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7. Abstract

This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in tank 241-U-202. This report supports the requirements of Tri-Party Agreement Milestone M-44-08.

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Tank Characterization Report for Single-Shell Tank 241-U-202

Date Published
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Prepared for the U.S. Department of Energy
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EXECUTIVE SUMMARY

Single-Shell tank 241-U-202 is an underground storage tank containing high-level radioactive waste. It is located in the U Tank Farm in the Hanford Site's 200 West Area. The tank was core sampled in May of 1995 and analysis was performed in accordance with the *Tank Safety Screening Data Quality Objective* , WHC-SD-WM-SP-004, Rev. 0 (Babad and Redus 1994). The analysis supports *Hanford Federal Facility Agreement and Consent Order* Milestone M-44-08 (Ecology et al. 1994).

The sixteen tanks in the U tank farm were built between 1943 and 1944. The tanks consist of twelve type II tanks, holding 2,010 kL (530 kgal) each of waste, and four type I tanks, holding 208 kL (55 kgal) each. Tank 241-U-202 is one of the 55,000-gallon tanks and currently contains 18.9 kL (5 kgal) of waste, 15.1 kL (4 kgal) of sludge, and 3.8 kL (1 kgal) of supernatant. Tank 241-U-202 received cladding waste and waste from the Reduction and Oxidation Facility starting in the first quarter of 1956. The tank was removed from service and labeled inactive in 1978. The supernatant was pumped in 1977 and the tank administratively interim stabilized in 1979. Intrusion prevention was completed in 1982. The tank waste level was adjusted in 1978 and 1979.

The Safety Screening Data Quality Objective is the only current data quality objective that applies to the tank 241-U-202 sampling event. The analyses required by that data quality objective included thermogravimetric analysis, differential scanning calorimetry, total alpha

activity, and flammable gas concentration. No exothermic activity was observed for any of the subsamples analyzed. From July of 1987 to July of 1993, the maximum temperature reading was 19 °C (67 °F). The average percent water for all samples was 44.9 wt%, significantly above the 17 wt% safety screening criteria. Total alpha was well within the data quality objective unit of 41 μCi/g. Based on this information, the waste does not appear to have any immediate safety concerns; however, the flammability of the gas in the headspace of the tank is another safety screening consideration, and the analysis of the tank's headspace is presently not available. No concerns, however, were raised from vapor sampling in support of this sampling event.

The current status of tank 241-U-202 is shown in Table ES-1, and a summary of the estimated historical inventory is shown in Table ES-2.

Table ES-1. Tank 241-U-202.

Tank description	
Type:	Single-shell
Constructed:	1943-44
In-service:	1956
Diameter:	6.1 m (20 ft)
Useable depth:	7.7 m (224 in.)
Capacity:	208 kL (55 kgal)
Bottom shape:	Dish
Ventilation:	Passive
Tank status	
Watch list:	Non-watch list
Contents:	Non-complexed waste
Total waste volume:	18.9 kL (5 kgal)
Sludge volume:	15.1 kL (4 kgal)
Supernatant volume:	3.8 kL (1 kgal)
Manual tape surface level:	67 cm (26.5 in.) (February 23, 1995)
Mean temperature: (July 1987 to July 1993)	18.7 °C (65.6 °F)
Integrity category:	Sound
Isolation status	
Interim stabilized:	1979
Intrusion prevention:	1982
Level adjustment:	1979

Table ES-2. Single-Shell Tank 241-U-202
Historical Inventory Estimates Summary.[1]

Physical properties			
Total solid waste	2.36 E+04 kg (4 kgal)		
Heat load	4.75 E-04 kW (2 Btu/h)		
Bulk density	1.56 g/mL		
Void fraction	0.814		
Water wt%	65.3		
Total organic carbon wt% (wet)	0.0		
Chemical constituents			
Analyte	mole/L	ppm	kg
Aluminum	6.26	1.08 E+05	2.56 E+03
Calcium	6.42 E-02	1.65 E+03	39.0
Chromium	6.61 E-03	220	5.20
Total iron	0.471	1.68 E+04	398
Nickel	2.90 E-02	1.09 E+03	25.7
Strontium	1.14 E-02	639	15.1
Si (SiO_3^{-2})	0.144	2.8 E+03	61.0
Sodium	3.57	5.26 E+04	1.24 E+03
Chloride	9.84 E-03	223	5.28
Carbonate	6.42 E-02	2.47 E+03	58.3
Hydroxide	22.7	2.47 E+05	5.84 E+03
Nitrate	0.790	3.14 E+04	741
Nitrite	1.16	3.41 E+04	805
Sulfate	3.30 E-03	203	4.81
Radiological constituents			
Cesium	2.85 E-03 Ci/L	1.83 μCi/g	43.2 Ci
Strontium	2.67 E-03 Ci/L	1.71 μCi/g	40.4 Ci
Plutonium		1.68 μCi/g	15.2 kg
Uranium	0.183 M	2.80 E+04 μg/g	661 kg

Note:

[1]Brevick et al. 1995, *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Area*, WHC-SD-WM-ER-352, Westinghouse Hanford Company, Richland, Washington.

REFERENCES

Babad, H., and K. S. Redus, 1994, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

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

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LIST OF TERMS

CWR	Cladding waste, Reduction-Oxidation Plant
DQO	data quality objectives
DSC	differential scanning calorimetry
RPD	relative percent difference
TCP	tank characterization plan
TGA	thermogravimetric analysis

LIST OF TERMS

BCS	Boeing Computer Services
CWR	REDOX cladding waste
CASS	Computer Automated Surveillance System
cm	Centimeter
ΔH	Change in enthalpy
DQO	Data quality objectives
DSC	Differential scanning calorimetry
ft	Feet
g	Gram
gal	Gallon
HTCE	Historical tank content estimate
ICP	Inductively coupled plasma spectroscopy
IDL	Instrument detection limit
hr	Hour
in.	Inch
J	Joules
kgal	Kilogallon
KHC	ICF Kaiser Hanford Company
kL	Kiloliter
L	Liter
m	Meter
μCi	Microcurie
μg	Microgram
mL	Milliliter
mmol	Millimole
mrad	Millirad
MW	Molecular weight
NA	Not applicable
NC	Not counted
NR	Not reported
ppm	Parts per million
QA/QC	Quality Assurance/Quality Control
REDOX	Reduction-oxidation
RPD	Relative percent difference
TCR	Tank characterization report
TGA	Thermo-gravimetric analysis
TIC	Total inorganic carbon
TLM	Tank layer model
TOC	Total organic carbon
TWINS	Tank Waste Information Network System
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

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1.0 INTRODUCTION

The tank characterization report presents an overview of single-shell tank 241-U-202 and its waste contents based upon an evaluation of historical information and push-mode core sampling and analysis events.

In May of 1995, single-shell tank 241-U-202 was sampled to meet safety screening requirements for Hanford Site nuclear waste tanks (Schreiber 1995, Babad et al. 1994). The analyses of the samples were used to complete this tank characterization report in order to comply with requirements specified in the *Hanford Federal Facility Agreement and Consent Order*, also known as the Tri-Party Agreement (Ecology et al. 1994). The analyses will also provide further information to the Tank Waste Remediation System in terms of tank safety.

The *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 0 (Babad et al. 1994), describes the sampling and analytical requirements used to screen waste tanks with no identified safety issues, tanks such as tank 241-U-202. Both watch-list and non-watch-list tanks are to be sampled and evaluated to identify safety conditions related to the ferrocyanide, organic, flammable gas, and criticality safety issues. The analytical requirements for safety screening are designed to identify a common set of primary analytes and waste characteristics. These include energetics, total alpha activity, moisture, and flammable gas concentrations.

This tank characterization report presents an overview of the tank sampling and analysis effort and contains observations regarding waste characteristics. Chemical information associated with tank 241-U-202, however, is united because the analysis of the waste was governed solely by the Tank Safety Screening Data Quality Objective (DQO), which requires that only limited analyses be performed. As a consequence, the concentration and inventory estimates of individual chemical species are based upon the estimates in *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Area*, WHC-SD-WM-ER-352, Rev. 0 (Brevick et al. 1995).

1.1 PURPOSE

The purpose of this report is to summarize the information about the use and contents of tank 241-U-202. When possible, this information will be used to assess issues associated with safety, operations, environmental, and process development activities. This report also provides a reference point for more detailed information about tank 241-U-202.

1.2 SCOPE

This report presents historical information based on process knowledge and early sampling and analysis. The results from the tank 241-U-202 1995 sample analyses for safety screening are then summarized and presented. The historical information is also compared with the actual waste measurements and recommendations are given based upon the current waste inventory and tank status where possible.

Because the 1995 screening analyses only included differential scanning calorimetry (DSC)/thermogravimetric analysis (TGA) and total alpha measurements, historical information is used for most chemical and radiochemical values. This report does not presently contain information on vapor space sampling and analysis for determination of the composition of tank headspace gases.

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-U-202 based on historical information. The first part of the section details the current condition of the tank, followed by discussions of the tank's background, transfer history, and process sources that contributed to the tank waste, including an estimate of the current contents based on the process history. Events that may be related to tank safety issues, such as potentially hazardous tank contents (ferrocyanide, organics) or off-normal operating temperature (tank damage, chemical reactions), are included. The final part of the section details any surveillance data available for the tank. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the solid/crust layers of the waste (i.e., slurry growth from gas evolution with subsequent burping and collapse or shrinkage resulting from drying). Drywell activity monitoring is noted where anomalies may suggest leaking of nearby tanks. Temperature data are provided to evaluate the heat generating characteristics of the waste.

2.1 TANK STATUS

As of the January 1995, Waste Status Report (Hanlon 1995), tank 241-U-202 contained 18.93 kL (5 kgal) of waste classified as non-complexed with a total depth of approximately 67 cm (26.5 in.). The composition of the waste is shown in Table 2-1.

Table 2-1. Summary Tank Contents Status.

Waste form	kL (kgal)
Total waste	18.9 (5)
Supernatant	3.8 (1)
Drainable interstitial liquid	0 (0)
Drainable liquid remaining	3.8 (1)
Pumpable liquid remaining	0 (0)
Sludge	15.1 (4)
Salt cake	0 (0)

The tank is identified as a low-heat tank, is passively ventilated, and is categorized as sound with interim stabilization and intrusion prevention completed in 1979 and 1982, respectively. Presently, the waste consists of cladding waste from the reduction and oxidation process (CWR). Tank 241-U-202 is not on a watch list.

2.2 TANK DESIGN AND BACKGROUND

The U Tank Farm is a first generation tank farm, built between 1943 and 1944, consisting of twelve, type II, 2,010 kL (530 kgal), 23 m (75 ft) diameter tanks and four, type I, 208 kL (55 kgal), 6.1 m (20 ft) diameter single-shell tanks. As with all first-generation tank farms, equipment to monitor and maintain the waste is sparse. A typical tank contains a manhole and eight risers that provide surface level access to the underground tank. Generally, there are four risers on each side of the tank and a condenser hatchway in the middle of the dome.

Tank 241-U-202 is one of the 208 kL tanks. This tank entered service in the first quarter of 1956. The single-shell tank is constructed of 300-mm (1-ft) thick reinforced concrete with a 6.4-mm (1/4-in.) mild carbon steel (ASTM A283 Grade C) liner on the bottom and sides and a 380-mm (1.25-ft) thick domed concrete top. Tank 241-U-202 has a 15-cm (6-in.) dish bottom, a 0.9-m (3-ft) radius knuckle, and a 7.7-m (18-ft 8-in.) operating depth. The tanks are set on a reinforced concrete foundation. A three-ply cotton fabric waterproofing was applied over the foundation and steel tank. Four coats of primer paint were sprayed on all exposed interior tank surfaces. Tank ceiling domes were covered with three applications of magnesium zincfluorosilicate wash. Lead flashing was used to protect the joint where the steel liner met the concrete dome. Asbestos gaskets were used to seal the manhole in the tank dome. The tanks were waterproofed on the sides and top with tar and gunnite. Each tank was covered with approximately 2.2 m (7.25 ft) of overburden. The U Tank Farm was designed for nonboiling waste with a maximum fluid temperature of 104 °C (220 °F). Tanks 241-U-201 through -204 each cascade to a junction box.

The surface level is monitored through riser 1 with a manual tape. A list of tank 241-U-202 risers, showing the size and relative location, is provided in Table 2-2. Figure 2-1 is a plan view of the riser configuration.

Figure 2-2 shows a cross section of the tank with the approximate waste level as well as a schematic of the tank equipment. tank -U-202 has two construction manholes and eight grade-level risers, of which risers 2 and 6 [300 mm (12 in.) in diameter] are available for use. If used as sampling ports, these risers will give access to two opposite sides of the tank, one by an inlet and the other approximately 180 degrees across. Four tank inlets are available. The inlets are located approximately 4.9 m (191.5 in.) from the tank bottom, as measured at the tank wall. Locations are shown in Figure 2-1.

The single thermocouple tree, located in tank 241-U-202, riser 4 has 11 thermocouples. Elevations are not available for the thermocouples. All thermocouples have been out of service since September of 1993, though temperatures are still taken semiannually (refer to Section 2.4.2). This tank is passively ventilated.

Table 2-2. Tank 241-U-202 Risers.

No.	Dia. (in.)	Description and comments
R1	4	Liquid level reel
R2	12	3" SMP and 4" flange
R3	12	DIP tubes (possibly removed & capped)
R4	4	Temperature
R5	4	WC in caisson
R6	12	OBSV port
R7	12	Dip tubes (possibly removed & capped)
R8	4	Breather filter
N1	3	Blanked in DIV box
N2	3	Blanked in DIV box
N3	3	Capped
N4	3	Capped

Figure 2-1. Tank Riser Location.

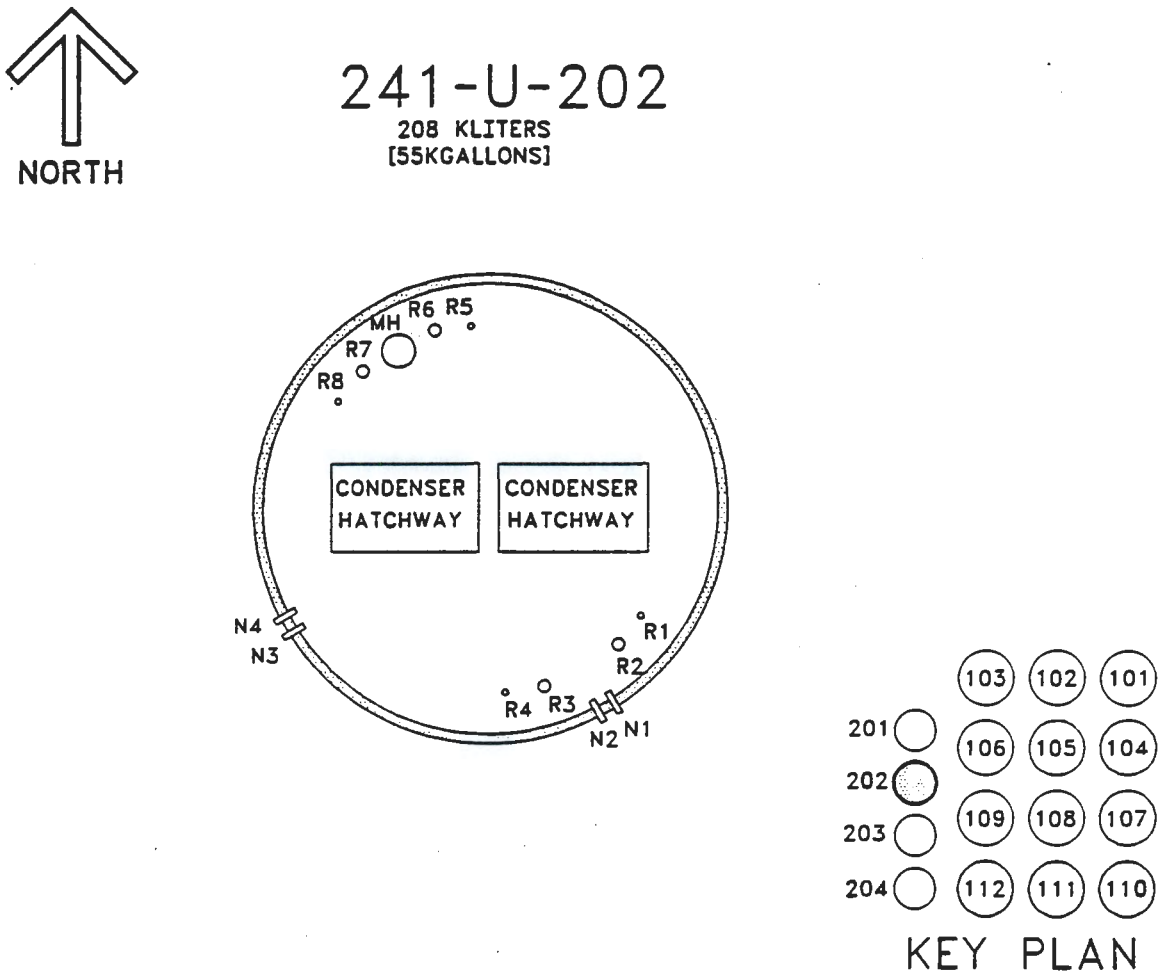
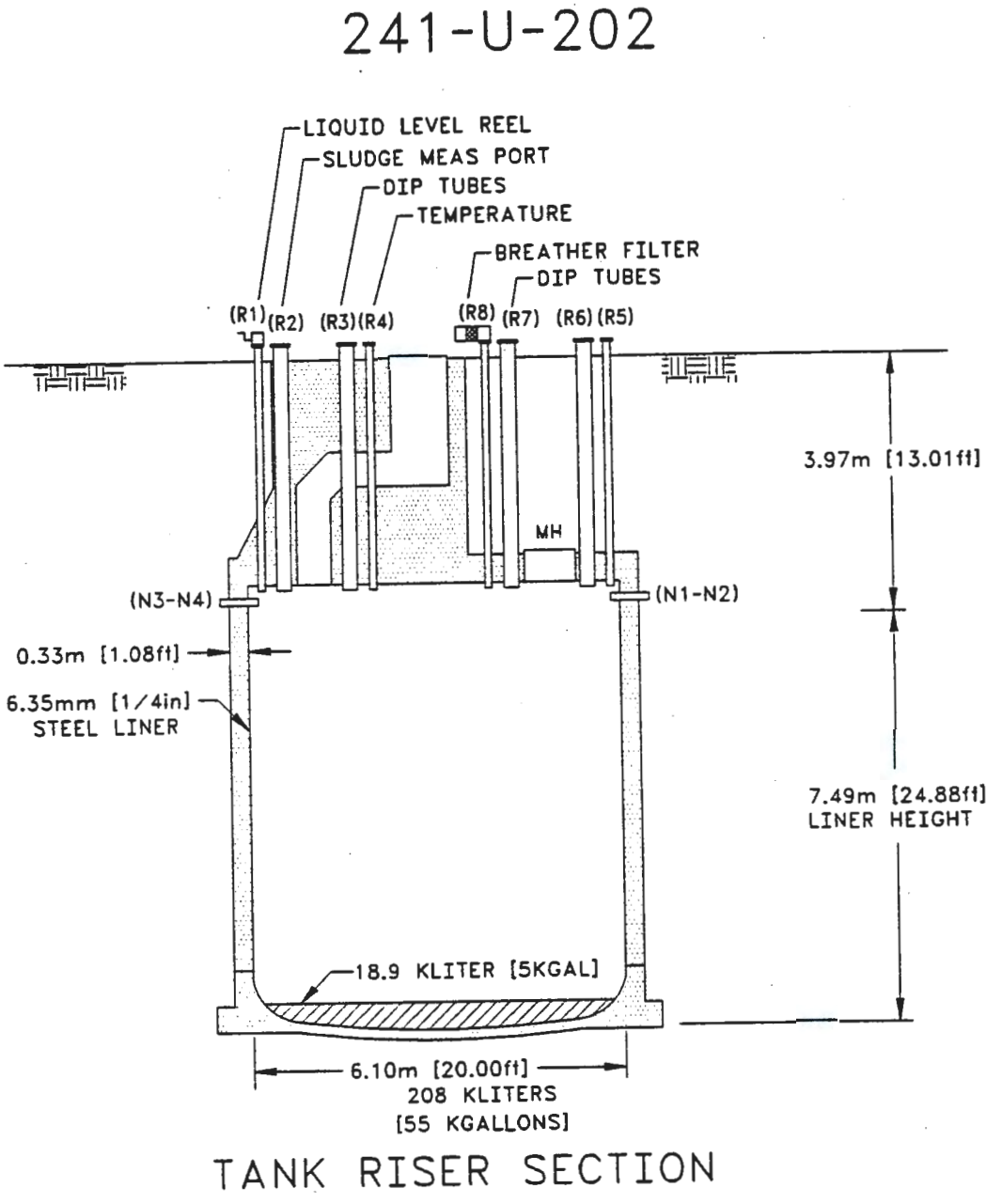


Figure 2-2. Tank Riser Section.



2.3 PROCESS KNOWLEDGE

These sections present the transfer history of tank 241-U-202. Section 2.3.1 and Table 2-3 present the major transfers along with a narrative describing the transfers.

2.3.1 Waste Transfer History

Tank 241-U-202 received no waste from the first quarter of 1944 to the first quarter of 1956. Water was initially added to tank 241-U-202 from an unknown source sometime during the first quarter of 1956. Also during the first quarter of 1956, supernatant was transferred to tank 241-U-202 from tank 241-U-110. Tank 241-U-202 received CWR during an unspecified time period as one of the last transfers. In 1977, the tank was pumped to an unspecified location. The transfer history for this is incomplete and unavailable.

Tank 241-U-202 was removed from service and declared inactive in the fourth quarter of 1978. It is categorized as sound, was interim stabilized in 1979, and intrusion prevention was completed in 1982. Presently, the waste contained in tank 241-U-202 has been classified as non-complexed.

Table 2-3. Tank 241-U-202 Major Transfers.*

Year: Qtr	Waste received by tank 241-U-202				Waste transferred out of tank 241-U-202		
	Transaction source	# T	Waste type	Waste amount kL (kgal)	# T	X TF (waste type)	Waste amount kL (kgal)
1956:1	Unknown	1	WTR	38 (10)	-	-	-
1956:1	U-110	1	SU	155 (41)	-	-	-
1977	--	--	--	--	Unknown	CWR	170 (45)

*Compiled from historical information provided in Brevick et al. 1995.
WTR = Water
SU = Supernatant. Liquid considered free of contamination to the extent it could be pumped to a crib.

2.3.2 Historical Estimation of Tank Contents

Based on the historical tank content estimate (Brevick et al. 1995) and the tank layer model (Agnew et al. 1994), tank 241-U-202 contains solely 15.1 kL (4 kgal) of CWR covered by a 3.7-kL (1-kgal) supernatant layer. Figure 2-3 shows a graphical representation of the estimated waste type(s) and volume(s) for the tank layer(s). The CWR waste type should contain large amounts of sodium, aluminum, uranium, nitrites, nitrates, and hydroxide. Also present will be quantities of silicates, strontium, and cesium (of which the amount of strontium is significantly larger than the amount of cesium). Therefore, this layer will have a slight activity. To further identify CWR, the following constituents will be totally or relatively absent from the waste. These constituents include but are not limited to, iron, bismuth, nickel, lead, and total organic carbon. The supernatant layer should consist mostly of water, but further evaluation of the remaining constituents cannot be performed because of the absence of data on this layer. Table 2-4, based on historical information, shows an estimate of the expected waste constituents and their concentrations.

2.4 SURVEILLANCE DATA

Tank 241-U-202 surveillance consists of liquid and solid surface-level measurements and temperature monitoring inside the tank waste and vapor space.

Liquid-level measurements are used to determined if there may have been a major leak from a tank. Solid surface-level measurements provide an indication of physical changes and consistency of the solid layers of a tank. Drywells located around the perimeter of the tank are used to detect increased radioactivity resulting from a leak to the soil.

2.4.1 Surface Level Readings

Tank 241-U-202's surface level is monitored daily with a manual tape. A graphical representation of these measurements can be found in Figure 2-4. The plot has two inaccurate data points that have been superseded by apparent reruns. Also, several unexplained readings exist. Tank 241-U-202 does not have a liquid observation well or drywells.

In 1978, the tank was labeled inactive and had a level adjustment. In 1979, a second level adjustment was performed, the supernatant was pumped from the tank, and the tank was interim stabilized. In 1982, tank intrusion prevention was performed.

Figure 2-3. Tank Layer Model (Not to Scale).

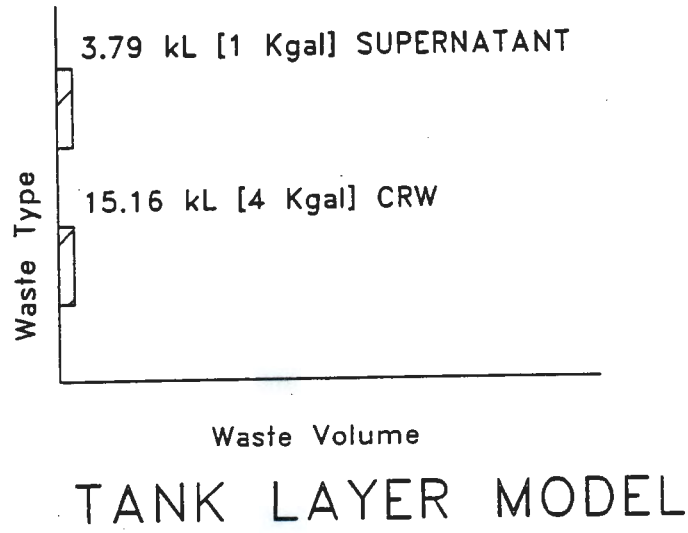


Table 2-4. Historical Tank Inventory Estimate.[1] (2 sheets)

Solids composite inventory estimate[2]			
Physical properties			
Total solid waste	2.36 E+04 kg (4 kgal)		
Heat load	4.75 E-04 kW (2 Btu/hr)		
Bulk density	1.56 (g/cc)		
Void fraction	0.814		
Water wt%	65.3		
TOC wt% C (wet)	0		
Chemical constituents	mole/L	ppm	kg
Na^{+1}	3.57	5.26 E+04	1.24 E+03
Al^{+3}	6.26	1.08 E+05	2.56 E+03
Fe^{+3} (total Fe)	0.471	1.68 E+04	398
Cr^{+3}	6.61 E-03	220	5.20
Bi^{+3}	0	0	0
La^{+3}	0	0	0
Ce^{+3}	0	0	0
Zr (as $ZrO(OH)_2$)	0	0	0
Pb^{+2}	0	0	0
Ni^{+2}	2.90 E-02	1.09 E+03	25.7
Sr^{+2}	1.14 E-02	639	15.1
Mn^{+4}	0	0	0
Ca^{+2}	6.42 E-02	1.65 E+03	39.0
K^{+1}	0	0	0
OH^{-1}	22.7	2.47 E+05	5.84 E+03
$NO3^{-1}$	0.790	3.14 E+04	741
$NO2^{-1}$	1.16	3.41 E+04	805
$CO3^{-2}$	6.42 E-02	2.47 E+03	58.3
$PO4^{-3}$	0	0	0
$SO4^{-2}$	3.30 E-03	203	4.81
Si (as SiO_3^{-2})	0.144	2.58 E+03	61.0
F^{1}	0	0	0
Cl^{-1}	9.84 E-03	223	5.28
$C_6H_5O_7^{-3}$	0	0	0
$EDTA^{-4}$	0	0	0
$HEDTA^{-3}$	0	0	0
NTA^{-3}	0	0	0
$glycolate^{-1}$	0	0	0
$acetate^{-1}$	0	0	0
$oxalate^{-2}$	0	0	0
DBP	0	0	0
NPH	0	0	0
CCl_4	0	0	0
hexone	0	0	0

Table 2-4. Historical Tank Inventory Estimate.[1] (2 sheets)

Solids composite inventory estimate[2]			
$FE(CN)_6^{-4}$	0	0	0
Radiological constituents			
Plutonium		1.68(μCi/g)	15.2(kg)
Uranium	0.183 (M)	2.80 E+04(μg/g)	661 (kg)
Cesium	2.85 E-03 (Ci/L)	1.83 (μCi/g)	43.2 (Ci)
Strontium	2.67 E-03 (Ci/L)	1.71 (μCi/g)	40.4 (Ci)

[1]Agnew, S. F., P. Baca, R. Corbin, T. Duran, and K. Jurgenson, 1994, *Tank Layer Model (TLM) Spreadsheet*, Los Alamos National Laboratory, Los Alamos, New Mexico.

[2]Composite inventory excludes supernatant, diatomaceous earth, and cement. Unknowns in tank inventory are assigned by Tank Layer Model.

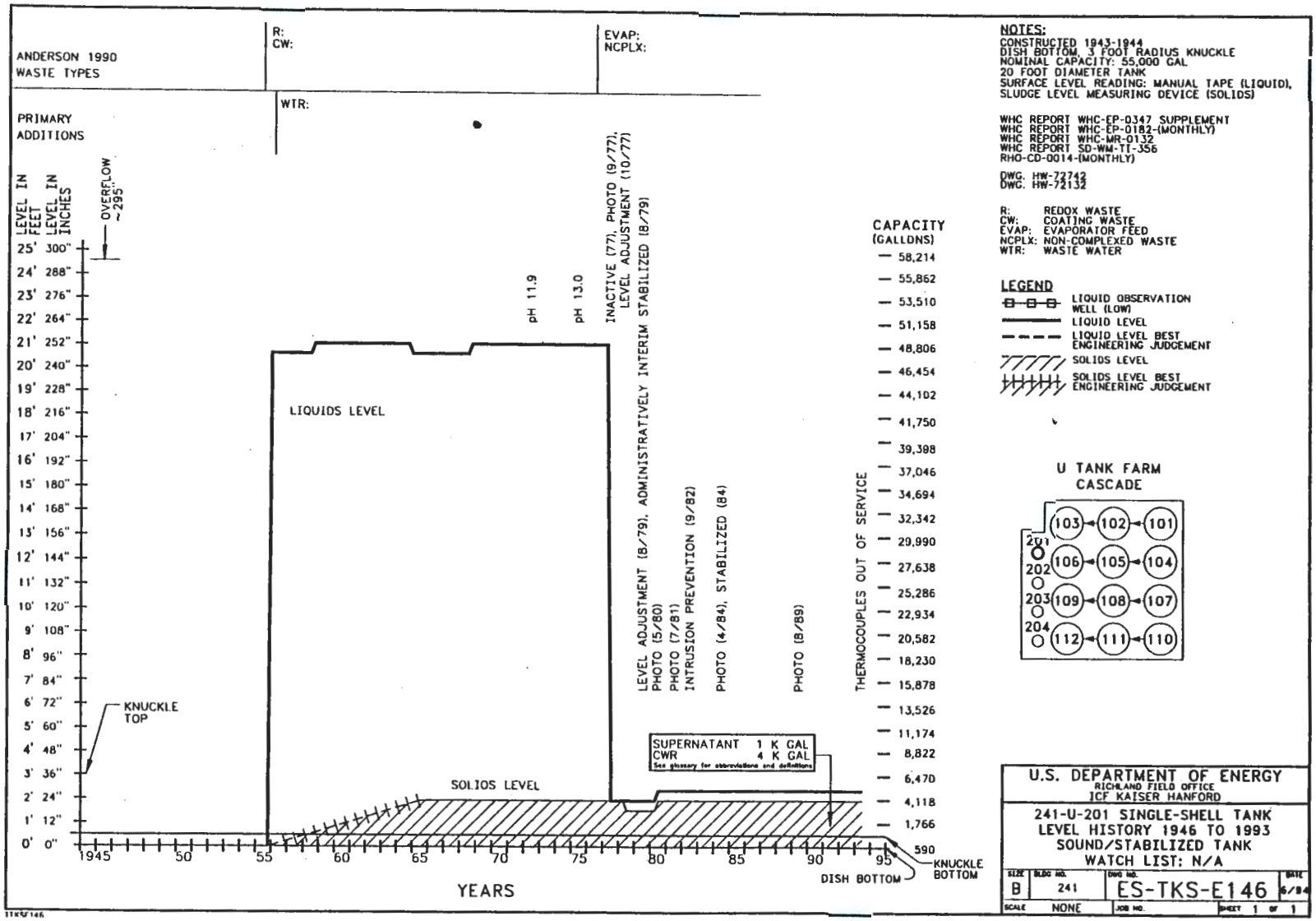


Figure 2-4. Tank Level History.

2.4.2 Internal Tank Temperatures

Tank 241-U-202 has a single thermocouple tree with 11 thermocouples to monitor the waste temperature. Elevations are not available for all the thermocouples. All of the 11 thermocouples have similar temperature readings.

The mean temperature of the first available electronic thermocouple readings was 19 °C (65.6 °F). From July 1987 to July 1993, the median temperature was 18 °C (65 °F), the minimum temperature was 14 °C (57 °F), and the maximum temperature was 19 °C (67 °F). Tank 241-U-202 is scheduled to have temperature data taken semiannually. Plots of the thermocouple readings can be found in the Supporting Documents to WHC-SD-WM-ER-352, Rev. 0 (Brevick et al. 1995). Plots of the weekly high thermocouple readings are provided in Figure 2-5.

2.4.3 Tank 241-U-202 Photographs

A picture has been assembled of the tank 241-U-202 interior from photographs taken on November 24, 1988. The photographs show a dark yellow supernatant surface surrounded by a light yellow sludge that is peppered with dark material. A yellowish-green residue is present on the lower tank walls. A manual tape, temperature probe, and a thermowell are visible in the photograph. The tank contains approximately 18.9 kL (5 kgal) of waste with a total depth of approximately 67 cm (26.5 in). Figure 2-6 should represent the current tank contents.

Figure 2-5. Tank 241-U-202 Weekly High Temperature Plot.

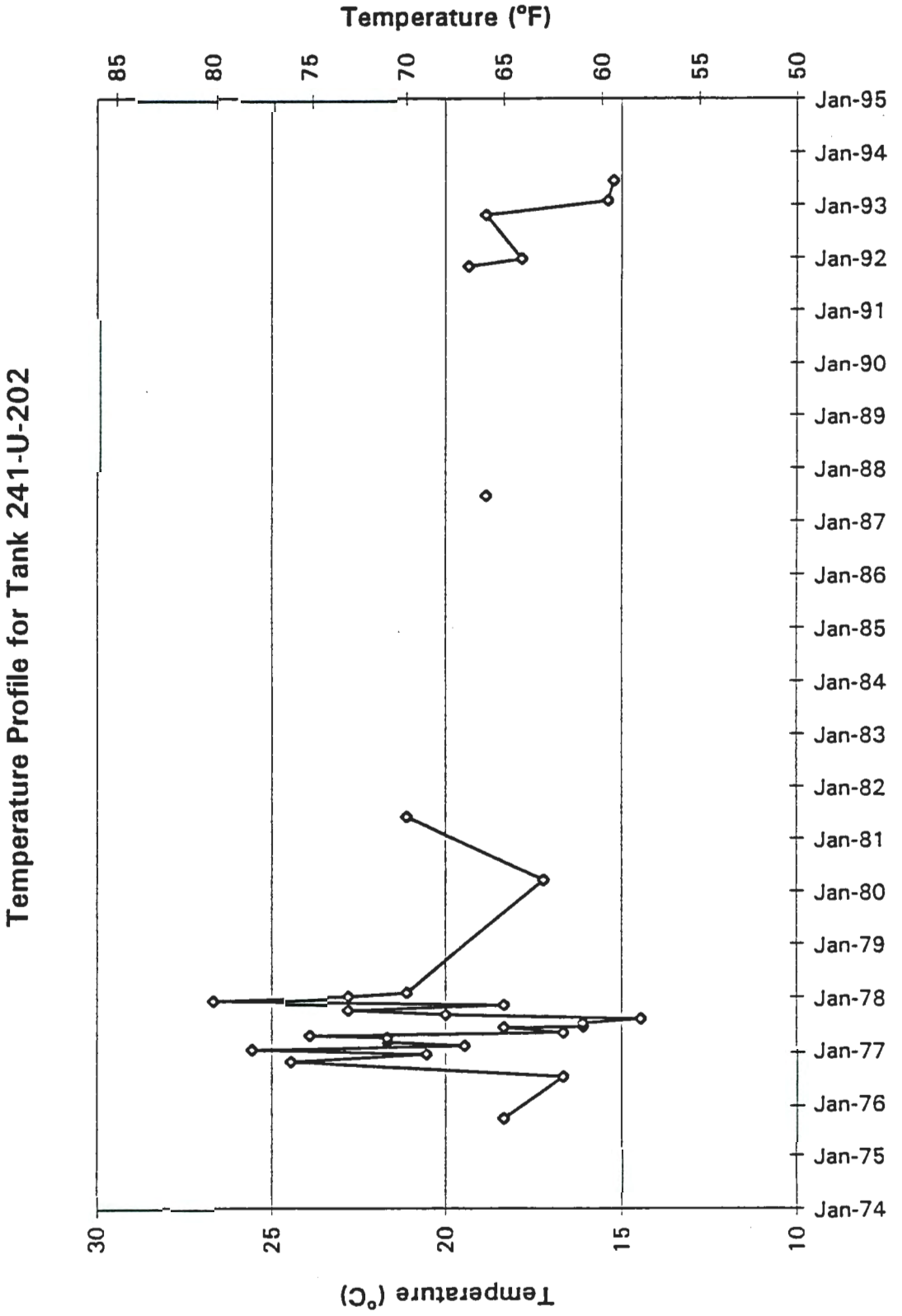
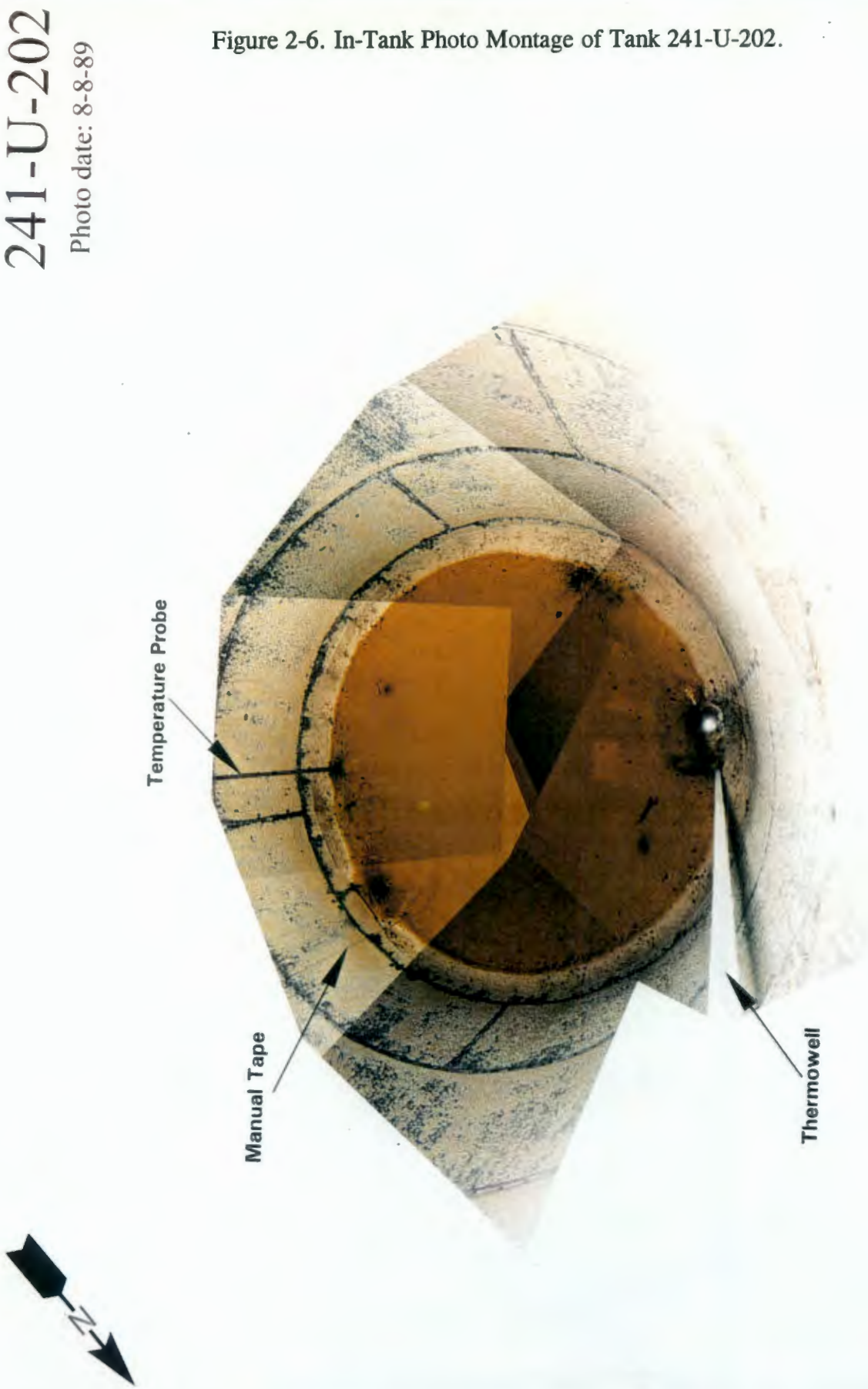


Figure 2-6. In-Tank Photo Montage of Tank 241-U-202.



3.0 TANK SAMPLING OVERVIEW

Because of the applicability and limited information on earlier sampling and analysis events, this chapter focuses primarily on the 1995 sampling event, the handling of the samples after the sampling event, and the analytical procedures performed on the samples.

3.1 DESCRIPTION OF 1975 SAMPLING EVENT

Historically, SST waste samples have been analyzed to characterize the supernatant, sludge, and/or salt cake in each tank. Data were compiled for samples obtained from the late 1950s to the present. Data have been located for one tank 241-U-202 sample obtained on September 25, 1975 and analyzed on October 23, 1975. These data are presented in Appendix A.

A description of the technique used to extract a sample from tank 241-U-202 is not available. The sample appears to be a liquid that was analyzed for primary cations and anions. The supernatant sample was a clear and yellow liquid. Most of the supernatant was pumped from the tank when it was stabilized in 1976, therefore the sample is not believed to accurately represent the tank waste. Therefore, the data will not be used for comparisons or for drawing conclusions.

3.1.1 Sample Handling

The sample (T-8245) was a clear yellow liquid with no solids. No procedure or explanation for the sample is available.

3.1.2 Sample Analysis

The sample was a liquid sample reported to consist of hydroxide, aluminum, sodium, nitrate, nitrite, phosphate, chloride, carbonate, and fluoride. The sample also contained a fair amount of strontium. The sample gives no indication of the tank's primary waste receiving assignments.

3.2 DESCRIPTION OF 1995 SAMPLING EVENT

Tank 241-U-202 was sampled on March 22, 1995; March 29, 1995; and March 30, 1995 using the core sample truck. Core sampling, in the push-mode, was selected as the sampling method for tank 241-U-202. Three two-segment cores were retrieved. Core 75 was collected from riser 2 on March 22 and delivered to the 222-S Laboratory on March 23, 1995. The two-segments of the core were labeled

Samples 95-050 and 95-051, respectively. Core 78, also a two-segment core, was collected from riser 6 on March 29, 1995 and delivered to the 222-S Laboratory on March 30, 1995. The segments were labeled Samples 95-057 and 95-058. As specified in WHC-SD-WM-SP-004, Rev. 0 (Babad et al. 1994), the risers to be analyzed were widely spaced. The third sample, also a two segment core, was a field blank and was collected and delivered to the 222-S Laboratory on March 30, 1995. For a description of core sampling procedures, refer to the *Tank Characterization Reference Guide*, WHC-SD-WM-TI-648, Rev. 0 (De Lorenzo et al. 1994).

3.2.1 Sample Handling

Three two-segment core samples were taken from tank 241-U-202. Two of the samples were analyzed and the other was archived for future needs. This archived sample was the first sample taken from riser 6 (Sample 95-055 and Sample 95-056) and is also referred to as the field blank. Table 3-1 details the sample recovery location and laboratory numbering system for these samples. No problems were encountered during the sampling.

The following sample descriptions were taken from WHC-SD-WM-DP-110, Rev. 0 (Jo 1995). Core 75, Segment 1, Sample 95-050, was extruded on April 3, 1995, with the total amount of solid material recovered being 119 g and drainable liquid recovered being 155 g. There was no liquid in the liner. The drainable liquid was yellow and the solid sample was light yellow with some black specks and large crystals. The first 3 inches of the solid sample retained the shape of the sampler. The final 2 inches "melted" on the extrusion tray. The solid material was damp and creamy. Analysis of the drainable liquids was run after settling the solids.

Core 75, Segment 2, Sample 95-051 was extruded on April 3, 1995 with the total amount of solid material recovered being 122 g, and drainable liquid recovered being 137 g. There were nine grams of fluid in the liner. The drainable liquid was yellow and the solid sample was a light-yellow sludge material. All other solid material was crystalline. The solid sample was divided into a crystalline half segment and a sludge half segment. All archiving requirements, in accordance with the tank characterization plan (TCP), were performed on the unfiltered liquid and solid subsamples, while all analysis of the drainable liquid was run after settling the solids. The liner liquid was not analyzed because the sample was too small.

Core 78, segment 1, sample 95-057 was extruded on April 4, 1995 , with the total amount of solid material recovered being 312 g, drainable liquid recovered being 42 g. No liner fluid was found. The drainable liquid was yellow and the solid sample was light yellow. The first 6 inches of extrusion was light-yellow soft sludge, followed by a 3-inch gap that contained drainable liquid, and the last six inches was yellow sludge. The sludge contained black specks on the surface, but no crystals were observed. The solid sample was

Table 3-1. Tank 241-U-202 Sample Numbers and Locations.

Riser number	Sample number	Date extruded	Subsample number
2	95-050	4/3/95	S95T000582 S95T000584 S95T000585
	95-051	4/3/95	S95T000588 S95T000590 S95T000591 S95T000598 S95T000599
6	95-057	4/4/95	S95T000621 S95T000630 S95T000632 S95T000633 S95T000634
	95-058	4/4/95	S95T000636 S95T000643 S95T000644 S95T000646 S95T000650
Field blank[1]	95-055 95-056	4/4/95	S95T000654

[1]First core, riser 6.

divided into a upper half segment and a lower half segment. All analysis of the drainable liquid was run after settling the solids.

Core 78, Segment 2, Sample 95-058 was extruded on April 4, 1995, with the total amount of solid material recovered being 184 g, drainable liquid recovered being 19 g. No liner fluid was found. The drainable liquid was yellow, and the solid sample was light yellow. The last eight inches of extrusion were solid, of which the first four inches were light-yellow crystals, and the last four inches were light-yellow sludge. The solid sample was divided into a upper half segment and a lower half segment. All analysis of the drainable liquid was run after settling the solid.

Core 78, the field blank, was extruded on April 4, 1995, with the total amount of liquid recovered being 286 g.

Figure 3-1 is a flowchart of the basic steps taken by the laboratory regarding the extrusion and analysis of samples.

Figure 3-1. Sample Handling and Analysis Flowchart. (3 sheets)

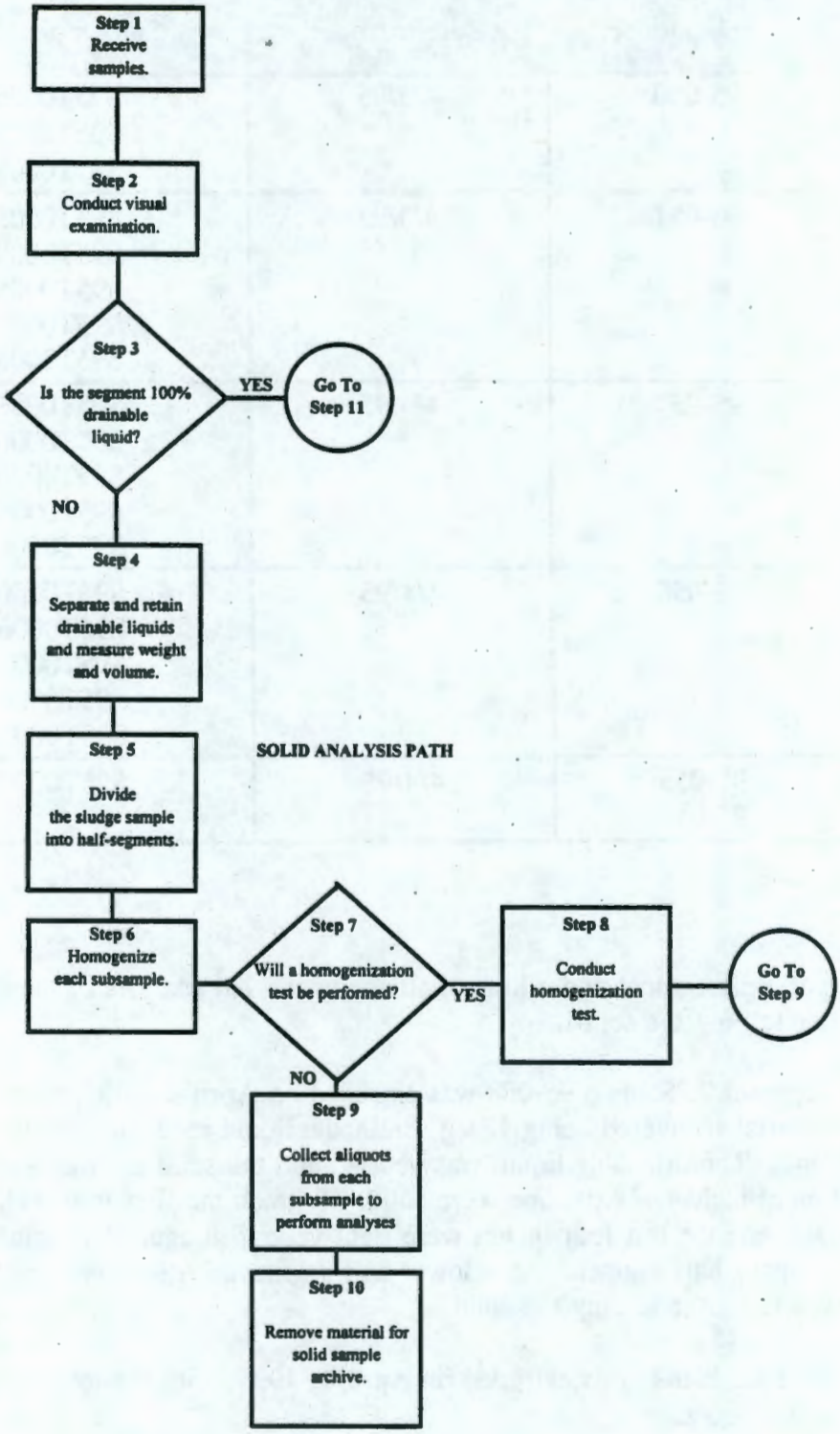


Figure 3-1. Sample Handling and Analysis Flowchart. (3 sheets)

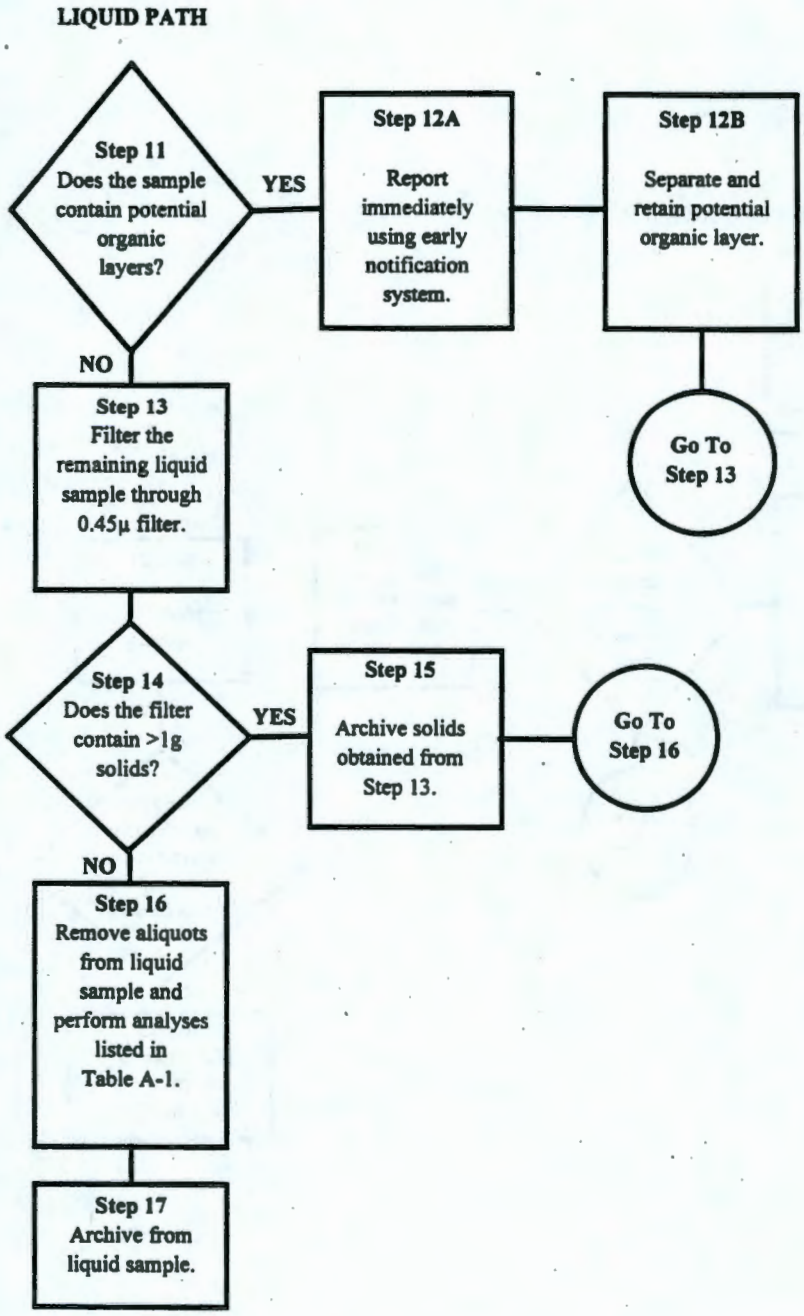
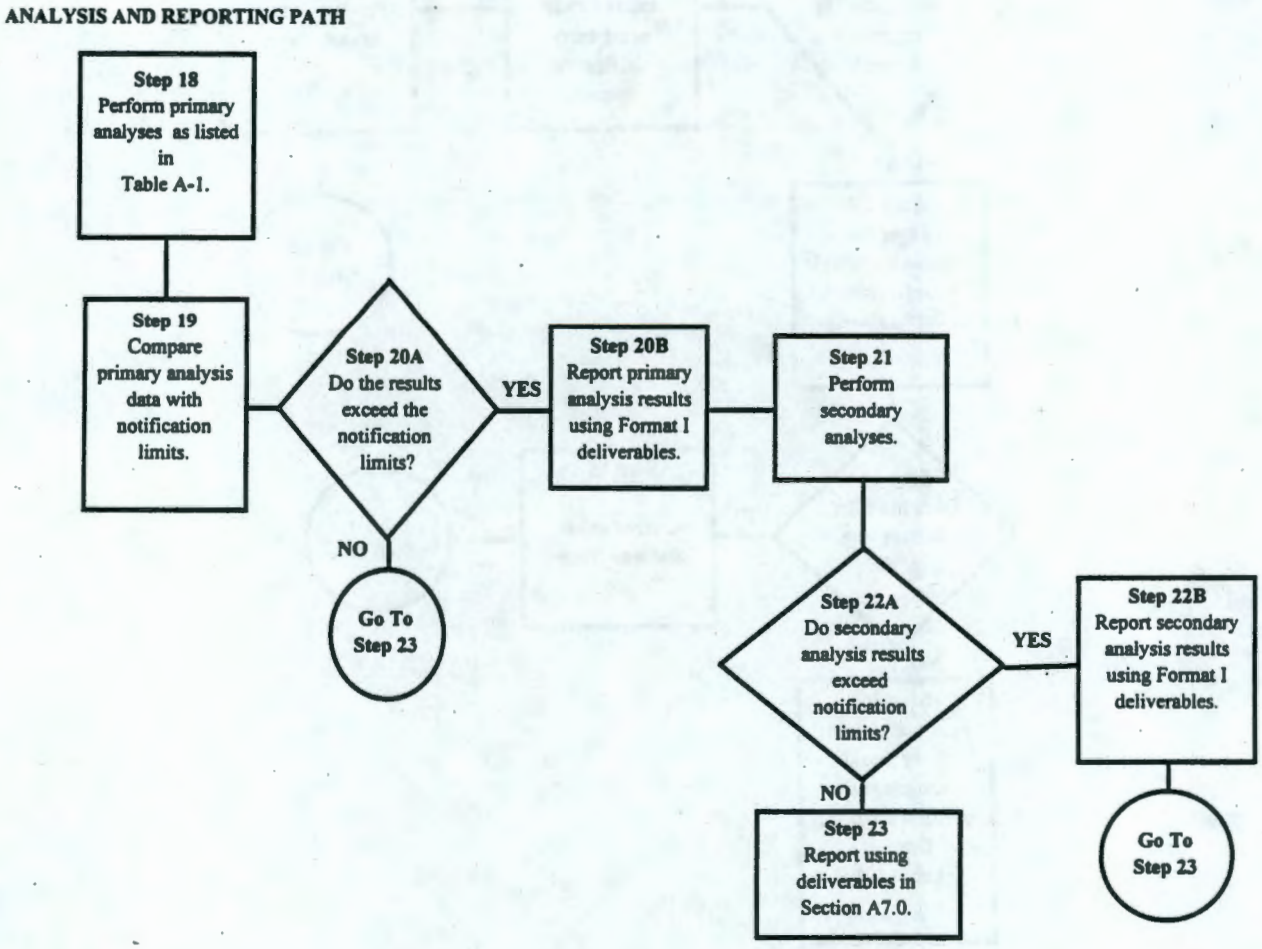


Figure 3-1. Sample Handling and Analysis Flowchart. (3 sheets)



The individual subsamples were homogenized. Sample homogenization is extremely critical for the DSC and TGA because of the very small amount of sample used in the analyses (10 to 50 mg). Even slight variations in sample consistency can result in relative percent differences (RPDs) between duplicate runs of greater than 10 percent (the Tank Safety Screening DQO limit (Babad et al. 1994).

The samples were only prepared for DSC/TGA and total alpha activity analyses. For a complete discussion of sample procedures see WHC-SD-WM-TI-648, Rev. 0 (De Lorenzo et al. 1994). There are no holding-time considerations because no regulatory analytes were requested. For further discussion of hold-time issues, refer to WHC-SD-WM-TI-648, Rev. 0 (De Lorenzo et al. 1994).

3.2.2 Sample Analyses

All analyses were performed at the 222-S Laboratory. The samples were received by the 222-S Laboratory on March 23, 1995 (Core 75) and March 30, 1995 (Core 78). The contents were extruded on March 24, 1995 (Core 75) and April 4, 1995 (Core 78).

The objectives of the sample analyses are to determine f weight percent water by TGA, exothermic activity by DSC, and total alpha content by alpha proportional counting. Procedures used for these analyses are listed in Table B-1 in Appendix B. Additional information on analytical methods can be obtained in WHC-SD-WM-TI-648, Rev. 0 (De Lorenzo et al. 1994).

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4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

The samples collected from tank 241-U-202 on March 22, March 29, and March 30 of 1995 are based upon the DQO process. The DQO that governs the data collection from these particular samples is the *Tank Safety Screening Data Quality Objective* (Babad et al. 1994). This DQO was developed to allow rapid classification of the tanks containing high-level waste and to support tank safety issues. The DQO specifies wt% water by TGA and DSC and total alpha activity be determined by laboratory analysis. The TGA, DSC, and total alpha results are presented in Table 4-1. Detailed quality control information concerning these three analyses is presented in Section 5.1.2.

Table 4-1. Analytical Presentation Tables.

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

4.2 DATA PRESENTATION

4.2.1 Total Alpha Activity

Total alpha analyses were performed on fusion digested samples, in accordance with procedure LA-508-101, Rev. D-2. All alpha total results were well within the DQO limit of 1.52 E+06 Bq/g (41 μCi/g). In addition, all samples exceeded the precision criteria. Table 4-2 presents the results for total alpha. If total alpha was not performed on a sample, it is not listed in the table. No quality control information such as matrix spike recoveries, standard recoveries, or duplicate results, are listed. This information may be obtained in WHC-SD-WM-DP-110, Rev. 0 (Jo 1995). The mean in the last column of Table 4-2 is a simple mean of the listed results.

A tank inventory estimate was not calculated for total alpha because of the lack of a current density value. The data tables present the data collected from the raw laboratory data sheets found in WHC-SD-WM-DP-110, Rev. 0 (Jo 1995).

Table 4-2. Tank 241-U-202 Total Alpha Activities.

Sample number	Result µCi/g	Duplicate µCi/g	Mean µCi/g
Riser 2			
S95T000585	1.19 E-03	1.07 E-03	<1.13 E-03
S95T000591	<1.18 E-03*	<1.20 E-03*	
S95T000599	<7.40 E-04*	<1.22 E-03*	
Riser 6			
S95T000633	1.52 E-03	1.10 E-03	<1.31 E-03
S95T000634	1.25 E-03	1.37 E-03	
S95T000646	<1.18 E-03*	<1.16 E-03*	
S95T000650	<5.32 E-03*	<4.71 E-03*	

Note: 1 Ci = 3.7 x 10^{10} Bq.
*All < values not used in calculating the mean.

4.3 PHYSICAL MEASUREMENTS AND THERMODYNAMIC ANALYSES

Tank 241-U-202 core samples were analyzed according to the *Tank Safety Screening Data Quality Objective* (Babad et al. 1994). Therefore, thermodynamic analyses were performed on the core samples. Density, percent solids, particle size, and rheology were neither requested nor performed.

4.3.1 Thermodynamic Analyses

TGA and DSC were performed on all subsegments of both core samples. All samples were homogenized before analysis. The results of the thermal analyses performed are summarized in Table 4-3.

The weight percent water by TGA was performed using Procedure LA-560-112, Rev. A-2 with a nitrogen purge. All results were above the notification limit (notification is made if the sample is analyzed at less that 17 wt% water), therefore no notifications were made. All samples met the precision and accuracy criteria stated in the TCP with an exception of Samples S95T000590, S95T000630, and S95T000643. See Section 5.1.2 for discussion of these anomalies.

Table 4-3. Tank 241-U-202 Thermogravimetric Analysis.

Sample number	Result (% water)	Duplicate result (% water)	Average (% water)	RPD	Third analysis (% water)
S95T000584	25.49	26.24	25.86	2.90	--
S95T000582	72.60	73.24	72.92	0.88	--
S95T000588	73.29	72.23	72.76	1.46	--
S95T000598	22.87	22.90	22.88	0.13	--
S95T000590	43.64	38.71	41.17	12.0	36.88
S95T000621	73.42	73.6	73.51	0.27	--
S95T000630	24.11	18.90	21.50	24.2	19.27
S95T000632	24.45	22.24	23.34	9.47	--
S95T000636	72.27	72.23	72.25	0.06	--
S95T000643	36.24	41.5	38.87	13.6	34.81
S95T000644	24.43	23.96	24.20	1.94	--

The differential thermal analyses were performed using Procedure LA-514-113, Rev. B-1, and a Mettler Model 20 and Procedure LA-514-114, Rev. B-0, and a Perkin Elmer DSC-7 Differential Scanning Calorimetry under nitrogen purge. All results were below the safety screen notification limit of 481 J/g (dry); therefore, no notifications were made. No exotherms were observed in any of the samples.

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

5.1.1 Field Observations

No field observations from the 1995 core sampling event impact the interpretation and use of the data.

5.1.2 Quality Control Assessment of Analytical Data

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

Matrix spikes are used to estimate the bias of the analytical method resulting from matrix interferences. Spike samples are prepared by splitting a sample into two aliquot portions and adding a known amount of a particular analyte to one portion to calculate a recovery percentage. The quality control criterion for matrix spikes is 100 $\pm$ 20% recovery. Spikes were only conducted on total alpha activity, and these samples demonstrated lower than normal spike recovery with values ranging from 50.8 to 89.6 percent recovery. Because the sample alpha activities were approximately three orders of magnitude less than the action limit for total alpha, rerun was not requested. The total alpha results are so low compared to the criticality criteria that the bias is not significant.

Duplicate analyses of samples provides an indication of laboratory precision and the homogeneity of the samples. To estimate this error, an RPD is calculated for each duplicate pair. The RPD is a measure of variability and is defined as the absolute value of one duplicate minus the other, divided by the mean. The TCP for tank 241-U-202 set the duplicate precision acceptance criterion at less than or equal to 10 percent RPD (Scheiber 1995). All samples met the precision and accuracy criteria stated in the TCP with three exceptions. TGA precision between the sample and the duplicate for Samples S95T000590, S95T000630, and S95T000643 exceeded the acceptance criteria, with RPD values of 12.0, 24.2, and 13.6, respectively. The sample result for S95T000590 was 43.64 percent water, the duplicate 38.71 percent. A third analysis was run with the result of 36.88 percent. The sample result for S95T000630 was 24.11 percent, and the duplicate was

18.90 percent. A third analysis was run with a result of 19.27 percent. The sample result for S95T000643 was 36.24 percent water, the duplicate was 41.52 percent. A third analysis was run with a result of 34.81 percent.

5.2 TANK WASTE INVENTORY AND PROFILE

The waste in tank 241-U-202 is composed of 80 percent sludge and 20 percent supernatant by volume (Hanlon 1995).

5.3 COMPARISON OF ANALYTICAL AND TRANSFER HISTORY

Because of the few analytical requirements for safety screening characterization, the comparison of the analytical results from the 1995 sampling event with the historical information is meaningless, based on the different models (assumption) for the data.

5.4 EVALUATION OF PROGRAM REQUIREMENTS

5.4.1 Safety Evaluation

This section provides selected results obtained from core sampling for some of the most pertinent analytes needed by the Waste Tank Safety Program. These data needs are defined in the *Tank Safety Screening Data Quality Objective* (Babad et al. 1994) and are used to determine that the 177 Hanford Site underground waste tanks have been appropriately categorized according to whether or not safety issues exist for each tank. The DQO establishes decision criteria for concentrations of analytes of concern. The decision criteria are used to determine whether a tank is safe or whether further investigation into the tank's safety is warranted. Table 5-1 tabulates the decision criteria required by the safety screening DQO and compares them to the recent analytical results.

The waste fuel energy value is determined using a DSC. No exothermic activity was observed for any of the subsamples analyzed.

The average percent water for all of the subsamples was 44.8 wt%. All results were above the notification limit (notification is made if the sample is analyzed at less that 17 wt% water), therefore no notifications were made.

Table 5-1. Safety Screening Data Quality Objective Decision Variables and Criteria.

Safety issue	Primary decision variable	Decision criteria threshold	Sample location	Average analytical value
Ferrocyanide/ organic	Total fuel content	115 cal/g	All	No exotherms observed
Ferrocyanide	Moisture content	[1]If fuel is above 8 wt%, then wt% H_2O < [(0.0932*DSC exotherm) - 10.7][2]	All	Ferrocyanide not applicable
Organic	Percent moisture	17 wt%	All	44.8 wt%
Criticality	Total alpha[3]	1.52 E+06 Bq/g (41 μCi/g) (1 g/L)[2]	Riser 2	Mean Bq/g (<1.1 E-03 μCi/g)
			Riser 6	Mean Bq/g (<2.2 E-03 μCi/g)
Flammable gas	Flammable gas	< 25% of the lower flammability limit	--	No data available

[1]For explanation of the moisture decision rule, See Figure 4-1 in Meacham et al. 1994.

[2]Fuel content is weight percent disodium nickel ferrocyanide, $Na_2NiFe(CN)_6$. The fuel percentage was calculated according to the following equation. The differential scanning calorimetry value is divided by 1430, which is the experimentally determined heat of reaction of $Na_2NiFe(CN)_6$ with nitrate in waste simulant (Postma et al. 1994).

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

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

$$\text{wt\% } H_20 < [(0.0932 \times \text{DSC exotherm}) - 10.7]$$

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

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

The heat generation rate based on historical radiochemical estimates is 4.75 E-04 kW (2 Btu/h). This is well below the high heat classification limit of 11.7 kW (4.0 E+04 Btu/h). In addition, the tank temperature history does not show any signs of excessive heat generation.

The potential for criticality is assessed using either total alpha or plutonium analyses. The safety screening criteria is 1 g/L of plutonium, which is equivalent to 41 μCi/g, assuming the waste has a density of 1.5 g/mL. All alpha total results were well within the DQO limit of 1.52 x E+06 Bq/g (41 μCi/g). In addition, all samples exceeded the precision criteria. The highest total alpha result for any semi-segment (1.4 μCi/g) is well below the limit of concern. In addition, the total alpha also contains activity from ^{241}Am.

The flammability of the gas in the headspace of the tank is another safety screening consideration. Analysis of the tank's headspace is presently not available.

6.0 CONCLUSIONS AND RECOMMENDATIONS

According to the *Tank Safety Screening Data Quality Objective* (Babad et al. 1994) and the comparisons and conclusions drawn from this document, evidence suggests there is no safety concern associated with tank 241-U-202. None of the Safety Screening decision criteria were exceeded. Although the flammability of the gas in the headspace of the tank is another safety screening consideration, the analysis of the tank's headspace is presently not available. No exothermic activity was observed for any of the subsamples analyzed. From July of 1987 to July of 1993, the maximum temperature reading was 19 °C (67 °F). The average percent water for all samples was 44.9 wt%, significantly above the 17 wt% safety screening criteria (Babad et al. 1994). Total alpha was well within the DQO unit of 41 μCi/g. Based on this information, the waste does no appear to have any immediate safety concerns.

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APPENDIX A

ANALYSIS OF 1975 SAMPLE

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Table A-1. Analysis Results.

Waste Tank 242-U-202		
Analysis of Tank Farm Sample		
October 23, 1975		
Received: September 25, 1975		
Sample: T-8245		
Component	Lab value	Lab unit
Physical data		
Vis-OTR	Clear, yellow. No solids 50 mrad/hr	
pH	12.8	
SpG	1.144	
H_2O	79.9	%
Chemical analysis		
OH	1.23	M
Al	0.294	M
Na	3.91	M
NO_2	0.237	M
NO_3	1.47	M
PO_4	1.40 E-02	M
Cl	2.49 E-02	M
F	9.55 E-04	M
CO_3	0.18	M
SO_4	Canceled	
Radiological analysis		
Pu	<3.78 E-06	g/gal
GEA: ^{134}Cs	1.66 E+02	μCi/gal
GEA: ^{137}Cs	1.24 E+05	μCi/gal
$^{89,90}Sr$	3.56	μCi/gal

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APPENDIX B

ANALYTICAL METHODS

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APPENDIX B

ANALYTICAL METHODS

Table B-1 contains the procedure number for each of the analytical methods performed on the tank 241-U-202 waste. All analyses were performed at the 222-S Laboratory.

Table B-1. Analytical Methods.

Analyte	Digestion	Procedure number
Thermogravimetric analysis	NA	LA-560-112[1]
Differential scanning calorimetry	NA	LA-514-113 LA-514-114[2]
Total alpha activity	Fusion	LA-508-101[3]

[1]*Determination of Weight Loss as Percent Water by Thermogravimetric Analysis (TGA)-Mettler TG 50*, LA-560-112, Rev. A-2, Westinghouse Hanford Company, Richland, Washington.

[2]*Differential Scanning Calorimetry (DSC)*, LA-415-113, Rev. B-1 and Mettler Model 20 and XXXXXX, LA-514-114, Rev. B-0 and a Perkin Elmer DSC-7 under nitrogen purge, Westinghouse Hanford Company, Richland, Washington.

[3]*Alpha and Beta in Liquid Samples*, LA-508-101, Rev. D-2, Westinghouse Hanford Company, Richland, Washington.

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