Accession #: D196071598

Document #: SD-WM-ER-535

Title/Desc:
TANK CHARACTERIZATION REPORT FOR SST 241-BX-103

Pages: 84

APR 03 1996 (2)

ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT № 615370

2. To: (Receiving Organization) Distribution	3. From: (Originating Organization) Data Assessment and Interpretation	4. Related EDT No.: N/A
5. Proj./Prog./Dept./Div.: Tank 241-BX-103/Waste Management/DAI/Characterization Technical Basis	6. Cog. Engr.: Kevin E. Bell	7. Purchase Order No.: N/A
8. Originator Remarks: This document is being released into the supporting document system for retrievability purposes.		9. Equip./Component No.: N/A
		10. System/Bldg./Facility: 241-BX-103
11. Receiver Remarks: For release.		12. Major Assm. Dwg. No.: N/A
		13. Permit/Permit Application No.: N/A
		14. Required Response Date: 04/03/96

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	WHC-SD-WM-ER-535	N/A	0	Tank Characterization Report for Single-Shell Tank 241-BX-103	N/A	2	1	1

16. KEY

Approval Designator (F)	Reason for Transmittal (G)	Disposition (H) & (I)
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

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(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.
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		QA		4/3/96							
		Safety									
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18.	19.	20.	21. DOE APPROVAL (if required)
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BD-7400-172-2 (04/94) GEF097

BD-7400-172-1

Tank Characterization Report for Single-Shell Tank 241-BX-103

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U.S. Department of Energy Contract DE-AC06-87RL10930

EDT/ECN: EDT-615370 UC: 2070
Org Code: 79400 Charge Code: N4G4D
B&R Code: EW 3120074 Total Pages: 78

Key Words: Tank Characterization Report, TCR, Single-Shell, SST, Tank 241-BX-103, Tank BX-103, BX-103, BX Farm

Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-BX-103. This report supports the requirements of Tri-Party Agreement Milestone M-44-09.

4/3/96

Release Approval Date

DATE: HANFORD RELEASE
STA: 4 ID: 2
APR 03 1996

Release Stamp

Approved for Public Release

Tank Characterization Report for Single-Shell Tank 241-BX-103

K. E. Bell

Date Published
April 1996

Prepared for the U.S. Department of Energy
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Management and Operations Contractor for the
U.S. Department of Energy under Contract DE-AC06-87RL10930

EXECUTIVE SUMMARY

This tank characterization report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell, underground storage tank 241-BX-103. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994), Milestone M-44-09.

Tank 241-BX-103 is one of 12 single-shell underground waste storage tanks located in the 200 East Area BX Tank Farm on the Hanford Site. It is the third tank in a cascade and is connected to tanks 241-BX-101 and 241-BX-102. Tank 241-BX-103 went into service in 1948. It received metal waste from the bismuth phosphate process (Agnew et al. 1994) and was full by January 1949. The tank was sluiced in 1954 and declared empty in July of that year (Rodenhizer 1987). It was filled with uranium recovery waste from tank 241-BY-108 during 1956; most of the waste in the tank was pumped to ditches during 1956 and 1957. In 1962, tank 241-BX-103 received PUREX cladding and organic wash wastes from tanks in the 241-C Tank Farm. From 1970 to 1976, it received wastes from the PUREX and B plants and other sources through tanks in the 241-B and -BX Tank Farms. During this time, discharges were directed to tanks in the 241-B, -BY, -BX, -C, -S, and -SX Tank Farms. After the final discharge in 1976, the tank contained about 598 kL of waste. It was saltwell pumped in 1977 and administratively stabilized in November 1983. Intrusion prevention was completed in October 1985; however, because of recent liquid level increases, the tank no longer meets the criteria for interim stabilization. To date, tank 241-BX-103 is classified as sound.

A description and status of tank 241-BX-103 are summarized in Table ES-1 and Figure ES-1. The tank, which has an operating capacity of 2,010 kL, contains 23 kL of supernate and 235 kL of sludge as of January 31, 1996. It was last core sampled in May 1995 and is not on any Watch List.

Although this report summarizes three sampling and analysis events, it focuses on the sampling and analysis events in May 1995 and March 1996, which were performed to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (DQO) (Babad and Redus 1994). Two cores, 86 and 87, were obtained using the push-mode core sampling method. In addition, flammability measurements were performed on the tank headspace in March 1996. The core samples were analyzed for moisture, energetics, and total alpha activity.

Because of the limited number of analyses performed on the recovered waste, estimates of the chemical and radionuclide composition have been taken from the *Tank Layer Model* (Agnew et al. 1995) and are summarized in Table ES-2. Four sludge samples and none of the drainable liquid samples showed exotherms in the differential scanning calorimetry (DSC) scans. The highest exotherm measured 161 J/g and was found on the upper half sludge sample from segment 1 of core 86. Calculated on a dry-weight basis, the result becomes 423 J/g, which is below the safety criterion of 480 J/g.

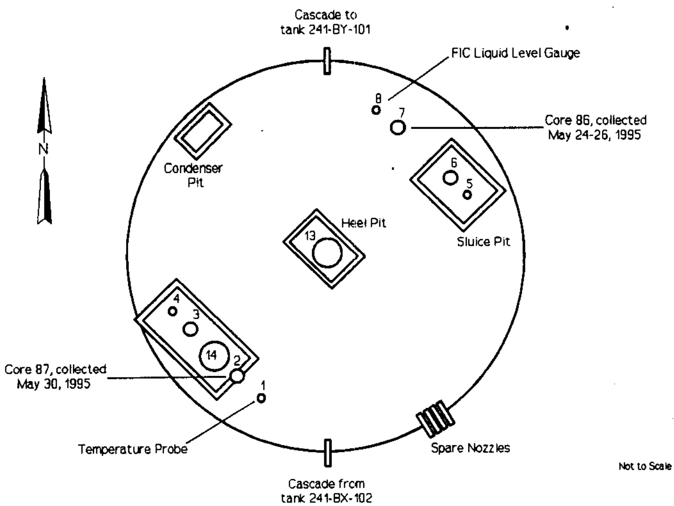
Table ES-1. Description and Status of Tank 241-BX-103.

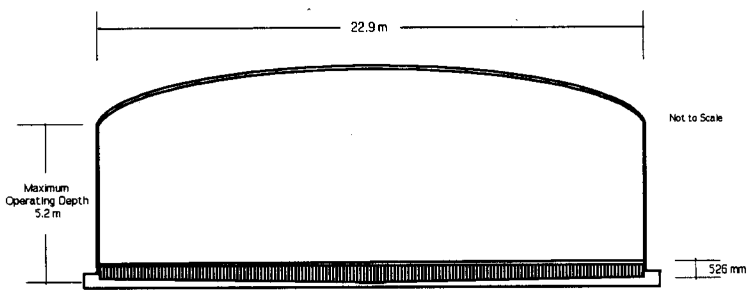
TANK DESCRIPTION	
Type	Single-shell
Constructed	1946-1947
In service	1948
Diameter	22.9 m
Operating depth	5.2 m
Capacity	2,010 kL
Bottom shape	Dish
Ventilation	Passive
TANK STATUS	
Total waste volume	258 kL
Sludge volume	235 kL
Supernate volume	23 kL
Waste surface level (April 1, 1996)	53.10 cm
Temperature (March 31, 1996)	16 to 19.4 °C
Integrity	Sound
Watch List status	None
SAMPLING DATES	
Supernate grab sampled (5 samples)	November 1974 - December 1975
Push-mode core sampled	May 1995
Headspace vapor samples	March 1996
SERVICE STATUS	
Out of service	1977
Intrusion prevention	1985[1]
Interim stabilized	1977

Note:

[1]The tank no longer meets the criteria for intrusion prevention because of recent liquid level increases.

Figure ES-1. Tank 241-BX-103 Configuration and Waste Profile.





Total Tank Volume: 2,010 kL
Waste Volume (August 1995): 257 kL
Sludge Volume (August 1995): 235 kL
Supernate Volume (August 1995): 23 kL

Table ES-2. Description and Status of Tank 241-BX-103.[1] (2 sheets)

Solids Composite Inventory Estimate[2]			
Physical Properties			
Total solid waste	3.54E+05 kg		
Heat load	132 W		
Bulk density	1.51 g/mL		
Void fraction	0.522		
Water wt%	59.0		
Total organic carbon	0% C		
Chemical Constituents			
Analyte	**Concentration (mol/L)**	**(µg/g)**	**Inventory (kg)**
Na^{+}	4.38	66,800	23,600
Al^{3+}	0.756	13,500	4,790
Fe^{3+} (total Fe)	0.447	16,600	5,860
Cr^{3+}	0.00441	152	53.8
Bi^{3+}	0	0	0
La^{3+}	0	0	0
Ce^{3+}	0	0	0
Zr (as $ZrO(OH)_2$)	0	0	0
Pb^{2+}	0	0	0
Ni^{2+}	0.0276	1,070	380
Sr^{2+}	0	0	0
Mn^{4+}	0	0	0
Ca^{2+}	0.0613	1,630	577
K^{+}	0	0	0
OH^{-}	9.23	1.04E+05	36,800
NO_3^{-}	0.191	7,860	2,780
NO_2^{-}	0.0280	854	302
CO_3^{2-}	0.703	28,000	9,900
PO_4^{3-}	0.412	26,000	9,180
SO_4^{2-}	0.787	50,200	17,700
Si (as SiO_3^{2-})	0.00180	33.6	11.9
F^{-}	0	0	0

Table ES-2. Description and Status of Tank 241-BX-103.[1] (2 sheets)

Solids Composite Inventory Estimate[2]			
Chemical Constituents (Continued)			
Analyte	**Concentration (mol/L)**	**Concentration (μg/g)**	**Inventory (kg)**
Cl^-	0.00354	83.3	29.5
$C_6H_5O_7^{3-}$	0	0	0
$EDTA^{4-}$	0	0	0
$HEDTA^{3-}$	0	0	0
NTA^{3-}	0	0	0
Glycolate$^-$	0	0	0
Acetate$^-$	0	0	0
Oxalate^{2-}	0	0	0
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	**Activity (Ci/L)**	**Activity (μCi/g)**	**Inventory (Ci)**
Pu	—	0.169	0.993 (kg)
U	0.916 (mol/L)	1.45E+05 (μg/g)	51,200 (kg)
Cs	0.00101	0.673	238
Sr	0.0829	55.0	19,400

Notes:

C = carbon
EDTA = ethylenediaminetetraacetic acid
HEDTA = N-(hydroxyethyl)-ethylenediaminetriacetic acid
NTA = nitrilotriacetic acid
DBP = dibutyl phosphate
NPH = normal paraffin hydrocarbon

[1]Agnew et al. (1995)

[2]These composite inventory data are estimated and cannot be used to make decisions. There are small (< 3 percent) differences between the analyte concentrations and the calculated inventories. This is currently being investigated.

An upper limit to a one-sided 95 percent confidence interval on the mean of 487 J/g was found for the lower half sludge from segment 1 of core 87. Thus, although no notification limits were exceeded on individual samples (Bell 1995b), the safety screening decision criterion at the 95 percent confidence limit was not met for energetics on the lower half segment portion of segment 1 of core 87. Values for percent water on sludge samples from the 1995 core samples ranged from 22.14 to 63.17 percent. The safety screening criterion was 17 percent. The percent water of the drainable liquid samples was in the 70 to 80 percent range. The total alpha results indicate that plutonium concentration is well below the safety screening criterion of 1 g/L. The highest total alpha activity measured was 5.17 μCi/g.

Based on analytical results, the tank appears to pose no safety concerns despite the energetics results on the lower half sludge from segment 1 of core 87. Analyses show that there is sufficient moisture in that sample to mediate the energetics concern according to the safety screening DQO. The tank headspace vapor flammability was determined to be 0 percent of the lower flammability limit (LFL) (WHC 1996). These results satisfy the safety screening DQO requirement that tank headspace flammability be $<$ 25 percent of the LFL. Tank headspace flammability was measured in the field in March 1996 on vapor samples drawn from below riser 7. The safety screening DQO also requires two full vertical profiles of the tank waste from risers as widely spaced as practical. This criterion was not strictly met for tank 241-BX-103 because no sample was obtained from the lower half of segment 2, core 87. Nevertheless, the safety screening data, obtained on the samples collected, do not indicate the presence of any safety concerns and support the non Watch List status of tank 241-BX-103.

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ANOVA	analysis of variance
C	Celsius
Ci	curies
cm	centimeter
DQO	data quality objective
DSC	differential scanning calorimetry
FIC	Food Instrument Corporation
g	gram
HDW	Hanford Defined Wastes
HTCE	historical tank content estimate
J	Joule
kg	kilogram
kL	kiloliter
L	liter
LFL	lower flammability limit
m	meter
mg	milligram
mL	milliliter
mm	millimeter
mol	moles
RPD	relative percent difference
TGA	thermogravimetric analysis
TLM	Tank Layer Model
W	Watts
WSTRS	Waste Status and Transaction Record Summary
wt%	weight percent
μCi	microcuries
μg	micrograms

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1.0 INTRODUCTION

This tank characterization report presents an overview of single-shell tank 241-BX-103 and its waste contents. It provides estimated concentrations and inventories for the waste components based on the latest sampling and analysis activities and background tank information, and it supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1994).

Tank 241-BX-103 began operation in 1948 and received waste until it was removed from service in 1977. Interim stabilization and intrusion prevention of the tank were completed in 1983 and 1985, respectively; therefore, the composition of the waste is not expected to 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.

1.1 PURPOSE

The purpose of this report is to summarize the information about the use and contents of tank 241-BX-103. 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-BX-103.

1.2 SCOPE

This characterization report presents the results of earlier supernate sampling and the only sampling events pertinent to the current contents of the tank, a core sampling event in May 1995 and tank headspace vapor measurements in March 1996. These sampling events supported the evaluation of the tank waste according to the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The data presented here will be evaluated against a more recent revision of the safety screening DQO document (Hunt 1995). From the two core samples, three analyses were performed as directed in the *Tank 241-BX-103 Tank Characterization Plan* (Bell 1995b). The safety screening analyses performed were differential scanning calorimetry (to evaluate fuel level and energetics), thermogravimetric analysis (to determine moisture content), total alpha activity analysis (to evaluate criticality potential) and percent of the Lower Flammability Limit (LFL) of the tank headspace. The results of these analyses are used to categorize a tank as "safe" or to identify it with a specific safety issue.

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2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-BX-103 based on historical information. The first part details the current condition of the tank. This is followed by a discussion 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 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 the section summarizes available surveillance data for the tank.

2.1 TANK STATUS

According to Hanlon (1996), tank 241-BX-103 contained 258 kL of noncomplexed waste as of January 31, 1996. The amounts of waste in the tank are shown in Table 2-1.

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

Waste Form	Volume (kiloliters)
Total waste	258
Sludge	235
Supernatant	23
Drainable interstitial liquid	0
Saltcake	0
Pumpable liquid remaining	0

Note:
[1]Hanlon (1996)

Tank 241-BX-103 is identified as a sound tank. Interim stabilization and intrusion prevention have been completed; however, the tank no longer meets the criteria for interim stabilization because of ongoing intrusions since 1986 (Hanlon 1996). The source of the intrusions is unknown, but may be a result of rainwater intrusions around one or more risers. A specific plan to identify and mediate the intrusions is contained in the *Single-Shell Waste Tank Intrusion Resolution Plan* (Huber 1994). Funds are available in the fiscal year 1996 Multi-Year Program Plan to identify the source of the intrusions. Tank 241-BX-103 is not on any Watch List. It was declared inactive in 1977 and is out of service. All tank monitoring systems were in compliance with documented standards as of January 31, 1995.

2.2 TANK DESIGN AND BACKGROUND

Tank 241-BX-103, located in the 200E Area of the Hanford Site, was constructed between 1946 and 1947. It is one of 12 100-series tanks in the 241-BX Tank Farm and began operations in September 1948. The tank has a capacity of 2,010 kL, a diameter of 22.9 m, and an operating depth of 5.2 m. Built as first-generation tanks, tanks in the BX Tank Farm were designed for nonboiling waste with a maximum fluid temperature of 104 °C. Tank 241-BX-103 is the last tank in a three-tank cascade that also includes tanks 241-BX-101 and 241-BX-102. The cascade overflow height is approximately 4.86 m from the tank bottom and 60 cm below the top of the steel liner.

Similar to other single-shell tanks, tank 241-BX-103 was constructed with a primary mild steel liner and a concrete dome with various risers. It has a dished bottom with a 1.2 m radius knuckle. The tank is set on a reinforced concrete foundation; the concrete tank dome is approximately 2.6 m below grade.

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. Photographic evaluation is used in determining liquid volume. A plan view of the riser configuration for tank 241-BX-103 is shown in Figure 2-1. Risers, ranging in diameter from 10 to 107 cm, and process nozzles are described in Table 2-2. Figure 2-2 shows a tank cross section with the approximate waste level and a schematic of tank equipment.

2.3 PROCESS KNOWLEDGE

This section provides information on the transfer history of tank 241-BX-103, describes the process wastes that made up the transfers, and estimates current tank contents based on transfer history.

2.3.1 Waste Transfer History

The waste transfer history information was gathered from Anderson (1990), Agnew et al. (1994), and Brevick et al. (1994). A summary of the major waste transfers is shown in Table 2-3.

The cascade lines for the cascade series that includes tank 241-BX-103 were opened during the first quarter of 1948 (Agnew et al. 1994). Metal waste from the bismuth phosphate process began cascading into tank 241-BX-103 from tank 241-BX-102 during September 1948. Transfers of metal waste to and from the cascade series continued until the first

Figure 2-1. Tank 241-BX-103 Riser Configuration

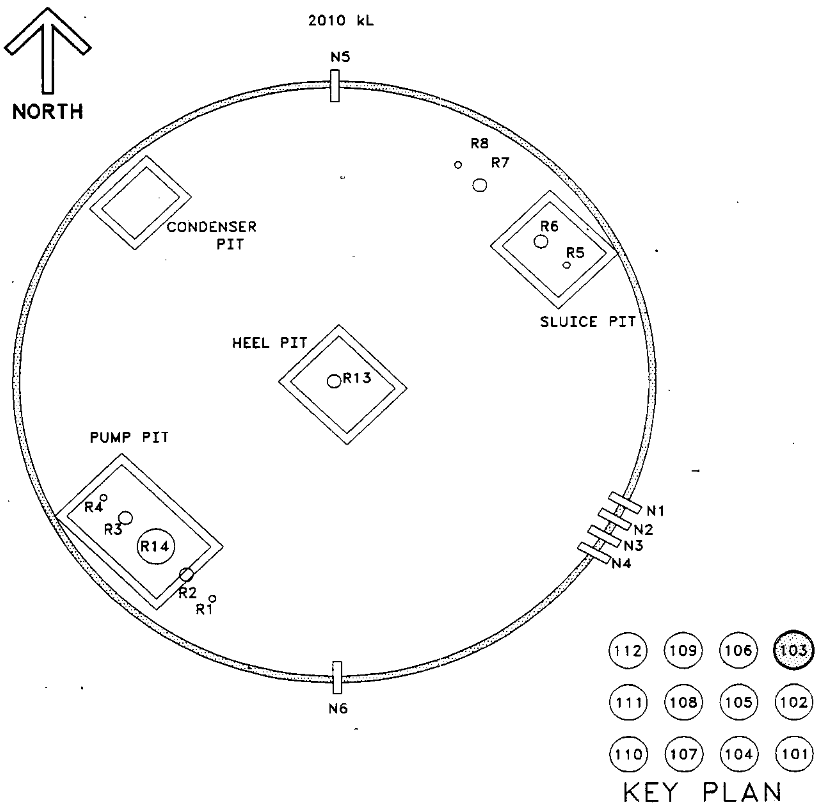


Table 2-2. Tank 241-BX-103 Risers and Nozzles.[1]

Riser Number	Diameter (Centimeters)	Description and Comments
1	10.2	Thermocouple tree
2	30.5	Flange/breather filter, G1 housing
3	30.5	Weather covered (sluicing nozzle)
4	10.2	Weather covered (recirculating dip tube)
5	10.2	Weather covered (recirculating dip tube)
6	30.5	Weather covered (sluicing nozzle)
7	30.5	Flange/B-222 observation port
8	10.2	Food Instrument Corporation gauge (bench mark)
13	107	Weather covered (heel jet)
14	107	Weather covered (manhole)
Nozzle Number	**Diameter (centimeters)**	**Description and Comments**
N1	37.62	Spare
N2	37.62	Spare
N3	37.62	Spare
N4	37.62	Spare
N5	37.62	Overflow outlet
N6	37.62	Inlet

Note:
[1]Alstad (1993), Agnew et al. 1995, and U.S. DOE (1986)

Figure 2-2. Tank 241-BX-103 Cross Section.

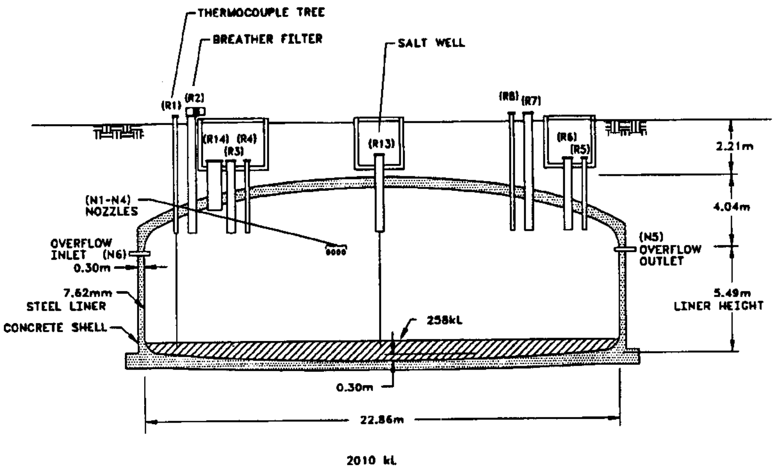


Table 2-3. Summary of Major Tank 241-BX-103 Waste Transfers.[1, 2]

Transfer Source	Waste Type Received	Time Period	Waste Volume Transferred (kiloliters)
from 241-BX-102	Metal waste	1948-1950	2,600
to U-Plant	Metal waste	1954	(2,100)
from 241-BY-108	Uranium recovery waste	1956	1,800
from 241-C-102	PUREX cladding and organic wash wastes	1962-1969	25,200
to 241-BX, B, BY farms	Supernatant	1968-1970	(34,900)
from 241-C-104	PUREX cladding and organic wash wastes	1969-1972	34,100
to 241-BY, SX, C farms	Supernatant	1970-1972	(14,900)
from 241-B-101	B Plant low-level waste, evaporator bottoms	1970-1975	2,160
from 241-BX-101	B Plant low-level and cesium recovery wastes, PUREX cladding and organic wash wastes, REDOX ion exchange waste	1970-1972	31,200
from 241-BX-105	Ion exchange waste	1972-1973	3,140
from 241-BX-106	B Plant low-level waste, PUREX cladding and organic wash wastes, REDOX ion exchange waste	1972-1973	1,910
from 241-C-104	Pacific Northwest National Laboratory wastes, PUREX cladding waste, N-Reactor and PUREX low-level wastes	1973	4,470
to 241-S, SX farms	Supernatant	1974-1976	(11,500)
from 241-BX-104	Ion exchange waste	1975-1976	9,120

Notes:

[1]Agnew et al. (1994)

[2]Anderson (1990)

quarter of 1951 when the cascade was closed. Tank 241-BX-103 remained filled with metal waste until it was sluiced for uranium recovery in 1954. It was declared empty in July of that year (Rodenhizer 1987).

In the third quarter of 1956, tank 241-BX-103 was filled with uranium recovery waste from tank 241-BY-108. During 1956 and 1957, most of the waste was pumped to ditches.

Transfer activity resumed in 1962 as tank 241-BX-103 began receiving a series of transfers of PUREX cladding and organic wash waste from tanks in the 241-C Tank Farm. From 1969 to 1976, the tank received wastes from the PUREX Plant, B Plant, and other sources through tanks 241-B-101, -BX-101, -BX-104, -BX-105, and -BX-106. During this time period, there also were frequent transfers from tank 241-BX-103 to tanks in the 241-B, -BY, -BX, -C, -S, and -SX tank farms. Approximately 598 kL of waste remained in tank 241-BX-103 after the final transfer from it in 1976.

Tank 241-BX-103 was saltwell pumped in 1977. It was administratively interim stabilized in November 1983; intrusion prevention was completed in October 1985.

2.3.2 Historical Estimate of Tank Contents

An estimate of the current contents of tank 241-BX-103 based on historical transfer data is available in the *Tank Layer Model for the Northeast, Southwest, and Northwest Quadrants* (TLM) (Agnew et al. 1995). The historical data used for the TLM prediction may be found in the *Waste Status and Transaction Record Summary for the Northeast Quadrant* (WSTRS) (Agnew et al. 1994), and *Hanford Defined Waste: Chemical and Radionuclide Compositions* (HDW) (Agnew 1995). The WSTRS document is a compilation of available waste transfer and volume status data. The HDW document provides the assumed typical compositions for Hanford Site waste types. The available data are incomplete, thereby reducing the usefulness of the transfer data and the modeling results from which it is derived. The TLM document uses WSTRS data to model the waste deposition processes, then, using additional data from HDW (which may introduce error), generates an estimate of the tank contents. Thus, these model predictions can only be considered estimates that require further evaluation using analytical data.

According to the TLM, the waste in tank 241-BX-103 exists in three layers: 203 kL of metal waste, 30 kL of unknown waste, and 15 kL of supernate. The metal waste is predicted to comprise the bottom layer, the top layer is supernate, and the middle layer is an unknown waste type, although Agnew et al. (1995) suggests that this layer is likely to be PUREX cladding waste. Metal waste sludge contains sodium, uranium, carbonate, phosphate, sulfate, and hydroxide. In addition, high concentrations of strontium and cesium should be present. The following constituents should be absent from metal waste: aluminum, iron, bismuth, nickel, lead, and organic carbon. Predicted waste constituents and concentrations for tank 241-BX-103 solids are shown in Table 2-4.

Table 2-4. Tank 241-BX-103 Historical Tank Content Estimate (2 sheets).[1]

Solids Composite Inventory Estimate[2]			
Physical Properties			
Total solid waste	3.54E+05 kg		
Heat load	132 W		
Bulk density	1.51 g/mL		
Void fraction	0.522		
Water wt%	59.0		
Total organic carbon	0% C		
Chemical Constituents			
Analyte	**Concentration**		**Inventory**
	(mol/L)	**(μg/g)**	**(kg)**
Na^+	4.38	66,800	23,600
Al^{3+}	0.756	13,500	4,790
Fe^{3+} (total Fe)	0.447	16,600	5,860
Cr^{3+}	0.00441	152	53.8
Bi^{3+}	0	0	0
La^{3+}	0	0	0
Ce^{3+}	0	0	0
Zr (as $ZrO(OH)_2$)	0	0	0
Pb^{2+}	0	0	0
Ni^{2+}	0.0276	1,070	380
Sr^{2+}	0	0	0
Mn^{4+}	0	0	0
Ca^{2+}	0.0613	1,630	577
K^+	0	0	0
OH^-	9.23	1.04E+05	36,800
NO_3^-	0.191	7,860	2,780
NO_2^-	0.0280	854	302
CO_3^{2-}	0.703	28,000	9,900
PO_4^{3-}	0.412	26,000	9,180
SO_4^{2-}	0.787	50,200	17,700
Si (as SiO_3^{2-})	0.00180	33.6	11.9
F^-	0	0	0
Cl^-	0.00354	83.3	29.5

Table 2-4. Tank 241-BX-103 Historical Tank Content Estimate (2 sheets).[1]

Solids Composite Inventory Estimate[2]			
Chemical Constituents (Continued)			
Analyte	Concentration (mol/L)	Concentration (µg/g)	Inventory (kg)
$C_6H_5O_7^{3-}$	0	0	0
$EDTA^{4-}$	0	0	0
$HEDTA^{3-}$	0	0	0
NTA^{3-}	0	0	0
$Glycolate^{-}$	0	0	0
$Acetate^{-}$	0	0	0
$Oxalate^{2-}$	0	0	0
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	Activity (Ci/L)	Activity (µCi/g)	Inventory (Ci)
Pu	—	0.169	0.993 (kg)
U	0.916 (mol/L)	1.45E+05 (µg/g)	51,200 (kg)
Cs	0.00101	0.673	238
Sr	0.0829	55.0	19,400

Notes:

C = carbon
EDTA = ethylenediaminetetraacetic acid
HEDTA = N-(hydroxyethyl)-ethylenediaminetriacetic acid
NTA = nitrilotriacetic acid
DBP = dibutyl phosphate
NPH = normal paraffin hydrocarbon

[1]Agnew et al. (1995)

[2]These composite inventory data are estimated and cannot be used to make decisions. There are small (< 3 percent) differences between the analyte concentrations and the calculated inventories. This is currently being investigated.

Although the TLM document predicts that most of the waste in tank 241-BX-103 consists of metal waste sludge and that analytical and segment extrusion data from the most recent sampling event is consistent with metal waste as the lower layer of sludge (See Section 5.4.2.), the transfer history of the tank suggests that little metal waste would remain. Historical records indicate the metal waste was removed when the tank was sluiced in 1954, and it received no further metal waste transfers (Rodenhizer 1987, Agnew et al. 1994, Anderson 1990). Based on the tank's transfer history, several different waste types may have contributed to the sludge now remaining in the tank. These include ion exchange waste, and PUREX cladding and organic wash waste.

2.4 SURVEILLANCE DATA

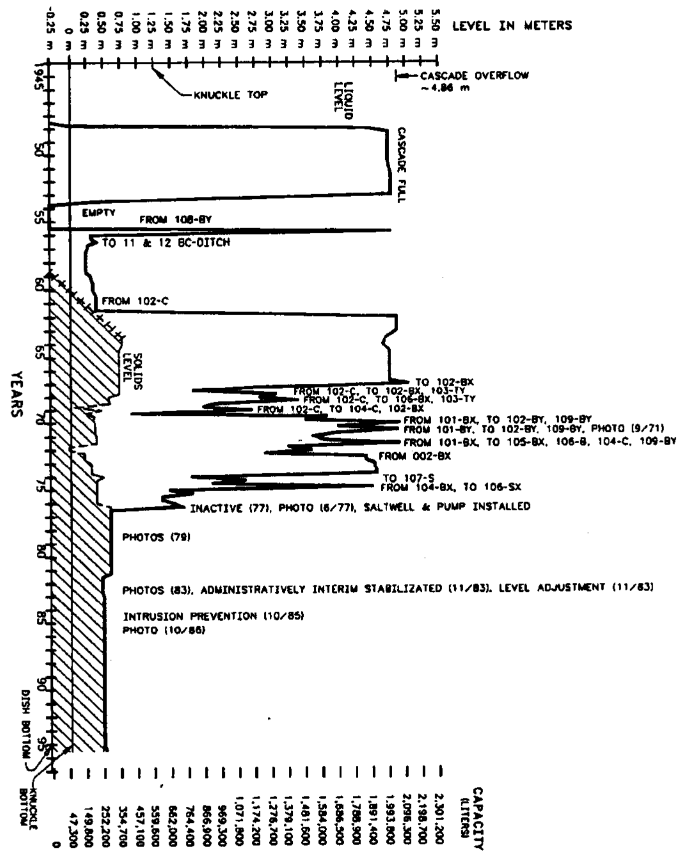
Tank 241-BX-103 surveillance consists of surface level measurements (liquid and solid), temperature monitoring inside the tank (waste and vapor space), and leak detection well (drywell) monitoring for radioactivity outside the tank. The data provide the basis for determining tank integrity. In-tank photography is another waste volume determination method used to resolve measurement anomalies and determine tank integrity.

Liquid level measurements are used to determine whether there is a leak from the tank. Solid surface level measurements indicate physical changes and consistency of the solid layers of a tank. Drywells located around the tank perimeter may detect increased radioactivity if there is a leak to the soil.

2.4.1 Surface Level Readings

The waste surface level within tank 241-BX-103 is monitored daily with an FIC gauge. A measurement of 53.1 cm was obtained on April 1, 1996. There have been erratic increases in the surface level measurements since January 1986 caused by an intrusion (Hanlon 1996). For this reason, the tank no longer meets the criteria for interim stabilization. The current leak detection baseline of 52 cm for this tank was set July 18, 1994. The surface level has remained within the 2.5 cm leak detection criterion for an increase. A graphical representation of quarterly surface level measurements from the time the tank entered service through 1995 is shown in Figure 2-3.

Figure 2-3. Tank 241-BX-103 Level History.



2.4.2 Internal Tank Temperatures

Temperature data for tank 241-BX-103 are recorded by 11 thermocouples although no elevations are known for the thermocouples (Tran 1993). The highest reading in the tank on March 31, 1996 was 19.4 °C, measured at thermocouple number 3. Plots of the individual thermocouple readings for tank 241-BX-103 can be found in Brevick et al. (1994). A graph of the weekly high temperatures recorded from 1975 to December 1995 is shown in Figure 2-4. No data were available between October 1982 and March 1994. As shown in the plot, temperatures have ranged between 16 and 33 °C.

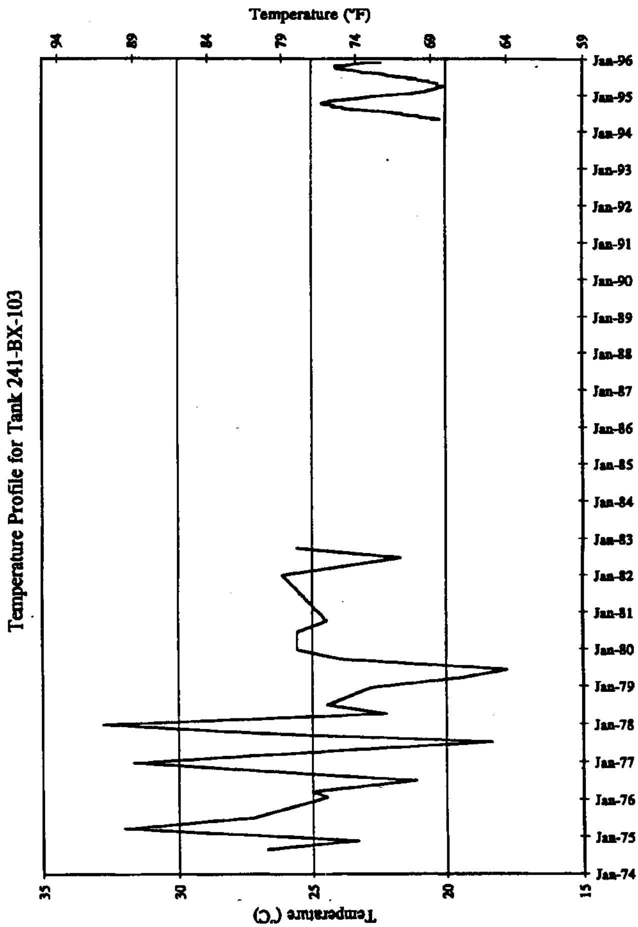
2.4.3 Drywells

Five drywells are associated with tank 241-BX-103 (Brevick et al. 1994). Three drywells have had readings above the 50 counts-per-second background radiation level. Of these, drywells 21-03-03 and 21-03-05 are no longer active; drywell 21-03-12 was active prior to 1990, but recent measurements have dropped below 200 counts per second. The previous activity is attributed to tank overflow and spillage during the active years of the tank farm's history (Welty 1988). Plotted readings from drywell 21-03-12 taken from 1990 to 1993 are available in Brevick et al. (1994).

2.4.4 Tank 241-BX-103 Photographs

The most recent photographs of the interior of tank 241-BX-103 were taken in 1986. They clearly show a dark brown supernate that appears to cover the entire surface although many photographs are hazy or overexposed making it difficult to identify all of the interior. Dark brown sludge is visible where the supernate meets the tank wall, and photographs show a light-yellow, crystalline ring around the tank wall approximately 10 to 20 cm above the waste surface. A more recent video taken in October 1994 indicates the waste volume or type has changed little since 1986. The crystalline ring is clearly visible at approximately the same relative position above the waste surface, which consists of dark-colored supernate. Any intrusions into the tank (see Section 2.1) apparently have not added a significant amount of material to the tank. Equipment, which is visible in photographs and the video, include an overflow nozzle, sluicing nozzles, a saltwell screen, a temperature probe, and an old level measurement tape.

Figure 2-4. Tank 241-Bx-103 Weekly High Temperature Plot.



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3.0 TANK SAMPLING OVERVIEW

This section focuses on the May 1995 and March 1996 sampling and analysis events for tank 241-BX-103. Two push-mode core samples and tank headspace vapor samples were taken to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The sampling and analyses were performed in accordance with the *Tank 241-BX-103 Tank Characterization Plan* (Bell 1995b). Further discussion of the sampling and analysis procedures can be found in the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

Analytical results have also been reported for five supernate samples taken from November 1974 to December 1975. Results from these analyses are presented in Appendix A. These results were not used to evaluate the present tank contents because of the age of the data and the lack of specifics about sample points. The data are included in this tank characterization report because the supernate analyses may provide some information on the content of liquids remaining in the tank.

3.1 DESCRIPTION OF 1974 AND 1975 SAMPLING EVENTS

From November 1974 to December 1975, five supernate samples were removed from tank BX-103 for analysis (Wheeler 1974, 1975a, 1975b, 1975c, 1975d). It appears the analyses were performed at the 222-S Laboratory although the above references are not clear in that regard. Furthermore, no riser numbers, chain-of-custody, or sample breakdown information is provided. The purpose of this series of sampling and analysis is not known, however, they most likely were taken to evaluate waste compatibility in preparation for waste transfer operations. This is shown by three supernate transfer operations from tank BX-103 to tank SX-110 from the fourth quarter of 1975 to the second quarter of 1976.

3.1.1 Supernate Sample Analysis (1975)

The sample preparation methods, which were used, are not clear in Wheeler (1974, 1975a, 1975b, 1975c, 1975d). It is believed that all analyses were performed directly or after dilution in acid or water. Furthermore, the analytical methods used are not indicated. Most likely, anion concentrations were determined by ion chromatography, and cations were determined by inductively coupled plasma spectroscopy. Tables containing the analytical results for these samples are in Appendix A.

3.2 DESCRIPTION OF THE 1995 PUSH-MODE SAMPLING EVENT

Two push-mode core samples of two segments each were collected from tank 241-BX-103 between May 24 and May 30, 1995. All samples were released to the laboratory within three days of being collected. Cores 86 and 87 were received, extruded, and analyzed at the 222-S Laboratory to support the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). Sampling and analytical requirements from the safety screening DQO are summarized in Table 3-1.

The push-mode core sampling method was chosen to obtain a vertical profile of the tank waste. Hydrostatic head fluid was not used to obtain the samples from segment 1 of core 86 or from either segment of core 87. The safety screening analyses include: total alpha content to evaluate criticality, differential scanning calorimetry (DSC) to ascertain the fuel energy value, and thermogravimetric analysis (TGA) for moisture content.

Table 3-1. Integrated Data Quality Objective Requirements for Tank 241-BX-103.[1]

Sampling Event	Applicable DQOs	Sampling Requirements	Analytical Requirements
Push-mode core sampling	Safety screening	Core samples from a minimum of two risers separated radially to the maximum extent possible.	Energetics Moisture Content Total Alpha
Tank headspace sampling	Safety screening	Tank headspace sampling below the riser.	Percent of the Lower Flammability Limit

Note:

[1]Bell (1995b)

3.3 SAMPLE HANDLING

Sampling information for the two core samples is shown in Table 3-2. Upon delivery to the 222-S Laboratory, cores 86 and 87 were extruded, photographed, and subsampled as prescribed in the *Tank 241-BX-103 Tank Characterization Plan* (Bell 1995b). The expected sample length is normally 48.3 cm except for the first segment which is often less.

Solid material from the extruded segments were divided into half segments labeled as upper and lower halves. The upper half is the material in the top half of the sampler; the material collected first. Only 10 cm of sample was expected from and realized for segment 1 of core 86.

Table 3-2. Tank 241-BX-103 Subsampling Scheme and Sample Description.[1]

Core: Seg: Riser	Date Sampled	Date Extruded	Liquid Recovered (grams)	Solids Recovered (grams)	Sample Description
86:1:7	5/24/95	5/31/95	221.71	33.91 (upper half)	Collected less than 5 mL of liner liquid, which was not retained. Recovered roughly 10 cm of solid material and about 210 mL of black, turbid drainable liquid. The solids were black, with a very wet consistency and a grainy texture. They were collected in one jar as the upper half of the segment.
86:2:7	5/26/95	5/31/95	21.39	86.68 (upper half) 195.54 (lower half)	Less than 5 mL of liner liquid was recovered, but not retained. Collected were 13 cm of sample from the upper half and 23 cm from the lower half of the segment. About 20 mL of black, turbid drainable liquid was also recovered. During the extrusion, the sampler push piston could not be pushed through the last 5 cm of the valve head, therefore the remaining solids were removed from the sampler with a spatula. All the solids were black and wet with a grainy texture. The interior of the sample contained a white, chalky material.
87:1:2	5/30/95	6/5/95	214.51	70.10 (upper half) 24.54 (lower half)	About 5 cm of solids were recovered shortly after the extrusion was begun and taken as the lower half of the segment. An additional 13 cm of solids sample was collected near the end of the extrusion and taken as the upper half of the segment. All solid material was black and shiny, with a grainy texture. Also collected were approximately 190 mL of turbid, black drainable liquid.
87:2:2	5/30/95	6/5/95	18.25	244.71 (upper half)	Prior to extrusion, less than 5 mL of liner liquid was recovered, but not retained. It was noted on the chain-of-custody form accompanying this sample that the "bottom alarm went off 16 3/4" into the stroke" such that only 42.5 cm of sample was expected. Collected toward the end of the extrusion were 23 cm of sludge, designated as the upper half of the segment. The solids were very dark brown sludge mixed with yellow-colored material. Most of the yellow material was present in the middle 13 cm of the sample. It was somewhat crumbly in some areas, but had a smooth consistency in other areas. The dark material generally had a smooth consistency. Approximately 15 mL of turbid, dark brown drainable liquid was also recovered.

Note:

[1]Bell (1995a)

3.4 SAMPLE ANALYSIS

The analyses performed at the half segment level on 1995 core samples 86 and 87 were limited to those needed to satisfy the safety screening requirements. The results of these analyses have been reported in the *90-Day Safety Screen Results and Final Report for Tank 241-BX-103, Push-Mode, Cores 86 and 87* (Bell 1995a). No individual sample results exceeded the safety screening DQO notification limits, although sample from the lower half sludge of segment 1, core 87 did exceed the safety screening limit of 480 J/g (dry) at the upper limit to the one-sided 95 percent confidence interval of the mean. That upper limit was 487 J/g (dry). A list of all samples by core, riser, segment portion, and sample number, and their associated analyses is shown in Table 3-3. The procedures used for these analyses are identified in Table 3-4. TGA and DSC analyses were performed under a nitrogen purge. Total alpha activity was determined by an alpha proportional counter on aliquots of samples that had been fused in potassium hydroxide and subsequently dissolved in hydrochloric acid.

Laboratory control standards, matrix spikes, and duplicate analysis quality control checks were applied to the total alpha activity analysis. Laboratory control standards and duplicate analysis quality control checks were used for the TGA and the DSC analyses. Assessment of the quality control data is in Section 5.1.2.

3.5 DESCRIPTION OF TANK HEADSPACE SAMPLING EVENT

Tank BX-103 headspace flammability was tested March 27, 1996, to satisfy the tank headspace flammability screening requirement of the safety screening DQO (Hunt 1995). Tank headspace flammability was determined in the field by means of a combustible gas meter while drawing a sample of the tank headspace from a point below riser 7 (WHC 1996). The combustible gas meter indicated that the tank headspace was 0 percent of the LFL, which satisfies the safety screening DQO requirement that the tank headspace be $<$ 25 percent of the LFL.

Table 3-3. Tank 241-BX-103 Sample Analysis Summary.[1]

Core:Segment:Riser	Segment Portion	Sample Number	Analyses
86:1:7	drainable liquid	S95T001032	TGA, DSC
	upper ½	S95T001021	TGA, DSC
		S95T001024	Total Alpha
86:2:7	drainable liquid	S95T001033	TGA, DSC
	upper ½	S95T001023	TGA, DSC
		S95T001026	Total Alpha
	lower ½	S95T001022	TGA, DSC
		S95T001025	Total Alpha
87:field blank	drainable liquid	S95T001036	TGA, DSC
87:1:2	drainable liquid	S95T001043	TGA, DSC
	upper ½	S95T001038	TGA, DSC
		S95T001041	Total Alpha
	lower ½	S95T001037	TGA, DSC
		S95T001042	Total Alpha
87:2:2	drainable liquid	S95T001044	TGA, DSC
	upper ½	S95T001039	TGA, DSC, % water by gravimetry
		S95T001040	Total Alpha

Note:

[1]Bell (1995a)

Table 3-4. Analytical Procedures.[1]

Analysis	Instrument	Preparation Procedure	Analytical Procedure
Energetics by DSC	Mettler® and Perkin-Elmer®	n/a	LA-514-113, Rev. B-1 LA-514-114, Rev. B-0
Percent water by TGA	Mettler® and Perkin-Elmer®	n/a	LA-560-112, Rev. A-2 LA-514-114, Rev. B-0
Percent water by gravimetry	n/a	n/a	LA-564-101, Rev. E-3
Total alpha activity	Alpha proportional counter	LA-549-141 Rev. C-2	LA-508-101, Rev. D-2
Tank headspace flammability	Combustible gas meter	n/a	Work package ES-96-177

Notes:

n/a = not applicable

Mettler is a registered trademark of Mettler Electronics, Anaheim, California.

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

[1]Bell (1995a)

4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

This section summarizes the analytical results associated with tank 241-BX-103 push-mode core samples taken in 1995 and tank headspace flammability data obtained in March 1996. The total alpha activity, percent water, and energetics results are shown in Tables 4-2, 4-3, and 4-5 respectively. The headspace flammability data are discussed in Section 4.2.5.

The 1995 core sampling and analysis efforts were performed to evaluate safety screening criteria as defined in the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The requirements of this document were transmitted to sampling and laboratory operations by a tank characterization plan (Bell 1995b). The DQO document specifies that sampling take place with the following analyses to be performed on the samples: weight percent water by TGA, DSC analysis for evaluation of fuel content and thermal output, and total alpha activity for criticality evaluation. The document also requires a measurement of the tank headspace flammability.

The sampling events prior to 1995 (see Section 3.0) resulted in characterizing supernate only. This is no longer representative of the waste in the tank. Thus, the only estimate of the chemical and radiochemical composition and inventory for the waste in tank 241-BX-103 is derived from the limited 1995 core sample data or the tank layering model (see Section 2.3.2 and Table 2-4). Estimates of the waste contents from the Tank Layer Model are based on historical waste transfer records. These data cannot be used to make decisions on the safety or disposition of tank waste.

The safety screening analyses results are summarized below. All reported analyses were performed in accordance with approved laboratory procedures. A list of the samples by core, segment, riser, segment portion, sample number, and their associated analyses is shown in Table 3-3. No deviations or modifications were noted by the laboratory.

4.2 DATA PRESENTATION

4.2.1 Density Calculations

An overall tank density is needed to calculate a projected inventory for total alpha activity. However, density measurements were not performed on the waste during the 1995 sampling and analysis effort. An estimated sludge density can be derived using the recovered segment lengths and masses.

To perform this calculation, a length-to-volume conversion factor is needed. This factor can be obtained from the dimensions of the sampler (length = 48.2 cm and volume = 309 mL) as shown in the following equation:

$$\left(\frac{309\ \text{mL}}{48.2\ \text{cm}}\right) = 6.41\ \frac{\text{mL}}{\text{cm}}$$

Using this factor, the length of the recovered segments can be converted into a volume. For example, 36 cm of sludge was recovered from segment 2 of core 86. This translated into 231 mL as shown in the following equation:

$$36\ \text{cm}\left(\frac{6.41\ \text{mL}}{\text{cm}}\right) = 231\ \text{mL}$$

The sludge density of segment 1 of core 86 was then determined by dividing the measured sludge mass (282 g) by the converted volume as shown in the following equation:

$$\frac{282\ \text{g}}{231\ \text{mL}} = 1.22\ \frac{\text{g}}{\text{mL}}$$

The sludge densities of the two segments of core 87 were similarly derived. No density was calculated for segment 1 of core 86 because the sludge was so wet that a significant amount of it 1) became mixed with the drainable liquid of the segment, and 2) was not recovered from the extrusion tray. Table 4-1 contains the estimated sludge densities.

Table 4-1. Estimated Sludge Densities.

Core	Segment	Estimated Sludge Density (g/mL)	Core Mean (g/mL)	Overall Sludge Density Estimate (g/mL)
86	2	1.22	1.22	1.23
87	1	0.82	1.24	
	2	1.66		

The estimated overall density of 1.23 g/mL from Table 4-1 was used in the projected inventory calculation for total alpha activity.

4.2.2 Total Alpha Activity

Analyses for total alpha were performed on all homogenized half-segment sludge subsamples from cores 86 and 87. No total alpha analyses were performed on drainable liquid samples. Duplicate aliquots of each half segment were prepared by fusion digestion and analyzed for total alpha activity. The results are summarized in Table 4-2. Standard recoveries were all within the laboratory lower and upper control limits but varied considerably. The laboratory attributed this variation to geometric variances in the standard sample mounts. Additionally, the relative percent difference (RPD) between the sample and duplicate runs on the upper half of segment 1 of core 86 (sample S95T001024) was slightly above the ± 10 percent limit specified in the BX-103 tank characterization plan (Bell 1995b) at 11.5 percent. A rerun was not performed because the analytical results were well below the 41 μCi/g notification limit.

Table 4-2 shows total alpha activity analytical results. The core mean was calculated by taking an average of the six sample results (three duplicate pairs) for each core. The one less-than value from core 87 was weighted equally with the detected values, and the resulting average was reported. The tank mean was derived by averaging the two core means. All results were roughly an order of magnitude less than the safety screening DQO notification limit of 41 μCi/g. The largest observed values were found in segment 1 of each core.

Quality control information for these samples is in Section 5.1.2. The absence of a total alpha activity analysis on drainable liquid is not expected to impact the reported results because the alpha-emitting radionuclides are not expected to be soluble in basic media.

4.2.3 Thermodynamic Analyses

Because tank 241-BX-103 push-mode samples were evaluated according to *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), the only physical analyses required on cores 86 and 87 at the half-segment level were TGA and DSC. The TGA provides an estimate of weight percent water, but it also is used to interpret the DSC results.

4.2.3.1 Thermogravimetric Analysis. Percent water by TGA was performed directly on all solid and drainable liquid subsamples from cores 86 and 87. Percent water was also measured gravimetrically on the upper half sludge from segment 2 of core 87. The results are in Tables 4-3 and 4-4. No samples had moisture levels below the safety screening criterion of 17 percent.

Analysis by TGA measures the mass of a sample while its temperature is increased at a constant rate. Nitrogen is passed over the sample during heating to remove any released gases. Any decrease in the weight of a sample during TGA represents a loss of gaseous matter from the sample through evaporation or through a reaction that forms gas phase products. The moisture content is estimated by assuming that all TGA sample weight loss up to a certain temperature (typically 150 to 200 °C) is caused by water loss. This upper temperature limit for moisture loss is chosen by the chemist at an inflection point on the

Table 4-2. Tank 241-BX-103 Total Alpha Activity Sludge Results.[1]

Sample Location Core:Seg	Segment Portion	Standard Recovery (%)	Spike Recovery (%)	Blank Result (µCi/g)	Result (µCi/g)	Duplicate (µCi/g)	RPD (%)	Mean Result (µCi/g)	95% Confidence Limit	Core Mean (µCi/g)
86:1	Upper ½	101.9	95.10	<0.0992	5.17[2]	4.61[2]	11.5	4.89	6.66	3.04
	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	
86:2	Upper ½	79.05	n/a	<0.276	1.65[3]	1.69[3]	2.40	1.67	1.80	
	Lower ½	101.9	n/a	<0.0992	2.47	2.64	6.65	2.56	3.09	
87:1	Upper ½	94.59	104.1	<0.130	4.98	4.80	3.68	4.89	5.46	3.23
	Lower ½	94.59	n/a	<0.130	4.38	4.33	1.15	4.36	4.51	
87:2	Upper ½	79.05	n/a	<0.0276	< 0.547[3]	0.339[3]	n/a	0.443	n/a	
	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	
Tank mean of sludge total alpha activity = 3.13 µCi/g Projected sludge total alpha inventory = 905 Ci										

Note:

[1]Bell (1995a)

[2]The value for RPD exceeds the limit specified in the Tank Characterization Plan (Bell 1995b).

[3]Associated standard recovery does not meet the criterion specified in the Tank Characterization Plan (Bell 1995b).

Table 4-3. Sludge Percent Water Results for Tank 241-BX-103.[1]

Sample Location	Segment Portion	Standard Recovery	Result	Duplicate	RPD	Mean Result	95% Lower Confidence Limit	Core Mean
Core:Seg		(%)	(% H_2O)	(% H_2O)	(%)	(% H_2O)	(%)	(% H_2O)
Thermogravimetric Analysis Results								
86:1	Upper ½	100.1	60.65	63.17	4.07	61.91	53.95	48.2
86:1	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	
86:2	Upper ½	99.24	40.75	39.25	3.75	40.00	35.26	
86:2	Lower ½	100.1	40.68	44.91	9.88	42.80	29.44	
87:1	Upper ½	98.41	55.20	57.21	3.58	56.20	49.86	36.1
87:1	Lower ½	99.24	59.61	62.98	5.50	61.30	50.66	
87:2	Upper ½	98.41	29.60[2]	25.01[2]	16.8	27.30	n/a	
87:2 (rerun)	Upper ½	101.0	24.14	22.14	8.64	23.14	16.83	
87:2:	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	
Gravimetric Analysis Results								
87:2	Upper ½	99.8	17.4[2]	32.4[2]	60.2	24.9	n/a	
87:2 (rerun)	Upper ½	98.76	23.6	23.4	0.85	23.50	22.87	
Tank mean of sludge weight percent water = 42.2%								

Note:

[1]Bell (1995a)

[2]The value for RPD exceeds the limit specified in the Tank Characterization Plan (Bell 1995b).

Table 4-4. Drainable Liquid Percent Water Results for Tank 241-BX-103.[1]

Sample Location	Standard Recovery	Result	Duplicate	RPD	Mean	95% Lower Confidence Limit	Core Mean
Core:Seg	(%)	(% H_2O)	(% H_2O)	(%)	(% H_2O)	(% H_2O)	(% H_2O)
86:1	97.49	78.13	78.78	0.83	78.45	76.40	76.84
86:2	97.49	75.31	75.16	0.20	75.23	74.76	
87:1	100.4	77.79	79.44	2.10	78.62	73.41	78.45
87:2	107.2	76.84	79.73	3.69	78.28	69.16	
Field Blank	97.47	100.9	100.8	0.10	100.8	n/a	
Tank mean of drainable liquid weight percent water = 77.6%							

Note:
[1]Bell (1995a)

TGA plot. Gravimetric analysis for water is determined by attributing to water evaporation all sample weight loss sustained by heating a sample at 120 °C for 20 hours.
The TGA scans on samples from Tank 241-BX-103 generally fall into two categories. Category one, which includes the drainable liquid samples from core 87 and all the sludge samples, is characterized by a rapid weight loss from 30 °C to 100-120 °C followed by a region of little or no weight loss until approximately 220 °C. A second weight loss of smaller magnitude is observed from roughly 220 to 300° C. This second region of weight loss varied from very small to 30 to 40 percent of the magnitude of the initial weight loss. The TGA scans for drainable liquid samples of core 86 displayed a different profile. The scans showed a rapid weight loss from 30 °C until 150-160 °C followed by little or no further weight loss through 500 °C. For samples in either category, the reported percent water values were determined using only the initial weight loss portion of the thermogram.

As expected, the drainable liquid samples were higher in moisture than the sludge samples. Table 4-4 shows the percent water results determined from gravimetric analysis of sludge from the upper half of segment 2 of core 87. That analysis was performed as requested in Johnson (1995) because the results by TGA were below 25 percent. There is good agreement between the percent water values by the two methods. The gravimetric results were used in the calculation of the tank mean for percent water.

All TGA analyses except sample S95T001038 from the upper half of segment 1, core 87 met the ±10 percent RPD criterion specified in the tank 241-BX-103 characterization plan. A rerun was performed on an sample aliquot with satisfactory results. The initial gravimetric result also was not satisfactory because of a high RPD between the sample and duplicate results. A rerun yielded a satisfactory RPD and an average result close to the original.

4.2.4 Differential Scanning Calorimetry

In a DSC analysis, heat absorbed or released by a sample is measured while it is exposed to a linear increase in temperature. Nitrogen is passed over the sample to remove any gases being released. A plot of the energy absorbed (a endothermic process) and/or released (an exothermic process) versus temperature defines the DSC scan.

The DSC analyses for tank 241-BX-103 were performed directly in duplicate on all segment-level drainable liquid samples and all homogenized half-segment sludge samples. Results are shown in Table 4-5 which displays the DSC results on a wet weight basis for both sludge and drainable liquid samples. Exothermic processes are denoted by a negative change in the enthalpy (ΔH) value. All DSC exothermic values were converted to a dry weight basis, and the results are tabulated in Table 4-6 using the respective average percent water for that sample as determined by TGA. Dry-weight results are given in Section 5.5.1 to compare the

Table 4-5. Differential Scanning Calorimetry Results for Tank 241-BX-103 (Wet Weight).[1]

Sample Core:Seg	Segment Portion	Standard Recovery	RPD	Run	Transition 1 Temp. range (°C)	Transition 1 ΔH (J/g)	Transition 2 Temp. range (°C)	Transition 2 ΔH (J/g)	Transition 3 Temp. range (°C)	Transition 3 ΔH (J/g)
Sludge Results										
86:1	Upper ½	91.74	3.34	1	30-160	1,020	250-370	-156.0	---	---
				2	30-160	992	240-390	-161.3	---	---
86:1	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
86:2	Upper ½	103.7	0	1	30-130	860	230-310	479	---	---
				2	30-130	868	230-310	468	---	---
	Lower ½	91.74	200	1	30-150	851	220-310	455	400-460	-48.4
				2	30-150	1010	220-310	299	---	---
				3	30-140	990	220-310	341	---	---
87:1	Upper ½	94.2	0.91	1	30-160	950	190-250	-19.2	290-410	-135.3
				2	30-150	1,470	190-250	-24.4	280-390	-128.7
	Lower ½	103.7	8.50	1	30-140	1,340	280-390	-155.7	---	---
				2	30-150	1,210	280-410	-143.0	---	---
87:2	Upper half	94.2	0	1	30-140	598	220-330	528	---	---
				2	30-150	683	220-330	553	---	---
	No lower ½	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Drainable Liquid Results										
86:1	drainable liquid	98.21	0	1	30-120	1,630	---	---	---	---
				2	30-130	1,690	---	---	---	---
86:2	drainable liquid	98.21	0	1	30-130	1,560	---	---	---	---
				2	30-140	1,760	---	---	---	---
87:1	drainable liquid	106.9	0	1	30-130	1,570	---	---	---	---
				2	30-130	1,560	---	---	---	---
87:2	drainable liquid	109.3	0	1	30-130	1,490	---	---	---	---
				2	30-140	1,290	---	---	---	---
Field Blank	n/a	97.47	0	1	30-120	2330	---	---	---	---
				2	30-120	1990	---	---	---	---

Note:

[1]Bell (1995a)

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BX-103 (Dry-Weight).

Sample (Core:Segment:Segment Portion)	%H2O by TGA[1]	Dry-Weight Exotherm Result 1 (J/g)	Dry-Weight Exotherm Result 2 (J/g)	Mean Result (J/g)	95% Upper Confidence Limit
86:1:Upper half sludge	61.91	410	423	417	455
86:2:Lower half sludge	42.80	84.6[2]	0[2,3]	28.3	111
87:1:Upper half sludge	56.20	353	350	351	361
87:1:Lower half sludge	61.30	402	370	386	487

Notes:

[1]Represents the average percent water determined by thermogravimetric analysis for each respective segment portion. The TGA data are presented in Table 4-3.

[2]The value for RPD exceeds the limit specified in the Tank Characterization Plan (Bell 1995b).

[3]A triplicate run on the same sample also displayed no exotherm. Both results of 0 J/g were used to calculate the mean.

analytical results with the requirements of the safety screening DQO. Conversions from wet weight to dry weight are performed as shown in the following equation:

$$\text{Exothermic result dry weight} = \frac{\text{exothermic result wet weight}}{(1 - \frac{\%\ H_2O}{100})}$$

None of the DSC sample results exceeded the safety screening DQO notification limit of 480 J/g on a dry weight basis, although sludge from the lower half of segment 1 of core 87 exceeded this limit at the upper limit to a one-sided 95 percent confidence interval on the mean. Exothermic transitions were observed in four samples; the highest exotherms were found in segment 1 of both cores with average dry weight values of 417 and 386 J/g for cores 86 and 87, respectively. An exotherm was observed in a sample from the lower half sludge of segment 2 of core 86; however, no exotherms were observed on the duplicate sample. A third aliquot was run because of the apparent discrepancy, but no exotherm was detected in the triplicate result. The laboratory report (Bell 1995a) noted that the sample had a grainy texture. It is therefore likely that the sample was somewhat heterogenous with respect to organic carbon or other material that could give rise to an exothermic process.

The information in Table 4-5 shows that the DSC scans of the sludge samples generally fall into three categories, and the drainable liquid samples fall into a fourth category. Sludge samples in the first category show a large endotherm from 30 °C to 140-160 °C followed by an exotherm from roughly 250 to 370° C. Other sludge samples also show a large endotherm from 30 °C to 130-150 °C and a second endotherm from approximately 220 to 320 °C. Sludge from the lower half of segment 2 of core 86, is considered to be in this category even though one aliquot displayed a relatively small exotherm from 400 to 460 °C. Upper half sludge from segment 1 of core 87, was unique in that it showed two exothermic events following the initial, large endotherm found in all the sludge samples. The drainable liquid DSC scans were not as interesting; they showed a large endothermic event from 30 to 130 °C and no further endotherms or exotherms.

The first transition in the DSC scans represents the endothermic process of the evaporation of free and interstitial water. The second endothermic transition correlates with the second significant weight loss in the TGA scan. This is consistent with the endothermic processes of sodium nitrate and sodium nitrite fusion, vaporization, and degradation. Exothermic reactions in which heat is released are probably the result of organic compounds in the sample reacting with the molten nitrate or nitrite salts. The energy required for the endothermic reactions are several times greater than the exothermic energy released. This indicates that a relatively large amount of energy would have to be applied to the waste before the exothermic reaction could be initiated.

4.2.5 Tank Headspace Flammability Results

The results of the tank headspace field analysis of March 27, 1996, indicated tank headspace flammable gases were present at 0 percent of the LFL (WHC 1996). This result satisfies the safety screening DQO requirement that the tank headspace be < 25 percent of the LFL. The tank headspace analysis also satisfied the safety screening DQO requirement that the tank headspace be sampled below the tank riser; a combustible gas meter was used to perform the analysis. All results from the tank 241-BX-103 headspace vapor survey are shown in Table 4-7.

Table 4-7. Tank 241-BX-103 Headspace Vapor Survey Results.[1]

Analyte	Concentration
LFL	0%
O_2	20.8%
NH_3	70 ppm
Total Organic Carbon	6.4 ppm

Notes:

ppm = parts per million

[1]Measured 10.7 m (35 ft) below riser 7. Data are from Work Package ES-96-177.

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

The purpose of this section is to evaluate the overall quality and consistency of the available analytical results for tank 241-BX-103 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 interpretation of the data. These factors are used to assess the overall quality and consistency of the data and to identify limitations in the use of the data. Normal consistency checks include the following: calculating a mass and charge balance, comparing total alpha activity results with the sum of individual alpha emitters, and comparing phosphorus and sulfur concentrations determined by inductively coupled plasma with phosphate and sulfate concentrations determined by ion chromatography. None of these comparisons were possible for tank 241-BX-103 because of very limited data. Therefore, the only measures of data quality for the safety screening results were the laboratory quality control information (see Section 5.1.2).

5.1.1 Field Observations

Only field observations relative to the 1995 core samples are discussed here. No field observation data is available from previous sampling events, and analytical results from those efforts are for comparison only rather than for calculating tank inventories.

There were specific differences in color, consistency, and drainable liquid content in the samples retrieved during the 1995 sampling event. The differences were slight and are not expected to impact the interpretation of the available data. Descriptions and photographs of the extruded core samples are in Section 3.3 and Appendix B, respectively. The photographs show there was poor correlation between expected and actual sample recovery. Resolution of the three primary safety screening DQO analytical requirements is at the half-segment level for solid samples. Furthermore, sampling requirements of the DQO are two full-depth core or auger samples. No sample was obtained from the lower half of segment 2 of core 87. This amounts to about 28 percent of the expected core sample and may preclude fulfillment of the safety screening requirements. However, none of the data collected suggest there are any safety concerns associated with tank 241-BX-103.

5.1.2 Quality Control Assessment

Quality control assessment of the 1995 push-mode core sample from tank 241-BX-103 includes an evaluation of blanks, duplicates, spikes, and standards that were performed in conjunction with the chemical analyses. The required, program-specific quality control elements are defined in Bell (1995b). They were conducted on the two 1995 push-mode core samples, allowing for an assessment regarding the accuracy and precision of the safety screening data. A summary of the quality control failures with respect to these data is shown in Table 5-1. It should be noted that all analyses met internal laboratory quality control requirements.

Table 5-1. 1995 Core Samples: Summary of Quality Control Failures[1].

Sample (Core:Seg:Portion)	Analysis	Quality Control Failure	Count Error (%)
86:1:upper half	total alpha	RPD 11.5%	9.9
86:2:upper half	total alpha	Std 79.05%	33.3
86:2:lower half	DSC	RPD 200%	n/a
87:2:upper half	TGA	RPD 16.8%[2]	n/a
87:2:upper half	% water by gravimetry	RPD 60.2%[2]	n/a
87:2:upper half	total alpha	Std 79.05%	170

Notes:

Std = standard
n/a = not applicable

[1]As defined by the tank 241-BX-103 characterization plan (Bell 1995b). Criteria include the following: standard recovery of 90 to 110 percent, spike recovery of 90 to 110 percent, and an RPD of duplicate analyses ≤ 10 percent.

[2]A rerun was performed with satisfactory results.

Standards contain the analytes of interest at known concentrations and are used to estimate the accuracy of the analytical method. They are evaluated once per batch prior to and concurrent with sample analysis. The criterion for an acceptable standard recovery for the 1995 core samples, as defined in the safety screening DQO document, is 90 to 110 percent. If a standard is above or below the criterion, the analytical results may be biased high or low, respectively. Standards are required on all safety screening analyses.

Matrix spikes are used to estimate the bias of the analytical method caused by matrix interferences. Spiked samples are prepared by splitting a sample into two aliquots and adding a known amount of a particular analyte to one aliquot to calculate a percent recovery. The safety screening quality control criterion for matrix spikes is 90 to 110 percent recovery. If a spike is above or below this criterion, the analytical results may be biased high or low, respectively. Spike recoveries are not applicable to the DSC and TGA analysis methods. The tank 241-BX-103 characterization plan requires a spike once per matrix on total alpha analyses; therefore, a spike was performed on one sludge sample from each core.

Method blanks document contamination resulting from the analytical process. They are prepared by filling sample containers with deionized, distilled water. They are not applicable to the DSC and TGA analysis methods. Blanks for total alpha activity analysis were carried through the complete sample preparation (fusion) and analytical procedure. Although the tank 241-BX-103 characterization plan requires a blank on total alpha activity analyses, the acceptance criteria are not defined. Field banks are used to assess any contamination originating from equipment used during sampling or extrusion activities.

The RPD between primary and duplicate samples provides an indication of sample homogeneity. It is defined as the absolute value of the difference between duplicate results divided by the mean. The criterion for an acceptable RPD for the 1995 core samples, as defined by the safety screening DQO document, is ≤ 10 percent.

5.1.2.1 Standard Recoveries of DSC, TGA, Percent Water by Gravimetry and Total Alpha Activity Analyses. None of the standards associated with the TGA or DSC analyses exceeded the quality control criterion of 90 to 110 percent recovery. At 79.05 percent, one standard run for total alpha was outside this range on one batch (two samples). This was within the laboratory upper and lower control limits and likely was caused by geometric variation of the standard sample mount. Nevertheless, the low recoveries suggest that the analytical results for total alpha may be biased low for the two samples.

5.1.2.2 Spike Recoveries of Total Alpha Activity Analyses. All total alpha activity spike recoveries from cores 86 and 87 are in the acceptable range.

5.1.2.3 Evaluation of the Field Blank and Total Alpha Activity Method Blanks. Samples of the field blank were analyzed by DSC and TGA. No exotherms were found in the DSC, and the percent water by TGA was 100.8 percent. Method blanks are not applicable to DSC and TGA methods. All total alpha activity method blanks were below the minimum detectable activity indicating that contamination was not a problem.

5.1.2.4 Duplicate Precision of DSC, TGA, Percent Water by Gravimetry, and Total Alpha Activity Analyses. Samples from four segment portions showed exotherms in their DSC scans. The sample/duplicate pair from the lower half of segment 2 of core 86 had a RPD value of 200 percent because only one of the sample/duplicate pairs showed an exotherm (85 J/g dry). A triplicate determination was performed and showed no exotherm. Only one RPD value for TGA exceeded the tank characterization plan quality control criterion. The sample was rerun with acceptable results. The TGA and DSC analyses use very small sample sizes (10 to 20 mg). The effects of small variations of a sample's composition are more pronounced the smaller the sample size. The one sample and duplicate pair that was submitted for analysis of percent water by gravimetry had an RPD of 60.2 percent. The sample was rerun with acceptable results.

One RPD exceeded the 10 percent criterion for total alpha at 11.5 percent. No rerun was performed because the average result is roughly eight times less than the notification limit. In addition, the RPD is not unusually high given the count error of 9.9 percent.

In summary, the tank 241-BX-103 sludge samples appear to be reasonably homogeneous, and there is no indication of sample contamination from sampling or extrusion equipment. Total alpha activity values may be biased low as indicated by a low standard recovery.

5.1.3 Data Consistency Checks

Insufficient data are available to perform data consistency checks.

5.2 COMPARISON OF HISTORICAL AND ANALYTICAL RESULTS

Regarding previous sampling events for tank 241-BX-103, the most recent sampling event prior to 1995 occurred in October 1975 (Brevick et al. 1994). This and all other historical samples available for this tank were supernatant samples. It appears that the supernate contents of tank 241-BX-103 have changed since they were last sampled because of waste transfers. Furthermore, these historical data are not validated and cannot be used to make decisions. The last supernate sample removed from the tank is described as yellow, while all drainable liquid obtained from the 1995 sampling activity was black or dark brown. For these reasons, no comparison between any 1995 safety screening data and the 1975 data was attempted.

5.3 TANK WASTE PROFILE

The ability to assess data trends between segments and between cores is limited for tank 241-BX-103 sampling and analysis events because of the limited cores taken and the limited analyses on discrete segments at different depths. Only analytical data resulting from the 1995 core sampling effort is considered here. Earlier data is of questionable quality, lacks sampling location and/or depth, or is not usable because waste has been added or removed from the tank since the particular sampling event. A comparison of the 1995 sludge results from different segments and core samples indicates the inventory profile for this tank. Visual differences between cores 86 and 87 and their affect on data interpretation are discussed in Section 5.1.1.

Based on the TLM document for tank 241-BX-103, three primary layers of waste are predicted in the tank: a bottom sludge layer composed of metal waste, a sludge layer of unknown waste, and a top layer of noncomplexed supernatant (Agnew et al. 1995). This scenario was supported by photographic and videographic evidence, which show an upper supernatant layer of dark, black liquid (Section 2.4.3).

During the May 1995 sampling event, cores were obtained from risers 2 and 7, fulfilling the tank characterization plan requirement of sampling from two risers located approximately 180° apart and near the outer edge of the tank (Bell 1995b). Sampling from these risers provided information on the spatial distribution of the percent water and total alpha results.

A statistical procedure known as the analysis of variance (ANOVA) was conducted on sludge and drainable liquid samples to determine whether there were any horizontal or vertical differences in analyte concentrations. The ANOVA model used was a random effects nested model. It was conducted on two segments from each riser. When a segment was subsampled into upper and lower halves (sludge only), as it was for segment 2 of core 86 and segment 1 of core 87, the results of the subsegments were combined to simplify the analysis. The ANOVA generates a p-value which 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 the sample means are significantly different. However, if a p-value is above 0.05, there is not sufficient evidence to conclude that the samples are significantly different.

The ANOVA results for the drainable liquid samples indicated there were no significant differences in percent water either horizontally or vertically. The ANOVA results for the sludge samples showed no significant horizontal differences for percent water or total alpha (p-values = 0.684 and 0.737, respectively), but both analytes showed significant differences vertically (p-value = 0.0001 for both).

The visual descriptions of the core samples indicated a general horizontal similarity and a vertical variation within the sludge layer. The sludge from segment 1 of both cores was described as moist and black. Segment 2 of core 86 was also moist and black on the outside, but subsampling revealed a white chalky material on the inside. Segment 2 of core 87 was very dark brown and swirled with yellow material. This evidence also strongly implies some vertical heterogeneity within the tank.

In summary, the information provided by the fill history, photographs, videotape, statistical analysis, and visual descriptions supports the conclusion that tank contents are generally uniform horizontally but show some vertical variation.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER HISTORY INFORMATION

Because of the few analytical requirements of the safety screening characterization, the comparison of analytical results from the 1995 sampling event with historical transfers and estimated inventories is very limited. Existing information is used to evaluate the tank layering model and tank inventory estimate.

5.4.1 Evaluation of Tank Layering Model

The TLM document (Agnew et al. 1995) concludes there are three waste layers in tank 241-BX-103. It should be pointed out that some mixing of waste layers should be expected so that distinct or abrupt changes in the waste may not be observed. Waste mixing may have occurred during waste additions or removals and from density differences in waste types, aging, and liquid pumping activities. The situation is complicated because waste entered tank 241-BX-103 on one side, cascaded from another location, and was pumped from a third location (see Figures 2-1 and 2-2).

The TLM is supported by in-tank photographs and video and extrusion and analytical results that indicate three layers of waste: supernate and two sludge layers. Although two apparent layers could be the same waste type differing only in the presence of small amounts of highly chromatic substances, data in Section 5.3 indicate there are statistically significant differences in total alpha activity between the first and second segments.

5.4.2 Evaluation of Solids Tank Inventory Estimate

The solids tank inventory estimate (see Table 2-4) indicates that tank 241-BX-103 solids contain 59 percent water. This does not compare well with the tank sludge average value of 42 percent found in the 1995 core samples. The discrepancy may be caused by uncertainty in the moisture content of the unknown waste type, which accounts for 15 percent of the sludge volume according to the TLM. The HDW document (Agnew 1995) predicts the percent water in the lower sludge (metal waste) layer at 38.3 percent. This compares very well with the 35.3 percent average found for the second segment of both 1995 core samples.

The supernate in the tank had many different sources and could not be compared with a specific waste type.

The comparison of the recent total alpha data with the historical information was also not favorable. The plutonium concentration in tank 241-BX-103 is 0.169 μCi/g according to the tank inventory estimate. If plutonium is the only source of alpha activity, it is low by a factor of about 19 (see Table 4-2). Activities of other alpha emitters is not known but almost certainly exist in the tank waste. The Pu activity calculated in Agnew (1995) for metal waste is only 0.00476 μCi/g, far below the total alpha activity actually detected in the 1995 samples.

Finally, the TLM predicts no total organic carbon (TOC) in the waste, yet four samples had exotherms in the DSC scans. It should be pointed out that the presence of these exotherms do not invalidate the TLM or preclude the absence of TOC in the waste since there are a number of possible chemical or physical processes that could account for an exotherm. It is unfortunate there are no TOC data to support or oppose the DSC data or the TLM.

In summary, the extrusion information and analytical results are consistent with the TLM predictions of three waste layers. However, the tank inventory estimate is not supported by the total alpha or the TGA data. The presence of unknown waste types or alpha emitters other than Pu may account for the poor correlation between the recent data and the inventory estimate. The TGA data do offer support for the existence of metal waste sludge as the lower sludge layer as the TLM indicates. However, the TLM conflicts with transfer records that indicate all metal waste was sluiced from the tank in 1954.

5.5 EVALUATION OF PROGRAM REQUIREMENTS

This section details data needs as defined in the *Tank Safety Screening Data Quality Objective* (Hunt 1995). The 1995 sampling and analysis effort was carried out under an earlier revision of the safety screening DQO document (Babad and Redus 1994). Evaluation of 1995 data will be made against the later revision cited above. The safety screening DQO document was developed to allow rapid classification of waste tanks into specific categories associated with safety issues. It also establishes decision criteria or notification limits for concentrations of analytes of concern. These decision criteria are used to determine whether a tank is safe or if further investigation into the tank's safety is warranted. If results from any primary analysis exceeds a decision criterion, the tank will not be classified "safe," and further analyses will be conducted to assure tank safety. The available data were insufficient to assess impacts on operational, environmental, or process development programs.

The decision criteria used to determine whether a tank is safe or should be identified with a specific safety issue are as follows: 1) energetics (no DSC exotherm exceeds 480 J/g), 2) flammable gas concentration (any flammable gas present in the tank headspace is less than 25 percent of the lower flammability limit), and 3) total alpha activity less than 1 g/L[1]. The earlier revision of the safety screening DQO, against which the sampling and analysis of the 1995 core samples was performed, also included as a criterion that percent water should be greater than 17 percent. The safety screening DQO document requires a 95 percent confidence that each decision criterion has been met (Hunt 1995). Thus, for a given analyte, the notification limit on an individual measurement does not have to be exceeded to preclude the tank from being declared safe, provided the notification limit is below the upper 95 percent confidence limit. This is the case for the DSC measurements discussed in Section 5.5.1. If a criterion is not met, further analyses are conducted to determine the specific safety issue with which the tank should be identified.

A statistical analysis of the safety screening data was performed to determine whether, with 95 percent confidence, the decision criteria were satisfied. The analytical results were compared with the decision criteria by computing upper limits to one-sided, 95 percent confidence intervals on the means. The 95 percent confidence limit for each analysis is given in Tables 4-2, 4-3, 4-4, and 4-6. For the DSC and total alpha analyses, the upper limit to the confidence interval was computed on the mean. If the upper limit is greater than 480 J/g or 41 μCi/g respectively, the decision criterion is exceeded. For percent moisture by TGA, the lower limit of the confidence interval was computed. If the lower limit is less than 17 percent, then the decision criterion is exceeded.

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[1]Although the actual decision criterion listed in the safety screening DQO is 1 g/L, total alpha is measured in μCi/g. To convert the criterion into the practical units of μCi/g, it was assumed that all alpha decay originated from ^{239}Pu. Assuming a sludge density of 1.5 and using the specific activity of ^{239}Pu, the decision criterion is derived as shown:

$$\left(\frac{1\ g}{L}\right)\left(\frac{1\ L}{10^3\ mL}\right)\left(\frac{1\ mL}{1.5\ g}\right)\left(\frac{0.0615\ Ci}{1\ g}\right)\left(\frac{10^6\ \mu Ci}{1\ Ci}\right) = 41\frac{\mu Ci}{g}$$

5.5.1 Safety Evaluation

With respect to the 1995 core samples, the specific requirement that a vertical profile of the tank be obtained from two widely spaced risers was not fulfilled. The lower half segment portion was missing from segment 2 of core 87. All four primary analytical requirements (energetics, total alpha activity, moisture content, headspace flammability) were met. The 95 percent confidence decision criteria thresholds were not met for TGA on the upper half of segment 2, core 87, and for DSC for the lower half of segment 1 of the same core, even though the notification limit on individual measurements was not exceeded.

The potential for criticality can be assessed from the total alpha data. None of the individual samples from the 1995 data contained total alpha activity greater than 5.17 μCi/g, and the overall mean result was 3.13 μCi/g. This was well below the notification limit of 41 μCi/g or 1 g/L as specified in the safety screening DQO.

Another factor in assessing the safety of tank waste is the heat generation and temperature of the waste. Heat is generated in the tanks from radioactive decay. The major radionuclides (^{90}Sr and ^{137}Cs) that contribute to heat generation were not analyzed in the 1995 sampling; therefore, an estimate based on sampling results cannot be made. However, the HTCE estimate for heat load is 132 W, well below the 11,700 W threshold that differentiates high- and low-heat load tanks. Furthermore, the historical temperature records do not indicate any sign of excessive heat generation.

The tank 241-BX-103 safety screening DQO decision criteria and results of the 1995 push-mode core sample and the 1996 tank headspace sample are summarized in Table 5-2. The tank BX-103 headspace flammability determination of March 27, 1996, revealed that the tank headspace flammability is 0 percent of the LFL (WHC 1996). This result meets the safety screening DQO requirement that the tank headspace flammability be $<$ 25 percent of the LFL. Information in the table indicates that the safety screening DQO sampling requirements may not have been met. However, there are no immediate safety concerns associated with the tank although TGA and DSC notification limits were exceeded at the 95 percent confidence level. The upper half of segment 2 of core 87, which showed low moisture in the solids (at the lower 95 percent confidence limit), showed no exotherm in the DSC scans. The lower half of segment 1 of core 87, which showed a large exotherm (at the upper 95 percent confidence level), contained adequate moisture at 61 percent to mediate any propagating, exothermic reaction.

Table 5-2. Tank 241-BX-103 Safety Screening Data Quality Objective Decision Criteria and Results.

Safety Screening Decision Criterion	Decision Criterion Threshold (at 95% Confidence)	Does decision criterion meet 95% confidence limit on all subsegments?
Sampling	Two complete vertical profiles from two risers	No[1]
Total fuel content	< 480 J/g (dry-weight basis)	No[2]
Percent moisture	> 17%	No[3]
Total alpha	< 1 g/L	Yes
Flammable gas	< 25% of lower flammability limit	Yes

Notes:

[1]The safety screening DQO does not specifically require a 95 percent confidence for sampling requirements. It is reasonable to assume the sampling requirements were not fulfilled because a full, vertical profile was not obtained from core 87.

[2]Pertains to the lower half sludge from segment 1 of core 87. Neither the sample nor duplicate result exceeded the decision criterion threshold, but that subsegment did not meet the threshold at the upper limit to a one-sided 95 percent confidence interval on the mean.

[3] Not a decision criterion per Hunt (1995).

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The most recent core sampling of tank 241-BX-103 was completed in May 1995; tank headspace flammability was determined in March 1996. The supernate analyses performed earlier do not represent an accurate waste composition. Because the 1995 core sampling event focused on safety screening criteria and because the safety screening analyses are the only ones available to represent the current waste in the tank, the chemical and radiochemical composition of the waste must be estimated from historical information. That information must be used with caution, but indicates the waste contains a layer of supernate and two layers of sludge with high concentrations of sodium, uranium, carbonate, phosphate, sulfate, and hydroxide. In addition, high concentrations of strontium and cesium should be present.

Analysis of the 1995 core samples indicates that the moisture content of tank 241-BX-103 sludge is significantly above the safety screening criterion of 17 percent with the exception of sludge from the upper half of segment 2, core 87. The lower 95 percent confidence limit on the mean for this segment portion was 16.8 percent water, and the mean of sample and duplicate runs was 23 percent. The next lowest moisture content for a sludge was 29.4 percent found in the lower half of segment 2, core 86. Exotherms were observed on four samples, the largest of which was 423 J/g on a dry-weight basis. Although all individual results were below the safety screening criterion of 480 J/g, the upper 95 percent confidence limit on the mean was 487 J/g (dry) for the sludge from the lower half of segment 1, core 87. However, the moisture in this segment portion was found to be 61.3 percent. All total alpha results were at least a factor of eight below the safety screening criterion. Additionally, the heat load of 132 W from the radioactive decay of radionuclides was well below the threshold of 11,700 W that separates high-heat from low-heat tanks.

The 1995 sampling and analysis event for tank 241-BX-103 does not appear to have met the sampling requirement of the safety screening DQO. Two risers radially separated by approximately 180° were sampled, but the lower half segment portion from segment 2 of core 87 was not obtained. All primary safety screening analyses on the sludge and drainable liquid were completed, and tank headspace flammability measurements were performed. Safety screening results from the sludge and drainable liquid samples at the 95 percent confidence level do not indicate any safety concerns. The tank headspace was measured at 0 percent of the LFL, thus tank headspace flammability is not an issue with tank 241-BX-103.

The analytical results for percent water and total alpha from the two core samples were compared to the TLM document in Section 5.4. Neither the TGA nor the total alpha data compare well with the tank inventory estimate. This discrepancy may be caused by a TLM over-estimation of the amount of metal waste in the tank, the presence of unknown waste types, and lack of activities of alpha-emitting isotopes other than Pu. It is possible that the sludge is largely composed of other waste types (see Section 2.3.2). The segment extrusion and TGA data do support the layering model found in the TLM document. Tank waste layering is apparent based on photographic and videographic information as well as the analytical results, the tank fill history, and the visual descriptions of the extruded samples.

It is recommended that the following activities be carried out to resolve deficiencies in meeting the safety screening DQO requirements for the 1995 push-mode core samples from tank 241-BX-103.

1. Sample from the lower half sludge of segment 1, core 87 should be re-evaluated by DSC and TGA analyses. If the upper limit to the one-sided 95 percent confidence limit on the mean for the DSC analysis still does not meet the safety screening criterion, perhaps sample material from that subsegment should be analyzed for secondary safety screening analytes to determine the specific safety category to which the tank belongs. The secondary analyses required are adiabatic calorimetry, TOC, and cyanide if the TOC content is less than 3 percent.

2. The characterization project should determine whether further sampling of tank 241-BX-103 is required to obtain sample from the lower half of segment 2.

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APPENDIX A

ANALYTICAL RESULTS FROM TANK 241-BX-103 1974-1975 SUPERNATE SAMPLES

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A1.0 INTRODUCTION

Appendix A provides the analytical data for the five supernate samples removed from tank 241-BX-103 from November 1974 to December 1975. A description of the sampling event and the sample is in Section 3.3.

The supernate samples most likely were taken in preparation for supernate waste transfers from tank 241-BX-103.

Analytical results are shown in Table A-1.

Table A-1. Analytical Results from Tank 241-BX-103, 1974-1975 Supernate Samples.

Sample Date mm/dd/yy	Desc	pH	SpG	Al	Na	NO_2	NO_3	PO_4	CO_3	OH	F	Pu	^{106}Ru/Rh	^{134}Cs	^{137}Cs	^{103}Ru	^{125}Sb	$^{89/90}$Sr	H_2O
				(M)	(M)	(M)	(M)	(M)	(M)	(M)	(M)	g/gal[1]	µCi/gal	µCi/gal	µCi/gal	µCi/gal	µCi/gal	µCi/gal	%
11/25/74[2]	Yellow, n/solids	12.9	1.063	0.0261	1.44	0.292	0.274	0.0401	0.204	0.353	1.11E-02	2.53	96.1E+3	n/a	1.12E+05	n/a	n/a	157	93.28
3/17/75	Brown <1% solids	12.7	1.0373	n/a	n/a	n/a	n/a	n/a	n/a	0.359	n/a	n/a	n/a	7.70E+03	6.52E+05	n/a	9.24E+03	n/a	81.93
6/18/75	Brown, n/solids	11.8	1.1243	0.00605	n/a	0.908	0.433	n/a	0.620	0.788	n/a	n/a	2.21E+05	924	1.79E+05	n/a	n/a	6.01E+03	99.14
4/14/75	Lt. brown, n/solids	12.0	1.1383	0.0238	2.02	0.582	0.504	0.0114	0.407	0.264	7.02E-03	4.62	1.62E+05	8.61E+02	2.19+05[3]	1.01E+03	n/a	3.48E+04	92.69
10/23/75	Yellow, n/solids	10.6	1.103	2.51E-03	1.98	0.433	0.339	0.0109	0.555	<4.74E-03	3.84E-03	4.92E-04	1.58E+05	846	1.09E+05	n/a	n/a	1.68E+04	86.16

Notes:

n/solids = no solids

[1]Data from Wheeler (1974) gives units in molarity and is most likely in error.

[2]Date of analytical report. Sample date is not given.

[3]Listed in Wheeler (1975b) as Cs-139 and is most likely in error.

APPENDIX B

PHOTOGRAPHS OF TANK 241-BX-103 EXTRUDED CORES 86 AND 87: 1995 CORE SAMPLES

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B1.0 INTRODUCTION

Appendix B contains photographs of extruded segments from cores 86 and 87 removed from tank 241-BX-103 in May 1995. Each core consisted of two segments. Each photograph is a composite of the two segments taken from the respective core sample. A continuous core sample from the bottom of the tank to the top can be visualized by arranging the segments so that the left (bottom) end of segment 1 is against the right (top) end of segment 2.

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Figure B-1. Tank 241-BX-103, Segment 1, Core 86.

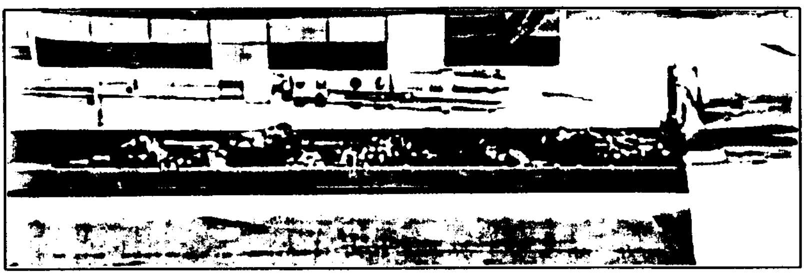

Figure B-2. Tank 241-BX-103, Segment 2, Core 86.

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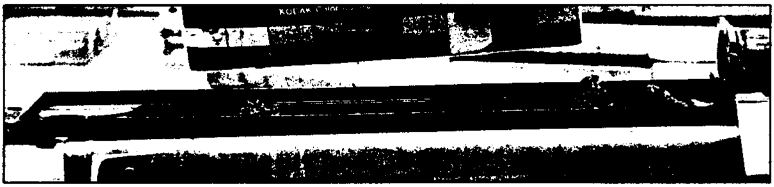

Figure B-3. Tank 241-BX-103, Segment 1, Core 87.

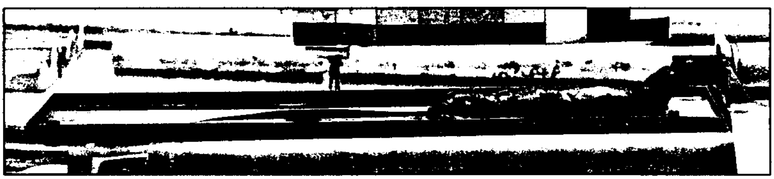

Figure B-4. Tank 241-BX-103, Segment 2, Core 87.

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