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7. 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-101. This report supports the requirements of Tri-Party Agreement Milestone M-44-08.

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Tank Characterization Report for Single-Shell Tank 241-BX-101

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EXECUTIVE SUMMARY

Single-shell tank 241-BX-101 is an underground storage tank containing high-level radioactive waste. It is located in the BX Tank Farm in the Hanford Site's 200 East Area. The tank was auger-sampled in June of 1994 and analysis was performed in accordance with WHC-SD-WM-SP-004, *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The analysis supports the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-08 (Ecology et al. 1994).

Tank 241-BX-101 went into service in 1948 receiving metal waste (produced in the bismuth phosphate process) from B Plant. Other waste types received by the tank during its service life were uranium recovery waste, Plutonium Uranium Extraction (PUREX) Facility cladding waste, evaporator bottoms, B Plant low-level waste, B Plant ion-exchange waste, PUREX organic wash wastes, and Reduction and Oxidation (REDOX) Facility ion-exchange waste.

Tank 241-BX-101 is the first tank in the 241-BX-101, 241-BX-102, 241-BX-103 cascade series. The tank has a capacity of 2,010 kL (530 kgal) and currently contains 163 kL (43 kgal) of waste, existing primarily as sludge, including 3.79 kL (1 kgal) of supernatant. The tank became a suspected leaker in 1973 and was removed from service. The tank was deactivated and the supernatant was pumped, interim stabilizing the tank in 1978. Intrusion prevention was completed in 1981.

The Safety Screening Data Quality Objective (DQO) (Babad and Redus 1994) is the only current DQO that has been applied to tank 241-BX-101. Therefore, only those analyses required by that DQO were performed in 1994. The analyses included thermogravimetric analysis, differential scanning calorimetry, and total alpha activity. The analyses revealed that the water content in three of the six major subsamples was below the DQO-required limit of 17 wt%, ranging from 11.3 wt% to 16.1 wt% in those three major subsamples. The three subsamples that satisfied the water content requirement ranged from 18.7 wt% to 26.3 wt%. The one subsample that was excluded contained a rock and had a water content of 1.5 wt%. These water content values may pose a safety concern though no exotherms were observed and all of the total alpha results were below the DQO decision limit (41 μCi/g). However, the total alpha results do exceed the transuranic classification criterion (100 nCi/g). The flammability of the gas in the headspace of the tank is another safety screening consideration; however, analysis of the tank's headspace is presently outside the scope of this report.

The current status of tank 241-BX-101 is shown in Table ES-1. A summary of the estimated historical inventory is shown in Table ES-2. The only chemical and radiochemical analytical data for the tank is from a 1976 sampling taken prior to stabilization. These values are based on the historical tank layer model estimates presented in Brevick and Redus (1994).

Table ES-1. Tank 241-BX-101.

Tank description	
Type:	Single-shell
Constructed:	1947
In-service:	1948
Diameter:	23 m (75 ft)
Usable depth:	4.9 m (16 ft)
Capacity:	2,010 kL (530 kgal)
Bottom shape:	Dish
Ventilation:	Passive
Tank status	
Watch list:	Non-watch list
Contents:	Non-complexed waste
Total waste volume:	163 kL (43 kgal)
Sludge volume:	159 kL (42 kgal)
Supernatant volume:	3.79 kL (1 kgal)
Manual tape surface level:	30.0 cm (11.8 in.)
Temperature:	20 °C (68 °F) (2/15/95)
Integrity category:	Assumed leaker
Isolation status	
Interim stabilized:	1978
Intrusion prevention:	1981
Level adjustment:	1982

Table ES-2. Single-Shell Tank 241-BX-101 Historical Inventory Estimates Summary.[a,b]

Physical properties			
Total waste	163 kL (43 kgal)		
Heat load	4.04 kW (13,800 Btu/h)		
Bulk density	1.66 g/cc		
Void fraction	0.584		
Water wt%	54.4		
Total organic carbon wt%	0.074		
Chemical constituents			
Chemical Constituents	mole/L	ppm	kg
Na (sodium)	4.96	6.88 E+04	1.81 E+04
Al (aluminum)	0.615	1.00 E+04	2.64 E+03
Fe (total iron)	0.815	2.75 E+04	7.23 E+03
Cr (chromium)	2.86 E-03	89.8	23.6
Ni (nickel)	0.116	4.11 E+03	1.08 E+03
Ca (calcium)	9.08 E-02	2.20 E+03	578
OH^- (hydroxide)	11.4[c]	1.17 E+05	3.08 E+04
NO^{-3} (nitrate)	0.520	1.95 E+04	5.13 E+03
NO^{-2} (nitrite)	2.50 E-03	69.4	18.3
CO_3^{-2} (carbonate)	0.662	2.40 E+04	6.32 E+03
PO_4^{-3} (phosphate)	0.329	1.89 E+04	4.97 E+03
SO_4^{-2} (sulfate)	0.651	3.78 E+04	9.94 E+03
SiO_3^{-2} (silicate)	0.379	6.43 E+03	1.69 E+03
Cl^- (chloride)	1.79 E-02	382	101
$C_6H_5O_7^{-3}$ (citrate)	3.75 E-03	428	113
Glycolate	4.99 E-02	2.26 E+03	596
Radiological constituents			
		μCi/g	
Pu		0.274	1.20 kg
U	1.11 *M*	1.60 E+05	4.22 E+04 kg
Cs	6.22 E-04 Ci/L	0.375	98.8 Ci
Sr	3.77 Ci/L	2.28 E+03	5.99 E+05 Ci

Note: 1 Ci = 3.7 E+10 Bq.

[a]Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[b]Composite inventory excludes supernatant, diatomaceous earth, and cement. Unknowns in tank inventory are assigned by Agnew et al. 1994).

[c]Believed to be the pH and not molar OH^-.

REFERENCES

Babad, H., J. W. Hunt, and L. S. Redus, 1995, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order*, as amended, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

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LIST OF TERMS

ANOVA	analysis of variance
DQO	data quality objectives
DSC	differential scanning calorimetry
PUREX	Plutonium Uranium Extraction (Facility)
RPD	relative percent difference
TGA	thermogravimetric analysis

TANK CHARACTERIZATION REPORT FOR SINGLE-SHELL TANK 241-BX-101

1.0 INTRODUCTION

This tank characterization report presents an overview of the use and contents of single-shell tank 241-BX-101 and its waste contents based upon an evaluation of historical information and sampling and analysis events.

In June of 1994, single-shell tank 241-BX-101 was sampled to meet safety screening requirements for Hanford Site nuclear waste tanks (Carpenter 1994, Babad and Redus 1994). The analyses of the samples were used to complete this tank characterization report in order to comply with requirements specified in the *Hanford Federal Facility Agreement and Consent Order*, also known as the Tri-Party Agreement (Ecology et al. 1994).

The *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004 (Babad and Redus 1994), describes the sampling and analytical requirements used to screen waste tanks with no identified safety issues, tanks such as tank 241-BX-101. Both watch list and non-watch list tanks are to be sampled and evaluated to identify safety conditions related to the ferrocyanide, organic, flammable gas, and criticality safety issues. The analytical requirements for safety screening are designed to identify a common set of primary analytes and waste characteristics; these include energetics, total alpha activity, moisture, and flammable gas concentrations.

This tank characterization report presents an overview of the tank sampling and analysis effort and contains observations regarding waste characteristics. Chemical information associated with tank 241-BX-101, however, is highly contained since the analysis of the waste was governed solely by the Tank Safety Screening Data Quality Objective (DQO) (Babad and Redus 1994), which requires that only limited analyses be performed. As a consequence, the concentration and inventory estimates of individual chemical species are based upon the estimates in WHC-SD-WM-ER-349, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area* (Brevick et al. 1994). The information presented represents the best estimate of the waste composition based on historical knowledge and the 1994 sampling and analysis event.

1.1 PURPOSE

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

1.2 SCOPE

This report presents historical information based on process knowledge and early sampling and analysis. The results from the 1994 tank 241-BX-101 sample analyses for safety screening are then summarized and presented. The historical information also is compared with the actual waste measurements and recommendations are given based upon the current waste inventory and tank status where possible.

Because the 1994 screening analyses only included differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and total alpha measurements, historical information is used for most chemical and radiochemical values. This report does not contain information on vapor space sampling and analysis for determination of the composition of tank headspace gases.

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-BX-101 based upon historical information. The first part of this section discusses the current status of the tank. This is followed by discussions of the tank's design and background. Process knowledge is then presented, including transfer history and process sources and an estimate of the current contents based upon the process history of the tank. The final subsection details current surveillance data on the tank.

2.1 TANK STATUS

The most recent waste inventory report (Hanlon 1995) for tank 241-BX-101 shows the tank contains 163 kL (43,000 gal) of non-complexed waste. This includes 3.79 kL (1,000 gal) of supernatant and 159 kL (42,000 gal) of sludge. The most recent temperature in tank 241-BX-101 was 20 °C (68 °F) (2/15/95). Liquid levels and tank temperatures are further discussed in Section 2.4.2. Tank 241-BX-101 is an inactive, single-shell, non-watch list tank. Ventilation for tank 241-BX-101 is through a passive breather filter, and all monitoring systems are currently in compliance with established standards (Hanlon 1995).

The tank is categorized as an assumed leaker, has been interim stabilized, and has undergone intrusion prevention. A tank is considered interim stabilized if it contains less than 189,000 L (50,000 gal) of drainable interstitial liquid and less than 18,900 L (5,000 gal) of supernatant liquid. Removing liquids minimizes the risk of waste leaking out of the tank. Intrusion prevention is the administrative designation reflecting the completion of the physical effort required to minimize inadvertent addition of liquids into an inactive storage tank (Hanlon 1995).

2.2 TANK DESIGN AND BACKGROUND

Tank 241-BX-101 is constructed of reinforced concrete with a mild steel liner covering its bottom and sides. The top of the tank is a concrete dome. The tank has a diameter of 23 m (75 ft), a usable depth of 4.9 m (16 ft), and a capacity of 2,010,000 L (530,000 gal) (Husa et al. 1993). The bottom of the tank is dish shaped. The basic design of tank 241-BX-101 is shown in Figure 2-1. Instrument access to tank 241-BX-101 is available through risers to monitor temperature, sludge level, and other bulk tank characteristics (Alstad 1991). The positions of these risers are found in Figure 2-2.

The 241-BX Tank Farm, built between 1946 and 1947, is on the eastern side of the 200 East Area. Figure 2-3 details the Hanford Site's 200 East Area and the location of the 241-BX Tank Farm. Figure 2-2 also shows the position of tank 241-BX-101 within the 241-BX Tank Farm.

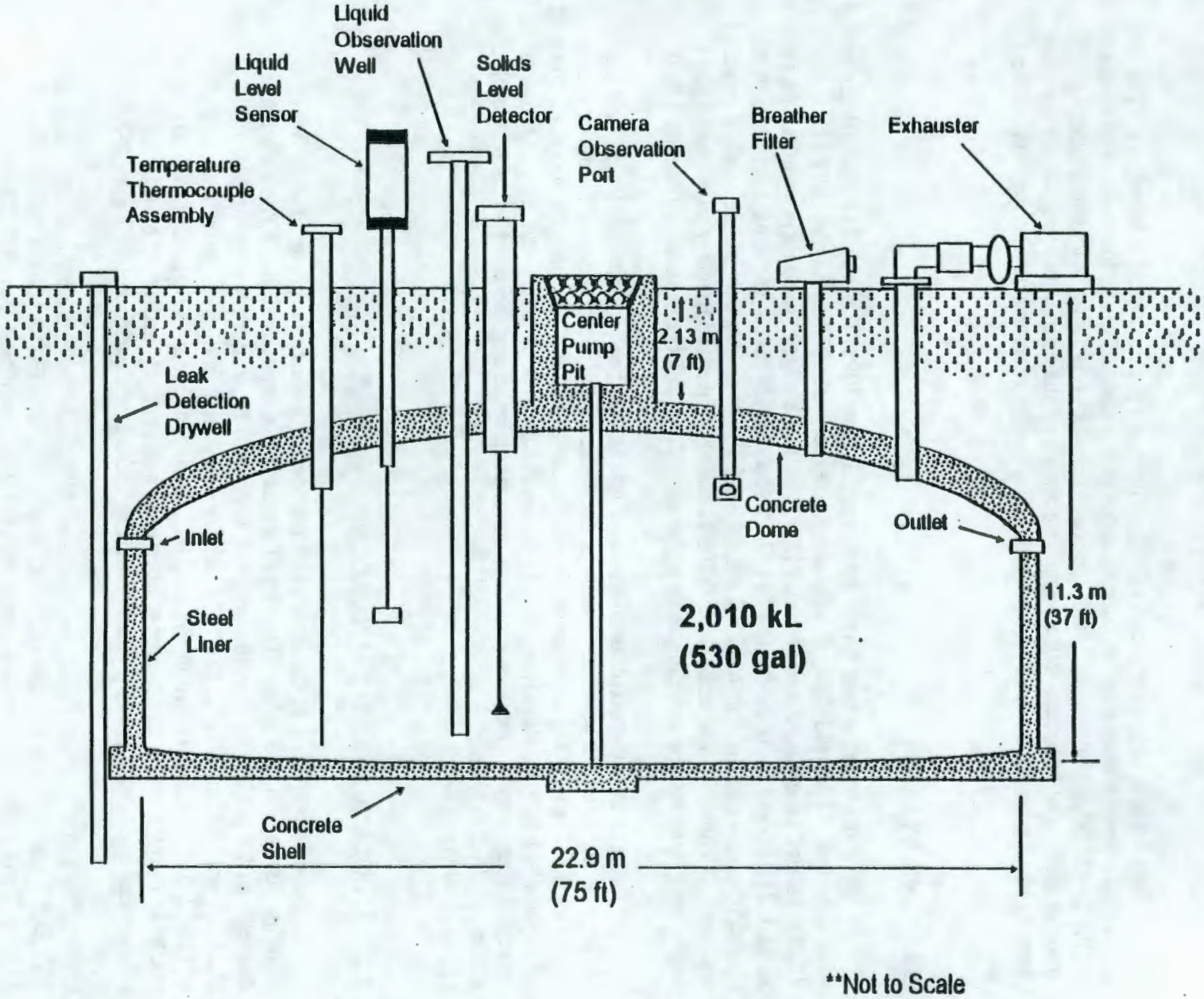
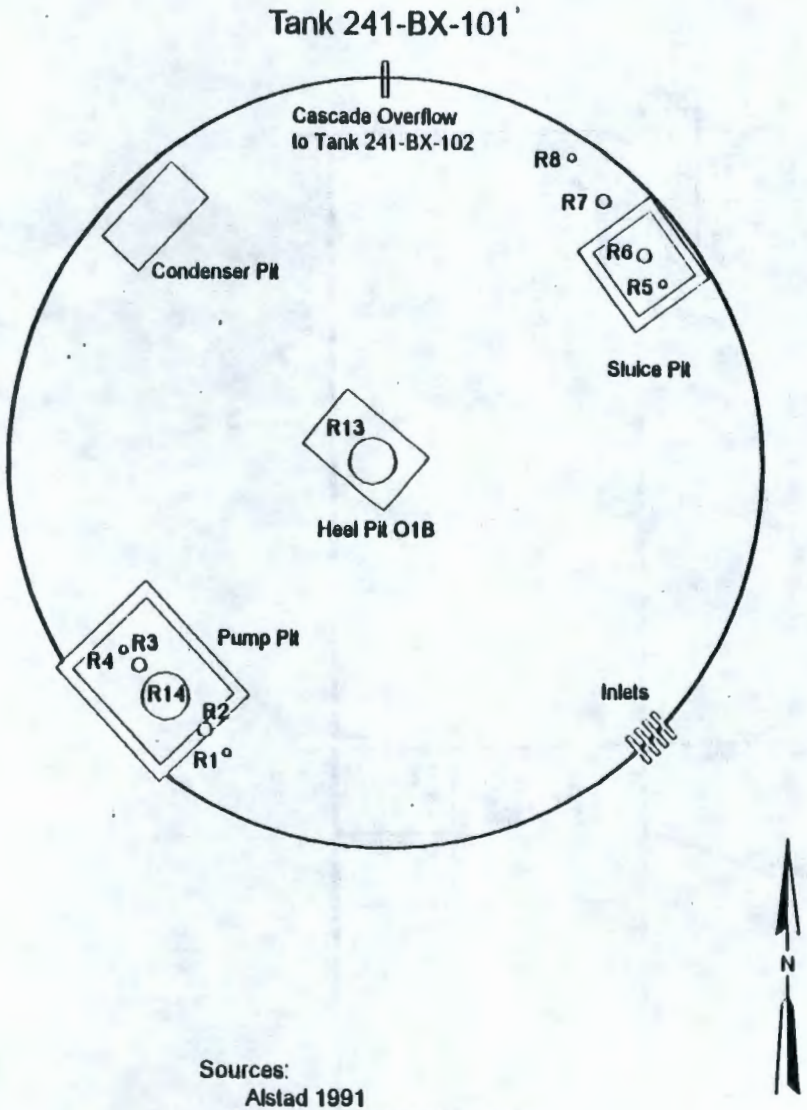


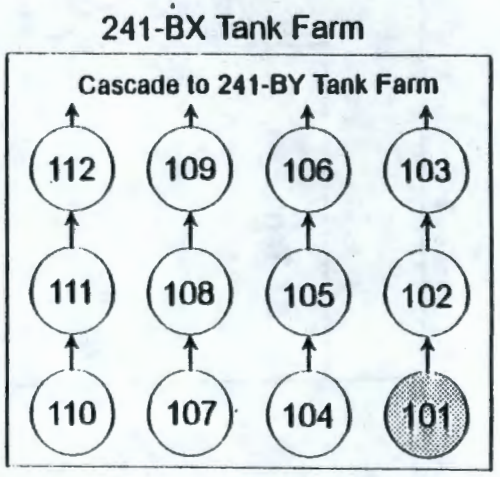
Figure 2-1. Basic Design of Tank 241-BX-101.

Figure 2-2. Riser Configuration for Tank 241-BX-101.



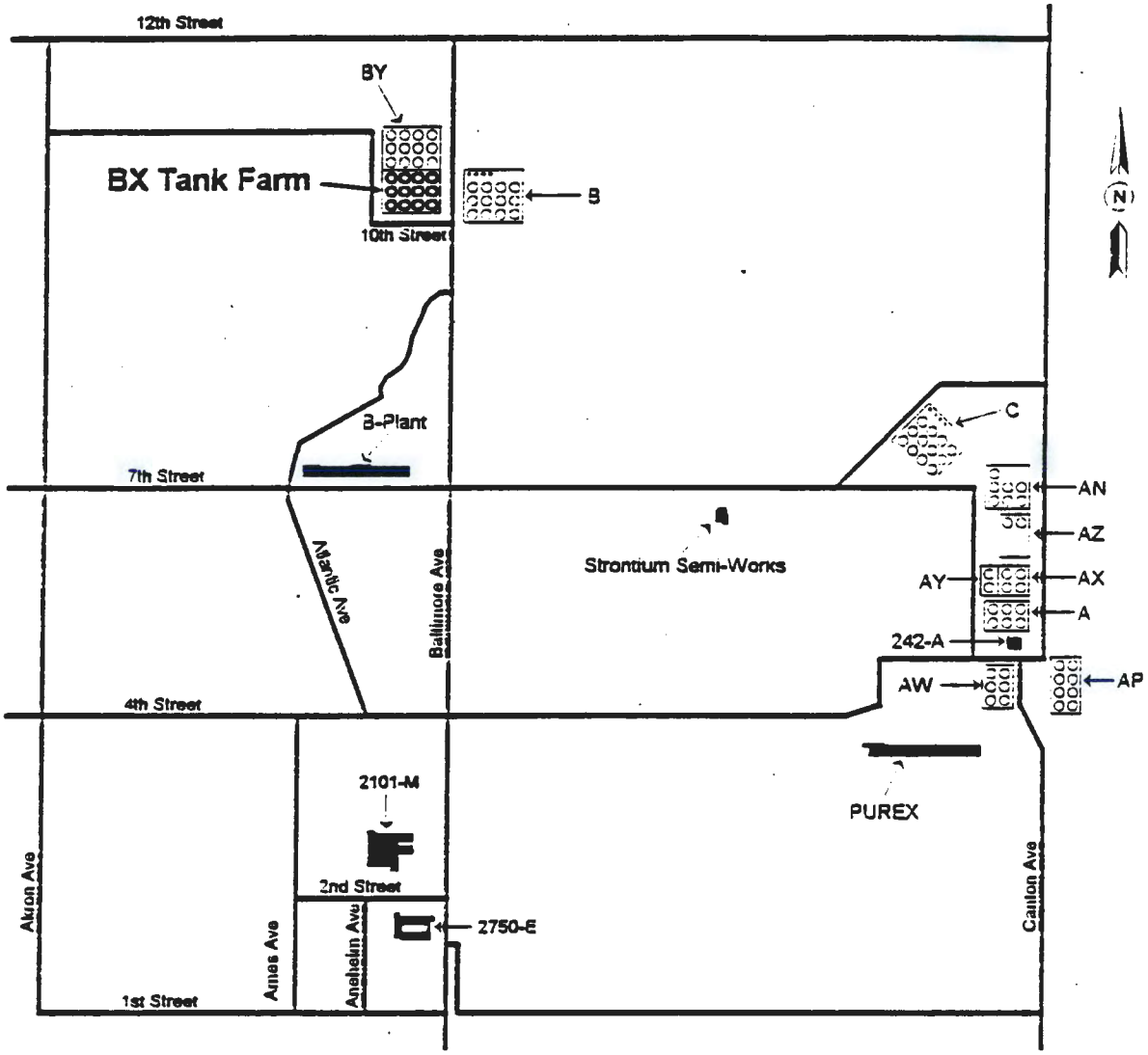
No.	Dia.	Description and Comments
R1	4"	Breather Filter, BM*
R2	12"	Temperature, WC**
R3	12"	Sluicing Nozzle, WC
R4	4"	Recirculating Dip Tube, WC
R5	4"	Recirculating Dip Tube, WC
R6	12"	Sluicing Nozzle, WC
R7	12"	Observation Port
R8	4"	Manual Tape
R13	36"	Heel Jet, WC
R14	42"	Pump, WC

*Benchmark
**WC-weather covered urethane foam



Sources:
Alstad 1991
Vitro Engineering Corp. 1986
Brevick, Gaddis, and Johnson 1994

Figure 2-3. Location of the 241-BX Tank Farm.



Tank 241-BX-101 is the first tank in a "cascade" connecting it to tanks 241-BX-102 and 241-BX-103. A cascade was a waste management system in which a number of tanks were connected in series by pipes located near the top of the tanks' working depths. Waste added to the first, or primary, tank in a cascade would flow to the following tanks when the waste reached the level of the tank's cascade piping. By using a cascade, fewer connections needed to be made during waste handling operations. This method reduced waste handling requirements, personnel exposure, and the chance of a loss of tank integrity from tank overflow. Another advantage of using the cascade system was waste volume reduction from the disposal of clarified liquid waste. Much of the entrained and precipitated solids would settle in the primary tank (in this case tank 241-BX-101) and the clarified liquids would flow through the cascade to the secondary tanks (241-BX-102 and 241-BX-103). This practice led to rapid accumulation of solids in the primary tank and disposal of clarified liquids from secondary tanks into cribs.

Tank 241-BX-101 went into service in 1948 and received a variety of waste types during its active service life. A comprehensive transfer history of the tank is found in the Section 2.3.

Tank 241-BX-101 was removed from service in 1975. The tank was interim stabilized in 1978 after salt well pumping was completed (Welty 1988). Intrusion prevention was completed in 1981. The administrative designation intrusion prevention replaced the designation interim isolation in June of 1993 and carries similar requirements (Hanlon 1995).

2.3 PROCESS KNOWLEDGE

2.3.1 Waste Transfer History

This section presents the transfer history of tank 241-BX-101 and describes the process wastes that made up those transfers. The majority of the transfer history information for this section is found in WHC-SD-WM-TI-615, *Waste Status and Transaction Record Summary for the Northeast Quadrant* (Agnew 1994a). Table 2-1 presents the transfer history of tank 241-BX-101. The following discussion accompanies and expands on the table. The text describes the characteristics of waste types that entered the tank and the conditions of their entry. Unless otherwise specified, information about waste type characteristics is taken from WHC-SD-WM-TI-629, *Hanford Defined Waste: Chemical and Radionuclide Compositions* (Agnew 1994b).

The first waste that tank 241-BX-101 received was metal waste, which began to enter the tank in January of 1948. This waste was added directly to tank 241-BX-101 and overflowed through cascade lines to tank 241-BX-102. Metal waste was produced early in the bismuth phosphate process at B Plant and contained a high concentration of sodium uranyl phosphate solids (Rodenhizer 1987).

Table 2-1. Waste Transfer History for Tank 241-BX-101.[a b] (3 sheets)

Year:	Waste received by tank 241-BX-101			Waste transferred out of tank 241-BX-101		
	Transaction source	Waste type	Waste amount[c] (L)	Transfer destination	Waste amount[c] (L)	Total tank volume[c] (L)
1948	B Plant	MW	5.87 E+06	241-BX-102	3.86 E+06	2.01 E+06
1948	B Plant	MW	1.25 E+03	241-BX-102	1.25 E+03	2.01 E+06
1950	B Plant	MW	6.29 E+06	241-BX-102	6.29 E+06	2.01 E+06
1951	B Plant	MW	2.01 E+06	241-BX-102 Leak	1.66 E+06 3.50 E+05	2.01 E+06
1952	–	–	0	–	–	2.01 E+06
1953	UNK	UNK	1.32 E+05	UNK	1.38 E+06	7.57 E+05
1954-1956	–	–	–	241-BY-103 UNK	4.16 E+05 3.41 E+05	0
1956	241-BY-106	UR	1.98 E+06	#13BC ditch	1.85 E+06	1.30 E+05
1957-1961	UNK 67 LD UNK	Water UNK UNK	2.54 E+05 4.9 E+04 1.17 E+05	–	–	5.49 E+05
1962-1967	241-C-103 241-C-102 UNK Correction	All UR, CWP	1.14 E+06 1.51 E+06 1.17 E+05 2.3 E+04	UNK Correction	1.19 E+06 7.57 E+03	2.14 E+06
1967-1968	Cell 23	EB	6.62 E+05	241-B-112	1.48 E+06	1.32 E+06
1968	Cell 23 B Plant (221-B)	EB BL	2.88 E+05 5.45 E+05	241-BX-102 241-BX-102	1.04 E+06 3.56 E+05	7.57 E+05
1969-1970	B Plant (221-B)	BL	1.27 E+06	UNK 241-C-103	1.32 E+05 1.46 E+06	4.35 E+05
1970	B Plant UNK 241-B-101 B Plant 241-C-104	IX BL Misc. supernatant IX OWW, CWP Misc. supernatant	3.32 E+06 3.79 E+03 1.65 E+06 2.08 E+06 3.21 E+06	241-BX-103 241-BX-106 241-BX-103	1.53 E+06 1.13 E+06 7.1 E+06	9.4 E+05

Table 2-1. Waste Transfer History for Tank 241-BX-101.[a b] (3 sheets)

Year:	Waste received by tank 241-BX-101			Waste transferred out of tank 241-BX-101		
	Transaction source	Waste type	Waste amount[c] (L)	Transfer destination	Waste amount[c] (L)	Total tank volume[c] (L)
1970-1971	241-C-104	OWW, CWP, TH, THL	7.43 E+06			
				241-BX-106	8.62 E+06	
	B Plant	IX	1.374 E+07			
				241-BX-103	1.637 E+07	
	241-B-101	Misc. supernatant	3.7 E+06			8.2 E+05
1971	241-B-101	Misc. supernatant	1.72 E+06			
	241-BY-101		2.96 E+06			
	241-C-104	Misc. supernatant	4.13 E+06			
	A-302 CT		4.5 E+04			
		OWW, CWP, UNK		241-BX-104	3.21 E+05	
				241-BX-106	8.7 E+06	
				241-BX-103	4.53 E+06	
	B Plant	Misc. supernatant	5.64 E+06			1.76 E+06
		UNK				
1971-1972	241-C-104	OWW, CWP	7.23 E+05			
				241-SX-103	1.36 E+06	
	B Plant	IX	1.5 E+07			
				241-SX-105	1.4 E+07	
				241-TX-101	7.57 E+05	
	241-B-101	Misc. supernatant	1.51 E+06			
				241-SX-101	1.6 E+06	1.28 E+06
1972	241-B-101	Misc. supernatant	3.66 E+06			
				241-SX-102	2.81 E+06	
	B Plant	IX	1.09 E+07			
				241-BX-103	1.68 E+06	
				241-SX-105	1.477 E+06	
				241-TX-101	6.65 E+06	
				241-T-101	1.88 E+06	
				241-UX CT	1.5 E+04	
				241-BX-104	7.76 E+05	5.52 E+06
1973-1976				UNK	1.5 E+04	
	UNK	BL, IX	8.71 E+04			
				241-BX-104	4.54 E+05	
	241-BX-104	Misc. Sup.	3.79 E+03			1.74 E+05

Table 2-1. Waste Transfer History for Tank 241-BX-101.[a,b] (3 sheets)

Year:	Waste received by tank 241-BX-101			Waste transferred out of tank 241-BX-101		
	Transaction source	Waste type	Waste amount[c] (L)	Transfer destination	Waste amount[c] (L)	Total tank volume[c] (L)
1976-1993	–	–	–	–	–	1.74 E+05
1993-present	–	–	–	UNK	1.14 E+0[d]	1.63 E+05

[a] Agnew, S. F., 1994, *Waste Status and Transaction Record Summary for the Northeast Quadrant*, WHC-SD-WM-TI-615, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[b] Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[c] All volume amounts are listed in liters. Conversion of liters into gallons is done using the equivalency of 1 L = 0.264 gal.

[d] No waste has been transferred from the tank. This volume corresponds to a level adjustment to an incomplete transfer history.

BL = B Plant low-level waste.
CT = catch tank.
CWP = cladding waste from the Plutonium Uranium Extraction Facility.
EB = evaporator bottoms waste.
IX = ion-exchange waste.
LD = line drainage.
MW = metal waste.
OWW = organic wash waste.
TH = thoria high-level waste or cladding waste.
THL = thoria low-level waste.
UNK = unknown.
UR = uranium recovery waste.

The metal waste from the bismuth phosphate process was so high in uranium that it was decided to remove the metal waste from the storage tanks to recover the uranium for reuse. This uranium recovery was accomplished at U Plant using the tributyl phosphate process. In 1953, tank 241-BX-101 was sluiced to remove its inventory of metal waste. Unknown transfers into the tank occurred in 1953. Following sluicing, the tank was declared empty in 1954, with only a small waste heel remained.

Tank 241-BX-101 was filled in the third quarter of 1956 with scavenged uranium recovery supernatant received from tank 241-BY-106. At that time, tank 241-BY-106 was the active receiver tank for U Plant. Uranium recovery waste contained comparatively large amounts of phosphate, sodium, sulfate, uranium, and ^{90}Sr. Tank 241-BY-106 was also a ferrocyanide settling tank. Scavenged supernatant was transferred here prior to its disposal to ground.

In the fourth quarter of 1956, much of the contents was transferred to a ditch. In the first quarter of 1957, the tank received waste from the line drainage.

The tank received uranium recovery waste and cladding waste in the second and fourth quarters of 1962 from tanks 241-C-102 and 241-C-103. The tank stood idle until the third quarter of 1967 when it transferred uranium recovery waste and cladding waste to tank 241-B-112. Cladding waste, produced during the dissolution of aluminum fuel cladding at the Plutonium Uranium Extraction (PUREX) Plant, was comparatively high in aluminum, silica, sodium, and hydroxide.

During the first and third quarters of 1968, the tank contained uranium recovery waste, cladding waste, and evaporator bottoms waste. The evaporator bottoms waste was received from cell 23 of B Plant. The evaporator bottoms waste was a salt slurry high in nitrates and sodium.

From the fourth quarter of 1968 to the second quarter of 1970, tank 241-BX-101 contained B Plant low-level waste from B-221. This waste was produced during processing of the PUREX acid stream in B Plant. The supernatant that tank 241-BX-101 received was comparatively high in nitrates, sodium, and glycolate. The waste also contained a modest concentration of citrate.

Starting in the second quarter of 1970, the tank received B Plant ion-exchange waste that had been generated during cesium removal in preparation for in-tank solidification. Ion-exchange waste was a dilute product of cesium recovery at B Plant. It contained significant concentrations of uranium and citrate and a trace of organic N-(2-hydroxyethyl)-ethylenediaminetriacetic acid.

Between the second quarter of 1970 and the second quarter of 1972, the tank received various wastes: organic wash waste, thoria high-level waste or cladding waste, thoria low-level waste, cladding waste, ion-exchange waste, and Reduction and Oxidation Plant ion-exchange waste. The waste was received from B Plant and from tanks 241-B-101 and 241-C-104. Some of this waste was transferred to tanks 241-BX-103, 241-BX-106, 241-SX-101, 241-SX-105 and 241-TX-101. The organic wash waste and thoria waste were from the PUREX Plant.

The tank received B Plant low-level waste and ion-exchange waste from the third quarter of 1972 until the first quarter of 1976. The tank became a suspected leaker in 1973, and the waste was transferred to tank 241-BX-104.

The process history of tank 241-BX-101 is presented graphically in Figure 2-4. Table 2-2 presents an estimate of the total volumes of the specific waste types that were added to the tank and an estimate of the volumes of specific waste types that remain in the tank.

Figure 2-4. Waste Volume History of Tank 241-BX-101.

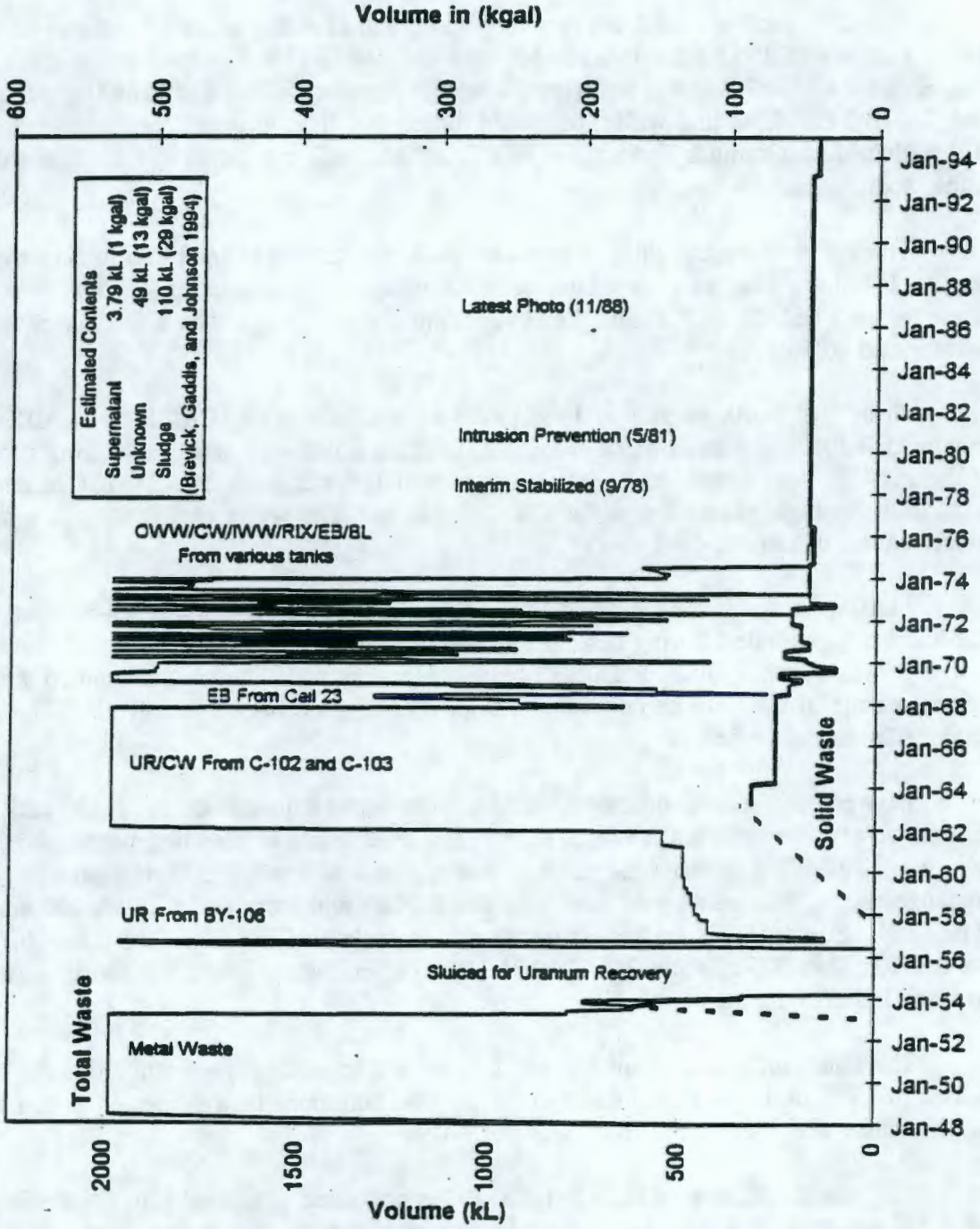


Table 2-2. Estimated Total and Current Volumes of Waste Types Received By Tank 241-BX-101.[a,b,c]

Waste type	Estimated volume L (gal)	Years received	Estimated current volume[d] L (gal)
MW	14.3 E+06 L (3.78 Mgal)	1948-1951	110,000 L (29,000 gal)
UR	1.98 E+06 L (523 kgal)	1956	–
UR, CWP	2.79 E+06 L (737 kgal)	1962-1967	–
EB	0.95 E+06 L (251 kgal)	1968	–
BL	1.82 E+06 L (481 kgal)	1968-1970	–
OWW, CWP	8.06 E+06 L (2.13 Mgal)	1970-1971	–
OWW, CWP, TH, THL	7.43 E+06 L (1.96 Mgal)	1971	–
Unknown / miscellaneous supernatant	21.2 E+06 L (5.6 Mgal)	1953-1976	49,300 L (13,000 gal)
BL, IX	87,100 L (23,000 gal)	1972-1976	–

[a] Agnew, S. F., 1994, *Waste Status and Transaction Record Summary for the Northeast Quadrant*, WHC-SD-WM-TI-615, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[b] Agnew, S. F., 1994, *Hanford Defined Wastes: Chemical and Radionuclide Compositions*, WHC-SD-WM-TI-629, Rev. 5, Westinghouse Hanford Company, Richland, Washington.

[c] Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[d] From tank layer model estimates; included in the tank layer model but not shown on the table is 3,790 L (1,000 gal) of supernatant.

BL = B Plant low-level waste.
CWP = waste from the dissolution of aluminum fuel cladding at the Plutonium Uranium Extraction Facility.
EB = evaporator bottoms waste.
IX = dilute ion-exchange waste returned from cesium recovery.
k = thousand.
M = million.
MW = metal waste from the $BiPO_4$ Process at B Plant.
OWW = organic wash waste.
TH = thoria high-level waste or cladding waste.
THL = thoria low-level waste.
UR = uranium recovery waste.

2.3.2 Historical Estimate of Tank Contents for Tank 241-BX-101

A tank layer model has been developed at Los Alamos National Laboratory (Agnew et al. 1994) that can be used to combine waste type inventories and historical waste stream data to arrive at an estimate of the current chemical contents of tank 241-BX-101 (Brevick et al. 1994). The estimated current waste volumes shown in Table 2-2 were derived using this model. The tank layer model uses these current volumes to calculate the current tank analytes and their concentrations. These estimates are given in Table 2-3.

2.4 SURVEILLANCE DATA

2.4.1 Surface Level Readings

To determine the surface level of the waste, tank 241-BX-101 is equipped with a manual tape surface level gauge. Surface level readings are currently being taken daily. Figure 2-5 graphically represents the surface level measurements from January 1991 to the present. The reading recorded on February 21, 1995, was 30.0 cm (11.8 in.). The surface level data for the past 3 years has ranged between 24.1 and 30.5 cm (9.5 and 12 in.).

Two dry wells have been identified for tank 241-BX-101. Graphical representations of the active drywells from January of 1990 to the present can be found in WHC-SD-WM-ER-311, *Supporting Document for the Northeast Quadrant Historical Tank Content Estimate Report for BX Tank Farm* (Brevick 1994). Each graph includes the peak counts per second (some wells have multiple peaks), the depth at which the peak occurred, and the date of the reading. The dry wells have remained stable during the review period.

2.4.2 Internal Tank Temperatures

Local tank temperatures are measured by a thermocouple tree (a probe with 14 thermocouples assembled in a pipe). The thermocouple tree monitors the waste temperatures at various levels in the tank. The thermocouple tree in tank 241-BX-101 is inserted through riser 2. See Figure 2-2 for riser locations. Thermocouples 1 through 12 on the tree in riser 2 have similar readings that span the time from 1974 to 1993. Two data points, from 1974 and from 1982, are available for thermocouple 13. No data are available for thermocouple 14, and no data are available for any thermocouples between 1985 and 1992. The tank is connected to the Tank Monitoring and Acquisition Control System, which continuously monitors the tank's thermocouples and downloads readings once a day to the Computer Automated Surveillance System.

Table 2-3. Single-Shell Tank 241-BX-101 Historical Inventory Estimates Summary.[a,b]

Physical properties			
Total waste	163 kL (43 kgal)		
Heat load	4.04 kW (13,800 Btu/h)		
Bulk density	1.66 g/cc		
Void fraction	0.584		
Water wt%	54.4		
Total organic carbon wt%	0.074		
Chemical constituents			
	mole/L	ppm	kg
Na (sodium)	4.96	6.88 E+04	1.81 E+04
Al (aluminum)	0.615	1.00 E+04	2.64 E+03
Fe (total iron)	0.815	2.75 E+04	7.23 E+03
Cr (chromium)	2.86 E-03	89.8	23.6
Ni (nickel)	0.116	4.11 E+03	1.08 E+03
Ca (calcium)	9.08 E-02	2.20 E+03	578
OH^- (hydroxide)	11.4[c]	1.17 E+05	3.08 E+04
NO^{-3} (nitrate)	0.520	1.95 E+04	5.13 E+03
NO^{-2} (nitrite)	2.50 E-03	69.4	18.3
CO_3^{-2} (carbonate)	0.662	2.40 E+04	6.32 E+03
PO_4^{-3} (phosphate)	0.329	1.89 E+04	4.97 E+03
SO_4^{-2} (sulfate)	0.651	3.78 E+04	9.94 E+03
SiO_3^{-2} (silicate)	0.379	6.43 E+03	1.69 E+03
Cl^- (chloride)	1.79 E-02	382	101
$C_6H_5O_7^{-3}$ (citrate)	3.75 E-03	428	113
Glycolate	4.99 E-02	2.26 E+03	596
Radiological constituents			
		μCi/g	
Pu		0.274	1.20 kg
U	1.11 M	1.60 E+05	4.22 E+04 kg
Cs	6.22 E-04 Ci/L	0.375 μCi/g	98.8 Ci
Sr	3.77 Ci/L	2.28 E+03	5.99 E+05 Ci

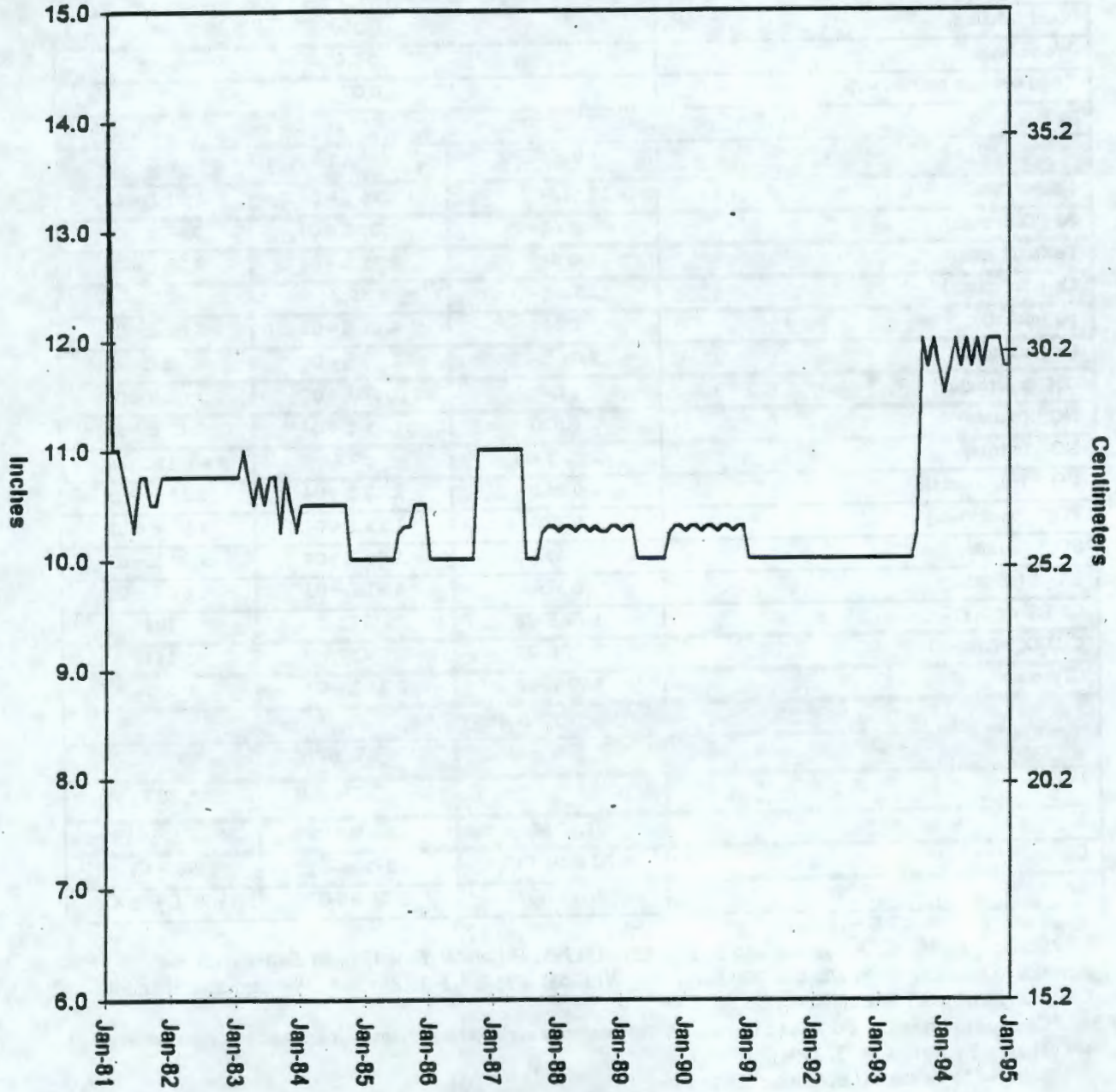
Note: 1 Ci = 3.7 E+10 Bq.

[a]Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[b]Composite inventory excludes supernatant, diatomaceous earth, and cement. Unknowns in tank inventory are assigned by Agnew et al. 1994).

[c]Believed to be the pH not molar OH^-.

Figure 2-5. Tank 241-BX-101 Surface Level Data.



Temperature readings for tank 241-BX-101 since 1975 are plotted in Figure 2-6. Each plotted temperature point is the highest of the readings recorded for that week by the thermocouples. High temperatures in the tank have tended to be registered by thermocouples 1 and 2. Only thermocouple 1 is actually in the waste. The others are in the tank's vapor space. The last available temperature reading for tank 241-BX-102 was 20 °C (68 °F) taken on February 15, 1995.

2.4.3 Tank 241-BX-101 Photographs

Figure 2-7 presents the 1988 photographic montage of tank 241-BX-101. It appears that the tank interior has a dark brown sludge ring around the perimeter with a major portion of the surface covered with translucent, reddish-brown liquid. The quality of these photographs is good and the equipment throughout the tank is clearly visible and has been identified and labelled. The picture was taken in 1988, when the tank held 3,790 L (1 kgal) of supernatant and 159,000 L (42 kgal) of sludge. There have been no changes in the tank that would affect the waste since these photographs were taken; therefore the picture should be representative of the tank's current contents (Brevick et al. 1994).

Figure 2-6. Tank 241-BX-101 Historical Temperature Data.

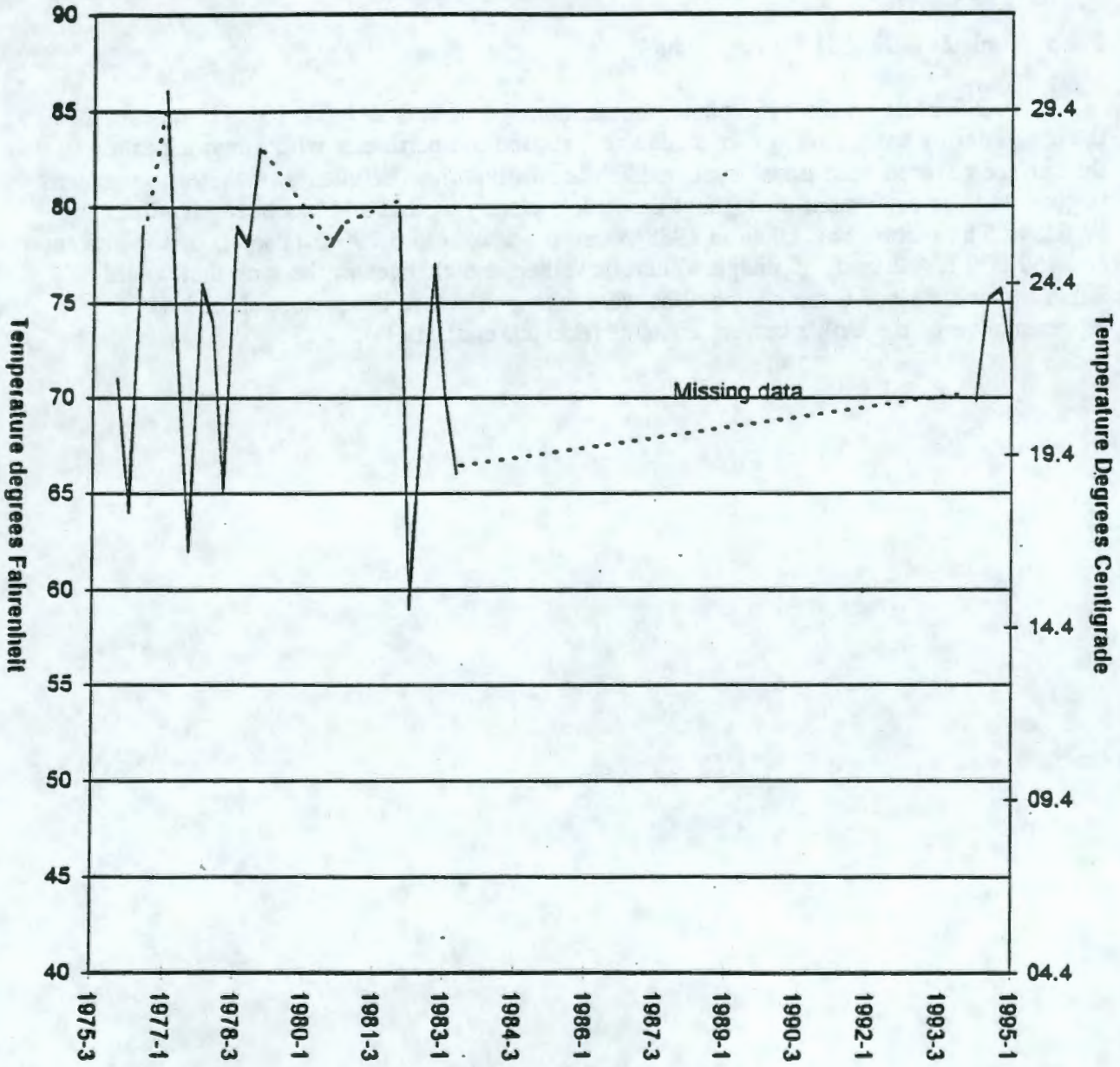
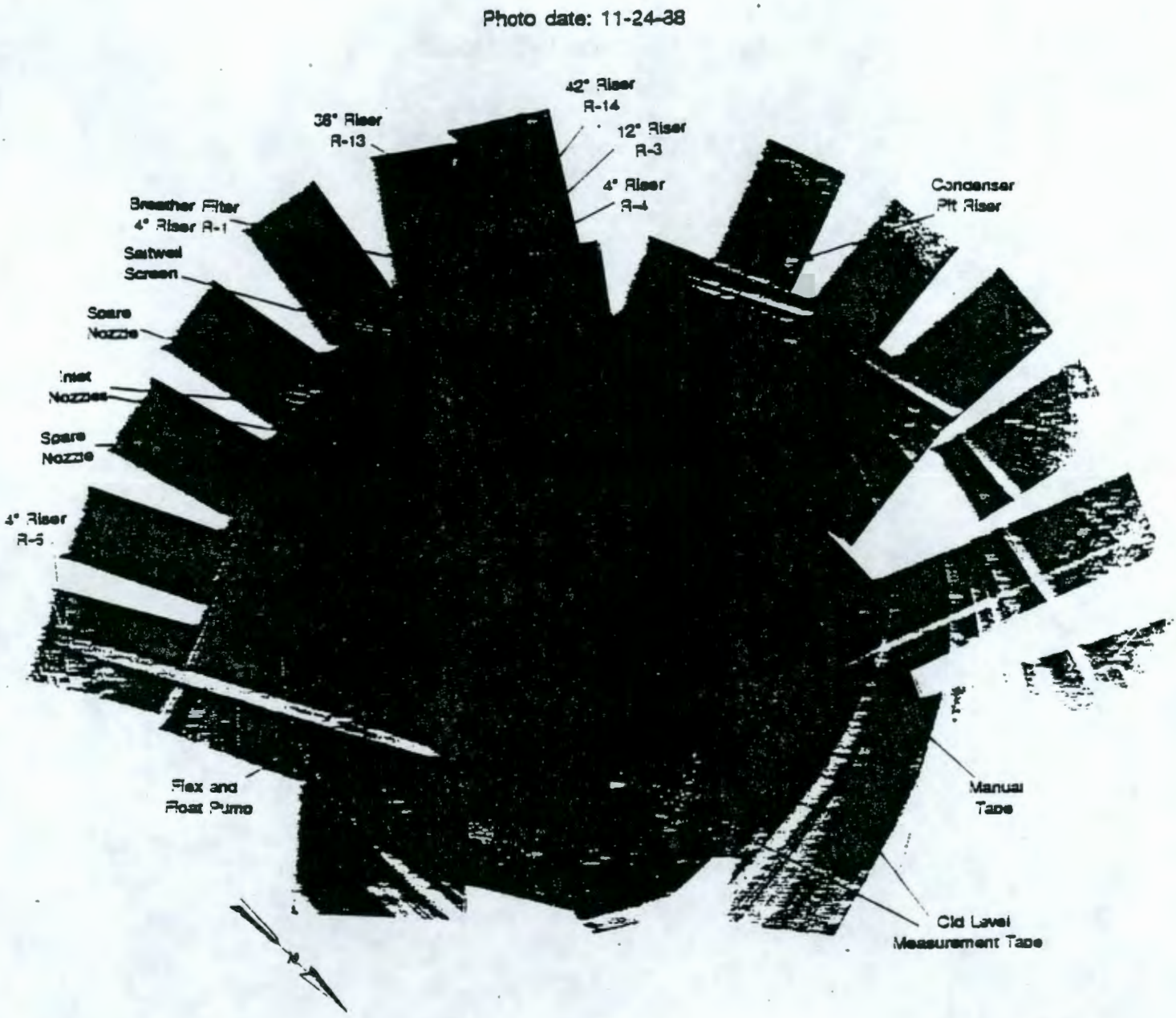


Figure 2-7. In-Tank Photo Montage of Tank 241-BX-101.



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3.0 TANK SAMPLING OVERVIEW

Because of the limited applicability of and information on earlier (1976) sampling and analysis events, this chapter focuses on the 1994 sampling event, the handling of the samples after the sampling event, and the analytical procedures performed on the samples.

3.1 DESCRIPTION OF 1994 SAMPLING EVENT

Tank 241-BX-101 was sampled on June 20 and 21, 1994, from two widely spaced risers, as specified in WHC-SD-WM-SP-004 (Babad and Redus 1994). Because of the shallow depth of the tank waste, auger sampling was selected as the most appropriate sampling method for tank 241-BX-101. The two auger samples were taken using a 10-in. auger. For a further description of auger sampling procedures, refer to WHC-SD-WM-TI-648, *Tank Characterization Reference Guide* (De Lorenzo et al. 1994). Table 3-1 lists tank farm sample numbers, transport cask numbers, and drill string dose rates for the two samples from tank 241-BX-101.

Table 3-1. Tank 241-BX-101 Sampling Information.

Sample number	Cask serial number	Riser number	Drill string dose rate mSv/h (mR/h)
94-AUG-004	C-1050	1	4.5 (450)
94-AUG-005	C-1052	7	2.0 (200)

3.1.1 Sample Handling

One auger sample was removed from each of two risers, 1 and 7, of tank 241-BX-101. Each 10-in. auger has eight flutes; flute 1 begins at the auger shaft while flute 8 is at the tip of the auger bit. Table 3-2 details the subsample locations and the laboratory numbering system for these samples.

The waste from riser 1 was present on all eight flutes of the auger. The waste from flute 1 was cream colored. The waste form flutes 2 and 4 was a darker brown color. The flute 3 sample was white to cream colored with the consistency of wet peanut butter. The sample combined from flutes 1, 2, and 4 had a similar consistency. However, the material from flutes 5 through 8 was damper but with a consistency similar to the upper flutes. Flutes 5 through 8 appeared dark brown to black on the outside but had some white to cream-colored material inside.

Table 3-2. Tank 241-BX-101 Sample Numbers and Locations.

Riser number	Lab jar number	Date extruded	Subsample location[a]
1	6044	6/23/94	R1/F3
	6045	6/23/94	R1/F1,2,4
	6046	6/23/94	R1/F5,6,7,8
7	6057	6/28/94	R7/F1
	6058	6/28/94	R7/F2,3,4,5
	6059	6/28/94	R7/F6,7,8
	Vial #6036	6/28/94	R7/rock[b]

[a] R and F denote the riser and flutes from which the sample was taken.
[b] Rock and ash found in extrusion tray.

The waste material from riser 7 was also present on all eight flutes. Flute 1 was damp and contained a white crusty material mixed with black material. Flutes 2 through 5 had a wet, smooth, peanut butter consistency. The material from flutes 6 through 8 was black and had the consistency of wet, smooth, peanut butter. A 30-g piece of rock-like material was also recovered from the extrusion tray (Kocher 1994). There was no drainable liquid present in either auger sample.

Some difficulty was encountered while extruding the sample from riser 7. A wire was stuck between the auger and the sleeve, making it necessary to use tools to extrude the sample. Some of the material fell to the tray during the process of extruding each auger. Additionally, some drying of each sample was noted.

This sample description is taken from WHC-SD-WM-DP-069, *Single-Shell Tank Waste Characterization for Tank 241-BX-101* (Kocher 1994). A total of 126.2 g of sample material was collected from riser 1 and 34.4 g from riser 7, although WHC-SD-WM-TP-210, *Tank 241-BX-101 Tank Characterization Plan* (Carpenter 1994), indicated that above 300 g of waste should be recovered. Sample material was present on all flutes of each auger. Table 3-3 describes the location, general appearance, and weight of the auger subsamples. Approximately 15 mL of liquid was observed in the liner of the riser 1 auger, about 10 mL of which were recovered. Nearly 5 g of liner liquid were observed for the riser 7 sample.

Table 3-3. Descriptive Summary of the 1994 Subsample.

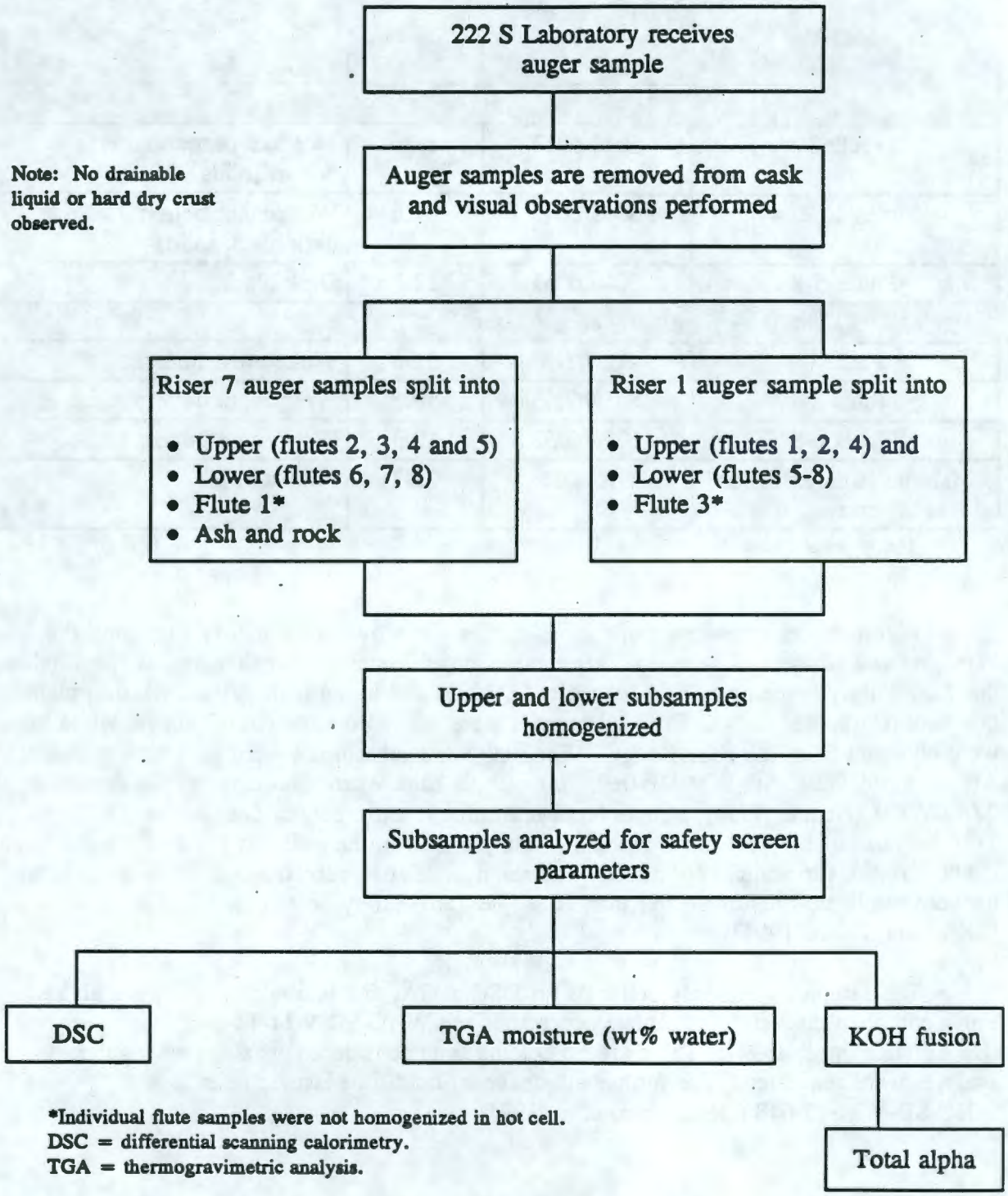
Subsample location	Sample jar or vial number	Net weight solids (g)	Sample description
Riser 1			
Flute 3	6044/6037	8.20	Wet like peanut butter; brown solids
Flutes 1, 2, 4	6045/6043	11.6	Wet peanut butter; dark black solids
Flutes 5-8	6046/6051	12.9	Dark black
Riser 7			
Flute 1	6057/NA	3.96	White crust material
Flutes 2-5	6058/6053	10.8	Wet peanut butter; black
Flutes 6-8	6059/6052	12.3	Wet peanut butter; black
Material from extrusion tray	NA/6036	2.97	Rock and ash

NA = not available.

Figure 3-1 is a flowchart of the basic steps taken by the laboratory regarding the extrusion and analysis of samples. After extrusion, subsamples were created as specified in the Tank Safety Screening DQO (Babad et al. 1995) and in the tank characterization plan for this tank (Carpenter 1994). Four subsamples were taken from the riser 7 auger, while three were obtained from the riser 1 auger. The individual subsamples were then homogenized. According to WHC-SD-WM-DP-069, *Single-Shell Tank Waste Characterization for Tank 241-BX-101* (Kocher 1994), sample homogenization is extremely critical for the DSC and TGA because of the very small amount of sample used in the analyses (10 to 50 mg). Even slight variations in sample consistency can result in relative percentage differences (RPDs) between duplicate runs of greater than 10% (the Tank Safety Screening DQO limit [Babad and Redus 1994]).

The samples were only prepared for DSC, TGA, and total alpha activity analyses. For a complete discussion of sample procedures see WHC-SD-WM-TI-648 (De Lorenzo et al. 1994). There are no holding time considerations since no regulatory analytes were requested. For further discussion of hold time issues, refer to WHC-SD-WM-TI-648 (De Lorenzo et al. 1994).

Figure 3-1. Sample Handling Flow Chart for Tank 241-BX-101.



*Individual flute samples were not homogenized in hot cell.
DSC = differential scanning calorimetry.
TGA = thermogravimetric analysis.

3.1.2 Sample Analyses

All analyses were performed at the 222-S Laboratory. The samples were received by the 222-S Laboratory on June 22, 1994. Laboratory personnel loaded the samples into the IE-2 hot cell, and the contents were mechanically extruded on June 23 and 28, 1994.

The objectives of the sample analyses are to provide the following: determination of weight percent water by TGA, exothermic activity by DSC, and total alpha content by alpha proportional counting. Procedures used for these analyses are listed in Table A-1 in Appendix A. Additional information on analytical methods can be obtained in WHC-SD-WM-TI-648 (De Lorenzo et al. 1994).

3.2 PREVIOUS SAMPLING EFFORTS

A sludge analysis for tank 241-BX-101 was conducted in March 1976. The analysis included chemical and physical property data and a heat generation rate based upon the $^{89/90}Sr$ and ^{137}Cs levels. Table B-1 in Appendix B presents the results of this analysis.

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4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

The samples collected on June 20 and 21, 1994, from tank 241-BX-101 are based upon the DQO process. The DQO that governs the data collection from these particular samples is WHC-SD-WM-SP-004 (Babad and Redus 1994). This DQO was developed to allow rapid classification of the tanks containing high-level waste and to support tank safety issues. The DQO specifies wt% water by TGA and DSC and total alpha activity be determined by laboratory analysis. The TGA, DSC, and total alpha results are presented in Table 4-1. Detailed quality control information concerning these three analyses is presented in Section 5.1.2.

Table 4-1. Analytical Presentation Tables.

Analysis	Tabulated result location
Total alpha	Table 4-2
Thermogravimetric analysis	Table 4-3
Differential scanning calorimetry	Table 4-4

4.2 DATA PRESENTATION

4.2.1 Total Alpha Activity

Total alpha analyses were performed on fusion digested samples, in accordance with procedure LA-508-101, *Alpha and Beta in Liquid Samples* (WHC 1994). Total alpha analyses were performed on all three samples from riser 1 and on two of the four samples from riser 7. The samples ranged from a high of 7.55×10^4 Bq/g (2.04 μCi/g) to a low of 2.25×10^4 Bq/g (0.608 μCi/g). The highest mean value was 5.29×10^4 Bq/g (1.43 μCi/g). All results were well within the DQO limit of 1.52×10^6 Bq/g (41 μCi/g).

Table 4-2 presents the results for total alpha. The table lists the sample numbers and sample points. If total alpha was not performed on a sample, it is not listed in the table. The results column is a simple mean of the original sample and its duplicate. No quality control information such as matrix spike recoveries, standard recoveries, or duplicate results, are listed. This information may be obtained in WHC-SD-WM-DP-069 (Kocher 1994). The mean in the last column is a simple mean of the listed results.

Table 4-2. Tank 241-BX-101 Total Alpha Activities.

Sample number[a]	Result Bq/g (μCi/g)	Mean Bq/g (μCi/g)
Riser 1		
R5738 R1/F3	2.41 E+04 (0.652)	2.93 E+04 (0.793)
R5739 R1/F1,2,4	2.28 E+04 (0.616)	
R5740 R1/F5,6,7,8	4.11 E+04 (1.11)	
Riser 7		
R5745 R7/F6,7,8	5.29 E+04 (1.43)	4.63 E+04 (1.25)
R5746 R7/F2,3,4,5	3.96 E+04 (1.07)	

Note: 1 Ci = 3.7 E+10 Bq.

[a] The first number is the laboratory tracking number. The second number is the customer ID number and identifies the riser and the auger flutes from which the sample came.

A tank inventory estimate was not calculated for total alpha because of the lack of a measured density value for the tank, which is needed to convert Bq/g to Bq (μCi/g to Ci). The data tables present the data collected from the raw laboratory data sheets found in WHC-SD-WM-DP-069 (Kocher 1994).

4.3 PHYSICAL MEASUREMENTS AND THERMODYNAMIC ANALYSES

Tank 241-BX-101 auger samples were analyzed according to WHC-SD-WM-SP-004 (Babad and Redus 1994). Therefore, thermodynamic analyses were performed on the auger samples; density, percent solids, particle size, and rheology were neither requested nor performed.

4.3.1 Thermodynamic Analyses

TGA and DSC were performed on all subsegments of both auger samples. All samples were homogenized prior to analysis. The results of the thermal analyses performed are summarized in Table 4-3. TGA results for four of the seven samples were below the DQO safety criterion of 17.0 wt% water. The first transition in each sample began at the lower temperature limit of the analysis (38 °C) and was complete at about 143 °C. In this region, the observed decreases in mass are mainly due to loss of bulk and interstitial water in the samples. The second transition began at 205 °C to 213 °C and was completed at about 493 °C. In this region, the covalently bound water and other reaction products are released.

The reaction of aluminum hydroxide plus heat, yielding aluminum oxide and water, is possible, since that reaction takes place above 300 °C and aluminum is present in the tank (see Appendix B). The percent weight loss listed in Table 4-3 for the second transition is with respect to the original weight of the sample.

Table 4-3. Tank 241-BX-101 Thermogravimetric Analysis.

Sample number[a]	Run	First transition		Second transition		Mean Total
		Range (38 °C - 143 °C)	Mean wt% loss	Range (205 °C- 493 °C)	Mean wt% loss	
Riser 1						
R5738 R1/F3	1	14.1	11.3	25.1	25.9	37.2
	2	8.50		26.6		
R5739 R1/F1,2,4	1	13.1	13.7	26.6	25.3	39.0
	2	14.2		24.0		
R5740 R1/F5,6,7,8	1	27.8	26.3	18.6	18.1	44.4
	2	24.8		17.5		
Riser 7						
R5749 R7/F1	1	16.4	16.1	22.9	23.2	39.3
	2	15.8		23.5		
R5746 R7/F2,3,4,5	1	18.4	21.1	19.8	18.9	40.0
	2	23.8		18.0		
R5745 R7/F6,7,8	1	15.4	18.7	19.0	16.1	34.8
	2	22.0		13.2		
R5750 R7/rock	1	2.13	1.5	29.0	29.6	31.1
	2	0.90		30.1		
		Average	15.52		22.42	37.95
		Standard deviation	7.60		4.71	4.11

[a] The first number is the laboratory tracking number. The second number is the customer ID number and identifies the riser and the auger flutes from which the sample came.

The DSC results are tabulated in Table 4-4. The range, peak temperature, and the change in enthalpy are given for one transition range (the exceptions being the first flute in riser 7 and the rock from riser 7 found in the extrusion tray in which the results from both transition ranges were integrated). No exothermic reactions were noted.

Table 4-4. Differential Scanning Calorimetry Energetic Results (Wet Weight) for Tank 241-BX-101.

Sample number[a]	Run	First transition			Second transition		
		Range °C	Peak °C	ΔH J/g[b]	Range °C	Peak °C	ΔH J/g
R5738 R1/F3	1	NI	NI	NI	208-335	280	380
	2	NI	NI	NI	200-320	282	396
R5739 R1/F1,2,4	1	NI	NI	NI	210-330	280	344
	2	NI	NI	NI	210-330	283	349
R5740 R1/5,6,7,8	1	NI	NI	NI	NI	NI	NI
	2	NI	NI	NI	NI	NI	NI
R5749 R7/F1	1	40-130	120	492	210-325	284	441
	2	50-130	94.7	274	190-318	282	538
R5746 R7/F2,3,4,5	1	NI	NI	NI	192-310	255	239
	2	NI	NI	NI	195-310	269	297
R5745 R7/F6,7,8	1	NI	NI	NI	200-320	271	198
	2	NI	NI	NI	170-310	265	187
R5750 R7/rock	1	90-120	101	32.0	210-340	300	552
	2	85-120	103	47.1	205-330	298	518

[a] The first number is the laboratory tracking number. The second number is the customer ID number and identifies the riser and the auger flute(s) from which the sample came.

[b] Negative sign denotes exothermic reaction.

NI = not integrated.

[c] Wet weight refers to the raw data seen by the DSC. Comparisons to the DQO are done on a dry weight basis.

5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

5.1.1 Field Observations

Several observations from the 1994 auger sampling event may impact the interpretation and use of the data. Since the amount of waste recovered from the riser 1 auger was only about 40%, and from the riser 7 auger only about 10%, of the expected quantity, the representativeness of the sample is uncertain.

The sampling plan (Carpenter 1994) indicated that about 20 cm (8 in.), or about 300 g, of waste was expected to be recovered. All of the auger flutes were expected to be full based upon the waste depth.

The small amount of waste recovered from riser 7 did not completely fill the voids around the flutes but was smeared out in relatively thin layers. The spreading out of small quantities of waste increases the potential for the waste to dry in the hot cell and during subsequent operations. This effect would bias the weight percent water values low; DSC results, which are corrected to a dry weight basis, would be biased high. It is not possible to determine whether this occurred or to assess the magnitude of this effect for tank 241-BX-101 data. However, the riser 7 average moisture result is slightly higher than the riser 1 results for which larger sample volumes had been obtained.

5.1.2 Quality Control Assessment of Analytical Data

An attempt is always made to quantify the different sources of error possible during the chemical analysis of a sample. When these errors are summarized, they give an indication of data reliability. If one or more of the error estimates are outside the acceptable limits, the accuracy of the concentration estimate is drawn into question. Possible sources of error are sample contamination, matrix interferences, analytical method error, and poor instrument calibration. Error estimates are determined from the analysis of standards, spike recoveries, blank contamination, and sample duplicate variation.

Standards are used to estimate the accuracy of the analytical method and are evaluated prior to and concurrent with sample analysis. Standards contain the analytes of interest at known concentrations. The criterion for standard recovery is 100 $\pm$ 10%. As can be seen in Table 5-1, all standard recoveries for percent water and total alpha were well within the limits. The three standard recoveries for DSC were also within limits (data not shown).

Matrix spikes are used to estimate the bias of the analytical method due to matrix interferences. Spike samples are prepared by splitting a sample into two aliquot portions and adding a known amount of a particular analyte to one portion to calculate a recovery

Table 5-1. Quality Control Summary for Tank 241-BX-101.

Riser	Analyte	Relative percent difference %[a]	Standard Recovery %[b]	Spike recovery %[c]
1	Percent water (flute 3)	50.4	99.4	NA
	Percent water (flutes 1,2,4)	7.83		
	Percent water (flutes 5-8)	11.3		
	Alpha (flute 3)	3.8	97.7	128.6
	Alpha (flutes 1,2,4)	2.4		120.4
	Alpha (flutes 5-8)	9.0		103.4
7	Percent water (ash & rock)	81.5	99.3	NA
	Percent water (flute 1)	3.80	--	--
	Percent water (flutes 2-5)	25.9	98.8	--
	Percent water (flutes 6-8)	35.5	--	--
	Alpha (flutes 2-5)	85.0	97.5	85.5
	Alpha (flutes 6-8)	13.8	--	90.5

[a] 10% limit.
[b] Range = 90-110%.
[c] Range = 80-120%.
NA = not available.

percentage. The quality control criterion for matrix spikes is 100 ± 20% recovery. Spikes were only conducted on total alpha activity, and two of three samples from riser 1 were slightly above the quality control limits. A rerun was requested that gave the same results (Kocher 1994). The total alpha results are so low compared to the criticality criteria that the high bias is not significant.

Method blanks document the contamination resulting from the analytical process and are prepared by filling sample containers with deionized, distilled water. They are carried through the complete sample preparation and analytical procedure and all reagents used in the sample processing are added in the same volumes. Blanks were conducted only on the total alpha data and contamination was not a problem.

Duplicate analyses of samples provides an indication of laboratory precision and the homogeneity of the samples. To estimate this error, an RPD is calculated for each duplicate pair. The RPD is a measure of variability and is defined as the absolute value of one duplicate minus the other, divided by the mean. The tank characterization plan for tank 241-BX-101 set the duplicate precision acceptance criterion at no RPD being larger than 10% (Carpenter 1994). Two of the subsample means from riser 7 exceeded the criterion for total

alpha (Table 4-2). Rerun of riser 7 sample and duplicate samples gave similar results indicating that the source of the difference was in sample preparation and probably caused by sample heterogeneity. The homogenization process in sample preparation is extremely critical for the DSC and TGA analyses because of the very small amount of sample analyzed (10 to 50 mg). Even slight variations in sample consistency can result in RPDs greater than 10%. The total alpha results are so low that these differences are not significant for evaluating criticality. Because of ALARA (as low as reasonably achievable) concerns and the probability that repeating the analyses would not improve the data, no reruns were requested (Kocher 1994).

5.2 TANK WASTE INVENTORY AND PROFILE

The waste in tank 241-BX-101 is composed of 97.7% sludge and 2.3% supernatant by volume (Hanlon 1995). According to the hot cell chemist's observations, the waste appeared to be heterogeneous because of the variations in color (Kocher 1994). However, distinct layers were not determined.

A statistical test known as the analysis of variance (ANOVA) and a similar but simplified version known as the t-test were conducted on the percent water and total alpha data to determine whether there were any differences between the two risers or between the vertically oriented subsamples taken from the auger. The ANOVA generates a statistic called a p-value that is compared with a standard significance level ($\alpha = 0.05$). If a p-value is below 0.05, there is sufficient evidence to conclude that the subsample means are significantly different from each other. However, if a p-value is above 0.05, there is not sufficient evidence to conclude that the subsamples are significantly different from each other.

For riser 1, the subsample from flute 3 was combined with the subsample from flutes 1, 2, and 4 in a weighted manner such that each of the 4 flutes was equally represented. This subsample was compared to the subsample from flutes 5 through 8 to determine whether any layering was apparent (flutes 1-4 = top, flutes 5-8 = bottom). For riser 7, the flute 1 subsample was combined with the subsample from flutes 2 through 5 in the same manner as riser 1, and then compared with the subsample from flutes 6 through 8 (flutes 1-5 = top, flutes 6-8 = bottom).

The results of the statistical tests are given in Table 5-2. The percent water indicated that at the 0.05 level, there was a significant difference between the top and bottom subsamples from riser 1 (p-value = 0.006), but not for those from riser 7 (p-value = 0.395). There was no significant difference between the two risers (p-value = 0.845). For total alpha, the ANOVA results again indicated a significant difference between the top and bottom for riser 1 (p-value = 0.005) but not for riser 7 (p-value = 0.306), as well as no significant difference between the two risers (p-value = 0.282). Thus, based upon the very small number of samples available, the distribution of percent water and total alpha in the

Table 5-2. Results of Statistical Tests.

Riser	Subsample	p-value percent water (top/bottom)	p-value percent water (riser/riser)	p-value total alpha (top/bottom)	p-value total alpha (riser/riser)
1	top	0.006	0.845	0.005	0.282
	bottom				
7	top	0.395		0.306	
	bottom				

tank appears to be fairly homogeneous horizontally. Vertically, mixed results were obtained, with riser 1 showing a higher value for percent water and total alpha as a function of depth, and riser 7 showing no significant vertical differences.

5.3 COMPARISON OF ANALYTICAL AND TRANSFER HISTORY

Because of the few analytical requirements for safety screening characterization, the comparison of the analytical results from the 1994 sampling event with the historical information is limited. Historical estimates of analyte concentrations, developed through the tank layer model (Agnew et al. 1994) and listed in Brevick et al. (1994) were available for comparison with the latest analytical results. The historical plutonium value has been considered the historical total alpha value and was compared to the total alpha lab result given in Table 4-2. The RPD for the historical and analytical results is 55.0%. These differences are considered reasonable considering the uncertainties in the historical model and the sampling and analysis results. The historical estimated quantities for the citrate and glycolate would not be expected to produce a significant exotherm and none was observed.

5.4 EVALUATION OF PROGRAM REQUIREMENTS

5.4.1 Safety Evaluation

This section provides selected results obtained from auger sampling for some of the most pertinent analytes needed by the Waste Tank Safety Program. These data needs are defined in WHC-SD-WM-SP-004 (Babad and Redus 1994) and are used to determine that the 177 Hanford Site underground waste tanks have been appropriately categorized as to whether or not safety issues exist for each tank. The DQO establishes decision criteria for concentrations of analytes of concern. The decision criteria are used to determine whether a tank is safe or whether further investigation into the tank's safety is warranted. Table 5-3 tabulates the decision criteria required by the safety screening DQO and compares them to the recent analytical results.

Table 5-3. Safety Screening Data Quality Objective Decision Variables and Criteria. (2 sheets)

Safety issue	Primary decision variable	Decision criteria threshold	Auger location	Average analytical value	Relative percent difference (%)
Riser 1					
Ferrocyanide/ organic	Total fuel content	115 cal/g	All flutes	No exotherms observed	
Ferrocyanide	Moisture content	[a]If fuel is above 8 wt%, then wt% H_2O < [(0.0932*DSC exotherm) - 10.7][b]	All flutes	Ferrocyanide not applicable.	
Organic	Percent moisture	17 wt%	Flute 3	11.3 wt%	50.4%
			Flutes 1,2,4	13.7 wt%	7.8%
			Flutes 5-8	26.3 wt%	11.3%
Criticality	Total alpha	1.52 E+06 Bq/g (41 μCi/g) (1 g/L)[c]	Flute 3	2.41 E+04 Bq/g (0.652 μCi/g)	3.8%
			Flutes 1,2,4	2.28 E+04 Bq/g (0.616 μCi/g)	2.4%
			Flutes 5-8	4.11 E+04 Bq/g (1.11 μCi/g)	9.0%
Flammable gas	Flammable gas	No data available	–	–	
Riser 7					
Ferrocyanide/ organic	Total fuel content	115 cal/g	All flutes	N/A (No exotherms observed)	
Ferrocyanide	Moisture content	[a]If fuel is above 8 wt%, then wt% H_2O < [(0.0932*DSC exotherm) - 10.7][b]	All flutes	Ferrocyanide not applicable	
Organic	Percent moisture	17 wt%	Flute 1	16.1 wt%	3.8%
			Flutes 2-5	21.1 wt%	25.9%
			Flutes 6-8	18.7 wt%	35.5%
			ash/ rock sample	1.51 wt%	81.5%
Criticality	Total alpha	1.52 E+06 Bq/g (41 μCi/g) (1 g/L)[c]	Flute 1	N/A	N/A
			Flutes 2-5	5.29 E+04 Bq/g (1.43 μCi/g)	85.0%
			Flutes 6-8	3.96 E+04 Bq/g (1.07 μCi/g)	13.8%
			ash/ rock sample	N/A	N/A

Table 5-3. Safety Screening Data Quality Objective Decision Variables and Criteria. (2 sheets)

Safety issue	Primary decision variable	Decision criteria threshold	Auger location	Average analytical value	Relative percent difference (%)
Flammable gas	Flammable gas	No data available	–	–	

[a] For explanation of the moisture decision rule, See Figure 4-1 in J. E. Meacham, R. J. Cash, G. T. Dukelow, H. Babad, J. W. Buck, C. M. Anderson, B. A. Pulsipher, J. J. Toth, and P. J. Turner, *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objective Process*, WHC-SD-WM-DQO-007, Westinghouse Hanford Company, Richland, Washington (1994).

[b] Fuel content is weight percent disodium nickel ferrocyanide, $Na_2NiFe(CN)_6$. The fuel percentage was calculated according to the following equation. The differential scanning calorimetry value is divided by 1430, which is the experimentally determined heat of reaction of $Na_2NiFe(CN)_6$ with nitrate in waste simulant according to A. K. Postma, J. E. Meacham, G. S. Barney, G. L. Borsheim, R. J. Cash, M. D. Crippen, D. R. Dickenson, J. M. Grigsby, D. W. Jeppson, M. Kummerer, J. M. McLaren, C. S. Simmons, and B. C. Simpson, *Ferrocyanide Safety Program: Safety Criteria for Ferrocyanide Watch List Tanks*, WHC-EP-0691, Westinghouse Hanford Company, Richland, Washington (1994).

$$\text{wt\% } Na_2NiFe(CN)_6 = \frac{[\text{DSC exotherm (cal/g dry weight)}]}{1430 \text{ cal/g}} \times 100.$$

Using this equation, the decision criterion of 4/3(wt% fuel - 8 wt%) listed in the DQO becomes:

$$\text{wt\% } H_2O < [(0.0932 \times \text{DSC exotherm}) - 10.7]$$

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

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

The waste fuel energy value is determined using a DSC. No exothermic activity was observed for the seven subsamples analyzed. There were no analyses for fuel sources such as total organic carbon and cyanide. Historical estimates indicate that relatively low (3,000 μg/g) concentrations of organics may be present.

The average percent water for all of the subsamples, except the rock identified in riser 7, was 17.8 wt% $\pm$ 5.6 wt% standard deviation. The moisture content of the waste on the lower flutes appear to be above the 17 wt% water safety screening criterion. However, moisture content of wastes on the upper flutes from riser 1 was less than the 17 wt% criterion. The reproducibility of three of the six subsamples (excluding rock and ash) significantly exceeded the desired 10% precision for the DQO. The average percent water result (17.8 wt%) is too near the safety screening criteria (17 wt%) and the variance too

large to confidently conclude that the moisture level in the tank is above 17 wt%. Because the average result is so near the limit, it is unlikely that the variance could be adequately improved through reanalysis. Some drainable or liner liquids were recovered for each auger. The 15 mL of liquid recovered for the riser 1 auger would represent about 12 wt% of the sample recovered assuming a density of 1.0 g/mL. The 5 g of liner liquid in riser 7 represents about 14.5 wt% of the total sample. If these liquid quantities are included with the TGA percent water results, the moisture content would be estimated to be considerably higher.

The heat generation rate based on historical radiochemical estimates is 4.04 kW (2.38×10^4 Btu/h) (decayed to 1995). This is still a factor of two below the high heat classification limit of 11.7 kW (4.0×10^4 Btu/h). In addition, the tank temperature history does not show any signs of excessive heat generation.

The potential for criticality is assessed using either total alpha or plutonium analyses. The safety screening criterion is 1 g/L of plutonium, which is equivalent to 41 μCi/g, assuming the waste has a density of 1.5 g/mL. The highest total alpha result (1.4 μCi/g) for any subsegment was well below the limit of concern. In addition, the total alpha also contains activity from ^{241}Am. Historical $^{239/240}$Pu estimates (0.58 μCi/g) also indicate that the plutonium should be well below this criterion. Even though some of the total alpha spike and duplicate results exceeded the desired criterion, their magnitude was not large enough to affect the criticality assessments.

The flammability of the gas in the headspace of the tank is another safety screening consideration. Analysis of the tank's headspace is presently outside the scope of this report.

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The sludge in tank 241-BX-101 was sampled and analyzed in 1976 and in 1994. The 1976 results may no longer be accurate because of subsequent salt well pumping and drying effects. Because the 1994 sampling event focused only on safety screening criteria, the chemical and radiochemical compositions of the waste must be estimated from historical information (Brevick et al. 1994). The tank is excepted to be mostly sludge with high concentrations of sodium, iron, and uranium phosphate and nitrate. The analysis of 1994 auger samples indicated that the plutonium concentration was well below criticality levels. No exotherms were observed for the DSC analyses. The average percent moisture results for the tank were very near the safety screening criterion of 17 wt%.

The 1994 sampling and analysis event for tank 241-BX-101 did not meet all of the requirements for the safety screening DQO (Babad and Redus 1994). Subsamples from the lower and upper areas of the auger samples from the two risers were analyzed, however waste recovery for the auger samples was lower than expected. Limited samples and data from different risers and waste depths make it difficult to determine accurately the variability of the waste composition in the tank. Variability for some of the TGA percent water and total alpha results were greater than desired, but did not have a significant impact on the assessment of the data. Historical information on the heat generation and tank temperature records does not indicate that excessive heat is being generated. Data and historical information do not indicate that tank 241-BX-101 has any immediate safety problems. The 1994 sampling and analysis event did not include an evaluation of the composition of the tank's headspace gases.

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APPENDIX A

ANALYTICAL METHODS

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APPENDIX A
ANALYTICAL METHODS

Table A-1 contains the procedure number for each of the analytical methods performed on the tank 241-BX-101 waste. All analyses were performed at the 222-S Laboratory.

Table A-1. Analytical Methods.

Analyte	Digestion	Procedure number
Thermogravimetric analysis	NA	LA-560-112[a]
Differential scanning calorimetry	NA	LA-514-113[b]
Total alpha activity	Fusion	LA-508-101[c]

[a] WHC, 1991, *Determination of Weight Loss as Percent Water by Thermogravimetric Analysis (TGA)-Mettler TG 50*, LA-560-112, Rev. A-1, Westinghouse Hanford Company, Richland, Washington.

[b] WHC, 1993, *Differential Scanning Calorimetry (DSC)*, LA-514-113, Rev. B-0, Westinghouse Hanford Company, Richland, Washington.

[c] WHC,1994, *Alpha and Beta in Liquid Samples*, Rev. D-1, Westinghouse Hanford Company, Richland, Washington.

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APPENDIX B

PREVIOUS SAMPLING RESULTS

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APPENDIX B
PREVIOUS SAMPLING RESULTS

Table B-1. Previous Sampling Analysis Results.

Analysis of sludge sample from tank 241-BX-101 March 8, 1976 (Received January 26, 1976)	
Physical data	
Visible: solids, dark brown in color	
Bulk density	1.68 g/mL
Particle density	1.98 g/mL
Percent water	29.1
Chemical analysis	
Analyte	μg/g
Al	1.61 E+05
Fe	1.13 E+04
Mg	1.16 E+03
Mn	6.54 E+03
Ca	2.38 E+03
Ba	817
Si	2.68 E+04
NO_3^-	1.48 E+05
PO_4^{3-}	9.50 E+03
Radiological analysis	
Radionuclide	Result
Pu	0.863 μg/g
$^{89/90}Sr$	3.96 E+13 Bq/g (1,070 μCi/g)
^{137}Cs	2.64 E+13 Bq/g (714 μCi/g)

Note: 1 Ci = 3.7 E+10 Bq.

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