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Tank Characterization Report for Double-Shell Tank 241-AN-107

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

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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-107. This report supports the requirements of Tri-Party Agreement Milestone M-44-09.

Release Approval 8/15/96 Date

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Tank Characterization Report for Double-Shell Tank 241-AN-107

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EXECUTIVE SUMMARY

This characterization report summarizes the available information on the historical uses and the current status of double-shell tank 241-AN-107, and presents the analytical results of the February 1996 grab sampling and analysis project. This report supports the requirements of *Hanford Federal Facility Agreement and Consent Order* Milestone M-44-09 (Ecology et al. 1996).

Tank 241-AN-107 is one of seven double-shell tanks located in the Hanford Site 200 East Area 241-AN Tank Farm. The tank went into service in the third quarter of 1981 by receiving water. A second transfer of water was received during the second quarter of 1982. Dilute non-complexed waste was received from tank 241-AN-102 during the second quarter of 1983, followed by a transfer of complexed concentrate waste (the largest single contributor to the present inventory) from tank 241-AZ-102 during the fourth quarter of 1983. In addition to the complexed concentrate waste, small portions of an unknown waste, most likely non-complexed concentrate waste originating from the plutonium-uranium extraction (PUREX) plant, were received during this time. The tank received a small amount of unknown waste in the third quarter of 1984. From 1984 to July 1996, no additional transfers have occurred into or out of the tank.

A description of tank 241-AN-107 is presented in Table ES-1. The tank has an operating capacity of 4,390 kL (1,160 kgal), and at the sampling date of February 13, 1996 the tank contained a total of 4,005 kL (1,058 kgal) of waste. Of this total, 3,500 kL (924 kgal) are supernatant, and 507 kL (134 kgal) are sludge (Hanlon 1996).

This report summarizes the collection and analysis of the grab samples acquired in February 1996. The sampling event was performed to satisfy the requirements listed in *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). The sampling and analyses were performed in accordance with the *Tank 241-AN-107 Grab Sampling and Analysis Plan* (Gooey 1996). The sampling effort involved taking 11 grab samples, including a field blank, of the tank waste from two widely spaced risers. The purposes of the safety screening data quality objective (DQO) are to identify any unknown safety issues and to verify the non-Watch List status. The DQO requires analyses for energetics using differential scanning calorimetry (DSC), percent water by thermogravimetric analysis (TGA), total alpha activity through alpha proportional counting, and bulk density, in addition to a visual check for the presence of an organic layer. The DQO also requires a determination of the flammability of the tank headspace gases. To satisfy this requirement, vapor samples were taken prior to grab sampling, and the flammability was measured as a percentage of the lower flammability limit (LFL) using a combustible gas meter.

Table ES-1. Description and Status of Tank 241-AN-107.

TANK DESCRIPTION	
Type	Double-shell
Constructed	1979-1980
In-service	3rd quarter 1981
Diameter	23 m (75 ft)
Operating depth	10.7 m (35.2 ft)
Capacity	4,390 kL (1,160 kgal)
Bottom shape	Flat
Ventilation	Active
TANK STATUS	
Waste classification	Complexed concentrate
Total waste volume (March 30, 1996)[1]	4,005 kL (1,058 kgal)
Sludge volume	507 kL (134 kgal)
Drainable interstitial liquid volume	34 kL (9 kgal)
Supernatant volume[2]	3,500 kL (924 kgal)
Waste surface level (February 13, 1996)	977 cm (384.8 in.)
Temperature (1/90 - 4/96)	29 °C (85 °F) to 39 °C (103 °F)
Integrity	Sound
Watch List	None
SAMPLING DATES	
Grab samples & tank headspace flammability	February 1996
Vapor samples	December 1994
Grab samples	February 1993, and May, 1994
SERVICE STATUS	
Active	Third quarter 1981 to present

Notes:

[1]No transfers have occurred since 1984.

[2]This is an active tank; waste volume may not represent future tank content.

Comparisons were made between the analytical results and the decision criteria thresholds defined in the safety screening DQO. All of the following results for DSC and total organic carbon (TOC) are based on a dry weight. All but one of the DSC measurements exhibited changes in enthalpy in excess of the decision criteria threshold of -480 J/g (dry weight basis). The largest magnitude sample-duplicate average DSC result was -1,304 J/g. The largest magnitude upper 95 percent confidence interval limit on the mean was -1,985 J/g. Because the DSC scans exhibited results above the threshold, TOC was measured as a secondary analyte and the reactive system screening test (RSST) was performed on the sludge sample with the highest DSC results. All TOC measurements were above the decision criteria threshold of 30,000 μg C/g (dry weight basis). The highest sample-duplicate result for the TOC measurement was 87,400 μg C/g, and the highest upper 95 percent confidence limit on the mean was 88,600 μg C/g (dry weight basis). The RSST analysis exhibited self-heating, complex, multi-step behavior, indicating the presence of organic compounds with widely varying stability toward molten nitrate. However, RSST results did not indicate rapidly self-propagating reaction conditions within the waste. Mean percent water values by thermogravimetric analyses (TGA) were 49.9 weight percent for the supernatant samples and 45.5 weight percent for the sludge samples. The mean total alpha activity results for the supernatant and sludge samples were 0.799 μCi/mL and 0.989 μCi/g, respectively. The highest 95 percent upper confidence interval limit on the mean for total alpha was 3.44 μCi/g, from sample S96T001445. All total alpha measurements were well below the DQO notification limit of 1 g/L, or 61.5 μCi/mL for the supernatant and 42.4 μCi/g for the sludge. The flammability of the tank 241-AN-107 headspace was measured at zero percent of the LFL (Esch 1996).

Table ES-2. Chemical Data Summary for Tank 241-AN-107.[1]

Analyte	Liquids			Solids		
	Overall Mean	RSD (Mean)	Projected Inventory	Overall Mean	RSD (Mean)	Projected Inventory
RADIONUCLIDES	μCi/mL	%	Ci	μCi/g	%	Ci
Total alpha	0.799	2.88	2,800	0.989	14.2	727
CARBON	μg C/mL	%	kg C	μg C/g	%	kg C
Total organic carbon	55,700	2.39	1.95E+05	42,700	8.34	31,400
PHYSICAL PROPERTIES		%	kg		%	kg
Weight percent water	49.9	0.347	2.41E+06	45.5	3.00	3.34E+05
Bulk density (g/mL)	1.38	0.605	---	1.45	2.47	---

Note:
[1]Esch (1996)

A summary of the analytical data, including relative standard deviations (RSD) and projected inventories, is presented in Table ES-2.

A tank heat load calculated based on analytical data found in Herting (1994) was 8,060 W (27,500 Btu/hr). The Historical Tank Content Estimate (HTCE) prediction was 7,500 W (25,600 Btu/hr) (Agnew et al. 1996a), while the heat load estimate by Kummerer (1994) was 7,910 W (27,000 Btu/hr). These estimates show good agreement and are well below the design specification of 20,500 W (70,000 Btu/hr) for the 241-AN tank farm (Harris 1992).

Waste stored at the Hanford Site is maintained in an alkaline state to minimize general and stress corrosion. Tank 241-AN-107 has a history of depletion of the caustic in the waste. At present, the concentration of caustic in the waste poses no general corrosion problems.

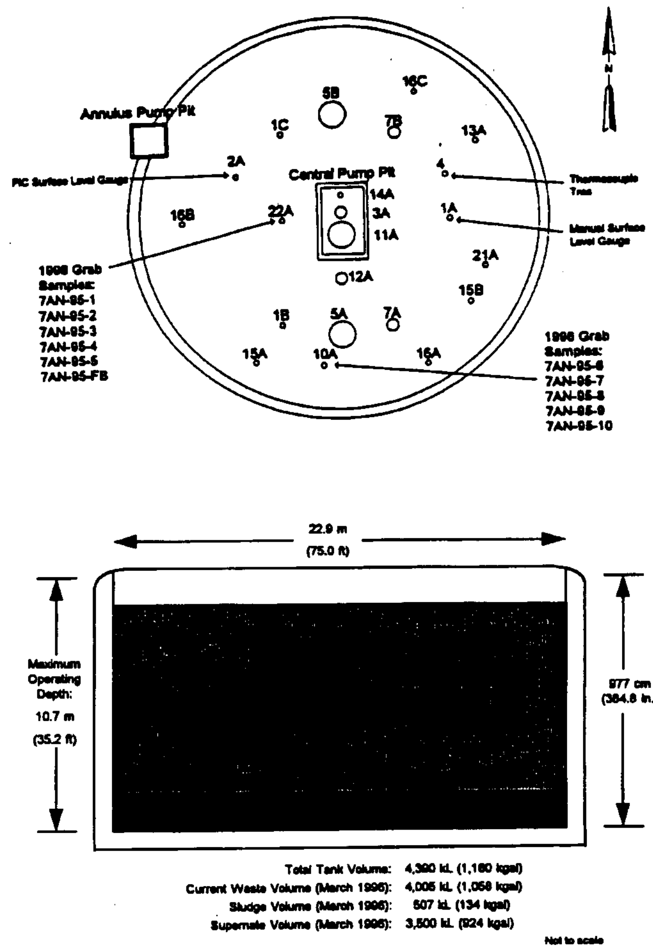
However, at the current levels of caustic, stress corrosion and failure could occur. This situation is being addressed in a two-phase plan. Phase 1 would add 19 M sodium hydroxide to the supernatant only. Phase 2 would thoroughly mix the sludge and supernatant layers as the sodium hydroxide was being added (Carothers 1992).

A profile of tank 241-AN-107 is provided in Figure ES-1.

The analytical results show that the waste exhibits total fuel content resulting in changes in enthalpy in excess of -480 J/g and TOC greater than 3 weight percent. However, the high moisture content places the tank in the "conditionally safe" category. The moisture in the waste must be maintained at greater than 17 weight percent in order to ensure that the tank remains in the "conditionally safe" category (Turner et al. 1995).

Finally, all analytical results indicate the feasibility of successful retrieval and disposal of the waste. However, the caustic depletion issue warrants further sampling or evaluation. Measures must also be taken to ensure that the moisture in the tank remains within the safety limits.

Figure ES-1. Profile of Tank 241-AN-107.



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LIST OF TERMS

ANOVA	analysis of variance
Btu/hr	British thermal units per hour
Ci	curies
Ci/L	curies per liter
cm	centimeters
DQO	data quality objective
DSC	differential scanning calorimetry
ECN	Engineering Change Notice
FIC	Food Instrument Corporation
ft	feet
g/L	grams per liter
g/mL	grams per milliliter
g/mole	grams per mole
HDW	Hanford Defined Waste
HTCE	Historical Tank Content Estimate
in.	inches
J/g	joules per gram
kg	kilograms
kg C	kilograms carbon
kgal	kilogallons
kL	kiloliters
LEL	lower explosive limit
LFL	lower flammability limit
m	meters
M	moles per liter
mg	milligrams
mL	milliliters
mm	millimeters
mole/g	moles per gram
N/A	not applicable
NFPA	National Fire Protection Association
ppm	parts per million
PUREX	plutonium-uranium extraction
QC	quality control
R/hr	roentgens per hour
RPD	relative percent difference

LIST OF TERMS (Continued)

RPM	revolutions per minute
RSD	relative standard deviation
RSST	reactive system screening tool
SACS	Surveillance Analysis Computer System
SAP	sampling and analysis plan
SMM	supernatant mixing model
SpG	specific gravity
TGA	thermogravimetric analysis
TLM	Tank Layer Model
TOC	total organic carbon
W	watts
WHC	Westinghouse Hanford Company
WSTRS	Waste Status and Transaction Record Summary
wt%	weight percent
°C	degrees Celsius
°C/min	degrees Celsius per minute
°F	degrees Fahrenheit
μCi/g	microcuries per gram
μCi/L	microcuries per liter
μCi/mL	microcuries per milliliter
μg C/g	micrograms carbon per gram
μg C/mL	micrograms carbon per milliliter
μg/g	micrograms per gram
ΔH	change in enthalpy

1.0 INTRODUCTION

This tank characterization report presents an overview of double-shell tank 241-AN-107 and its waste components. It provides estimated concentrations and inventories for the waste constituents based on the latest sampling and analysis activities, in combination with background tank information. Tank 241-AN-107 was grab sampled in February 1996 in accordance with the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995) and the *Tank 241-AN-107 Grab Sampling and Analysis Plan* (Gooey 1996).

Tank 241-AN-107 contains concentrated complexant waste, which must be segregated from non-complexant wastes. Thus, although the volume may change, it is unlikely that the composition will change substantially, with the exception of hydroxide. Although the tank is in service, it has not received waste since 1984. The concentration and inventory values reported in this document reflect the best estimates based on the most recent analytical data. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996)

1.1 PURPOSE

This report summarizes information about the use and contents of tank 241-AN-107. Where possible, this information will be used to assess issues associated with safety, operational, environmental, and process activities. This report also provides a reference point for more detailed information about tank 241-AN-107.

1.2 SCOPE

The February 1996 grab sampling event for tank 241-AN-107 supported the evaluation of the tank waste according to the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). The purpose of the safety screening DQO is to identify any unknown safety issues and to verify the non-Watch List status. The primary safety screening analyses, including DSC (to evaluate fuel level and energetics), TGA (to determine the moisture content), total alpha activity analysis (to evaluate the criticality potential), specific gravity or density, and a visual check for an organic layer (liquids only), were performed on the samples. Combustible gas meter readings of the tank headspace vapors were also taken to address flammability concerns. Because of high energetics, TOC was also analyzed.

Three sludge samples and two supernatant samples were taken from tank 241-AN-107 in May 1994 to support characterization of the waste in the tank, and to evaluate the depletion of the caustic. Also, four vapor samples from tank 241-AN-107 were obtained through the ventilation access duct on December 21, 1994. After a purge of the tubing and a leak check of the sample system were performed, a sample of ambient air was drawn to establish background levels. The samples were collected through flow-through canisters. The samples were received by the Inorganic Mass Spectrometry Laboratory at the Pacific Northwest National Laboratory on December 29, 1994.

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-AN-107 based on historical information. The first part details the current condition of the tank. The next part contains discussions of the tank's design, transfer history, and the process sources that contributed to the tank waste, including an estimate of the current contents based on the process history. Events that may be related to tank safety issues, such as potentially hazardous tank contents or off-normal operating temperatures, are included. The final part summarizes available surveillance data for the tank. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the solid layers of the tank. Temperature data are provided to evaluate the heat-generating characteristics of the waste.

2.1 TANK STATUS

As of March 31, 1996, tank 241-AN-107 contained an estimated 4,005 kL (1,058 kgal) of waste classified as complexed concentrate (Hanlon 1996). Liquid waste volume was estimated using a combination of Food Instrument Corporation (FIC) and manual tape surface level gauges. Solid waste volume was last estimated using a sludge level measurement device on August 22, 1989. The amounts of various waste phases existing in the tank are presented in Table 2-1.

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

Waste Form	Estimated Volume[2,3]	
	kL	kgal
Total waste	4,005	1,058
Supernatant liquid	3,500	924
Sludge	507	134
Saltcake	0	0

Notes:

[1]Hanlon (1996)

[2]Volumes in kL are not additive due to rounding error in conversion from kgal.

[3]Tank 241-AN-107 is an active tank, therefore the volume will change when transfers occur.

Tank 241-AN-107 is categorized as sound and is not on any Watch List. This tank is actively ventilated. All monitoring systems were in compliance with documented standards as of March 31, 1996 (Hanlon 1996).

2.2 TANK DESIGN AND BACKGROUND

The 241-AN Tank Farm was constructed from 1979 to 1980 in the 200 East Area. The 241-AN Tank Farm contains seven double-shell tanks. These tanks have a capacity of 4,390 kL (1,160 kgal), a diameter of 23 m (75 ft), and an operating depth of 10.7 m (35.2 ft). Tank 241-AN-107 began receiving waste in September 1983. The tanks in the 241-AN tank farm were designed to hold boiling waste with a maximum design temperature of 177 °C (350°F) (Brevick et al. 1995, Leach and Stahl 1993).

Tank 241-AN-107 was constructed with a primary carbon steel liner (heat-treated and stress-relieved), a secondary carbon steel liner (not heat-treated), and a reinforced concrete shell. The bottom of the primary liner is 13 mm (0.5 in.) thick, the lower portion of the sides is 19 mm (0.75 in.) thick, the upper portion of the sides is 13 mm (0.5 in.) thick, and the dome liner is 9.5 mm (0.375 in.) thick. The secondary liner is 9.5 mm (0.375 in.) thick. The concrete walls are 46.0 cm (1.5 ft) thick and the dome is 38.0 cm (1.25 ft) thick. The tank has a flat bottom. The bottoms of the primary and secondary liners are separated by an insulating concrete layer. There is a grid of drain slots in the concrete foundation beneath the secondary steel liner. The grid's function is to collect any waste that may leak from the tank and divert it to the leak detection well.

Tank 241-AN-107 has 43 risers ranging in diameter from 100 mm (4 in.) to 1.1 m (42 in.), that provide access to the tank. The tank has 37 risers that provide access to the annulus. Table 2-2 shows numbers, diameters, and descriptions of the risers (annular risers not included). A plan view that depicts the riser configuration is shown as Figure 2-1. Risers 10A, 15A, and 21A (each 100 mm [4 in.] in diameter) and risers 7B and 12A (each 300 mm [12 in.] in diameter) are classified as spares (Lipnicki 1996). A tank cross-section showing the approximate waste level along with a schematic of the tank equipment is in Figure 2-2.

Table 2-2. Tank 241-AN-107 Risers.[1,2,3,4] (2 sheets)

New Riser Number[5]	Old Riser Number	Diameter (inches)	Description and Comments[6]
102	1A	4	Sludge measurement port
103	1B	4	Sludge measurement port, P/CP (12-in. cover)
101	1C	4	Sludge measurement port
104	2A	4	Liquid level, level indicating transmitter
105	3A	12	Supernatant pump, central pump pit
106	4	4	Thermocouple tree
108	5A	42	Manhole
107	5B	42	Manhole
112	7A	12	Tank ventilation
111	7B	12	Spare
125	10A	4	Spare
126	11A	42	Slurry distributor, central pump pit
127	12A	12	Observation port, spare
128	13A	4	Tank pressure
129	14A	4	Supernatant return, central pump pit, dropleg nozzle
131	15A	4	Spare, P/CP (12-in. cover)
130	15B	4	High liquid level sensor
134	16A	4	Sludge measurement port
132	16B	4	Sludge measurement port, P/CP (12-in. cover)
133	16C	4	Sludge measurement port
155	21A	4	Spare
156	22A	4	Sludge measurement port
165	24A	6	Air lift circulator (12-in. cover)
166	24B	6	Air lift circulator (12-in. cover)
167	24C	6	Air lift circulator (12-in. cover)
160	24D	6	Air lift circulator (12-in. cover)
162	24E	6	Air lift circulator (12-in. cover)
163	24F	6	Air lift circulator (12-in. cover)
164	24G	6	Air lift circulator (12-in. cover)
161	25A	6	Air lift circulator (12-in. cover)

Table 2-2. Tank 241-AN-107 Risers.[1,2,3,4] (2 sheets)

New Riser Number[5]	Old Riser Number	Diameter (inches)	Description and Comments[6]
178	25B	6	Air lift circulator (12-in. cover)
179	25C	6	Air lift circulator (12-in. cover)
180	25D	6	Air lift circulator (12-in. cover)
168	25E	6	Air lift circulator (12-in. cover)
169	25F	6	Air lift circulator (12-in. cover)
170	25G	6	Air lift circulator (12-in. cover)
171	25H	6	Air lift circulator (12-in. cover)
172	25J	6	Air lift circulator (12-in. cover)
173	25K	6	Air lift circulator (12-in. cover)
174	25L	6	Air lift circulator (12-in. cover) (Was 25E, ECN #613266, 1/20/95)
175	25M	6	Air lift circulator (12-in. cover)
176	25N	6	Air lift circulator (12-in. cover)
177	25P	6	Air lift circulator (12-in. cover)

Notes:

P/CP = riser recessed below a concrete pad, with access plate at grade.

[1]Salazar (1994)

[2]WHC (1992)

[3]WHC (1995a)

[4]WHC (1995b)

[5]Riser numbers changed by Engineering Change Notice (ECN) 613266, dated 1/20/95, to the referenced drawings.

[6]In the case of a discrepancy between the documents and the drawing, the drawing took precedence.

Figure 2-1. Riser Configuration for Tank 241-AN-107.

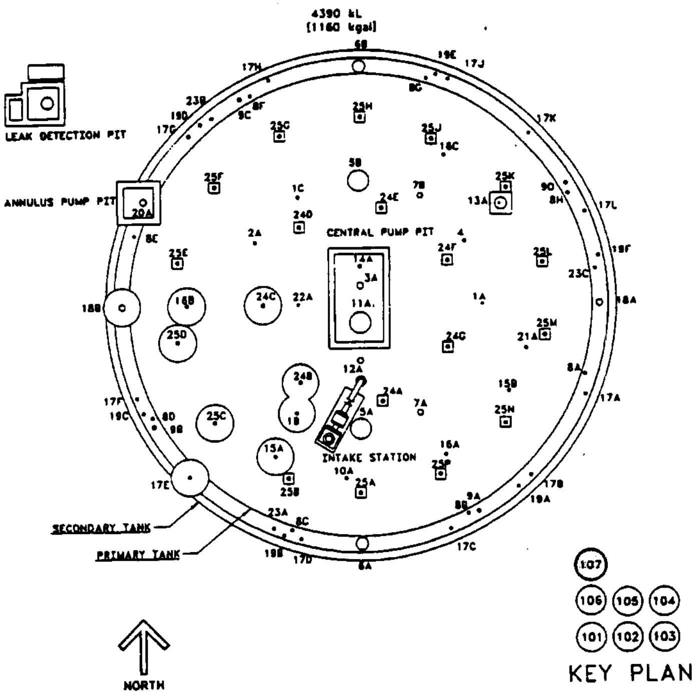
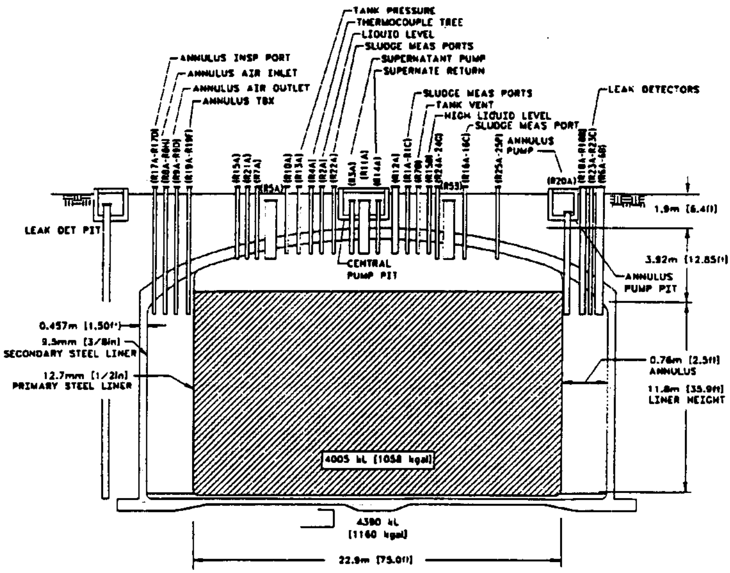


Figure 2-2. Tank 241-AN-107 Cross-Section.



2.3 PROCESS KNOWLEDGE

These sections present the waste transfer history of tank 241-AN-107. The major waste receipts and transfers involving tank 241-AN-107 are presented in Section 2.3.1, Table 2-3. Section 2.3.2 is from a database developed for waste volume projections and the data have not been validated. Section 2.3.2 is included to provide comment on transfers occurring after January 1, 1994, and describes the historical estimation of the tank's waste contents.

2.3.1 Waste Transfer History

Tank 241-AN-107 first received water in the third quarter of 1981 for integrity testing. A second addition of water in the second quarter of 1982 completed the testing phase for the tank. Dilute non-complexed waste was transferred into tank 241-AN-107 from tank 241-AN-102 during the second quarter of 1983. Another transfer of waste occurred during the fourth quarter of 1983 with an addition of complexed concentrate waste from tank 241-AZ-102. Records indicate that a portion of the waste transferred could have had a small amount of non-complexed concentrate waste originating from plutonium-uranium extraction (PUREX) miscellaneous streams.

Tank 241-AN-107 received 30 kL (8 kgal) of an unknown waste type in the third quarter of 1984. Tank 241-AN-107 did not receive or transfer any more waste, though several unknown losses and gains were noted in the historical records, totaling a loss of 140 kL (37 kgal) of waste from the tank. Since the average loss due to evaporation is approximately 50,000 gal/yr, most of the losses are most likely due to evaporation.

Table 2-3. Summary of Tank 241-AN-107 Waste Received History.[1,2]

Transfer Source	Waste Type Received and Removed	Time Period	Estimated Waste Volume	
			kL	kgal
241-AN-102	Dilute non-complexed	1983	1,760	465
241-AZ-102	Complexed concentrate	1983	2,529	668
Unknown	Waste removed	1983-1984	-114	-30
		1984-1996	-140	-37

Notes:

[1]Agnew et al. (1996b)

[2]Waste volumes and types are best estimates based on historical data.

2.3.2 Historical Estimation of Tank Contents

The following is an estimate of the contents for tank 241-AN-107 based on historical transfer data. The historical data used for the estimate is from the *Waste Status and Transaction Record Summary for the Southeast Quadrant* (WSTRS) (Agnew et al. 1996b) and the *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev. 3* (Agnew et al. 1996a). The Hanford Defined Waste (HDW) Model Rev. 3 document contains the HDW list, the Supernatant Mixing Model (SMM) the Tank Layer Model (TLM), and the Historical Tank Content Estimate (HTCE). The WSTRS is a compilation of available waste transfer and volume status data. The HDW provides the assumed typical compositions for 50 separate waste types. In most cases, the available data are incomplete, reducing the reliability of the transfer data and the derived modeling results. The SMM is a model installed on a spreadsheet and describes the supernatant and concentrates within each of the tanks. The SMM uses information from both the WSTRS and the TLM. The TLM, using the WSTRS 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 estimates that require further evaluation using analytical data.

Based on the model results, tank 241-AN-107 contained 4,012 kL (1,060 kgal) of waste, of which 507 kL (134 kgal) was concentrated supernatant solids. Figure 2-3 shows a graphical representation of the estimated waste type and volume for each tank layer. Presently, modeling data do not exist on the exact contents of the supernatant or concentrated supernatant solids layers. Table 2-4 shows an estimate of the expected waste constituents and concentrations.

2.4 SURVEILLANCE DATA

Tank 241-AN-107 surveillance consists of surface level measurements (liquid and solid), temperature monitoring inside the tank (waste and headspace), and leak detection well monitoring for radioactive liquids outside the primary tank. The data provide the basis for determining tank integrity.

Solid surface level measurements will provide an indication of the physical changes in and consistencies of the solid layers of a tank. However, due to the nature of the waste tank 241-AN-107 received, solids are either suspended or dissolved and are not directly measured as a part of the total waste volume. Leak detection systems within the annulus of the tank will detect leaks from the primary tank. These systems indicate that the tank is sound and not leaking.

Figure 2-3. Tank Layer Model for Tank 241-AN-107.

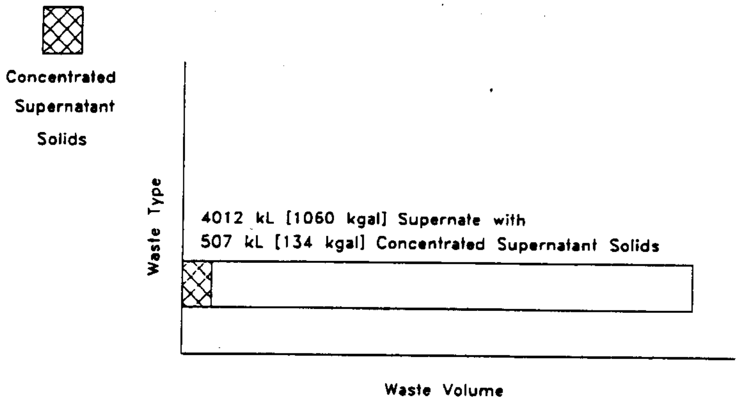


Table 2-4. Tank 241-AN-107 Inventory Estimate.[1,2,3] (2 sheets)

Total Inventory Estimate			
Physical Properties			
Total solid waste	6.21E+06 kg (1,060 kgal)		
Heat load	7,500 W (25,600 Btu/hr)		
Bulk density	1.54 (g/mL)		
Water wt%	39.0		
Total Organic Carbon wt% Carbon (wet)	1.48		
Chemical Constituents	**M**	**ppm**	**kg**
Na^+	11.4	1.70E+05	1.06E+06
Al^{3+}	1.68	29,400	1.82E+05
Fe^{3+} (total Fe)	0.00571	206	1,280
Cr^{3+}	0.0430	1,450	9,000
Bi^{3+}	0.00122	166	1,030
La^{3+}	1.22E-05	1.10	6.81
Hg^{2+}	1.02E-05	1.33	8.24
Zr (as $ZrO(OH)_2$)	5.85E-04	34.6	215
Pb^{2+}	0.00147	197	1,230
Ni^{2+}	0.00469	178	1,110
Sr^{2+}	4.06E-06	0.231	1.43
Mn^{4+}	0.00494	176	1,090
Ca^{2+}	0.0258	670	4,160
K^+	0.0582	1,470	9,150
OH^-	7.31	80,500	5.00E+05
NO_3^-	4.21	1.69E+05	1.05E+06
NO_2^-	2.32	69,000	4.29E+05
CO_3^{2-}	0.542	21,100	1.31E+05
PO_4^{3-}	0.0981	6,030	37,500
SO_4^{2-}	0.287	17,900	1.11E+05
Si (as SiO_3^{2-})	0.0691	1,260	7,820
F^-	0.0766	943	5,860
Cl^-	0.207	4,740	29,500
citrate	0.0343	4,210	26,100

Table 2-4. Tank 241-AN-107 Inventory Estimate.[1,2,3] (2 sheets)

Chemical Constituents (cont'd)	M	ppm	kg
$EDTA^{4-}$	0.0367	6,850	42,500
$HEDTA^{3-}$	0.0638	11,300	70,400
glycolate	0.142	6,910	42,900
acetate	0.0304	1,160	7,220
oxalate	1.04E-05	0.595	3.69
DBP	0.0291	3,030	18,800
butanol	0.0291	1,400	8,670
NH_3	0.0320	352	2,190
$Fe(CN)_6^{4-}$	0	0	0
Radiological Constituents			
Pu	---	0.0443 (μCi/g)	4.58 (kg)
U	0.00929 (M)	1,430 (μg/g)	8,900 (kg)
Cs	0.260 (Ci/L)	168 (μCi/g)	1.05E+06 (Ci)
Sr	0.0963 (Ci/L)	62.4 (μCi/g)	3.87E+05 (Ci)

Notes:

[1]Agnew et al. (1996a)

[2]The HTCE predictions have not been validated and should be used with caution.

[3]Differences appear to exist among the inventory above and the inventories calculated from the two sets of concentrations.

2.4.1 Surface Level Readings

The waste surface level is monitored with a Food Instrument Corporation (FIC) gauge and a manual tape. The surface level reading from the automatic FIC gauge on February 13, 1996 was 9.77 m (384.7 in.), which equals approximately 4,005 kL (1,058 kgal). The baseline for tank 241-AN-107 is 9.76 m (384.5 in.). A graphical representation of the volume measurements is presented as a level history graph in Figure 2-4.

2.4.2 Internal Tank Temperatures

Temperature data for tank 241-AN-107 are recorded by 18 thermocouples on one thermocouple tree located in riser 4A. Temperature data from the Computer Automated Surveillance System, recorded from July 1983 to June 1991, are available for all 18 thermocouples. Temperature data from the Surveillance Analysis Computer System (SACS), recorded from January 1990 to April 1996, are available for 12 thermocouples. There are several small breaks in the temperature data. The average temperature of all SACS data for tank 241-AN-107 from January 1990 to April 1996 was 35 °C (95 °F), the minimum temperature was 29 °C (85 °F), and the maximum temperature was 39 °C (103 °F). Over the last year, the average temperature was 34.9 °C (94.9 °F), the minimum temperature was 30.1 °C (86.18 °F), and the maximum temperature was 39.3 °C (102.74 °F). A weekly high temperature graph, generated from the SACS data, can be found in Figure 2-5. On April 22, 1996 the minimum temperature was 31.9 °C (89.42 °F) on thermocouple 18 and the maximum was 37.1 °C (98.78 °F) on thermocouple 2. Plots of the individual thermocouple readings for tank 241-AN-107 can be found in the supporting documents for the HTCE (Brevick et al. 1995).

2.4.3 Tank 241-AN-107 Photographs

The 1988 photographic montage of the tank 241-AN-107 interior lacks clarity and a good definition of the waste surface color. It is dark and too hazy to determine the waste color with any confidence. A dark brown liquid appears to be on the surface, but this could be due to the lighting or underlying solid material. When the photographs were taken, the tank contained approximately 4,122 kL (1,089 kgal) of waste, which equals approximately 10 m (396 in.).

Figure 2-4. Tank 241-AN-107 Level History.

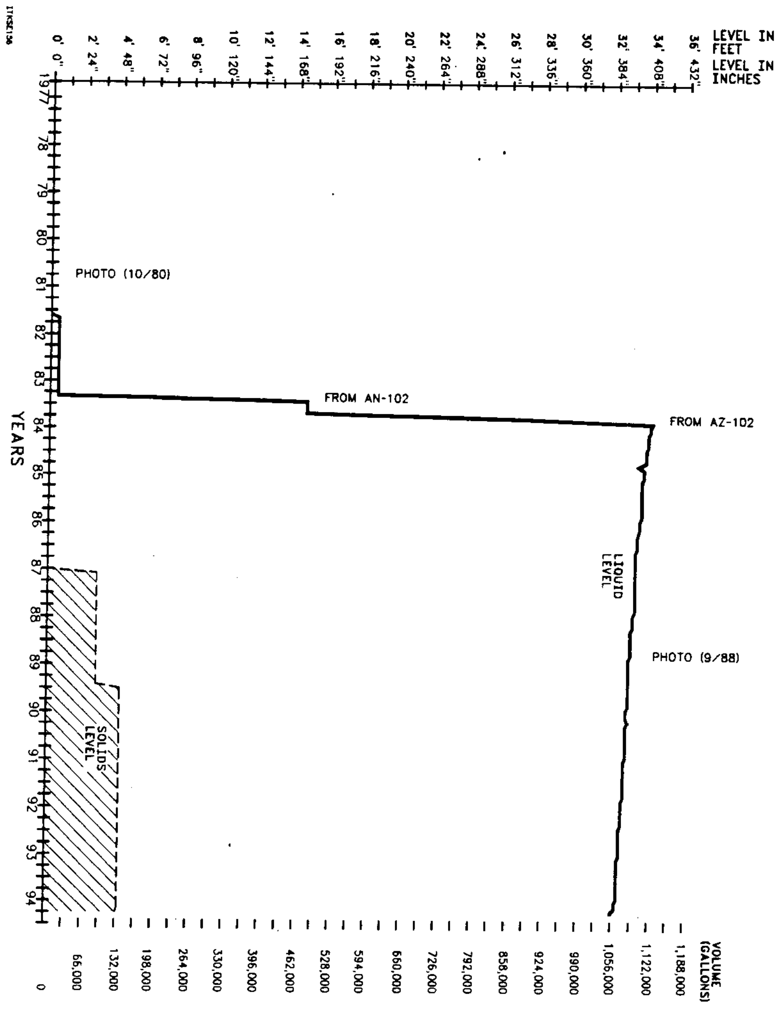
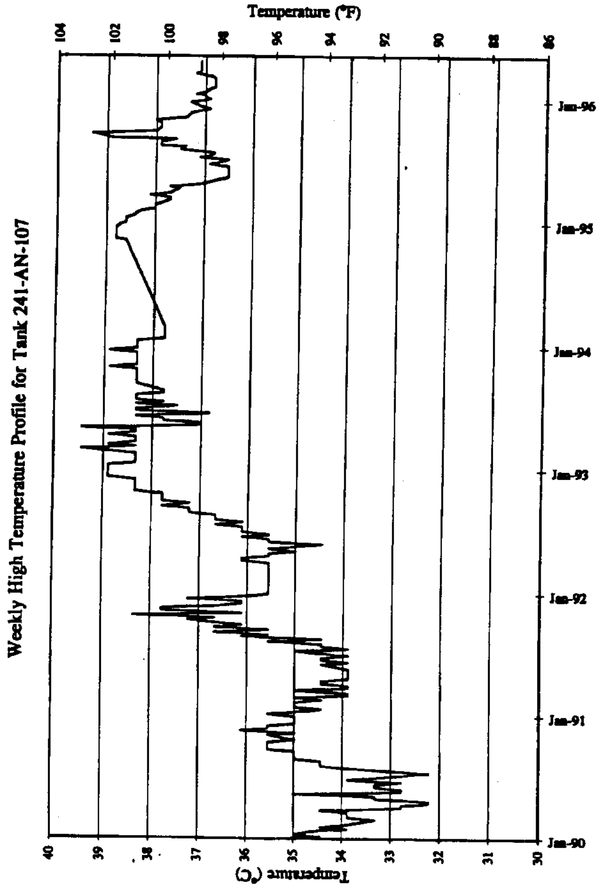


Figure 2-5. Tank 241-AN-107 Weekly High Temperature Plot.



3.0 TANK SAMPLING OVERVIEW

This section describes the February 1996 grab sampling and analysis event for tank 241-AN-107. Grab samples were obtained to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). The sampling and analyses were performed in accordance with the *Tank 241-AN-107 Grab Sampling and Analysis Plan* (Jo 1996). Further discussions of the sampling and analysis procedures can be found in the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

3.1 DESCRIPTION OF SAMPLING EVENT

The samples acquired on February 13 and 14, 1996 consisted of three supernatant samples and two sludge samples from each of the two risers. Samples 7AN-95-1 through 7AN-95-5 and a field blank 7AN-95-FB were acquired from riser 22A on February 14, 1996, and samples 7AN-95-6 through 7AN-95-10 were obtained from riser 10A on February 13, 1996. All samples were received by the Westinghouse Hanford Company (WHC) 222-S Laboratory on the day the samples were obtained.

The bottle-on-a-string sampling method was used to obtain the grab samples. The tank headspace was sampled from 6 m (20 ft) below risers 10A and 22A, and analyzed for flammable gas as prescribed by the safety screening DQO. Table 3-1 summarizes the sampling mode, applicable DQOs, and sampling and analytical requirements for the sampling events.

Table 3-1. Integrated Data Quality Objective Requirements for Tank 241-AN-107.[1]

Sampling Event	Applicable DQOs	Sampling Requirements	Analytical Requirements
February 1996 grab sampling	Safety screening (Dukelow et al. 1995)	Vertical profiles from two widely spaced risers	▸ Energetics ▸ Moisture content ▸ Total alpha activity ▸ Headspace gas flammability ▸ TOC[2] ▸ Visual check for presence of organic layer ▸ Density

Note:

[1]Jo (1996)

[2]TOC is a secondary analyte that was measured because of high DSC results, in accordance with Turner et al. (1995).

3.2 SAMPLE HANDLING

The grab samples were shipped to the 222-S Laboratory for subsampling and analysis. Samples were assigned LABCORE numbers as listed in Table 3-2. All samples were subjected to visual inspection for color, clarity, and solids content. The radiation dose rate on contact was also measured. All samples were dark yellow or dark brown in appearance, with no visible organic layer. The supernatant samples were then subsampled for the different analyses and for archiving.

Table 3-2. Grab Sample Information and Description.[1]

Customer ID	Laboratory ID	Sample Elevation[2] m (in.)	Sample Type	Volume Settled Solids %	Dose on Contact R/hr
Riser 22A					
7AN-95-1	S96T000719	7.62 (300)	Supernatant	Trace	1.1
7AN-95-2	S96T000720	5.49 (216)	Supernatant	Trace	1.8
7AN-95-3	S96T000721	3.35 (132)	Supernatant	Trace	1.8
7AN-95-4	S96T000738	0.914 (36)	Sludge	77.1 % settled	2.0
7AN-95-5	S96T000739	0.838 (33)	Sludge	89.2 % settled	2.2
7AN-95-FB	S96T000722	N/A	Field blank	none	< 0.005
Riser 10A					
7AN-95-6	S96T000661	7.62 (300)	Supernatant	Trace	1.2
7AN-95-7	S96T000662	5.49 (216)	Supernatant	Trace	2.5
7AN-95-8	S96T000663	3.35 (132)	Supernatant	Trace	2.0
7AN-95-9	S96T000695	0.813 (32)	Sludge	87.5 % settled	2.0
7AN-95-10	S96T000696	0.610 (24)	Sludge	88.0 % settled	2.5

Notes:
ID = identification

[1]Esch (1996)
[2]Sample elevation is measured from the tank bottom to the mouth of the sample bottle.

After measurement of the settled solids, the sludge samples were shaken and transferred to centrifuge cones for the measurement of volume percent solids by centrifugation and for separation of supernatant from the sludge. Bulk densities for the wet sludge, the centrifuged sludge, and the decanted supernatant were determined. Subsamples for analysis and archive were created from both the centrifuged sludge and the decanted supernatant portions.

Although the centrifuged liquids and solids from the sludge samples were analyzed separately, the analytical results were combined to give a better representation of the sludge, which is composed of liquids and solids. Solid material that resembled corroded metal was found in the decanted solid samples. Any of the material remaining in the sample bottles after the sample was transferred to the centrifuge cones was left in the sample bottle to minimize effects on sample homogeneity. The material found in samples 7AN-95-4 and 7AN-95-5 was removed and archived. In addition to resembling corroded metal, sample 7AN-95-5 contained some chunks of salt-like material, which was removed and archived. The volume percent settled solids were measured for the sludge samples.

3.3 SAMPLE ANALYSIS

Samples 7AN-95-1 through 7AN-95-10, and the field blank 7AN-95-FB were analyzed in accordance with the safety screening DQO. Analytes include energetics by differential scanning calorimetry (DSC) to ascertain the fuel energy value, weight percent water by thermogravimetric analysis (TGA), total alpha activity for determining the criticality potential, specific gravity (SpG), a visual check for the presence of an organic layer, and bulk density. The total organic carbon (TOC) and reactive system screening tool (RSST) adiabatic calorimetry were performed as secondary analyses because the DSC results exceeded the decision criteria threshold. The RSST was performed on the sludge sample that exhibited the highest exothermic energy (Esch 1996).

Analysis of the tank headspace for flammable gases is also required by the safety screening DQO. Tank headspace flammability was determined in the field by means of a combustible gas meter. The tank headspace vapor sample was drawn from 6 m (20 ft) below risers 10A and 22A. Tank headspace vapor sampling is further discussed in Section 4.1.3.

Quality control (QC) checks included, where appropriate, laboratory control standards, matrix spikes, duplicate analyses, and blanks. Results of the QC tests and the implications for data quality are discussed in Section 5.1.2.

All reported analyses were performed in accordance with approved laboratory procedures. A list of the analyses performed on specific samples is presented in Table 3-3. Table 3-4 summarizes the analytical procedure titles, instruments, and preparation methods used in the analysis of the grab samples.

Table 3-3. Tank 241-AN-107 Sample Analysis Summary.[1] (2 sheets)

Customer ID	Laboratory ID	Analysis
7AN-95-1	S96T000719	Bulk density
	S96T000723	DSC, TGA, TOC, total alpha
	S96T000727	Archive
7AN-95-2	S96T000720	Bulk density
	S96T000724	DSC, TGA, TOC, total alpha
	S96T000728	Archive
7AN-95-3	S96T000721	Bulk density
	S96T000725	DSC, TGA, TOC, total alpha
	S96T000729	Archive
7AN-95-4	S96T000738	Volume percent solids, bulk density
	S96T000740 (solid)	Archive
	S96T000744	DSC, TGA, TOC, bulk density
	S96T000748	Total alpha
	S96T000742 (liquid)	Archive
	S96T000746	DSC, TGA, TOC, bulk density, total alpha
7AN-95-5	S96T000739	Volume percent solids, bulk density
	S96T000741 (solid)	Archive
	S96T000745	DSC, TGA, TOC, bulk density
	S96T000749	Total alpha
	S96T000743 (liquid)	Archive
	S96T000747	DSC, TGA, TOC, SpG, total alpha
7AN-95-6	S96T000661	Bulk density
	S96T000697	DSC, TGA, TOC, total alpha
	S96T000702	Archive
7AN-95-7	S96T000662	Bulk density
	S96T000698	DSC, TGA, TOC, total alpha
	S96T000703	Archive
7AN-95-8	S96T000663	Bulk density
	S96T000699	DSC, TGA, TOC, total alpha
	S96T000704	Archive

Table 3-3. Tank 241-AN-107 Sample Analysis Summary.[1] (2 sheets)

Customer ID	Laboratory ID	Analysis
7AN-95-9	S96T000695	Volume percent solids, bulk density
	S96T000715 (solid)	Archive
	S96T000707	DSC, TGA, TOC, bulk density
	S96T000717	Total alpha
	S96T001445	Total alpha
	S96T000705 (liquid)	Archive
	S96T000700	DSC, TGA, TOC, bulk density, total alpha
7AN-95-10	S96T000696	Volume percent solids, bulk density
	S96T000716 (solid)	Archive
	S96T001825	RSST
	S96T000708	DSC, TGA, TOC, bulk density
	S96T000718	Total alpha
	S96T001446	Total alpha
	S96T000706 (liquid)	Archive
	S96T000701	DSC, TGA, TOC, bulk density, total alpha
7AN-95-FB (Field blank)	S96T000722	DSC, TGA, total alpha, SpG, archive
Vapor tests	N/A	Combustible gas meter readings for flammable gas concentration and oxygen, organic vapor monitor for total organic vapors and ammonia

Notes:

ID = Identification

[1]Esch (1996)

Table 3-4. Analytical Procedures.[1]

Analysis	Instrument	Preparation Procedure	Procedure Number[2]
Energetics by DSC	Mettler™	N/A	LA-514-113, Rev. C-1
Percent water by TGA	Mettler™		LA-560-112, Rev. B-1
Specific gravity	N/A		LA-510-112, Rev. C-3
Bulk Density	N/A		LO-160-103, Rev. B-0
TOC	Furnace oxidation/ Coulometer		LA-344-105, Rev. C-0
			LA-342-100, Rev. C-0
Total alpha activity	Alpha proportional counter	LA-549-141, Rev. E-0, for the sludge samples	LA-508-101, Rev. D-2
		Direct for supernatant samples.	
Visual for organic layer	N/A	N/A	LA-519-151
Flammable gas	Combustible gas meter	N/A	WHC-IP-0030 IH 1.4
Organic vapor	Organic vapor monitor	N/A	WHC-IP-0030 IH 2.1
Reactive system screening tool	N/A	N/A	WHC-SD-WM-TP-104

Notes:

Rev. = revision

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

[1]Esch (1996)

[2]Internal procedures of Westinghouse Hanford Company, Richland, Washington.

3.4 PREVIOUS SAMPLING EVENTS

Historically, waste samples have been analyzed to characterize the waste composition within the tank. Data for tank 241-AN-107 samples reported from 1980 through 1988 were obtained from Rockwell Hanford Operations internal letters and Westinghouse Hanford Company internal memoranda. Sample and analysis descriptions and analytical results are presented in Appendix B. Data from 1990 through 1994 sample events are reported in the following sections.

3.4.1 Description of the May 1990 DSC Analyses

On May 4, 1990 a supernatant sample was drawn from tank 241-AN-107. The purpose of the sampling and analysis was to examine exothermic reactions that exist at low scanning rates. The cover gas was air. The pans were open stainless steel. Runs were made from 50 to 500 °C at 5 degrees/minute, and at 20 degrees/minute. The exothermic reactions were consistent with the oxidation of an organic carbon. The 5 degree/minute scan produced a change in enthalpy of -1,199 J/g, and the 20 degree/minute scan produced a change in enthalpy of -817.7 J/g (Bechtold 1990).

3.4.2 Description of the June 1990 Caustic Consumption Experiment

In May of 1990, the results of an experiment were reported in which a sample was drawn from tank 241-AN-107 for the purposes of investigating possible interactions of added caustic with the complexed concentrate waste. A sample bottle from the tank was agitated to uniformly distribute the solids, and an aliquot was drawn. After centrifugation, two samples of the supernatant were drawn as controls, and were allowed to rest for 24 hours and then were recentrifuged prior to analysis. The results of the analysis of control samples A and C are reported in Table 3-5 (control sample B was not analyzed). The experimental results indicated a possible reaction between TOC and the added caustic. Stronger evidence was observed that alpha-emitting species reacted with the added caustic and formed precipitates (Washington 1990).

3.4.3 Description of the May 1994 Sampling Event

Three sludge samples and two supernatant samples were taken from tank 241-AN-107 to support characterization of the waste in the tank, and to evaluate the depletion of the caustic. The sludge samples, R04047 and R04748, were taken at depths of 2.5 m (627 in.) and 2.6 m (652 in.), respectively (Herting 1994). The supernatant samples were taken from the supernatant layer; no depth was specified (Herting 1993). A summary of the analytical results is presented in Table 3-6.

Table 3-5. Analytical Results of Control Samples for Caustic Consumption Experiment.[1]

Analyte	Experiment A control			Experiment C control		
	Super-natant	Water-washed solids	Acid-washed solids	Super-natant	Water-washed solids	Acid-washed solids
METALS	M	mole/g	mole/g	M	mole/g	mole/g
Aluminum	---	6.65E-05	5.95E-05	0.0762	---	3.36E-04
Calcium	---	1.24E-05	3.77E-06	0.0154	9.30E-06	2.19E-06
Chromium	---	2.58E-06	7.85E-07	0.00428	1.37E-06	1.72E-06
Iron	---	1.86E-05	4.95E-06	0.0309	5.60E-06	8.25E-06
Magnesium	---	2.98E-06	---	0.0112	4.54E-06	---
Sodium	---	6.30E-03	3.58E-04	8.22	5.16E-03	5.56E-04
Nickel	---	7.23E-06	---	0.01	5.44E-06	---
Phosphorus	---	1.42E-05	5.15E-06	---	---	8.05E-06
Anions	M	M	M	M	M	M
Carbonate	0.0216	---	---	1.19	---	---
Nitrate	---	---	---	---	---	---
Nitrite	1.09	---	---	1.01	---	---
Hydroxide	0.0525	---	---	0.0603	---	---
Radionuclides	μCi/L	μCi/L	μCi/L	μCi/L	μCi/L	μCi/L
Total alpha	695	0.5125	0.0618	691	0.3769	0.0681
^{60}Co	506	0.3150	0.02	---	0.2787	---
^{137}Cs	387,000	297.5	16.43	370,000	249.07	13.43
^{154}Eu	1,650	1.1250	0.19	---	0.963	---
^{155}Eu	2,070	1.2775	0.24	---	1.148	---
Physical properties						
TOC (g/L C)	46.7	---	---	1.19	---	---
pH	12.7	---	---	12.78	---	---

Note:
[1]Washington (1990)

Table 3-6. Composition of Sludge and Supernatant Samples from Tank 241-AN-107.[1,2]
(2 sheets)

Analysis	Supernatant Samples			Sludge Samples	
	R2686	R3154	R3155	R4047	R4048
Physical Properties					
SpG	1.047	1.392	1.384	1.464	1.473
DSC (J/g)	694	650	704	442	462
TGA (wt% water)	49.7	49.55	49.25	45.45	45.83
Total carbon	μg/g	μg/g	μg/g	μg/g	μg/g
TOC	29,900	29,900	32,400	26,300	27,900
TIC	12,200	11,200	8,800	10,000	9,600
Metals	μg/g	μg/g	μg/g	μg/g	μg/g
Aluminum	840	850	890	14,630	18,060
Nickel	320	320	330	330	330
Iron	1,050	1,070	1,110	3,200	4,660
Calcium	380	380	410	410	460
Chromium	120	120	130	380	530
Phosphorus	290	280	290	1,390	1,290
Lead	230	240	240	310	350
Potassium	1,230	1,460	1,880	1,080	1,112
Manganese	350	350	360	480	540
Sodium	144,310	141,080	142,750	140,300	140,700
Sulfur	1,850	1,890	2,180	1,770	1,780

Table 3-6. Composition of Sludge and Supernatant Samples from Tank 241-AN-107.[1,2] (2 sheets)

Analysis	Supernatant Samples			Sludge Samples	
	R2686	R3154	R3155	R4047	R4048
Anions	μg/g	μg/g	μg/g	μg/g	μg/g
Fluoride	ND	ND	ND	1,200	1,110
Chloride	2,500	2,300	1,900	1,300	1,400
Nitrite	29,500	33,500	43,200	38,600	45,800
Nitrate	178,800	166,700	164,100	134,000	160,400
Phosphate	300	400	400	410	400
Sulfate	7,900	11,600	10,000	8,300	8,500
Radionuclides	μCi/g	μCi/g	μCi/g	μCi/g	μCi/g
Total alpha	1.07	1.09	1.01	0.75	0.96
$^{239/240}Pu$	0.035	0.033	0.034	0.08	0.09
^{241}Am	0.66	0.58	0.66	0.60	0.79
^{137}Cs	302	259	197	334	276
^{90}Sr	92	89	99	108	121

Notes:
[1]Herting (1994)
[2]Herting (1993)

3.4.4 Description of the December 1994 Vapor Sampling Event

Four vapor samples from tank 241-AN-107 were obtained through the ventilation access duct on December 21, 1994. After a purge of the tubing and a leak check of the sample system were performed, a sample of ambient air was drawn to establish background levels. The samples were collected through flow-through canisters. The samples were received by the Inorganic Mass Spectrometry Laboratory at Pacific Northwest National Laboratory on December 29, 1994. The samples were analyzed in accordance with Carpenter (1994), using procedure number PNL-MA-599-ALO-284[1]. The analytical results are presented in Table 3-7 (Goheen 1994).

[1]Internal procedure of Pacific Northwest National Laboratory, Richland, Washington.

Table 3-7. 1994 Vapor Samples.[1]

Analyte	Sample identification number			
	S4086-AO1.WC1	S4086-AO2.WC2	S4086-AO3.WC3	S4086-AO4.WC4
	Mole percent	Mole percent	Mole percent	mole percent
Argon	0.94	0.94	0.94	0.94
Carbon dioxide	0.044	0.039	0.039	0.039
Carbon monoxide	< 0.01	< 0.01	< 0.01	< 0.01
Helium	< 0.001	< 0.002	< 0.001	< 0.001
Hydrogen	< 0.001	< 0.001	< 0.002	< 0.0018
Methane	< 0.001	< 0.001	< 0.001	< 0.001
Nitrogen	78.2	78	78	78
Oxygen	20.8	21	21	21
Nitrous oxide	< 0.001	< 0.001	< 0.001	< 0.001
Other nitrogen oxides	< 0.001	< 0.001	< 0.001	< 0.001
Other hydro-carbons	< 0.001	< 0.001	< 0.001	< 0.001

Note:

[1]Goheen (1994)

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4.0 ANALYTICAL RESULTS

Section 4.0 presents a summary of the analytical results associated with the February 1996 grab sampling of tank 241-AN-107. The sampling and analysis parameters governing this event were integrated by and described in the SAP (Jo 1996). Analysis of the grab samples was performed at the WHC 222-S Laboratory.

Data locations for this characterization report are displayed in Table 4-1. As noted in Table 4-1, the complete analytical data set can be found in Appendix A. Only analyte overall means are reported in Section 4.0.

Table 4-1. Analytical Data Presentation Tables.

Data Type	Tabulated Location
Chemical data summary	Table 4-2
Headspace flammability screening results	Table 4-3
Comprehensive analytical data	Appendix A

4.1 DATA PRESENTATION

The analytical results from the February 1996 sampling event involving tank 241-AN-107 have been summarized in Section 4.1. The data were originally reported in *Final Report for Tank 241-AN-107, Grab Samples 7AN-95-1 through 7AN-95-10 and 7AN-95-FB* (Esch 1996). Section 4.1.1 presents the chemical data, Section 4.1.2 contains the physical data, and Section 4.1.3 presents the headspace flammability results.

4.1.1 Chemical Data Summary

Data from the grab samples were used to derive overall means for all analytes except DSC, which does not require calculation of a mean. Separate means were reported for the supernatant and the sludge. The sludge means include the results of the centrifuged liquid analysis. All analyte means reported are weighted based on sample location. The overall means were calculated by first averaging the primary and duplicate results for each sample. The sample means from a given riser were then averaged to derive a riser mean. Finally, the riser means were averaged to derive the overall tank mean.

All information contained in Table 4-2 was taken from the Appendix A tables. Table 4-2 is divided into two sections, presenting the supernatant and sludge data. The first column of Table 4-2 contains the name of the analyte. The second and fifth columns contain the overall means for each analyte. The third and sixth columns display the relative standard deviation (RSD) of the mean, defined as the standard deviation (of the mean) divided by the mean, multiplied by 100. The RSDs were determined by using standard analysis of variance (ANOVA) statistical techniques. The projected inventories listed in the fourth and seventh columns were derived based on the waste volumes of 3,500 kL (924 kgal) of supernatant and 507 kL (134 kgal) of sludge. The projected inventories for the sludge phase of the waste were calculated using the mean density of the sludge.

Table 4-2. Chemical Data Summary for Tank 241-AN-107.[1]

Analyte	Supernatant			Sludge		
	Overall Mean	RSD (Mean)	Projected Inventory	Overall Mean	RSD (Mean)	Projected Inventory
RADIONUCLIDES	µCi/mL	%	Ci	µCi/g	%	Ci
Total alpha	0.799	2.88	2,800	0.989	14.2	727
CARBON	µg C/mL	%	kg C	µg C/g	%	kg C
Total organic carbon	55,700	2.39	1.95E+05	42,700	8.34	31,400
PHYSICAL PROPERTIES		%	kg		%	kg
Weight percent water	49.9	0.347	2.41E+06	45.5	3.00	3.34E+05
Bulk density (g/mL)	1.38	0.605	---	1.45	2.47	---

Note:

[1]Esch (1996)

4.1.2 Physical Data Summary

Thermal analyses and bulk density were performed on the tank 241-AN-107 grab samples to satisfy the requirements of the safety screening DQO (Dukelow et al. 1995).

4.1.2.1 Thermogravimetric Analysis. During a TGA, the mass of a sample is measured while its temperature is increased at a constant rate. Nitrogen is passed over the sample during the heating to remove any released gases. Any decrease in the weight of a sample represents a loss of gaseous matter from the sample either through evaporation or through a reaction that forms gas phase products. The moisture content is estimated by assuming that all TGA sample weight loss up to a certain temperature (typically 150 °C [302 °F]) is from water evaporation. Weight percent water by TGA was performed by the 222-S Laboratory on a Mettler™ instrument.

The TGA results for tank 241-AN-107 are presented in Appendix A, Table A-3. All samples exhibited a large weight loss between the ambient temperature and 200 °C (392 °F). In most of the runs, the weight loss occurred in two steps, or transitions. Again, this weight loss is attributed to the evaporation of water. The overall mean weight percent water values for the tank supernatant and sludge were 49.9 and 45.5, respectively.

4.1.2.2 Differential Scanning Calorimetry. During DSC, 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, 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 an exothermic (characterized by or causing the release of heat) event is determined graphically. Analyses by DSC were performed on a Mettler™ instrument.

The DSC results, including peak temperatures and magnitude of enthalpy changes on a wet basis, are presented in Appendix A, Table A-4. All of the samples exhibited an initial endothermic reaction, which represents the evaporation of free and interstitial water. All but one of the samples exhibited exothermic reactions in the second transition. Peak temperatures for these reactions ranged from 239.3 to 422.3 °C (462.7 to 792.1 °F), and exothermic changes in enthalpy from -297.8 to -664.8 J/g. One sample (S96T000744 from riser 22A at 0.914 m sample elevation) exhibited an endothermic reaction in the second transition, followed by an exothermic reaction in the third transition. The exothermic reactions for this sample exhibited peak temperatures of 423.9 and 277.4 °C (795.0 and 531.3 °F) and changes in enthalpy of -326 and -287.4 J/g for the primary and duplicate runs, respectively.

The exothermic reactions in all but one of the runs exceeded the safety screening action limit of a change in enthalpy of -480 J/g on a dry weight basis (Esch 1996). Ninety-five percent confidence intervals on the mean are tabulated in Section 5.5.1.

4.1.2.3 Reactive System Screening Tool. Because the safety screening action limit for DSC was exceeded, RSST adiabatic calorimetry was performed on the sludge sample with the highest energetics. The sample displayed complex, multi-step behavior, suggesting the presence of organic compounds of widely varying stabilities towards molten nitrate (Esch 1996). The RSST analysis exhibited a self-heating reaction that began at approximately 150 °C (302 °F) and ended at 600 °C (1,112 °F) with a peak temperature change of 900 °C/min (1,652 °F/min). The reaction, while exothermic, was not rapidly self-propagating (Esch 1996).

The water weight loss during sample preparation was 39.74 percent. The total weight loss including water loss during sample preparation and reaction products loss during the analysis was 57.77 percent. The total weight loss is low due to condensation on the sample insulation sheath. The maximum rate of temperature change was in excess of 900 °C/min for a few seconds and the maximum temperature was 617 °C (1,142.6 °F). An equation for specific exothermic energy on a dry weight basis was derived as follows:

$$\hat{Q}_{dry}\ (J/g) = 307(\overline{C}_{ps}^{react}) + 44.3$$
$$\text{where } \overline{C}_{ps}^{react} = \text{average sample heat capacity during reaction}$$
$$\hat{Q} = \text{specific exothermic energy.}$$

The average sample heat capacity in the above equation must be estimated because a measured value is unavailable (Esch 1996).

The gas production per gram of sample (on a dry weight basis) calculated by two different methods was 4.90E-03 mole/g and 5.12E-03 mole/g. The corresponding average molecular weight of gases produced was 61 and 59 g/mole, respectively. Error is introduced into the computed average molecular weight by the evolution of condensate, which contributes to weight loss but not to pressure.

4.1.2.4 Bulk Density. Bulk density measurements were performed on the samples. Specific gravity was measured instead of bulk density for one of the samples (S96T000747) because bulk density could not be measured due to the presence of suspended solids. The bulk density and specific gravity results are presented in Table A-5. The overall mean bulk density values for the tank supernatant and sludge were 1.38 and 1.45 g/mL, respectively.

In addition to the supernatant and sludge, bulk density measurements were performed on the wet sludge samples (prior to centrifugation). The overall mean bulk density for the wet sludge was 1.48 g/mL.

4.1.2.5 Volume Percent Solids. Volume percent solids by centrifugation measurements were performed on the four sludge samples. The overall mean volume percent solids for the four sludge samples was 60.6 percent.

4.1.3 Headspace Flammability Screening Results

As requested in the SAP (Jo 1996), the tank 241-AN-107 headspace was sampled and analyzed for the presence of flammable gases prior to grab sampling. The safety screening decision criteria threshold for flammable gas concentration is 25 percent of the lower flammability limit (LFL) (Dukelow et al. 1995). The combustible gas meter used to sample the tank headspace reports results as a percentage of the lower explosive limit (LEL). Because the National Fire Protection Association defines the terms LEL and LFL identically, the two terms may be used interchangeably (NFPA 1995). The reported flammable gas result of 0 percent of the LEL was well below the safety screening decision criteria threshold. In addition to flammable gases, the concentrations of oxygen, ammonia, and total organic carbon vapor were determined. The results of the gas monitoring are presented in Table 4-3.

In addition to the vapor samples obtained prior to the 1996 grab sampling event, the tank headspace was vapor sampled December 21, 1994. The sampling event is discussed in Section B-9, and the results are presented in Table B-10.

Table 4-3. Headspace Flammability Screening for Tank 241-AN-107.[1]

Vapor Characteristic Measured	Results	
	Riser 10A, 20 ft inside riser	Riser 22A, 20 ft inside riser
Flammability gas concentration	0% of LEL	0% of LEL
Volume percent oxygen gas	N/A	20.6%
Ammonia gas concentration	65 ppm	< 200 ppm
Total organic carbon vapor concentration	2.7 ppm	9.6 ppm

Note:
[1]Esch (1996)

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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-107 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 use of the data. Most of the usual consistency checks were not possible given the limited scope of analyses.

5.1.1 Field Observations

A vertical profile of the tank was obtained from two widely spaced risers as required by the safety screening DQO (Dukelow et al. 1995). A horizontal and vertical comparison of the analytical results between the two grab samples was possible, providing an estimate of the distribution of waste constituents in the sludge and supernatant (see Section 5.3). No problems during sampling or transportation to the laboratory were reported.

5.1.2 Quality Control Assessment

The usual QC assessment includes an evaluation of the appropriate blanks, duplicates, matrix spike recoveries, and standard recoveries performed in conjunction with the chemical analyses. All of the pertinent QC tests were conducted on the 1996 grab samples and reported in Esch (1996). The *Hanford Analytical Services Quality Assurance Plan* (DOE 1995) established the specific accuracy and precision criteria for the QC checks.

All of the total alpha activity standard recoveries were within the target level. The TOC spike recovery for one of the supernatant samples was below the lower limit. The precisions (estimated by the relative percent difference [RPD], which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, multiplied by one hundred) between all TGA and TOC sample/duplicate pairs were within the limits. All four sludge samples had RPD results for total alpha activity that exceeded the limits. Samples 7AN-95-9 and 7AN-95-10 were redigested and reanalyzed, with sample results for 7AN-95-9 still outside the limits. The RPD of one supernatant DSC sample/duplicate pair was outside the criteria. Finally, none of the samples exceeded the criterion for preparation blanks; thus, contamination was not a problem for any of the analyses.

The majority of the QC results were within the boundaries specified in DOE (1995). Although a few were outside their target levels, they were not found to substantially impact either the validity or the use of the data.

5.1.3 Data Consistency Checks

Comparisons of different analytical methods can help to assess the consistency and quality of the data. Examples would be the comparison of phosphorous as determined by inductively coupled plasma spectroscopy versus phosphate as determined by ion chromatography, the calculation of mass and charge balances, and the comparison of total alpha to the sum of the alpha emitters. Due to the lack of data, no consistency checks were possible.

5.2 COMPARISONS AMONG HISTORICAL ANALYTICAL RESULTS AND HTCE

The results of the 1993/1994 sample and analysis event (Herting 1993 and 1994) were compared with the HTCE. The comparisons are shown in Table 5-1. As can be seen in the table, most analytes exhibited RPDs greater than 50 percent. Two analytes (^{90}Sr and ^{137}Cs) exhibited RPDs less than 50 percent and greater than 25 percent. Density, weight percent water, plutonium, nitrate, sodium, potassium and lead all showed RPDs less than 25 percent.

Table 5-1. Comparison of 1993/1994 Historical Analytical Results with HTCE.[1,2] (2 sheets)

Analyte	1993/1994 Analytical Result[3]	HTCE[4]	Relative percent difference
METALS	µg/g	µg/g	%
Aluminum	2,870	29,400	164
Calcium	396	670	51.4
Chromium	166	1,450	159
Iron	1,440	206	150
Lead	249	197	23.2
Manganese	374	176	71.9
Nickel	324	178	58.2
Potassium	1,470	1,470	0
Sodium	1.42E+05	1.70E+05	17.9

Table 5-1. Comparison of 1993/1994 Historical Analytical Results with HTCE.[1,2] (2 sheets)

Analyte	1993/1994 Analytical Result[3]	HTCE[4]	Relative percent difference
ANIONS	µg/g	µg/g	%
Chloride	2,120	4,740	76.4
Fluoride	150	943	125
Nitrate	167,000	1.69E+05	1.24
Nitrite	36,300	69,000	62.1
Phosphate	846	6,030	151
Sulfate	9,650	17,900	59.9
RADIONUCLIDES	µCi/g	µCi/g	%
$^{239/240}Pu$	0.0406	0.0443	8.64
^{137}Cs	259	168	42.8
^{90}Sr	96.0	62.4	42.5
TOTAL CARBON	µg/g	µg/g	%
TOC	30,300	14,800	68.6
TIC	10,600	4,220	86.2
PHYSICAL PROPERTIES			%
Density	1.27	1.54	19.2
Weight percent water	49.0	39.0	22.7

Note:

[1]Herting (1993 and 1994)

[2]Brevick (1995)

[3]Supernatant and sludge results were averaged for the comparison. The averages were weighted using the relative proportions of supernatant and sludge, based on the volume of the tank as of May 31, 1994 (Hanlon 1994). Of a total of 4,021 kL (1,062 kgal) of waste, 507 kL (134 kgal) were sludge, and 3,512 kL (928 kgal) were supernatant.

[4]The data are not validated and should be used with caution.

In addition to the comparison between the HTCE and the 1993/1994 data (Herting 1993 and 1994), a comparison was made between the combined 1996 analytical data and the 1993/1994 data. The 1996 data were combined by weighting their average according to the relative proportions of the supernatant and sludge volumes. The comparison is presented in Table 5-2. As can be seen in the table, good agreement exists between 1996 analytical data and the 1993/1994 data.

Table 5-2. Comparison of Combined 1996 Data and 1993/1994 Data.[1,2]

Analyte	1996 combined results	1993/1994 results	Relative percent difference
Bulk density (g/mL)	1.39	1.27	9.02
Water (wt %)	49.3	49.0	0.610
Total alpha (μCi/g)	0.824	1.03	22.3
TOC (μg C/g)	40,700	30,300	29.3

Notes:
[1]Esch (1996)
[2]Herting (1993 and 1994)

Comparisons were also made between the 1996 sludge and supernatant data and those from 1990 (Washington 1990). The supernatant results from 1996 were compared directly with the supernatant results from 1990, which are the averages of the results from two samples. The water wash and the acid wash results for the 1990 sludge analyses, also the average of two samples, were added prior to comparison because the water and acid washes were performed on the same material in succession. The sludge density from 1996 (1.45 g/mL) was used to convert the 1990 total alpha results from μCi/L to μCi/g. The comparison is presented in Table 5-3.

Table 5-3. Comparison of 1996 Sludge and Supernatant Data with 1990 Historical Data.[1,2]

Analyte	1996 sludge result	1990 sludge result	RPD
Total alpha (μCi/g)	0.989	2.54E-04	200
Analyte	**1996 supernatant result**	**1990 supernatant result**	**RPD**
Total alpha (μCi/mL)	0.799	0.693	14.2
TOC (μg C/mL)	55,700	23,900	79.9

Notes:
[1]Esch (1996)
[2]Washington (1990)

5.3 TANK WASTE PROFILE

According to the estimate of Hanlon (1996), tank 241-AN-107 contains 3,500 kL (924 kgal) of supernatant and 507 kL (134 kgal) of sludge. The sludge is estimated to contain 34 kL (9 kgal) of drainable interstitial liquid. The photographic montage of the waste surface suggests a dark brown liquid (Section 2.4.3). The visual description of all samples was as opaque and dark yellow/brown. However, the samples collected near the bottom of the tank had a large percentage of solids. The TLM (Figure 2-3) is in fairly close agreement with Hanlon (1996), indicating 4,012 kL (1,060 kgal) of supernatant and 507 kL (134 kgal of sludge.

Waste samples were obtained from different depths and different risers. Consequently, nested random effects ANOVA models (riser and depth terms) were fit to the total alpha, weight percent water, and TOC data for both the liquid and solid portions of the samples. Because of the lack of duplicate analyses, no ANOVA model was fit to the density data.

The results from these models can be used to test whether mean analyte concentrations vary significantly in the vertical and horizontal directions.

The results from the ANOVA models showed that there were no significant differences in the mean concentrations for total alpha, weight percent water and TOC between risers (horizontal variability). This was true for both the liquid and solid portion of the samples.

For the solid portion of the samples, there were significant differences (0.05 level of significance) in mean concentrations between depths (vertical variability) for weight percent water. For the liquid portion of the samples, there were significant differences in mean concentrations between depths for total alpha and TOC.

In summary, the information available from the Hanlon (1996) estimates, the photographic montage, the visual descriptions of the samples, the TLM, and the statistical results is not consistent regarding the disposition of the waste. Vertical tank heterogeneity is indicated, but the information available does not suggest lateral homogeneity.

5.4 COMPARISON OF HTCE WITH ANALYTICAL RESULTS

The HTCE data (from Table 2-4) for tank 241-AN-107 were compared to the 1996 combined analytical results for bulk density, percent water, total alpha activity, and TOC. Two of the four analytes (bulk density, and water) exhibited RPDs less than 25. The HTCE underestimated the total alpha activity and TOC concentrations. However, the total alpha activity comparison may be biased low, because the HTCE prediction of this analyte only includes plutonium. Table 5-4 presents the comparison.

Table 5-4. Comparison of Transfer History with 1996 Analytical Results.

Analyte	1996 Combined Results[1]	HTCE Estimate[2,4]	Relative Percent Difference
Bulk density (g/mL)	1.39	1.54	10.2%
Water (wt%)	49.3	39.0	23.3%
Total alpha (μCi/g)	0.824	0.0443[3]	180%
TOC (μg C/g)	40,700	14,800	93.3%

Note:
[1]Weighted mean taken from Esch (1996)
[2]Agnew et al. (1996a)
[3]This value includes total plutonium only.
[4]The data are not validated and should be used with caution.

5.5 EVALUATION OF PROGRAM REQUIREMENTS

Tank 241-AN-107 is classified as a non-Watch List tank. The grab samples retrieved from the tank were acquired to meet the requirements of the safety screening DQO (Dukelow et al. 1995). A discussion of the specific requirements of the DQO and a comparison of the analytical data to defined decision limits are presented in this section.

5.5.1 Safety Evaluation

Data criteria identified in the safety screening DQO are used to assess the tank's safety and to check for unidentified safety issues. The DQO requires at least two vertical profiles of the tank waste; after the data from these two profiles are reviewed, more profiles may be required. An assessment was made of the results from the 1996 sampling event, and it was decided that further sampling was not needed. Of the five primary analyses required by the DQO, three have decision criteria thresholds which, if exceeded, could warrant further investigation to ensure tank safety. These three analyses include DSC (to measure the fuel content), total alpha activity (to evaluate the criticality potential), and a measurement of the tank headspace flammability.

The safety screening DQO has established a criteria threshold limit of a change in enthalpy of -480 J/g (dry weight basis) for exothermic reactions detected during the DSC analysis. All samples except one duplicate exhibited exothermic reactions greater than the decision criteria threshold; the highest sample-duplicate pairs exhibited a change in enthalpy of -1,304 J/g (dry weight) and the largest 95 percent confidence interval upper limit on the mean was -1,985 J/g. As presented in Esch (1996), Table 5-5 lists the DSC results, the means, and the upper limit to 95 percent confidence intervals on the mean for all samples.

The results for the solid samples were consistently lower than those for the liquid samples, which supports the suggestion that the organic fuel is soluble.

Table 5-5. DSC Exothermic Reactions on a Dry Weight Basis at a 95% Confidence Level.[1] (2 sheets)

Sample Number	Sample ΔH	Duplicate ΔH	Mean ΔH	Standard Deviation ΔH	95% Upper Confidence Limit on the Mean ΔH
	J/g	J/g	J/g	J/g	J/g
Supernatant					
S96T000723	-1,047	-1,139	-1,093	65.1	-1,383
S96T000724	-1,278	-1,243	-1,260	24.7	-1,371
S96T000725	-1,077	-1,167	-1,122	63.6	-1,406
S96T000697	-1,264	-1,345	-1,304	57.3	-1,560
S96T000698	-1,224	-1,303	-1,263	55.9	-1,513
S96T000699	-969	-1,247	-1,108	196.3	-1,985
Sludge					
S96T000744	-528	-466	-498	43.8	-693
S96T000745	-530	-497	-514	23.8	-620
S96T000746	-1,148	-1,114	-1,131	24.0	-1,238
S96T000747	-1,174	-1,167	-1,170	4.9	-1,193
S96T000707	-628	-566	-597	43.7	-792
S96T000708	-638	-652	-645	9.2	-686
S96T000700	-1,124	-963 (dup.) -1,173 (trip.)	-1,087	109.9	-1,272
S96T000701	-1,115	-1,034	-1,074	57.3	-1,330

Notes:

dup. = duplicate
trip. = triplicate
ΔH = change in enthalpy

[1]Esch (1996)

Total organic carbon was analyzed because the DSC results exceeded the decision criteria threshold. The following results are given on a dry weight basis (Table 5-6). All TOC samples exceeded the decision criteria threshold of 30,000 μg C/g; the highest sample-duplicate result was 87,400 μg C/g. However, with a moisture content greater than 17 weight percent, the tank can be considered "conditionally safe" in accordance with the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995). Table 5-6 presents the TOC results along with the 95 percent confidence interval upper limits. The high value for the upper limit of the 95 percent confidence interval was 88,600 μg C/g. Reactive systems screening testing (RSST) was performed on the sludge sample with the highest fuel content as determined by DSC (S96T000701). The RSST results indicated a self-heating reaction that began at 150 °C (302 °F) and ended at 600 °C (1,112 °F), with a peak temperature change of 900 °C/min (1,652 °F/min). The reaction, while exothermic, was not rapidly self-propagating.

Table 5-6. Total Organic Carbon on Dry Weight Basis at 95% Confidence Level.[1]

Sample Number	Sample	Duplicate	Mean	Standard Deviation	95% Upper Confidence Limit
Supernatant	μg C/g	μg C/g	μg C/g	μg C/g	μg C/g
S96T000723	87,700	87,100	87,400	220	88,400
S96T000724	81,200	81,700	81,500	175	82,200
S96T000725	74,400	78,500	76,400	1,430	82,800
S96T000697	75,300	74,000	74,700	432	76,600
S96T000698	80,400	79,800	80,100	214	81,100
S96T000699	82,300	82,800	82,600	176	83,400
Sludge	μg C/g	μg C/g	μg C/g	μg C/g	μg C/g
S96T000744	43,700	43,800	43,700	50.5	44,000
S96T000745	45,500	47,100	46,300	537	48,700
S96T000746	86,900	86,000	86,400	288	87,700
S96T000747	82,700	83,500	83,000	276	84,300
S96T000700	74,400	74,500	74,500	31.9	74,600
S96T000701	86,900	85,400	86,200	540	88,600
S96T000707	54,400	48,600	51,500	2,040	60,600
S96T000708	54,700	54,400	54,600	128	55,100

Note:

[1]Data were taken from Esch (1996). All data were converted to a dry weight basis, in units of μg C/g.

The safety screening DQO requires the measurement of cyanide for sludge samples in which the DSC results exceed the energy equivalent of the TOC measurement. In order to evaluate the need for cyanide analysis, the TOC results from the two sludge samples from riser 22A (exhibiting the lowest TOC) were converted to equivalent energy using the following equation. The 632 J/g value represents the energy equivalent of 5 weight percent TOC, based on a sodium acetate average energetics standard.

$$Energy\ Equivalent = wt\%\ TOC\ (dry\ weight)\ \left[\frac{632\ J/g}{5}\right]$$

The energy equivalent of the lower of the two samples from riser 22A (S96T000744) was -552 J/g. The DSC result from that same sample was -498 J/g. Because the TOC accounted for the energetics in the DSC analysis, no cyanide measurement was required.

The safety screening DQO limit for criticality is 42.4 μCi/g for the sludge, and 61.5 μCi/mL for supernatant and is assessed from the total alpha activity. All results were well below the limit. The mean sludge result was 0.989 μCi/g and the mean supernatant result was 0.799 μCi/mL. The highest 95 percent upper confidence interval limit on the mean for total alpha was 3.44 μCi/g, from sample S96T001445.

The DQO notification limit for flammable gas concentration in the tank headspace is 25 percent of the LFL (as discussed in Section 4.0). Combustible gas meter readings taken at the time of the 1996 sampling revealed the concentration of flammable gases to be 0 percent of the LFL.

Table 5-7 lists the safety issues, the analytes of concern along with their decision thresholds, and the corresponding analytical results.

Another factor in assessing tank waste safety is heat generation and temperature of the waste. Heat is generated in the tanks from radioactive decay. Analytical data from sampling and analysis events in February 1993 and May 1994 were used to calculate the tank heat load. A result of 8,060 W (27,500 Btu/hr) was obtained (Herting 1994). The HTCE prediction was 7,500 W (25,600 Btu/hr), while the heat load estimate by Kummerer (1994) was 7,910 W (27,000 Btu/hr). These estimates are well below the design specification of 20,500 W (70,000 Btu/hr) for the 241-AN tank farm (Harris 1992). Because an upper temperature limit has been exhibited (Section 2.4.3), it may be concluded that any heat generated from radioactive sources throughout the year is dissipated.

The integrity of tank systems that contain high-level radioactive waste is maintained by addition of caustic compounds such as sodium hydroxide, resulting in the elevation of the concentration of free hydroxide radicals. This is normally a stable situation requiring little or no adjustment. Tank 241-AN-107, however, has a history of consumption or depletion of the free hydroxide radicals. The present concentration of hydroxide is not a general corrosion problem, but can potentially cause stress corrosion cracking. A plan has been

Table 5-7. Safety Screening and Organic Data Quality Objective Decision Variables and Criteria.[1,2,3,4]

Issue	Primary Decision Variable	Decision Criteria Threshold	Analytical Result
Ferrocyanide/ Organics	Total fuel content	-480 J/g (dry weight)	All samples exceeded the limit. Largest sample-duplicate mean value of exotherm -1,304 J/g (dry weight).
Organics	Total Organic Carbon	30,000 μg C/g (dry weight)	All samples exceeded the limit. High sample-duplicate mean value of 87,400 μg C/g (dry weight)
Criticality	Total alpha	42.4 μCi/g (sludge) 61.5 μCi/mL (supernatant)	0.989 μCi/g (sludge) 0.799 μCi/mL (supernatant)
Flammability	Flammable gas	25 percent of the LFL	0 percent of the LFL

Note:
[1]Dukelow et al. (1995)
[2]Turner et al. (1995)
[3]Jo (1996)
[4]Esch (1996)

developed (Carothers 1992) whereby the hydroxide concentration will be adjusted to the point at which corrosion stress cracking can be avoided. The plan calls for the addition of 19 M sodium hydroxide to the tank waste, in two phases. The first phase will add caustic to the supernatant only, with no mixing of the sludge layer. This will protect most of the tank surface, since most of the waste is supernatant. The second phase will uniformly mix the tank contents, including the sludge, during the caustic addition.

A sample of the waste in tank 214-AN-107 was acquired in order to study the affects of hydroxide addition to the waste (Washington 1990). It was noted that alpha-emitting radionuclides tended to precipitate after caustic addition. Total alpha was found to decrease in the supernatant samples. Analysis of the solids in the samples using an acid digestion accounted for the decreased total alpha activity in the supernatant. Some evidence was noted that the levels in the supernatant for TOC and carbonate also decreased after caustic addition.

6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-AN-107 has been sampled and analyzed for the purposes of safety screening according to the requirements listed in the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). The tank was grab sampled in February 1996. To assess tank safety, the safety screening DQO required analyses for energetics, total alpha activity, weight percent water, density, a check for the presence of a separable organic layer, and the flammable gas concentration of the tank headspace. The sample analyses were performed at the WHC 222-S Laboratory.

The safety screening DQO has established a decision limit of a change in enthalpy of -480 J/g (dry weight basis) for exothermic reactions detected during the DSC analysis. All the samples except one duplicate exhibited exothermic reactions greater than the decision limit; the highest exothermic reaction measured was -1,304 J/g (dry weight). The highest upper 95 percent confidence interval limit for the DSC analysis was -1,985 J/g on a dry weight basis.

Because the DSC results exceeded the decision limit, total organic carbon was analyzed. All TOC samples exceeded the decision limit of 30,000 μg C/g (dry weight); the highest sample-duplicate mean result on a dry weight basis was 87,400 μg C/g. The highest upper 95 percent confidence interval limit on the mean on a dry weight basis for the TOC analysis was 88,600 μg C/g. However, because its contents have a moisture content greater than the criterion of 17 weight percent (the tank contents measured > 40 percent water), the tank can be considered "conditionally safe" in accordance with the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995).

The safety screening DQO limit for criticality is 42.4 μCi/g for the sludge and 61.5 μCi/mL for the supernatant, and is assessed from the total alpha activity. All results were well below the limit. The mean sludge result was 0.989 μCi/g and the mean supernatant result was 0.799 μCi/mL. The highest upper 95 percent confidence interval limit on the mean was 3.44 μCi/g for the sludge and 1.23 μCi/mL for the supernatant.

The heat load for tank 241-AN-107 according to the HTCE was 7,500 W (25,600 Btu/hr), while the heat load estimate by Kummerer (1994) was 7,910 W (27,000 Btu/hr). Analytical data from sampling and analysis events in February 1993 and May 1994 were used to calculate the heat load. A result of 8,060 W (27,500 Btu/hr) was obtained. These estimates are well below the design specification of 20,500 W (70,000 Btu/hr) for the 241-AN tank farm (Harris 1992).

The DQO notification limit for flammable gas concentration is 25 percent of the LFL (as discussed in Section 4.0). Combustible gas meter readings taken at the time of the 1996 sampling revealed the concentration of flammable gases to be 0 percent of the LFL.

Finally, all analytical results indicate the feasibility of successfully retrieving and disposing of the waste. However, the caustic depletion issue warrants further monitoring. Measures must also be taken to ensure that the moisture in the tank remains within limits.

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APPENDIX A

ANALYTICAL RESULTS

DOUBLE-SHELL TANK 241-AN-107

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A.1 INTRODUCTION

Appendix A presents the chemical and radiological characteristics of tank 241-AN-107 in a tabular form, in terms of the specific concentrations of total alpha activity, total carbon, and physical properties.

The data table for each analyte lists the laboratory sample number, sample location (riser), an original and duplicate result for each sample, a sample mean, an overall mean for the tank, a relative standard deviation, and a projected inventory for the tank. The data are listed in standard notation for values greater than 0.001 and less than 100,000. Values outside these limits are listed in scientific notation.

The tables are numbered sequentially. The following table presents the table numbers of specific analyte groups.

Analyte Characteristic	Table Number
Total alpha	A-1
Total organic carbon	A-2
Physical properties	A-3 through A-6

A.2 ANALYTE TABLE DESCRIPTION

The "Sample Number" column lists the laboratory sample number for which the analyte was measured. Sampling rationale, locations, and descriptions of the sampling event are discussed in Section 3.0.

The "Sample Location" column lists the riser from which the sample was collected.

The "Result" and "Duplicate" columns are self-explanatory. The "Sample Mean" column is the average of the result and duplicate values. Superscript letters on the mean values are quality control flags. The quality control flags are defined as follows:

- QC:a indicates a standard recovery below the QC limit.
- QC:b indicates a standard recovery above the QC limit.
- QC:c indicates a spike recovery below the QC limit.
- QC:d indicates a spike recovery above the QC limit.
- QC:e indicates that the RPD was outside the QC limit.
- QC:f indicates the presence of blank contamination.

The "Overall Means" were calculated from the sample means, and were weighted based on sample location. The sample means from a given riser were averaged to derive a riser mean. The riser means were then averaged to derive the overall mean. Separate means were calculated for the supernatant and sludge. The sludge means contain the results of the centrifuged liquids. This is discussed further in Sections 3.2 and 4.1.1.

The "Relative Standard Deviation" is a measure of variance defined as the standard deviation divided by the mean. This number is expressed as a percentage.

The "Projected Inventory" is the inventory estimate for the tank calculated from the overall means and the waste volume at the time of sampling: 3,500 kL (924 kgal) of supernatant and 507 kL (134 kgal) of sludge. The density of the centrifuged solids was used to calculate the projected inventories for the solid waste phase.

Table A-1. Tank 241-AN-107 Analytical Results: Total Alpha Activity.

Sample Number	Riser Number	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)	Projected Inventory
Supernatant		µCi/mL	µCi/mL	µCi/mL	µCi/mL	%	Ci
S96T000723	22A	0.839	0.8250	0.8320	0.799	2.88	2,800
S96T000724		0.854	0.8050	0.8300			
S96T000725		0.823	0.7880	0.8050			
S96T000697	10A	0.796	0.7170	0.7380			
S96T000698		0.787	0.7760	0.7820			
S96T000699		0.721	0.8610	0.7910			
Sludge		µCi/g	µCi/g	µCi/g	µCi/g	%	Ci
S96T000700	10A	0.983	1.100	1.042	0.989	14.2	727
S96T000701		1.150	1.120	1.135			
S96T000717		0.804	1.060	0.9320[QC:e]			
S96T000718		0.671	1.130	0.9000[QC:e]			
S96T001445		1.580	0.8800	1.230[QC:e]			
S96T001446		1.540	1.490	1.515			
S96T000746	22A	0.838	0.8390	0.8390			
S96T000747		1.030	1.000	1.015			
S96T000748		0.723	0.5800	0.6510[QC:e]			
S96T000749		1.020	0.7190	0.8690[QC:e]			

Table A-2. Tank 241-AN-107 Analytical Results: Total Organic Carbon.

Sample Number	Riser Number	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)	Projected Inventory
Supernatant		μg C/mL	μg C/mL	μg C/mL	μg C/mL	%	kg C
S96T000723	22A	60,300	60,500	60,400	55,700	2.39	1.95E+05
S96T000724		56,700	56,900	56,800			
S96T000725		51,400	53,700	52,600			
S96T000697	10A	51,300	51,600	51,400			
S96T000698		55,900	55,500	55,700			
S96T000699		56,900	57,200	57,000			
Sludge		μg C/g	μg C/g	μg C/g	μg C/g	%	kg C
S96T000744	22A	27,000	26,900	27,000	42,700	8.34	31,400
S96T000745		27,600	27,900	27,800			
S96T000746		59,200	58,300	58,800			
S96T000747		56,800	56,300	56,600			
S96T000700	10A	50,900	50,900	50,900			
S96T000701		58,900	58,500	58,700[QC:c]			
S96T000707		30,700	28,100	29,400			
S96T000708		32,800	32,600	32,700			

Table A-3. Tank 241-AN-107 Analytical Results: Weight Percent Water. (2 sheets)

Sample Number	Riser Number	Run	Transition 1		Transition 2		Total	Sample Mean	Overall Mean	RSD (Mean)
Supernatant			% H_2O	Temperature Range (°C)	% H_2O	Temperature Range (°C)	% H_2O	% H_2O	% H_2O	%
S96T000723	22A	1	40.96	35 - 90	9.22	90 - 195	50.18	49.92	49.9	0.347
		2	39.71	35 - 95	9.95	95 - 200	49.66			
S96T000724		1	36.04	30 - 110	13.38	110 - 180	49.42	49.48		
		2	36.98	35 - 110	12.57	110 - 180	49.55			
S96T000725		1	34.61	35 - 110	15.34	110 - 180	49.95	50.18		
		2	36.21	35 - 110	14.20	110 - 190	50.41			
S96T000697	10A	1	38.89	35 - 100	12.72	100 - 185	51.61	50.55		
		2	41.29	35 - 100	8.21	100 - 175	49.50			
S96T000698		1	42.52	35 - 95	7.11	95 - 175	49.63	49.62		
		2	42.69	35 - 95	6.92	95 - 170	49.61			
S96T000699		1	41.00	35 - 90	8.91	90 - 190	49.91	49.93		
		2	40.57	35 - 90	9.38	90 - 195	49.95			

Table A-3. Tank 241-AN-107 Analytical Results: Weight Percent Water. (2 sheets)

Sample Number	Riser Number	Run	Transition 1		Transition 2		Total	Sample Mean	Overall Mean	RSD (Mean)
Sludge			% H_2O	Temperature Range (°C)	% H_2O	Temperature Range (°C)	% H_2O	% H_2O	% H_2O	%
S96T000744	22A	1	22.89	40 - 110	15.28	110 - 155	38.17	38.39	45.5	3.00
		2	26.53	35 - 100	12.07	100 - 135	38.60			
S96T000745		1	28.76	35 - 85	10.63	85 - 120	39.39	40.05		
		2	28.73	35 - 70	11.98	70 - 110	40.71			
S96T000746		1	43.69	35 - 95	6.92	95 - 170	50.61	50.75		
		2	44.37	35 - 95	6.53	95 - 165	50.90			
S96T000747		1	46.17	35 - 110	4.06	110 - 165	50.23	50.68		
		2	51.13	35 - 165	---	---	51.13			
S96T000700	10A	1	39.51	35 - 95	10.94	95 - 165	50.45	50.48		
		2	39.03	35 - 90	11.48	90 - 160	50.51			
S96T000701		1	42.40	35 - 100	8.51	100 - 170	50.91	50.64		
		2	35.00	35 - 100	15.37	100 - 180	50.37			
S96T000707		1	43.55	30 - 155	---	---	43.55	42.87		
		2	42.19	35 - 170	---	---	42.19			
S96T000708		1	40.07	35 - 170	---	---	40.07	40.05		
		2	40.04	35 - 170	---	---	40.04			

Table A-4. Tank 241-AN-107 Analytical Results: Energetics by DSC. (2 sheets)

Sample Number	Riser Number	Run	Sample Weight	Transition 1		Transition 2		Transition 3	
Supernatant			mg	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)
S96T000723	22A	1	14.473	112.1	1,394.4	239.5	-524	---	---
		2	14.372	119.8	1,565.7	239.5	-570.3	---	---
S96T000724		1	27.960	111.3	874.0	245.9	-646	---	---
		2	33.581	113.3	823.2	244.0	-627.7	---	---
S96T000725		1	29.651	115.3	843.4	420.1	-537	---	---
		2	33.639	115.3	727.1	420.2	-581.5	---	---
S96T000697	10A	1	18.341	119.3	1,131.6	243.8	-625	---	---
		2	21.369	117.3	1,195.3	422.3	-664.8	---	---
S96T000698		1	14.983	119.8	1,351.9	241.4	-616	---	---
		2	13.947	114.1	1,316.2	239.3	-656.2	---	---
S96T000699		1	14.222	121.7	1,523.4	239.4	-485[QC:e]	---	---
		2	13.827	121.6	1,519.1	420.2	-624.4[QC:e]	---	---

Table A-4. Tank 241-AN-107 Analytical Results: Energetics by DSC. (2 sheets)

Sample Number	Riser Number	Run	Sample Weight	Transition 1		Transition 2		Transition 3	
Sludge			mg	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)
S96T000744	22A	1	19.254	119.9	741.5	252.4	27.7	423.9	-326
		2	16.710	114.6	946.1	250.3	51.1	277.4	-287.4
S96T000745		1	28.032	119.3	960.8	281.6	-318	---	---
		2	6.845	94.0	1,021.0	279.2	-297.8	---	---
S96T000746		1	15.653	115.9	1,229.4	241.5	-565	---	---
		2	17.927	119.6	1,132.2	241.6	-548.3	---	---
S96T000747		1	14.714	119.8	1,109.5	239.5	-579	---	---
		2	14.400	119.8	1,105.2	239.5	-575.4	---	---
S96T000700	10A	1	18.861	113.6	1,195.6	241.7	-556	---	---
		2	20.083	114.0	1,032.7	239.8	-476.9	---	---
		3	19.77	117.5	1,090.6	241.8	-580.8	---	---
S96T000701		1	16.717	119.7	1,106.1	239.7	-550	---	---
		2	16.542	119.5	1,247.9	239.4	-510.3	---	---
S96T000707		1	40.791	113.3	1,095.5	280.5	-359	---	---
		2	35.070	113.3	1,165.1	280.1	-323.3	---	---
S96T000708		1	28.502	119.3	859.9	273.9	-383	---	---
		2	24.290	119.3	1,127.8	273.8	-390.5	---	---

Note:

ΔH = change in enthalpy (negative sign denotes exothermic reaction).

Table A-5. Tank 241-AN-107 Analytical Results: Bulk Density. (2 sheets)

Sample Number	Riser Number	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)
Supernatant		g/mL	g/mL	g/mL	g/mL	%
S96T000719	22A	1.360	---	---	1.38	0.605
S96T000720		1.380	---	---		
S96T000721		1.370	---	---		
S96T000661	10A	1.380	---	---		
S96T000662		1.400	---	---		
S96T000663		1.380	---	---		
Sludge		g/mL	g/mL	g/mL	g/mL	%
S96T000744	22A	1.590	---	---	1.45	2.47
S96T000745		1.560	---	---		
S96T000746		1.370	---	---		
S96T000747[1]		1.367	1.346	1.357		
S96T000700		1.340	---	---		
S96T000701		1.390	---	---		
S96T000707		1.570	---	---		
S96T000708		1.530	---	---		

Table A-5. Tank 241-AN-107 Analytical Results: Bulk Density. (2 sheets)

Sample Number	Riser Number	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)
Settled solids		g/mL	g/mL	g/mL	g/mL	%
S96T000738	22A	1.510	---	---	1.48	0.638
S96T000739		1.470	---	---		
S96T000695	10A	1.480	---	---		
S96T000696		1.470	---	---		

Note:
[1]Specific gravity

Table A-6. Tank 241-AN-107 Analytical Results: Volume Percent Centrifuged Solids.

Sample Number	Riser Number	Result	Overall Mean	RSD (Mean)
Solids		Volume % Solids	Volume % Solids	%
S96T000738	22A	63.00	60.6	2.8
S96T000739		56.10		
S96T000695	10A	63.30		
S96T000696		60.00		

APPENDIX B

DOUBLE-SHELL TANK 241-AN-107

HISTORICAL SAMPLING

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B.0 HISTORICAL SAMPLING

Historically, waste samples have been analyzed to attempt to characterize the waste composition within the tank. Data have been compiled for samples obtained from the 1980s to the present. Data have been located for tank 241-AN-107 samples reported from October 10, 1980 through May 16, 1990. The data were obtained from Rockwell Hanford Operations internal letters and Westinghouse Hanford Company internal memoranda.

B.1 Description of the 1980 Sampling Event

Two samples of the caustic heel in tank 241-AN-107 were received by the Separations Process Development Unit for analysis. Neither a description of the techniques used to extract samples from tank 241-AN-107 nor the sample depths or the riser used to obtain the samples were available. The samples were colorless liquids. Sample #5439 contained rust-colored solids, and sample #5440 contained only a trace of solids (Herting 1980). The samples were filtered and the filtrate analyzed.

B.1.1 Sample Handling and Analysis

The samples were filtered and all analyses were performed on the resulting filtrates. Total alpha and total beta analyses were also performed on the filtered solids. No other sample handling data were available.

The results of the analyses are shown in Table B-1. The radiation dose rate for both samples was zero and the levels of alpha, beta, and gamma radiation were below the detection limits of WHC Analytical Laboratories.

Table B-1. Supernatant Samples.[1,2] (2 sheets)

Waste Tank 241-AN-107		
Analyses of Waste Tank Samples from Tank 107-AN		
October 10, 1980		
Component	Sample #5439	Sample #5440
	Lab Result	Lab Result
Physical Data		
Vis-OTR	Colorless liquid with rust colored solids	Colorless liquid with a trace of solids
pH	12.3	12.2

Chemical Analysis		
OH^-	0.229 M	0.243 M
NO_2^-	0.258 M	0.258 M
Radiological Analysis		
Total alpha	< 1.73E-04 μCi/L	< 1.43E-04 μCi/L
Total beta	< 5.08E-04 μCi/L	< 1.03E-04 μCi/L
Total alpha (solids)	< 8.64E-05 μCi/L	< 1.50E-04 μCi/L
Total beta (solids)	< 5.25E-04 μCi/L	< 4.89E-04 μCi/L

Note:
[1]Herting (1980)
[2]The reliability of these data is questionable due to the lack of proper QC documentation.

B.2 Description of the 1984 Sampling Event

Two samples were received by the Chemical Laboratory Unit from tank 241-AN-107. The sampling techniques, procedures, and sampled riser were not available from records, but it was noted that the samples were taken from 0.6 m (24 in.) below the waste surface (#R8843) and 1.2 m (48 in.) below the waste surface (#R8844). The samples were noted as being a dark material, but a visual assessment of the solids was not available (Jansky 1984).

B.2.1 Sample Handling and Analysis

Aliquots of each sample were taken and submitted for analysis. The results showed that the two samples were virtually identical in composition with regard to the components analyzed. Sample R8843 was used for a batch boildown at reduced pressure. No other sample handling data were available.

The results for these samples are located in Table B-2. It was noted in the literature that the samples appeared to contain no solids, though the dark nature of the samples made it impossible to be certain.

Table B-2. Supernatant Samples.[1,2]

Waste Tank 241-AN-107		
Tank 241-AN-107 Samples		
April 23, 1984		
Component	Sample #8843	Sample #8844
	Lab Result	Lab Result
Chemical Analysis		
HEDTA	0.040 M	0.037 M
EDTA	0.038 M	0.036 M
TOC	46.0 g/L	44.4 g/L
NO_3^-	0.84 M	0.81 M
NO_3^-	4.08 M	3.98 M
Boildown Analysis of Sample #8843		
Pressure (Torr)	% Waste Volume Reduction	Temperature (°C)
60	0	49.6
60	10	50.6
60	20	52.0
63[3]	25[4]	52.4
63	30	52.4

Notes:

[1]Jansky (1984)

[2]The reliability of these data is questionable due to the lack of proper QC documentation.

[3]Pressure fluctuating due to foaming

[4]Apparent nucleation

B.3 Description of the 1985 Sampling Event

Four samples were taken from tank 241-AN-107, though the report only contained information regarding two of the samples. The sampling techniques, procedures, and sampled riser were not available from records, but it was noted that three of the samples were taken from 4.5 m (15 ft) from the bottom of the tank (#R3635, #R3636, and #R3637) and one near the bottom of the tank (#R3638). The samples from tank 241-AN-107 were part of eight samples examined, so precise details of the visual assessment of the sample for tank 241-AN-107 are not well defined. The report noted that the samples varied from opaque coffee-black to translucent brilliant red. It was noted that these colors are indicative of the formation of complexed organometallic species in the waste.

Two reports were used to present the sample results from tank 241-AN-107. The first report on February 28, 1985 (Bratzel 1985a) contained the supernatant results from the two samples. The second report on March 14, 1985 (Bratzel 1985b) contained the solids analysis.

B.3.1 Sample Handling and Analysis

Both reports indicated matching analysis procedures for the samples. The supernatant samples were centrifuged to separate any suspended solids and submitted for analysis to WHC Analytical Laboratories. Aliquots were also submitted to the Analytical Process Development Unit for characterization of organics as well as plutonium and americium concentrations.

After percent solids on the bottom slurry sample were obtained, the aliquots were centrifuged and the centrate separated and analyzed. The centrifuged solids were weighed, dried at 110 °C, and dissolved in a 12 M HNO_3/0.2 M hydrofluoric acid wash. The acid wash from the centrifuged solids was analyzed and reported.

The results for these samples are located in Tables B-3, B-4, and B-5.

B.4 Description of the 1986 Sampling Event

A sample was tested to determine the hydroxide concentration of the complexed concentrate waste in tank 241-AN-107. No information concerning the sampling event or the techniques used to obtain this sample was given in the historical records (Herting 1986).

Table B-3. Complex Concentrate Supernatant Sample #R3637.[1,2] (2 sheets)

Waste Tank 241-AN-107		
Complex Concentrate Supernatant Analysis Results		
Sample Depth - 15 feet from tank bottom		
February 28, 1985		
Component	Lab Value	Lab Unit
Physical Data		
SpG	1.41	g/mL
Chemical Analysis		
Fe	3.31E-02	M
Al	1.98E-01	M
Mg	1.04E-03	M
Na	1.01E+01	M
Mo	< 6.21E-04	M
Cr	4.42E-03	M
Zn	6.23E-04	M
Mn	6.44E-03	M
Cd	5.45E-04	M
Ca	1.73E-02	M
Cu	4.15E-04	M
Pb	1.82E-03	M
K	4.39E-02	M
Zr	7.98E-04	M
Si	< 8.00E-04	M
Ba	5.03E-05	M
La	2.77E-04	M
Ni	8.80E-03	M
OH^-	< 4.00E-02	M
Cl^-	4.10E-02	M
F^-	< 2.22E-01	M
NO_3^-	3.54E+00	M
NO_2^-	8.29E-01	M

Table B-3. Complex Concentrate Supernatant Sample #R3637.[1,2] (2 sheets)

Waste Tank 241-AN-107		
Complex Concentrate Supernatant Analysis Results		
Sample Depth - 15 feet from tank bottom		
February 28, 1985		
Component	Lab Value	Lab Unit
CO_3^{2-}	1.27E+00	M
PO_4^{3-}	2.33E-02	M
SO_4^{2-}	6.33E-02	M
TOC	4.43E+01	g/L Carbon
Radiological Analysis		
U	1.07E-01	g/L
$^{239/240}Pu$[3]	1.08E+02	µCi/L
$^{239/240}Pu$[4]	(8.9 + 0.28)E+01	µCi/L
^{241}Pu[4]	(7.4 + 0.3)E+02	µCi/L
^{241}Am[3]	1.11E+02	µCi/L
^{241}Am[4]	(1.30+0.14)E+02	µCi/L
^{60}Co	8.59E+02	µCi/L
^{137}Cs	4.50E+05	µCi/L
^{154}Eu	2.69E+03	µCi/L
^{155}Eu	5.31E+03	µCi/L
$^{89/90}Sr$	1.48E+05	µCi/L

Notes:

[1]Bratzel (1985a)

[2]The reliability of these data is questionable due to the lack of proper QC documentation.

[3]Analytical Laboratory

[4]Analytical Process Development Unit

Table B-4. Complex Concentrate Sludge Sample #R3638.[1,2] (2 sheets)

Waste Tank 241-AN-107		
Complex Concentrate Liquid Analysis Results		
Sample Depth - Tank bottom		
February 28, 1985		
Component	Lab Value	Lab Unit
Physical Data		
SpG	1.35	g/mL
Chemical Analysis		
Fe	5.26E-02	M
Al	2.11E-01	M
Mg	9.14E-04	M
Na	9.80E+00	M
Mo	< 6.21E-04	M
Cr	5.14E-03	M
Zn	7.76E-04	M
Mn	< 2.42E-02	M
Cd	< 1.48E-03	M
Ca	1.59E-02	M
Cu	3.43E-04	M
Pb	< 2.60E-03	M
K	4.25E-02	M
Zr	< 3.35E-03	M
Si	5.26E-04	M
Ba	2.42E-04	M
La	1.01E-03	M
Ni	8.16E-03	M
OH^-	< 6.58E-02	M
Cl^-	< 5.93E-02	M
F^-	< 1.86E-01	M
NO_3^-	3.51E+00	M
NO_2^-	7.76E-01	M
CO_3^{2-}	1.17E+00	M
PO_4^{3-}	2.07E-02	M

Table B-4. Complex Concentrate Sludge Sample #R3638.[1,2] (2 sheets)

Waste Tank 241-AN-107		
Complex Concentrate Liquid Analysis Results		
Sample Depth - Tank bottom		
February 28, 1985		
Component	Lab Value	Lab Unit
Chemical Analysis		
SO_4^{2-}	< 1.81E-01	M
TOC	3.88E+01	g/L Carbon
Radiological Analysis		
U	1.72E-01	g/L
$^{239/240}Pu$[3]	1.22E+02	μCi/L
$^{239/240}Pu$[4]	(8.6 + 0)E+01	μCi/L
^{241}Pu[4]	(3.3 + 0.18)E+02	μCi/L
^{241}Am[3]	8.75E+02	μCi/L
^{241}Am[4]	(1.35+0.2)E+02	μCi/L
^{137}Cs	3.76E+05	μCi/L
$^{89/90}Sr$	1.64E+05	μCi/L

Notes:

[1]Bratzel (1985a)
[2]The reliability of these data is questionable due to the lack of proper QC documentation.
[3]Analytical Laboratory
[4]Analytical Process Development Unit

Table B-5. Complex Concentrate Sludge Sample #R3638.[1,2] (2 sheets)

Waste Tank 241-AN-107				
Complex Concentrate Solids Analysis Results				
Sample Depth - Tank bottom				
March 14, 1985				
Component	Total Solids Characterization[3]		Acid Soluble Fraction[4]	
	Lab Value	Lab Unit	Lab Value	Lab Unit
Chemical Analysis				
Fe	1.02	wt%	1.37	wt%
Al	2.51	wt%	0.38	wt%
Cr	0.11	wt%	0.16	wt%
Ni	4.7E-02	wt%	4.4E-02	wt%
Si	< 1.6E-02	wt%	0.86	wt%
Pb	N/A	wt%	4.0E-02	wt%
Zn	9.4E-03	wt%	1.1E-02	wt%
Zr	4.7E-02	wt%	4.4E-02	wt%
Ca	0.12	wt%	0.56	wt%
La	< 1.6E-02	wt%	< 0.03	wt%
Mg	5.4E-02	wt%	0.31	wt%
Cu	3.9E-03	wt%	< 6.5E-03	wt%
Mo	< 5.4E-02	wt%	< 0.16	wt%
Cd	< 1.6E-02	wt%	< 6.5E-03	wt%
Mn	0.16	wt%	< 0.20	wt%
Ba	< 3.9E-03	wt%	0.23	wt%
Hg	N/A	wt%	1.1E-02	wt%
Na	2.8E+01	wt%	N/A	wt%
Pd	< 6.2E-02	wt%	N/A	wt%
Ce	< 5.4E-02	wt%	N/A	wt%
K	< 3.1E-01	wt%	N/A	wt%
Sr	< 8.0E-03	wt%	N/A	wt%
Nd	< 3.1E-02	wt%	N/A	wt%

Table B-5. Complex Concentrate Sludge Sample #R3638.[1,2] (2 sheets)

Waste Tank 241-AN-107				
Complex Concentrate Solids Analysis Results				
Sample Depth - Tank bottom				
March 14, 1985				
Component	Total Solids Characterization[3]		Acid Soluble Fraction[4]	
	Lab Value	Lab Unit	Lab Value	Lab Unit
Chemical Analysis				
Cl	< 0.43	wt%	< 0.98	wt%
CO_3	2.43[5]	wt%	N/A	wt%
U	1.07E-01	wt%	5.6E-02	wt%
TOC	6.65[5]	wt%	9.50	wt%
Radiological Analysis				
^{60}Co	N/A	μCi/L	0.83	μCi/L
^{137}Cs	4.16E+02	μCi/L	4.1E-02	μCi/L
$^{89/90}$Sr	2.84E+02	μCi/L	0.72	μCi/L
Pu	0.312	μCi/L	0.26	μCi/L
Am	1.48	μCi/L	0.76	μCi/L

Notes:

[1]Bratzel (1985b).

[2]The reliability of these data is questionable due to the lack of proper QC documentation.

[3]Dissolved solids fraction (fuming H_2SO_4).

[4]Acid leached fraction (12<u>M</u> HNO_3/0.1<u>M</u> hydrofluoric acid). Represents incomplete solids dissolution.

[5]Determined by direct analysis of dried solids.

B.4.1 Sample Handling and Analysis

The report noted that the results of the analysis showed that no "free" hydroxide or NaOH are present in complexed concentrate waste. The report also indicated that the calculated hydroxide concentration precisely matched the analyzed sample at 0.44<u>M</u>. No other specifics on the sample or the sampling event were noted in the historical report. Table B-6 contains results for analytical analysis of this sample.

Table B-6. Complex Concentrate Waste Sample #R8650.[1,2]

Waste Tank 241-AN-107		
Analysis of direct sample of CC waste from tank 107-AN		
December 4, 1986		
Component	Lab Value	Lab Unit
Physical Data		
pH	12.2	
Chemical Analysis		
OH	< 0.05	M
CO_3	1.11	M
NO_2	0.83	M
NO_3	2.7	M
SO_4	0.06	M
PO_4	0.03	M
Na	9.15	M
Al	0.14	M
Fe	0.033	M
TOC	39.1	g/L
Radiological Analysis		
Total Alpha	689	µCi/L

Note:

[1]Herting (1986)

[2]The reliability of these data is questionable due to the lack of proper QC documentation.

B.5 Description of the 1988 Sampling Event

Two slurry samples from tank 241-AN-107 were analyzed for viscosity and percent settled solids in support of caustic addition requirements and concentrated complexant pretreatment studies. No information concerning the sampling event or the techniques used to obtain this sample were given in the historical records (Prignano 1988).

B.5.1 Sample Handling and Analysis

The percent settled solids was determined by solids formation in an ice bath. An aliquot of a well mixed slurry sample (approximately 10 mL) was placed in a graduated cone and the cone placed in an ice bath after cooling initially to room temperature. The test did not result in any solids formation.

Density of the supernatant was determined by allowing the samples to warm to room temperature.

Viscosities were determined at three temperatures (30, 18, and 7°C) for each sample with a Haake viscometer, model number M-150. The viscometer was calibrated and the samples analyzed. The decrease in viscosity with increasing shear rate indicated a pseudoplastic liquid. The report indicated that the shear thinning effect was reversible, and the slurry samples regained their original higher viscosity when the shearing was slowed.

The analytical results for these samples are shown in Table B-7.

Table B-7. Viscosity and Percent Settled Solids Determination on Samples #R-8569 and #R-8374.[1,2] (2 sheets)

Waste Tank 241-AN-107					
Viscosity and Percent Settled Solids Determination					
July 6, 1988					
Received: May 17, 1988					
Physical Data					
Component	R-8569		R-8374		
Sample Depth	12 m (40 ft)		16 m (53 ft)		
% solids	0		10		
Description	~110 mL, brown amber color		~100 mL, brown amber		
Supernatant SpG	2.1		1.4		
Viscosity Analysis					
Temperature	16 (RPM) 86.6 (1/sec)	32 (RPM) 173 (1/sec)	64 (RPM) 346 (1/sec)	128 (RPM) 692 (1/sec)	256 (RPM) 1385 (1/sec)
Sample R-8569					
30 °C	---	---	6.8	5.8	4.9
18 °C	---	11.0	9.7	8.2	6.9
7 °C	---	16.5	14.1	11.9	---

Table B-7. Viscosity and Percent Settled Solids Determination on Samples #R-8569 and #R-8374.[1,2] (2 sheets)

Sample R-8374					
30 °C	---	---	9.0	8.1	7.4
18 °C	---	14.9	13.6	12.3	---
7 °C	---	23.8	21.5	---	---

Note:

[1]Prignano (1988)

[2]The reliability of these data is questionable due to lack of proper QC documentation.

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