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Tank Characterization Report for Double-Shell Tank 241-AN-102

Jaiduk Jo

Westinghouse Hanford Company, Richland, WA 99352
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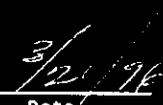
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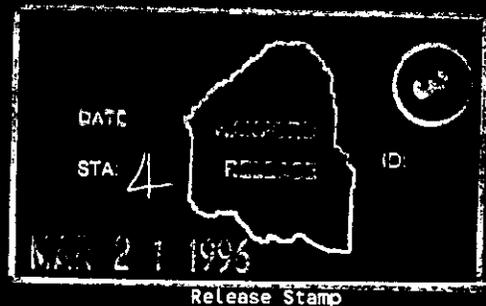
Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-AN-102. This report supports the requirements of the Tri-Party Agreement Milestone M-44-09.

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Tank Characterization Report for Double-Shell Tank 241-AN-102

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EXECUTIVE SUMMARY

This tank characterization report summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in double-shell underground storage tank 241-AN-102. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* Milestone M-44-09 (Ecology et al. 1994).

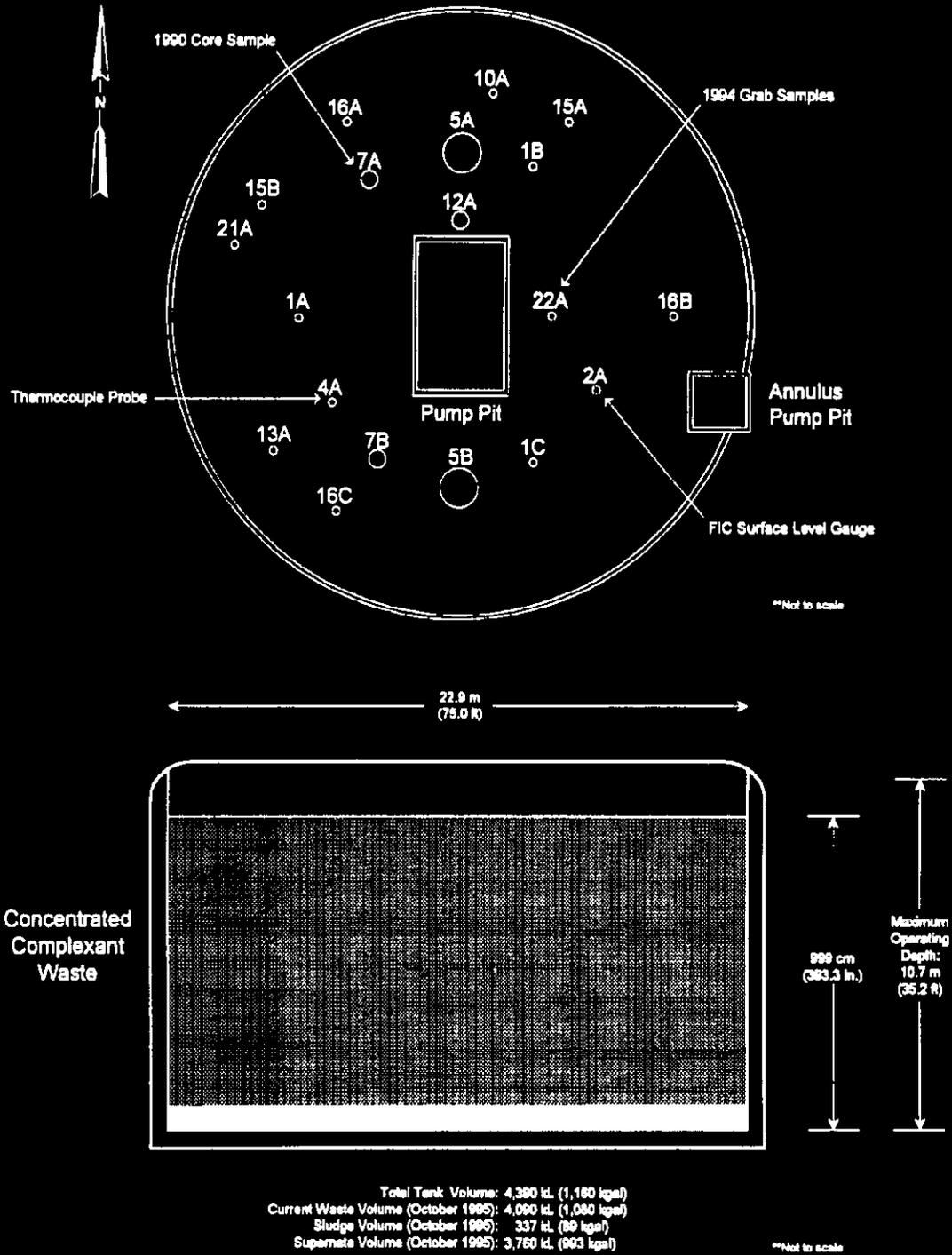
Tank 241-AN-102 is one of seven double-shell tanks located in the AN Tank Farm in the Hanford Site's 200 East Area. It went into service in 1981 with a waste transfer from tank 241-SY-102. The tank was nearly emptied in July 1984, leaving only 129 kL (34 kgal) of waste. During the fourth quarter of 1984, the tank was filled with concentrated complexant waste from tank 241-AW-101. Between 1984 and 1992, tank 241-AN-102 received only minor amounts of Plutonium-Uranium Extraction Plant (PUREX) miscellaneous waste and water, and has received no waste since 1992. Therefore, the waste currently in the tank is considered to be concentrated complexant waste. Tank 241-AN-102 is sound and is not included on any Watch List.

A description and status of tank 241-AN-102 are summarized in Table ES-1 and Figure ES-1. The tank, which has a capacity of 4,390 kL (1,160 kgal), presently contains 4,090 kL (1,080 kgal) of waste, 337 kL (89 kgal) existing as sludge and 3,760 kL (993 kgal) as supernate (Hanlon 1996).

Table ES-1. Description and Status of Tank 241-AN-102.

TANK DESCRIPTION	
Type	Double-shell
Constructed	1980-1981
In-service	1981
Diameter	22.9 m (75.0 ft)
Maximum operating depth	10.7 m (35.2 ft)
Capacity	4,390 kL (1,160 kgal)
Bottom shape	Flat
Ventilation	Operating exhauster
TANK STATUS (as of June 1995)	
Waste classification	Complexant concentrate
Total waste volume	4,090 kL (1,080 kgal)
Sludge volume	337 kL (89 kgal)
Drainable interstitial liquid	11 kL (3 kgal)
Supernate volume	3,760 kL (993 kgal)
Waste surface level (January 17, 1996)	999 cm (393.3 in.)
Temperature (1983 - 1995)	18.3 °C (65 °F) to 40 °C (104 °F)
Integrity	Sound
SAMPLING DATES	
Grab sampled	October 1994
Core sampled	June 1990
SERVICE STATUS	
In service	

Figure ES-1. Description and Status of Tank 241-AN-102.



This report summarizes two sampling and analysis events: (1) a three-segment core sample of the solids in 1990; and (2) supernate grab samples in 1994. Sludge composition and properties are based on the 1990 core sample taken to support waste vitrification, grout, pretreatment, and retrieval activities. Because the core sample was obtained before the existence of data quality objectives (DQOs), the sampling and analysis may not comply with the safety screening DQO (Dukelow et al. 1995) requirements. Supernate composition is based on the 1994 grab samples. Because the samples were taken for process control purposes, no DQOs were applicable to the sampling event. An assessment was still made using the results from this sampling event and the decision criteria of the waste compatibility DQO.

For informational purposes, the analytical results from the 1990 and 1994 samplings were compared with the decision limits of the safety screening DQO. Differential scanning calorimetry (DSC) was performed on centrifuged fractions of a composite from the 1990 sludge sampling event. Exothermic behavior exceeding the -480 J/g (dry weight) DQO limit was observed in the centrifuged solids and in a "crust" sample that was found floating on top of the centrifuged liquid. The dry weight DSC values were -757 J/g for the centrifuged solids and -933 J/g for the crust material. Water contents of both these samples were at least 25 weight percent, as determined by thermogravimetric analysis (TGA). Exothermic behavior is not surprising due to the complexant concentrate waste stored in the tank. Complexant concentrate contains high levels of organic carbon, nitrate, and nitrite. No DSC analyses were performed on the supernate.

Because of the high levels of organic carbon, a comparison was made between the total organic carbon (TOC) results and the TOC limit presented in the organic DQO (Turner et al. 1995). The DQO has established a 30,000- $\mu\text{g C/g}$ limit. On a dry weight basis, the sludge was found to contain 27,300 $\mu\text{g C/g}$ of TOC. The measured supernate TOC concentration of 37,200 $\mu\text{g C/g}$ (dry weight) exceeded the organic DQO limit.

The heat generated by the radioactivity in the tank waste is estimated to be 9,890 watts (33,800 Btu/hr), which is within the operating specification limit of 20,500 watts (70,000 Btu/hr) (Harris 1994). Average waste temperatures have remained consistently around 33 °C (91 °F) since 1985. Consequently, the heat load and thermal history do not indicate that the waste can radiologically generate temperatures high enough to initiate an exothermic reaction.

Total alpha activity results from both sampling events were well below the safety screening limits. A comparison with the final safety screening analytical requirement, the flammable gas concentration, was not possible because sampling of the tank headspace has not occurred.

Based on the analytical findings, a recommendation is suggested. A DSC analysis should be run on a sample of the supernate to measure the energetics and determine the contribution of the TOC to any exothermic reactions.

The concentration and tank inventories for the major constituents and analytes of concern in the tank waste are summarized in Table ES-2. Because the waste is concentrated complexant, it can be expected to contain high concentrations of aluminum, sodium, nitrate, nitrite, phosphate, sulfate, hydroxide, ^{137}Cs , and both organic and inorganic carbon. However, the analytical results did not support the expected results for all of the mentioned analytes. The analytical results for sodium, nitrate, nitrite, ^{90}Sr , and total organic carbon were higher than anticipated. For hydroxide, the measured value was much less than the expected value.

Table ES-2. Concentrations and Inventories for Major Analytes and Analytes of Concern in Tank 241-AN-102.

Physical Properties	Sludge Results ¹		Supernate Results ¹	
Density	1.5 g/mL		1.40 g/mL	
Percent unbound water (weight)	40.3		50.0	
Heat load	9,890 watts (33,800 Btu/hr)			
Chemical Constituents	Sludge Concentration ¹	Sludge Inventory ¹	Supernate Concentration ¹	Supernate Inventory ¹
Metals	µg/g	kg	µg/mL	kg
Al (Aluminum)	12,200	6,170	15,000	56,000
K (Potassium)	< 1,740	< 888	3,900	15,000
Na (Sodium)	2.34E+05	1.18E+05	2.57E+05	9.66E+05
Anions	µg/g	kg	µg/mL	kg
Cl ⁻ (Chloride)	2,060	1,040	3,900	15,000
F ⁻ (Fluoride)	< 890	< 450	2,000	7,500
OH ⁻ (Hydroxide)	---	---	4,100	15,000
NO ₃ ⁻ (Nitrate)	1.12E+05	56,700	2.21E+05	8.31E+05
NO ₂ ⁻ (Nitrite)	39,300	19,900	83,400	3.14E+05
PO ₄ ³⁻ (Phosphate)	3,030	1,530	4,900	18,000
SO ₄ ²⁻ (Sulfate)	25,900	13,100	14,000	53,000
Total Carbon	µg C/g	kg C	µg C/mL	kg C
Total Inorganic Carbon	12,300	6,220	13,500	50,800
Total Organic Carbon	16,300	8,250	26,100	98,100
Radionuclides	µCi/g	Ci	µCi/mL	Ci
¹³⁷ Cs	285	1.44E+05	382	1.44E+06
⁹⁰ Sr	169	85,500	73.7	2.77E+05

Note:

¹All analytical results are reported on a wet weight basis.

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LIST OF TERMS

ANOVA	analysis of variance
Btu/hr	British thermal units per hour
C	Celsius
Ci	curies
Ci/L	curies per liter
cP	centipoise
DQO	data quality objective
DSC	differential scanning calorimetry
EDTA	ethylenediaminetetraacetic acid
F	Fahrenheit
ft	feet
g	grams
g/L	grams per liter
g/mL	grams per milliliter
HDW	Hanford defined wastes
HEDTA	N-(hydroxyethyl)-ethylenediaminetriacetic acid
HTCE	historical tank content estimate
IC	ion chromatography
ICP	inductively coupled plasma
in.	inches
J/g	joules per gram
kg	kilograms
kg C	kilograms of carbon
kgal	kilogallons
kL	kiloliters
L	liters
M	molarity (moles per liter)
m	meters
mg	milligrams
mL	milliliters
mm	millimeters
mmol	millimole
mol/L	moles per liter
Pa	Pascals
ppm	parts per million
psi	pounds per square inch
PUREX	Plutonium-Uranium Extraction Plant
RPD	relative percent difference
RSD	relative standard deviation

LIST OF TERMS (Continued)

TGA	thermogravimetric analysis
TIC	total inorganic carbon
TLM	tank layer model
TOC	total organic carbon
WSTRS	Waste Status and Transaction Record Summary
$\mu\text{Ci/g}$	microcuries per gram
$\mu\text{Ci/L}$	microcuries per liter
$\mu\text{Ci/mL}$	microcuries per milliliter
$\mu\text{eq/g}$	microequivalents per gram
$\mu\text{g/g}$	micrograms per gram
$\mu\text{g/mL}$	micrograms per milliliter
$\mu\text{g C/g}$	micrograms of carbon per gram
$\mu\text{g C/mL}$	micrograms of carbon per milliliter
μm	micrometers
ΔH	change in enthalpy

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1.0 INTRODUCTION

This report presents an overview of double-shell tank 241-AN-102 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. This characterization report describes the results of two sampling events: a core sample of the solids taken in 1990, and a grab sampling of the supernate in 1994 (Herting 1994). Although the tank is in service, it has not received waste since 1992. However, it may become active in the future, which may or may not change the composition of the waste.

Tank 241-AN-102 contains concentrated complexant waste, which must be segregated from non-complexant wastes (Fowler 1995). Thus, although the volume may change, it is unlikely that the composition will change substantially. The concentration and inventory estimates reported in this document may not reflect the exact composition of the waste but do represent the best estimates based on the most recent analytical data. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994) Milestone M-44-09.

1.1 PURPOSE

The purpose of this report is to summarize information about the use and contents of tank 241-AN-102. When possible, this information will be used to assess issues associated with safety, operational, environmental, and process development activities. This report also provides a reference point for more detailed information about tank 241-AN-102.

1.2 SCOPE

The core sample acquired in 1990 was taken to support waste vitrification, grout, pretreatment, and retrieval activities (DiLiberto 1990). Extensive characterization work was performed, including analysis of chemical, radiochemical, physical, and rheological properties (Douglas 1996). Other than total organic carbon, no specific organic analyses were performed.

The grab samples obtained in 1994 were taken for process control purposes. The immediate objective of the sampling was to determine whether the free hydroxide concentration in the waste was within tank corrosion control specifications (Herting 1994). Chemical components and a few radionuclides were measured for the supernate. In addition, limited testing of physical properties was done. No organic analyses were performed, with the exception of total organic carbon (TOC). Because this sampling was done for process control reasons, no tank characterization plan was written, and no data quality objectives (DQOs) were applicable.

This report does not include any information on headspace sampling and analysis to determine the composition of the tank headspace gases.

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-AN-102 based on historical information. The first part of the section details the current condition of the tank, followed by discussions of the tank's background, transfer history, and process sources that contributed to the tank waste, including an estimate of the current contents based on the process history. Events that may be related to tank safety issues are included. The final part of the section details surveillance data available for the tank. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the waste layers. Temperature data are provided to evaluate the heat-generating characteristics of the waste.

2.1 TANK STATUS

As of October 31, 1995, tank 241-AN-102 contained 4,090 kL (1,080 kgal) of waste classified as complexant concentrate (Hanlon 1996). The amounts of the various phases comprising this waste are shown in Table 2-1.

Table 2-1. Status Summary of Tank Contents.

Waste Form	Kiloliters (kilogallons)
Total waste	4,090 (1,080)
Supernate	3,760 (993)
Sludge	337 (89)
Drainable interstitial liquid	11 (3)
Drainable liquid remaining	3,770 (996)
Pumpable liquid remaining	3,760 (993)
Saltcake	0 (0)

Tank 241-AN-102 is in service at the present time; however, there have been no waste transfers to or from the tank since 1992. The tank's integrity is classified as sound. It is not on any Watch List. All monitoring systems were in compliance with documented standards as of October 31, 1995 (Hanlon 1996).

2.2 TANK DESIGN AND BACKGROUND

The AN Tank Farm, built between 1980 and 1981, incorporated the newest-generation double-shell tank farm design. This tank farm consists of seven, type 100 series, 4,390-kL (1,160-kgal) tanks. These tanks were designed for waste with a maximum fluid temperature of 177 °C (350 °F). Tank 241-AN-102 has 61 risers ranging in size from 10 cm (4 in.) to

1.07 m (42 in.) in diameter that provide surface level access to the underground tank. This tank has four risers available for use: three 10-cm (4-in.)-diameter risers (numbers 10A, 15A, and 21A), and one 30-cm (12-in.)-diameter riser (number 7A). If used as sampling ports, the risers would give access to a wide area of the northern half of the tank.

This double-shell tank is constructed of 46-cm (1.5-ft)-thick concrete walls and a 38-cm (1.25-ft) thick concrete dome. The mild carbon steel liner on the bottom is 1.3 cm (0.5 in.) thick, while the lower portion of the sides are 1.9 cm (0.75 in.) thick. The upper portion of the sides are 1.3 cm (0.5 in.) thick and the dome liner is 0.95-cm (0.375-in.)-thick steel. The inner liner has been heat treated and stress relieved. The outer liner is made of 0.95-cm (0.375-in.) mild carbon steel. The outer liner has not been heat treated. This tank has a flat bottom and a liner height with a maximum operating depth of 10.7 m (35.2 ft). In addition, the tank has a grid of drains slots beneath the steel-liner bottom. The grid is designed to collect any tank leakage and divert it to a leak detection well. The tank is set on an insulated, reinforced concrete foundation. Various coatings and sealants were used to prevent leaks or intrusions.

The tank waste level is monitored with a Food Instrument Corporation gauge. A list of tank 241-AN-102 risers, showing the size and general use, is provided in Table 2-2. A plan view that depicts the riser configuration and locations is shown in Figure 2-1. This constitutes all installed equipment for tank 241-AN-102. A tank cross-section showing the approximate waste level along with a schematic of the tank equipment is found in Figure 2-2.

2.3 PROCESS KNOWLEDGE

These sections present the transfer history of tank 241-AN-102 and an estimate of the composition of the tank waste based on its process history.

2.3.1 Waste Transfer History

Waste was initially added to tank 241-AN-102 in September 1981 with the transfer of dilute evaporator feed from tank 241-SY-102 (Agnew et al. 1995b). The transfers of waste from tank 241-SY-102 continued until the third quarter of 1982. Water was added to tank 241-AN-102 intermittently from the second quarter of 1982 until the third quarter of 1983.

Table 2-2. Tank 241-AN-102 Risers. (2 sheets)

Number	Diameter (inches)	Description and Comments
1A	4	Sludge measurement port
1B	4	Sludge measurement port
1C	4	Sludge measurement port P/CP (12-in. CVR)
2A	4	Liquid level gauge
3A	12	Supernate pump (central pump pit)
4A	4	Thermocouple probe
5A	42	No surface access
5B	42	No surface access
6A	24	Annulus access, spare
6B	24	Annulus access, spare
7A	12	Spare
7B	12	Tank ventilation
8A	4	Annulus air inlet
8B	4	Annulus air inlet
8C	4	Annulus air inlet
8D	4	Annulus air inlet
8E	4	Annulus air inlet
8F	4	Annulus air inlet
8G	4	Annulus air inlet
8H	4	Annulus air inlet
9A	8	Annulus air outlet
9B	8	Annulus air outlet
9C	8	Annulus air outlet
9D	8	Annulus air outlet
10A	4	Spare
11A	42	Slurry distributor (central pump pit)
12A	12	Observation port, spare
13A	4	Tank pressure
14A	4	Dropleg nozzle (central pump pit)
15A	4	Vessel vent drain
15B	4	High liquid level sensor
16A	4	Sludge measurement port

Table 2-2. Tank 241-AN-102 Risers. (2 sheets)

Number	Diameter (inches)	Description and Comments
16B	4	Sludge measurement port
16C	4	Sludge measurement port, P/CP (12-in. CVR)
17A	4	Annulus inspection port
17B	4	Annulus inspection port
17C	4	Annulus inspection port
17D	4	Annulus inspection port
17E	4	Annulus inspection port
17F	4	Annulus inspection port
17G	4	Annulus inspection port
17H	4	Annulus inspection port, P/CP (12-in. CVR)
17J	4	Annulus inspection port
17K	4	Annulus inspection port, P/CP (12-in. CVR)
17L	4	Annulus inspection port
18A	12	Annulus access
18B	12	Annulus access
19A	4	TBX 102-4, no identification
19B	4	TBX 102-5, no identification
19C	4	TBX 102-6
19D	4	TBX 102-1
19E	4	TBX 102-2
19F	4	TBX 102-3
20A	12	Annulus pump (annulus pump pit)
21A	4	Spare
22A	4	Sludge measurement port
23A	4	Leak detector 102-4
23B	4	Leak detector 102-2
23C	4	Leak detector 102-3

Notes:

CVR = metal cover plate
P/CP = riser is recessed below a cement pad with an access plate at grade
TBX = instrument leads

Figure 2-1. Tank 241-AN-102 Riser Location.

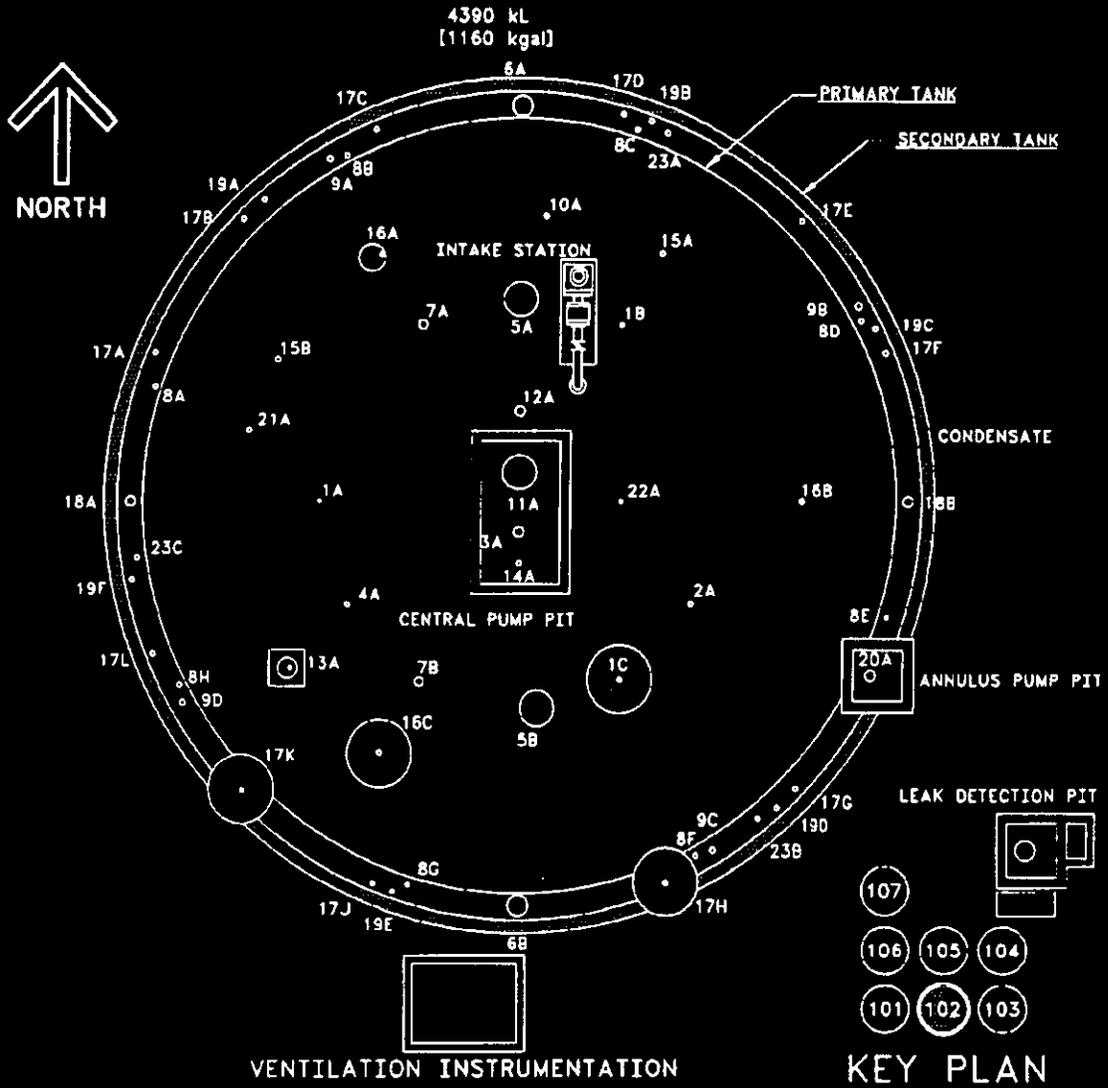
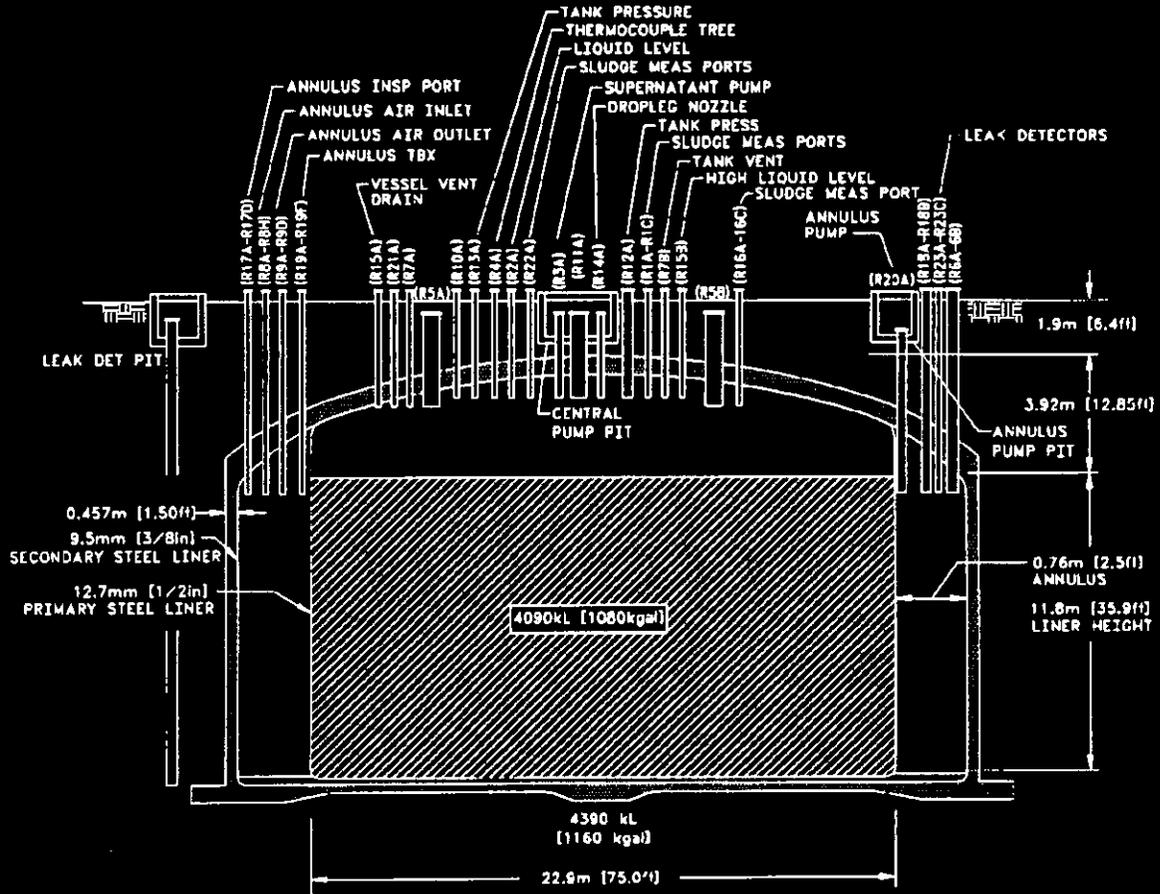


Figure 2-2. Tank 241-AN-102 Cross-Section.



During the third quarter of 1982, most of the waste in tank 241-AN-102 was transferred to the 242-A Evaporator feed tank (tank 241-AW-102). Tank 241-AN-102 then received noncomplexed waste processed in the 242-A Evaporator (Brager 1994, Agnew et al. 1995b). Most of this waste was removed during 1983 leaving about 125 kL (33 kgal) of waste in tank 241-AN-102. The tank was then filled with evaporator feed from various tanks; most of this waste was removed in July 1984 for an evaporator campaign leaving a heel of about 129 kL (34 kgal) in tank 241-AN-102. By the fourth quarter of 1984, tank 241-AN-102 was nearly filled with complexant concentrate waste (from a previous 242-A Evaporator campaign) stored in tank 241-AW-101 (Brager 1994). Since then, tank 241-AN-102 has received only small amounts of miscellaneous waste from the PUREX Plant and waste water (Agnew et al. 1995b, Koreski and Strode 1994).

A chronological summary of the major transfers of waste through 241-AN-102 is shown in Table 2-3.

Table 2-3. Tank 241-AN-102 Waste Transfer Summary.^{1,2}

Transfer	Time Period	Volume Received kiloliters (kilogallons)	Volume Removed
Evaporator feed and water	1981-82	3,930 (1,040)	3,800 (1,000)
Concentrated noncomplexed waste from Tank 241-AW-102	1982	1,760 (465)	1,760(465)
Evaporator feed and water	1983-84	4,030 (1,070)	4,020 (1,060)
Concentrated complexant waste from Tank 241-AW-101	1984	4,090 (1,080)	0
PUREX miscellaneous waste and water	1984-95	121 (32)	0
Unknown	1984-94	8 (2)	182 (48)

Notes:

¹Agnew et al. (1995b)

²Koreski and Strode (1994)

2.3.2 Historical Estimation of Tank Contents

The Historical Tank Content Estimate (HTCE) (Brevick et al. 1995) is a prediction of the contents for tank 241-AN-102 based on historical transfer data. The historical data used for the estimate are from the Waste Status and Transaction Record Summary (WSTRS) (Agnew et al. 1995b), the Hanford Defined Waste (HDW) (Agnew 1995) list, and the Tank Layer Model (TLM) (Agnew et al. 1995a). TheWSTRS is a compilation of available waste transfer and volume status data. The HDW provides the assumed typical compositions for 50 separate waste types. In some cases, the available data are incomplete, reducing the usability of the transfer data and the modeling results. The TLM takes theWSTRS data, models the waste deposition processes, and using additional data from the HDW which may introduce more error, generates an estimate of the tank contents. Thus, these model predictions can only be considered as estimates which require further evaluation using analytical data.

Based on the HTCE (Brevick et al. 1995), tank 241-AN-102 contains 337 kL (89 kgal) of concentrated supernatant solids and 3,808 kL (1,006 kgal) of supernate. Figure 2-3 shows a graphical representation of the estimated waste types and volumes for the tank layers. The HTCE total waste inventory for tank 241-AN-102 is presented in Table 2-4. The waste should contain large amounts of sodium, aluminum, nitrates, nitrites, carbonate, phosphate, sulfate, and hydroxide. The waste should also be rich in organic complexants; therefore, the total organic carbon content should be high. Quantities of strontium and cesium (of which the amount of cesium is significantly larger than the amount of strontium) should be present.

2.4 SURVEILLANCE DATA

Tank 241-AN-102 surveillance consists of surface level measurements (liquid and solid), and temperature monitoring inside the tank (waste and headspace). The annulus is ventilated and continually monitored by radiation and leak detectors for evidence of primary tank leakage.

2.4.1 Waste Level Readings

The tank waste surface level is monitored with a Food Instrument Corporation gauge. A surface level measurement of 999 cm (393.3 in.) was obtained on January 17, 1996. Changes in the waste level have shown a slight decreasing trend during the last three years. A graphical representation of the surface level history can be found in Figure 2-4. The average of three sludge level readings obtained from three separate risers on June 29, 1989, was 82.6 cm (32.5 in.) (Sasaki 1989).

Figure 2-3. Tank Layer Model Estimate.

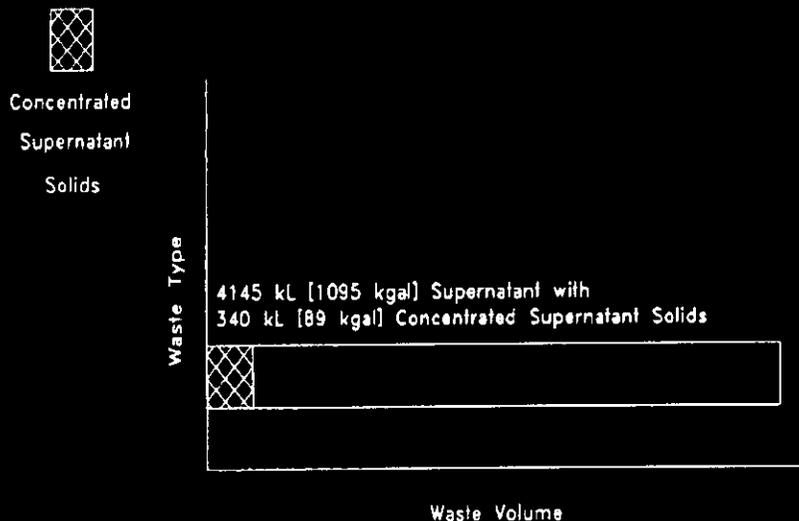


Table 2-4. Tank 241-AN-102 Historical Tank Content Estimate (2 sheets).¹

Total Tank Inventory Estimate			
Physical Properties			
Total Waste	5.26E+06 kg (1,100 kgal)		
Heat Load	4.17 kilowatts (14,200 Btu/hr)		
Bulk Density ²	1.27 (grams/cubic centimeter)		
Water wt% ²	62.8		
TOC wt% C (wet) ²	0.736		
Chemical Constituents	mol/L	ppm	kg
Na ⁺	5.90	1.07E+05	5.62E+05
Al ³⁺	0.728	15,500	81,400
Fe ³⁺ (total Fe)	0.00341	150	788
Cr ³⁺	0.0210	861	4,530
Bi ³⁺	7.32E-04	120	634
La ³⁺	4.43E-06	0.485	2.55
Hg ²⁺	5.05E-06	0.797	4.20
Zr (as ZrO(OH) ₂)	4.69E-04	33.7	177
Pb ²⁺	6.48E-05	10.6	55.7
Ni ²⁺	0.00297	137	722
Sr ²⁺	4.68E-06	0.323	1.70
Mn ⁴⁺	0.00718	311	1,640
Ca ²⁺	0.0238	752	3,960

Table 2-4. Tank 241-AN-102 Historical Tank Content Estimate (2 sheets).¹

Total Tank Inventory Estimate			
Chemical Constituents (Cont'd)	mol/L	ppm	kg
K ⁺	0.0206	635	3,340
OH ⁻	3.24	43,400	2.28E+05
NO ₃ ⁻	2.07	1.01E+05	5.33E+05
NO ₂ ⁻	1.24	44,800	2.36E+05
CO ₃ ²⁻	0.302	14,300	75,100
PO ₄ ³⁻	0.108	8,080	42,500
SO ₄ ²⁻	0.162	12,300	64,700
Si (as SiO ₃ ²⁻)	0.0303	670	3,520
F ⁻	0.0506	757	3,990
Cl ⁻	0.0998	2,780	14,700
citrate ³⁻	0.0235	3,490	18,400
EDTA ⁴⁻	0.0112	2,530	13,300
HEDTA ³⁻	0.0182	3,930	20,700
glycolate ⁻	0.0896	5,290	27,800
acetate ⁻	0.0133	617	3,250
oxalate ²⁻	1.67E-05	1.16	6.10
dibutyl phosphate	0.0125	1,580	8,330
butanol	0.0125	728	3,830
NH ₃	0.0330	442	2,330
Fe(CN) ₆ ⁴⁻	0	0	0
Radiological Constituents	Ci/L	μCi/g	Ci
Pu	—	0.0212	1.86 (kg)
U	0.00402 (mol/L)	753 (μg/g)	3,960 (kg)
Cs	0.201	158	8.33E+05
Sr	0.00949	7.47	39,300

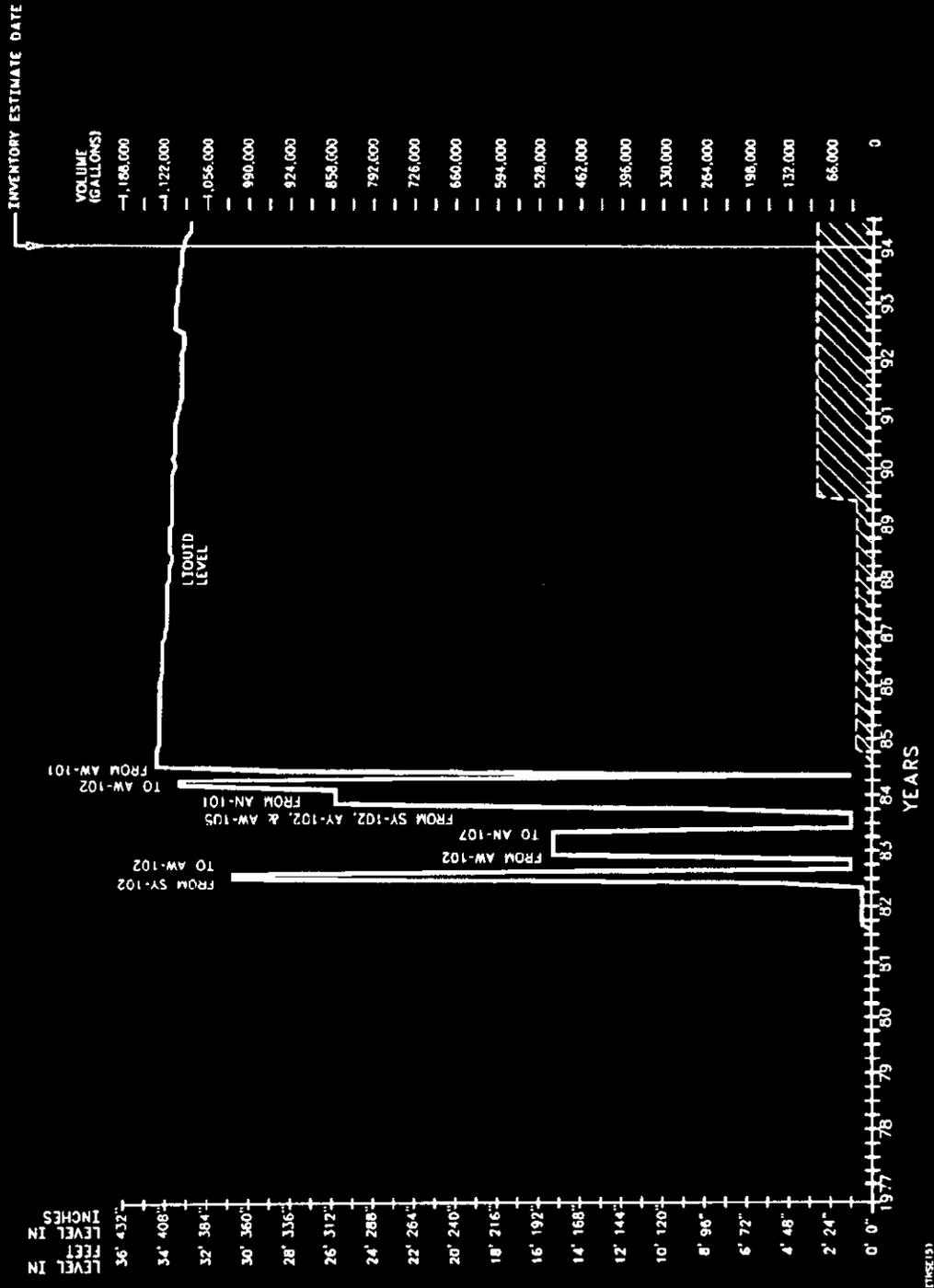
Notes:

wt% = weight percent

¹Brevick et al. (1995)

²Volume average for density, mass average water wt%, and total organic carbon wt% C

Figure 2-4. Tank 241-AN-102 Waste Level History.



2.4.2 Internal Tank Temperatures

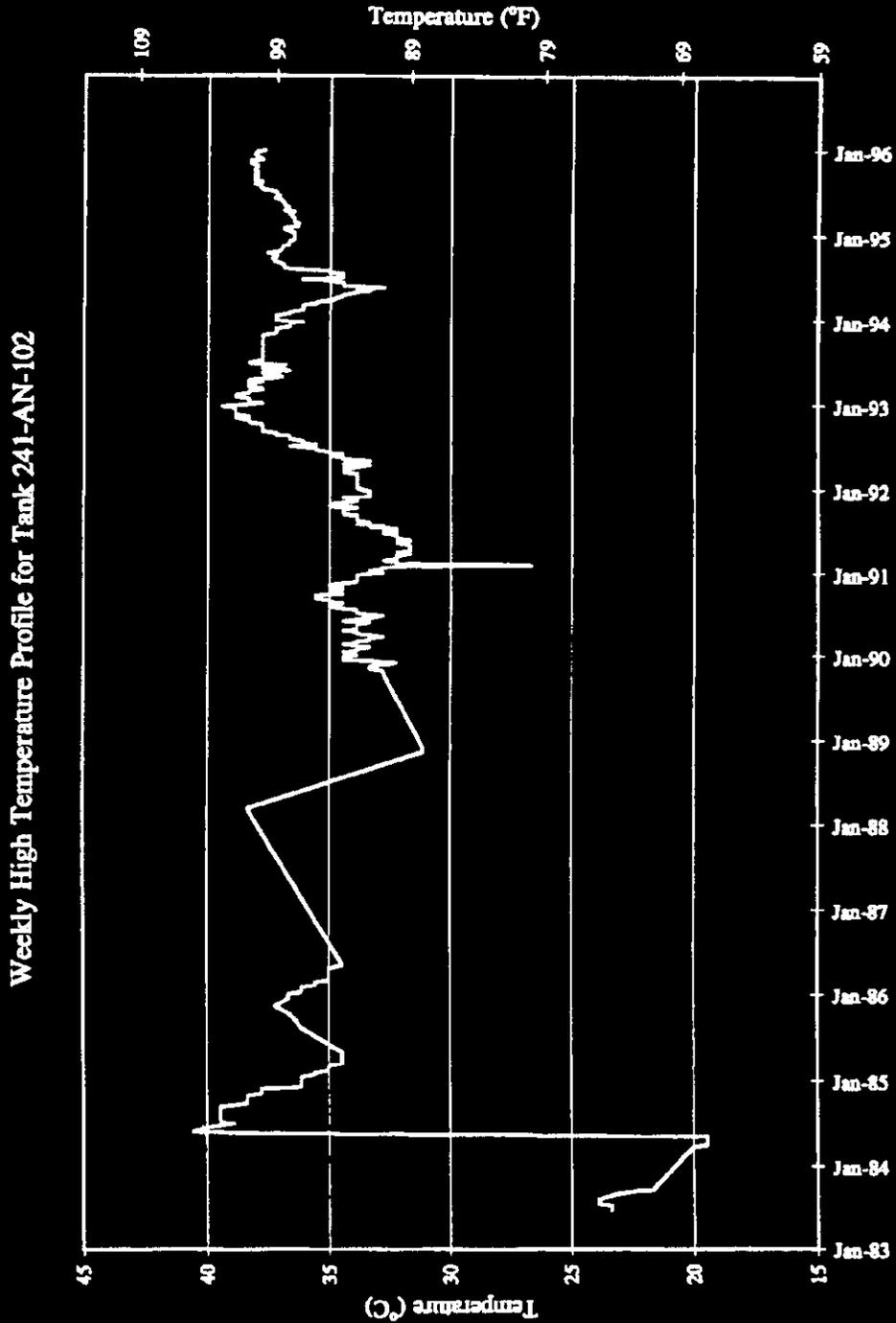
Temperature data for tank 241-AN-102 are recorded by 18 thermocouples attached at varying levels to one thermocouple tree located in riser 4A. Temperature data recorded from July 1983 to November 1990 are available for all 18 thermocouples, but the quality of data varied for each thermocouple. Temperature data recorded from January 1990 to September 1993 are available for six thermocouples.

The average temperature during these time periods was 32.6 °C (90.7 °F) with a standard deviation of 5 °C (9 °F). The minimum temperature was 18.3 °C (65 °F) and the maximum temperature was slightly above 40 °C (104 °F). These temperatures are within operating specifications (Harris 1994). The thermocouple plots can be found in Brevick et al. (1995). A graphical representation of the weekly high temperature can be found in Figure 2-5.

2.4.3 Tank 241-AN-102 Photographs

No in-tank photographs are available for tank 241-AN-102.

Figure 2-5. Tank 242-AN-102 Weekly High Temperature Plot.



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3.0 TANK SAMPLING OVERVIEW

This section describes the two sampling events used in the characterization of tank 241-AN-102: grab samples obtained in 1994 and a core sample obtained in 1990. The 1994 samples were collected for process control purposes. The 1990 sample was taken to support waste vitrification, grout, pretreatment, and retrieval activities (DiLiberto 1990). Descriptions of two additional sampling events (1989 and 1985) used for historical comparisons (see Section 5.2) are also briefly discussed. For a detailed discussion of sampling and analytical procedures, see the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

3.1 DESCRIPTION OF 1994 SAMPLING EVENT

Four grab samples, three supernate and one sludge, were obtained from riser 22A of tank 241-AN-102 on October 21, 1994 (Herting 1994; Jones 1994). Sampling depths and sample numbers are presented in Table 3-1. The immediate objective of the sampling was to determine whether the tank waste free-hydroxide concentration was within tank corrosion control specifications. Because the sampling was performed for process control purposes, no tank characterization plan was required. For the same reason, no field/trip blank was required. No problems were noted with the sampling event.

Table 3-1. Grab Sampling Elevations.

Sample Number	Sample Type	Sample Elevation ¹ centimeters (inches)
102-AN-1	Supernate	884 (348)
102-AN-2	Supernate	541 (213)
102-AN-3	Sludge	13 (5)
102-AN-4	Supernate	127 (50)

Note:

¹Sample elevation is measured from the tank bottom to the mouth of the sample bottle.

3.1.1 Sample Handling (1994)

The four samples were delivered to the Westinghouse Hanford Company 222-S Laboratory in 120-mL sample bottles on October 24, 1994. The sludge sample was archived for future characterization and caustic demand studies.

The three supernate samples were described as being very dark brown, almost black. Each sample was agitated by shaking before two 15-mL aliquots were removed from each bottle. The aliquots were then transferred into separate centrifuge cones.

Each aliquot was centrifuged for one hour, after which the liquid phase was decanted from each cone into a sample vial. All six cones contained two distinct solids layers of roughly equal volume. The weight percent centrifuged solids for the cones ranged from 1.09 to 1.30, with a mean of 1.23. The top layer was dark brown and the bottom layer was white. Each layer of solids was analyzed by polarized light microscopy and by x-ray diffraction. The white solids were composed mostly of octahedral crystals of sodium fluoride diphosphate. The dark brown solids were made up of submicron-sized particles that could not be identified by either method.

3.1.2 Sample Analysis (1994)

In addition to physical measurements (density, pH, and percent water), the clear liquid was analyzed for seven metals, eight anions, total organic carbon, total inorganic carbon, two radionuclides, and total alpha activity (Jones 1994). Identification numbers for the supernate samples are provided in Table 3-2. Analysis procedure numbers were not reported with the analytical results. Analytical results are tabulated in Appendix A and summarized in Section 4.

Table 3-2. Supernate Sample Identification Numbers.¹

Sample Number	Liquid from Centrifuged Aliquot	Description
102-AN-1	HE38459A	Liquid decanted from centrifuge cone A.
	HE38459D	Liquid decanted from centrifuge cone D.
102-AN-2	HE38459B	Liquid decanted from centrifuge cone B.
	HE38459E	Liquid decanted from centrifuge cone E.
102-AN-4	HE38459C	Liquid decanted from centrifuge cone C.
	HE38459F	Liquid decanted from centrifuge cone F.

Note:

¹Herting (1994)

3.2 DESCRIPTION OF 1990 SAMPLING EVENT

Two three-segment core samples were obtained from riser 7A of tank 241-AN-102 on May 24, 1990. Surface and sludge levels at the time of the sampling event indicated almost two full segments of sludge and one full segment of supernate could be expected from each core (Strasser 1990). One core was shipped to the Pacific Northwest National Laboratory for extrusion and chemical, radiochemical, and physical analyses. The other was extruded at the 222-S Process Chemistry Laboratory and archived. No chain-of-custody forms are available; therefore, drill string dose rates are not reported.

3.2.1 Sample Handling and Analysis (1990)

On July 10 and 11, 1990, the three segments delivered to the Pacific Northwest National Laboratory were extruded from the core sample collected from tank 241-AN-102. The segment descriptions presented in Table 3-3 come from laboratory core characterization worksheets; Douglas (1996) describes the subsequent sample preparation, analytical methods, and analytical results. A composite was prepared from the three segments. Some of the composite was archived for future analyses. The balance of the composite sample was either analyzed directly, or centrifuged into liquid and solids fractions that were analyzed for physical, chemical, and radiochemical properties (the laboratory identified the centrifuged solids fraction as AN-102-SOL and the liquid fraction as AN-102-SUP). Rheological analysis was also performed on the centrifuged solids fraction. The characterization of the core sample is outlined in Figure 3-1. The analytical results are tabulated in Appendix B and summarized in Section 4.

Inductively coupled plasma was used to determine concentrations for the metals in both the liquid and solids core composite fractions. Prior to analysis, solids centrifuged from the core composite were chemically fused using two separate fusions. A sodium hydroxide fusion was run in a zirconium crucible, while a potassium hydroxide fusion was run in a nickel crucible. The fused solid material was then dissolved in hydrochloric acid. All metals were prepared for analysis using sodium hydroxide fusion except for sodium and zirconium. Analyses for sodium and zirconium, along with additional determinations for aluminum, calcium, chromium, iron, manganese, potassium, and phosphorus, were performed after digestion with a potassium hydroxide fusion. Anion determinations in water leachates of the solids fraction were made using ion chromatography. Ion chromatographic analysis of the liquid fraction was performed by direct column injection of the liquid.

Both the liquid and solids fractions were analyzed for their transuranic element content. Concentrations were determined using both mass spectrometry and alpha energy analysis. Separation of americium and curium fractions from plutonium was accomplished using standard ion exchange techniques. The plutonium and americium/curium fractions were then analyzed by alpha counting followed by alpha energy analysis. Concentrations of plutonium and uranium were found to be too low for mass spectrometry determination.

Table 3-3. Tank 241-AN-102 Extruded Core Segments.

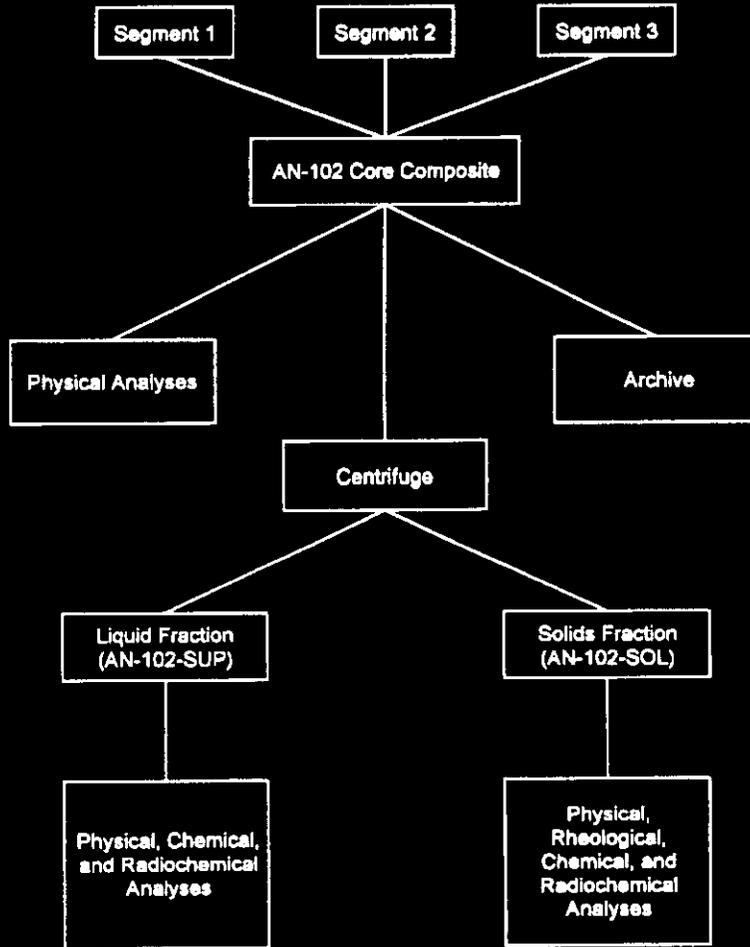
Segment	Length	Sample Recovery ¹ (%)	Liner Liquid (mL)	Gross Weight (grams)	Description
1	Not given	Not given	15	496	Mostly a noncohesive slurry; medium brown in color.
2	41 cm (16 in.)	84	10	538	Semi-solid. The bottom 15 cm (6 in.) were gray-brown; the remainder of the segment was light brown. The waste had a glossy surface; it was sticky, becoming less cohesive in the upper 10 to 12 cm (4 to 5 in.).
3	44 cm (17.5 in.)	92	10	544	Entire segment was semi-glossy. The lowest 2.5 cm (1 in.) of the sample was a dark brown and semi-solid; a 3.8-cm (1.5-in.) space with a trace of liquid followed. The space was followed by 5 cm (2 in.) of dark brown semi-solid waste, 15 cm (6 in.) of light brown semi-solid waste, and then 22 cm (8.5 in.) of a light brown waste of pudding consistency.

Note:

¹Sample recovery is an approximation derived by dividing the length of the recovered segment by the length of the sampler (48 cm [19 in.]).

Carbon-14 activity was measured on both the composite core solids and supernate materials by scintillation counting. Prior to analysis, oxidation (hot acidic persulfate method) and extraction of the carbon were accomplished using an acidification module. Tritium activity was measured using scintillation counting on water leachates of the solids samples. Activity in the supernate samples was determined directly. Precipitation or ion exchange methods were used to purify ⁶³Ni, ⁷⁹Se, and ⁹⁹Tc; activities were determined using beta or liquid scintillation counting. Activity for ²³⁷Np was measured directly by alpha energy analysis.

Figure 3-1. Characterization Flowchart for 1990 Core Sample.



3.3 DESCRIPTION OF 1989 SAMPLING EVENT

A supernate sample was obtained from tank 241-AN-102 in 1989. Other than the sample status report included as an attachment to Herting (1994), no other information was available, including descriptions of the tank location from which the sample was obtained and collection techniques used. Because the sample is supernate, it was likely collected using the bottle-on-a-string method. The sample was described as being dark brown and aqueous with solids present. The sample was analyzed for density and pH, 11 metals, four anions, total organic carbon, and six radionuclides. Analytical results are presented in Appendix C. Comparisons between results from this sampling event and the 1994 data are provided in Section 5.2.

3.4 DESCRIPTION OF 1984 SAMPLING EVENT

Two samples were collected from tank 241-AN-102 in 1984 and analyzed at the 222-S Laboratory (Bratzel 1985). A sludge sample (R-3640) was obtained from the bottom of the tank and a supernatant sample (R-3639) was obtained from 4.5 m (15 ft) above the tank bottom; however, a description of the techniques used to extract the samples was not available. The samples were centrifuged to separate suspended solids; aliquots were then analyzed. Solids were weighed, dried, and dissolved in 12 M HNO₃/0.2 M HF. A pretreatment procedure was used to destroy organics in the sample with the potential to interfere with plutonium and americium determinations. Plutonium and americium were separated using anion exchange, precipitation, and solvent extraction; activity was determined using scintillation counting. Metal cation analyses were determined by inductively coupled plasma spectroscopy, and anions were determined by ion chromatography. The total organic carbon content was determined by coulometric titration. Analytical results are presented in Appendix C. Comparisons between results from this sampling event and the 1994 and 1990 data are given in Section 5.2. Results showed that the waste stored in tank 241-AN-102 approached the transuranic categorization threshold of 100 nanocuries per gram.

4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

The purpose of this section is to summarize the sampling and analytical results from the most recent sludge and supernate samplings and to provide an estimate of the analyte concentrations for both waste phases. In addition, an estimate of the total amount (inventory) of the analyte in both the sludge and supernate is presented based on the estimated volumes of each respective layer. Table 4-1 summarizes where the data can be found in this document. Data regarding the physical characteristics of the sludge portion of the waste are presented and discussed in Section 4.3. All sampling events from which the reported results were derived are described in Section 3.

Table 4-1. Analytical Presentation Tables.

Analysis	Tabulated Result Location
Tank 241-AN-102 Chemical Composition Summary	Table 4-2
Analytical Results for the 1994 Supernate Sampling	Appendix A
Analytical Results for the 1990 Sludge Sampling	Appendix B

The sludge composition is based on the analysis of a three-segment core sample taken in 1990 (Douglas 1996). The three segments were combined to create a core composite, which was then centrifuged to form centrifuged solids and centrifuged liquid samples. The chemical analyses were performed on these centrifuged fractions. The overall sludge concentrations and inventories are calculated by summing the centrifuged solids and centrifuged liquid results (see Appendix B for an explanation of this calculation). The overall sludge chemical composition is summarized in Table 4-2, and individual results from analyses of the centrifuged fractions are tabulated in Appendix B. With regard to the centrifuged solids, two fusion digestion methods were used in preparing samples for inductively coupled plasma (ICP) analysis when analyzing for certain metals. For these metals, the results from the two fusion digestions were averaged. No comments concerning precision were provided.

When both the centrifuged solids and centrifuged liquid values were less than an instrument's detection limit, the calculated sludge concentration was determined based on the detection limits and reported as non-detect. The calculated projected inventory was then also reported as non-detect. In the case of one detected concentration result and one non-detect, a weighted average of the two results was taken and the calculated sludge concentration and projected inventory were recorded as non-detected values.

The supernate composition and inventory for the tank was based on the results from the 1994 grab samples (Herting 1994) as presented in Appendix A. A simple mean was calculated from the results for each analyte. This mean, along with the projected tank inventory, is presented in Table 4-2. This composition and inventory may change if transfers into and out of the tank occur.

4.2 DATA PRESENTATION

The best estimates regarding the chemical and radiochemical composition of the sludge and supernate (Herting 1994) portions of tank 241-AN-102 are listed in Table 4-2. Although minor additions of dilute non-complexed waste and water were added to the tank after the most recent sludge samples were taken, they did not make a substantial contribution to the sludge layer. The 1994 grab samples were taken after the last waste additions, so the analytical results should accurately represent the current contents. The analyte given in column one is followed by the mean sludge concentration and total projected sludge inventory in columns two and three, respectively. The mean supernate concentration and total projected supernate inventory are given in columns four and five, respectively. An explanation of how the supernate values were derived is given in Appendix A, and an explanation for the calculation of the sludge values is found in Appendix B.

Table 4-2. Sludge and Supernate Results for Tank 241-AN-102. (4 sheets)

Analyte	1990 Sludge Sample		1994 Supernate Samples	
	Sludge Concentration	Projected Inventory	Supernate Concentration	Projected Inventory
METALS	µg/g	kg	µg/mL	kg
Al	12,200	6,170	15,000	56,000
As	< 166	< 84.0	---	---
Sb	< 516	< 261	---	---
Ba	25.5	12.9	---	---
Be	< 0.712	< 0.360	---	---
B	< 792	< 401	---	---
Cd	< 31.1	< 15.7	---	---
Ca	811	410	440	1,700
Ce	< 403	< 204	---	---
Cr	1,370	693	290	1,100
Co	< 568	< 287	---	---
Cu	58.6	29.7	---	---
Dy	< 14.0	< 7.08	---	---

Table 4-2. Sludge and Supernate Results for Tank 241-AN-102. (4 sheets)

Analyte	1990 Sludge Sample		1994 Supernate Samples	
	Sludge Concentration	Projected Inventory	Supernate Concentration	Projected Inventory
Fe	1,500	759	---	---
La	< 29.5	< 14.9	---	---
Pb	< 272	< 138	---	---
Li	< 89.0	< 45.0	---	---
Mg	116	58.7	---	---
Mn	479	242	---	---
Mo	39.4	19.9	---	---
Nd	< 28.0	< 14.2	---	---
Ni	425	215	400	1,500
P	< 2,170	< 1,100	1,600	6,000
K	< 1,740	< 880	3,900	15,000
Re	< 58.1	< 29.4	---	---
Rh	< 295	< 149	---	---
Ru	< 225	< 114	---	---
Se	< 618	< 313	---	---
Si	1,360	688	---	---
Ag	< 1.59	< 0.805	---	---
Na	2.34E+05	1.18E+05	2.57E+05	9.66E+05
Sr	19.9	10.1	---	---
S	---	---	4,770	17,900
Te	< 195	< 98.7	---	---
Tl	< 6,310	< 3,190	---	---
Th	< 270	< 137	---	---
Ti	< 25.3	< 12.8	---	---
U	1,590	805	---	---
V	< 15.0	< 7.59	---	---
Zn	81.3	41.1	---	---
Zr	554	280	---	---
IONS	µg/g	kg	µg/mL	kg
Br	< 448	< 227	---	---
Cl	2,060	1,040	3,900	15,000

Table 4-2. Sludge and Supernate Results for Tank 241-AN-102. (4 sheets)

Analyte	1990 Sludge Sample		1994 Supernate Samples	
	Sludge Concentration	Projected Inventory	Supernate Concentration	Projected Inventory
Cr ⁶⁺	< 18.2	< 9.21	---	---
F ⁻	< 890	< 450	2,000	7,500
OH ⁻	---	---	4,100	15,000
NO ₃ ⁻	1.12E+05	56,700	2.21E+05	8.31E+05
NO ₂ ⁻	39,300	19,900	83,400	3.14E+05
PO ₄ ⁻³	3,030	1,530	4,900	18,000
SO ₄ ⁻²	25,900	13,100	14,000	53,000
RADIONUCLIDES	μCi/g	Cl	μCi/mL	Cl
²⁴¹ Am	0.584	296	---	---
¹²⁵ Sb	< 0.768	< 389	---	---
¹⁴ C	0.00128	0.648	---	---
¹⁴⁴ Ce	< 1.08	< 547	---	---
¹³⁴ Cs	< 0.130	< 65.8	---	---
¹³⁷ Cs	285	1.44E+05	382	1.44E+06
⁶⁰ Co	0.283	143	---	---
²⁴² Cm	1.59E-04	0.0805	---	---
^{243/244} Cm	0.0349	17.7	---	---
¹⁵² Eu	< 0.0966	< 48.9	---	---
¹⁵⁴ Eu	0.982	497	---	---
¹⁵⁵ Eu	1.05	531	---	---
¹⁵³ Gd	< 0.397	< 201	---	---
²³⁷ Np	< 9.43E-04	< 0.477	---	---
⁹⁴ Nb	1.74E-04	0.0880	---	---
²³⁸ Pu	0.0237	12.0	---	---
^{239/240} Pu	0.0705	35.7	---	---
¹⁰⁶ Ru	< 1.20	< 607	---	---
⁷⁹ Se	0.00199	1.01	---	---
⁹⁰ Sr	169	85,500	73.7	2.77E+05
⁹⁹ Tc	0.0982	49.7	---	---
¹¹³ Sn	< 0.965	< 488	---	---
³ H	0.00251	1.27	---	---

Table 4-2. Sludge and Supernate Results for Tank 241-AN-102. (4 sheets)

Analyte	1990 Sludge Sample		1994 Supernate Samples	
	Sludge Concentration	Projected Inventory	Supernate Concentration	Projected Inventory
Total Alpha	---	---	0.167	628
Total Beta	1,380	6.98E+05	---	---
PHYSICAL PROPERTIES				
Density	1.5 ¹ g/mL		1.40 g/mL	
pH	---		13.1	
% Water	40.3		50.0	
CARBON	µg C/g	kg C	µg C/mL	kg C
TIC	12,300	6,220	13,500	50,800
TOC	16,300	8,250	26,100	98,100

Note:

¹The reported density value is from a density determination on the core composite; it was not calculated by taking a weighted mean from the centrifuged solids and centrifuged liquid results.

4.3 PHYSICAL MEASUREMENTS

The limited physical properties measured on the 1994 supernate samples (density and percent water) are reported in Table 4-2, and are not discussed in this section unless otherwise indicated. Extensive physical characterization was performed on the 1990 core sample. The properties measured include density, percent solids, rheology, particle size, percent water, and energetics (Douglas 1996). These measurements will aid in the development of pretreatment and retrieval systems. A discussion of physical testing methodology and interpretation of results can be found in *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

4.3.1 Density, Percent Solids, Percent Oxides, and pH

Density, percent solids, percent oxides, and pH determinations for the 1990 core sample (Douglas 1996) are presented in Table 4-3. The weight percent solids measurement on the core composite was performed by drying a sample for 24 hours at 105 °C (219 °F). The result, 59.7 weight percent, was subtracted from 100 to derive the water weight percent in the sludge. This calculation yielded a weight percent water value of 40.3 percent. Percent oxides is a ratio of the mass of the sample remaining after heating to 1,050 °C (1922 °F) (after all volatile fractions have been driven off and the remaining constituents have been

converted to oxides) and the original mass of the sample, multiplied by 100. This value provides an indication of the amount of solids that will remain if the waste is vitrified. The centrifuged liquid and solid fractions were derived from centrifuged core composite material. Specific information concerning the analytical methods was not available.

Table 4-3. Physical Properties of Tank 241-AN-102 Sludge (1990).

Property	Measurement
Core Composite	
Density (g/ml)	1.5
Weight % Solids	59.7
Weight % Water	40.3
Weight % Oxides	28.7
Weight % Centrifuged Liquid	44.6
Weight % Centrifuged Solids	55.4
Volume % Centrifuged Liquid	50.5
Volume % Centrifuged Solids	49.5
Centrifuged Liquid	
Density (g/ml)	1.4
pH	14.5
Centrifuged Solids	
Density (g/ml)	1.7

A weight percent centrifuged solids determination was also performed for the 1994 supernate samples. Two aliquots were taken from each of the three grab samples, producing six samples. All six samples were centrifuged for one hour, after which the liquids were clear. The weight percent centrifuged solids was measured for each of the six samples; the results are tabulated in Table 4-4.

Table 4-4. Supernate Weight Percent Centrifuged Solids Results (1994).

Sample	Weight Percent Solids	Mean	Relative Standard Deviation
	wt %	wt %	%
102-AN-1	1.25	1.23	5.6
	1.09		
102-AN-2	1.28		
	1.25		
102-AN-4	1.20		
	1.30		

Note:
wt % = weight percent

4.3.2 Rheology

Rheological properties, including shear strength, penetration resistance, shear stress vs. shear rate, and viscosity, were measured on different components of the tank 241-AN-102 sludge (Douglas 1996). Some of the rheological analyses were performed on the core composite, while others were performed on centrifuged fractions of the composite.

4.3.2.1 Shear Strength and Penetration Resistance. Shear strength was measured on two core composite samples that had previously been used for a settled solids measurement (after sitting for 83 days). The average measured shear strength was 3,050 dynes per square centimeter at 33 °C (91.4 °F).

Penetration resistance was evaluated at 33 °C on a centrifuged solids sample that had sat for 61 days. The analysis was performed in a 50-mL centrifuge cone, which had a diameter of 2.86 cm. The solids were over 4 cm deep. Probes of 0.64 and 2.54 cm diameters were used in the analysis. The penetration resistance of 20 psi obtained with the 0.64-cm-diameter probe indicates that the centrifuged solids are cohesive. However, the penetration resistance of 315 psi acquired using the 2.54-cm-diameter probe indicates that the centrifuged solids are dilatant. The penetration resistance obtained with the larger probe is likely not a valid measure of the true penetration resistance due to the relative diameter of the probe to the diameter of the sample and its container (2.86 cm).

4.3.2.2 Shear Stress as a Function of Shear Rate. Shear stress versus shear rate was measured on the core composite. The rheogram, presented in Figure 4-1, demonstrated that the waste exhibited yield-pseudoplastic behavior and had a yield point of 2.61 Pa. The data from the rheogram were fit to the following non-linear yield power law model:

$$\tau = \tau_y + kD^\gamma$$

where

- τ = shear stress
- τ_y = yield point
- D = shear rate
- k = consistency factor
- γ = flow behavior index.

The power law model describes the change in shear stress as a function of shear rate and the flow behavior index. Table 4-5 displays the power law model parameters. As can be seen in Figure 4-1, the data fit to the power law model was excellent, with an r^2 value of 0.9998. Also note in the figure that the fitted yield point was 0.631 Pa. The fitted yield point is determined manually by drawing tangents to the initial portion of the curve and the curve at about 25 s^{-1} . 1993). The fitted yield point is considered a more representative value than 2.61 Pa, because the data are usually unreliable at low shear rates. Values for the critical flow rate and Reynold's number were not determined.

Table 4-5. Power Law Model Parameters.

Yield Point, τ_y	Consistency Factor, k	Flow Behavior Index, γ
Pa	Pa	
2.61	0.605	0.781

4.3.2.3 Viscosity. Viscosity was measured on the core composite. A plot of the viscosity as a function of shear rate is provided in Figure 4-2. The figure reveals that the viscosity varied from 275 cP at a shear rate of 50 s^{-1} to 155 cP at a shear rate of 470 s^{-1} .

Figure 4-1. Shear Stress Versus Shear Rate.

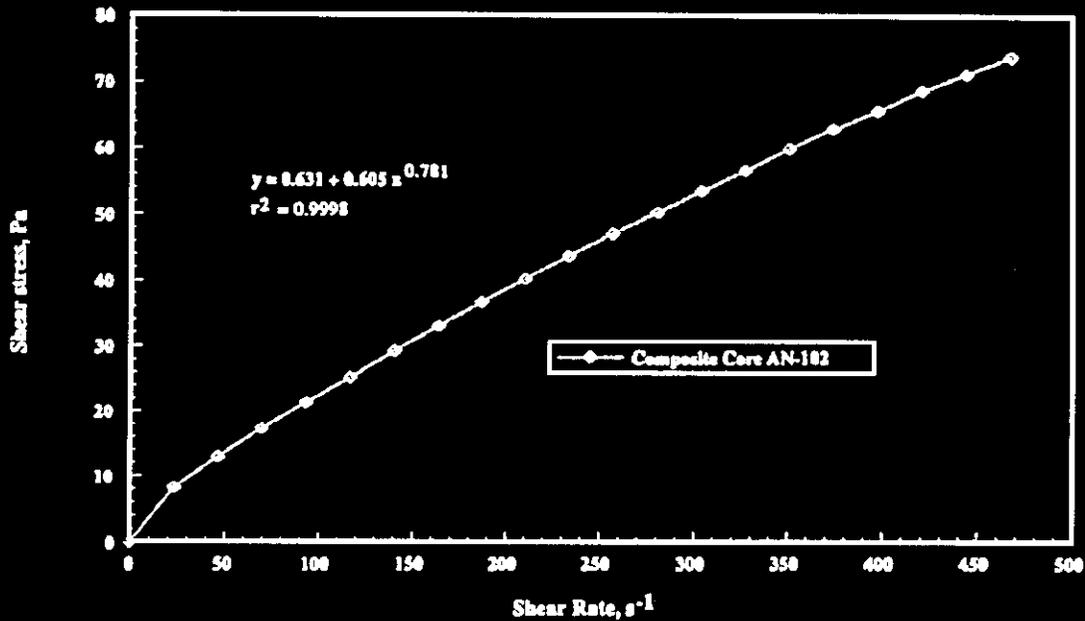
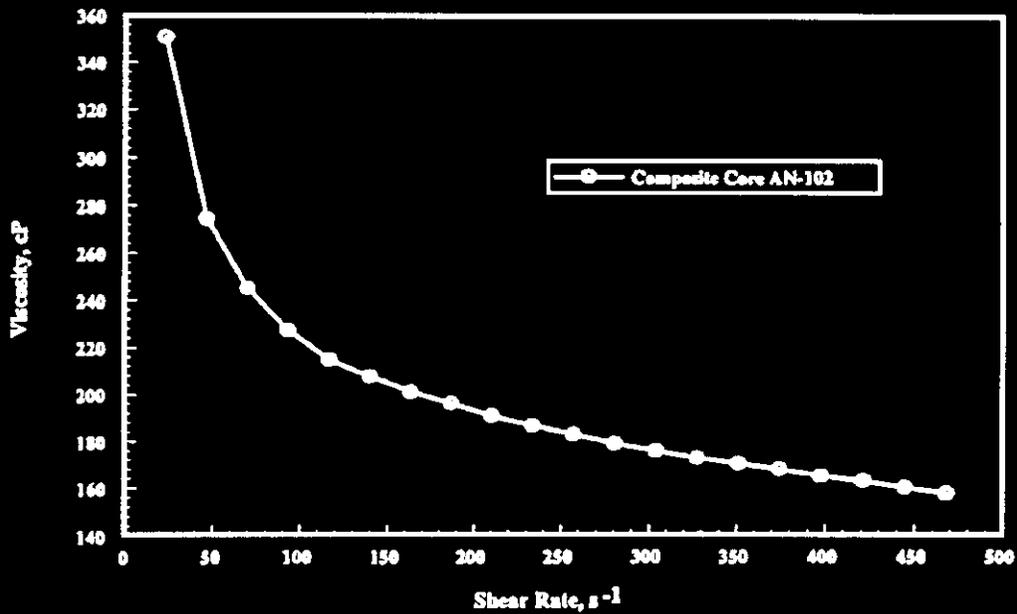


Figure 4-2. Viscosity Versus Shear Rate.



4.3.3 Particle Size

A particle size analysis was performed on the centrifuged solids sample using a Brinkman 2010 particle size analyzer (Douglas 1996). This apparatus determines particle size by measuring the amount of light that is attenuated as a laser beam is passed through the sample (DeLorenzo et al. 1994). The sample was suspended in a 50 percent glycerine/waste solution to minimize sample dissolution into the suspension medium (which would result in an inaccurate determination). Figures 4-3 and 4-4 present the particle size results.

The number density particle size distribution is shown in Figure 4-3. The most frequently encountered particle size was 0.75 μm , while the mean and median particle sizes were 1.06 μm and 0.90 μm , respectively. Figure 4-4 displays the particle size distribution data on a volume density basis. Particle size distribution based on number density is converted to volume density by assuming a mean particle size in each range measured and a spherical geometry for each particle. The most numerous particle size on a volume density basis was 1.51 μm ; the mean was 3.47 μm and the median was 3.27 μm . The volume distribution was fairly even for particles ranging from 2 to 7 μm .

4.3.4 Thermal Analyses

This section presents the results of thermogravimetric and differential scanning calorimetric analyses performed on centrifuged samples of the composited core from the 1990 sampling event (Douglas 1996). After centrifuging the core composite material, some light-colored solids were found floating on top of the centrifuged liquid. These solids were collected and identified as a "crust." Both thermal analyses were performed on this crust material and the centrifuged solids. No thermal analyses were done on the centrifuged liquid, or on the tank supernatant.

4.3.4.1 Thermogravimetric Analysis. A thermogravimetric analysis (TGA) was performed on both the centrifuged solids and the crust material from the 1990 sampling event. Thermogravimetric analysis measures the mass of a sample while the temperature of the sample is increased at a constant rate. Any decrease in the weight of a sample represents loss of volatile matter either through evaporation or through a reaction that forms gas phase products.

The TGA results are compiled in Table 4-6. Three transitions were recorded in each TGA scan. The weight loss in the first transition is attributed to sample water loss through evaporation. These values are considered the weight percent water. As can be seen in Table 4-6, the weight percent water in the centrifuged solids was 30 percent, while the crust material contained 25 percent water by weight. Typical sample sizes for TGA range from 2 to 10 mg; the actual sample sizes for the centrifuged solids and the crust material were 7.55 mg and 2.58 mg, respectively. Figure 4-5 displays the centrifuged solids TGA scan, while Figure 4-6 presents the TGA plot for the crust material.

Figure 4-3. Number Density Particle Size Distribution.

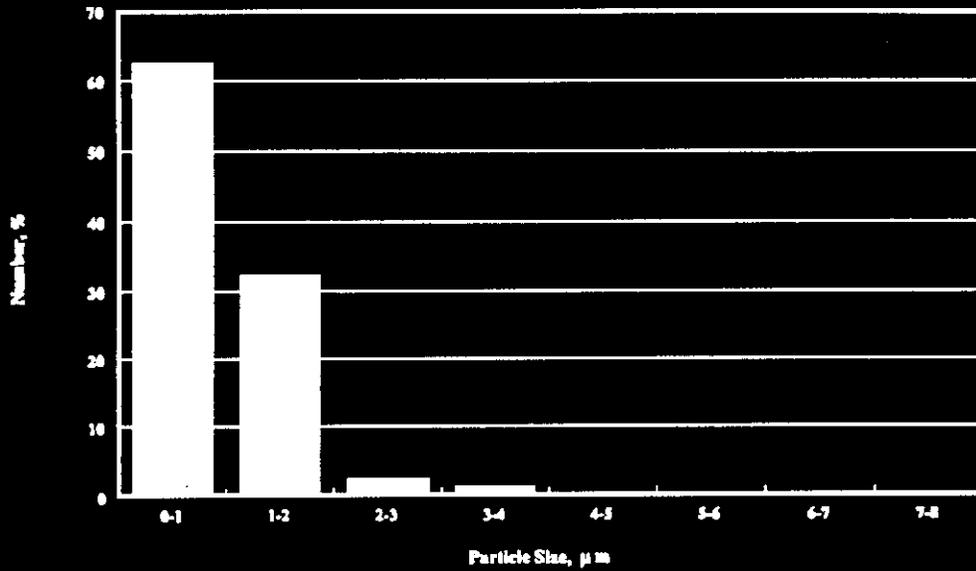


Figure 4-4. Volume Density Particle Size Distribution.

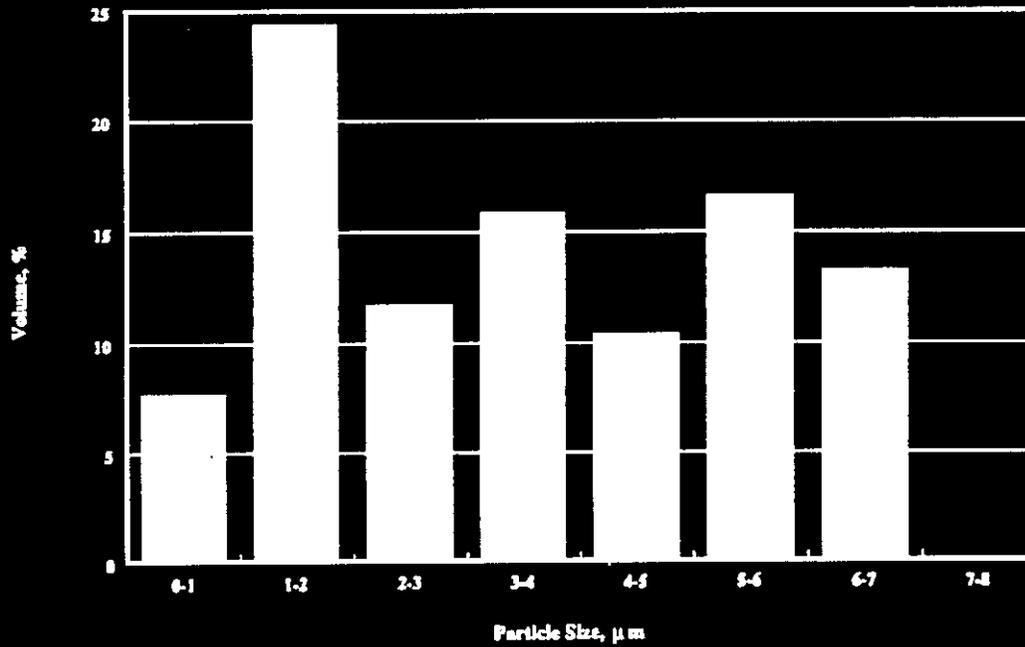


Table 4-6. Thermogravimetric Analysis Results for Tank 241-AN-102 (1990).

Sample	Transition 1		Transition 2		Transition 3	
	Temp. Range (°C)	Weight % Loss	Temp. Range (°C)	Weight % Loss	Temp. Range (°C)	Weight % Loss
Centrifuged solids	50 to 190	30	210 to 330	3	390 to 480	5
Crust material	50 to 160	25	210 to 320	7	Not determined	8

Note:

Temp. = Temperature

4.3.4.2 Differential Scanning Calorimetry. Differential scanning calorimetry (DSC) experiments were also run on both the centrifuged solids and the crust material (Douglas 1996). No DSC was performed on the centrifuged liquid or supernatant. In a DSC analysis, heat absorbed or emitted by a substance is measured while the substance is exposed to a linear increase in temperature. While the substance is being heated, a gas such as nitrogen is passed over the waste material to remove any gases being released. The onset temperature for an endothermic (characterized by or causing the absorption of heat) or exothermic (characterized by or causing the release of heat) event is determined graphically.

Similar to the TGA scans, three transitions were observed in the DSC plots. The first and third transitions for both samples were endothermic. Exothermic behavior was noted in the second transition for each sample. Exothermic behavior is not surprising given that tank 241-AN-102 contains complexant concentrate waste, which is high in organic and nitrate/nitrite concentrations. Duplicate runs on the centrifuged solids gave exothermic reactions of -250 and -530 J/g (wet weight). These results were converted to a dry weight basis for comparison to the safety screening DQO limit of -480 J/g by dividing the change in enthalpy (ΔH) by the solid fraction of the waste (1 - weight fraction water). The equivalent dry weight values (converted using the TGA result of 30 weight percent water) for these exotherms were -357 and -757 J/g. An exothermic reaction of -700 J/g (wet weight) was observed in the crust material. Its equivalent dry weight, using the TGA value of 25 weight percent water, was -933 J/g. Consequently, two of the three DSC measurements exceeded the safety screening DQO limit of -480 J/g (dry weight). Typical DSC sample sizes range from 2 to 10 mg; the actual sample sizes for the DSC runs were 7.23 mg for the centrifuged solids and 7.21 mg for the crust material.

The DSC results are presented in Table 4-7. The temperature range and the magnitude of the enthalpy change are provided for each transition. Figures 4-7 and 4-8 display the respective DSC scans for the centrifuged solids and the crust material.

Figure 4-5. Thermogravimetric Analysis Scan for the Centrifuged Solids Sample.

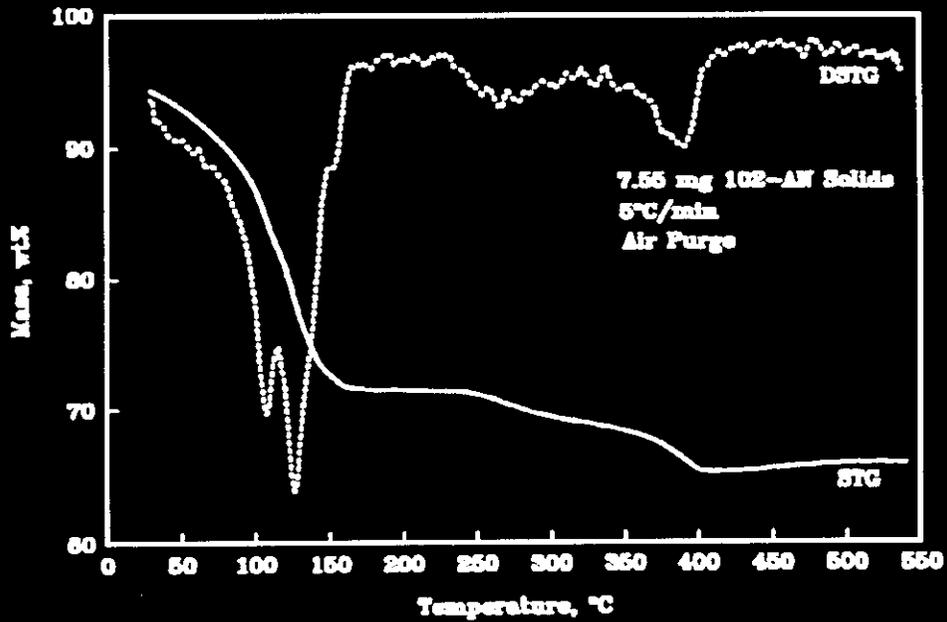


Figure 4-6. Thermogravimetric Analysis Scan for the Crust Material.

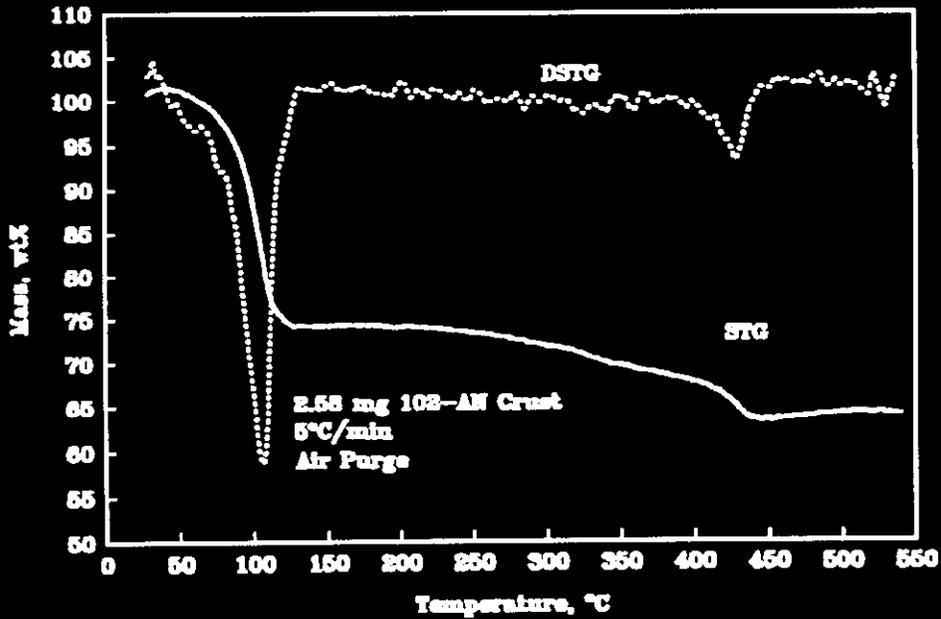


Table 4-7. Differential Scanning Calorimetry Results for Tank 241-AN-102 (1990)¹.

Sample	Run	Transition 1		Transition 2		Transition 3	
		Temp. Range (°C)	ΔH (J/g)	Temp. Range (°C)	ΔH (J/g)	Temp. Range (°C)	ΔH (J/g)
Centrifuged solids	1	50 to 190	1,500	210 to 330	-250; -530 ²	390 to 480	482
Crust material	1	50 to 160	380	210 to 320	-700	Not determined	Endothermic (ΔH not determined)

Notes:

Temp. = Temperature

¹Results reported on a wet-weight basis

²Duplicate results were reported for this transition only

Figure 4-7. Differential Scanning Calorimetry Scan for the Centrifuged Solids.

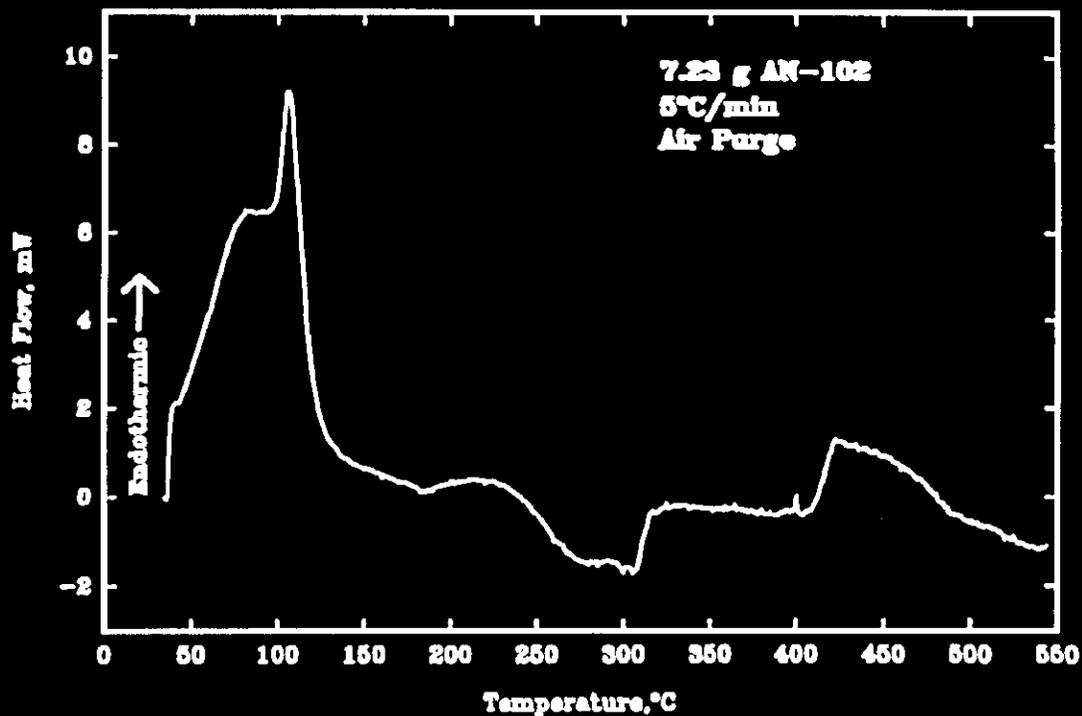
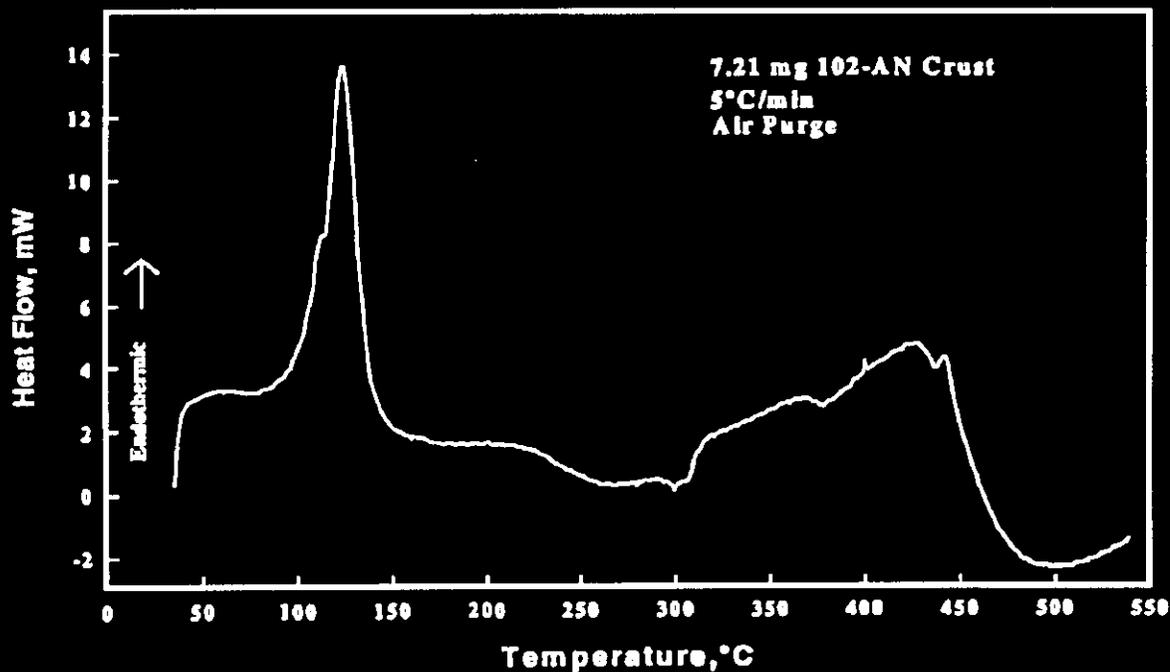


Figure 4-8. Differential Scanning Calorimetry Scan for the Crust Material.



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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

The purpose of this chapter is to evaluate the overall quality and consistency of the available results for tank 241-AN-102 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 any limitations in the data's use. Some consistency checks were not possible due to the limited number of analyses performed.

5.1.1 Field Observations

Samples were taken from only one riser in both the 1990 and 1994 sampling events, thereby eliminating any assessment of horizontal waste disposition within the tank and increasing the possible bias in the data obtained. The 1994 supernate sampling was the only time in which multiple depths within a phase were sampled, allowing some assessment of the vertical homogeneity in the supernate. No information was made available regarding the sample recovery for this sampling event. Estimated sample recoveries for two of the three 1990 core segments was good (84 and 92 percent); a recovery could not be calculated for the third segment. Also, the 1990 event presented data based on a single analysis of a composite sample; this prevents an estimation of the possible analytical uncertainty associated with the analysis.

5.1.2 Quality Control Assessment

The usual quality control assessment includes an evaluation of the appropriate blanks, duplicates, spikes, and standards that were performed in conjunction with the chemical analyses. However, only limited quality control information was available for the 1994 sampling event, while none was available for the 1990 event. Spikes were not performed during the 1994 analysis.

The 1994 sampling included one standard run in conjunction with each analyte, which provided an estimate of the accuracy of the analysis. The accuracy criteria established by the laboratory for standards was 80 to 110 percent recovery for the radionuclides and 80 to 120 percent recovery for the inorganic analytes (DOE 1995). If a standard is above or below

a certain criterion, then the analytical results may be biased high or low, respectively. None of the standards conducted on the 1994 supernate analytes violated the criteria (Herting 1994).

The laboratory analytical precision criterion for the radionuclides is ≤ 20 relative percent difference (RPD) when the sample and duplicate results are at least five times the detection limit (DOE 1995). For the inorganic analytes, the criterion is the same except that the sample and duplicate results must be at least ten times the detection limit. These precision requirements are calculated by the RPD, which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times 100. All of the 1994 analytes had three pairs of samples to evaluate for precision, with the exception of the ion chromatography (IC) analytes. For the IC analytes, one of the six duplicate samples gave consistently high values, indicating a possible bias. Therefore, those values were not used in calculating the mean concentrations or RPDs for this data set (Herting 1994). The only analyte to have any RPDs outside the quality control target level was potassium, which had two of three outside the limits (28 and 60 percent).

In summary, the vast majority of the quality control results were within the laboratory boundaries (DOE 1995). Consequently, the data are considered reliable and no limitations have been applied to their use.

5.1.3 Data Consistency Checks

Comparisons of results from different analytical methods, such as phosphorus and sulfur by inductively coupled plasma (ICP) to IC phosphate and sulfate results, along with mass and charge balances, help to assess the overall consistency of the data. These comparisons are provided below. Other consistency checks, such as total alpha or total beta compared to the sum of the alpha or beta emitters, were not possible due to a lack of data.

5.1.3.1 Phosphate/Phosphorus and Sulfate/Sulfur Comparisons. This data consistency check compares the phosphorus and sulfur results determined from ICP with the phosphate and sulfate results determined from IC. A close correlation between the two strengthens the credibility of both results.

Two phosphorus comparisons were possible from the different sampling events of tank 241-AN-102. The first comparison was from the 1994 supernate results. The ICP phosphorus results gave a value of 1,600 $\mu\text{g/mL}$, which converted to a phosphate value of 4,900 $\mu\text{g/mL}$. This showed excellent data consistency as the IC phosphate result was 4,900 $\mu\text{g/mL}$. The second comparison was for the centrifuged liquid from the 1990 sampling event. The ICP phosphorus result was 1,150 $\mu\text{g/g}$, which converted to a phosphate value of 3,520 $\mu\text{g/g}$. This value is 52 percent larger than the IC phosphate result of 2,310 $\mu\text{g/g}$.

A comparison of ICP sulfur to IC sulfate was possible for the 1994 supernate results. The ICP sulfur value of 4,770 $\mu\text{g/mL}$ converts to 14,300 $\mu\text{g/mL}$ of sulfate. This compares very favorably with the IC sulfate result of 14,000 $\mu\text{g/mL}$.

5.1.3.2 Mass and Charge Balance. The principle objective in performing a mass and charge balance is to determine if the measurements are self-consistent. Because supernate comprises nearly 92 percent of the tank waste, mass and charge balances were done only for this waste phase. In calculating the balances, only supernate analytes listed in Table 4-2 that were detected at a concentration of 2,000 $\mu\text{g/g}$ or greater were considered. The analyte concentrations listed in Tables 5-1 and 5-2 were derived by dividing the values listed in Table 4-2 (in $\mu\text{g/mL}$) by the supernate density (1.40 g/mL) to convert to units of $\mu\text{g/g}$.

Aluminum was assumed to be present as the aluminate anion, AlO_2^- , which was calculated stoichiometrically from the inductively coupled plasma data for aluminum. There may be some argument whether the species in question is aluminate or tetrahydroxoaluminate, but the difference in their molecular weights has a minimal effect on the overall mass balance and no effect on the charge balance. Although smaller concentrations of other forms of aluminum are probably also present in the waste, they are not included in order to keep the mass and charge balance calculations simple and consistent. The acetate and carbonate data were derived from the total organic carbon and total inorganic carbon analyses, respectively. The other anionic analytes listed in Table 5-2 were assumed to be present as sodium and potassium salts and were expected to balance the positive charge exhibited by the cations. Sulfur is considered to be present as the sulfate ion, and appears to be completely water soluble. As discussed in section 5.1.3.1, the sulfate concentration calculated from the ICP result was 14,300 $\mu\text{g/mL}$ (10,200 $\mu\text{g/g}$) as compared to the IC result of 14,000 $\mu\text{g/mL}$ (10,000 $\mu\text{g/g}$). Thus, sulfur appears only in the anion mass and charge calculations. The concentrations of the cationic species in Table 5-1, the anionic species in Table 5-2, and the percent water were ultimately used to calculate the mass balance. The uncertainty estimates (RSDs) associated with each analyte, along with the uncertainty for the cation and anion totals, are also given in the tables.

Table 5-1. Cation Mass and Charge Data (1994).

Analyte	Concentration ($\mu\text{g/g}$)	RSD (Mean) (%)	Charge ($\mu\text{eq/g}$)
Potassium	2,790	6.7	71
Sodium	184,000	2.7	8,000
Totals	187,000	2.7	8,070

Note:

RSD (Mean) = relative standard deviation of the mean

Table 5-2. Anion Mass and Charge Data (1994).

Assumed Species	Concentration (µg/g)	RSD (Mean) (%)	Charge (µeq/g)
Aluminate ¹	23,400	0.30	397
Acetate (TOC) ²	45,700 (18,600)	1.9	775
Carbonate (TIC) ²	48,200 (9,640)	1.2	1,610
Chloride	2,790	3.5	79
Hydroxide	2,930	0	172
Nitrate	158,000	0.4	2,550
Nitrite	59,600	0.6	1,300
Phosphate	3,500	2.9	111
Sulfate	10,000	3.2	208
Totals	354,000	0.4	7,200

Notes:

¹The concentration of aluminate (AlO₂⁻) was derived stoichiometrically from the aluminum concentration of 10,700 µg/g (15,000 µg/mL ÷ 1.4 g/mL).

²The values in parentheses are from the TOC and TIC analytical results, and were used to derive the acetate and carbonate values on the left.

The mass balance was calculated from the formula below. The conversion factor from µg/g to weight percent is 0.0001.

$$\begin{aligned} \text{Mass balance} &= \% \text{ Water} + 0.0001 \times \{\text{Total Analyte Concentration}\} \\ &= \% \text{ Water} + 0.0001 \times \{K^+ + Na^+ + AlO_2^- + C_2H_3O_2^- + CO_3^{2-} + Cl^- + OH^- + NO_3^- + \\ &\quad NO_2^- + PO_4^{3-} + SO_4^{2-}\} \end{aligned}$$

The analyte concentrations calculated from the above equation totaled 541,000 µg/g. The mean weight percent water obtained from thermogravimetric analysis reported in Table 4-2 is 50.0 percent, or 500,000 µg/g. The mass balance resulting from adding the percent water to the total analyte concentration is 104 percent (Table 5-3).

The following equations demonstrate the derivation of the total cationic and total anionic charge; the charge balance is the ratio of these two values.

$$\text{Total cations (microequivalents)} = Na^+/23.0 + K^+/39.1 = 8,070 \text{ microequivalents}$$

Total anions (microequivalents) = $\text{AlO}_2^-/59 + \text{C}_2\text{H}_3\text{O}_2^-/59.0 + \text{CO}_3^{2-}/30.0 + \text{Cl}^-/35.5 + \text{OH}^-/17.0 + \text{NO}_3^-/62.0 + \text{NO}_2^-/46.0 + \text{PO}_4^{3-}/31.7 + \text{SO}_4^{2-}/48.1 = 7,200$ microequivalents

The charge balance obtained by dividing the sum of the positive charge by the sum of the negative charge was 1.12.

In summary, the above calculations yield reasonable (close to 1.00 for charge balance and 100 percent for mass balance) mass and charge balance values, giving a strong indication that the analytical results for the supernate are fairly self-consistent and, therefore, reliable.

Table 5-3. Mass Balance Totals.

	RSD (Mean) (%)	Concentrations ($\mu\text{g/g}$)
Total from Table 5-1	2.7	187,000
Total from Table 5-2	0.4	354,000
Water %	0	500,000
Grand Total	0.5	1,041,000

5.2 COMPARISON OF HISTORICAL AND ANALYTICAL RESULTS

Comparison of results from two different sampling events gives an indication of the precision of the analyses, assuming that the tank contents have remained relatively unchanged between the events. This is a fairly good assumption for tank 241-AN-102, given that only small quantities of dilute non-complexed waste and water have been added to the tank since early 1984.

Table 5-4 gives the results of three different sampling events of the upper, supernate portion of tank 241-AN-102. The data from 1984 and 1989 are limited in that they were both based on a single analysis of a single sample, while the 1994 results were derived from three pairs of duplicate samples. A single data point precludes an estimation of the analytical uncertainty associated with the analysis. In addition, no quality control information was available for the 1984 and 1989 sampling events. The comparisons show very good agreement, with only two analytes (potassium and hydroxide) showing slightly greater than a two-fold difference.

Table 5-4. Historical Comparisons within the Supernate Layer of Tank 241-AN-102.

Analyte	1984 Supernate	1989 Supernate	1994 Supernate
METALS	mol/L	mol/L	mol/L
Al	0.537	0.460	0.55
Ca	0.0123	0.0101	0.011
Cr	0.00796	0.00605	0.0055
K	0.0531	0.0407	0.10
Na	10.4	7.65	11.2
Ni	0.00643	0.00577	0.006
P	---	0.0497	0.052
IONS	mol/L	mol/L	mol/L
Cl ⁻	0.0949	---	0.11
F ⁻	< 0.118	---	0.11
NO ₃ ⁻	3.61	3.54	3.57
NO ₂ ⁻	1.32	1.36	1.81
OH ⁻	0.201	0.445	0.24
PO ₄ ⁻³	0.0473	---	0.052
SO ₄ ⁻²	0.114	---	0.15
RADIONUCLIDES	μCi/L	μCi/L	μCi/L
¹³⁷ Cs	3.10E+05	4.00E+05	3.82E+05
PHYSICAL PROPERTIES	g/ml	g/ml	g/ml
Density	1.39	1.34	1.40
CARBON	g/L	g/L	g/L
TIC	10.1	13.2	13.5
TOC	33.7	27.3	26.1

Table 5-5 gives the results of two sampling events comparing liquid obtained from centrifuging slurry samples from the bottom of the tank into solids and liquid portions. Most of the analytes show fairly good agreement between the two samples, with a few notable discrepancies. Manganese appears to show a greater than ten-fold difference, but the larger value is a non-detect. Nitrate and nitrite showed five-fold and four-fold differences, respectively. These differences may be due to the methods of sampling. The 1984 sample was probably obtained using the "bottle-on-a-string" method and may possibly have been

contaminated by supernate during withdrawal from the tank. The 1990 sample was acquired using the core sampling method, and is less likely to be contaminated by tank supernate. Different sampling locations could also be a factor. The sampling riser for the 1984 sampling event was not reported.

Table 5-5. Historical Comparisons of Centrifuged Liquid Within the Sludge Layer of Tank 241-AN-102.

Analyte	1984 Centrate ¹	1990 Centrate ²
METALS	mol/L	mol/L
Al	0.538	0.48
Ca	0.0203	0.011
Cr	0.0101	0.0077
Fe	0.00468	0.0053
K	0.0468	0.049
Mn	< 0.0155	0.0013
Na	see footnote 3	7.6
Zr	< 0.00139	1.6E-05
IONS	mol/L	mol/L
Cl ⁻	0.0903	0.042
F ⁻	< 0.132	0.13
NO ₂ ⁻	1.28	0.30
NO ₃ ⁻	3.38	0.66
PO ₄ ⁻³	0.0480	0.034
SO ₄ ⁻²	0.0196	0.045
RADIONUCLIDES	μCi/L	μCi/L
¹³⁷ Cs	4.48E+05	2.00E+05
⁹⁰ Sr	1.01E+05	45,000
PHYSICAL PROPERTIES	g/ml	g/ml
Density	1.39	1.4

Notes:

¹Centrate from centrifuged solids slurry sample.

²Liquid from centrifuged core composite material.

³The value reported in the source document (Bratzel 1985) is 40.6 mol/L. This number is considered unreliable, so no comparison is possible.

Table 5-6 gives the results of two sampling events comparing the solids within the sludge layer of tank 241-AN-102. The results generally show good agreement for only aluminum, calcium, ¹³⁷Cs and ^{239/240}Pu. The problems in agreement for the remaining analytes could be due to heterogeneity of the waste on the tank bottom. Different sampling locations may also have an effect. Because the 1984 sampling riser was not identified, it is not known whether the two samples were removed from the same riser.

Table 5-6. Historical Comparisons of Solids Within the Sludge Layer of Tank 241-AN-102.

Analyte	1984 Solids	1990 Solids ¹
METALS	µg/g	µg/g
Al	14,000	14,600
Ca	1,100	1,210
Cr	850	2,240
Fe	710	2,530
Mn	< 850	824
Mo	< 300	44.1
Na	35,000	3.22E+05
Zr	< 260	1,000
IONS	µg/g	µg/g
Cl ⁻	< 2,300	2,870
RADIONUCLIDES	µCi/g	µCi/g
²⁴¹ Am	0.38	0.99
¹³⁷ Cs	410	400
^{239/240} Pu	0.060	0.087
⁹⁰ Sr	160	280
CARBON	µg C/g	µg C/g
TIC	6,260	20,700
TOC	71,700	26,900

Note:

¹If both the sodium/zirconium and potassium/nickel fusion methods were used for a given analyte, the larger value of the two was reported for the above comparison. For both values see Table B-1, Appendix B.

5.3 TANK WASTE PROFILE

Based on the sampling history of the tank, two distinct layers of waste were expected in tank 241-AN-102: an upper supernatant layer comprising nearly 92 percent of the waste volume; and a lower sludge layer. According to Brevick et al. (1995), the waste is entirely composed of concentrated complexant waste. However, it is possible that a minor heel of non-complexed waste exists below the concentrated complexant waste, because a 129-kL (34-kgal) heel of non-complexed slurry was left in the tank when it began receiving the concentrated complexant waste in 1984. The supernate consists entirely of concentrated complexant waste. Only the 1994 supernatant sampling provided enough data for a statistical analysis. Even this sampling event removed waste from only one riser; thus, the horizontal disposition of the waste cannot be determined. The 1994 event did sample from three different depths within the supernatant layer, so some estimate of the vertical disposition of the waste is possible within this layer.

A statistical procedure known as the analysis of variance (ANOVA) was conducted on the 1994 supernatant data to determine if there were any vertical differences in analyte concentrations (Herting 1994). 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 from each other. However, if a p-value is above 0.05, there is not sufficient evidence to conclude that the samples are significantly different from each other.

The results of the ANOVA tests indicated that none of the analytes listed in Appendix A showed significant differences as a function of depth (all p-values > 0.05). Also, the visual descriptions of the three samples described them as all being similar in appearance. This evidence strongly implies homogeneity within the supernatant layer.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER DATA

The HTCE values for tank 241-AN-102 are given in Table 5-7 along with the inventory estimates of the supernate and sludge layers based on analytical results (from Table 4-2). The HTCE estimate in column four is a single value for each analyte for the entire tank contents; separate estimates for the supernate and sludge were not provided. Thus, individual comparisons for the sludge and supernate layers were not possible. The analytical results are given separately in columns two and three; the total of columns two and three could be compared to column four.

Comparison of the HTCE estimates with the analytical values gives varied results. Some analytes are reasonably close in their estimates (chloride and sulfate) while others are very different (^{90}Sr and TOC). In general, most comparisons are of the same order of magnitude.

Table 5-7. Comparisons of Analytical Data to Historical Tank Content Estimate Values.

Analyte	1990 Sludge	1994 Supernate	BTCE
METALS	kg	kg	kg
Al	6,170	56,000	81,400
Ca	410	1,700	3,960
Cr	693	1,100	4,530
K	< 880	15,000	3,340
Na	1.18E+05	9.66E+05	5.62E+05
Ni	215	1,500	722
Zr	280	---	177
IONS	kg	kg	kg
Cl ⁻	1,040	15,000	14,700
F ⁻	< 450	7,500	3,990
OH ⁻	---	15,000	2.28E+05
NO ₃ ⁻	56,700	8.31E+05	5.33E+05
NO ₂ ⁻	19,900	3.14E+05	2.36E+05
PO ₄ ⁻³	1,530	18,000	42,500
SO ₄ ⁻²	13,100	53,000	64,700
RADIONUCLIDES	Cl	Cl	Cl
¹³⁷ Cs	1.44E+05	1.44E+06	8.33E+05
⁹⁰ Sr	85,500	2.77E+05	39,300
PHYSICAL PROPERTIES	g/mL	g/mL	g/mL
Density	1.50	1.40	1.27
CARBON	kg C	kg C	kg C
TIC	6,220	50,800	15,000
TOC	8,250	98,100	7,360

5.5 EVALUATION OF PROGRAM REQUIREMENTS

Tank 241-AN-102 is classified as a non-Watch List tank. Although DQOs were not applicable to any of the sampling events discussed in this report, the decision criteria listed in the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995) can still be

compared to one or more sets of analytical results to determine whether they meet current safety standards. This comparison is provided for informational purposes only, because none of the sampling events and analyses were performed to the DQO specifications. Consequently, analytical data found to be in compliance with the decision criteria may not necessarily be used to demonstrate a tank's safety.

The safety screening DQO establishes decision criteria or notification limits for concentrations of analytes of concern. The decision criteria are used to determine if a tank is safe, or if further investigation into the tank's safety is warranted. If results from one of the primary analyses exceed any of the decision criteria, the tank is not classified as "safe" and further analyses are conducted to assure the safety of the tank (Dukelow et al. 1995). An evaluation of operational issues was conducted using the waste compatibility DQO. Although the 1994 supernate sampling was not done for waste compatibility purposes, comparisons were still made between the analytical results and the DQO criteria.

5.5.1 Safety Evaluation

The primary analytical requirements identified in the safety screening DQO (Dukelow et al. 1995) are energetics, total alpha activity, and flammable gas concentration. Both the 1994 and 1990 sampling events failed to meet all the requirements of the safety screening data quality objective. The specific requirement that a vertical profile of the tank be obtained from two widely spaced risers was not met. One riser was sampled rather than two, making a horizontal assessment of the tank contents impossible.

The waste fuel content was determined by DSC. No DSC analyses were performed on the supernatant (1994 grab samples); DSC data were available from the 1990 sludge sampling event. During the 1990 analysis, DSC was performed on a centrifuged solids sample and a "crust" material found floating on top of the centrifuged liquid. No DSC was done on the centrifuged liquid itself. Exothermic reactions exceeding the -480 J/g safety screening limit (dry weight) were observed in both samples. The dry weight exothermic value for the centrifuged solids was -757 J/g, while the dry weight exothermic value for the crust material was -933 J/g. Such substantial exothermic behavior is not surprising considering that the concentrated complexant waste stored in the tank contains high levels of organic carbon and nitrate/nitrite concentrations. Both samples did have notable water contents as determined by TGA (30 weight percent for the centrifuged solids and 25 weight percent for the crust material).

Other indicators of fuel content are the TOC and cyanide concentrations. The organic safety program has established a dry weight TOC concentration limit of 3 weight percent, or 30,000 $\mu\text{g/g}$ (Turner et al. 1995). The TOC result (wet weight) for the 1994 supernatant sampling was 26,100 $\mu\text{g/mL}$, while the wet weight TOC result from the 1990 sludge sampling was 16,300 $\mu\text{g/g}$. The corresponding dry weights for these values were 37,200 $\mu\text{g/g}$ and 27,300 $\mu\text{g/g}$, respectively. The result for the 1994 sampling demonstrates that the

TOC concentration in the supernate exceeds the 3 weight percent organics DQO limit. A supernate sampling in 1984 found 24,200 $\mu\text{g/g}$ of TOC (wet weight). A conversion of this value to a dry weight was not possible due to the lack of a percent water value. However, assuming a percent water of 50 percent (figure obtained from the 1994 supernate sampling), the corresponding TOC concentration on a dry weight basis would be 48,400 $\mu\text{g/g}$. A DSC analysis of the supernate is recommended to determine the fuel content of the supernate and to ascertain the contribution of the TOC to any exothermic reactions.

Cyanide analyses were not performed on either tank sample. Based on the process history of the tank, there is no basis to expect the presence of cyanide.

The potential for criticality can be assessed from the total alpha data. None of the individual samples from the 1994 data contained alpha activity greater than 0.125 $\mu\text{Ci/g}$, and the mean result was 0.119 $\mu\text{Ci/g}$. This result was well below the safety screening DQO notification limit of 1 gram per liter (g/L), which is equivalent to 43.9 $\mu\text{Ci/g}$ using the supernate density of 1.40 g/ml. The sludge total alpha content can be estimated from the 1990 $^{239/240}\text{Pu}$ and ^{241}Am data. The data revealed that the total alpha concentration was far below 1 g/L, as only 0.655 $\mu\text{Ci/g}$ were present.

The flammability of the gas in the tank headspace is another safety screening consideration. No tank headspace flammability results are reported because tank headspace sampling and analysis were not performed.

Large amounts of moisture reduce the potential for propagating exothermic reactions in the waste. The percent water content of the waste was determined by TGA. Although the latest safety screening DQO does not contain decision criteria for weight percent water (Dukelow et al. 1995), the previous version specified that the waste must have at least 17 weight percent water (Babad et al. 1995). All samples from the 1994 sampling event and the 1990 sludge sampling event were above this 17 percent limit.

Table 5-8 presents a comparison of the safety screening criteria from Dukelow et al. (1995) and the recent analytical results. Again, because none of the sampling events were governed by the safety screening DQO, they may not have been performed to the DQO specifications; consequently, this comparison is provided for informational purposes only.

Table 5-8. Safety Screening Data Quality Objective Criteria.¹

Safety Issue	Primary Decision Variable	Decision Criteria Threshold	Supernate Analytical Result	Sludge Analytical Result
Ferrocyanide/ Organics	Total fuel content	-480 J/g ²	Not performed	Centrifuged solids = -757 J/g ²
				"Crust" material = -933 J/g ²
Criticality	Total alpha	1 g/L ³ (supernate = 43.9 μCi/g) (sludge = 41 μCi/g)	0.119 μCi/g	0.655 μCi/g
Flammable gas	Flammable gas	25% of the lower flammability limit	Not performed	Not performed

Notes:

¹Because neither of the sludge or supernate sampling events were governed by the safety screening DQO, this comparison is for informational purposes only.

²Dry weight basis.

³Although 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 ²³⁹Pu. The 43.9 μCi/g notification limit for the supernate is derived using the supernate density of 1.40 and the specific activity of ²³⁹Pu (0.0615 Ci/g). The notification limit for the sludge is determined similarly, using the sludge density of 1.5. The following equation displays the derivation method for the two notification limits:

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

Another factor in assessing the safety of tank waste is the heat generation and temperature of the waste. Heat is generated in waste tanks primarily from radioactive decay. Table 5-9 lists the primary heat-producing radionuclides and their data results (from Table 4-2) for the sludge and supernate layers in columns two and three, respectively. Column four sums the two values for a total, which is then converted into watts in column five. The estimate of 9,890 watts (33,800 Btu/hr) was below the operating specification limit of 20,500 watts (70,000 Btu/hr) (Harris 1994). As a comparison, the HTCE estimate was 4,170 watts (14,200 Btu/hr). Since an upper temperature limit is exhibited (Section 2.4.2), it may be concluded that any heat generated from radioactive sources throughout the year is dissipated.

Table 5-9. Tank 241-AN-102 Projected Heat Load.

Radionuclide	1990 Sludge Result	1994 Supernate Result	Total	Total
	Curies	Curies	Curies	Watts
¹³⁷ Cs	1.44E+05	1.44E+06	1.58E+06	7,460
⁹⁰ Sr	85,500	2.77E+05	3.63E+05	2,430
Total			1.94E+06	9,890

5.5.2 Operational Evaluation

The immediate objective of the 1994 sampling event was to determine whether the free hydroxide concentration was within the tank farm corrosion specification limit. The criterion for free hydroxide is set forth in the *Unclassified Operating Specifications for the 241-AN, AP, AW, AY, AZ, and SY Tank Farms* (Harris 1994), and is given in Table 5-10. This specification applies when the nitrate concentration exceeds 3.0 moles per liter (*M*), which is the case as shown by the 1994 supernate data ($\text{NO}_3^- = 3.57M$). The results indicated that for all three samples, the average free hydroxide concentration was 0.24*M*, which is below the criterion given in Table 5-10 and causes the tank to be out of its operational specification limit. The other two specifications listed in Table 5-9, $\text{OH}^- + \text{NO}_2^-$ and NO_3^- , both met their specification limits.

Table 5-10. Free Hydroxide Criterion for Nitrate Greater Than 3.0 Molarity.

Variable	Specification Limit	Analytical Result
OH^-	$0.3M \leq \text{OH}^- < 10M$	0.24 <i>M</i>
$\text{OH}^- + \text{NO}_2^-$	$\geq 1.2M$	2.05 <i>M</i>
NO_3^-	$\leq 5.5M$	3.57 <i>M</i>

Note:

M = moles per liter

According to the waste compatibility DQO (Fowler 1995), if a transfer involving tank 241-AN-102 is to occur, certain factors need to be investigated before proceeding. Because the waste stored in tank 241-AN-102 is concentrated complexant waste, it must remain segregated, with minimal mixing with non-complexant waste types. In addition to the corrosion problem already discussed, the specific gravity may be of concern. The specific gravity of the supernate is 1.40, which is greater than the 1.3 initial limit listed in the waste compatibility DQO (Fowler 1995). It also is approaching the 1.41 limit that the commingled waste must have after the transfer. The remaining waste compatibility DQO criteria were satisfied.

6.0 CONCLUSIONS AND RECOMMENDATIONS

Characterization of the waste stored in tank 241-AN-102 is based on two sampling and analysis events. Sludge composition and properties are taken from a three-segment core sample obtained in 1990. Concentration estimates for the supernate were gathered from three grab samples acquired in 1994. Data quality objectives were not applicable to either sampling event; however, a rudimentary safety assessment has been performed by comparing the analytical results with the decision criteria of the safety screening DQO (Dukelow et al. 1995).

Exothermic behavior exceeding the -480-J/g safety screening limit was observed in the waste material obtained during the 1990 sampling event. The DSC analyses were run on centrifuged fractions of the sludge core composite. The centrifuged solids contained an exothermic reaction of -757 J/g (dry weight), and a crust material found floating on the centrifuged liquid yielded an exothermic reaction of -933 J/g (dry weight). The water contents of these two samples were at least 25 weight percent. To corroborate these results, DSC and TGA measurements on the archived 1994 sludge sample are suggested.

Because the tank contains relatively high concentrations of organic carbon, a comparison was made between the TOC results and the TOC limit presented in the organic DQO (Turner et al. 1995). The TOC concentration in the supernate was determined to be 37,200 $\mu\text{g/g}$ (dry weight), exceeding the 3 weight percent limit (30,000 $\mu\text{g/g}$) specified in the DQO. A DSC analysis of the supernate is recommended to measure the energetics and determine the contribution of the TOC to any exothermic reactions. A TGA is also recommended for the supernate. The sludge TOC concentration (dry weight) was 27,300 $\mu\text{g/g}$.

The total alpha activity results were far below the safety screening limit. The calculated heat load from the radioactive decay of isotopes was 9,890 watts (33,800 Btu/hr), well within the operating specification limit of 20,500 watts (70,000 Btu/hr). Because sampling of the tank headspace has not been performed, the concentration of the tank gases is unknown.

Comparisons between the analytical data and the estimated concentrations taken from the Historical Tank Content Estimate produced mixed results. Many of the analytes agreed well. Others, such as TOC and ^{90}Sr , were present in quantities much higher than projected.

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APPENDIX A
ANALYTICAL RESULTS FOR 1994 GRAB SAMPLES

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A.1.0 INTRODUCTION

Table A-1 gives the analytical results for the 1994 supernate sampling event (Herting 1994). The first column contains the name of the analyte or physical characteristic. Column two lists the laboratory sample from which the analyte was measured. Sampling rationale, locations, and descriptions are contained in Section 3.0. No quality control data, such as matrix spikes, serial dilutions, or duplicate analyses, are listed. Columns three and four, "Result," list the mean of the primary and duplicate sample from the centrifuged supernate for a given sampling location. The third column lists the analyte concentration in mol/L or $\mu\text{Ci/L}$, while column four gives a converted concentration in units of $\mu\text{g/mL}$ or $\mu\text{Ci/mL}$. These conversions were performed for the metals, ions, and total inorganic carbon (TIC) using the following formula:

$$\text{Result} \left(\frac{\text{mol}}{\text{L}} \right) * \text{molecular wt.} \left(\frac{\text{g}}{\text{mol}} \right) * \left(\frac{1 \text{ L}}{1000 \text{ mL}} \right) * \left(\frac{1.0\text{E}+06 \mu\text{g}}{\text{g}} \right) = \text{Concentration} \left(\frac{\mu\text{g}}{\text{mL}} \right).$$

For the radionuclides, the conversion formula used was:

$$\text{Result} \left(\frac{\mu\text{Ci}}{\text{L}} \right) * \left(\frac{1 \text{ L}}{1,000 \text{ mL}} \right) = \text{Concentration} \left(\frac{\mu\text{Ci}}{\text{mL}} \right).$$

For total organic carbon (TOC) the conversion was:

$$\text{TOC Result} \left(\frac{\text{g}}{\text{L}} \right) * \left(\frac{1 \text{ L}}{1,000 \text{ mL}} \right) * \left(\frac{1.0\text{E}+06 \mu\text{g}}{\text{g}} \right) = \text{TOC Concentration} \left(\frac{\mu\text{g}}{\text{mL}} \right).$$

Column five lists the mean, which is a simple average of the individual results from the three different sampling locations. Column six, relative standard deviation (RSD), is a measure of variance defined as the standard deviation divided by the mean. This number is expressed as a percentage. The last column is the total projected inventory of tank 241-AN-102 at the time of sampling, and is calculated for the metals, ions, TIC, and TOC as follows:

$$\text{Concentration} \left(\frac{\mu\text{g}}{\text{mL}} \right) * \left(\frac{1 \text{ kg}}{1.0\text{E}+09 \mu\text{g}} \right) * \left(\frac{1,000 \text{ mL}}{\text{L}} \right) * (3.76\text{E}+06 \text{ L}) = \text{Inventory} (\text{kg}).$$

Total inventory for the radionuclides was calculated as follows:

$$\text{Concentration} \left(\frac{\mu\text{Ci}}{\text{mL}} \right) * \left(\frac{1,000 \text{ mL}}{\text{L}} \right) * \left(\frac{1 \text{ Ci}}{1.0\text{E}+06 \mu\text{Ci}} \right) * (3.76\text{E}+06 \text{ L}) = \text{Inventory} (\text{Ci}).$$

Total inventory was not applicable to the density, pH, and percent water data.

Table A-1. Analytical Summary from the 1994 Supernate Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Sample Identification	Result	Result	Mean	RSD ¹ (Mean)	Projected Inventory
METALS		mol/L	µg/mL	µg/mL	%	kg
Aluminum	102-AN-1/324	0.55	15,000	15,000	0.30	56,000
	102-AN-2/459	0.55	15,000			
	102-AN-4/622	0.56	15,000			
Calcium	102-AN-1/324	0.011	440	440	1.5	1,700
	102-AN-2/459	0.011	440			
	102-AN-4/622	0.011	440			
Chromium	102-AN-1/324	0.0055	290	290	0	1,100
	102-AN-2/459	0.0055	290			
	102-AN-4/622	0.0055	290			
Nickel	102-AN-1/324	0.006	400	400	2.8	1,500
	102-AN-2/459	0.007	400			
	102-AN-4/622	0.006	400			
Phosphorus	102-AN-1/324	0.052	1,600	1,600	0.6	6,000
	102-AN-2/459	0.053	1,600			
	102-AN-4/622	0.052	1,600			
Potassium	102-AN-1/324	0.089	3,500	3,900	6.7	15,000
	102-AN-2/459	0.098	3,800			
	102-AN-4/622	0.112	4,400			
Sodium	102-AN-1/324	11.8	271,000	2.57E+05	2.7	9.66E+05
	102-AN-2/459	10.7	246,000			
	102-AN-4/622	11.1	255,000			
Sulfur	102-AN-1/324	0.149	4,780	4,770	0.6	17,900
	102-AN-2/459	0.147	4,720			
	102-AN-4/622	0.150	4,820			
IONS		mol/L	µg/mL	µg/mL	%	kg
Chloride	102-AN-1/324	0.10	3,600	3,900	3.5	15,000
	102-AN-2/459	0.11	3,900			
	102-AN-4/622	0.12	4,300			

Table A-1. Analytical Summary from the 1994 Supernate Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Sample Identification	Result	Result	Mean	RSD ¹ (Mean)	Projected Inventory
IONS (Cont'd)		mol/L	µg/mL	µg/mL	%	kg
Fluoride	102-AN-1/324	0.10	1,900	2,000	4.3	7,500
	102-AN-2/459	0.10	1,900			
	102-AN-4/622	0.12	2,300			
Hydroxide	102-AN-1/324	0.24	4,100	4,100	0	15,000
	102-AN-2/459	0.24	4,100			
	102-AN-4/622	0.24	4,100			
Nitrate	102-AN-1/324	3.55	220,000	2.21E+05	0.4	8.31E+05
	102-AN-2/459	3.57	221,000			
	102-AN-4/622	3.60	223,000			
Nitrite	102-AN-1/324	1.79	82,300	83,400	0.6	3.14E+05
	102-AN-2/459	1.81	83,300			
	102-AN-4/622	1.84	84,600			
Phosphate	102-AN-1/324	0.050	4,800	4,900	2.9	18,000
	102-AN-2/459	0.051	4,800			
	102-AN-4/622	0.055	5,200			
Sulfate	102-AN-1/324	0.14	13,000	14,000	3.2	53,000
	102-AN-2/459	0.14	13,000			
	102-AN-4/622	0.16	15,000			
RADIONUCLIDES		µCi/L	µCi/mL	µCi/mL	%	Ci
¹³⁷ Cs	102-AN-1/324	382,000	382	382	0.5	1.44E+06
	102-AN-2/459	385,000	385			
	102-AN-4/622	378,000	378			
⁹⁰ Sr	102-AN-1/324	74,300	74.3	73.7	0.8	2.77E+05
	102-AN-2/459	74,300	74.3			
	102-AN-4/622	72,500	72.5			
Total Alpha	102-AN-1/324	170	0.170	0.167	0.9	628
	102-AN-2/459	165	0.165			
	102-AN-4/622	166	0.166			

Table A-1. Analytical Summary from the 1994 Supernate Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Sample Identification	Result	Result	Mean	RSD ¹ (Mean)	Projected Inventory
PHYSICAL PROPERTIES					%	
Density	102-AN-1/324	1.40		1.40 g/mL	0	
	102-AN-2/459	1.40				
	102-AN-4/622	1.40				
pH	102-AN-1/324	13.1		13.1	0.1	
	102-AN-2/459	13.1				
	102-AN-4/622	13.1				
% Water	102-AN-1/324	50.0		50.0	0	
	102-AN-2/459	50.0				
	102-AN-4/622	50.0				
CARBON			µg C/mL	µg C/mL	%	kg C
TIC	102-AN-1/324	1.15 mol/L	13,800	13,500	1.2	50,800
	102-AN-2/459	1.12 mol/L	13,400			
	102-AN-4/622	1.11 mol/L	13,300			
TOC	102-AN-1/324	27.1 g/L	27,100	26,100	1.9	98,100
	102-AN-2/459	25.0 g/L	25,000			
	102-AN-4/622	26.2 g/L	26,200			

Note:

¹RSDs (of the mean) were calculated on the original duplicate pairs using the original units (mol/L or µCi/L). The means of the original duplicate pairs are included in column 3. For the individual duplicate pair results, refer to Herting (1994).

APPENDIX B
ANALYTICAL RESULTS FOR 1990 CORE SAMPLE

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B.1.0 INTRODUCTION

The three tables given in this appendix list analytical data that were obtained from a 1990 sampling of the sludge layer in tank 241-AN-102 (Douglas 1996). The sludge recovered was composited and then centrifuged, creating two fractions: a centrifuged solids sample and a centrifuged liquid sample. These two samples were chemically analyzed separately, and the two separate sets of results are presented in Tables B-1 and B-2. Table B-3 combines the data from the first two tables to present an estimate of the overall sludge concentration and a total inventory for the sludge layer. Although small quantities of dilute non-complexed waste and water were added to the tank after the 1990 core sample was taken, they did not contribute substantially to the contents of the sludge layer.

B.1.1 CENTRIFUGED SOLIDS RESULTS

The best estimates for the chemical and radiochemical composition of the centrifuged solids portion of the sludge in tank 241-AN-102 were derived from the 1990 sampling event and are listed in Table B-1. These results are based on the centrifuged solids formed from the segments 1-3 slurry composite. The solids were prepared for analysis using two different fusions, thus providing measures of the elements masked by the containment crucibles and reagents used to solubilize the solids. The two fusion systems employed were NaOH with a zirconium crucible (NaOH/Zr) and KOH with a nickel crucible (KOH/Ni); the reported K and Ni concentrations were determined for the NaOH/Zr fusion and vice versa for the Na and Zr concentrations. The sample identification number for all analytes was 102AN-SOL. Table B-1 shows the analytes, the concentrations as originally reported in millimoles per gram (mmol/g), and the concentrations converted to $\mu\text{g/g}$.

B.1.2 CENTRIFUGED LIQUID RESULTS

Table B-2 lists the best estimates for the chemical and radiochemical composition of the centrifuged liquid portion of the sludge in tank 241-N-102. The centrifuged liquid portion is derived from the 1990 sampling event. This sample was centrifuged from the segments 1-3 slurry composite, and given the sample number 102AN-SUP. Table B-2 shows the analytes, the concentrations in mol/L or $\mu\text{Ci/mL}$, and the concentrations converted to $\mu\text{g/g}$ using the supernate density of 1.4 g/mL.

Table B-1. Centrifuged Solids Results from the 1990 Sampling Event for Tank 241-AN-102. (4 sheets)

Analyte	Concentration	Concentration
METALS	mmol/g ¹	µg/g ¹
Ag	< 1.8E-04	< 1.94
Al	0.54	14,600
As	< 0.0039	< 292
B	< 0.13	< 1,410
Ba	3.2E-04	43.9
Be	< 1.3E-04	< 1.17
Ca	0.030	1,210
Cd	< 2.4E-04	< 27.0
Ce	< 0.0051	< 715
Co	< 0.017	< 1,000
Cr	0.043	2,240
Cu	0.0014	89.0
Dy	< 1.5E-04	< 24.4
Fe	0.045	2,530
K	< 0.052 ³	< 2,030 ³
La	< 3.2E-04	< 44.5
Li	< 0.023	< 160
Mg	0.0084	204
Mn	0.015	824
Mo	4.6E-04	44.1
Na	14 ²	3.22E+05 ²
Nd	< 2.3E-04	< 33.2
Ni	0.0098 ³	575 ³
P	< 0.097	< 3,000
Pb	< 0.0018	< 373

Table B-1. Centrifuged Solids Results from the 1990 Sampling Event for Tank 241-AN-102. (4 sheets)

Analyte	Concentration	Concentration
METALS (Cont'd)	mmol/g ¹	µg/g ¹
Re	< 5.6E-04	< 104
Rh	< 0.0051	< 525
Ru	< 0.0039	< 394
Sb	< 0.0076	< 925
Se	< 0.014	< 1,110
Si	0.084	2,360
Sr	3.9E-04	34.2
Te	< 0.0027	< 345
Th	< 0.0020	< 464
Ti	< 9.4E-04	< 45.0
Tl	< 0.055	< 11,200
U ⁵	0.012	2,860
V	< 5.2E-04	< 26.5
Zn	0.0021	137
Zr	0.011 ²	1,000 ²
IONS	mmol/g	µg/g
Cr ⁶⁺	< 5.8E-04	< 30.2
Br ⁻	< 0.01	< 799
Cl ⁻	0.081	2,870
F ⁻	< 0.01 ⁵	< 190 ⁵
NO ₃ ⁻	2.89	1.79E+05
NO ₂ ⁻	1.37	63,000
PO ₄ ³⁻	0.038	3,610
SO ₄ ²⁻	0.46	44,200

Table B-1. Centrifuged Solids Results from the 1990 Sampling Event for Tank 241-AN-102. (4 sheets)

Analyte	Concentration	Concentration
RADIONUCLIDES		μCi/g^a
²⁴¹ Am		0.76 (0.99')
¹⁴ C		0.0019
¹⁴⁴ Ce		< 1.8
²⁴² Cm		Not Detected
^{243/244} Cm		0.059
⁶⁰ Co		0.43
¹³⁴ Cs		< 0.22
¹³⁷ Cs		400
¹⁵² Eu		< 0.17
¹⁵⁴ Eu		1.6
¹⁵⁵ Eu		1.7
¹⁵³ Gd		< 0.66
³ H		0.0035
⁹⁴ Nb		3.1E-04
²³⁷ Np		0.0017
²³⁸ Pu		0.029
^{239/240} Pu		0.087
¹⁰⁶ Ru		< 2.0
¹²⁵ Sb		< 1.3
⁷⁹ Se		0.0035
¹¹³ Sn		1.5
⁹⁰ Sr		280
⁹⁹ Tc		0.16
Total Beta		940

Table B-1. Centrifuged Solids Results from the 1990 Sampling Event for Tank 241-AN-102. (4 sheets)

Analyte	Concentration	Concentration
CARBON	mmol C/g	µg C/g
TIC	1.72	20,700
TOC	2.24	26,900
PHYSICAL PROPERTIES		
% Water by TGA		30
Density		1.7 g/mL

Notes:

¹Average for the two independent analyses of the solids prepared by the two separate fusions.

²Single analysis from the KOH fusion in a nickel crucible.

³Single analysis from the NaOH fusion in a zirconium crucible.

⁴Reported U concentration determined by fluorescence.

⁵Matrix interference noted.

⁶Concentrations of fission products and total beta content decay corrected to January 1, 1991.

⁷Second concentration determined using gamma energy analysis (GEA).

B.1.3 COMBINED SLUDGE RESULTS

Table B-3 combines the results presented in Tables B-1 and B-2 (shown in Table B-3 in columns two and three, respectively) in order to estimate the total concentration of a given analyte in the sludge layer (column four). A weighted mean was calculated using the results from each centrifuged fraction and multiplying by the respective weight percent that each fraction represented in the composite. The total calculated sludge concentration was derived by multiplying the centrifuged solids concentration by the value of weight percent centrifuged solids (55.4%). Similarly, the centrifuged liquid concentration was multiplied by 44.6%.

These two values were then added to arrive at the overall sludge number, listed in column four of Table B-3. For example, using the values for aluminum in Table B-3, column four would be derived by the following calculation:

$$\text{Sludge Aluminum Concentration} = \left(\frac{14,600 \mu\text{g}}{\text{g}} \right) * (0.554) + \left(\frac{9,250 \mu\text{g}}{\text{g}} \right) * (0.446).$$

The estimated total inventory for a given analyte with respect to the sludge layer is then given in column five. The total inventory estimates were calculated by the following equation for the metals and ions:

$$\text{Concentration} \left(\frac{\mu\text{g}}{\text{g}} \right) * \left(\frac{1 \text{ kg}}{1.0\text{E}+09 \mu\text{g}} \right) * (5.06\text{E}+08 \text{ g}) = \text{Sludge Inventory (kg)}.$$

For the radionuclides, the conversion formula used was:

$$\text{Concentration} \left(\frac{\mu\text{Ci}}{\text{g}} \right) * \left(\frac{1 \text{ Ci}}{1.0\text{E}+06 \mu\text{Ci}} \right) * (5.06\text{E}+08 \text{ g}) = \text{Sludge Inventory (Ci)}.$$

For TIC and TOC, the conversion formula used was:

$$\text{Concentration} \left(\frac{\mu\text{g C}}{\text{g}} \right) * \left(\frac{1 \text{ kg C}}{1.0\text{E}+09 \mu\text{g C}} \right) * (5.06\text{E}+08 \text{ g}) = \text{Sludge Inventory (kg C)}.$$

Table B-2. Centrifuged Liquid Results from the 1990 Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Concentration	Concentration
METALS	mol/L	µg/g
Ag	< 1.5E-05	< 1.16
Al	0.48	9,250
As	1.9E-04	10.2
B	0.0032	24.7
Ba	2.6E-05	2.55
Be	2.2E-05	0.142
Ca	0.011	315
Cd	4.5E-04	36.1
Ce	< 1.6E-04	< 16.0
Co	< 7.3E-04	< 30.7
Cr	0.0077	286
Cu	4.6E-04	20.9
Dy	< 8.6E-06	< 0.998
Fe	0.0053	211
K	0.049	1,370
La	1.1E-04	10.9
Li	< 1.6E-04	< 0.793
Mg	3.3E-04	5.73
Mn	0.0013	51.0
Mo	4.9E-04	33.6
Na	7.6	1.25E+05
Nd	2.1E-04	21.6
Ni	0.0057	239
P	0.052	1,150
Pb	9.9E-04	147
Re	< 8.6E-06	< 1.14
Rh	< 1.2E-04	< 8.82
Ru	2.2E-04	15.9
Sb	< 9.0E-05	< 7.83
Se	< 1.4E-04	< 7.90

Table B-2. Centrifuged Liquid Results from the 1990 Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Concentration	Concentration
METALS (Cont'd)	mol/L	µg/g
Si	0.0056	112
Sr	3.4E-05	2.13
Te	9.8E-05	8.93
Th	1.8E-04	29.8
Ti	< 2.5E-05	< 0.855
Tl	< 0.0016	< 234
U ¹	2.9E-05	4.93
V	< 2.2E-05	< 0.801
Zn	2.6E-04	12.1
Zr	1.6E-05	1.04
IONS	mol/L	µg/g
Cr ⁶⁺	< 8.6E-05	< 3.19
Br	< 0.0002	< 11.4
Cl ⁻	0.042	1,060
F ⁻	0.13	1,760
NO ₃ ⁻	0.66	28,800
NO ₂ ⁻	0.30	9,860
PO ₄ ³⁻	0.034 ²	2,310
SO ₄ ²⁻	0.045	3,090
AMMONIA	mol/L	µg/g
NH ₃	0.0026	31.6
RADIONUCLIDES	µCi/mL³	µCi/g³
²⁴¹ Am	0.089 (0.11 ⁴)	0.0636 (0.0786 ⁴)
¹⁴ C	7.1E-04	5.07E-04
¹⁴⁴ Ce	< 0.26	< 0.186
²⁴² Cm	5.0E-04	3.57E-04
^{243/244} Cm	0.0068	0.00486
⁶⁰ Co	0.14	0.100
¹³⁴ Cs	< 0.026	< 0.0186
¹³⁷ Cs	200	143

Table B-2. Centrifuged Liquid Results from the 1990 Sampling Event for Tank 241-AN-102. (3 sheets)

Analyte	Concentration	Concentration
RADIONUCLIDES (Cont'd)	$\mu\text{Ci/mL}^1$	$\mu\text{Ci/g}^2$
¹⁵² Eu	< 0.0076	< 0.00543
¹⁵⁴ Eu	0.30	0.214
¹⁵⁵ Eu	0.33	0.236
¹⁵³ Gd	< 0.10	< 0.0714
³ H	0.0018	0.00129
⁹⁴ Nb	6.5E-06	4.64E-06
²³⁷ Np	< 2.3E-06	< 1.64E-06
²³⁸ Pu	0.024	0.0171
^{239/240} Pu	0.070	0.0500
¹⁰⁶ Ru	< 0.28	< 0.200
¹²⁵ Sb	< 0.15	< 0.107
⁷⁹ Se	1.7E-04	1.21E-04
¹¹³ Sn	< 0.42	< 0.300
⁹⁰ Sr	45	32.1
⁹⁹ Tc	0.030	0.0214
Total Beta	2,700	1,930
CARBON	mol/L	$\mu\text{g C/g}$
TIC	0.23	1,970
TOC	0.37	3,170
PHYSICAL PROPERTIES		
Density	1.4 g/mL	

Notes:

¹Reported U concentration determined by fluorescence.

²Matrix interference noted.

³Concentrations of fission products and total beta content decay corrected to January 1, 1990.

⁴Second concentration determined using GEA.

Table B-3. Calculated Sludge Results for Tank 241-AN-102. (4 sheets)

Analyte	Centrifuged Solids Concentration	Centrifuged Liquid Concentration	Calculated Sludge Concentration	Total Projected Sludge Inventory
METALS	µg/g	µg/g	µg/g	kg
Ag	< 1.94	< 1.16	< 1.59	< 0.805
Al	14,600	9,250	12,200	6,170
As	< 292	10.2	< 166	< 84.0
B	< 1,410	24.7	< 792	< 401
Ba	43.9	2.55	25.5	12.9
Be	< 1.17	0.142	< 0.712	< 0.360
Ca	1,210	315	811	410
Cd	< 27.0	36.1	< 31.1	< 15.7
Ce	< 715	< 16.0	< 403	< 204
Co	< 1,000	< 30.7	< 568	< 287
Cr	2,240	286	1,370	693
Cu	89.0	20.9	58.6	29.7
Dy	< 24.4	< 0.998	< 14.0	< 7.08
Fe	2,530	211	1,500	759
K	< 2,030	1,370	< 1,740	< 880
La	< 44.5	10.9	< 29.5	< 14.9
Li	< 160	< 0.793	< 89.0	< 45.0
Mg	204	5.73	116	58.7
Mn	824	51.0	479	242
Mo	44.1	33.6	39.4	19.9
Na	3.22E+05	1.25E+05	2.34E+05	1.18E+05
Nd	< 33.2	21.6	< 28.0	< 14.2
Ni	575	239	425	215

Table B-3. Calculated Sludge Results for Tank 241-AN-102. (4 sheets)

Analyte	Centrifuged Solids Concentration	Centrifuged Liquid Concentration	Calculated Sludge Concentration	Total Projected Sludge Inventory
METALS (Cont'd)	µg/g	µg/g	µg/g	kg
P	< 3,000	1,150	< 2,170	< 1,100
Pb	< 373	147	< 272	< 138
Re	< 104	< 1.14	< 58.1	< 29.4
Rh	< 525	< 8.82	< 295	< 149
Ru	< 394	15.9	< 225	< 114
Sb	< 925	< 7.83	< 516	< 261
Se	< 1,110	< 7.90	< 618	< 313
Si	2,360	112	1,360	688
Sr	34.2	2.13	19.9	10.1
Te	< 345	8.93	< 195	< 98.7
Th	< 464	29.8	< 270	< 137
Ti	< 45.0	< 0.855	< 25.3	< 12.8
Tl	< 11,200	< 234	< 6,310	< 3,190
U	2,860	4.93	1,590	805
V	< 26.5	< 0.801	< 15.0	< 7.59
Zn	137	12.1	81.3	41.1
Zr	1,000	1.04	554	280
IONS	µg/g	µg/g	µg/g	kg
Br ⁻	< 799	< 11.4	< 448	< 227
Cl ⁻	2,870	1,060	2,060	1,040
Cr ⁶⁺	< 30.2	< 3.19	< 18.2	< 9.21
F ⁻	< 190	1,760	< 890	< 450
NO ₃ ⁻	1.79E+05	28,800	1.12E+05	56,700

Table B-3. Calculated Sludge Results for Tank 241-AN-102. (4 sheets)

Analyte	Centrifuged Solids Concentration	Centrifuged Liquid Concentration	Calculated Sludge Concentration	Total Projected Sludge Inventory
IONS (Cont'd)	µg/g	µg/g	µg/g	kg
NO ₂ ⁻	63,000	9,860	39,300	19,900
PO ₄ ⁻³	3,610	2,310	3,030	1,530
SO ₄ ⁻²	44,200	3,090	25,900	13,100
RADIONUCLIDES	µCi/g	µCi/g	µCi/g	Ci
²⁴¹ Am	0.99 ¹	0.0786 ¹	0.584	296
¹⁴ C	0.0019	5.07E-04	0.00128	0.648
¹⁴⁴ Ce	< 1.8	< 0.186	< 1.08	< 547
²⁴² Cm	Not Detected	3.57E-04	1.59E-04	0.0805
^{243/244} Cm	0.059	0.00486	0.0349	17.7
⁶⁰ Co	0.43	0.100	0.283	143
¹³⁴ Cs	< 0.22	< 0.0186	< 0.130	< 65.8
¹³⁷ Cs	400	143	285	1.44E+05
¹⁵² Eu	< 0.17	< 0.00543	< 0.0966	< 48.9
¹⁵⁴ Eu	1.6	0.214	0.982	497
¹⁵⁵ Eu	1.7	0.236	1.05	531
¹⁵³ Gd	< 0.66	< 0.0714	< 0.397	< 201
³ H	0.0035	0.00129	0.00251	1.27
⁹⁴ Nb	3.1E-04	4.64E-06	1.74E-04	0.0880
²³⁷ Np	0.0017	< 1.64E-06	< 9.43E-04	< 0.477
²³⁸ Pu	0.029	0.0171	0.0237	12.0
^{239/240} Pu	0.087	0.0500	0.0705	35.7
¹⁰⁶ Ru	< 2.0	< 0.200	< 1.20	< 607
¹²⁵ Sb	< 1.3	< 0.107	< 0.768	< 389

Table B-3. Calculated Sludge Results for Tank 241-AN-102. (4 sheets)

Analyte	Centrifuged Solids Concentration	Centrifuged Liquid Concentration	Calculated Sludge Concentration	Total Projected Sludge Inventory
RADIONUCLIDES (Cont'd)	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	Cl
⁷⁹ Se	0.0035	1.21E-04	0.00199	1.01
¹¹³ Sn	1.5	< 0.300	< 0.965	< 488
⁹⁰ Sr	280	32.1	169	85,500
⁹⁹ Tc	0.16	0.0214	0.0982	49.7
Total Beta	940	1,930	1,380	6.98E+05
PHYSICAL PROPERTIES				
% Water	40.3			
Density	1.7 g/mL	1.4 g/mL	1.5 ³ g/mL	
CARBON	$\mu\text{g C/g}$	$\mu\text{g C/g}$	$\mu\text{g C/g}$	kg C
TIC	20,700	1,970	12,300	6,220
TOC	26,900	3,170	16,300	8,250

Notes:

¹Based on GEA data in order to provide the most conservative estimate.

²The weight percent water for the sludge was taken from a percent solids determination on the core composite (done by drying at 105 °C [221 °F] for 24 hours). A weight percent water based on TGA data could not be calculated because TGA was not performed on the centrifuged liquid.

³The reported density value is from a density determination on the core composite; it was not calculated by taking a weighted mean from the centrifuged solids and centrifuged liquid results.

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APPENDIX C
HISTORICAL ANALYTICAL RESULTS

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C.1.0 INTRODUCTION

Appendix C contains analytical results from two historical samplings, one in 1984 and the other in 1989. The 1984 sampling included both a sludge and supernate sample. The sludge sample was taken from the bottom of the tank, and the supernate sample was obtained from 4.5 m (15 ft) above the tank bottom. The sample handling and preparation procedures are discussed in Section 3.4. Analyses were performed at the 222-S Laboratory, and the results were published in Bratzel (1985). Table C-1 contains the results from analyses of these samples. The second column displays the supernate results, while the third and fourth columns tabulate the results from centrifuged fractions of the sludge sample. The centrate is the centrifuged liquid, while the solids are centrifuged solids. The units used are those reported by the laboratory. Conversions of these units have been performed as needed throughout the document.

Table C-1. 1984 Analytical Results for Tank 241-AN-102. (2 sheets)

Analyte	Supernate	Centrate	Solids
METALS	mol/L	mol/L	weight %
Al	0.537	0.538	1.4
Ba	< 1.25E-04	1.33E-04	< 0.0021
Ca	0.0123	0.0203	0.11
Cd	5.03E-04	4.48E-04	< 0.0085
Cr	0.00796	0.0101	0.085
Cu	< 5.41E-04	< 4.33E-04	0.0030
Fe	0.00180	0.00468	0.071
K	0.0531	0.0468	---
La	< 0.00114	< 9.10E-04	< 0.0085
Mg	3.57E-04	0.00378	0.0090
Mn	0.0184	< 0.0155	< 0.085
Mo	< 0.00895	< 0.00718	< 0.030
Na	10.4	40.6 ¹	3.5
Nd	---	---	< 0.017
Ni	0.00643	0.0655	0.037
Pb	< 0.00114	0.00105	---
Si	< 0.00561	< 0.00450	< 0.0085
Sr	---	---	5.8E-04
Zn	< 4.22E-04	7.00E-04	< 0.0038
Zr	< 0.00173	< 0.00139	< 0.026
IONS	mol/L	mol/L	weight %
Cl ⁻	0.0949	0.0903	< 0.23
CO ₃ ²⁻	0.840	1.46	3.13
F ⁻	< 0.118	< 0.132	---
NO ₃ ⁻	3.61	3.38	---
NO ₂ ⁻	1.32	1.28	---
OH ⁻	0.201	0.648	---
PO ₄ ³⁻	0.0473	0.0480	---
SO ₄ ²⁻	0.114	0.0196	---

Table C-1. 1984 Analytical Results for Tank 241-AN-102. (2 sheets)

Analyte	Supernate	Centrate	Solids
RADIONUCLIDES	μCi/L	μCi/L	μCi/g
^{239/240} Pu	13.8	24.6	0.060 (Pu)
²⁴¹ Am	147	268	0.38 (Am)
⁶⁰ Co	551	635	---
¹³⁷ Cs	3.10E+05	4.48E+05	410
¹⁵⁴ Eu	850	---	---
¹⁵⁵ Eu	1,260	---	---
^{89/90} Sr	1.24E+05	1.01E+05	160
U	0.0247 g/L	< 0.107 g/L	0.090 wt %
PHYSICAL PROPERTIES			
Specific gravity	1.39	1.39	---
CARBON	g C/L	g C/L	weight %
TOC	33.7	29.8	7.17

Note:

¹The 40.6 mol/L sodium result for the centrate is considered unreliable and should not be used in any calculations or comparisons.

A supernate sample was obtained from tank 241-AN-102 in 1989. Other than the Sample Status Report included as an attachment to Herting (1994), no other information was available. The sample was depicted as being dark brown and aqueous with solids present. The Sample Status Report has been reproduced in Table C-2.

Table C-2. Analytical Results for 1989 Supernate Sample. (2 sheets)

Analyte	Concentration
METALS	mol/L
Al	0.460
B	0.00270
Ca	0.0101
Cr	0.00605
Cu	3.27E-04
Fe	0.00193
K	0.0407
La	1.26E-04
Na	7.65
Ni	0.00577
P	0.0497
ANIONS	mol/L
CO ₃ ²⁻	1.10
NO ₂ ⁻	1.36
NO ₃ ⁻	3.54
OH ⁻	0.445
RADIONUCLIDES	μCi/L
²⁴¹ Am	140
⁶⁰ Co	324
¹³⁷ Cs	4.00E+05
¹⁵⁴ Eu	539
¹⁵⁵ Eu	616
^{239/240} Pu	8.52
CARBON	g C/L
TOC	27.3

PROPERTIES	
Density	1.34 g/mL
pH	14

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DISTRIBUTION SHEET

G. H. Beeman	S7-71	X
S. F. Bobrowski	K7-28	X
N. G. Colton	K3-75	X
* J. R. Gormsen	K7-28	
S. A. Hartley	K5-12	X
J. G. Hill	K7-94	X
L. K. Holton	K9-73	X
G. J. Lumetta	P7-25	X
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