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# ENGINEERING DATA TRANSMITTAL

Page 1 of 1

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# Tank Characterization Report for Single-Shell Tank 241-B-101

**Ruth D. Schreiber**
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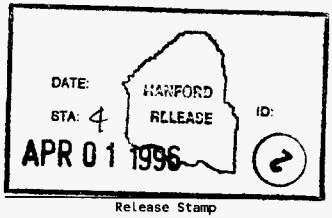
Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-B-101. This report supports the requirements of Tri-Party Agreement Milestone M-44-09.



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# Tank Characterization Report for Single-Shell Tank 241-B-101

R. D. Schreiber
J. G. Douglas
Westinghouse Hanford Company

T. T. Tran
Los Alamos Technical Associates, Inc.

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Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Westinghouse Hanford Company
P.O Box 1970
Richland, Washington

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## EXECUTIVE SUMMARY

This tank characterization report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in tank 241-B-101. This report supports requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994), Milestone M-44-09.

Tank 241-B-101 is a single-shell underground waste storage tank located in the 200 East Area B Tank Farm on the Hanford Site. The tank was placed in service in the second quarter of 1945 when it received metal waste from the bismuth phosphate process at B Plant (Agnew et al. 1995b). After being declared full in October of 1945, the tank was inactive, except for cascades to tank 241-B-102, until the first quarter of 1953 when it was sluiced. A small sludge heel was left after the sluicing activity (Agnew et al. 1995b). Evaporator bottoms waste was transferred to the tank from tank 241-B-105 during the fourth quarter of 1953 and the first quarter of 1954. During the third quarter of 1957, supernatant waste was transferred from tank 241-B-101 to tank 241-C-109 for ferrocyanide scavenging. Cladding waste from the Plutonium-Uranium Extraction (PUREX) Plant was transferred to tank 241-B-101 from tanks 241-C-103 and 241-C-106 during the third quarter of 1963, and from tank 241-C-102 during the third and fourth quarters of 1963. B Plant high-level supernatant waste was pumped through tank 241-B-101 from the second quarter of 1969 to the second quarter of 1970. B Plant low-level waste was transferred to tank 241-B-101 from the third quarter of 1970 until the first quarter of 1973. Liquid waste was intermittently removed from the tank throughout the 1969 to 1973 B Plant transfers. Tank 241-B-101 was removed from service in 1974 and was declared inactive in 1976 (Agnew et al. 1995b).

A description and status record of tank 241-B-101 are given in Tables ES-1 and ES-2 and Figure ES-1. The tank has an operating capacity of 2,010 kL (530 kgal), and presently contains 428 kL (113 kgal) of waste. While the *Waste Tank Summary Report for Month Ending November 30, 1995* (Hanlon 1996) states that this waste is entirely sludge, the Historical Tank Content Estimate (Brevick et al. 1994a) identifies the waste as both sludge and saltcake material, which is supported by the photographs taken of the core segments during extrusion. This report summarizes the collection and analysis of samples from one sampling event, which was performed to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). Two cores from opposite sides of the tank were obtained using the push-mode core sampling method. The cores were analyzed for their moisture content using thermogravimetric analysis (TGA), for their energetics content using differential scanning calorimetry (DSC), and for their total alpha activity using a fusion digestion and an alpha proportional counter. Further analyses are pending; a future revision to this characterization report will incorporate the additional data. A field measurement for tank headspace flammability was made during a separate sampling event.

Several samples exhibited percent water results below the limit listed in the tank safety screening DQO. However, the corresponding DSC results were well below the notification limit; consequently, the low percent water was not deemed a safety hazard because a low percent water content in itself is not considered to be an unsafe condition. Subsegments were reanalyzed using both the TGA method and alternative percent water methods (gravimetry) in an attempt to overcome inconsistencies in the results (Schreiber 1995c).

Table ES-1. Description and Status of Tank 241-B-101.

| | |
|---|---|
| **Tank Description** | |
| Type | Single-shell |
| Constructed | 1943-1944 |
| In-service | 1945 |
| Diameter | 22.9 m (75 ft) |
| Maximum operating depth | 5.18 m (17 ft) |
| Capacity | 2,010 kL (530 kgal) |
| Bottom shape | Dish |
| Ventilation | Passive |
| **Tank Status** | |
| Waste classification | Non-complexed |
| Total waste volume | 428 kL (113 kgal) |
| Solids volume | 428 kL (113 kgal) |
| Drainable interstitial liquid | 23 kL (6 kgal) |
| Supernatant volume | 0 |
| Waste surface level (February 23, 1996) | 86.4 cm (34 in.) |
| Temperature (January 9, 1996) | 41.8 °C (107.3 °F) to 30.9 °C (87.7 °F) |
| Integrity | Assumed leaker, 1974 |
| Watch List status | None |
| **Sampling Dates** | |
| Push-mode core sampled | June 1995 |
| Tank headspace flammability measurement | March 1996 |
| **Service Status** | |
| Out of service | 1974 |
| Interim stabilized | March 1981 |
| Intrusion prevention | May 1981 |

Table ES-2. Analytical Data Summary for Tank 241-B-101.

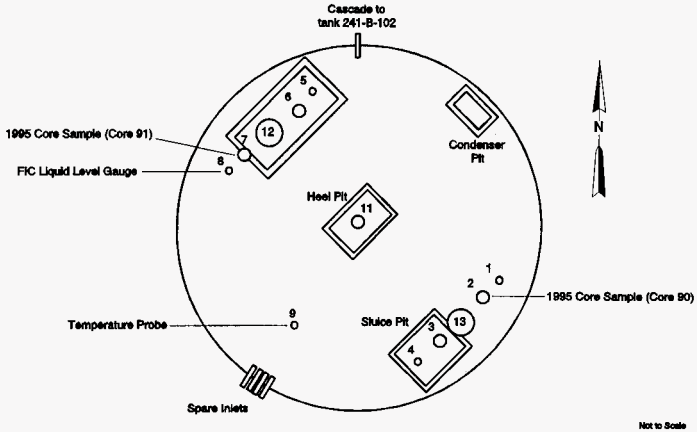
| Physical Properties | | Result | RSD (Mean) (%) |
|---|---|---|---|
| Density[1] | | 1.48 g/mL | N/A |
| Percent water by TGA[2] | | 32.5 | 11 |
| Percent water by Gravimetry[2] | | 27.1 | 19.7 |
| **Anions[2]** | **Concentration (μg/g)** | **RSD (Mean) (%)** | **Estimated Inventory (kg)** |
| $Cl^-$ | 556 | 27.5 | 352 |
| $F^-$ | 269 | 11.9 | 170 |
| $NO_2^-$ | 65,900 | 26.9 | 41,700 |
| $NO_3^-$ | 2.32E+05 | 14.8 | 1.47E+05 |
| $Oxalate^{2-}$ | < 1,620 | N/A | < 1,030 |
| $PO_4^{3-}$ | 5,820 | 21.4 | 3,690 |
| $SO_4^{2-}$ | 68,300 | 55 | 43,300 |
| **Radionuclides[2]** | **μCi/g** | **%** | **Ci** |
| Total Alpha | 2.91 | 70.1 | 1,840 |

Notes:

RSD (Mean) = relative standard deviation of the mean
N/A = not applicable

[1]Derived from the recovered segment masses and lengths as discussed in Section 4.2.
[2]Schreiber (1995c)

Figure ES-1. Profile of Tank 241-B-101.



The TGA percent water results for 8 of 32 individual samples measured were below 17 percent, the lowest value being 6.2 percent. Mean percent water content as measured by TGA was 32.5 percent, and by gravimetry was 27.1 percent. The accompanying uncertainties for these results, expressed as relative standard deviations of the mean, were 11 percent and 19.7 percent, respectively.

The subsegment whose exothermic reaction produced the highest result, -364 J/g (dry weight basis), came from the lower half solids of core 90, segment 1. The corresponding water content for this sample was 42 weight percent.

The mean total alpha activity was 2.91 $\mu$Ci/g, roughly one twelfth of the notification limit.

Tank headspace flammability as measured at a depth of 10 meters (33 feet) in riser 2 was 0 percent of the lower flammability limit.

Based on temperature surveillance data, tank 241-B-101 does not appear to have a heat-load issue. Recent surveillance data show a waste temperature range of 41.8 °C (107.3 °F) to 30.9 °C (87.7 °F) and a waste level of 86.4 cm (34 in.).

Four of a total of six drywells associated with the tank have had readings above the 50 counts per second background level (Brevick et al. 1994b). The highest reading was 1,928 counts per second in 1975. A tank leak is purported to be the cause of the high readings (Welty 1988).

## CONTENTS

**CONTENTS (Continued)**

## LIST OF FIGURES



## LIST OF TABLES

## LIST OF TABLES (Continued)

## LIST OF TERMS

| | |
|---|---|
| ANOVA | analysis of variance |
| B | B Plant high-level waste |
| BL | B Plant low-level waste |
| B SltCk | B Plant saltcake waste |
| cal/g | calories per gram |
| Ci/L | curies per liter |
| Ci | curies |
| cm | centimeter |
| CW | cladding waste |
| DBP | dibutyl phosphate |
| DQO | data quality objective |
| DSC | differential scanning calorimetry |
| EDTA | ethylenediaminetetraacetic acid |
| FIC | Food Instrument Corporation |
| ft | feet |
| g/mL | grams per milliliter |
| g | grams |
| g/L | grams per liter |
| HDW | Hanford Defined Wastes |
| HEDTA | N-(hydroxyethyl)-ethylenediaminetriacetic acid |
| HHF | hydrostatic head fluid |
| HTCE | Historical Tank Content Estimate |
| IC | ion chromatography |
| ICP | inductively coupled plasma spectroscopy |
| ID | identification |
| in. | inches |
| J/g | joules per gram |
| kg | kilograms |
| kgal | kilogallon |
| kL | kiloliter |
| LFL | lower flammability limit |
| m | meter |
| mg | milligram |
| mL | milliliter |
| mol/L | moles per liter |
| MW | metal waste |
| ND | not detected |
| NPH | normal paraffin hydrocarbons |
| NTA | nitrilotriacetate |
| ppm | parts per million |
| PUREX | Plutonium-Uranium Extraction Plant |
| QC | quality control |

## LIST OF TERMS (Continued)

| | |
|---|---|
| RSD | relative standard deviation |
| TCP | tank characterization plan |
| TGA | thermogravimetric analysis |
| TLM | tank layer model |
| WHC | Westinghouse Hanford Company |
| WSTRS | Waste Status and Transaction Record Summary |
| wt% | weight percent |
| °C | degrees Celsius |
| °F | degrees Fahrenheit |
| $\mu$Ci/g | microcuries per gram |
| $\mu$g/g | micrograms per gram |
| $\mu$m | micrometers |

# 1.0 INTRODUCTION

This tank characterization report presents an overview of single-shell tank 241-B-101 and its waste contents. It provides estimated concentrations and inventories for the waste components based on the latest sampling and analysis activities, in combination with background tank information. This characterization report presents the results of a core sampling event from June 1995 and, for informational purposes only, a historical sludge sampling event from 1976.

Tank 241-B-101 began operation in 1945 and received waste until it was declared inactive in 1974. Interim stabilization and intrusion prevention of the tank were completed in 1981; therefore, with the exception of drying and radioactive decay and barring an intrusion, the composition of the waste should not change until pretreatment and retrieval activities commence. The analyte concentrations reported in this document reflect the best composition estimates of the waste based on the available analytical data and historical models. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994) Milestone M-44-09.

## 1.1 PURPOSE

The purpose of this report is to summarize the information about the use and contents of tank 241-B-101. Where possible, this information will be used to assess issues associated with safety, operations, environmental, and process development activities. This report also serves as a reference point for more detailed information concerning tank 241-B-101.

## 1.2 SCOPE

The June 1995 core sampling event for tank 241-B-101 supported the evaluation of the tank waste according to the *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). From the two core samples, three primary analyses were performed as directed in the *Tank 241-B-101 Tank Characterization Plan* (Schreiber 1995d). These analyses were differential scanning calorimetry (DSC) to evaluate fuel level and energetics, thermogravimetric analysis (TGA) to determine moisture content, and total alpha activity analysis to evaluate criticality potential. Lithium concentration was also measured to check for sample contamination by the hydrostatic head fluid used during the push-mode core sampling process. The number of analyses was limited due to the narrow focus of the sampling event: verification of the non-Watch List status of the tank and/or identification of any unknown safety issues associated with the tank. More analyses are pending; the additional data will be included in a later revision of this report.

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# 2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-B-101 based on historical information. The first part of the section details the current condition of the tank. This is followed by discussions of the tank's background, transfer history, and the process sources that contributed to the tank waste, including an estimate of the current contents based on the process history. Events that may be related to tank safety issues, such as potentially hazardous tank contents or off-normal operating temperatures, are included. The final part of this section summarizes available surveillance data for the tank. Analytical and physical property data generated prior to May, 1989, and presented in this report are for information only; pre-May, 1989 data have not been validated and are not to be used for decision-making purposes.

## 2.1 TANK STATUS

As of November 30, 1995, tank 241-B-101 contained 428 kL (113 kgal) of waste classified as non-complexed (Hanlon 1996). Amounts of the various waste phases existing in the tank are presented in Table 2-1. Although saltcake is not identified in the tank contents status, the tank layer model (TLM) predicts 284 kL (75 kgal) of saltcake (Agnew et al. 1995a).

Table 2-1. Summary Tank Contents Status.[1]

| Waste Form | Volume | |
|---|---|---|
| | kL | kgal |
| Total waste | 428 | 113 |
| Supernatant liquid | 0 | 0 |
| Drainable interstitial liquid | 23 | 6 |
| Drainable liquid remaining | 23 | 6 |
| Pumpable liquid remaining | 0 | 0 |
| Sludge | 428 | 113 |
| Saltcake | 0 | 0 |

Note:
[1]Hanlon (1996)

Tank 241-B-101 was identified as an assumed leaker in 1974 with an estimated waste volume loss of 30 kL (8 kgal) (Welty 1988). The tank was removed from service in 1974 and declared inactive in 1976. Interim stabilization was completed in March 1981 and intrusion prevention was finished in May 1981. Tank 241-B-101 is not on any Watch Lists. All tank monitoring systems were in compliance with documented standards as of November 30, 1995.

## 2.2 TANK DESIGN AND BACKGROUND

This section summarizes the design and background of tank 241-B-101. Further detail can be found in the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994). The tank was constructed between 1943 and 1944. It is one of twelve 100 series tanks in the 241-B Tank Farm. The tank has a capacity of 2,010 kL (530 kgal), a diameter of 22.9 m (75 ft), and an operating depth of 5.18 m (17 ft). Tank 241-B-101 first went into operation in May 1945. First-generation tanks, like those in the B Tank Farm, were designed for non-boiling waste with a maximum fluid temperature of 104 °C (220 °F). The cascade overflow height is approximately 4.78 m (15.68 ft) from the tank bottom and 61 cm (24 in.) below the top of the steel liner.

Tank 241-B-101 was constructed with a primary mild steel liner and a concrete dome with various risers, similar to other single-shell tanks. It has a dished bottom with a 1.2-m (4-ft) radius knuckle. The tank is set on a reinforced concrete foundation, and is covered with approximately 2.2 m (7.25 ft) of overburden.

The surface level is monitored through riser 8 with a Food Instrument Corporation (FIC) gauge. Solid waste volume is also determined by the FIC gauge, while a photographic evaluation is used in determining liquid volume. The thermocouple tree is in riser 9. A plan view that depicts the riser configuration is shown in Figure 2-1, and risers and process nozzles are described in Table 2-2. A tank cross-section showing the approximate waste level and a schematic of the tank equipment is shown in Figure 2-2.

Figure 2-1. Riser Configuration for Tank 241-B-101.

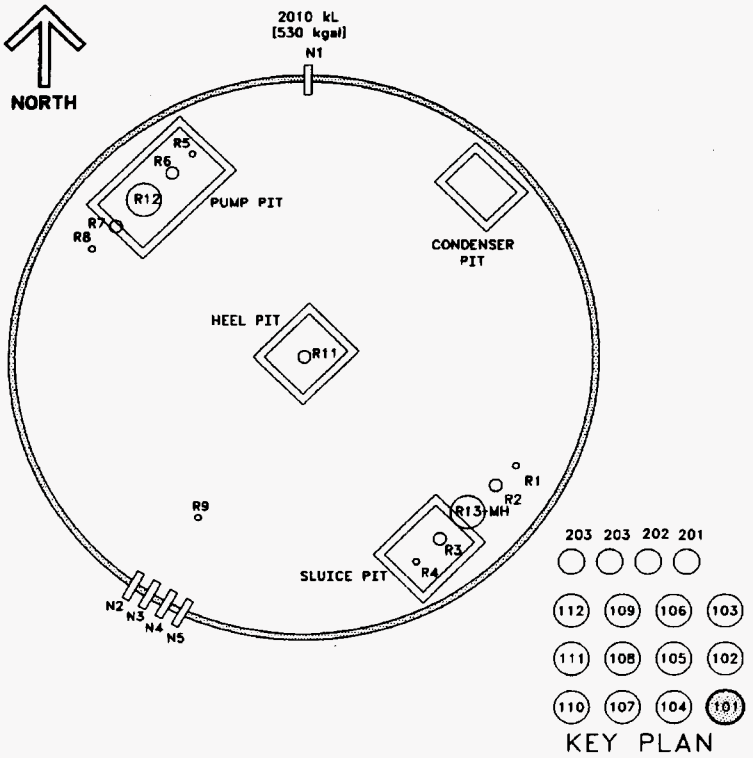


Table 2-2. Tank 241-B-101 Risers.[1]

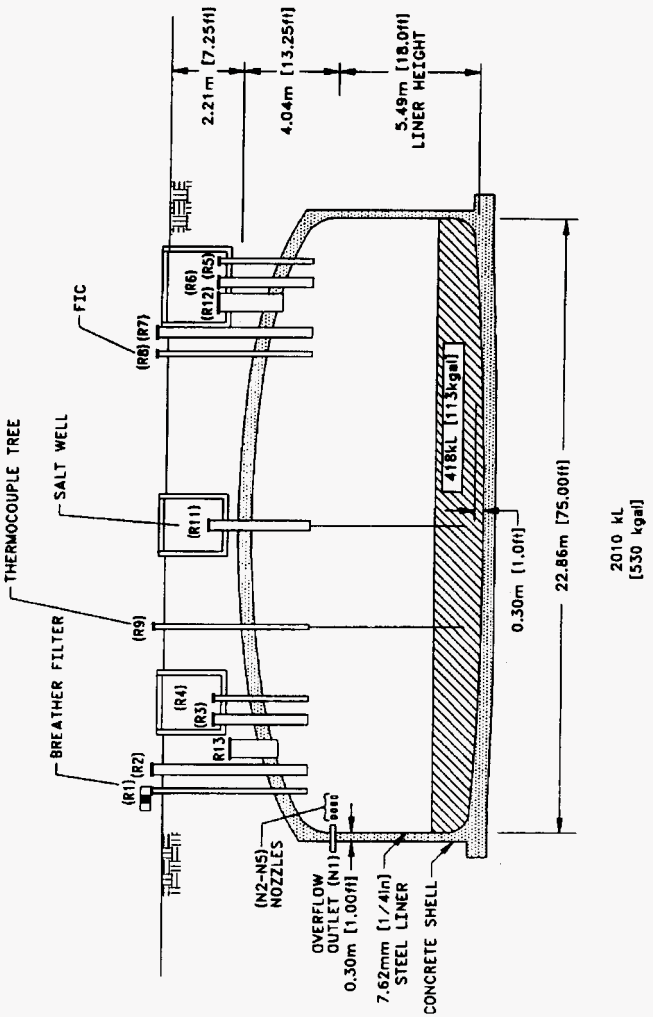
| Riser number | Diameter (inches) | Description and comments |
|---|---|---|
| 1 | 4 | Breather filter |
| 2[2] | 12 | Flange/B-222 observation port |
| 3 | 12 | Weather covered |
| 4 | 4 | Weather covered |
| 5 | 4 | Weather covered |
| 6 | 12 | Weather covered |
| 7[2] | 12 | Weather covered |
| 8 | 4 | Food Instrument Corporation gauge (bench mark) |
| 9 | 4 | Thermocouple tree (bench mark) |
| 11 | 12 | Weather covered (saltwell screen & pump) |
| 12 | 42 | Weather covered |
| 13 | 42 | Below grade (manhole cover) |
| **Nozzle number** | **Diameter (inches)** | **Description and comments** |
| N1 | 3 | Cascade outlet |
| N2 | 3 | Process inlet nozzles |
| N3 | 3 | Process inlet nozzles |
| N4 | 3 | Process inlet nozzles |
| N5 | 3 | Process inlet nozzles |

Notes:

[1]Alstad (1993); Brevick et al. (1994b)

[2]Risers available for sampling.

Figure 2-2. Tank 241-B-101 Configuration.



## 2.3 PROCESS KNOWLEDGE

This section presents the transfer history of tank 241-B-101 and describes the process wastes that made up these transfers. This is followed by an estimate of the current tank contents based on transfer history.

### 2.3.1 Waste Transfer History

From the second quarter of 1945 until the first quarter of 1946, metal waste (MW) from the bismuth phosphate process (in B Plant) was directed to tank 241-B-101 (Agnew et al. 1995b). In October 1945, the tank was declared full and there was little activity until the first quarter of 1953 (transfers from October 1945 through the first quarter of 1946 resulted in cascading of waste to tank 241-B-102). In the first quarter of 1953, the contents of the tank were sluiced, leaving a small sludge heel of MW (Agnew et al. 1995b). During the fourth quarter of 1953 and the first quarter of 1954, tank 241-B-101 received evaporator bottoms waste from tank 241-B-105; however, no solid waste measurements were recorded until the second quarter of 1957. During the third quarter in 1957, supernatant waste in tank 241-B-101 was transferred to tank 241-C-109 for ferrocyanide scavenging. In mid-1957, the solid waste volume in tank 241-B-101 was recorded at 1,190 kL (315 kgal).

During the third quarter of 1963, tank 241-B-101 received PUREX cladding waste (CW) from tanks 241-C-103 and 241-C-106. During the third and fourth quarters of 1963, tank 241-B-101 received CW from tank 241-C-102. Just prior to these transfers, a noticeable drop in solids measured was observed, from 1,192 kL (315 kgal) to 765 kL (202 kgal). A second drop was noticed in 1965 when the solids level fell to 609 kL (161 kgal). This drop is presently attributed to dissolution of saltcake. In the first quarter of 1969, a large transfer of supernatant occurred to tank 241-BY-103 and another decrease in solids was measured. From the second quarter of 1969 to the second quarter of 1970, B Plant high-level supernatant waste was sent to tank 241-B-101 with intermittent transfers to tanks 241-B-102, 241-BX-103 and 241-BX-101. According to the tank layer model (Agnew et al. 1995a), at this time there was a fourth measurable drop in solids, again most likely due to dissolution. From the third quarter of 1970 to the first quarter of 1973, additions of B Plant low-level waste into tank 241-B-101 occurred, as well as transfers out of tank 241-B-101 to tanks 241-B-102, 241-BX-101, and 241-BX-104.

Tank 241-B-101 was declared an assumed leaker in 1974 (Hanlon 1996). During the second quarter of 1974, a transfer of supernatant from the tank to tank 241-BX-106 took place. Tank 241-B-101 was removed from service during the fourth quarter of 1974, and two final transfers of supernatant were made to tank 241-BX-103 between this time and the first quarter of 1975. In 1976, tank 241-B-101 was declared inactive.

The transfer history of tank 241-B-101 is summarized in Table 2-3, and the tank level history is shown in Figure 2-3.

Table 2-3. Summary of Tank 241-B-101 Waste Transfer History.[1]

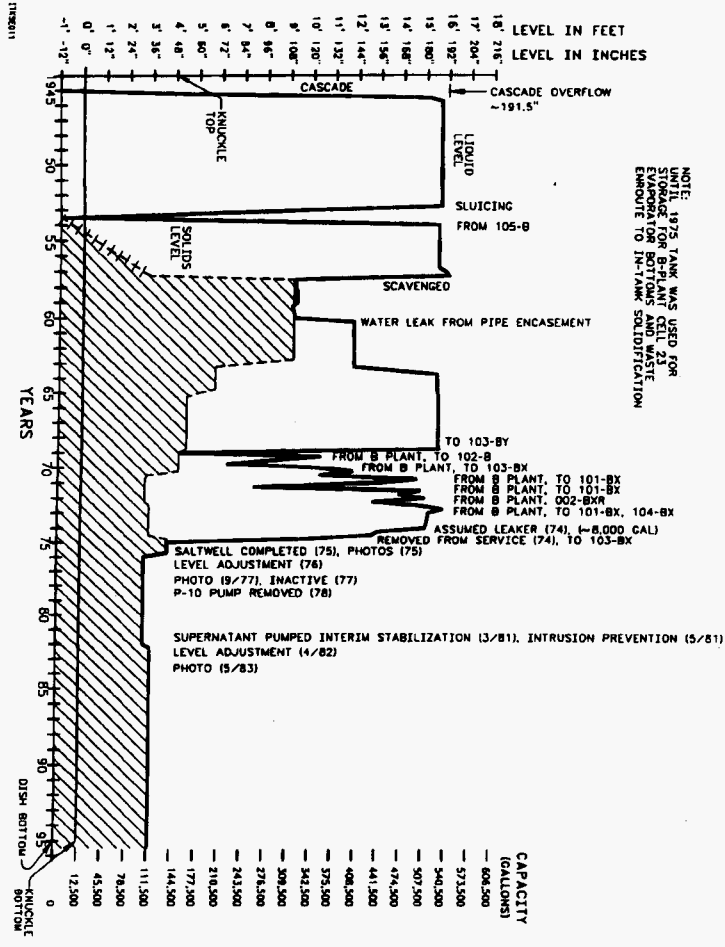
| Transfer source | Waste type received | Time period | Solid waste volume[2] kL (kgal) | Comments |
|---|---|---|---|---|
| B Plant | Metal waste | 1945 to 1946 | 2,010 (530) | Tank full, cascading to 241-B-102. |
| | | 1953 | 0 | Tank sluiced and declared empty, although small heel of metal waste remained. |
| 241-B-105 | Evaporator bottoms | 1953 to 1954 | 1,995 (527) | Saltcake added on top of metal waste heel. |
| | | 1957 | 1,190 (315) | Solids recorded. Supernatant waste removed for ferrocyanide scavenging, solid waste remaining. |
| Various C Farm Tanks | Supernatant Cladding waste | 1963 to 1965 | 609 (161) | Supernatant cladding waste, added in 1963, decrease in solids attributed to saltcake solids dissolution. Second solids decrease in 1965. |
| B Plant | B Plant high-level supernatant wastes | 1969 to 1970 | 572 (151) | Solids measurement decrease attributed to saltcake solids dissolution. Multiple transfers to tanks 241-BY-103, 241-B-102, 241-BX-101 and 241-BX-103. |
| B Plant | B Plant low-level wastes | 1970 to 1973 | 413 (109) | Sludge deposition as well as saltcake solids dissolution. Multiple transfers out to tanks 241-B-102, 241-BX-101 and 241-BX-104. |

Notes:

[1]Agnew et al. (1995b)

[2]Solids not recorded until 1957; pre-1957 volumes are total for tank 241-B-101.

Figure 2-3. Tank Level History.



### 2.3.2 Historical Estimation of Tank Contents

An estimate of the current contents of tank 241-B-101 based on historical transfer data is available from the Historical Tank Content Estimate (HTCE) (Brevick et al. 1994a). The historical data used for the HTCE prediction include the Waste Status and Transaction Record Summary (WSTRS) (Agnew et al. 1995b), the Hanford Defined Wastes (HDW) (Agnew 1995), and the tank layer model (TLM) (Agnew et al. 1995a). The WSTRS is a compilation of available waste transfer and volume status data. The HDW provides the assumed typical compositions for Hanford Site waste types. In some cases, the available data are incomplete, reducing the usability of the transfer data and the modeling results derived from it. The TLM takes the WSTRS data, models the waste deposition processes, and, using additional data from the HDW (which may introduce additional error), generates an estimate of the tank contents. Thus, these model predictions can only be considered an estimate that requires further evaluation using analytical data.

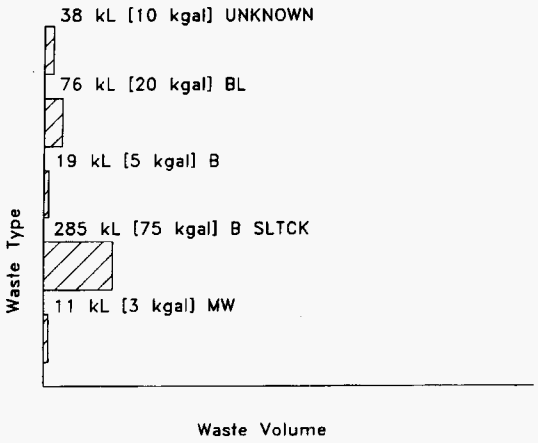
Figure 2-4 shows a graphical representation of the estimated waste type and volumes for the tank layers. As stated previously, the bottom layer has been estimated to be metal waste (MW). It should contain large amounts of sodium, uranium, carbonate, phosphate, sulfate, hydroxide, and a trace amount of plutonium. Also present will be quantities of strontium and cesium (of which the amount of strontium is significantly larger than the amount of cesium); therefore, this layer will have a slight activity. To further identify MW, certain constituents should be totally or relatively absent from the waste. These constituents include but are not limited to: aluminum, iron, bismuth, nickel, lead, and total organic carbon.

The B Plant saltcake (B SltCk) layer should contain large amounts of sodium, nitrate, nitrite, fluoride, sulfate, phosphate, carbonate, and hydroxide. Also present will be aluminum, iron, bismuth, zirconium, and a trace amount of plutonium. Cesium and strontium quantities will be low; therefore, the activity should be low.

The next layer is B Plant high-level waste (B waste). This waste type contains very large amounts of iron and hydroxide, large amounts of sodium, aluminum, nickel, nitrate, and sulfate, smaller amounts of chromium, nitrite, and total organic carbon, and a trace amount of lead. Also, the waste should contain extremely large quantities of strontium (the strontium concentration is approximately 149 times the cesium concentration) and large quantities of cesium. Therefore, the B waste layer has a very large activity associated with it. Some plutonium should also be present in this layer, but at levels significantly less than those of cesium and strontium. This layer of B waste can be distinguished from the MW and B SltCk waste types by the presence of total organic carbon, nickel, lead, and a very large activity. Also, B waste does not contain bismuth and zirconium (both are found in B SltCk, but not in MW).

The waste layer located above the B waste layer is defined as B Plant low-level waste (BL waste). This BL waste will contain large amounts of uranium, hydroxide, sodium, aluminum, iron, silicate, and nitrate. Quantities of nickel, carbonate, total organic carbon, and a small amount of plutonium also will be found. Cesium is not present, but a large concentration of strontium will exist. Therefore, there will be a notable activity associated with this layer. The BL waste layer can be distinguished from the MW, B SltCk, and B waste layers by the presence of total organic carbon (not found in MW or B SltCk waste, but found in a much smaller quantity in B waste) and the absence of zirconium (found only in B SltCk) and cesium. Another distinguishing factor of this waste type is the activity, which will be significantly higher than the MW and B SltCk waste layers, but lower than the B waste layer. The unknown waste layer constituents are, of course, undefined; however, Agnew et al. (1995a) suggests this waste layer may be BL waste. Table 2-4 shows the historical estimate of the expected waste constituents and their concentrations.

Figure 2-4. Tank Layer Model.




BL = B Plant low-level waste
B = B Plant high-level waste
B SLTCK = B Plant Saltcake
MW = metal waste

Table 2-4. Tank 241-B-101 Historical Tank Content Estimate.[1] (2 sheets)

| Solids Composite Inventory Estimate[2] | | | |
|---|---|---|---|
| **Chemical Constituents** | | | |
| **Analyte** | **mol/L** | **ppm (μg/g)** | **kg** |
| $Na^{+}$ | 8.15 | 1.13E+05 | 80,100 |
| $Al^{3+}$ | 0.609 | 9,910 | 7,030 |
| $Fe^{3+}$ (total Fe) | 0.790 | 26,600 | 18,900 |
| $Cr^{3+}$ | 0.0084 | 264 | 187 |
| $Bi^{3+}$ | 0.00407 | 514 | 364 |
| $La^{3+}$ | 0 | 0 | 0 |
| $Ce^{3+}$ | 0 | 0 | 0 |
| Zr (as $ZrO(OH)_2$) | 0.00815 | 448 | 318 |
| $Pb^{2+}$ | 3.08E-08 | 0.00385 | 0.00273 |
| $Ni^{2+}$ | 0.103 | 3,650 | 2,590 |
| $Sr^{2+}$ | 0 | 0 | 0 |
| $Mn^{4+}$ | 0 | 0 | 0 |
| $Ca^{2+}$ | 0.190 | 4,590 | 3,250 |
| $K^{+}$ | 0 | 0 | 0 |
| $OH^{-}$ | 6.92 | 71,000 | 50,300 |
| $NO_3^{-}$ | 2.33 | 87,200 | 61,800 |
| $NO_2^{-}$ | 0.129 | 3,590 | 2,540 |
| $CO_3^{2-}$ | 0.317 | 11,500 | 8,130 |
| $PO_4^{3-}$ | 0.974 | 55,800 | 39,600 |
| $SO_4^{2-}$ | 0.467 | 27,000 | 19,200 |
| Si (as $SiO_3^{2-}$) | 0.378 | 6,410 | 4,540 |
| $F^{-}$ | 0.543 | 6,230 | 4,410 |
| $Cl^{-}$ | 0.0554 | 1,180 | 840 |
| $Citrate^{3-}$ | 0.00352 | 401 | 285 |
| $EDTA^{4-}$ | 0 | 0 | 0 |
| $HEDTA^{3-}$ | 0 | 0 | 0 |
| $NTA^{3-}$ | 0 | 0 | 0 |
| $Glycolate^{-}$ | 0.0428 | 1,940 | 1,370 |
| $Acetate^{-}$ | 0 | 0 | 0 |
| $Oxalate^{2-}$ | 0 | 0 | 0 |

Table 2-4. Tank 241-B-101 Historical Tank Content Estimate.[1] (2 sheets)

| Solids Composite Inventory Estimate[2] | | | |
|---|---|---|---|
| **Chemical Constituents (Continued)** | | | |
| **Analyte** | **mol/L** | **ppm (μg/g)** | **kg** |
| DBP | 0 | 0 | 0 |
| NPH | 0 | 0 | 0 |
| $CCl_4$ | 0 | 0 | 0 |
| Hexone | 0 | 0 | 0 |
| $Fe(CN)_6^{4-}$ | 0 | 0 | 0 |
| **Radiological Constituents** | | | |
| **Analyte** | **Ci/L** | **μCi/g** | **Ci** |
| Pu | ——— | 0.782 | 9.24 (kg) |
| U | 0.380 (mol/L) | 54,600 (μg/g) | 38,700 (kg) |
| Cs | 0.0218 | 13.2 | 9,330 |
| Sr | 4.71 | 2,840 | 2.02E+06 |
| **Physical Properties** | | | |
| Total solid waste | 7.09E+05 kg (113 kgal) | | |
| Heat load | 13,600 watts (46,500 Btu/hr) | | |
| Bulk density | 1.66 grams per cubic centimeter | | |
| Void fraction | 0.666 | | |
| Water wt% | 54 | | |
| Total organic carbon wt% C (wet) | 0.065 | | |

Notes:

[1]Brevick et al. (1994a); data in this table are presented for information only and are not to be used for decision-making purposes.

[2]The composite inventory excludes supernatant. Unknowns in the tank inventory are assigned by the tank layer model (Agnew et al. 1995a).

## 2.4 SURVEILLANCE DATA

Tank 241-B-101 surveillance consists of surface level measurements (liquid and solid), temperature monitoring inside the tank (waste and headspace), and leak detection well (drywell) monitoring for radioactivity outside the tank. The data are significant because they provide the basis for determining tank integrity.

Liquid level measurements are used to determine if there may be a major leak from or intrusion into the tank. In-tank photography is another waste volume determination method used to resolve measurement anomalies and determine tank integrity. Solid surface level measurements provide an indication of physical changes and consistency of the solid layers of a tank. Drywells located around the perimeter of the tank may detect increased radioactivity if there is a leak to the soil.

### 2.4.1 Surface Level Readings

Tank 241-B-101 surface level is monitored with an FIC gauge through riser 8. The gauge is set in the intrusion mode for a 2.5-cm (1-in.) increase. Data are not available for a surface level plot due to the FIC gauge setting. The latest surface level measurement, taken on February 23, 1996, was 86.4 cm (34 in.) as measured from the tank centerline.

### 2.4.2 Internal Tank Temperatures

Tank 241-B-101 has a single thermocouple tree with 17 thermocouples to monitor the waste temperature through riser 9. Thermocouple 1 is 40.5 cm (16 in.) from the bottom of the tank centerline. Thermocouples 2 though 12 are located at 61-cm (24-in.) intervals above thermocouple 1. Elevations for thermocouples 13 through 17 are unavailable (Tran 1993). Considering the position of thermocouple 1 and the depth of the waste, thermocouple 1 is definitely in contact with the tank waste. However, the thermocouple data suggest that thermocouple 2 may also be in contact with the tank waste. Thermocouple 2 is at an elevation of 102 cm (40 in.) from the tank bottom; the most recent surface level reading was 86.4 cm (34 in.). If the surface of the waste is sufficiently uneven, it is entirely possible that thermocouple 2 is indeed in contact with the waste. The remaining thermocouples are in the headspace.

Internal tank temperature data, from the date thermocouples began recording until 1974, are sporadic. From May 1974 to 1993, thermocouples 1 through 11 have similar readings. Data spanning from 1974 to 1987 for thermocouples 12 through 14 show trends similar to the first 11 thermocouples, and data for thermocouples 15 and 16 are available from 1984 to 1986. Only one data point is available for thermocouple 17 (Brevick et al. 1994b).

The average temperature from the first recorded data for thermocouples 1 through 14 is 47 °C (117 °F). Thermocouples 1 and 2 have recorded temperatures about 8 °C (15 °F) to 11 °C (20 °F) higher than the other thermocouples for May 1974 to the present. From May 1974 to the present, the median temperature is 35 °C (95 °F), the minimum temperature is 4 °C (40 °F), and the maximum temperature is 58 °C (137 °F). The most recent waste temperature readings, taken on January 9, 1996, ranged from 41.8 °C (107.3 °F) (thermocouple 1) to 30.9 °C (87.7 °F) (thermocouple 7). Tank 241-B-101 is a low-heat-load, non-Watch List tank, and is scheduled to be monitored semi-annually in January and July. Plots of the individual thermocouple readings can be found in Brevick et al. (1994b). A graph of the semi-annual high temperatures (from the thermocouple with the highest reading) can be found in Figure 2-5.

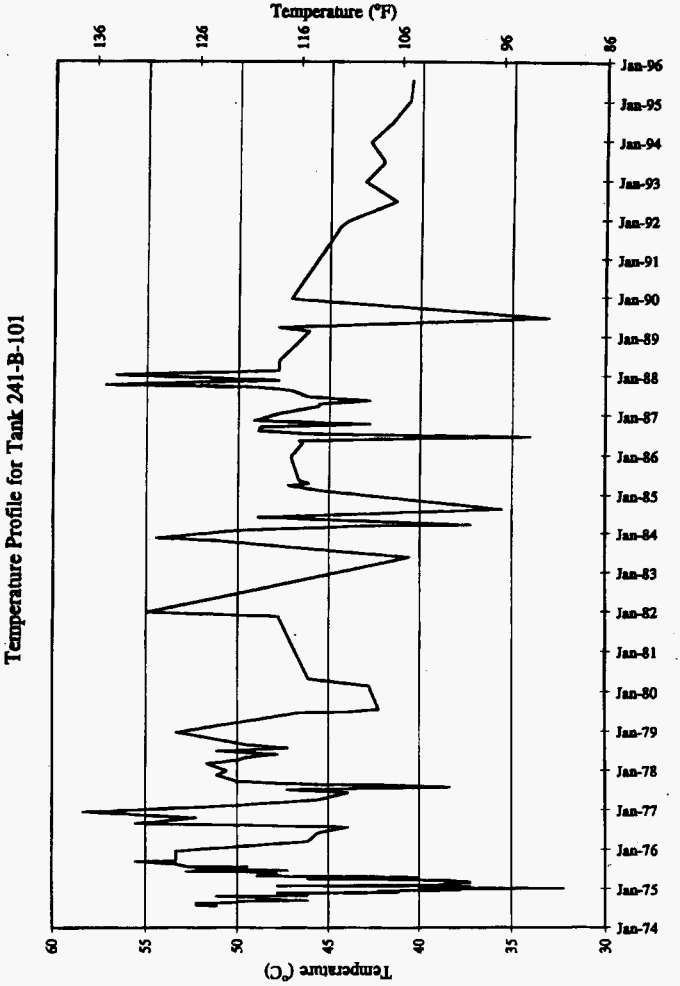
### 2.4.3 Drywells

Six drywells are associated with tank 241-B-101 (Brevick et al. 1994b). Four drywells, no longer active, have had readings above the 50 counts-per-second background radiation level. Of these, drywell 20-01-01 had the highest readings in 1975 at 1,928 counts per second. The radioactivity is attributed to a tank leak (Welty 1988). Plotted readings from drywells are available in Brevick et al. (1994b).

### 2.4.4 Tank 241-B-101 Photographs

Photographs taken on May 19, 1983 indicate a dark, rough surface with no visible liquid (Welty 1988). While most of the photographs for tank 241-B-101 lack clarity to allow interpretation of all the interior details, the photographs appear to represent the current tank contents.

Figure 2-5. Tank 241-B-101 High Temperature Plot.



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# 3.0 TANK SAMPLING OVERVIEW

This section describes the June 1995 sampling and analysis event for tank 241-B-101. Push-mode core samples were taken to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). The sampling and analyses were performed in accordance with the *Tank 241-B-101 Tank Characterization Plan* (Schreiber 1995d). Further discussions of the sampling and analysis procedures can be found in the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

## 3.1 DESCRIPTION OF SAMPLING EVENT

Two push-mode core samples were collected from tank 241-B-101 between June 19 and June 23, 1995. The first core sample was collected from riser 2 on June 19, 1995 and received by the 222-S Laboratory on June 21, 1995. The second core sample was obtained from riser 7 on June 23, 1995 and received by the 222-S Laboratory on June 26, 1995.

The push-mode core sampling method was chosen based on surface photographs taken of the waste. Although rotary-mode core sampling could have been performed, it would have been substantially more expensive and was not expected to yield better results. Auger sampling was not appropriate because the depth of the waste would not have allowed the DQO-required full vertical profile to be obtained. The safety screening analyses performed on the tank 241-B-101 samples included: total alpha activity to determine the potential for a criticality event; DSC to ascertain the fuel energy value; and TGA to obtain the moisture content of the waste material.

The tank safety screening DQO also requires a measurement of tank headspace vapor flammability (Babad et al. 1995). On March 26, 1996, a field measurement was performed to determine tank headspace vapor flammability. The measurement was performed using a combustible gas meter while sampling at a depth of 10 m (33 ft) through riser number 2 (WHC 1996). Table 3-1 summarizes the sampling and analytical requirements from the safety screening DQO.

Table 3-1. Integrated Data Quality Objective Requirements for Tank 241-B-101.[1]

| Sampling event | Applicable DQOs | Sampling requirements | Analytical requirements |
|---|---|---|---|
| June, 1995 push-mode core sampling | Safety screening | Core samples from a minimum of two risers separated radially to the maximum extent possible. | ▸Energetics<br>▸Moisture content<br>▸Total alpha activity |
| March, 1996 tank headspace sampling | Safety screening | Tank headspace vapor sample below the riser | ▸Flammable gas concentration |

Note:
[1]Schreiber (1995d)

## 3.2 SAMPLE HANDLING

The riser 2 core sample, identified as core 90, was extruded by the 222-S Laboratory on June 23, 1995. The sample was composed of two separate segments that were labeled with distinct identification numbers. Segment 1 was identified as sample 95-099, and segment 2 was given sample number 95-100. The riser 7 sample was extruded by the 222-S Laboratory on June 28, 1995. This sample, designated core 91, was also comprised of two separate segments. These segments were labeled as sample 95-101 (segment 1) and sample 95-102 (segment 2). Appendix A shows extrusion photographs of segments one and two of cores 90 and 91. Table 3-2 gives the subsampling scheme and sample description. Table 3-3 provides the samples and the analyses performed, and Table 3-4 identifies the procedures used. More analyses are pending, and the results will be included in a future revision to this report.

Sample 95-099 contained 388.3 g of solids and no drainable liquid. Less than 5 mL of liner liquid was also observed; however, because this amount of sample is insufficient for subsampling and analysis, it was not retained. The extruded sample was 41 cm (16 in.) in length and appeared as a smooth, damp solid that was able to retain its shape. A gap was observed in the upper portion of the segment in which no material was obtained. The top 32 cm (12.5 in.) of sample, which was dark brown in color, was subdivided into an upper half-segment weighing 150.4 g and a lower half-segment weighing 168.1 g. The bottom 9 cm (3.5 in.) of material weighed 69.8 g, appeared lighter brown in color, and was analyzed as a separate facies.

Sample 95-100 had an extruded length of approximately 20 cm (8 in.) and contained 195 g of solids. Approximately 50 mL (74.2 g) of opaque, brown drainable liquid was recovered. The solid material was divided into half-segments in accordance with the tank characterization plan (TCP). The upper half-segment appeared as a smooth, damp, light to medium brown sludge weighing 89.2 g. The lower half-segment contained white crystalline flakes and weighed 105.6 g. Subsamples for the upper half-segment and the decanted drainable liquid were submitted to the laboratory for analysis. At the request of the safety program, the lower half-segment was not initially analyzed due to uncertainty regarding the laboratory homogenization capabilities (see Attachment 1 of Schreiber [1995c]). However, the large size of the subsample made it logical to proceed with the analyses, and this half-segment was later analyzed and the results reported in Schreiber (1995b).

Sample 95-101 contained 393.6 g of solids and no drainable liquid. As with sample 95-099, less than 5 mL of liner liquid was observed but was not retained for analysis. The extruded solid sample was 40 cm (16 in.) in length, appeared smooth, and varied in color from medium to dark brown. There was an 8-cm (3-in.) gap in the upper portion of the sample in which no material was obtained. The segment was subdivided into half-segments at the point of the color change. Specifically, the upper 30 cm (12 in.) of sample was designated as the upper half-segment. This half-segment appeared as a medium brown sludge and weighed 291.3 g. Within this upper half-segment, a piece of flat, hard material was discovered that was approximately 1 cm (1/2-in.) long and had a mass of 2.4 g. This item was segregated from the rest of the sample and archived as sample S95T001209. The bottom 10 cm (4 in.) of sample material was designated to be the lower half-segment. This lower half-segment weighed 99.9 g and appeared dark brown in color.

Sample 95-102 produced a 30-cm (12-in.) solid portion weighing 271.1 g, and approximately 30 mL of opaque, dark brown drainable liquid weighing 47.1 g. As with the previous samples, less than 5 mL of liner liquid was recovered but not analyzed. The upper 15 cm (6 in.) of the solid portion weighed 152.2 g and appeared as dark brown soft sludge. This portion of the sample was labeled as the upper half-segment. The next 5 cm (2 in.) of sample resembled a light brown sludge and weighed 46 g. This portion of sample was identified as the lower half-segment. The bottom 10 cm (4 in.) of sample (72.9 g) was light brown in color, crystalline, and brittle. This portion of waste, identified as a separate facies, was initially archived, like the lower half of sample 95-100, due to the homogenization uncertainty in the laboratory. However, the relatively large size of the subsample made it logical to proceed with the analyses, and the sample was later evaluated and the results reported in Schreiber (1995b). If homogenization issues are resolved in the future, sufficient quantity of this sample will be archived for the analyses to be repeated.

In addition to the two segment samples, a field blank was also obtained from core 91. The field blank was extruded on June 28, 1995 and appeared as a clear, colorless liquid weighing 270.6 g. Less than 5 mL of liquid from the liner was acquired but not retained for analysis. As requested by the TCP, a subsample of the field blank material was submitted to the laboratory for analysis (percent water, DSC, and lithium only), and an archive sample was retained in the hot cell.

Hydrostatic head fluid (HHF) was used during the sampling process, which may have biased some results. To determine the extent of possible sample contamination, the samples were analyzed by inductively coupled plasma spectroscopy (ICP). This analysis is in addition to the safety screening analyses and is used to determine the presence of lithium, the metallic component of the salt lithium bromide, which is used as a tracer element in the HHF.

## 3.3 DESCRIPTION OF HISTORICAL SAMPLING EVENT

Results from historical sampling events are used to corroborate the recent analytical results. Because tank 241-B-101 was actively receiving waste until it was removed from service in 1974, sampling events prior to this date are no longer representative of the current tank contents. Only one set of pertinent historical sampling and analysis data has been identified for this tank. This sample was received on January 5, 1976 and the analytical results were released on February 24, 1976. The sample was noted as being soft, dark brown sludge. Documentation describing the sampling location (riser number and sample depth) and the sampling method used was not available. The sample handling procedure was described in Horton (1976). The sample was prepared for analysis by fusing 1.2 mL of damp sludge with potassium hydroxide, dissolving the melt in concentrated hydrochloric acid, and diluting to 250 mL with water. The analytical results are tabulated in Appendix B, and comparisons with the 1995 core sampling results are made in Section 5.2. Because the sampling location of the 1976 sample is unknown, the comparison between the 1976 sample and the 1995 sample is for information only; no conclusions should be inferred from the comparison.

Table 3-2. Tank 241-B-101 Subsampling Scheme and Sample Description.[1]

| Core | Riser | Segment (sample ID) | Segment portion | Sample length | Weight (g) | Sample characteristics |
|---|---|---|---|---|---|---|
| 90 | 2 | 1(95-099) | upper half | 32 cm (12.5 in.) | 150.4 | dark brown sludge, smooth, damp, and retains shape |
| | | | lower half | | 168.1 | |
| | | | facies[2] | 9 cm (3.5 in.) | 69.8 | lighter brown sludge, smooth, damp, and retains shape |
| | | 2(95-100) | upper half | 20 cm (8 in.) | 89.2 | smooth, damp, light to medium brown sludge |
| | | | lower half | | 105.6 | white crystalline flakes |
| | | | drainable liquid | --- | 74.2 | opaque, brown |
| 91 | 7 | 1(95-101) | upper half | 30 cm (12 in.) | 291.3 | medium brown sludge |
| | | | lower half | 10 cm (4 in.) | 99.9 | dark brown sludge |
| | | | object | 1 cm (0.5 in.) | 2.4 | flat, hard (archived) |
| | | 2(95-102) | upper half | 15 cm (6 in.) | 152.2 | dark brown, soft sludge |
| | | | lower half | 5 cm (2 in.) | 46 | light brown sludge |
| | | | facies[3] | 10 cm (4 in.) | 72.9 | light brown, crystalline, brittle |
| | | | drainable liquid | --- | 47.1 | opaque, dark brown |
| | | field blank | --- | --- | 270.6 | clear, colorless liquid |

Notes:

[1]Schreiber (1995a)

[2]Represents the bottom 9 cm (3.5 in.) of core 90, segment 1. It was labeled as a separate facies because of its distinct color relative to the remaining segment portion.

[3]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies due to its distinct color and texture.

Table 3-3. Tank 241-B-101 Sample Analysis Summary.[1] (2 sheets)

| Core | Riser | Segment (sample ID) | Segment portion | Sample number | Analyses |
|---|---|---|---|---|---|
| 90 | 2 | 1(95-099) | upper half | S95T001214 | TGA, DSC |
| | | | | S95T001215 | Total Alpha, ICP |
| | | | lower half | S95T001217 | TGA, DSC |
| | | | | S95T001218 | Total Alpha, ICP |
| | | | facies[2] | S95T001220 | TGA, DSC |
| | | | | S95T001221 | Total Alpha, ICP |
| | | | | S95T001541 | Gravimetry |
| | | 2(95-100) | upper half | S95T001229 | TGA, DSC |
| | | | | S95T001230 | Total Alpha, ICP |
| | | | | S95T001543 | Gravimetry |
| | | | | S95T001544 | IC |
| | | | | S95T002532 | Gravimetry, TGA |
| | | | lower half | S95T001551 | TGA, DSC |
| | | | | S95T001552 | Total Alpha, ICP |
| | | | | S95T001553 | IC |
| | | | | S95T002533 | Gravimetry, TGA |
| | | | drainable liquid | S95T001223 | TGA, DSC |
| | | | | S95T001224 | ICP |
| | | | | S95T001542 | IC |
| 91 | 7 | 1(95-101) | upper half | S95T001238 | TGA, DSC |
| | | | | S95T001336 | Total Alpha, ICP |
| | | | lower half | S95T001235 | TGA, DSC |
| | | | | S95T001236 | Total Alpha, ICP |
| | | | | S95T001546 | IC |

Table 3-3. Tank 241-B-101 Sample Analysis Summary.[1] (2 sheets)

| Core | Riser | Segment (sample ID) | Segment portion | Sample number | Analyses |
|---|---|---|---|---|---|
| 91 | 7 | 2(95-102) | upper half | S95T001244 | TGA, DSC |
| | | | | S95T001245 | Total Alpha, ICP |
| | | | | S95T001549 | IC |
| | | | lower half | S95T001241 | TGA, DSC |
| | | | | S95T001337 | Total Alpha, ICP |
| | | | | S95T001547 | Gravimetry |
| | | | facies[3] | S95T001555 | TGA, DSC |
| | | | | S95T001556 | Total Alpha, ICP |
| | | | | S95T001557 | IC |
| | | | | S95T002534 | Gravimetry, TGA |
| | | | drainable liquid | S95T001247 | TGA, DSC |
| | | | | S95T001248 | ICP |
| | | | | S95T001550 | IC |
| | | field blank | --- | S95T001232 | TGA, DSC |
| | | | | S95T001233 | ICP |

Notes:

[1]Schreiber (1995c)

[2]Represents the bottom 9 cm (3.5 in.) of core 90 segment 1, and was labeled as a separate facies because of its distinct color relative to the remaining segment portion.

[3]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies due to its distinct color and texture.

Table 3-4. Analytical Procedures.[1]

| Analysis | Instrument | Preparation procedure | Procedure number |
|---|---|---|---|
| Energetics by DSC | Mettler[2]<br>Perkin-Elmer[3] | N/A | LA-514-113, Rev. B-1<br>LA-514-114, Revs. B-0 & C-0 |
| Percent water by TGA | Mettler™<br>Perkin-Elmer™ | N/A | LA-560-112, Revs. A-2 & B-0<br>LA-514-114, Revs. B-0 & C-0 |
| Total alpha activity | Alpha proportional counter | LA-549-141, Rev. D-0 | LA-508-101, Rev. D-2 |
| Lithium by ICP | Inductively coupled plasma spectrometer | LA-549-141, Rev. D-0<br>LA-505-158, Rev. A-4 | LA-505-161, Rev. A-1<br>LA-505-151, Rev. D-3 |
| Bromide by IC | Ion chromatography | LA-504-101, Rev. D-0 | LA-533-105, Rev. D-0 |
| Percent water by gravimetry | N/A | N/A | LA-564-101, Rev. F-1 |

Notes:

N/A = not applicable
Rev. = revision

[1]Schreiber (1995c)

[2]Mettler™ is registered trademark of Mettler Electronics, Anaheim, California.

[3]Perkin-Elmer™ is a registered trademark of Perkins Research and Manufacturing Company, Inc., Canoga Park, California.

# 4.0 ANALYTICAL RESULTS

## 4.1 OVERVIEW

This section presents the analytical results associated with the June 1995 sampling of tank 241-B-101. The sampling and analyses were performed for evaluation of safety screening criteria defined in *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). The DQO stipulates that the samples are to be analyzed on the half-segment level. These analyses include: weight percent water by TGA; DSC for evaluation of fuel content and thermal output; and total alpha analyses for criticality evaluation. Samples of the tank headspace are also required by the DQO to quantify any flammable gas accumulation. In addition, this section includes inventory estimates for lithium and anions, the concentrations of which were determined while assessing the samples for contamination by HHF. These analyses were requested because HHF was used during the push-mode core sampling process, and lithium bromide was added as a tracer element in the HHF. The ICP results are used to determine the extent, if any, of HHF contamination during the sampling process. Lithium analysis is requested first and, depending on the results, a bromide analysis (by ion chromatography [IC]) may be requested for corroboration. As a result of the DQO, a TCP (Schreiber 1995d) was generated to outline the characterization process for tank 241-B-101. All analytical information contained in this section was taken from Schreiber (1995c), and parts of the narrative were taken from subsections of Schreiber (1995a, 1995b, and 1995c).

A change to Babad et al. (1995) was issued while the tank 241-B-101 samples were being analyzed (Schreiber 1995d). This change deleted the notification limit for the TGA analysis and made it unnecessary to perform additional secondary analyses for the determination of the water content. However, inconsistent percent water results indicated that reanalysis of some subsegments was needed. Additional aliquots from some segment portions were taken from the archived samples and were reanalyzed using a gravimetric percent water method. This is discussed in more detail in Section 4.4.1.

Where applicable, overall analyte means were calculated as described in this paragraph. The sample/duplicate means (more than one if reruns were conducted) were averaged to obtain a subsegment mean. The subsegment means were averaged to obtain a main segment mean, the main segment means were averaged to obtain a core mean, and the core means were averaged to obtain an overall mean. When data were missing for main segments or subsegments, the overall mean was calculated by weighting more heavily those main segments and subsegments with data. When drainable liquid results were reported, they were treated as a separate subsegment and incorporated into the overall mean estimates. All less than (<) values listed are the analytical instrument's detection limit for the analyses, and are also included in all mean calculations. This overall mean value was then multiplied by the appropriate conversion factors to obtain an inventory estimate for a particular analyte.

Analytical results are reported in Tables 4-3 through 4-17. Included in the tables are sample numbers, segments and segment portions, core numbers, results, analyte means, and inventory estimates where applicable. An overall relative standard deviation (RSD) of the mean concentration was calculated for an analyte only when that analyte's reported concentration exceeded the detection limit in over half of the sludge and drainable liquid samples. If half or more of the sample results were non-detected values, an RSD was not calculated. The RSD (mean) (in percent) is defined as 100 times the standard deviation of the mean divided by the tank mean.

The standard deviation of the mean was estimated using a hierarchical statistical model to fit the data using standard analysis of variance techniques. The four quality control (QC) parameters assessed on the tank 241-B-101 samples were standards, spikes, duplicates, and blanks. The QC results for cores 90 and 91 are summarized in Section 5.1.2. More specific QC information is provided in each of the analyte data summary tables found in this section. Sample and duplicate pairs in which any of the QC parameters were outside their specified limits are footnoted appropriately. Table 4-1 lists the reported analytes and their respective table numbers.

Table 4-1. Locations of Tabulated Analytical Data.

| Analyte | Table Number |
|---|---|
| Total alpha activity | Table 4-3 |
| Thermogravimetric and gravimetric analysis | Table 4-4 |
| Comparison of TGA with gravimetric results | Table 4-5 |
| Differential scanning calorimetry (wet weight basis) | Table 4-6 |
| Differential scanning calorimetry (dry weight basis) | Table 4-7 |
| Lithium | Table 4-8 |
| Bromide | Table 4-9 |
| Estimated bromide concentrations | Table 4-10 |
| Chloride | Table 4-11 |
| Fluoride | Table 4-12 |
| Nitrite | Table 4-13 |
| Nitrate | Table 4-14 |
| Oxalate | Table 4-15 |
| Phosphate | Table 4-16 |
| Sulfate | Table 4-17 |

## 4.2 DENSITY CALCULATIONS

An overall tank density is needed to calculate projected inventories for the measured waste constituents. However, density measurements were not performed on the waste during the 1995 sampling and analysis effort. An estimated tank density can be derived using the recovered segment lengths and masses. To perform this calculation, a length-to-volume conversion factor is needed; such a factor can be obtained from the dimensions of the sampler (length = 19 in. and volume = 309 mL) as follows:

$$\left[\frac{309\ \text{mL}}{19\ \text{inches}}\right] = 16.26\ \frac{\text{mL}}{\text{inch}}$$

Using this factor, the length of the recovered segments can be converted into a volume. For example, 16 in. of waste were recovered from segment 1 of core 90. This translated into 260.2 mL as shown:

$$16\ \text{inches}\left[\frac{16.26\ \text{mL}}{\text{inch}}\right] = 260.2\ \text{mL}$$

The density of segment 1 (core 90) was then determined by dividing the measured segment mass (388.3 g) by the converted volume as displayed:

$$\frac{388.3\ \text{g}}{260.2\ \text{mL}} = 1.49\ \frac{\text{g}}{\text{mL}}$$

The densities of the other segment from core 90 and the two segments of core 91 were similarly derived. It should be noted that in deriving the densities for segment 2 from both cores, the recovered drainable liquid volumes and masses were added into the total volumes and masses, so that the density is truly an overall waste density and accounts for the drainable liquid. Also, for segment 1 of core 91, the mass of the flat, hard object (2.4 g) that was archived was not included in the total mass of the segment. Table 4-2 contains the estimated segment densities and the overall density. The overall density was calculated in the same manner as the analyte means; each segment and core was weighted equally.

Table 4-2. Estimated Densities.

| Core | Segment | Estimated Density (g/mL) | Core Mean (g/mL) | Overall Density Estimate (g/mL) |
|---|---|---|---|---|
| 90 | 1 | 1.49 | 1.49 | 1.48 |
| | 2 | 1.49 | | |
| 91 | 1 | 1.50 | 1.46 | |
| | 2 | 1.41 | | |

The estimated overall density of 1.48 g/mL from Table 4-2 has been used in all projected inventory calculations. The value of 1.48 g/mL compares favorably with the 1.66 g/mL estimate taken from the HTCE (Brevick et al. 1994), as well as the 1.59 g/mL estimate from the 1976 sampling event (Horton 1976).

## 4.3 TOTAL ALPHA ACTIVITY

The total alpha activity analyses were performed on a fusion digested sample using an alpha proportional counter according to procedure LA-508-101, revision D-2. The fusion dilutions were prepared using procedure LA-549-141, revision D-0. All total alpha activity results were below the TCP notification limit of 41 $\mu$Ci/g, with the highest observed value of any sample or duplicate result being 14.2 $\mu$Ci/g. Samples S95T001245 and S95T001337 each had results below the analytical instrument's calibrated detection limit.

Based on the DQO requirements, the TCP requested total alpha activity analysis for the solids only on a half-segment level. Table 4-3 presents the total alpha activity data for tank 241-B-101. The table identifies the sample by number, the sample segment, and the portion of the segment from which the samples were derived. The projected inventory is calculated by multiplying the weighted overall mean by the estimated density of 1.48 g/mL (see Section 4.2) and the volume of solid waste in the tank, 428 kL (113 kgal) (Hanlon 1996), along with the appropriate conversion factors.

Table 4-3. Analytical Results for Total Alpha Activity.[1] (2 sheets)

| Sample | Segment (portion) | Result (duplicate) | Sample mean | Segment mean | Core mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|---|---|---|
| | | μCi/g | μCi/g | μCi/g | μCi/g | μCi/g | % | Ci |
| **Core 90** | | | | | | 2.91 | 70.1 | 1,840 |
| S95T001215 | 1 (upper half) | 4.35 (4.79) | 4.57[2] | 2.21 | 1.17 | | | |
| S95T001218 | 1 (lower half) | 1.95 (1.83) | 1.89 | | | | | |
| S95T001221 | 1 (facies[3]) | 0.202 (0.124) | 0.163[4] | | | | | |
| S95T001230 | 2 (upper half) | 0.249 (0.228) | 0.238 | 0.132 | | | | |
| S95T001552 | 2 (lower half) | 0.0211 (0.0299) | 0.0255[4] | | | | | |
| **Core 91** | | | | | | | | |
| S95T001336 | 1 (upper half) | 3.66 (4.64) | 4.15[4] | 9.08 | 4.65 | | | |
| S95T001236 | 1 (lower half) | 13.8 (14.2) | 14 | | | | | |
| S95T001245 | 2 (upper half) | < 7.00E-04 (< 6.39E-04) | < 6.70E-04[5] | 0.210 | | | | |

Table 4-3. Analytical Results for Total Alpha Activity.[1] (2 sheets)

| Sample | Segment (portion) | Result (duplicate) | Sample mean | Segment mean | Core mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|---|---|---|
| | | μCi/g | μCi/g | μCi/g | μCi/g | μCi/g | % | Ci |
| **Core 91 (Continued)** | | | | | | 2.91 | 70.1 | 2,060 |
| S95T001337 | 2 (lower half | < 0.527 (0.363) | 0.445 | | | | | |
| S95T001556 | 2 (facies[6]) | 0.191 (0.176) | 0.183 | | | | | |

Notes:

[1]Schreiber (1995c)

[2]Spike recovery above the QC limit.

[3]Represents the bottom 9 cm (3.5 in.) of core 90, segment 1.

[4]RPD was outside the QC limit.

[5]Spike recovery below the QC limit.

[6]Represents the bottom 10 cm (4 in.) of core 91, segment 2.

## 4.4 THERMAL ANALYSES

The physical analyses required by the DQO were TGA and DSC, which determine the thermal stability or reactivity of a material. Because selected subsegments exhibited low TGA results, gravimetric analysis was requested on these samples as a secondary analyte.

### 4.4.1 Thermogravimetric and Gravimetric Analysis

In TGA, the mass of a sample is measured while its temperature is increased at a constant rate. A gas, such as nitrogen or air, is passed over the sample during the heating to remove any gaseous matter. Any decrease in the weight of a sample represents a loss of gaseous matter from the sample either through evaporation or through a reaction that forms gas phase products.

Analyses for percent water by TGA were performed under a nitrogen atmosphere using procedures LA-560-112, Revisions A-2 and B-0, (Mettler™) and LA-514-114, Revisions B-0 and C-0 (Perkin-Elmer™). Analyses for percent water were also performed by a gravimetric method (LA-564-101, Revision F-1). Percent water results are presented in Table 4-4. The overall means and RSDs were calculated as described in Section 4.1.

The ICP results for samples S95T001224, S95T001230, S95T001236, S95T001245, and S95T001248 produced higher than expected lithium results, indicating that an incursion of water from the HHF used during sampling may have contaminated these samples and thus biased high the TGA and gravimetry estimates of percent water (see Section 4.4.1). These samples correspond to the drainable liquid from core 90 segment 2; the upper half solid segment from core 90 segment 2; the lower half solid segment from core 91 segment 1; the upper half solid segment from core 91 segment 2; and the drainable liquid from core 91 segment 2. To corroborate these lithium results, the samples were also analyzed for bromide. Bromide was detected in the same five samples in which lithium was detected, as well as in sample S95T001557, which corresponds to the facies from core 91 segment 2 (see Section 4.4.2). Therefore, corrections for this contamination were made for all six of the duplicate pairs in which bromide was detected. These corrections used the estimated bromide concentrations listed in Tables 4-9 and 4-10 rather than lithium because lithium often precipitates out of solution and could thus give less reliable results. Hydrostatic head fluid blank results needed for the correction formula were not available for this sampling event; the blank results obtained for tank 241-B-104 were used instead, because both tanks were push-mode core sampled in June of 1995 (Jo 1995). These HHF corrected values are given in parentheses beside the original TGA and gravimetric results and were used in the calculation of the overall means and RSDs listed in Table 4-4 in place of the original results. Corrected lower limits for the 95 percent confidence intervals on those samples affected by HHF intrusion are given in Appendix C. The discussion in the following paragraphs, however, relates to the uncorrected percent water results because only those values were available to use to make the decisions regarding reruns.

Table 4-4. Thermogravimetric and Gravimetric Analysis Results for Tank 241-B-101.[1] (2 sheets)

| Sample number | Segment | Segment portion | Result[2] % $H_2O$ | Duplicate[2] % $H_2O$ | Mean[2] % $H_2O$ |
|---|---|---|---|---|---|
| **THERMOGRAVIMETRIC ANALYSIS** | | | | | |
| **Core 90** | | | | | |
| S95T001217 | 1 | lower half | 41.51 | 42.49 | 42.00 |
| S95T001214 | | upper half | 33.07 | 33.09 | 33.08 |
| S95T001220 | | facies[3] | 18.75 | 18.74 | 18.74 |
| S95T001229 | 2 | upper half | 16.42 (11.4) | 14.69 (9.50) | 15.55 (10.4)[4] |
| S95T002532 | | | 13.57 (8.40) | 11.54 (6.20) | 12.55 (7.30)[4] |
| S95T001551 | | lower half | 18.75 | 20.97 | 19.86[4] |
| S95T002533 | | | 48.82 | 47.58 | 48.20 |
| S95T001223 | | drainable liquid | 49.63 (47.5) | 49.41 (47.3) | 49.52 (47.4) |
| **Core 91** | | | | | |
| S95T001238 | 1 | upper half | 33.48 | 34.72 | 34.10 |
| S95T001235 | | lower half | 41.68 (39.2) | 44.13 (41.7) | 42.91 (40.4) |
| S95T001244 | 2 | upper half | 38.58 (35.6) | 48.11 (45.9) | 43.34 (40.8)[4] |
| S95T001241 | | lower half | 29.27 | 9.20 | 19.23[4] |
| S95T001241 Rerun | | | 26.79 | 25.55 | 26.17 |
| S95T001555 | | facies[5] | 18.48 (15.6) | 17.56 (14.9) | 18.02 (15.3) |
| S95T002534 | | | 16.62 (13.7) | 13.80 (11.1) | 15.21 (12.4)[4] |
| S95T001247 | | drainable liquid | 50.07 (47.6) | 50.86 (48.4) | 50.47 (48.0) |
| Grand average for thermogravimetric analysis | | | | | 32.5% |
| Relative standard deviation of the mean | | | | | 11% |
| **GRAVIMETRIC ANALYSIS** | | | | | |
| **Core 90** | | | | | |
| S95T001541 | 1 | facies[3] | 23.45 | 23.25 | 23.35 |
| S95T002532 | 2 | upper half | 11.70 (6.40) | 13.70 (8.50) | 12.70 (7.40)[4] |
| S95T001543 | | | 40.78 (37.3) | 41.26 (37.7) | 41.02 (37.5) |
| S95T002533 | | lower half | 50.60 | 50.60 | 50.60 |

Table 4-4. Thermogravimetric and Gravimetric Analysis Results for Tank 241-B-101.[1] (2 sheets)

| Sample number | Segment | Segment portion | Result[2] % $H_2O$ | Duplicate[2] % $H_2O$ | Mean[2] % $H_2O$ |
|---|---|---|---|---|---|
| Core 91 | | | | | |
| S95T001547 | 2 | lower half | 28.44 | 28.03 | 28.23 |
| S95T002534 | | facies[5] | 18.90 (16) | 18.00 (15.4) | 18.45 (15.7) |
| Grand average for gravimetric analysis | | | | | 27.1% |
| Relative standard deviation of the mean | | | | | 19.7% |

Notes:

[1]Schreiber (1995c)

[2]The values given in parentheses were corrected for HHF contamination based on the bromide concentrations.

[3]Represents the bottom 9 cm (3.5 in.) of core 90, segment 1.

[4]RPD was outside the QC limit.

[5]Represents the bottom 10 cm (4 in.) of core 91, segment 2.

The primary and duplicate results for the core 90, segment 2, upper half sample (sample number S95T001229) and the duplicate result for the core 91, segment 2, lower half sample (S95T001241) were below the TCP notification criteria of 17 weight percent moisture. The results for the core 90, segment 1 facies material (S95T001220) were very near the limit. Because of the low TGA results obtained, percent moisture by gravimetry was performed as a secondary analysis on these samples in an attempt to corroborate the TGA results. For each of these subsegments, additional sample material from archive had to be used. This additional material was reanalyzed under different sample numbers (sister samples), as follows: Sample number S95T001543 was used for the core 90, segment 2, upper half sample (sister to sample number S95T001229); sample number S95T001547 was generated to identify the core 91, segment 2, lower half sample (sister to S95T001241); and sample number S95T001541 was made as a sister sample to S95T001220 and represented the core 90, segment 1 facies material. These TGA and gravimetry results are compared (sample/duplicate means only) in Table 4-5 for all reruns to clarify the discussion. The gravimetry and TGA results for S95T001541 and S95T001220, respectively, were fairly consistent, as were the results for S95T001547 and S95T001241 with the exception of the original duplicate result for sample S95T001241. However, the gravimetry results for S95T001543 and the TGA results for S95T001229 were inconsistent by nearly a factor of three.

Table 4-5. Comparison of TGA with Gravimetric Results.[1]

| Core | Segment | Segment Portion | TGA[2] % $H_2O$ (Mean) | Gravimetric[2] % $H_2O$ (Mean) |
|---|---|---|---|---|
| 90 | 1 | Facies[3] | 18.74 | 23.35 |
| 90 | 2 | Upper half | 15.55 (10.4) | 12.70 (7.40) |
| 90 | 2 | Upper half | 12.55 (7.30) | 41.02 (37.5) |
| 90 | 2 | Lower half | 19.86 | 50.60 |
| 90 | 2 | Lower half | 48.20 | --- |
| 91 | 2 | Lower half | 19.23 | 28.23 |
| 91 | 2 | Lower half | 26.17 | --- |
| 91 | 2 | Facies[4] | 18.02 (15.3) | 18.45 (15.7) |
| 91 | 2 | Facies[4] | 15.21 (12.4) | --- |

Notes:

[1]Schreiber (1995c)

[2]The values given in parentheses were corrected for HHF contamination based on the bromide concentration.

[3]Represents the bottom 9 cm (3.1 in.) of core 90, segment 1.

[4]Represents the bottom 10 cm (4 in.) of core 91, segment 2.

In an effort to produce a consistent percent moisture value, a second archive sample from the core 90, segment 2, upper half material was obtained and identified as sample number S95T002532. This sample underwent both gravimetry and TGA analysis, and yielded percent moisture results consistent with the original TGA analysis. The difference exhibited by the first gravimetry analysis (on S95T001543) was attributed to difficulties in homogenization.

Difficulties in homogenization led to the initial decision not to analyze two of the samples (core 90, segment 2, lower half [sample number S95T001551] and core 91, segment 2, facies [sample number S95T001555]). Later, however, it was decided to proceed with the analysis. The TGA results for both samples showed the percent moisture was below the safety screening limit at a 95 percent confidence level (the results prior to this calculation were above the limit). Similar to the subsegments discussed above, the segment portions were resampled from the archive materials, analyzed for gravimetry, and reanalyzed for TGA under sample numbers S95T002533 and S9500T2534, respectively. Both the gravimetric and TGA results for S95T002534 corroborated the original TGA results for sample number S95T001555. However, neither the TGA nor the gravimetric reanalysis results for

S95T002533 corroborated the original TGA results for sample number S95T001551. See Section 5.1.3.1 for additional discussion regarding the comparison of the TGA and gravimetric methods for determining percent water.

Although some of the percent water results were below the 17 weight percent limit, their corresponding DSC results were at least an order of magnitude below the DSC notification limit (Schreiber 1995b). Provided that DSC results do not exceed the notification limit of -481 J/g, low TGA results do not constitute unsafe conditions.

### 4.4.2 Differential Scanning Calorimetry

In a DSC analysis, heat absorbed or emitted by a substance is measured while the substance is exposed to a linear increase in temperature. While the substance is being heated, a gas such as nitrogen is passed over the waste material to remove any gases being released. The onset temperature for an endothermic (characterized by or causing the absorption of heat) or exothermic (characterized by or causing the release of heat) event from a DSC run is determined graphically.

Analyses by DSC for the core samples were performed under a nitrogen atmosphere using procedures LA-514-113, Revision B-1 and LA-514-114, Revisions B-0 and C-0. No results exceeded the safety screening notification action limit of -481 J/g; therefore, no notifications were required.

The DSC wet weight basis results are presented in Table 4-6. The temperature at maximum enthalpy change ($\Delta H$) and the magnitude of the enthalpy change are provided for each transition. The first transition represents the endothermic reaction associated with the evaporation of free and interstitial water. The second transition probably represents the energy (heat) required to remove bound water from hydrated compounds such as aluminum hydroxide or to melt salts such as sodium nitrate. The third transition is generally exothermic and is probably caused by the fuel components of the sample reacting with the nitrate salts. Exothermic reactions are denoted by a minus sign in front of the $\Delta H$ value. For each subsample showing an exothermic reaction, the wet weight basis DSC results were converted to a dry weight basis by the following equation:

$$\text{Exotherm (dry weight)} = \frac{\text{exotherm (wet weight)}}{\left[1 - \frac{\%\ \text{Water}}{100}\right]}$$

The percent water value used was the TGA sample and duplicate mean result (from Table 4-4) that corresponded to a particular DSC sample number. These dry weight results are presented in Table 4-7, and can be directly compared to the safety screening limit of -481 J/g.

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-B-101.[1] (2 sheets)

| Sample number | Segment (portion) | Run | Sample weight (mg) | Transition 1 | | Transition 2 | | Transition 3 | | Transition 4 | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) |
| **Core 90** | | | | | | | | | | | |
| S95T001214 | 1 (upper half) | 1[2] | 14.68 | 105.7 | 736.2 | 229.0 | 13.7 | 385.7 | -193.1 | --- | --- |
| | | 2 | 11.0 | 108.1 | 775.6 | 225.2 | 13.3 | 313.5 | -174.5 | --- | --- |
| S95T001217 | 1 (lower half) | 1[2] | 8.35 | 121.9 | 1,004.2 | 229.2 | 28.7 | 315.5 | -211.0 | --- | --- |
| | | 2 | 25.50 | 131.7 | 911.5 | 224.8 | 20.7 | 321.5 | -156.6 | --- | --- |
| | | 3 | 23.80 | 125.8 | 869.3 | 232.7 | 39.9 | 433.7 | -186.7 | --- | --- |
| | | 4 | 21.20 | 128.5 | 862.8 | 232.8 | 41.8 | 439.7 | -182.3 | --- | --- |
| S95T001220 | 1 (facies[3]) | 1 | 11.80 | 100.4 | 516.6 | 289.7 | 588.8 | --- | --- | --- | --- |
| | | 2 | 34.50 | 124.7 | 483.2 | 285.3 | 597.3 | --- | --- | --- | --- |
| S95T001223 | 2 (drainable liquid) | 1 | 14.5 | 124.7 | 1091.3 | 227.1 | 49.7 | 429.7 | -26.3 | --- | --- |
| | | 2 | 13.58 | 118.8 | 1158.0 | 223.1 | 57.2 | 397.7 | -26.3 | --- | --- |
| S95T001229 | 2 (upper half) | 1 | 32.80 | 124.5 | 434.2 | 295.4 | 472.5 | --- | --- | --- | --- |
| | | 2 | 19.90 | 104.3 | 365.1 | 293.1 | 467.4 | 436.9 | 30.3 | --- | --- |
| S95T001551 | 2 (lower half) | 1 | 43.72 | 140.01 | 462.11 | 286.61 | 56.07 | --- | --- | --- | --- |
| | | 2 | 27.67 | 138.15 | 483.45 | --- | --- | --- | --- | --- | --- |

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-B-101.[1] (2 sheets)

| Sample number | Segment (portion) | Run | Sample weight (mg) | Transition 1 | | Transition 2 | | Transition 3 | | Transition 4 | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) | Peak (°C) | ΔH (J/g) |
| **Core 91** | | | | | | | | | | | |
| S95T001235 | 1 (lower half) | 1 | 20.70 | 126.6 | 971.3 | 231.0 | 21.5 | 315.6 | -24.0 | 447.6 | 1.3 |
| | | 2 | 16.48 | 121.0 | 899.8 | 182.9 | 13.1 | 231.0 | 24.8 | 315.5 | -25.4 |
| S95T001238 | 1 (upper half) | 1 | 30.15 | 123.3 | 722.1 | 224.8 | 29.4 | 331.5 | -163.6 | --- | --- |
| | | 2 | 22.8 | 120.4 | 831.3 | 228.8 | 50.0 | 419.7 | -165.4 | --- | --- |
| S95T001241 | 2 (lower half) | 1 | 35.10 | 123.8 | 644.2 | 287.1 | 279.6 | 395.0 | 42.4 | --- | --- |
| | | 2 | 21.45 | 113.5 | 620.6 | 293.6 | 768.1 | --- | --- | --- | --- |
| S95T001244 | 2 (upper half) | 1[4] | 14.60 | 122.7 | 1,162.0 | 229.0 | 48.7 | 427.6 | -2.9 | --- | --- |
| | | 2 | 14.60 | 122.7 | 1,201.7 | 229.0 | 47.5 | 427.6 | -2.7 | --- | --- |
| S95T001555 | 2 (facies[5]) | 1 | 51.93 | 140.51 | 266.94 | 319.94 | 93.64 | 461.83 | 590.03 | --- | --- |
| | | 2 | 38.04 | 140.49 | 460.32 | 288.99 | 79.0 | --- | --- | --- | --- |
| S95T001247 | 2 (drainable liquid) | 1 | 14.6 | 120.1 | 1,231.6 | 222.9 | 48.9 | 427.6 | -3.1 | --- | --- |
| | | 2 | 16.50 | 116.5 | 1,138.9 | 222.9 | 49.1 | 425.5 | -3.2 | --- | --- |
| S95T001232 | field blank | 1 | 9.58 | 101.3 | 1,781.4 | --- | --- | --- | --- | --- | --- |
| | | 2 | 12.79 | 101.3 | 1,373.3 | --- | --- | --- | --- | --- | --- |

Notes:

[1]Schreiber (1995c)
[2]RPD was outside the QC limit.
[3]Represents the bottom 9 cm (3.5 in.) of core 90 segment 1.
[4]Standard recovery was above the QC limit.
[5]Represents the bottom 10 cm (4 in.) of core 91, segment 2.

Table 4-7. Dry Weight Basis for DSC Results Showing Exotherms.[1]

| Sample Number | Segment | Segment Portion | Results[2] (J/g Dry) | Duplicate (J/g Dry) | Mean (J/g Dry) |
|---|---|---|---|---|---|
| **Core 90** | | | | | |
| S95T001214 | 1 | upper half | -289.0 | -261.0 | -275.0 |
| S95T001217 | 1 | lower half | -364.0 | -270.0 | -317.0 |
| | | | -322.0 | -314.0 | -318.0 |
| S95T001223 | 2 | drainable liquid | -52.0 | -52.0 | -52.0 |
| **Core 91** | | | | | |
| S95T001235 | 1 | lower half | -42.0 | -44.5 | -43.3 |
| S95T001238 | 1 | upper half | -248.0 | -251.0 | -249.5 |
| S95T001244 | 2 | upper half | -5.0 | -5.0 | -5.0 |
| S95T001247 | 2 | drainable liquid | -6.0 | -6.0 | -6.0 |

Notes:

[1]Schreiber (1995c)

[2]The TGA percent water values used to convert each DSC run from a wet weight to dry weight basis are found as the corresponding sample means in Table 4-4.

## 4.5 INDICATOR ANALYTES FOR HYDROSTATIC HEAD FLUID CONTAMINATION

### 4.5.1 Lithium

The ICP analysis was performed using procedures LA-505-151, Revision D-3 and LA-505-161, Revision A-1. Solid subsamples were prepared by fusion per procedure LA-549-141, Revision D-0, and liquid subsamples were either analyzed directly or diluted with acid according to procedure LA-505-158, Revision A-4. The ICP analysis is used to determine whether lithium is present in the samples. Lithium bromide is used as a tracer element in the HHF, and the presence of large concentrations of lithium in a sample may indicate that the sample has been contaminated during the sampling process. An overall mean is not calculated for lithium because it is not a constituent of the tank waste; small amounts of it are added deliberately on occasion during the sampling process. As noted in the TGA results, the ICP results for samples S95T001224, S95T001230, S95T001236, S95T001245, and S95T001248 reveal that these samples may have been contaminated with lithium, indicating an incursion of water from the HHF used during sampling. Table 4-8 summarizes the ICP analysis results for lithium in the tank 241-B-101 samples.

Table 4-8. Lithium Analytical Results.[1] (2 sheets)

| Sample number | Segment (portion) | Sample µg/g | Duplicate µg/g | Mean µg/g | RPD % |
|---|---|---|---|---|---|
| **Core 90** | | | | | |
| S95T001221 | 1 (facies[2]) | < 44.82 | < 45.22 | < 45.02 | N/A |
| S95T001215 | 1 (upper half) | < 45.42 | < 46.28 | < 45.85 | N/A |
| S95T001218 | 1 (lower half) | < 46.98 | < 46.66 | < 46.82 | N/A |
| S95T001230 | 2 (upper half) | 79.25 | 70.87 | 75.06[3] | 11.2 |
| S95T001552 | 2 (lower half) | < 47.01 | < 47.19 | < 47.10 | N/A |
| S95T001224 | 2 (drainable liquid) | 147.0 | 144.7 | 145.85 | 1.65 |
| **Core 91** | | | | | |
| S95T001336 | 1 (upper half) | < 55.43 | < 55.07 | < 55.25 | N/A |
| S95T001236 | 1 (lower half) | 84.70 | 74.83 | 79.77[3] | 12.4 |
| S95T001245 | 2 (upper half) | 104.5 | 110.4 | 107.45[4] | 5.5 |
| S95T001337 | 2 (lower half) | < 43.04 | < 45.79 | < 44.42 | N/A |
| S95T001556 | 2 (facies[5]) | < 52.47 | < 52.37 | < 52.42 | N/A |
| S95T001248 | 2 (drainable liquid) | 135.0 | 135.4 | 135.2 | 0.22 |

Notes:

N/A = not applicable

[1]Schreiber (1995c)

[2]Represents the bottom 9 cm (3.5 in.) of core 90, segment 1.

[3]RPD was outside the QC limit.

[4]Spike recovery was below the QC limit.

[5]Represents the bottom 10 cm (4 in.) of core 91, segment 2.

### 4.5.2 Bromide

Ion chromatography results for bromide were used to corroborate the lithium results from the ICP analysis. The samples from tank 241-B-101 were prepared for IC analysis by a water digestion (procedure LA-504-101, Revision D-0) and were analyzed using procedure LA-533-105, Revision D-0. Table 4-9 presents the results of the IC analysis for bromide. The high detection limit values can be attributed to the very high levels of nitrate and nitrite in the tank, which made it necessary for the samples to be analyzed at high dilution factors. While the bromide concentration of the diluted samples often fell below the *calculated*

detection limit of the ion chromatograph, a bromide peak was still detected. For those diluted samples with detectable bromide peaks, bromide concentrations were estimated from a linear extrapolation of the calibration curve. The extrapolated bromide values, presented in Table 4-10, should be considered qualitative. An overall mean for bromide was not calculated because it is an analyte used to determine sample contamination and is not a tank constituent.

Table 4-9. Analytical Results for Bromide.[1]

| Sample number | Segment (portion) | Result | Duplicate | Mean | RPD |
|---|---|---|---|---|---|
| | | µg/g | µg/g | µg/g | % |
| **Core 90** | | | | | |
| S95T001542 | 2 (drainable liquid) | < 1,340 | < 1,340 | < 1,340[2] | N/A |
| S95T001544 | 2 (upper half) | 1,500 | 1,510 | 1,500 | 0.66 |
| S95T001553 | 2 (lower half) | < 3,540 | < 3,540 | < 3,540 | N/A |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | < 2,280 | < 2,280 | < 2,280 | N/A |
| S95T001549 | 2 (upper half) | < 2,190 | < 2,190 | < 2,190 | N/A |
| S95T001557 | 2 (facies[3]) | < 2,080 | < 2,080 | < 2,080 | N/A |
| S95T001550 | 2 (drainable liquid) | 1,740 | 1,770 | 1,760 | 1.71 |

Notes:

N/A = not applicable

[1]Schreiber (1995c)

[2]Spike recovery was below the QC limit.

[3]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

Table 4-10. Estimated Bromide Concentrations.[1]

| Sample Number | Segment (portion) | Concentration (μg/g) | |
|---|---|---|---|
| | | Sample | Duplicate |
| **Core 90** | | | |
| S95T001542 | 2 (drainable liquid) | 1,460 | 1,480 |
| S95T001553 | 2 (lower half) | ND | ND |
| **Core 91** | | | |
| S95T001546 | 1 (lower half) | 1,100 | 1,120 |
| S95T001549 | 2 (upper half) | 1,220 | 1,100 |
| S95T001557 | 2 (facies[2]) | 904 | 815 |

Note:

[1]Schreiber (1995c)

[2]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

## 4.6 ION CHROMATOGRAPHY ANALYSIS

Analyses for anions other than bromide were performed in the process of analyzing for bromide. Results of the IC analyses are presented in Tables 4-11 through 4-17. A projected inventory was calculated for each of the non-bromide anions, using a density value of 1.48 g/mL (see Section 4.2) and a waste volume of 428 kL (113 kgal) (Hanlon 1996). The "Sample Mean" column is an average of the sample and duplicate analyses. The overall mean and RSD (mean) estimates in the fourth and fifth columns are calculated as discussed in Section 4.1.

Table 4-11. Analytical Results for Chloride.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| **Core 90** | | | 556 | 27.5 | 352 |
| S95T001542 | 2 (drainable liquid) | 686 | | | |
| S95T001544 | 2 (upper half) | 350[2] | | | |
| S95T001553 | 2 (lower half) | 1,268[3] | | | |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | < 299 | | | |
| S95T001549 | 2 (upper half) | 302 | | | |
| S95T001557 | 2 (facies[4]) | < 273[5] | | | |
| S95T001550 | 2 (drainable liquid) | 591 | | | |

Notes:

[1]Schreiber (1995c)

[2]RPD was outside the QC limit.

[3]Spike recovery was below the QC limit.

[4]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

[5]Spike recovery was above the QC limit.

Table 4-12. Analytical Results for Fluoride.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| **Core 90** | | | 269 | 11.9 | 170 |
| S95T001542 | 2 (drainable liquid) | 238 | | | |
| S95T001544 | 2 (upper half) | 143 | | | |
| S95T001553 | 2 (lower half) | < 348 | | | |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | 365 | | | |
| S95T001549 | 2 (upper half) | < 215[2] | | | |
| S95T001557 | 2 (facies[3]) | < 204[2] | | | |
| S95T001550 | 2 (drainable liquid) | 253[4] | | | |

Notes:

[1]Schreiber (1995c)

[2]Spike recovery was above the QC limits.

[3]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

[4]Spike recovery was below the QC limits.

Table 4-13. Analytical Results for Nitrite.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| **Core 90** | | | 65,900 | 26.9 | 41,700 |
| S95T001542 | 2 (drainable liquid) | 1.32E+05[3] | | | |
| S95T001544 | 2 (upper half) | 64,000[2,3] | | | |
| S95T001553 | 2 (lower half) | 19,800[2,3] | | | |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | 44,800[2,3] | | | |
| S95T001549 | 2 (upper half) | 72,400[2,3] | | | |
| S95T001557 | 2 (facies[4]) | 20,800[2] | | | |
| S95T001550 | 2 (drainable liquid) | 1.32E+05 | | | |

Notes:

[1]Schreiber (1995c)

[2]RPD was outside the QC limit.

[3]Spike recovery was above the QC limits.

[4]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

Table 4-14. Analytical Results for Nitrate.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | μg/g | μg/g | % | kg |
| **Core 90** | | | 2.32E+05 | 14.8 | 1.47E+05 |
| S95T001542 | 2 (drainable liquid) | 3.76E+05 | | | |
| S95T001544 | 2 (upper half) | 1.94E+05 | | | |
| S95T001553 | 2 (lower half) | 2.12E+05[2,3] | | | |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | 1.39E+05 | | | |
| S95T001549 | 2 (upper half) | 1.64E+05[2,4] | | | |
| S95T001557 | 2 (facies[5]) | 2.98E+05[2] | | | |
| S95T001550 | 2 (drainable liquid) | 3.36E+05 | | | |

Notes:

[1]Schreiber (1995c)

[2]RPD was outside the QC limit.

[3]Spike recovery was above the QC limits.

[4]Spike recovery was below the QC limits.

[5]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

Table 4-15. Analytical Results for Oxalate.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| Core 90 | | | < 1,620 | N/A | < 1,030 |
| S95T001542 | 2 (drainable liquid) | < 1,050 | | | |
| S95T001544 | 2 (upper half) | < 1,000 | | | |
| S95T001553 | 2 (lower half) | < 2,780 | | | |
| Core 91 | | | | | |
| S95T001546 | 1 (lower half) | < 1,790 | | | |
| S95T001549 | 2 (upper half) | < 1,720 | | | |
| S95T001557 | 2 (facies[2]) | < 1,630 | | | |
| S95T001550 | 2 (drainable liquid) | < 1,050 | | | |

Notes:

N/A = not applicable

[1]Schreiber (1995c)

[2]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

Table 4-16. Analytical Results for Phosphate.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected Inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| **Core 90** | | | 5,820 | 21.4 | 3,690 |
| S95T001542 | 2 (drainable liquid) | 7,380 | | | |
| S95T001544 | 2 (upper half) | 3,760[2] | | | |
| S95T001553 | 2 (lower half) | < 3,340 | | | |
| **Core 91** | | | | | |
| S95T001546 | 1 (lower half) | 6,850 | | | |
| S95T001549 | 2 (upper half) | 12,400[3] | | | |
| S95T001557 | 2 (facies[4]) | < 3,120 | | | |
| S95T001550 | 2 (drainable liquid) | 4,800 | | | |

Notes:

[1]Schreiber (1995c)

[2]Spike recovery was above the QC limit.

[3]RPD was outside the QC limit.

[4]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

Table 4-17. Analytical Results for Sulfate.[1]

| Sample number | Segment (portion) | Sample mean | Overall mean | RSD (Mean) | Projected inventory |
|---|---|---|---|---|---|
| | | µg/g | µg/g | % | kg |
| Core 90 | | | 68,300 | 55 | 43,300 |
| S95T001542 | 2 (drainable liquid) | 22,800[2] | | | |
| S95T001544 | 2 (upper half) | 16,000[3] | | | |
| S95T001553 | 2 (lower half) | 2.41E+05[2,3] | | | |
| Core 91 | | | | | |
| S95T001546 | 1 (lower half) | 10,600 | | | |
| S95T001549 | 2 (upper half) | 11,800 | | | |
| S95T001557 | 2 (facies[4]) | 1.95E+05 | | | |
| S95T001550 | 2 (drainable liquid) | 21,200 | | | |

Notes:

[1]Schreiber (1995c)

[2]Spike recovery was above the QC limits.

[3]RPD was outside the QC limit.

[4]Represents the bottom 10 cm (4 in.) of core 91, segment 2, and was labeled as a separate facies because of its distinct color relative to the remaining core portions.

## 4.7 TANK HEADSPACE FLAMMABILITY

The *Tank Safety Screening Data Quality Objective* (Babad et al. 1995) requires that tank headspace flammability be determined as a percentage of the lower flammability limit (LFL). On March 26, 1996, a field measurement was made for tank headspace flammable gases using a combustible gas monitor. The measurement was made while sampling at a depth of 10 m (33 ft) through riser 2 and yielded a reading of 0 percent of the LFL (WHC 1996).

# 5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

The purpose of this chapter is to evaluate the overall quality and consistency of the available results for tank 241-B-101 and to assess and compare these results against historical information and program requirements.

## 5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

This section evaluates sampling and analysis factors that may impact the use or interpretation of the data. These factors are used to assess the overall quality and consistency of the data and to identify any limitations in the use of the data. Most of the usual data consistency checks were unable to be conducted due to the lack of data. These consistency checks will be done in a future revision after completion of the pending analyses.

### 5.1.1 Field Observations

Sample recoveries from all of the extruded segments were high (> 80 percent). There were 41 cm (16 in.) of sample recovered from two of the segments, 30 cm (12 in.) from a third, and 20 cm (8 in.) from the fourth. Also, core 90, segment 1 and core 91, segment 1 showed 8-cm (3-in.) gaps in the sample upon extrusion. These observations may draw into question how well the samples represent the entire tank contents. Percent water corrections were needed for six samples due to contamination by the HHF. There may also have been some homogenization difficulties with the crystalline samples from the bottom of segment 2 for both cores.

### 5.1.2 Quality Control Assessment

The usual quality control (QC) assessment includes an evaluation of the appropriate blanks, duplicates, spikes, and standards that are performed in conjunction with a chemical analysis. All four pertinent quality control tests were conducted with the 1995 core samples; this section provides a general evaluation of the results. The TCP (Schreiber 1995d) establishes the specific accuracy and precision criteria for three of the QC checks. The fourth, blank contamination, has a criterion set by the laboratory. Samples that had one or more QC results outside the criteria have been identified (by footnoting) in the Section 4 data tables. The original data reports (Schreiber 1995a, 1995b, and 1995c) should be consulted for more detailed QC information.

The standards conducted for all of the analytes were within the defined criterion with the exception of one of eight conducted on the DSC solid samples. This result was slightly above the criterion at 112.1 percent recovery. Spike recoveries are not applicable to the percent water or DSC analyses. For total alpha activity, two of ten spikes conducted were

outside the target level, one slightly above and one substantially below (60%). The low spike recovery was most likely caused by a high solids content on the sample mount and subsequent self-shielding. Also, both the primary and duplicate results for that sample were below the detection limit, and almost five orders of magnitude below the notification limit of 41 $\mu$Ci/g. Regardless, these two deviations were not substantial enough to affect the criticality evaluation. One of ten spikes conducted in conjunction with the lithium analyses was slightly low, and at least one spike recovery was outside the limits for all anions except oxalate. As mentioned in Section 4.4.2, the detection limits for bromide were high, as were the dilution factors. This situation affected all of the IC analyses and in turn caused poor or meaningless spike recoveries.

The laboratory analytical precision is estimated by the relative percent difference (RPD), which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times one hundred. A number of duplicate pairs for several analytes had RPDs larger than the SAP target level, but most of these were due to sample heterogeneity or large sample dilution (IC only). The crystalline facies material did not easily lend itself to complete homogenization. Finally, none of the samples exceeded the criterion for preparation blanks; thus, contamination was not a problem for any of the analyses.

To summarize, the vast majority of QC results were within the boundaries specified in the TCP (Schreiber 1995d). Although some QC results were outside their target levels, they were not found to substantially impact either the validity or the use of the data.

### 5.1.3 Data Consistency Checks

Comparisons of different analytical methods can help to assess the consistency and quality of the data. Examples would be the comparison of percent water by TGA versus gravimetry, phosphorus by ICP versus phosphate by IC, total alpha or total beta activity compared to the sum of alpha or beta emitters, and the calculation of a mass and charge balance to check the overall consistency of the data. Due to the limited data, only the comparison of percent water by TGA and gravimetry was possible.

**5.1.3.1 Comparison of Percent Water Results by TGA and Gravimetry.** The percent water data for the tank 241-B-101 core segment subsamples revealed a bias between the two methods, TGA and gravimetry, used to generate the data (Cromar 1996). The results of those samples that had been determined by both TGA and gravimetry (see Table 4-4) were subjected to a randomized complete block design ANOVA. The ANOVA results showed a significant bias between the two analytical methods at the 95% probability level; the gravimetric method appears to be biased toward higher percent water values than the TGA method. One possible reason for the bias between the two methods is that the small sample size (approximately 10 mg) for TGA could cause significant water loss during sample

preparation prior to the analysis. While the two analytical methods may be biased with respect to each other, both methods showed similar trends in the water contents of the various subsegments.

## 5.2 COMPARISON OF ANALYTICAL RESULTS FROM DIFFERENT SAMPLING EVENTS

Only limited comparisons were possible between the 1976 and 1995 analytical results. Table 5-1 shows the common solids data for these two sampling events. Complete analytical results for the 1976 and 1995 sampling events are given in Appendix B and Section 4.0, respectively. This comparison should be viewed with caution, because: (1) supernatant was removed between 1976 and 1995 as a result of stabilization efforts; and (2) the 1976 values were based on a single sample, whereas the means from 1995 were averages from different segments and cores. Because tank 241-B-101 waste exhibited significant heterogeneity (see Section 5.3), results based on one segment would be expected to differ from averages of several segments. Analytical ranges from the 1995 event are also shown in Table 5-1 for comparison. As can be seen in the table, poor correlation is found between the results of the two sampling events, especially for the anions. Because the 1976 data predate May, 1989, the 1976 data are not validated and may not be used for decision-making purposes.

Table 5-1. Comparison of Sludge Data from 1976 and 1995.

| Analyte | 1976 Result[1] | 1995 Result[2] | |
|---|---|---|---|
| | | Mean | Analytical Range |
| Percent water | 20.1 % | TGA: 32.5% | TGA: 7.30 - 48.20 % |
| | | Gravimetry: 27.1% | Gravimetry: 7.40 - 50.60 % |
| $NO_3^-$ | 8.62E+05 μg/g | 2.32E+05 μg/g | 1.39E+05 - 2.98E+05 μg/g |
| $PO_4^{3-}$ | 1.02E+05 μg/g | 5,820 μg/g | < 3,120 - 12,400 μg/g |

Notes:

[1]Horton (1976)

[2]Schreiber (1995c)

## 5.3 TANK WASTE PROFILE

One objective of the 1995 sampling event was to obtain a vertical profile from two widely spaced risers (Schreiber 1995d). This objective was met, as risers 2 and 7 were approximately 180° apart and near the outer edge of the tank. The sample analyses from these risers provided information on the horizontal and vertical distribution of the tank waste

for many of the analytes. Information on the vertical disposition of the waste was also available from the tank layer model (TLM) (Agnew et al. 1995) (Figure 2-4). According to the TLM, the waste was composed of five layers. Two-thirds of the waste consisted of a layer of saltcake from the 242-B Evaporator, under which was a small amount of metal waste. Above the saltcake was a layer of high-level B-plant waste, followed by low-level B Plant waste. A layer of unknown waste was expected on top. This number of waste types indicated that the tank contents should be vertically heterogeneous.

The visual descriptions of the samples also indicated some vertical differences, but did not reveal any obvious horizontal trends (Table 3-2). The sludge generally became lighter in color as a function of depth, although the consistency did not seem to change. The very bottom subsegments from both cores were markedly different in appearance than the other subsegments. The bottom 11 cm (4.5 in.) of core 90 and the bottom 10 cm (4 in.) of core 91 were both described as crystalline flakes, although the color was white for one core and light brown for the other. This material is most likely saltcake from the 242-B Evaporator.

The fact that two risers with multiple segments were sampled allowed a statistical procedure known as the analysis of variance (ANOVA) to be conducted on the 1995 core samples. The specific ANOVA model was a random effects nested model, and was used to determine if there were any horizontal or vertical differences (spatial variability) in analyte concentrations. The analysis was conducted on the combined solid/drainable liquid data in the same weighted manner in which the analyte means were calculated. Only analytes that had more than half of the solid data results above the detection limit were used in this analysis, and for a given analyte, only those detected values were used. The ANOVA generates a p-value that is compared with a standard significance level ($\alpha = 0.05$). If a p-value is below 0.05, there is sufficient evidence to conclude that at least some of the sample means are significantly different from each other. However, if a p-value is above 0.05, there is not sufficient evidence to conclude that any of the samples are significantly different from each other.

Sufficient data were available to analyze seven analytes: total alpha activity, nitrate, nitrite, phosphate, sulfate, and percent water by both the TGA and gravimetric method. The results of the ANOVA tests indicated that there were significant horizontal differences in analyte concentrations for nitrite, phosphate, and sulfate (all three p-values were $< 0.000$). The segment-level tests showed no significant vertical differences between the main segments for any of the seven analytes. However, there were significant vertical differences on the subsegment level for all of the analytes except percent water by gravimetry (p-value = 0.0512). Based on the evidence of the tank layer model, the visual descriptions of the core samples, and the statistical analysis, the waste in tank 241-B-101 shows significant differences in analyte concentration as a function of both depth and radial location within the tank.

## 5.4 COMPARISON OF TRANSFER HISTORY AND ANALYTICAL RESULTS

The Historical Tank Content Estimate (HTCE) (Brevick et al. 1994b) data for tank 241-B-101 were compared to the 1995 results. These two sets of data only had a limited number of analytes in common, as shown in Table 5-2. This comparison applies to solids data only.

Table 5-2. Comparison of Historical Tank Content Estimate and 1995 Analytical Data.

| Analyte | Historical Tank Content Estimate Solids Inventory[1] | 1995 Core Sample Sludge Results[2] |
|---|---|---|
| Percent water | 54% | TGA: 32.5%<br>Gravimetry: 27.1% |
| $NO_3^-$ | 61,800 kg | 1.47E+05 kg |
| $PO_4^{3-}$ | 39,600 kg | 3,690 kg |

Notes:

[1]Brevick et al. (1994a)

[2]Schreiber (1995c)

The comparison generally demonstrated poor agreement. Both percent water and phosphate HTCE values were higher than the 1995 analytical results. However, the nitrate HTCE value was less than half of the nitrate inventory calculated from the analytical results. At least some of the observed discrepancies are probably due to the variation in the tank waste noted in the previous section. Another cause of the differences could be that not all of the waste types projected by the TLM were sampled. Specifically, 11 kL (3 kgal) of MW is estimated by the TLM to compose the bottom layer of the tank 241-B-101 waste (Agnew et al. 1995b). Due to the dished tank bottom and the location of the risers, it is likely that this waste was not sampled. The HTCE estimates, however, account for MW analytes in their predictions.

## 5.5 EVALUATION OF PROGRAM REQUIREMENTS

The 1995 tank 241-B-101 core samples were obtained to screen the tank waste for unidentified safety issues as prescribed in *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). A discussion of the requirements of this DQO and a comparison of the analytical data to defined concentration limits are presented in this section. Evaluation of data in terms of operational, environmental, or process development requirements is not possible due to the limited number of analyses performed.

### 5.5.1 Safety Evaluation

Data criteria identified in the safety screening DQO (Babad et al. 1995) are used to assess the safety of the waste in tank 241-B-101. For a proper safety assessment, the DQO requires samples from two widely spaced risers. This requirement was met, because the sampling risers (risers 2 and 7) were on opposite sides of the tank. Four primary analyses are required by the safety screening DQO: (1) DSC for evaluation of tank waste energetics; (2) TGA for measurement of the weight percent water; (3) determination of the total alpha activity; and (4) determination of tank headspace flammability as a percentage of the LFL. For each of the required analyses, a notification threshold is established by the DQO which, if exceeded, may warrant further investigation to assure the safety of the tank. In addition to the safety screening analyses, the tank 241-B-101 samples were analyzed for lithium and bromide to determine the extent of contamination by HHF. Table 5-3 displays the notification threshold for each safety screening decision variable. Also included are the analytical results and 95 percent confidence interval limits to provide comparisons with the decision limits.

With respect to the DSC results, several samples from tank 241-B-101 did display exothermic behavior. However, none of the exothermic reactions exceeded the -481 J/g DQO limit (dry weight). Statistical analyses were performed to determine the 95 percent confidence intervals for each DSC sample and duplicate pair, and the upper limits from these confidence intervals were compared to the decision limit of -481 J/g (dry weight basis). The results of these calculations are given in Table C-1 of Appendix C. All of the upper limits from the confidence interval were below the decision limit, indicating that, with 95 percent certainty, tank 241-B-101 does not pose a threat for an energetics event. The highest upper limit result, -439.1 J/g, was obtained from the lower half of segment 1 from core 90.

Table 5-3. Safety Screening DQO Decision Variables and Criteria.

| Safety Issue | Primary Decision Variable | Decision Criteria Threshold | Analytical Value | 95% Confidence Interval Limit[3] |
|---|---|---|---|---|
| Ferrocyanide/ organics | Total fuel content | -481 J/g (-115 cal/g) | Largest exothermic reaction (dry weight) = -364 J/g (lower half of segment 1 from core 90) | -439.1 J/g (lower half of segment 1 from core 90) |
| Organics | Percent moisture | 17 wt% | 32.5%[1] (mean) | 0% (segment 2, lower half, cores 90 and 91) |
| Criticality | Total alpha | 41 $\mu$Ci/g (1 g/L)[2] | 2.91 $\mu$Ci/g (mean) | 15.26 $\mu$Ci/g (lower half of segment 1, core 91) |
| Flammable gas | Headspace flammability | 25% of LFL | 0% of LFL[4] | N/A |

Notes:

[1]Limit excursions for individual samples have been discussed in detail in the accompanying text.

[2]Although the actual decision criterion listed in the DQO is 1 g/L, total alpha is measured in $\mu$Ci/g rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it was assumed that all alpha decay originates from $^{239}$Pu. As indicated in the TCP, assuming a tank density of 1.5 g/mL and using the specific activity of $^{239}$Pu (0.0615 Ci/g), the decision criterion may be converted to 41 $\mu$Ci/g as shown:

$$\left(\frac{1\ g}{L}\right)\left(\frac{1\ L}{10^3\ mL}\right)\left(\frac{1}{density}\frac{mL}{g}\right)\left(\frac{0.0615\ Ci}{1\ g}\right)\left(\frac{10^6\ \mu Ci}{1\ Ci}\right) = \frac{61.5}{density}\frac{\mu Ci}{g}$$

[3]See Appendix C for complete results. Value listed is highest upper limit results for DSC and total alpha activity, and lowest lower limit result for TGA.

[4]Flammable gas measured in tank headspace at a depth of 10 m (33 ft) through riser 2 (WHC 1996).

As can be seen in Table 5-3, the overall percent water mean determined by TGA was well above the 17 weight percent safety screening DQO limit. However, some of the individual samples did have results that violated the limit. The upper half portion of segment 2, core 90 (sample S95T001229) had results of 11.4 for the original and 9.50 for the duplicate; the facies from segment 2 of core 91 (sample S95T001555) had results of 15.6 for the original and 14.9 for the duplicate; and sample S95T001241, the lower half portion of segment 2, core 91, had a duplicate result of 9.200. Due to the large RPD between the original and duplicate results for sample S95T001241, a second TGA analysis was done that yielded results above the notification limit (Schreiber 1995c).

Statistical analyses were performed to determine the 95 percent confidence intervals for the TGA data, and the lower limits from the confidence intervals were compared to the decision limit of 17 weight percent. This would identify any samples in which the confidence interval lower limit was below the 17 percent limit. Table C-2 in Appendix C presents the results of these calculations. Obviously, the three samples already discussed which had sample results below the limit also had confidence interval lower limits below the limit. Two samples were discovered that had 95 percent confidence interval lower limits below 17 percent, but had overall means greater than 17 percent. Sample S95T001551 (lower half sample from segment 2 of core 91) had a mean of 19.86 percent, but a 95 percent confidence interval lower limit of 12.85 percent. Likewise, sample S95T001244 (upper half from segment 2 of core 91) had a mean of 40.8 percent and a 95 percent lower limit of 9.30 percent. One additional sample, S95T001220 (facies from segment 1 of core 90), had a 95 percent confidence interval lower limit close to 17 percent (18.71 percent).

For all five samples that had 95 percent confidence interval lower limits below 17 percent, and the single sample that had a lower limit near 17 percent (with the exception of sample S95T001244), a secondary percent water analysis by gravimetry was performed. Results showed that four of the five samples had greater than 17 weight percent water. The gravimetric result from the facies from segment 2 of core 91 was below the 17 percent limit with a value of 15.7 percent. An anomaly was discovered for the upper half sample from segment 2 of core 90. The gravimetric result of 37.5 percent was nearly three times the TGA measurement. To resolve this discrepancy, a second gravimetric analysis was performed. The result from this second gravimetric analysis, 7.40 percent, was more consistent with the TGA value, indicating that the measurements from the first gravimetric run were erroneous (Schreiber 1995a).

Although not specified in the DQO, additional TGA runs were performed on three of the five samples just discussed. The lower half sample from segment 2 of core 90 and the facies from segment 2 of core 91 were subjected to a second TGA analysis due to the low original TGA results. A second TGA run was performed on the upper half sample from segment 2 of core 90 because of the large difference between the initial TGA result and the initial gravimetry result as mentioned previously. Results from the segment 2, core 90, upper half sample and the core 91, segment 2, facies were below the 17 percent limit, with respective values of 7.30 and 12.4 percent. The result for the core 90, segment 2, lower half sample was well above the limit. However, the 95 percent confidence interval test revealed that this sample, along with the other two, had lower limits below 17 percent.

Even though results from several of the TGA and gravimetric analyses did not meet the required minimum of 17 weight percent water, no exothermic reactions that exceeded the safety screening DQO limit were found in any of these samples. Consequently, the low moisture content of these samples should not constitute unsafe conditions (Schreiber 1995a).

The criticality potential of a tank is assessed using the total alpha activity of the waste material. Although the safety screening criterion for this analysis is 1 g/L, the laboratory reports total alpha activity in units of $\mu$Ci/g. The 1 g/L threshold can be converted into

$\mu$Ci/g using the HTCE model tank density of 1.66 g/mL, which produces a result of 37 $\mu$Ci/g. The TCP, however, assumed a density of 1.5 g/mL, which produced a notification limit of 41 $\mu$Ci/g. Regardless of which limit is used, all of the total alpha activity results were far below the limits. The mean total alpha activity for tank 241-B-101 was found to be 2.91 $\mu$Ci/g, as is shown in Table 5-3. Table C-3 in Appendix C shows the upper limits for the 95 percent confidence intervals calculated based on the total alpha activity results. By comparing these upper limits to the TCP notification limit of 41 $\mu$Ci/g, it is seen that the highest value is less than half of the notification limit, indicating that tank 241-B-101 does not pose a criticality concern.

Tank headspace flammable gases were measured in the field by means of a combustible gas monitor. The measurement was performed while sampling at a depth of 10 m (33 ft) through riser 2 and yielded a result of 0 percent of the LFL (WHC 1996).

Another factor in assessing the safety of the tank waste is the heat generation and waste temperature. Heat is generated in the tanks from radioactive decay. An estimate of the tank heat load from the 1995 data was not possible because the primary heat-producing radionuclides, $^{137}Cs$ and $^{90}Sr$, have not yet been evaluated; these determinations are pending and the results will be reported in a future revision of this report. Radiochemical results are available from the 1976 sampling event, but a single sample from a single, unknown point in the tank provides insufficient information to make a heat load estimate for the entire tank.

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# 6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-B-101 has been sampled and analyzed for the purposes of safety screening in accordance with the requirements listed in the *Tank Safety Screening Data Quality Objective* (Babad et al. 1995). The June 1995 push-mode core sampling effort is the most recently recorded sampling event for this tank. As mandated by the safety screening DQO, analyses for percent water, energetics, and total alpha activity were performed. As part of the evaluation of the percent water measurement, analyses for lithium and bromide were performed in order to detect any contamination by the hydrostatic head fluid. In the process of measuring the bromide concentration, the concentrations of several other anions were determined. These concentrations were used to calculate inventory estimates for the anions. Further analyses are pending, and the additional data will be included in a later revision of this report. The concentrations and inventories of other waste constituents were taken from the Historical Tank Content Estimate (Brevick et al. 1994a).

All analytical results satisfied the requirements of the DQO with the exception of the percent water content of several of the segment portions. Percent water determinations of segment subsamples from both cores yielded results near or below the tank safety screening DQO limit of 17 wt%. Repeat determinations of percent water were performed for those samples with low moisture values using both TGA and gravimetry. While several inconsistencies occurred between samples and duplicates, between initial and repeat determinations, and between the TGA and gravimetric methods, the observed inconsistencies did not substantially alter the data interpretation. In order to further evaluate the percent water results, additional measurements were performed by both gravimetry and thermogravimetric analysis. Difficulties in homogenization were hypothesized to cause most of the inconsistencies. After reanalysis and repeat sampling, the low percent water content of one segment subsample, core 90, segment 2, upper half, was reported to cognizant safety program personnel. Although the percent water content for some segment subsamples were below the DQO limit, the violations were not deemed a safety concern because all differential scanning calorimetry results were well within safety screening limits (Schreiber 1995c). All total alpha activity results were also within the DQO limit.

A flammable gas result of 0 percent of the LFL was obtained during the sampling event of March 26, 1996. The gas sampling was performed within the tank headspace at a depth of 10 m (33 ft) through riser 2; therefore the flammable gas results meet the requirements of the tank safety screening DQO.

Hydrostatic head fluid with a lithium bromide tracer was used to obtain the push-mode core samples, and contamination was detected in six of the subsamples. The bromide concentrations were used to correct the percent water results for the contamination. However, during the bromide analysis by ion chromatography, the nitrate and nitrite concentration levels made it necessary to dilute the samples to a point at which the bromide concentrations had to be estimated by linear extrapolation of the calibration curve.

Radiochemical data from the 1995 sampling event are not yet available to calculate a theoretical heat load for tank 241-B-101. When the radiochemical data become available, a theoretical heat load will be calculated and presented in the next revision of this report. The historical temperature data for tank 241-B-101 do not indicate a significant problem with heat generation in this tank.

The waste exhibited heterogeneity both vertically and horizontally. Because of the limited number of analyses performed at this time, a definite validation of the TLM and the HTCE estimates could not be performed. Results from the pending analyses will allow validation in the future.

Based on the recent safety screening analyses, certain tank 241-B-101 subsegments did not meet the tank safety screening DQO criterion of 17 wt% water; however, no other tank safety screening DQO criteria, including energetics, were violated.

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# APPENDIX A

# EXTRUSION PHOTOGRAPHS FROM 1995 CORE SAMPLES

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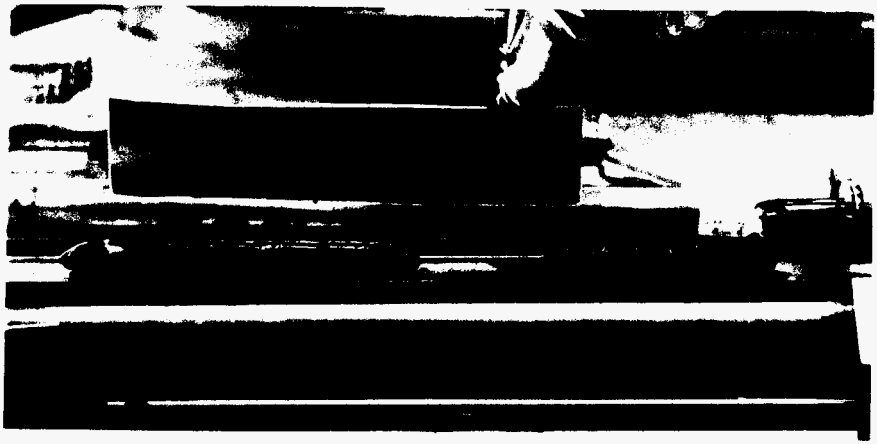
Figure A-1. Tank 241-B-101 Core 90 Segment 1 and 2 Extrusion Photograph.

Segment 1



Segment 2

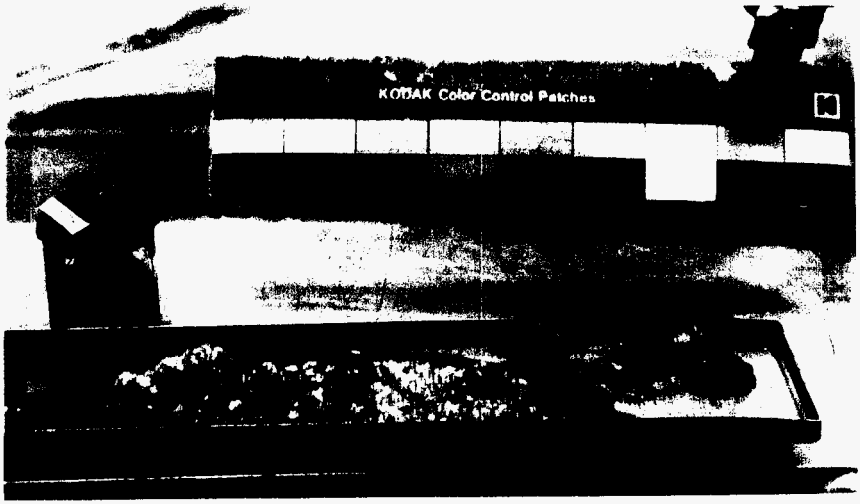
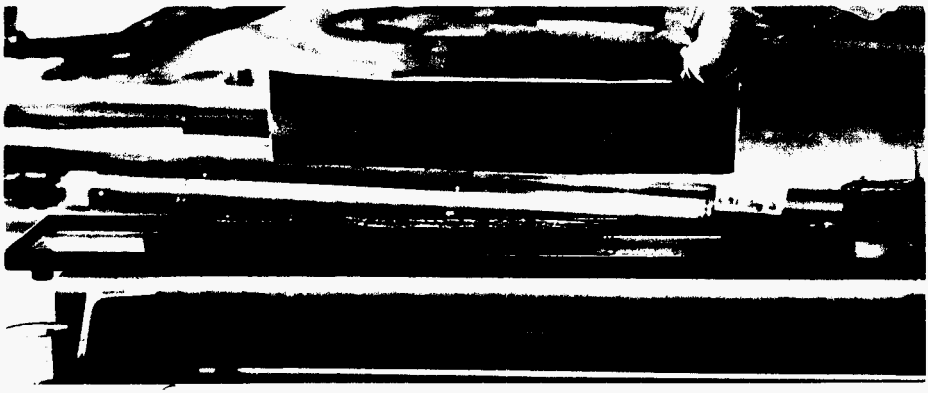
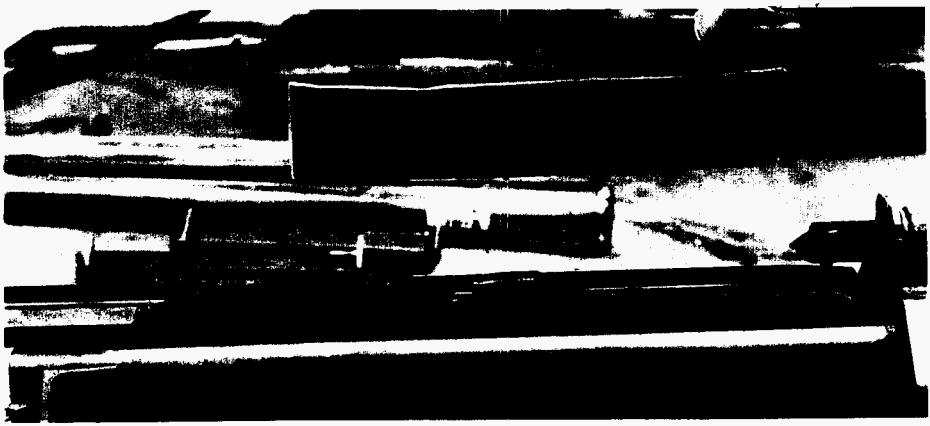


Figure A-1. Tank 241-B-101 Core 91 Segment 1 and 2 Extrusion Photograph.

Segment 1



Segment 2



# APPENDIX B

# HISTORICAL ANALYTICAL RESULTS

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**NOTE:** The data presented in this appendix are for information only. Analytical and physical property data generated prior to May, 1989 are not validated and may not be used for decision-making purposes.

Table B-1. Tank 241-B-101 1976 Sludge Analytical Results.[1,2]

| **Physical properties** | | |
|---|---|---|
| Bulk density | 1.59 g/mL | |
| Percent water | 20.1% | |
| **Analyte** | **Concentration** | |
| **Metals** | **mol/L** | **μg/g** |
| Al | 4.95 | 84,000 |
| Ba | < 0.05 | < 4,320 |
| Ca | < 0.24 | < 6,050 |
| Fe | 1.3 | 45,700 |
| Mg | 0.05 | 760 |
| Mn | 0.13 | 4,490 |
| Si | 0.05 | 900 |
| Pu | 0.0188 g/L | 11.8 |
| **Anions** | **mol/L** | **μg/g** |
| $NO_3^-$ | 22.1 | 8.62E+05 |
| $PO_4^{3-}$ | 1.7 | 1.02E+05 |
| **Radionuclides** | **Ci/L** | **μCi/g** |
| $^{89/90}Sr$ | 2.5 | 1,570 |
| $^{137}Cs$ | 0.67 | 421 |

Notes:

[1]Brevick et al. (1994b)

[2]Horton (1976)

Table B-2. Tank 241-B-101 1976 Particle Size Distribution.[1]

| Particle size ($\mu$m) | Average diameter ($\mu$m) | Weight percent |
|---|---|---|
| 5 | 8.25 | 99.98 |
| 10 | 16.5 | 99.6 |
| 20 | 25.9 | 44.9 |
| 30 | 35.7 | 24.7 |
| 40 | 45.5 | 24.4 |
| 50 | 60.4 | 18.03 |
| 70 | 75.3 | 9. |
| 80 | 85.3 | 6.2 |
| 90 | 95.3 | 3.5 |
| 100 | 105.2 | 1.66 |

Note:

[1]This table is reproduced from Horton (1976) exactly as found. No text or specific explanations were given.

# APPENDIX C

# CONFIDENCE INTERVAL TEST RESULTS

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Appendix C lists the 95 percent confidence limits for DSC, percent water, and total alpha activity. The number of measurements and standard deviation for a given sample are also included, as they are needed to mathematically derive the 95 percent confidence limits. All uncertainty values are standard deviations of the data with the exception of the single DSC sample and the three percent water samples in which four individual results went into the calculation. In these cases, the correct terminology is standard deviation of the mean.

Table C-1. 95% Confidence Interval Upper Limits for DSC (joules/gram dry).[1]

| Sample Number | Core | Segment | Mean | Number of Measurements | Standard Deviation (Data) | Upper Limit |
|---|---|---|---|---|---|---|
| S95T001214 | 90 | Segment 1, UH | 275.00 | 2 | 14 | 363.4 |
| S95T001217 | | Segment 1, LH | 317.50[2] | 4 | 19.3[3] | 439.1 |
| S95T001220 | | Segment 1, facies | 0.00 | 2 | 0.00 | 0.00 |
| S95T001229 | | Segment 2, UH | 0.00 | 2 | 0.00 | 0.00 |
| S95T001551 | | Segment 2, LH | 0.00 | 2 | 0.00 | 0.00 |
| S95T001223 | | Segment 2, DL | 52.00 | 2 | 0.00 | 52.00 |
| S95T001238 | 91 | Segment 1, UH | 249.50 | 2 | 1.5 | 258.97 |
| S95T001235 | | Segment 1, LH | 42.5 | 2 | 0.5 | 45.66 |
| S95T001244 | | Segment 2, UH | 5.00 | 2 | 0.00 | 5.00 |
| S95T001241 | | Segment 2, LH | 0.00 | 2 | 0.00 | 0.00 |
| S95T001555 | | Segment 2, facies | 0.00 | 2 | 0.00 | 0.00 |
| S95T001247 | | Segment 2, DL | 6.00 | 2 | 0.00 | 6.00 |

Notes:

UH = upper half
LH = lower half
DL = drainable liquid

[1]Schreiber (1995a)

[2]Mean includes all four results.

[3]Standard deviation of the mean.

Table C-2. 95% Confidence Interval Lower Limits for Percent Water.[1]

| Sample Number | Core | Segment | Mean[2] | Number of Measurements | Standard Deviation (Data) | Lower Limit[2] |
|---|---|---|---|---|---|---|
| S95T001214 | 90 | Segment 1, UH | 33.08 | 2 | 0.01 | 33.02 |
| S95T001217 | | Segment 1, LH | 42.00 | 2 | 0.49 | 38.91 |
| S95T001220 | | Segment 1, facies | 18.74 | 2 | 0.0055 | 18.71 |
| S95T001229 | | Segment 2, UH | 15.55 (10.4) | 2 | 0.87 (0.911) | 10.09 (4.65) |
| S95T001229 S95T002532 | | | 14.06 (8.85) | 4 | 1.50 (0.996)[3] | 4.58 (2.56)[4] |
| S95T001551 | | Segment 2, LH | 19.86 | 2 | 1.11 | 12.85 |
| S95T001551 S95T002533 | | | 34.03 | 4 | 5.45[3] | 0.00[4,5] |
| S95T001223 | | Segment 2, DL | 49.52 (47.4) | 2 | 0.1 (0.111) | 48.83 (46.7) |
| S95T001238 | 91 | Segment 1, UH | 34.10 | 2 | 0.62 | 30.19 |
| S95T001235 | | Segment 1, LH | 42.91 (40.4) | 2 | 1.22 (1.27) | 35.17 (32.4) |
| S95T001244 | | Segment 2, UH | 45.17[6] (40.8) | 3 | 3.30 (4.99) | 24.32 (9.30) |
| S95T001241 | | Segment 2, LH | 22.70 | 2 | 4.57 | 0.00[5] |
| S95T001555 | | Segment 2, facies | 18.02 (15.3) | 2 | 0.458 (0.507) | 15.12 (12.1) |
| S95T001555 S95T002534 | | | 15.21 (13.9) | 4 | 1.406 (0.998)[3] | 7.74 (7.60)[4] |
| S95T001247 | | Segment 2, DL | 50.47 (48.0) | 2 | 0.4 (0.428) | 47.97 (45.3) |

Notes:

UH = upper half
LH = lower half
DL = drainable liquid

[1]Schreiber (1995c)
[2]Values in parentheses were corrected for HHF contamination.
[3]Standard deviation of the mean.
[4]Lower limit of the 95% confidence interval on the mean was based on the two pair of primary and duplicate sample results.
[5]Result was negative, so reported as zero.
[6]Mean includes triplicate result.

Table C-3. 95% Confidence Interval Upper Limits for Total Alpha Activity ($\mu$Ci/g).[1]

| Sample Number | Core | Segment | Mean | Number of Measurements | Standard Deviation (Data) | Upper Limit |
|---|---|---|---|---|---|---|
| S95T001218 | 90 | Segment 1, UH | 4.57 | 2 | 0.223 | 5.96 |
| S95T001221 | | Segment 1, LH | 1.89 | 2 | 0.063 | 2.27 |
| S95T001215 | | Segment 1, facies | 0.16 | 2 | 0.045 | 0.41 |
| S95T001230 | | Segment 2, UH | 0.24 | 2 | 0.01 | 0.30 |
| S95T001552 | | Segment 2, LH | 0.03 | 2 | 0.0045 | 0.05 |
| S95T001236 | 91 | Segment 1, UH | 4.15 | 2 | 0.490 | 7.24 |
| S95T001236 | | Segment 1, LH | 14.00 | 2 | 0.2 | 15.26 |
| S95T001245 | | Segment 2, UH | 0.00 | 2 | 0.00 | 0.00 |
| S95T001237 | | Segment 2, LH | 0.45 | 2 | 0.1 | 0.96 |
| S95T001556 | | Segment 2, facies | 0.18 | 2 | 0.0077 | 0.23 |

Notes:

UH = upper half
LH = lower half
DL = drainable liquid

[1]Schreiber (1995c)

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| M. L. Dexter | R1-51 | X | | | |
| J. G. Douglas | L5-55 | X | | | |
| G. L. Dunford | S7-81 | X | | | |
| D. B. Engelman | R1-49 | X | | | |
| G. D. Forehand | S7-21 | X | | | |
| J. S. Garfield | H5-49 | X | | | |
| * J. D. Guberski | R2-06 | | | | X |
| D. L. Herting | T6-09 | X | | | |
| N. A. Homan | H6-25 | X | | | |
| J. W. Hunt | R2-12 | X | | | |

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| Ogden Environmental<br>101 East Wellsian Way<br>Richland, WA 99352 | | | | | |
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| Sandia National Laboratory<br>P.O. Box 5800<br>MS-0744, Dept. 6404<br>Albuquerque, NM 87815 | | | | | |
| D. Powers | | X | | | |
| Nuclear Consulting Services Inc.<br>P. O. Box 29151<br>Columbus, OH 43229-01051 | | | | | |
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| Chemical Reaction Sub-TAP<br>P.O. Box 271<br>Lindsborg, KS 67456 | | | | | |
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| Tank Characterization Panel<br>Senior Technical Consultant<br>Contech<br>7309 Indian School Road<br>Albuquerque, NM 87110 | | | | | |
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| U. S. Department of Energy - Headquarters<br>Office of Environmental Restoration and Waste Management EM-563<br>12800 Middlebrook Road<br>Germantown, MD 20874 | | | | | |
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| T. J. Kelley | S7-21 | X | | | |
| N. W. Kirch | R2-11 | X | | | |
| M. J. Kupfer | H5-49 | X | | | |
| D. L. McGrew | R3-25 | X | | | |
| J. E. Meacham | S7-15 | X | | | |
| W. C. Miller | R1-30 | X | | | |
| C. T. Narquis | T6-16 | X | | | |
| R. J. Nicklas | R1-43 | X | | | |
| D. E. Place | H5-27 | X | | | |
| D. A. Reynolds | R2-11 | X | | | |
| L. M. Sasaki | R2-12 | X | | | |
| F. A. Schmittroth | H0-35 | X | | | |
| R. D. Schreiber | R2-12 | X | | | |
| N. J. Scott-Proctor | S5-01 | X | | | |
| L. W. Shelton, Jr. | H5-49 | X | | | |
| B. C. Simpson | R2-12 | X | | | |
| G. L. Troyer | T6-50 | X | | | |
| D. A. Turner | S7-15 | X | | | |
| D. J. Washenfelder | H5-27 | X | | | |
| M. S. Waters | S6-30 | X | | | |
| L. R. Webb | T6-06 | X | | | |
| K. A. White | S5-13 | X | | | |
| TFIC (Tank Farm Information Center) | R1-20 | X | | | |
| Central Files | A3-88 | X | | | |
| EDMC | H6-08 | X | | | |
| ERC (Environmental Resource Center) | R1-51 | X | | | |
| TCRC (10) | R2-12 | X | | | |

* Dope