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7. Abstract

This document summarizes the information on the historicall uses, present status, and the sampling and analysis results of waste stored in tank 241-SX-113. 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-SX-113

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EXECUTIVE SUMMARY

This tank characterization report summarizes the information on the historical uses, present status, and sampling and analysis results of waste stored in the single-shell underground storage tank 241-SX-113. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-08.[1]

Tank 241-SX-113 is one of 15 single-shell tanks located in the SX Tank Farm in the 200 West Area of the Hanford Site. It is the first tank in a three-tank cascade series. The tank went into service in 1958 with receipt of reduction oxidation (REDOX) waste. Other than diatomaceous earth, the only other waste received by tank 241-SX-113 was REDOX waste from tank 241-SX-114. In 1962, the tank was removed from service and declared an assumed leaker. Interim stabilization was completed in November 1978 and intrusion prevention was completed in September 1982. Tank 241-SX-113 is not on any Watch Lists. The tank, which has an operational capacity of 3,790 kiloliters (1,000 kilogallons), contains 98 kiloliters (26 kilogallons) of waste, existing completely as sludge. No liquid remains.[2]

This report summarizes the only available sampling event, an auger sampling in May 1995.

[1]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.

[2]Hanlon, B.M., 1995, *Waste Tank Summary for Month Ending April 30, 1995*, WHC-EP-0182-85, Westinghouse Hanford Company, Richland, Washington.

The sampling and analysis were performed in accordance with the *Tank Safety Screening Data Quality Objective*.[3] Consequently, only total alpha activity, percent water, and energetics were evaluated. Samples were archived in accordance with the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification*.[4]

No safety screening notification thresholds were exceeded. No exothermic reactions were observed in the samples, all samples contained over 17 wt% water, and all total alpha activities were well below the 41-microcurie per gram limit. A calculated tank heat load was not possible because only total alpha activity was measured. The Historical Tank Content Estimate for heat load is 45.6 watts (156 British thermal units per hour).[5] Based on the safety screening analyses, it appears that tank 241-SX-113 poses no immediate safety concerns. However, this report does not contain any vapor space sampling and analysis information.

The lower portion of the waste containing the REDOX waste should have substantial concentrations of aluminum, iron, chromium, sodium, and nitrates. REDOX waste also contained significant amounts of cesium and strontium.

[3]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.

[4]Kupfer, M.J., J.M. Conner, R.A. Kirkbridge, and J.R. Mobley, 1994, *Interim Data Quality Objectives for Waste Pretreatment and Vitrification*, WHC-SD-WM-DQO-011, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

[5]Brevick, C.H., L.A. Gaddis, and W.W. Pickett, 1995, *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Areas*, ICF Kaiser Hanford Company, WHC-SD-WM-ER-352, Rev. 1, ICF Kaiser Hanford Company, Richland, Washington.

A description and status of tank 241-SX-113 are summarized in Table ES-1 and Figures ES-1 and ES-2. Table ES-2 summarizes the physical properties and chemical and radiochemical composition estimates taken from the Historical Tank Content Estimate[6] for tank 241-SX-113 waste.

[6]Brevick, C.H., L.A. Gaddis, and W.W. Pickett, 1995, *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Areas*, ICF Kaiser Hanford Company, WHC-SD-WM-ER-352, Rev. 1, ICF Kaiser Hanford Company, Richland, Washington.

Table ES-1. Description and Status of Tank 241-SX-113.

TANK DESCRIPTION	
Type	Single-shell
Constructed	1953 to 1954
In-service	1958
Diameter	23 m (75 ft)
Usable depth	9.1 m (30 ft)
Operating capacity	3,790 kL (1,000 kgal)
Bottom shape	Dished
Ventilation	Passive
TANK STATUS (as of April 1995)	
Waste type	Non-complexed
Total waste volume	98 kL (26 kgal)
Sludge volume	98 kL (26 kgal)
Supernate volume	-0-
Manual tape surface level (July 2, 1995)	38 cm (15 in.)
Temperature (January 1988 to January 1995)	23 °C (73 °F) to 27 °C (81 °F)
Integrity	Assumed leaker (1962)
Watch List	None
SAMPLING DATES	
Auger sampled	May 9 and 10, 1995
SERVICE STATUS	
Removed from service	1962
Interim stabilization	1978
Intrusion prevention	1982
Assumed leaker	1962

Notes:
- ft = feet
- m = meters
- in. = inches
- kL= kiloliters
- kgal = kilogallons
- cm = centimeters
- C = Celsius
- F = Fahrenheit

Figure ES-1. Tank 241-SX-113.

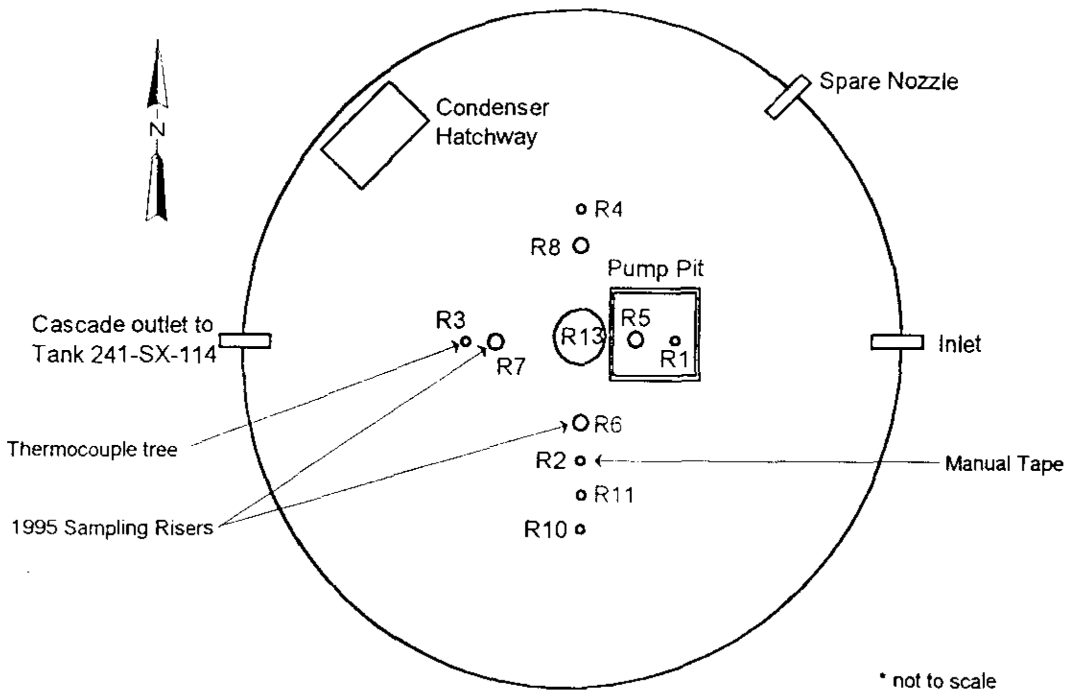

Figure ES-2. Waste Profile of Tank 241-SX-113.

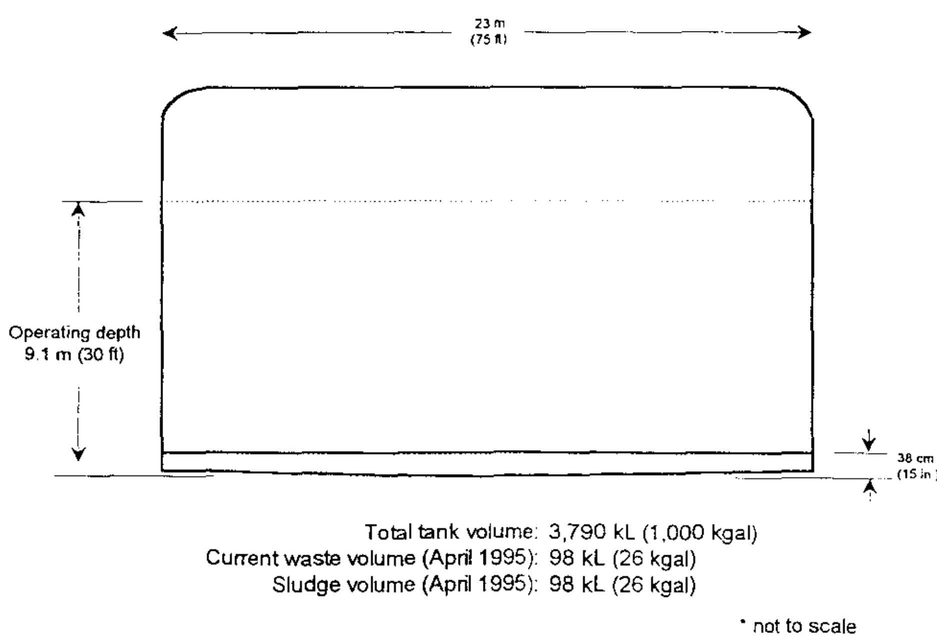

Table ES-2. Historical Tank Content Estimate for the Solids in Tank 241-SX-113.[1]

Physical Properties			
Total Solid Waste	10,600 kg (2 kgal)		
Heat Load	45.6 watts (156 Btu/hr)		
Bulk Density	1.40 g/mL		
Void Fraction	0.825		
Water wt %	68.9		
Chemical Constituents			
Analyte	**mole/liter**	**μg/g**	**kg**
Na^+	2.31	38,000	402
Al^{3+}	4.60	88,900	939
Fe^{3+} (total Fe)	0.513	20,500	217
Cr^{3+}	0.457	17,000	180
Ni^{2+}	0.0265	1,120	11.8
Ca^{2+}	0.0616	1,770	18.7
OH^-	17.0	2.07E+05	2,190
NO_3^-	2.06	91,300	966
CO_3^{2-}	0.0616	2,650	28.0
SO_4^{2-}	0.0159	1,090	11.6
Si (as SiO_3^{2-})	0.0123	248	2.62
Cl^-	0.0371	942	9.96
Radiological Constituents			
Radionuclide			
Pu		0.0118 μCi/g	0.00207 kg
U	0.0129 mole/liter	2,200 μg/g	23.3 kg
Cs	0.0731 Ci/L	52.4 μCi/g	554 Ci
Sr	0.844 Ci/L	605 μCi/g	6,390 Ci

Notes:
kgal = kilogallons
Btu/hr = British thermal units per hour
kg = kilograms
g/mL = grams per milliliter
μCi/g = microcuries per gram
μg/g = micrograms per gram
Ci/L = curies per liter
Ci = curies
[1]Brevick, C.H., L.A. Gaddis, and W.W. Pickett, 1995, *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Areas*, WHC-SD-WM-ER-352, Rev. 1, ICF Kaiser Hanford Company, Richland, Washington. The Historic Tank Content Estimate includes only 8 kL (2 kgal) of REDOX waste; constituents of the other 90 kL (24 kgal) are not included in the estimated inventory.

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LIST OF TERMS

ANOVA	analysis of variance
DQO	data quality objective
DSC	differential scanning calorimetry
HDW	Hanford Defined Waste
REDOX	reduction oxidation
RPD	relative percent difference
R1	REDOX waste (1952 to 1958)
TGA	thermogravimetric analysis
TLM	Tank Layer Model

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1.0 INTRODUCTION

This tank characterization report provides an overview of single-shell tank 241-SX-113 and its waste contents based on evaluation of historical information and sampling and analysis results. The chemical concentrations and inventories presented are based on estimates from the Tank Layer Model (TLM) (Agnew et al. 1995) and reported in the Historical Tank Content Estimate (Brevick et al. 1995). Evaluation of the model has not been completed, and the quantification of the tank contents is limited by the accuracy of the model. The May 1995 analysis of auger samples was used to evaluate safety screening criteria (Babad and Redus 1994) for the tank and does not provide detailed chemical and radiochemical information about the tank contents. Interim stabilization and intrusion prevention have been completed. Therefore, it is unlikely that the tank contents will change until pretreatment activities commence. The information presented in this report represents the best estimate of the present waste composition based on historical knowledge and the May 1995 sampling and analysis event. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-08 (Ecology et al. 1994).

1.1 PURPOSE

This report summarizes the information on the use and contents of tank 241-SX-113. When possible, this information will be used to address issues associated with safety, operations, environmental, and process development activities. This report also provides an initial reference point for more detailed information on tank 241-SX-113.

1.2 SCOPE

The auger samples collected in May of 1995 were obtained and analyzed to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The following three types of analyses, required by the data quality objective, were performed: differential scanning calorimetry to determine fuel content and energetics, thermogravimetry to measure percent moisture, and total alpha activity analysis to evaluate criticality. Samples were also archived in accordance with the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994).

Because of the limited analyses completed on the waste material collected in May of 1995, the chemical and radiochemical concentrations for most components are estimated from the TLM (Agnew et al. 1995).

This report does not contain information on vapor sampling and analysis to determine the composition of the tank's headspace gases.

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2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-SX-113 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 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/crust layers of the tank (i.e., slurry growth from gas evolution with subsequent burping and collapse, or shrinkage due to 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 April 1995 Waste Tank Summary (Hanlon 1995), tank 241-SX-113 contained 98 kiloliters (kL) (26 kilogallons [kgal]) of waste classified as non-complexed. Approximate volumes of the different waste phases are shown in Table 2-1.

Table 2-1. Summary Tank Contents Status.

Waste form	Volume kL (kgal)
Sludge	98 (26)
Salt cake	0 (0)
Total waste	98 (26)
Supernatant	0 (0)
Drainable interstitial liquid	0 (0)
Drainable liquid remaining	0 (0)
Pumpable liquid remaining	0 (0)

Notes:
kL = kiloliters
kgal = kilogallons

The tank is identified as a low-heat load tank, is passively ventilated, and is categorized as an assumed leaker. The tank was stabilized with diatomaceous earth in 1972 and was administratively interim stabilized in November 1978. Intrusion prevention was completed in September 1982. The waste is composed primarily of reduction oxidation (REDOX) waste (R1) and diatomaceous earth. Tank 241-SX-113 is not on any Watch Lists.

2.2 TANK DESIGN AND BACKGROUND

The 241-SX Tank Farm, built between 1953 and 1954, is a third-generation single-shell tank farm, consisting of 15 type IV 3,790-kL (1,000-kgal) tanks. Nine of these tanks were the first tanks designed for self-boiling waste, and the original construction included underground duct headers connected to a common condenser-ventilation system. These tanks were designed for a maximum waste temperature of 121 degrees Celsius (°C) (250 degrees Fahrenheit [°F]), pH values from 8 to 10, and a boiling period of 1 to 5 years.
Tank 241-SX-113 has four air lift circulators to control waste temperatures. This tank is equipped to cascade to tank 241-SX-114 and is first in a three-tank cascade series.
Tank 241-SX-113 has 11 risers ranging in size from 10 centimeters (cm) (4 in.) to 107 cm (42 in.) in diameter that provide surface level access to the underground tank. For more information about the 241-SX Tank Farm and single-shell tanks in general see the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

Tank 241-SX-113 is constructed of 0.6-m (2-ft) thick concrete on the lower portion of the tank walls and 0.381-m (1.25-ft) thick reinforced concrete on the upper part of the walls. The liner is a 3/8-inch mild carbon steel liner (American Society for Testing and Materials A283 Grade C) on the bottom and sides. The dome of the tank is a .381-m (1.25-ft) thick concrete top. This tank has a 30-cm (12-in.) dish bottom, no knuckle, a 9.53-m (31.3-ft) liner height and a 9.1-m (30-ft) operating depth. The tank is set on a reinforced concrete foundation. Various coatings and sealants were used to seal the inside and outside of the tank liner, dome, risers, and manholes to ensure that no leaks and intrusions existed. The tank was covered with approximately 2.2 m (7.25 ft) of overburden.

The tank surface level is monitored quarterly with a manual tape through riser 2. A list of tank 241-SX-113 risers showing size and use is provided in Table 2-2. A plan view depicting the riser configuration and relative locations is shown as Figure 2-1. Two tank inlets are available and are also shown in Figure 2-1. This constitutes all installed equipment for tank 241-SX-113. A tank cross-section showing the approximate waste level along with a schematic of the tank equipment is shown in Figure 2-2.

2.3 PROCESS KNOWLEDGE

This section presents the transfer history of tank 241-SX-113. Section 2.3.1 and Table 2-3 present the major transfers of waste that involved tank 241-SX-113 along with a narrative describing the transfers.

Table 2-2. Tank 241-SX-113 Risers.

Number	Diameter (inches)	Description and comments
R1	4	Drain
R2	4	Liquid level reel
R3	4	Temperature probe, benchmark
R4	4	Flange with cover
R5	12	Pump
R6	12	Breather filter
R7	12	Observation port
R8	12	Weather covered air circulator
R10	4	Under concrete slab - below grade
R11	4	Post (old liquid level reel riser)
R13	42	Below grade
N1	3	Spare
N2	3	Inlet nozzle overflow
N3	3	Outlet nozzle overflow

Table 2-3. Tank 241-SX-113 Waste Transfer Synopsis.

Waste type	Total volume kL (kgal)
REDOX high-level waste received (1958)	1,840 (487)
Condensate removed (1958)	284 (75)
Supernate removed (1958)	1,150 (305)
241-SX-114 waste added (1962)	787 (208)
241-SX-114 waste removed (1962)	681 (180)
Diatomaceous earth added (1972)	91 (24)

Notes:
kL = kiloliters
kgal = kilogallons

Figure 2-1. Riser Configuration of Tank 241-SX-113.

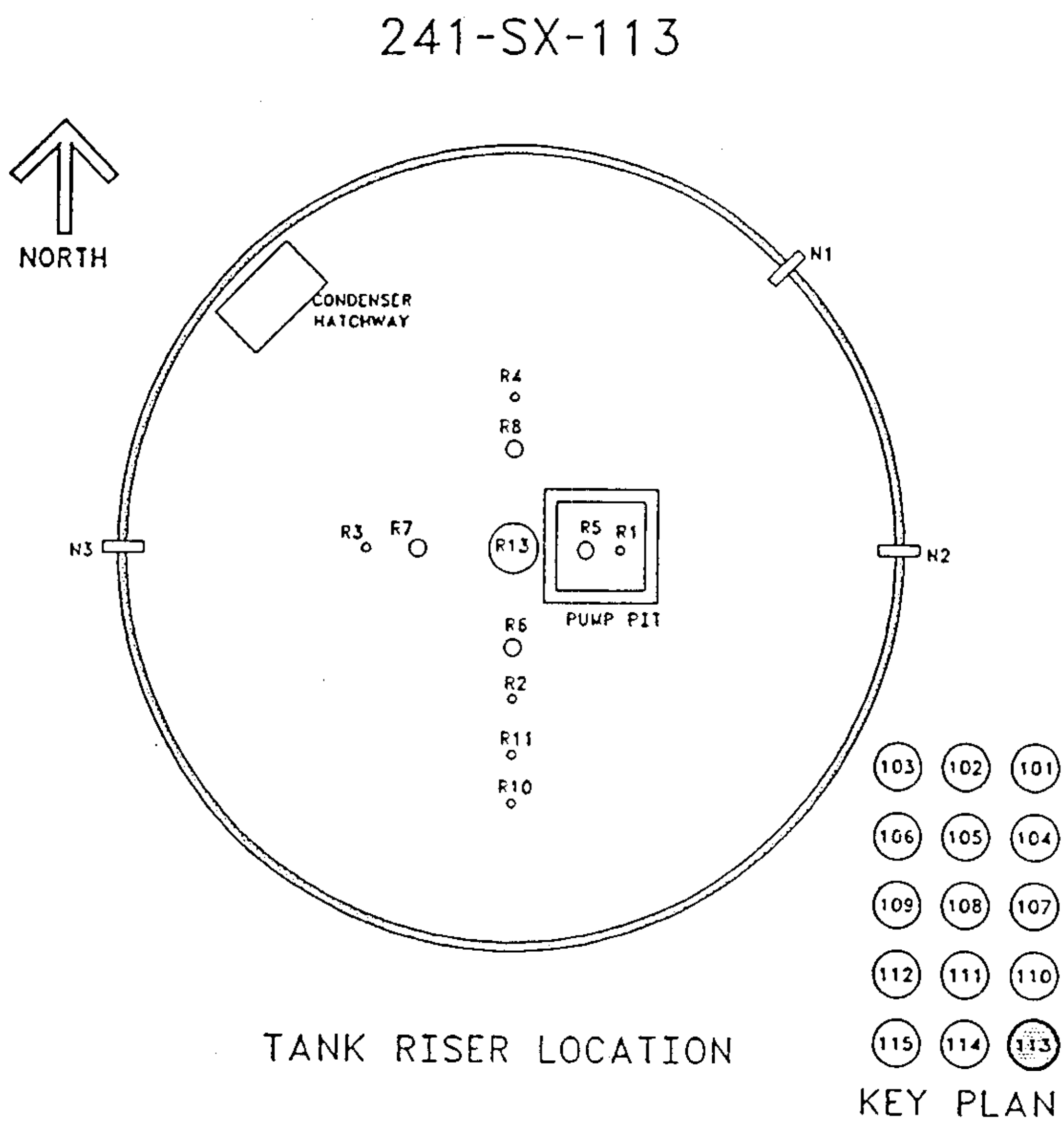
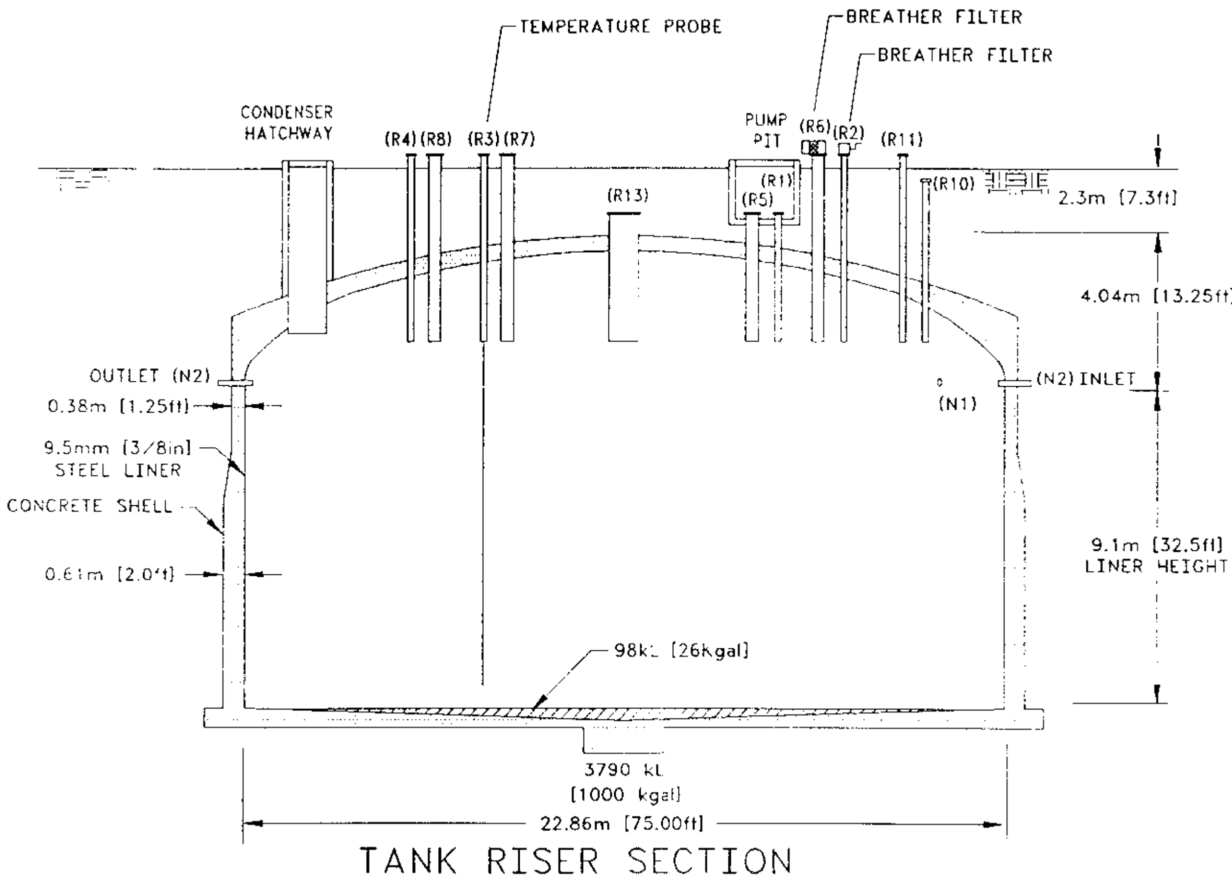


Figure 2-2. Cross-Section View of Tank 241-SX-113.



2.3.1 Waste Transfer History

Tank 241-SX-113 was brought into service in February 1958. Although this tank is in a cascade series, its fill history indicates waste was not cascaded through it (Agnew et al. 1994). During the first and second quarters of 1958, the tank received a total of about 1,840 kL (487 kgal) of R1 waste, which originated from the process that used hexone to extract plutonium and uranium from irradiated nuclear fuel (Agnew et al. 1994). The waste in tank 241-SX-113 began self-concentrating (i.e., the concentration of radionuclides in the waste contributed sufficient decay heat to cause the waste to boil) during the second quarter of 1958; about 284 kL (75 kgal) of condensate were removed from the tank during the second and third quarters of 1958 (Anderson 1990). In May 1958, measurements obtained by inserting rods into the tank through risers indicated the bottom of the tank liner had lifted about 1.2 m (4 ft); the liner dropped to its original position over several days (Hanson 1962; Welty 1988). This brought the integrity of the tank into question and most of the waste was removed leaving a heel of R1 waste. Shortly thereafter, the tank liner bulged a second time; this bulge also receded over time.

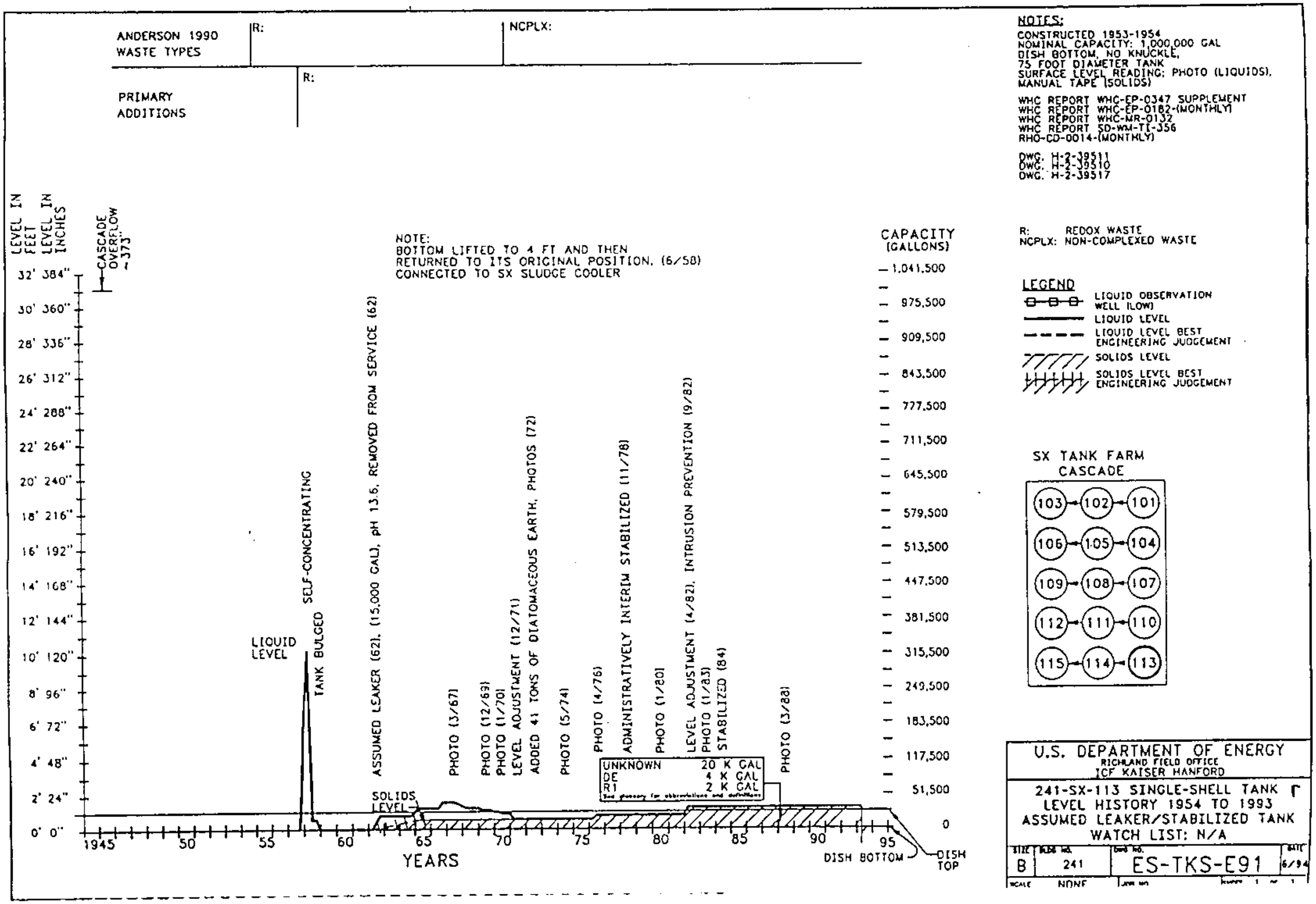
During October and November 1962, tank 241-SX-113 received a transfer of R1 waste from tank 241-SX-114 totaling about 787 kL (208 kgal) as part of a leak test procedure (Hanson 1962). Calculations showed about 57 kL (15 kgal) of the waste leaked, and most of the waste remaining in tank 241-SX-113 was then returned to tank 241-SX-114. Diatomaceous earth was added to the tank during the first quarter of 1972 to immobilize the residual supernatant liquid.

Information presented in Table 2-3 was taken from Agnew et al. (1994). The difference between the volume of waste transferred to the tank over its process history and the volume of waste removed from it does not correspond to the amount of waste currently remaining there. This is likely due to incomplete or inaccurate historical information. The fill history of tank 241-SX-113 is presented graphically in Figure 2-3. This chart was prepared from quarterly surface level information that does not illustrate all transfer activity.

2.3.2 Historical Estimation of Tank Contents

This section presents the Historical Tank Content Estimate for tank 241-SX-113 (Brevick et al. 1995). The historical data used for the estimate is the Waste Status and Transaction Record Summary (Agnew et al. 1994), the Hanford Defined Waste (HDW) list (Agnew 1995), and the Tank Layer Model (TLM) (Agnew et al. 1995). The Waste Status and Transaction Summary is a compilation of available waste transfer and volume status data. The HDW provides the assumed typical compositions for 50 separate waste types. In some cases, the available data is incomplete, thereby reducing the usefulness of the transfer data and the modeling results derived from it. The TLM takes the 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 an estimate that requires further evaluation using analytical data.

Figure 2-3. Tank Fill History.



Based on the TLM, tank 241-SX-113 contains 8 kL (2 kgal) of R1 waste, 15 kL (4 kgal) of diatomaceous earth, and 76 kL (20 kgal) of an unknown waste type. The layering chronology of the TLM indicates R1 was the first waste in the tank, followed by diatomaceous earth, and then unknown waste. However, Agnew et al. (1995) note that the layering chronology assumed by the TLM is questionable due to possible measurement errors. A review of the known transfer history of the tank suggests the waste consists of R1 waste from the 1958 transfers and waste (mostly likely R1) transferred between tank 241-SX-113 and tank 241-SX-114 during the 1962 leak test procedure; the top layer would consist of diatomaceous earth placed in the tank in 1972. In-tank photographs support this interpretation suggesting the waste surface consists of diatomaceous earth (see Section 2.4.3).

The top waste layer should consist primarily of SiO_2. The R1 waste layer should contain significant quantities of aluminum, iron, chromium, sodium, and nitrates. This waste should also contain substantial quantities of strontium and cesium. Table 2-4 presents an estimate of the waste constituents for the 8-kL (2-kgal) R1 waste layer predicted by the TLM. The Historical Tank Content Estimate for tank 241-SX-113 does not include diatomaceous earth, nor does it predict the composition of the unknown layer.

Figure 2-4. Tank Layer Model.

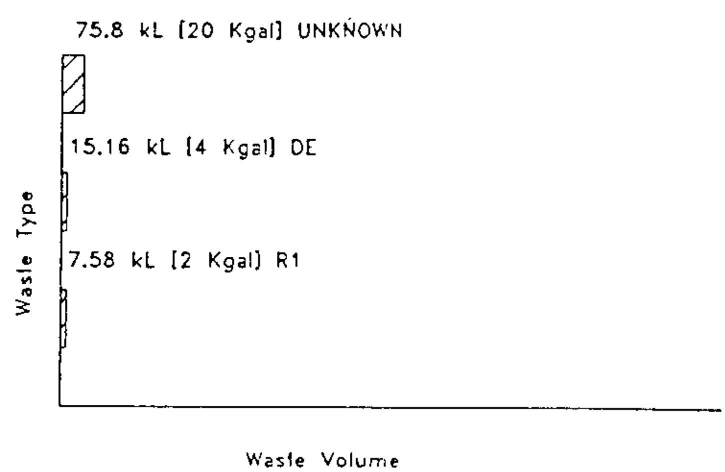


Table 2-4. Historical Tank Content Estimate (2 sheets).[1]

Physical Properties			
Total solid waste	10,600 kg (2 kgal)[2]		
Heat load	45.6 watts (156 Btu/hr)		
Bulk density	1.40 g/mL		
Void fraction	0.825		
Water wt%	68.9		
Total Organic Carbon wt% C (wet)	0		
Chemical constituents			
Analyte	**mole/liter**	**μg/g**	**kg**
Na^{+}	2.31	38,000	402
Al^{+3}	4.60	88,900	939
Fe^{+3} (total Fe)	0.513	20,500	217
Cr^{+3}	0.457	17,000	180
Bi^{+3}	0	0	0
La^{+3}	0	0	0
Ce^{+3}	0	0	0
Zr (as $ZrO(OH)_2$)	0	0	0
Pb^{+2}	0	0	0
Ni^{+2}	0.0265	1,120	11.8
Sr^{+2}	0	0	0
Mn^{+4}	0	0	0
Ca^{+2}	0.0616	1,770	18.7
K^{+}	0	0	
OH^{-}	17.0	207,000	2,190
NO_3^{-}	2.06	91,300	966
NO_2^{-}	0	0	0
CO_3^{-2}	0.0616	2,650	28.0
PO_4^{-3}	0	0	0
SO_4^{-2}	0.0159	1,090	11.6
Si (as SiO_3^{2-})	0.0123	248	2.62
F^{-}	0	0	0
Cl^{-}	0.0371	942	9.96
$C_6H_5O_7^{-3}$	0	0	0
$EDTA^{-4}$	0	0	0

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

Chemical constituents (continued)			
Analyte	mole/liter	μg/g	kg
$HEDTA^{-3}$	0	0	0
NTA^{-3}	0	0	0
$glycolate^{-}$	0	0	0
$acetate^{-}$	0	0	0
$oxalate^{-2}$	0	0	0
DBP	0	0	0
NPH	0	0	0
CCl_4	0	0	0
hexone	0	0	0
$Fe(CN)_6^{-4}$	0	0	0
Radiological constituents			
Radionuclide	**Concentration**		
Pu		0.0118 (μCi/g)	0.00207 (kg)
U	0.0129 mole/liter	2,200 (μg/g)	23.3 (kg)
Cs	0.0731 (Ci/L)	52.4 (μCi/g)	554 (Ci)
Sr	0.844 (Ci/L)	605 (μCi/g)	6,390 (Ci)

Notes:
kW = kilowatts
Btu/hr = British thermal units per hour
kg = kilograms
kgal = kilogallons
g/mL = grams per milliliter
μCi/g = microcuries per gram
μg/g = micrograms per gram
Ci/L = curies per liter
Ci = curies
[1]Data taken from Brevick, C.H., L.A. Gaddis, and W.W. Pickett, 1995, *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Areas*, WHC-SD-WM-ER-352, Rev. 1, ICF Kaiser Hanford Company, Richland, Washington.
[2]The Historic Tank Content Estimate includes only 8 kL (2 kgal) of REDOX waste.

2.4 SURVEILLANCE DATA

Tank 241-SX-113 surveillance consists of surface level measurements, temperature monitoring inside the tank (waste and vapor space), and leak detection well (drywell) monitoring for radioactivity outside the tank. The data are significant because they provide the basis for determining tank integrity.

Surface level measurements provide an indication of physical changes and consistency of the waste surface of a tank. Drywells located around the perimeter of the tank are used to detect increased radioactivity in the event of a leak to the soil. Radiation readings from the single drywell for tank 241-SX-113 have been below the 50 counts per second background level (Welty 1988).

2.4.1 Surface Level Readings

The tank 241-SX-113 surface level is monitored quarterly with a manual tape. The surface level has remained steady for the past 3 years, ranging between 38 cm (15 in.) and 37.5 cm (14.8 in.). On July 2, 1995, a surface level measurement of 38 cm (15 in.) was recorded. A graphical representation of quarterly surface level readings was presented in Figure 2-3.

2.4.2 Internal Tank Temperatures

Tank 241-SX-113 has a single thermocouple tree with 18 thermocouples in riser 3 to monitor the waste temperature. Elevations are not available for the thermocouples.
Tank 241-SX-113 is scheduled to have temperature data taken semiannually.

The median temperature of thermocouple readings from January 1988 to January 1995 was 26 °C (78.9 °F), the minimum temperature was 23 °C (73 °F), and the maximum temperature was 27 °C (81 °F). Plots of the thermocouple readings for the tank can be found in the supporting documents to the Historical Tank Content Estimate (Brevick et al. 1994). A graphical representation of the weekly high temperature can be found in Figure 2-5.

2.4.3 Tank 241-SX-113 Photographs

The photograph for tank 241-SX-113 was taken in 1988 (Figure 2-6). Diatomaceous earth was added to tank 241-SX-113 in 1972 and appears to cover the entire surface. The surface is completely dry. Most of the tank is a steel grey color except for a few spots which appear to be rust. The equipment is clearly shown in the photograph. The air lift circulators, a variety of risers, the manual tape, a turbine pump, two temperature probes, and a spare inlet nozzle can be seen in the picture. A discarded measurement tape can be seen on the waste surface. There have been no changes in the tank waste since the photographs were taken, so the photographs should represent the current tank contents.

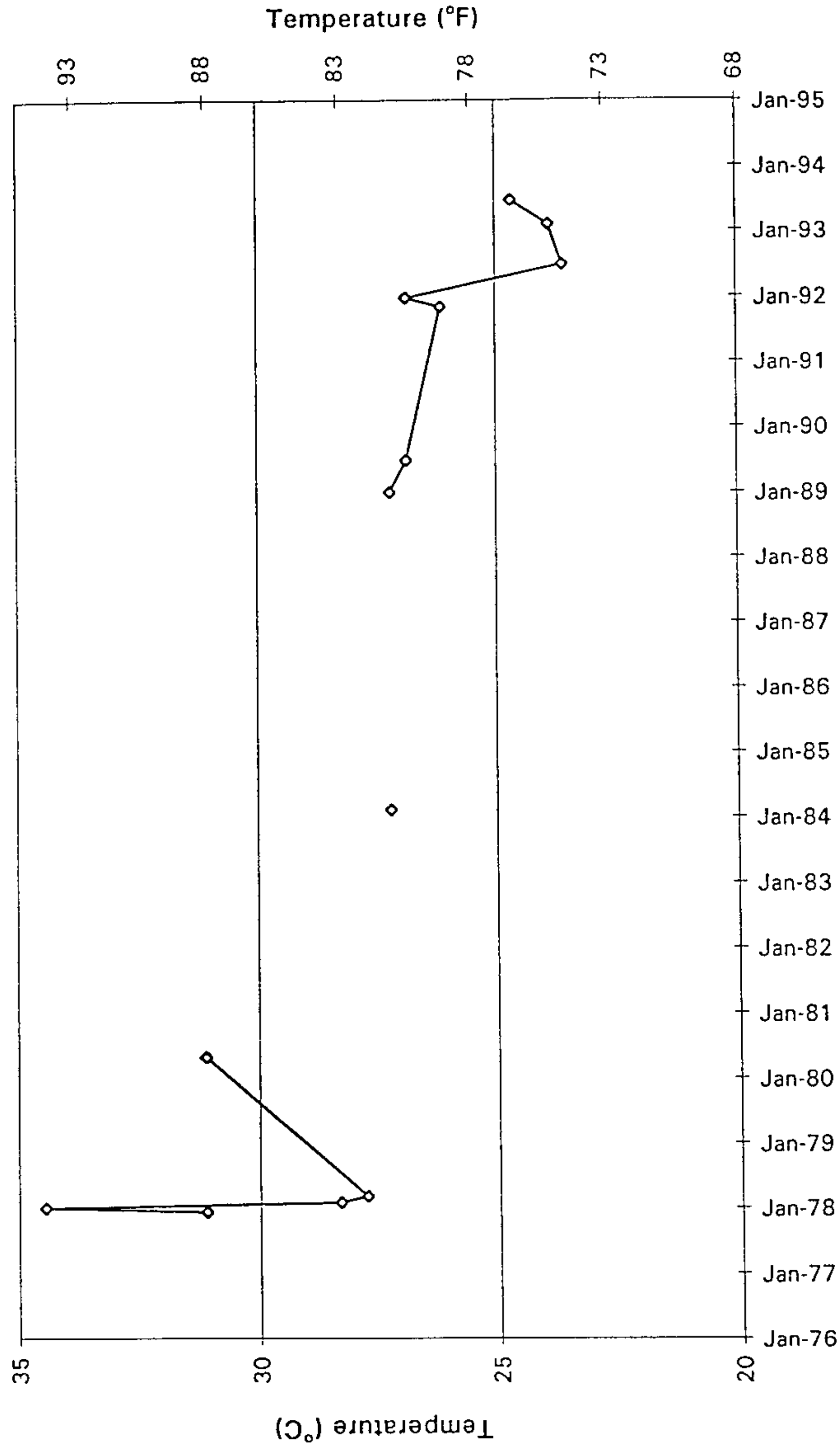
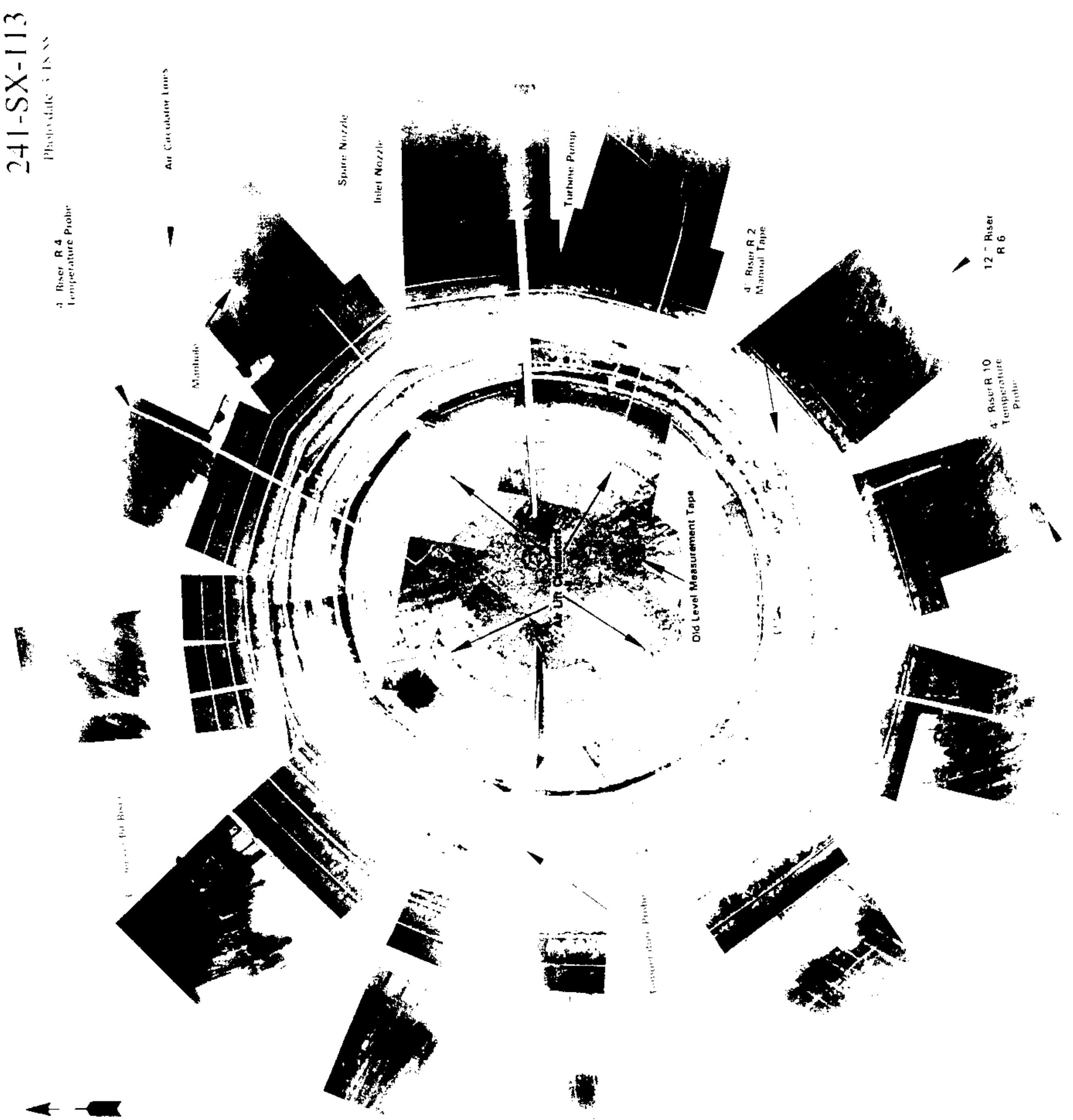


Figure 2-5. Tank 241-SX-113 Weekly High Temperature Plot.

Figure 2-6. Tank 241-SX-113 Waste Surface Photographs.



3.0 TANK SAMPLING OVERVIEW

This section describes an auger sampling event associated with tank 241-SX-113. Two auger samples were collected from the tank on May 9 and 10, 1995, to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994), and *Tank 241-SX-113 Tank Characterization Plan* (Sasaki 1995a). Sample handling and reported results may be found in the *45-Day Safety Screen Results and Final Report for Tank 241-SX-113, Auger Samples 95-AUG-028 and 95-AUG-029* (Sasaki 1995b). Further discussion of sampling and analytical procedures can be found in the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

3.1 DESCRIPTION OF SAMPLING EVENT (1995)

Two samples were collected from tank 241-SX-113 using 20-inch augers. Sample 95-AUG-028 was collected from riser 7 and sample 95-AUG-029 was collected from riser 6. Auger sampling was used because the waste material was expected to consist of sludge approximately 38 centimeters (cm) (15 inches [in.]) in depth. The 20-inch augers were expected to recover about 14 cm (5.5 in.) for sample 95-AUG-028 and 17.8 cm (7 in.) for sample 95-AUG-029. Sample 95-AUG-028 had waste material on all flutes of the auger for a total recovery of 249 grams. Most waste material was on the lower half of the auger for sample 95-AUG-029 for a total recovery of 97.8 grams. The dose rates through the drill strings were 50 milliroentgens/hour (mR/hr) for both samples. The smearable contamination was less than the alpha and beta-gamma detection limits. No drainable liquid was recovered from the auger samples. Table 3-1 summarizes the sampling data and describes the material collected on the augers. No problems related to the sample collections were noted.

3.1.1 Sample Handling (1995)

Auger samples 95-AUG-028 and 95-AUG-029 were received at the Westinghouse Hanford Company 222-S Laboratory May 11, 1995, and extruded in the hot cell on May 11 and 12, 1995, respectively. Color photographs were taken but were not provided with the data package. No discrete layers were noted in the samples, but differences in textures and colors were noted for sample 95-AUG-029.

Both auger samples were divided into half segments for analysis; archive specimens of each half segment were retained in the hot cell. Homogenized specimens removed from the augers were analyzed for thermal properties, percent water, and total alpha activity. Subsampling information, sample identification, and the completed analyses are provided in Table 3-2. In addition, samples were archived as requested in the pretreatment data quality objective (Kupfer et al. 1994).

Table 3-1. Tank 241-SX-113 Sampling Summary.

Sample ID	Riser	Amount Collected (grams)	Dose Rate (mR/hr)	Description
95-AUG-028	7	249	50	Most of the material resembled gray clay. Material texture was such that a large majority of sample adhered to the auger. Flutes 1-7 had more material than 8-18. Material on flutes 8-18 appeared crumbly. No crust or liquid was visible.
95-AUG-029	6	97.8	50	Most of the material was on the lower half of auger. Lower half material brown and moist with a texture like wet soil. Material on upper half light gray to beige with clay- or putty-like texture. Small piece of cellophane like material on top flute; material removed but not analyzed.

Notes:
mR/hr = milliroentgens per hour

Table 3-2. Tank 241-SX-113 Sample Data Summary.

Sample ID	Core ID	Segment	Preparation	Analyses
S95T000919	95-AUG-028	Upper half	Direct	TGA/DSC
S95T000921	95-AUG-028	Upper half	Fusion/Digest	Total Alpha
S95T000923	95-AUG-028	Lower half	Direct	TGA/DSC
S95T000925	95-AUG-028	Lower half	Fusion/Digest	Total Alpha
S95T000927	95-AUG-029	Upper half	Direct	TGA/DSC
S95T000929	95-AUG-029	Upper half	Fusion/Digest	Total Alpha
S95T000931	95-AUG-029	Lower half	Direct	TGA/DSC
S95T000933	95-AUG-029	Lower half	Fusion/Digest	Total Alpha
S95T000924	95-AUG-028	Lower half	NA	Archive
S95T000920	95-AUG-028	Upper half	NA	Archive
S95T000928	95-AUG-029	Upper half	NA	Archive
S95T000932	95-AUG-029	Lower half	NA	Archive

Notes:
DSC = differential scanning calorimetry
TGA = thermogravimetric analysis
NA = not applicable

3.1.2 Sample Analysis (1995)

Following the requirements of the safety screening data quality objective, energetics by differential scanning calorimetry (DSC), percent water by thermogravimetric analysis (TGA), and total alpha activity analysis were completed on the samples. The results of these analyses are presented in Section 4.0 of this document and the *45-Day Safety Screen Results and Final Report for Tank 241-SX-113, Auger Samples 95-AUG-028 and 95-AUG-029* (Sasaki 1995b). No results exceeded the safety screening notification limits.

Upper and lower specimens from both auger samples were analyzed. The DSC and TGA analyses were done on 5- to 20-milligram specimens of the waste material under a nitrogen atmosphere. The total alpha activity specimens were dissolved before analysis; this was accomplished by fusing a solid aliquot (0.2 to 0.5 grams) of the waste material in potassium hydroxide and dissolving, or digesting, the resultant fluxed material in hydrochloric acid. Total alpha activity was then determined on a liquid aliquot of the dissolved waste material.

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

All reported analyses were completed using approved laboratory procedures. A list of these procedures is presented in Table 3-3. No deviations or modifications to the procedures were noted by the laboratory. Total alpha activity spike recoveries have typically been below the specified lower limit when solids are observed on the sample mount, as occurred during these analyses.

Table 3-3. Laboratory Preparation and Analytical Procedures.

Analysis	Instrument	Preparation Procedure	Analytical Procedure
Energetics by DSC	Mettler™ and Perkin-Elmer™	NA	LA-514-113, Rev, B-1 LA-514-114, Rev. B-0
%Water by TGA	Mettler™ and Perkin-Elmer™	NA	LA-560-112, Rev. A-2 LA-514-114, Rev. B-0
Total Alpha	NA	LA-549-141, Rev. C-2	LA-508-101, Rev. D-2

Notes:
DSC = differential scanning calorimetry
NA = not applicable
TGA = thermogravimetric analysis
Mettler™ is a registered trademark of Mettler Electronics, Anaheim, California.
Perkin-Elmer™ is a registered trademark of Perkins Research and Manufacturing Company, Inc., Canoga Park, California.

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4.0 ANALYTICAL RESULTS

4.1 OVERVIEW

The May 1995 auger samples collected for tank 241-SX-113 are based on the data quality objective (DQO) process. The DQO that governs the sampling and subsequent sample analysis is the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). This DQO was developed to allow rapid classification of tanks containing high-level radioactive waste and to support the tank safety issue. For tank 241-SX-113, the DQO specifies that the following properties will be evaluated in the samples: weight percent water by thermogravimetric analysis (TGA), energetics by differential scanning calorimetry (DSC), and total alpha. The TGA, DSC, and total alpha results are presented within this document as indicated in Table 4-1. A tank characterization plan (Sasaki 1995a) was generated to outline the characterization process for tank 241-SX-113. A detailed discussion of the sampling process was presented in Section 3.0.

A total tank inventory was not calculated for total alpha because of the lack of a density value for the waste. The density value is required to convert the result from microcuries per gram to a projected inventory in curies.

Table 4-1. Analytical Data Presentation Tables.

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

The data tables present the results collected from the raw laboratory data sheets found in *45-Day Safety Screen Results and Final Report for Tank 241-SX-113 Auger Samples 95-AUG-028 and 95-AUG-029* (Sasaki 1995b).

4.2 TOTAL ALPHA

Analyses for total alpha activity were performed on a fusion digested sample on an alpha proportional counter according to procedure LA-508-101, Rev. D-2. All total alpha results were well below the safety screening DQO notification limit of 41 microcuries per gram (μCi/g), with the highest observed value of any sample or duplicate at 0.481 μCi/g. Relative percent differences (RPD), where calculable, were within the DQO requirement of 10 percent. Spike recoveries for two samples were within the requirement of 90 to 110 percent. Spike recoveries for the other two samples were low at 84.1 and 88.1 percent (these values are typical for alpha analyses). No reruns were requested for these samples as the total alpha activity results were at least a factor of 85 below the notification limit.

Table 4-2 presents the results for total alpha activity. The table lists the sample numbers and locations from which the samples were derived. The mean column is an average of the original sample and its duplicate. These results are a specific concentration of the analyte at different sampling points. No quality control data such as matrix spikes or serial dilutions are listed. The upper-half samples from riser six and the duplicate sample from the lower-half sample from riser six produced values that were below the analytical instrument's calibrated detection limit (denoted as <). For the riser six upper-half sample, the larger less-than value is presented in the mean column; this value was not included in the combined mean. The detected riser six lower-half sample result is repeated in the mean column and included in the combined mean; the duplicate is not included since it is a less-than value. This information may be obtained from Sasaki (1995b).

Table 4-2. Tank 241-SX-113 Analytical Data: Total Alpha.[1]

Analyte	Sample number	Sample location	Result	Duplicate	Mean	Combined mean[2]
Radionuclide						
Digested Solid		**Riser:section**	**μCi/g**	**μCi/g**	**μCi/g**	**μCi/g**
Total alpha	S95T000921	7:upper ½	0.0526	0.0526	0.0526	0.198
	S95T000925	7:lower ½	0.0617	0.0598	0.0607	
	S95T000929	6:upper ½	< 0.119	< 0.0744	< 0.119	
	S95T000933	6:lower ½	0.481	< 0.452	0.481	

Notes:
μCi/g = microcuries per gram

[1]Data derived from Sasaki (1995b).
[2]The combined mean includes riser seven sample data means and the riser six lower-half sample result; values below detection limits were not included in the combined mean.

4.3 THERMODYNAMIC ANALYSES

Tank 241-SX-113 auger samples were evaluated according to *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994); therefore, the only physical analyses required were TGA and DSC. These analyses determine the thermal stability or reactivity of a material. Density, percent solids, particle size, and rheology were not requested or performed.

4.3.1 Thermogravimetric Analysis

Thermogravimetric analysis measures the mass of a sample while the sample temperature is increased at a constant rate. A gas, such as nitrogen or air, is passed over the sample during heating to remove any gaseous matter. Any decrease in the sample weight represents a loss of gaseous matter either through evaporation or through a reaction that forms gas phase products.

Determination of weight percent water by TGA was performed under a nitrogen purge using either procedure LA-560-112, Rev. A-2 on a Mettler™ TG 50 instrument or procedure LA-514-114, Rev. B-0 on a Perkin-Elmer™ TGA 7 instrument. Analytical results satisfied the safety screening DQO requirement of 17 weight percent for all samples. The samples averaged 42.0 to 53.0 weight percent water. The RPDs between the samples and duplicates were below 10 percent for all but one sample. Sample S95T000931 (the lower half of the auger from riser 6) had an RPD of 23.0 percent. A third analysis was run on this sample with a result of 36.0 weight percent water and a relative standard deviation of 16.5 percent, still above the 10-percent criterion. However, all three results for the sample were well above the 17 weight percent water requirement.

The recorded sample weight losses are relative to the original sample weight. The TGA plots associated with the samples obtained from tank 241-SX-113 displayed one broad transition ranging from ambient temperature to approximately 160 degrees celsius (°C). The loss of sample weight within this temperature range is attributed to the evaporation of free water and the dissociation of covalently bonded water molecules. The TGA results are listed in Table 4-3.

Table 4-3. Thermogravimetric Analysis Results for Tank 241-SX-113.[1]

Sample location	Laboratory sample number	Method	Result	Duplicate	Mean	RPD
Riser:section			$\%H_2O$	$\%H_2O$	$\%H_2O$	%
7:Upper ½	S95T000919	Mettler™	52.1	53.8	53.0	3
7:Lower ½	S95T000923		46.1	42.7	44.4	8
6:Upper ½	S95T000927	Perkin-Elmer™	46.4	47.8	47.1	3
6:Lower ½	S95T000931		46.8	37.2	42.0	23
Estimated Tank Weight Percent Water					**46.6**	

Notes:

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

[1]Data derived from Sasaki (1995b).

4.3.2 Differential Scanning Calorimetry

In DSC analysis, heat absorbed or emitted by a substance is measured while the substance is exposed to a linear increase in temperature. While the substance is being heated, a gas such as nitrogen is passed over the waste material to remove any gases being released. The onset temperature for an endothermic (characterized by or causing the absorption of heat) or exothermic (releasing, as opposed to absorbing heat) event from a DSC analysis is determined graphically.

Analyses by DSC were performed under a nitrogen atmosphere using either procedure LA-514-113, Rev. B-1 on a Mettler™ DSC 20 instrument for the riser seven samples, or procedure LA-514-114, Rev. B-0 on a Perkin-Elmer™ DSC 7 instrument for the riser six samples. No exothermic reactions were observed in any of the samples or their respective duplicates.

The DSC results for tank 241-SX-113 are given in Table 4-4. The temperature range, temperature at maximum enthalpy change, and the magnitude of the enthalpy change in joules per gram are provided for each transition. The first transition represents the endothermic reaction associated with the evaporation of free and interstitial water. The second transition probably represents the energy (heat) required to remove the bound water from hydrated compounds such as aluminum hydroxide, or to melt salts such as sodium nitrate.

Table 4-4. Differential Scanning Calorimetry Results for Tank 241-SX-113.[1]

Sample number	Sample location Riser:section	Run	Sample weight mg	Transition 1 temperature range ambient to 170 °C Peak °C	Transition 1 ΔH J/g	Transition 2 temperature range 255 to 320 °C Peak °C	Transition 2 ΔH J/g
S95T000919	7:upper ½	1	15.9	108	1,310	---	---
		2	15.6	122	1,250	---	---
S95T000923	7:lower ½	1	15.0	108	953	279	10.1
		2	10.8	121	1,120	291	23.4
		3	10.8	119	1,280	287	31.5
S95T000927	6:upper ½	1	7.53	110	906	273	7.23
		2	11.4	117	896	---	---
S95T000931	6:lower ½	1	9.36	115	850	295	69.4
		2	16.7	116	886	304	137

Notes:

mg = milligrams
°C = degrees Celsius
J/g = joules per gram
ΔH = change in enthalpy

[1]Data derived from Sasaki (1995b).

5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

The purpose of this section is to evaluate the overall quality and consistency of the available results for tank 241-SX-113 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 data. These factors are used to assess the overall quality and consistency of the data and to identify any limitations in its use. Because of the limited sampling and analysis for tank 241-SX-113, evaluation of many items of data consistency is not possible.

5.1.1 Field Observations

No events or conditions were noted that may have impacted the quality or usefulness of the data.

5.1.2 Quality Control Assessment

The appropriate blanks, duplicates, spikes, and standards for quality control measures were performed on the 1995 auger sampling analyses (thermogravimetric analysis [TGA], total alpha activity, and differential scanning calorimetry [DSC]). The tank characterization plan (Sasaki 1995a) established a criterion of plus or minus 10 percent for the accuracy and precision of the data.

The standard and spike results provide an estimate of analysis accuracy. If a standard or spike is above or below the criterion, the analytical results may be biased high or low, respectively. All standards conducted on TGA, total alpha activity, and DSC were in the acceptable range. Two of four spikes conducted on total alpha activity were below the criterion (84.1 and 88.1 percent), indicating that the analytical results may be biased low. Spikes are not applicable to the TGA and DSC methods.

Evaluation of blanks is only applicable to total alpha analyses. All blanks were below the detection limit, indicating that contamination was not a problem. The precision requirements are calculated by the relative percent difference (RPD) between primary and duplicate samples. No RPDs were calculated for DSC because no exothermic reactions were detected. The RPDs for total alpha activity met the criterion, but one RPD (23.0 weight percent recovery) for percent water did not. A triplicate analysis was run on this sample with the results still above the quality control criterion. However, all three results for the sample were well above the 17 weight percent minimum requirement (Sasaki 1995b).

In summary, it appears that other than the low spike recoveries for total alpha activity, the quality control results were good.

5.2 COMPARISON OF ANALYTICAL DATA

Data other than the tank safety screening data are not available.

5.3 TANK WASTE PROFILE

Based on the transfer history of the tank, three primary layers of waste are expected: an upper layer of diatomaceous earth, a middle layer of unknown waste (most likely REDOX waste), and a lower REDOX waste layer. It is difficult to verify this profile given the small number of analytes tested and the shallow depth of the waste. Also, the two risers sampled were only about 10 feet apart, which is not ideal for a horizontal comparison of the tank contents. Therefore an analysis of variance (ANOVA), normally conducted on the weight percent water data to determine if there were any horizontal or vertical differences in the water concentration, was not performed. This test was also not conducted on the DSC results because there were no exotherms, or on the total alpha because of several non-detects (Sasaki 1995b).

5.4 COMPARISON OF ANALYTICAL AND HISTORICAL TANK CONTENT ESTIMATE DATA

A comparison can be made between total alpha activity measured in the safety screening auger samples (Sasaki 1995a) and the plutonium activity as modeled in Brevick et al. (1995). This comparison is not totally valid, as not all of the alpha-emitting radionuclides are taken into consideration, as well as the fact that the model only takes into consideration the known REDOX waste on the bottom of the tank. The safety screening results for total alpha activity were 0.198 μCi/g, and the plutonium concentration from Brevick et al. (1995) was 0.0118 μCi/g. In addition to the total alpha activity comparison, the weight percent water value by TGA from Sasaki (1995b) may be compared to the modeled value from Brevick et al. (1995). The safety screening result was 46.6 weight percent, and that from the tank model was 68.9 weight percent. However, it should be noted that the percent water value by TGA is attributed only to the evaporation of free water and to the dissociation of covalently bonded water molecules, while that from the tank model represents the total water.

5.5 COMPARISON OF RESULTS TO PROGRAM REQUIREMENTS

The 1995 auger sampling of tank 241-SX-113 was performed to meet the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). This data quality objective (DQO) defines data needs used to screen the 177 Hanford Site underground waste tanks for unidentified safety issues and to determine if the tanks have been placed into the appropriate safety category. The DQO establishes decision criteria or notification limits for concentrations of analytes of concern. If results from one of the primary analyses exceed any of the decision criteria, the tank is not classified as "safe" and further analyses are conducted to ensure the safety of the tank (Babad and Redus 1994). Table 5-1 tabulates the decision criteria required by the safety screening DQO and compares them to the recent analytical results. There are insufficient data to evaluate operational, environmental, or process development program requirements.

Also specified in the safety screening DQO are the sampling intervals and desired precision. The DQO requirement that the sampling interval be every half segment was achieved by the 222-S Laboratory. One TGA analysis sample exceeded the plus or minus 10 percent DQO precision threshold, measured by RPD. RPDs were not given for the DSC results because no exothermic reactions were observed. The calculated total alpha activity RPDs were within the plus or minus 10 percent criterion.

As can be seen in Table 5-1, none of the safety screening notification limits were exceeded. No exothermic reactions were observed in the DSC analyses, the TGA analysis results were all above the required 17 weight percent water, and the total alpha activity results were well below the safety screening notification limit of 41 μCi/g (Sasaki 1995b). It should be noted that flammable gas emissions were not evaluated for this tank.

The amount of heat generated in the tank by radioactive decay is not available because radiochemical analyses other than total alpha activity were not performed. An estimated heat load of 45.6 watts (156 Btu/hr) from the Historical Tank Content Estimate (Brevick et al. 1995) was presented previously in Section 2.0.

Table 5-1. Safety Screening Data Quality Objective Decision Variables and Criteria (2 sheets).

Safety issue	Primary decision variable	Decision criteria threshold	Auger location	Analytical value	RPD (%)
Riser 6					
Ferrocyanide/organic	Total fuel content	115 cal/g (481 J/g)	All segments	No exotherms observed	---
Organic	Percent moisture	17 wt%	upper ½	47.1 wt%	3
			lower ½	42.0 wt %	23
Criticality	Total alpha	41 μCi/g (1 g/L)[a]	upper ½	< 0.119 μCi/g	NA
			lower ½	0.481 μCi/g	NA
Flammable gas	Flammable gas	25% lower flammability limit for all flammable gases	No data	No data	
Riser 7					
Ferrocyanide/organic	Total fuel content	115 cal/g (481 J/g)	All segments	No exotherms observed	---
Organic	Percent moisture	17 wt%	upper ½	53.0	3
			lower ½	44.4	8
Criticality	Total alpha	41 μCi/g (1g/L)[a]	upper ½	0.0526 μCi/g	0
			lower ½	0.0607 μCi/g	3

Table 5-1. Safety Screening Data Quality Objective Decision Variables and Criteria (2 sheets).

Safety issue	Primary decision variable	Decision criteria threshold	Auger location	Analytical value	RPD (%)
Riser 7 (continued)					
Flammable gas	Flammable gas	25% lower flammability limit for all flammable gases	No data	No data	

Notes:

RPD = relative percent difference
cal/g = calories per gram
wt% = weight percent
DSC = differential scanning calorimetry
μCi/g = microcuries per gram
g/L = grams per liter
NA = not applicable
No data = TCP does not address

[a]Although the actual decision criterion listed in the DQO is 1g/L, the total alpha is measured in μCi/g rather than g/L. To convert the notification limit for total alpha into a value more readily usable by the laboratory, it was assumed that all alpha decay originates from Plutonium-239. Using the specific activity of Plutonium-239 (0.0615 curies per gram [Ci/g]) and assuming a tank density of 1.5, the decision criterion may be converted to 41 μCi/g as follows:

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

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The solid waste in tank 241-SX-113 has been sampled and analyzed for the purposes of safety screening in accordance with the requirements listed in *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The May 1995 auger sampling is the only recorded sampling event for this tank. As mandated by the safety screening DQO, analyses for weight percent water, energetics, and total alpha activity were performed. Because only these properties have been measured, the chemical and radiochemical composition of the waste must be approximated solely from estimates based on the tank waste history (Brevick et al. 1995). Prior to waste retrieval, a full suite of analyses should be conducted.

All analytical results satisfied the specifications of the safety screening DQO. No exothermic reactions were observed. All of the percent water values were above the 17-percent criterion. None of the total alpha activity results came close to approaching the 41 microcurie per gram limit. No heat load determination was available from analytical data, but a heat load estimate of 45.6 watts (156 Btu/hr) was made in the Historical Tank Content Estimate (Brevick et al. 1995). As discussed in section 5.5, these estimates should be used with caution. Based on the analytical results and the safety screening requirements, the waste in tank 241-SX-113 does not appear to pose any immediate safety concerns. However, the results from the 1995 auger sampling do not include an evaluation of the tank headspace vapors.

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