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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-AZ-102. This report supports the requirements of Tri-Party Agreement Milestone M-44-08.

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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 double-shell underground storage tank 241-AZ-102. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-08 (Ecology et al. 1994).

Tank 241-AZ-102 is located in the AZ Tank Farm in the Hanford Site's 200 East Area. The tank was designed to provide storage space for high-level aging waste generated at Plutonium-Uranium Extraction (PUREX) Plant and went into service in 1976 upon receipt of aging waste from PUREX and high strontium wastes from B Plant. The tank later received complexant concentrate waste from operation of the 242-A Evaporator. In 1986, waste was removed leaving only a minimal heel. Since then the major waste types received by the tank have been aging waste (or neutralized current acid waste) from PUREX and waste water. The last transfer of aging waste to tank 241-AZ-102 occurred in 1990. The tank remains in active service for waste management operations.

A description and status of tank 241-AZ-102 are summarized in Table ES-1, Table ES-2, and Figure ES-1 in this executive summary. Future plans for the tank will change the composition of the supernatant and possibly the sludge. The tank, which has an

Table ES-1. Tank 241-AZ-102 Description and Status.

Tank description	
Type:	Double-shell
Constructed:	1971-1975
In-service:	1976
Diameter:	23 m (75 ft)
Usable depth:	8.8 m (29 ft)
Operating capacity:	3,710 kL (980 kgal)
Bottom shape:	Flat
Ventilation:	Operating exhauster
Tank status	
Current waste volume:	3,590 kL (949 kgal)
Current sludge volume:	360 kL (95 kgal)
Current supernatant volume:	3,230 kL (854 kgal)
FIC/manual tape surface level:	877 cm (345 in.)
Highest temperature recorded in sludge:	83 °C (182 °F) (May 1, 1995)
Integrity category:	Sound
Service status	
Active service	

Table ES-2. Double-Shell Underground Storage Tank 241-AZ-102 Concentrations and Inventories for Major Analytes and Analytes of Concern. (2 sheets)

Physical properties	Sludge results		Liquid results	
Density (g/mL)	1.49		1.10	
Percent water	51%		84.1%	
pH	--		12.9	
Heat load	<58,441 W (<199,457 Btu/hr)			
Chemical constituents	Sludge[a] concentration	Sludge inventory	Liquid[b] concentration	Liquid inventory
Metals	(μg/g)	(kg)	(μg/g)	(kg)
Aluminum	38,688.9	19,196.3	140.9	501.5
Cadmium	10,800.5	5,358.9	--	--
Chromium	1,628.5	808.0	879.0	3,128.9
Iron	94,322.7	46,800.1	<9.1	<32.4
Lanthanum	3,240.4	1,607.8	--	--
Sodium	54,340.0	26,961.9	4,836.4	17,215.6
Nickel	6,379.7	3,165.4	--	--
Silicon	3,361.1	1,667.7	484.0	1,722.8
Uranium	9,464.0	4,695.8	1,400.0	4,983.4
Zirconium	13,033.0	6,466.6	--	--
TIC	13,203.2	6,551.0	5,311.8	18,907.9
Ions	(μg/g)	(kg)	(μg/g)	(kg)
NO_3^-	3,424.4	1,699.1	21,636.4	77,016.9
NO_2^-	11,877.6	5,893.3	25,090.9	89,313.6
SO_4^{2-}	8,040.8	3,989.6	15,727.3	55,982.9

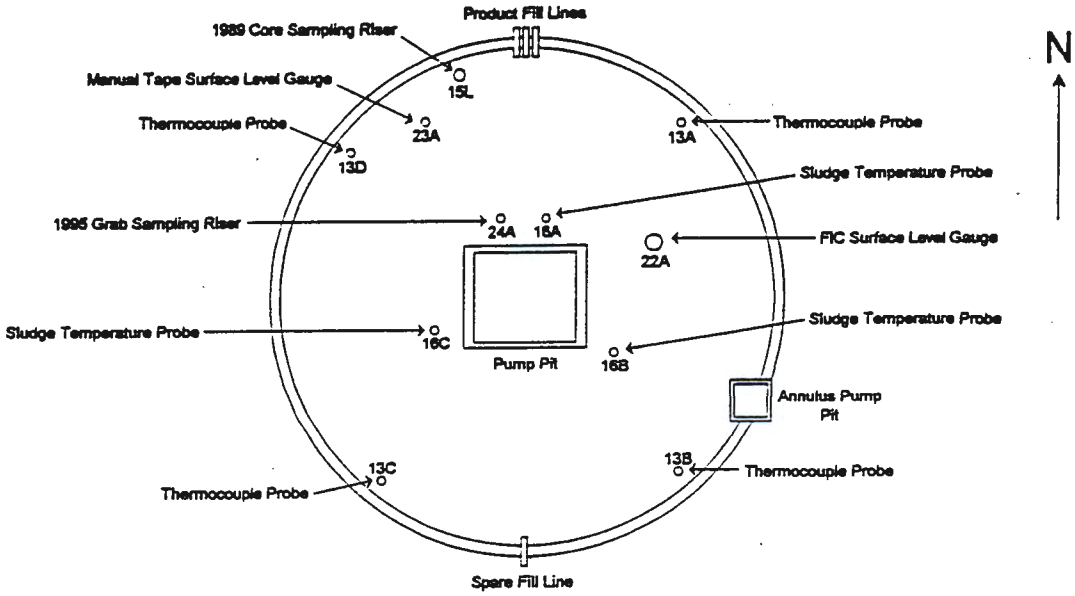
Table ES-2. Double-Shell Underground Storage Tank 241-AZ-102 Concentrations and Inventories for Major Analytes and Analytes of Concern. (2 sheets)

Physical properties	Sludge results		Liquid results	
Organics	(μg/g)	(kg)	(μg/g)	(kg)
Total organic carbon	2,650.4	1,315.0	1,366.4	4,863.8
Radionuclides	(μCi/g)	(Ci)	(μCi/g)	(Ci)
^{241}Am	72.7	36,081.7	<4.3 E-03	<15.3
^{137}Cs	842.8	342,801.2	945.5	3,370,000.0
^{106}Ru	<2,903.9	<3,570.3	--	--
^{90}Sr	15,841.7	6,366,730.3	1.6	5,802.1

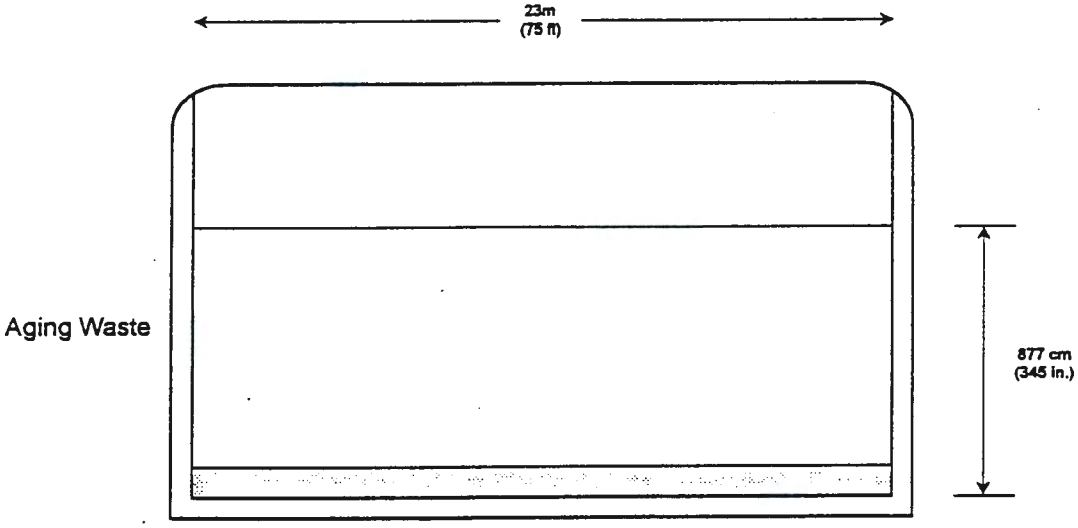
Note: 1 Ci = 3.7 E+10 Bq.
[a]Based on 1989 sampling and analysis, sludge with interstitial liquids.
[b]Based on 1995 sampling and analysis, supernatant only.
-- Indicates information/value not measured.

Figure ES-1. Tank 241-AZ-102.

Tank 241-AZ-102



Waste Profile of Tank 241-AZ-102



Total Tank Volume: 3,790 kL (1,000 kgal)
Current Waste Volume: 3,590 kL (949 kgal)
Current Sludge Volume: 360 kL (95 kgal)
Current Supernate Volume: 3,230 kL (854 kgal)

operational capacity of 3,710 kL (980 kgal), currently contains 3,590 kL (949 kgal) of waste, 3,230 kL (854 kgal) existing as supernatant and 360 kL (95 kgal) in the form of sludge (Hanlon 1995). The sludge measured 333 kL (88 kgal) when last sampled (May 1989), while the supernatant constituted 3,236 kL (855 kgal) upon its most recent sampling (February 1995).

This report summarizes four sampling and analysis events. The first two sampling events occurred in August and October of 1987 and the results are presented and used in this report only for comparison with the more recent sampling events. The third sample was taken in 1989 to support retrieval, pretreatment, and disposal and is used to represent sludge composition and properties. Finally, supernatant composition is based on grab samples taken in February of 1995 to evaluate waste compatibility. The grab samples were taken as prescribed in the *Data Quality Objectives for the Waste Compatibility Program*, WHC-SD-WM-DQO-001 (Carothers 1994).

The fuel content of the supernatant was measured by differential scanning calorimetry (DSC) and no exotherms were found, denoting that the fuel content of the supernatant is low. Although a similar analysis was not performed on the core sample, the presence of excessive fuel is unlikely when the total organic carbon (TOC) content and process history of the tank are considered. If the sludge is sampled again the analyses should include an evaluation of the fuel content by DSC to provide an estimate of the future compatibility of this sludge with other waste types. The waste is approximately 90% supernatant, and the sludge moisture

content was found to be 51% (by weight), which more than satisfies the 17 wt% water requirement of the safety screening DQO (Babad and Redus 1994). The tank has a substantial heat load of <58,441 W (<199,457 Btu/hr), which is to be expected due to the high concentration of ^{90}Sr in aging waste. Although notable, the heat load is still far below the 4,000,000 Btu/hr design limit (Bergmann 1989). The estimated level of $^{239/240}Pu$ in the tank sludge, 3.14 μCi/g, is below the safety screening threshold of 41.3 μCi/g (Babad and Redus 1994). Based on this information, the waste in this tank does not appear to have any immediate safety concerns; however, this report does not include any tank head space vapor sampling and analysis information for evaluation.

The 1995 supernatant analysis indicates that the liquid meets compatibility assessment criteria. The supernatant $^{239/240}Pu$ and ^{241}Am levels are below the transuranic classification limit of 100 nCi/g. The TOC concentration in the supernatant is 1.50 g/L, well below the organic complexant classification criteria of 10 g TOC/L. It should be noted, however, that the transfer of the waste in tank 241-AZ-102 to a non-aging waste DST will likely result in a violation of heat load limits for the receiving tank.

The concentration and tank inventory estimates for the major constituents and analytes of concern in the sludge and liquid above the sludge are summarized in Table ES-2. The sludge contained high concentrations of Fe, Na, and Al, and relatively high concentrations of zirconium, chromium, cadmium, uranium, nickel, SO_4^{2-}, NO_2^-, and NO_3^-. The results for iron, sodium, aluminum, uranium, SO_4^{2-}, NO_2^-, and NO_3^- are consistent with what was

expected to be in aging waste, and nickel and chromium are expected corrosion products from PUREX piping and process equipment. Also expected due to aging waste composition were the high levels of ^{90}Sr, ^{137}Cs, ^{106}Ru, and ^{241}Am.

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LIST OF TERMS

ACSL	Analytical Chemistry Service Laboratories
DOE	U.S. Department of Energy
DQO	Data Quality Objectives
DSC	differential scanning calorimetry
Ecology	Washington State Department of Ecology
EPA	U.S. Environmental Protection Agency
FIC	Food Instrument Corporation
HWVP	Hanford Waste Vitrification Plant
ICP	inductively coupled plasma
ND	not detected
NCAW	neutralized current acid waste
PCL	Process Control Laboratories
PNL	Pacific Northwest Laboratories
PUREX	Plutonium-Uranium Extraction (Plant)
RPD	relative percent difference
SpG	specific gravity
TCP	tank characterization plan
TGA	thermo-gravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
WHC	Westinghouse Hanford Company

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1.0 INTRODUCTION

This report presents an overview of double-shell underground storage tank 241-AZ-102 (tank 241-AZ-102) and its waste contents. It provides estimated concentrations and inventories for the waste components based on the latest sampling and analysis activities and background tank information. This tank characterization report for tank 241-AZ-102 describes the results of the four most recent sampling events. The first occurred in August of 1987 with the taking of a sludge and supernatant sample (Herting 1987). The second took place in October of 1987 and also included a sludge and supernatant sample (Herting 1988). A core sample consisting of two segments was obtained in 1989 (Gray et al. 1993). Finally, the tank waste was grab sampled in February of 1995 (Rollison 1995). Tank 241-AZ-102 is in active service; future plans include combining the waste in tank 241-AZ-102 with that in tank 241-AZ-101. Therefore, the composition of the tank waste can be expected to change. This report will be revised periodically to reflect new sample information and other changes. 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

The report summarizes the information about the use and contents of tank 241-AZ-102. 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-AZ-102.

1.2 SCOPE

The samples taken in 1987 were primarily intended to determine the composition of the solids which had accumulated on the floor of the tank. Chemical and radiochemical waste components were measured on the supernatant and sludge. No physical or thermodynamic analyses were performed. Other than total organic carbon (TOC), no specific organic analyses were performed.

The core sample obtained in 1989 was taken to characterize the neutralized current acid waste (NCAW) stored in tank 241-AZ-102 for support of retrieval, pretreatment, and disposal processes. Early characterization of NCAW was particularly important because at the time it was expected to be the first waste retrieved and vitrified in the Hanford Waste Vitrification Plant (Gray et al. 1993). Chemical, radiochemical and physical properties were measured on the supernatant and sludge phases of this sample. Other than TOC, no specific organic analyses were performed; and thermodynamic analyses were not conducted.

The supernatant grab samples acquired in 1995 were taken to support tank operations in assessing the compatibility of tank 241-AZ-102 supernatant with other waste for transfer purposes. The technical basis for this compatibility assessment is described in the *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994). The sampling and analysis were performed as described in *Tank 241-AZ-102 Tank Characterization Plan* (Schreiber 1995). The supernatant was analyzed for a smaller set of metals and radiochemical constituents than the 1989 core sampling event to comply with the requirements of Schreiber (1995). Thermodynamic analyses including thermo-gravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis were performed. The grab samples were not safety screened since the sampling event did not meet the criteria defined in the *Safety Screening Data Quality Objectives* (Babad and Redus 1994), which specifies that at least widely spaced two risers be sampled at 3 different waste levels to achieve a vertical waste profile (Schreiber 1995).

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

Terms such as waste types, waste generating processes, etc., generally are not defined in this document; detailed explanations of these and many other Tank Farm/Hanford Site terms can be found in the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

2.0 HISTORICAL TANK INFORMATION

Tank 241-AZ-102 is in active service and can be expected to have additional transfers to and from the tank that will alter the volume and composition status of the tank. The most current volume is provided in reports that are routinely updated (e.g., Hanlon [1995]). Tank history includes tank design information, waste transfer history, and waste temperature and level surveillance information.

2.1 TANK STATUS

According to Hanlon (1995), tank 241-AZ-102 contains 3,590 kL (949 kgal) of waste; 360 kL (95 kgal) are sludge and 3,230 kL (854 kgal) are supernatant. The sludge volume estimate reported in Hanlon (1995) is based on a measurement obtained on 6/4/92 by an in-tank sludge measurement device. The waste depth has been measured by both manual tape and Food Instrument Corporation (FIC) automatic surface level gauge to be 877 cm (345 in.) (Hanlon 1995). The current designation of the tank's contents is aging waste.

A maximum internal tank temperature of 83 °C (182 °F) was recorded on May 1, 1995. Waste levels and tank temperatures are further discussed in Section 2.4. The tank is classified as a structurally sound, non Watch List tank. There are no unreviewed safety questions associated with Tank 241-AZ-102 at this time. All tank monitoring instruments are in compliance with documented standards with the exception of erratic leak detection pit radiation readings (Hanlon 1995).

Tank 241-AZ-102 is actively ventilated and remains in service. The in service designation allows the tank to continue to receive waste to support continued operations.

2.2 TANK DESIGN

Tank 241-AZ-102 is a double-shell tank consisting of a heat-treated (stress-relieved) primary steel liner (resting on a concrete insulating pad) inside a second steel liner encased in a reinforced concrete shell (WHC 1994). The tank has a design capacity of 3,790 kL (1,000 kgal); however, safety considerations limit the maximum operating volume to about 3,710 kL (980 gal) (Hanlon 1995; Baumgartner, Pines, and Soldat 1983; ICF Kaiser 1994; and Welty 1988). The tank has a diameter of 22.9 m (75 ft) and a usable depth of about 8.8 m (29 ft). Aging waste tanks have special support systems to allow the tanks to store high-heat generating wastes with minimum probability of loss of integrity. These systems include a tank preheating system comprised of a steam coil, a waste mixing system comprised of 22 airlift circulators, and an exhaust condenser system to control condensate in the exhaust stream (WHC 1994). Maximum design temperatures for this tank are: 179 °C (355 °F) for sludge, 127 °C (260 °F) for liquid, and 104 °C (220 °F) for vapor (WHC 1994). Instruments access the tank through risers and monitor the pressure,

temperature, liquid level, sludge level, and other bulk tank characteristics. The locations of these risers are depicted in Figure 2-1. Waste entered the tank through piping which penetrated the sidewall; waste could be removed by a transfer pump entering the tank through riser 6 near the center of the tank (Vitro Engineering 1973). A diagram of a double-shell aging waste tank is presented in Figure 2-1. For more information about the AZ Tank Farm and double-shell tanks in general, see the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

Construction of tank 241-AZ-102 was completed in 1975 and was brought into service in 1976 as a receiver for high-heat producing waste from PUREX Plant and B Plant (Baumgartner, Pines, and Soldat 1983). It is one of two tanks that comprise the AZ Tank Farm located near the eastern fence of the 200 East Area. Figure 2-2 depicts the location of the AZ Tank Farm.

2.3 PROCESS KNOWLEDGE

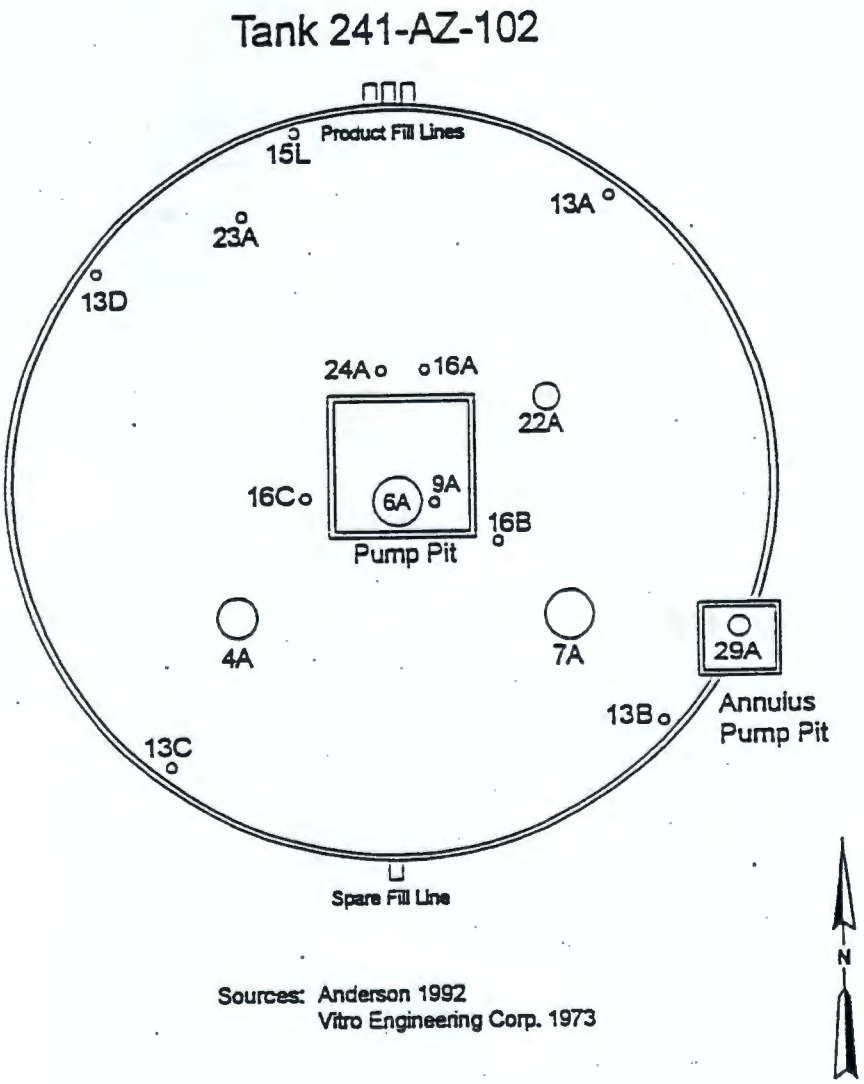
Most of the waste now stored in the tank consists of water and aging waste from PUREX (Agnew et al. 1995). In addition to the large amounts of cesium and strontium, the sludge is expected to contain significant concentrations of some transuranic (TRU) radionuclides. A summary of the transfer history for this tank is found in Section 2.3.1.

The fill history of Tank 241-AZ-102 can be described as receiving high-level B Plant waste and concentrated wastes from 1976-80. Transfer history documents indicate the tank was last emptied in 1986; however, a minimal heel of settled solids may have remained in the tank. The tank was then refilled with PUREX aging waste which, along with water, has remained in the tank. The tank has received only small transfers of water and dilute wastes since 1990. Waste transfer information in this section comes from Agnew et al. (1995), Jungfleisch (1984), and Anderson (1990). Table 2-1 uses a compilation of transaction records to present an estimate of the total volume of the various waste types that have entered the tank from 1986 to January 1995. Figure 2-3 illustrates the service life of Tank 241-AZ-102 from 1976 to January 1995.

2.3.1 Waste Transfer History

Tank 241-AZ-102 first received aging and high strontium wastes in 1976 (Anderson 1990; Agnew et al. 1995). Further additions of residual liquor and complexant concentrate waste brought the tank to near capacity by late 1980. From 1980 to 86 large amounts of supernatant were removed from and added to the tank in conjunction with 242-A Evaporator operations.

Figure 2-1. Tank 241-AZ-102 Riser Configuration.



Sources: Anderson 1992
Vitro Engineering Corp. 1973

No.	Dia.	Description and Comments
4A	20"	Tank Ventilation
6A	42"	Transfer Pump
7A	42"	Steam Coils
9A	4"	Pump Pit Drain
13A	4"	Thermocouple Probe
13B	4"	Thermocouple Probe
13C	4"	Thermocouple Probe
13D	4"	Thermocouple Probe
15L	6"	Spare
16A	4"	Sludge Temperature Probe
16B	4"	Sludge Temperature Probe
16C	4"	Sludge Temperature Probe
22A	16"	FIC Gauge
23A	4"	Manual Tape
24A	4"	Sludge Measurement Port
29A	12"	Annulus Pump

Figure 2-2. Aging Waste Tank Design.

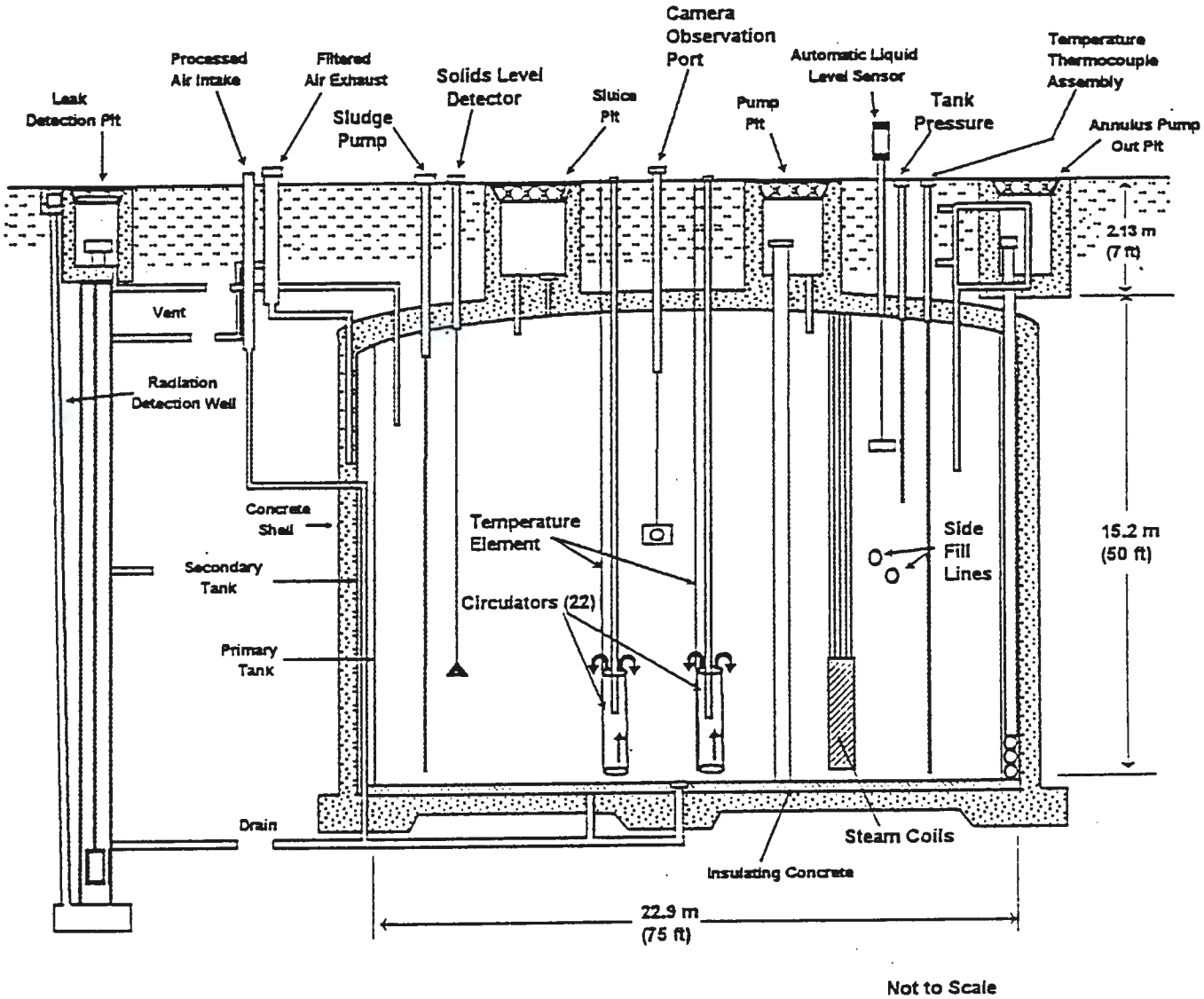


Table 2-1. Estimated Waste Types Received by Tank 241-AZ-102.[a]

Waste type	Estimated cumulative volume		Years received
	(kL)	(kgal)	
PUREX NCAW	1,525	403	1986 to 1990
Water[b]	3,267	863	1986 to 1995
PUREX miscellaneous waste	45	12	1988 to 1990

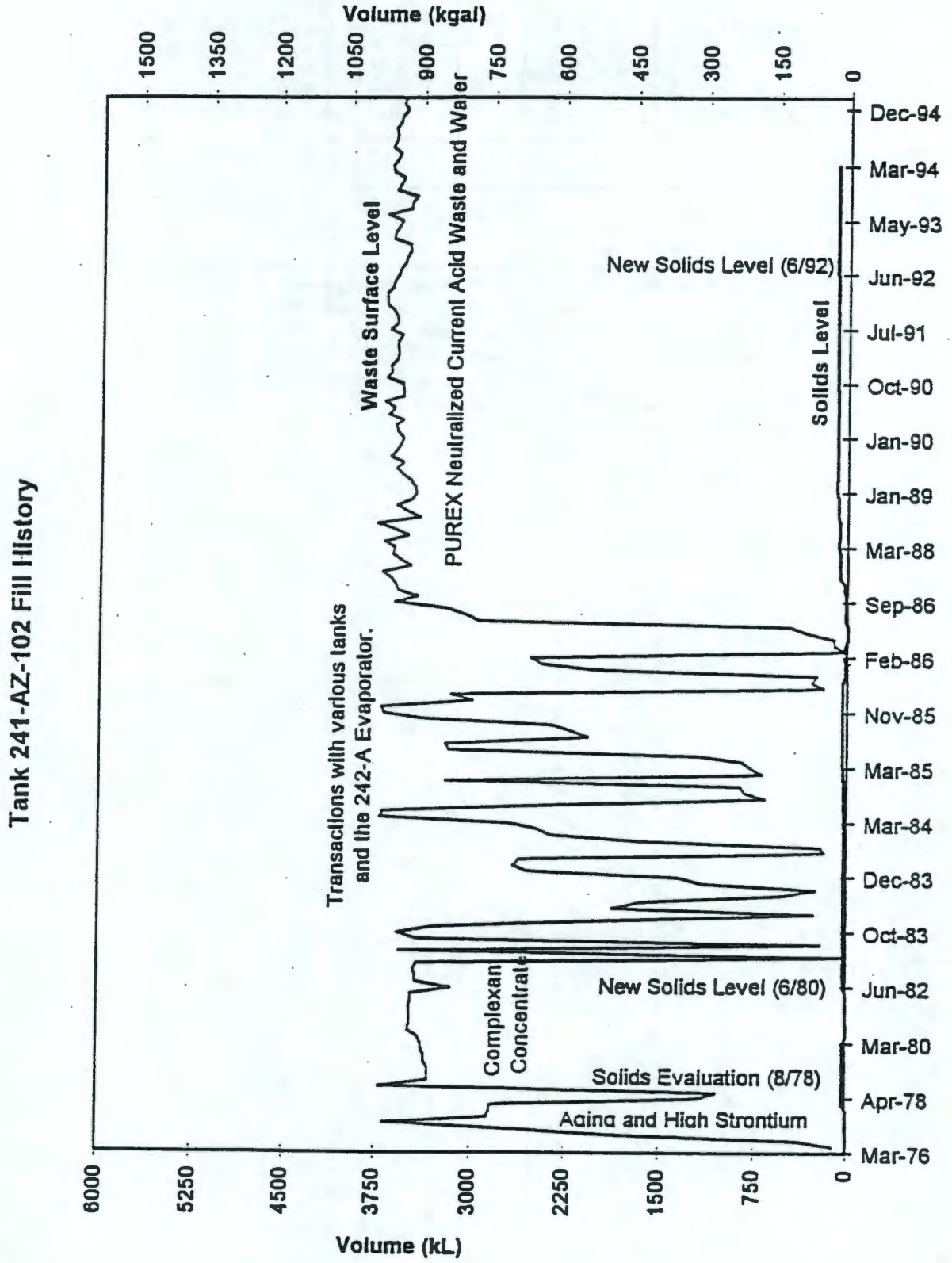
[a]Agnew, S. F., P. Baca, R. Corbin, T. Duran, and K. Jurgensen, 1995, *Waste Status and Transaction Record Summary for the Northwest Quadrant*, WHC-SD-WM-TI-689, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

[b]As NCAW is added to the tank, water is evaporated and removed; the liquid level in the tank remains constant, but the concentration of chemicals increases.

PUREX = Plutonium-Uranium Extraction (Plant).

NCAW = neutralized current acid waste.

Figure 2-3. Tank 241-AZ-102 Fill History.



In 1986, the tank was pumped to a minimal heel, refilled with a pre-determined amount of dilute waste/water to allow air lift circulator operation, and began receiving PUREX NCAW. NCAW is derived from the waste stream produced from the first-cycle solvent extraction step at PUREX (Cruzen et al. 1988). Following the removal of cladding, irradiated nuclear fuel was dissolved in nitric acid. The dissolved nuclear fuel was then fed to a solvent extraction system that removed most of the neptunium, plutonium, and uranium. The solvent extraction system includes 14 solvent extraction columns. The waste stream was then concentrated and most of the nitric acid was extracted; the waste was further de-nitrated with sucrose and neutralized with sodium hydroxide before being transferred to aging-waste tanks for storage. The resultant waste (NCAW) produces significant decay heat and can self-boil if tank ventilation is not provided. The estimated composition of typical PUREX NCAW is presented in Table 2-2. The volumes of waste received by tank 241-AZ-102 since it was emptied in 1986 are presented in Table 2-1. The tank received its final transfer of PUREX NCAW in 1990 bringing its total waste volume to 3,600 kL (951 kgal). Since 1986, water has been added and removed in small amounts, small transfers of dilute PUREX miscellaneous waste (i.e., ^{237}Np containing waste from the PUREX F-16 tank) and dilute waste from tank 241-AZ-101 were received.

Table 2-2. PUREX Aging (High-Level) Waste Composition.[a]

Component	Predicted Sludge (μg/g)	Predicted Supernatant (μg/L)
Sodium	1.17 E+05	1.93 E+07
Iron	8.47 E+04	1.17 E+05
OH^-	1.19 E+05	1.67 E+05
NO_3^-	2.68 E+03	3.53 E+07
NO_2	3.61 E+01	4.74 E+05
SO_4^{2-}	1.51 E+05	1.15 E+07
SiO_3^{2-}	2.69 E+04	1.52 E+06
Uranium	9.74 E+04	9.52 E+05
Plutonium	2.45 (μCi/g)	4.81 E+01 (μCi/L)
Cesium	3.23 E+02 (μCi/g)	4.25 E+06 (μCi/L)[b]
Strontium	5.27 E+04 (μCi/g)[b]	3.35 E+04 (μCi/L)

[a]Agnew, S. F., P. Baca, R. Corbin, T. Duran, and K. Jurgensen, 1995, *Waste Status and Transaction Record Summary for the Northwest Quadrant*, WHC-SD-WM-TI-689, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

[b]When compared with PUREX process/operations logs, these values seem to be extremely exaggerated.

2.3.2 Historical Estimation of the Contents of Tank 241-AZ-102

Tank inventory estimates based on the Tank Layer Model, the Waste Status and Transaction Record Summary, and the Hanford Defined Wastes Types developed by Los Alamos National Laboratory were not yet available for tank 241-AZ-102 when this report was compiled.

The current contents of tank 241-AZ-102 consists of NCAW from the PUREX process and dilute noncomplexed waste. A minimal remnant heel of solids left in the tank from the waste it stored prior to 1986 may exist below the solids that have settled from the aging waste. Based on current information (Hanlon 1995), the waste is expected to have two primary layers; the bottom layer is composed of 360 kL (95 kgal) of sludge, and the top layer is composed of 3,230 kL (854 kgal) of supernatant (Hanlon 1995). Iron solids are expected to be a main component of the sludge due to the large quantity of iron sulfamate used in the PUREX process (Nguyen 1988). Additionally, corrosion iron from piping and process vessels is expected to exceed the amount of iron added as essential material at PUREX. The exact composition of NCAW was continually changing with each processing run. However, there were several analytes that were always present in each run. These analytes, in addition to those listed in Table 2-2, are also indicators of NCAW. These analytes include ^{241}Am, chromium, nickel, ^{95}Nb, ^{95}Zr, ^{106}Ru, and ^{144}Ce (Cruzen et al. 1988 and Allison 1986). Minor amounts of zirconium that escaped removal during the decladding process are likely. Cadmium was also introduced into the waste stream as a neutron poison.

Historical sampling of this tank also provides an estimation of the waste constituents and is discussed in detail in Sections 3 and 4.

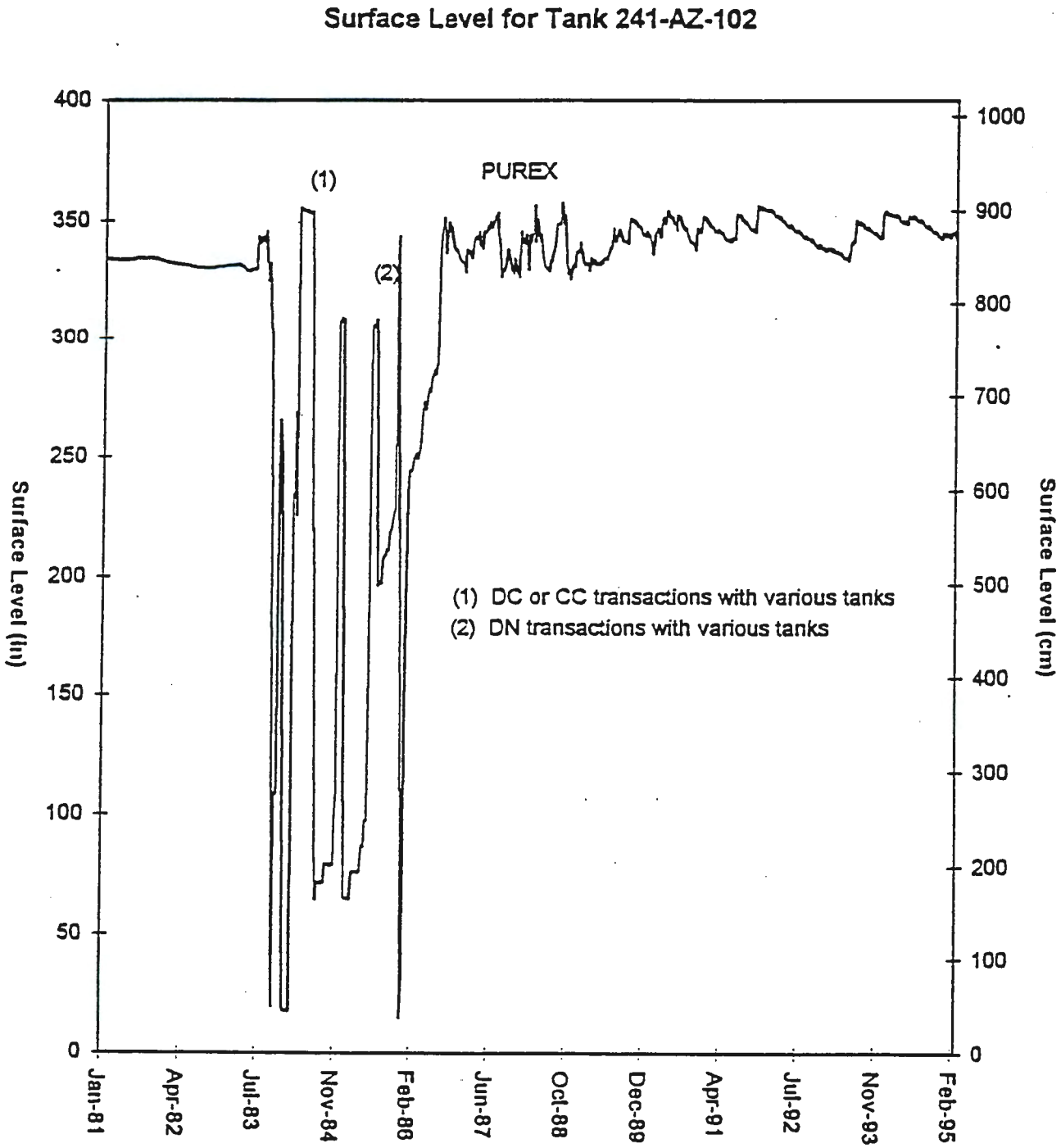
2.4 SURVEILLANCE DATA

2.4.1 Surface Level Readings

To determine the surface level of the waste, tank 241-AZ-102 is equipped with both a liquid level gauge manufactured by the FIC which can be monitored either automatically or manually and a manual tape. The FIC gauge uses a conductivity probe to detect the level of the tank's contents and, in the automatic mode is electrically connected to the Computer Automated Surveillance System.

The manual tape is used for level detection when the FIC indicator is out of service. The surface level of the tank is measured daily and is presented graphically in Figure 2-4 for the period of January 1981 to February 1995. The last surface level measurement was 877 cm (345 in.) according to Hanlon (1995).

Figure 2-4. Tank 241-AZ-102 Surface Levels
January 1981 to February 1995.



2.4.2 Internal Tank Temperatures

To measure in-tank temperatures, probes with 18 thermocouples assembled in a pipe (called a thermocouple tree) are inserted into tank 241-AZ-102 at Risers 13A, 13B, 13C, and 13D (Figure 2-5). Each thermocouple tree monitors temperatures at 3 elevations within the tank. The type of thermocouple used in this tank is an Iron-Constantan (type J). All thermocouple trees are currently operating. Temperature readings are downloaded to the Surveillance Analysis Computer System weekly.

Maximum sludge temperature readings, as well as the average solution temperatures for tank 241-AZ-102 from December 1993 through December 1994 are plotted in Figure 2-6. This was the only data available at the time this report was published. As illustrated in Figure 2-6, maximum sludge temperatures in the tank are stable; they range between 84 °C (183 °F) and 79 °C (174 °F). The plotted solution temperature appears to increase slightly over the period plotted. All recorded temperatures are within design limits of the tank and no temperature anomalies were identified.

2.4.3 In-Tank Photographs

The interior of tank 241-AZ-102 was last photographed on December 1984 (Hanlon 1995). This tank is currently in use and has been very active since the photograph was taken. This photograph no longer reflects the current appearance of the waste surface and is not included in this report.

Figure 2-5. 200 East Area and Location of the 241-AZ Tank Farms.

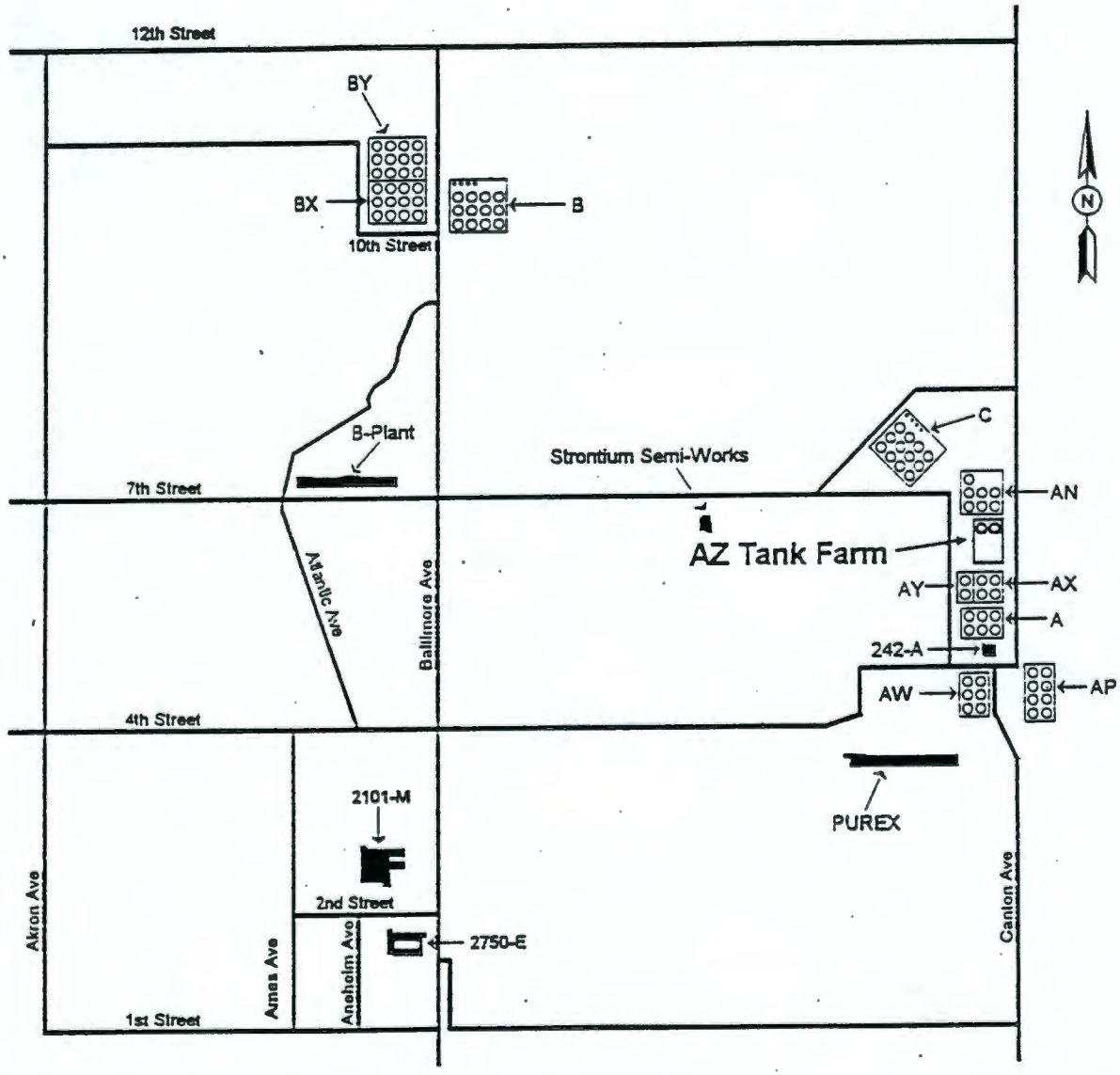
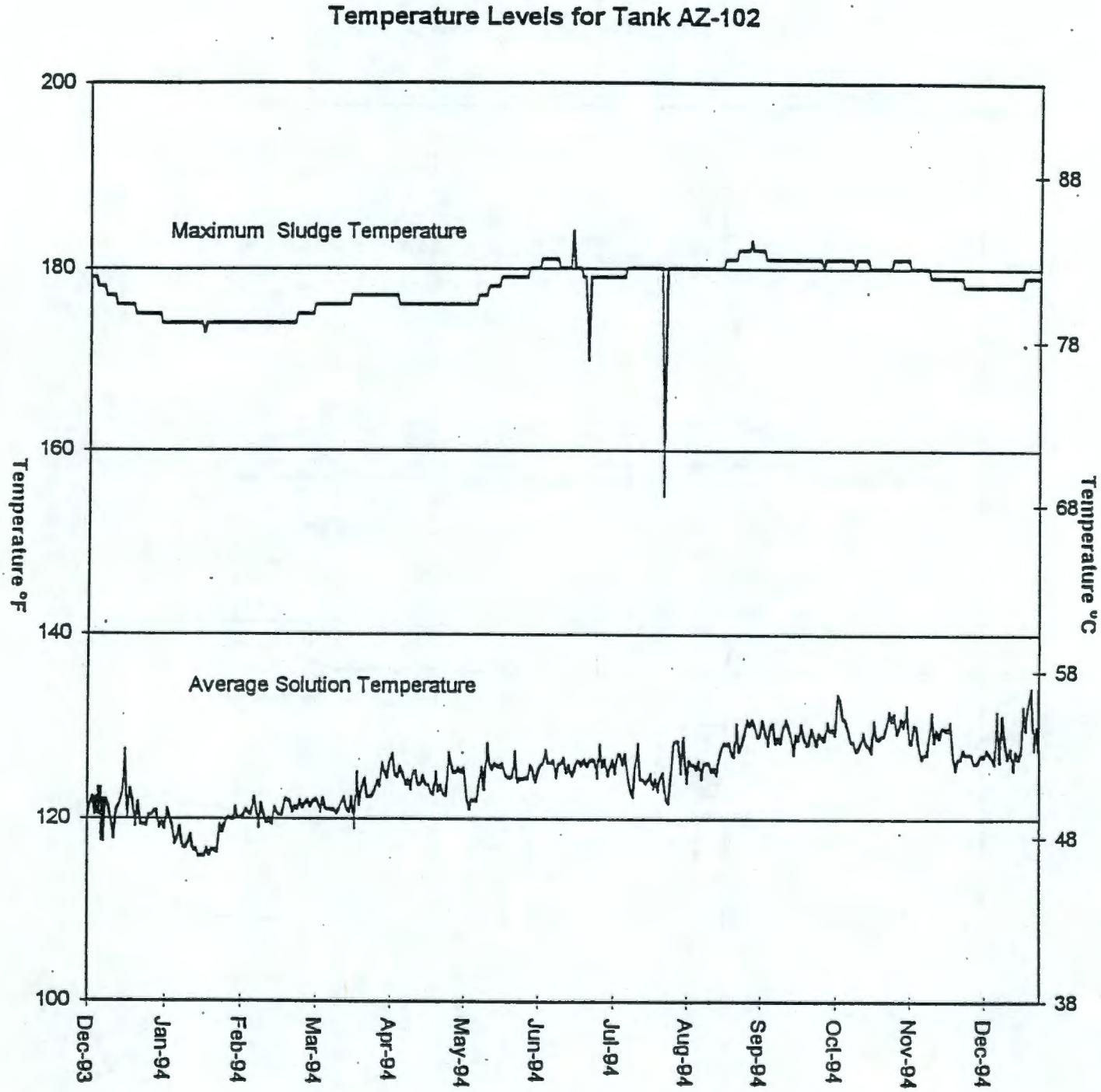


Figure 2-6. Tank 241-AZ-102 Temperatures
December 1993 to December 1994.



3.0 TANK SAMPLING OVERVIEW

This section describes the four latest sampling and analysis events for tank 241-AZ-102. Grab samples of liquid and sludge were taken in both August and October of 1987 and a core sample was taken in May 1989. Grab samples of tank liquids and sludge were most recently taken in February 1995. Sludge components can be best estimated from the 1989 core sample since the other sludge samples (1987) were taken by the grab method. The lone sludge grab sample taken in 1995 for process testing purposes has not yet been analyzed. Analysis of this sludge sample is planned and the results will be included in a future revision of this document. Physical measurements were performed on both the 1989 sludge and 1995 supernatant samples. The 1987 and 1989 sample events were performed to support characterization, retrieval, and handling of the waste in the tank and the 1995 samples were performed to evaluate the compatibility of the waste for future transfers. A further description of the sampling procedures may be found in the *Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

Supernatant and sludge sampling occurred prior to 1987 for various purposes and the results of these analyses are not considered to be an accurate representation of the sludge/supernatant currently in the tank because in 1986 the tank was pumped to a minimum heel to support PUREX operations with the receipt of NCAW.

3.1 DESCRIPTION OF AUGUST 1987 SAMPLING EVENT

In August of 1987, a supernatant and a sludge sample were obtained from tank 241-AZ-102. The supernatant sample, numbered T-3495, was a clear, pale yellow liquid with a trace of reddish-brown solids. The sludge sample, numbered T-3496, was 100% dark brown solid material. The riser(s) that these samples were taken from is unknown from existing documentation.

3.1.1 Sample Handling (August 1987)

The supernatant and sludge samples were delivered to the Process Chemistry Laboratories (PCL). An undiluted sample of the supernatant was delivered to the Analytical Chemistry Service Laboratories (ACSL) for analysis.

Two aliquots of the sludge sample were taken and labeled "Sample A" and "Sample B." The aliquots were centrifuged and the supernatant discarded. The solids were water washed and recentrifuged. The water wash supernatants were decanted and submitted to the ACSL for analysis. The centrifuged solids were dissolved in acid and submitted to the ACSL for analysis.

3.1.2 Sample Analysis (August 1987)

The water wash supernatant samples were analyzed for anions, a few metals, pH and specific gravity (SpG), TOC, and three radionuclides. The solid samples were analyzed for total metals, TOC, and seven radionuclides. The analytical results can be found in Appendix C. Lists of the analytical procedures for this and subsequent sampling events can be found in Appendix D. Since laboratory procedures and their identifying numbers are periodically revised over the years, those listed in Appendix D are not necessarily the exact analyses conducted by the laboratory in 1987. Furthermore, procedure numbers are not specifically stated within the 1987 data packages.

3.2 DESCRIPTION OF OCTOBER 1987 SAMPLING EVENT

In October of 1987, a supernatant and a sludge sample were received by the PCL. The supernatant sample was a pale yellow liquid with approximately 0.15 vol% solids, which were brown in color. The sludge sample was very dark brown in color, and contained 65 wt% solids. The riser from which the samples were obtained is unknown.

3.2.1 Sample Handling and Analysis (October 1987)

A sample of the supernatant was submitted without dilution to the ACSL for analysis. Two solid samples were submitted to the ACSL for analysis after water wash, centrifugation, and dissolution, as described above in Section 3.1.1. The samples were labeled "Sample A" and "Sample B." The supernatant and the solid samples were analyzed for three metals, eight anions, TOC, pH, and seven radionuclides. The results can be found in Appendix C.

3.3 DESCRIPTION OF MAY 1989 SAMPLING EVENT

In May and July of 1989, the Pacific Northwest Laboratories (PNL) received and extruded a 2-segment core sample from tank 241-AZ-102. The core was obtained through riser 15L using a core sample collection system, mounted on a special truck (Gray et al. 1993).

The core sample from tank 241-AZ-102 consisted of two segments, described in Table 3-1. The first segment (Sampler #89-016) weighed 293 g, of which 268 g were solids and 25 g were liquid. The segment was dark brown with a glossy surface. Its length was 39.4 cm, and it held its shape after extrusion. The second segment (Sampler #89-017) weighed 287 g, and contained no liquids. It had a length of 46 cm, with a very dry and crumbly texture.

Table 3-1. Tank 241-AZ-102 May 1989 Core Sample Description.

Segment	Solids (g)	Liquids (g)	Length (cm)	Color	Texture
Top	268	25	39.4	Dark brown and glossy	Cohesive
Bottom	287	0	46	Light brown (bottom end) and chocolate brown (remainder)	Dry and crumbly

3.3.1 Sample Handling (May 1989)

After receipt and extrusion of the two segments from core 1, the samples were subjected to various decanting, centrifuging, and compositing processes in order to prepare the samples for analysis. The analyses included those for characterization of the waste, a test which simulated B Plant waste, and a test of the washed solids slurry to evaluate the B Plant simulation. The B Plant waste simulation and evaluation tests are not discussed in this report.

As presented in Figure 3-1, the two segments underwent extensive preparation for physical and chemical analyses. Subsequent to extrusion and prior to preparation, samples were obtained from the segments for Layer Tests, and physical tests were performed. A composite solid sample was formed from both segments. A portion of the composite sample was then centrifuged and the supernatant added to that from the top segment. After physical tests were performed on the top segment and composite sample, the liquid and solid material from both segments were combined in a composite sample which was analyzed for density prior to centrifugation. A complete list of constituents was analyzed for subsequent to centrifugation. The calculated analyte concentrations and projected inventory estimates reported in Section 4 and Appendix A are based on the results from these analyses. Portions of the composite solids from the bottom segment were combined with portions of the supernatant from the top segment to form a 40% and a 10% slurry solution, which were used for physical tests.

3.3.2 Sample Analysis (May 1989)

As stated in Section 3.1.2, a list of sample procedures and numbers can be found in Appendix D. Two fusions were used for the inductively coupled plasma (ICP)/atomic emission spectroscopy analyses and most of the radiochemical analyses of solid samples: one using sodium peroxide in a zirconium crucible, the other using potassium hydroxide in a nickel crucible. Following fusion, the samples were dissolved in hydrochloric acid and diluted to a known volume. Most of the other analyses were performed either directly

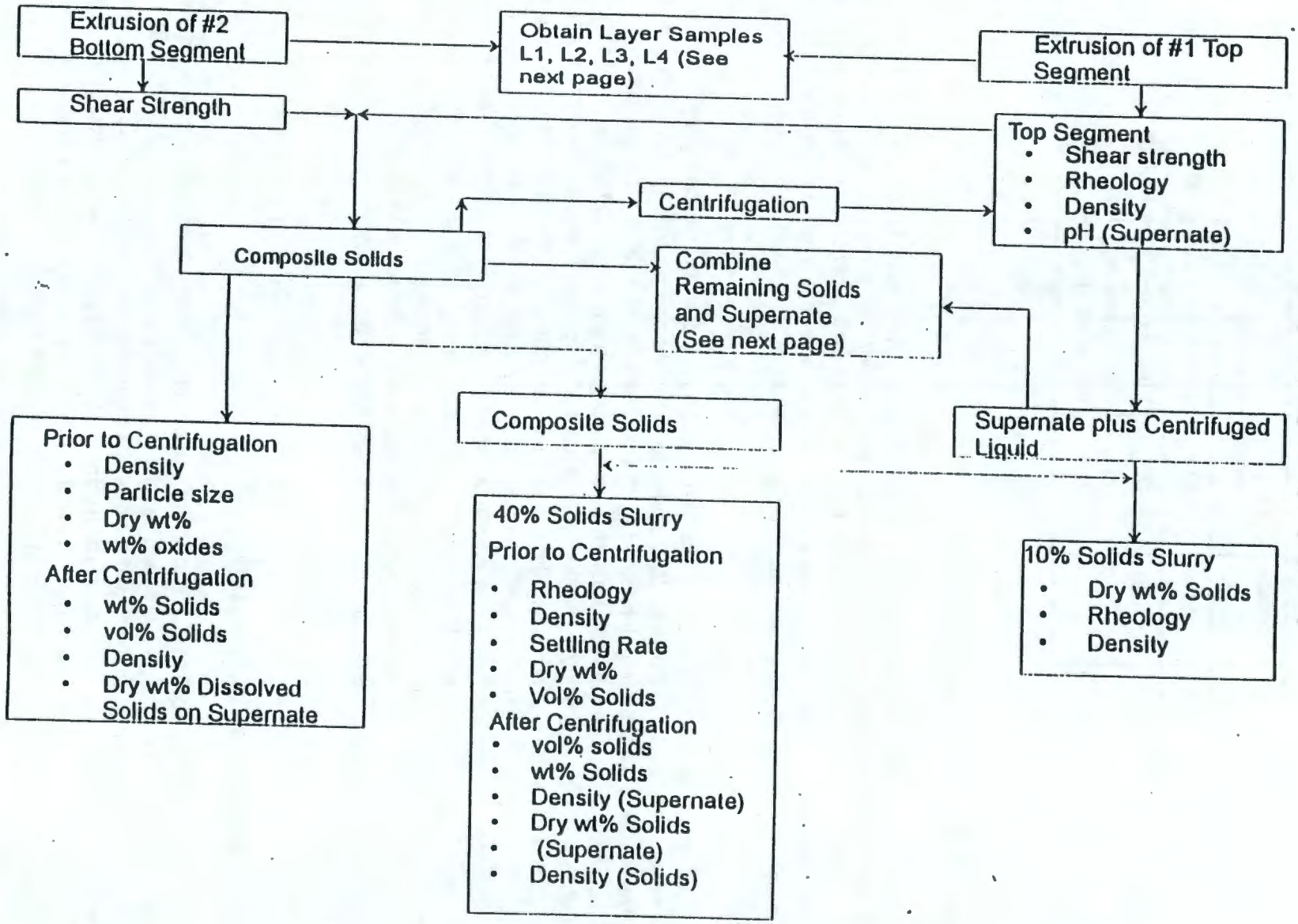


Figure 3-1. Flow Chart for Sample Preparation and Analysis (May 1989). (2 sheets)

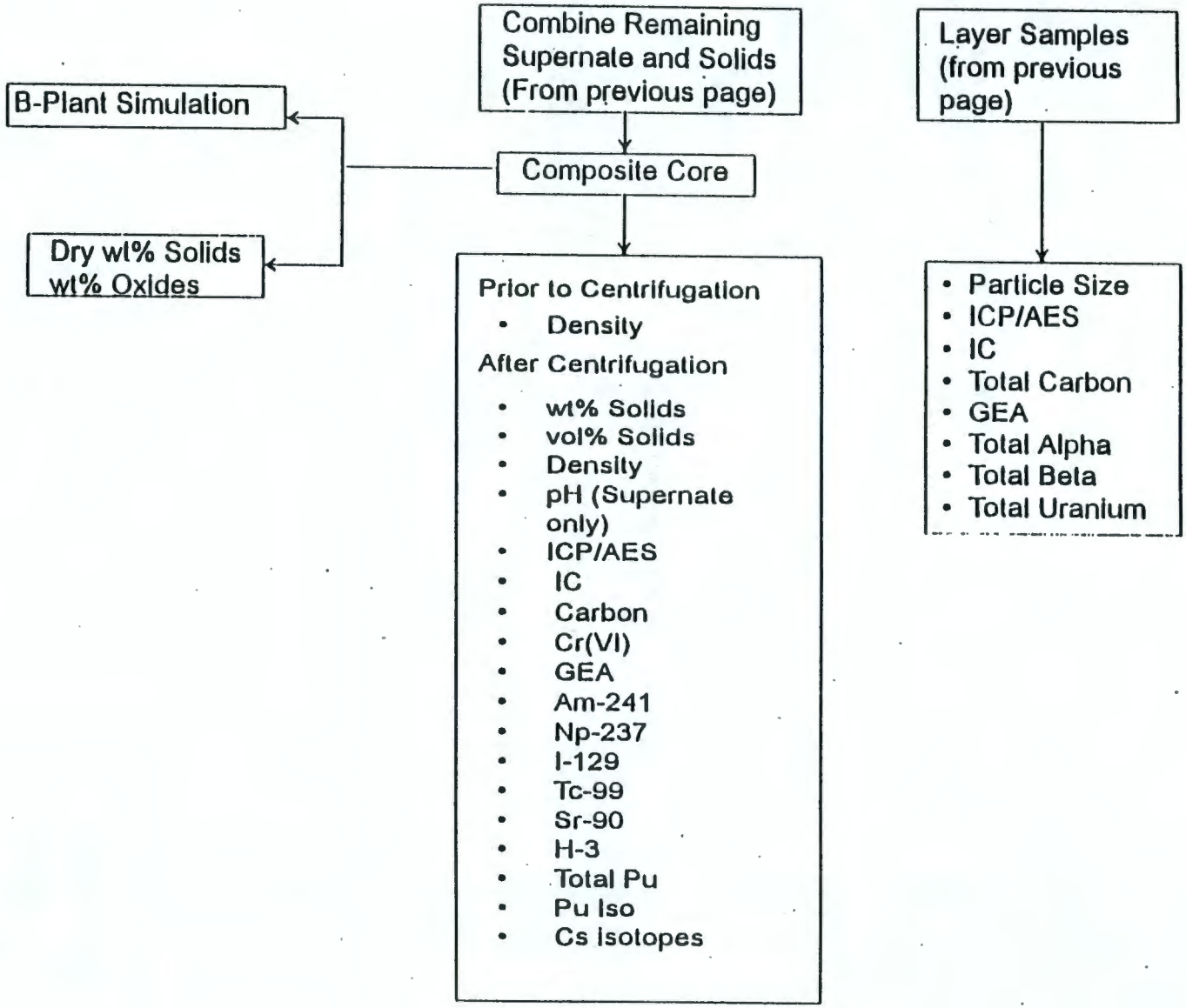


Figure 3-1. Flow Chart for Sample Preparation and Analysis (May 1989). (2 sheets)

on the sample, or on a leachate from the sample. The analytical results of the 1989 sample event were used in conjunction with the 1995 supernatant samples, described in Section 3.4, to characterize the tank. The chemical and radiochemical results from the 1989 sampling event are tabulated in Appendix A. Results of the physical property measurements can be found in Section 4.3.

3.4 DESCRIPTION AND HANDLING OF FEBRUARY 1995 SAMPLING EVENT

On February 23, 1995, four samples were obtained from tank 241-AZ-102 and submitted to the 222-S Laboratory. The samples were taken from riser 24A using the bottle-on-a-string sampling method (Schreiber 1995). The samples were acquired to determine the compatibility of the waste in the tank for future receipt of waste (Carothers 1994). No field blank was taken because of the expected high concentrations of analytes in the tank (Schreiber 1995). No physical description of the samples or the sample preparation steps was available from existing documentation. Table 3-2 presents the riser number, sample numbers, and the depths for the grab samples.

Table 3-2. Tank 241-AZ-102 February 1995 Grab Sampling Depths.[a]

Sample number	Sample type	Sample location	Sample depth[b] (m)
102-AZ-1	Supernatant	Riser 24A	8.38
102-AZ-2	Supernatant	Riser 24A	12.4
102-AZ-3	Supernatant	Riser 24A	16.2
102-AZ-4	Sludge	Riser 24A	16.9

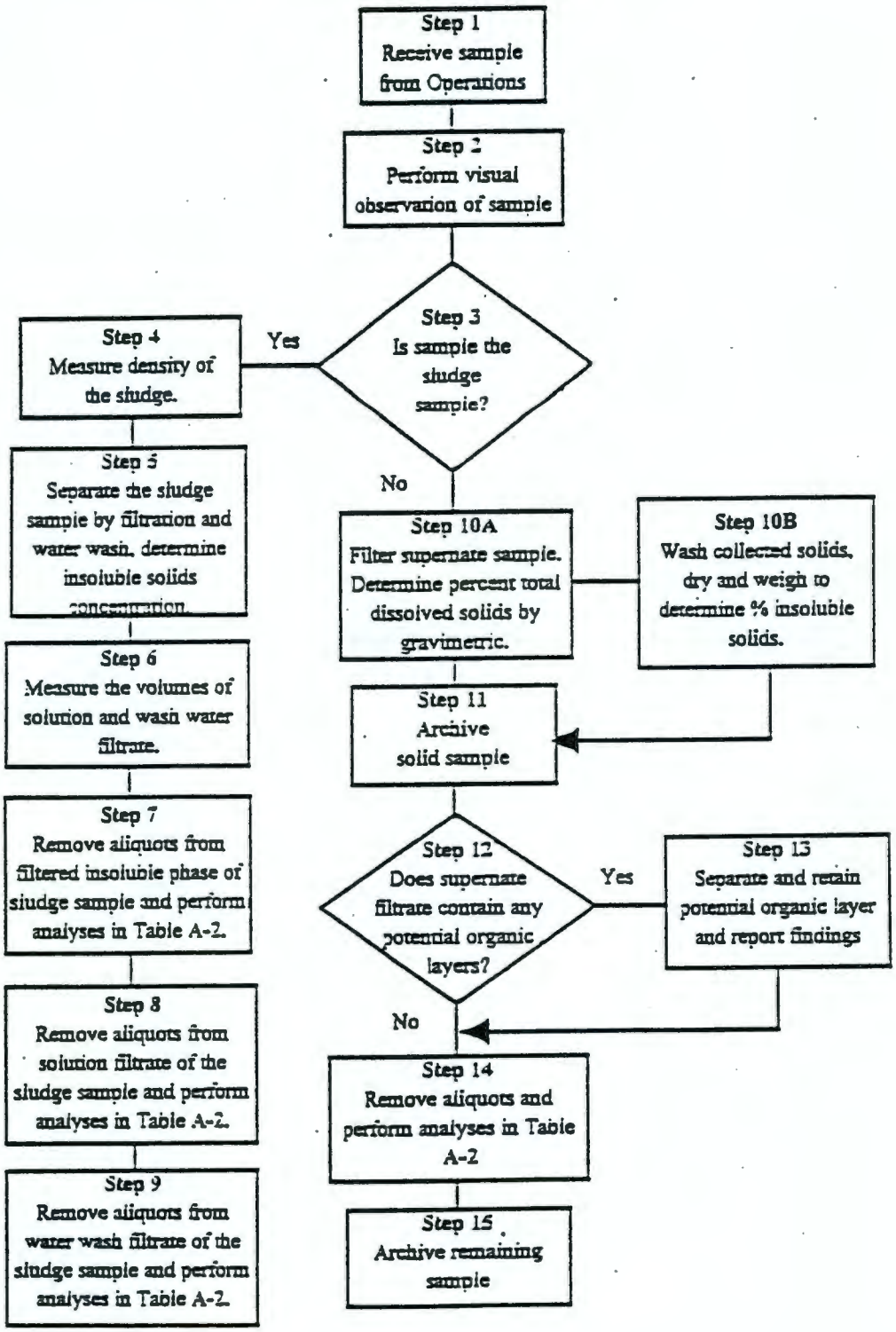
[a]Schreiber, R. D., 1995, *Tank 241-AZ-102 Tank Characterization Plan*, WHC-SD-WM-TP-228, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[b]Sample depth is defined as the distance from the top of the riser to the mouth of the sample bottle.

3.4.1 Sample Analysis (February 1995)

The 1995 grab samples were analyzed in accordance with the requirements of Schreiber (1995) and Figure 3-2. A list of the analytical procedures can be found in Appendix D. The results of the analyses were used, along with the results of the 1989 core samples, to estimate the total inventories of each evaluated analyte. The results are tabulated in Appendix B. To date, only the three supernatant samples have been evaluated. Analysis of the lone sludge sample (102-AZ-4) was requested in the tank characterization plan (TCP) (Shreiber 1995), but as of yet has not been performed. Since no analysis results are available for this sample, any further discussion of this sample is excluded from this revision of this report.

Figure 3-2. Flow Chart for Data Collection and Preparation (February 1995).



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4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

The purpose of this section is to summarize the sampling and analytical results from the most recent sludge and supernatant samplings, as reported in the appendices. This section provides an estimate of the analyte concentrations in both the sludge and supernatant. In addition, an estimate of the total amount (inventory) of the analyte in both waste phases is presented based on the volumes of the sludge and supernatant layers estimated to be in the tank. The summary tables and appropriate appendices where the data can be found in this document are summarized in Table 4-1. Data regarding the physical characteristics of the sludge portion of the waste are presented and discussed in Section 4.3. All sampling events from which the reported results were derived are described in Section 3.

Table 4-1. Analytical Presentation Tables.

Analysis	Tabulated result location
Tank 241-AZ-102 Chemical Composition Summary	Table 4-2
Analytical Summary for the Sludge	Appendix A and Table A-1
1995 Grab Sample Results	Appendix B

The sludge composition is based on the analyses of tank 241-AZ-102, core 1, taken in 1989. The data from these analyses were obtained from Gray et al. (1993), are reported in Appendix A, and are summarized in Table 4-2. The calculated analyte concentrations for the sludge are a summation of the results acquired from centrifuged solid and centrifuged liquid samples from the sludge (refer to Appendix A for an explanation of this calculation). With regard to the centrifuged solids, the samples designated for ICP analysis were prepared by two separate fusion methods. All of the metals with the exception of potassium and nickel were analyzed after a fusion performed by potassium hydroxide in a nickel crucible. A sodium peroxide fusion in a zirconium crucible was used to evaluate potassium and nickel. No comments concerning precision were provided in existing documentation.

The supernatant composition and inventory for the tank was based on the results from the 1995 grab samples (Rollison 1995) as presented in Appendix B. A simple mean was calculated from the results for each analyte. This composition and inventory may change if transfers into and out of the tank occur.

Table 4-2. Analytical Summary for Waste in Tank 241-AZ-102. (5 sheets)

Analyte	1989 Sludge sample				1995 Grab samples	
	Centrifuged sludge concentration	Interstitial liquid concentration	Calculated average sludge concentration[a]	Total projected sludge inventory[b]	Liquid concentration[c]	Total projected supernatant inventory[d]
Metal	µg/g	µg/g	µg/g	kg	µg/g	kg
Al	53,700.0	89.1	38,688.9	19,196.3[e]	140.9	501.5
Sb	<3,410.0	<9.0	ND	ND	--	--
As	<592.0	3.8	<427.3	<212.0	--	--
Ba	553.0	0.7	398.4	197.7	--	--
Be	13.5	0.1	9.7	4.8	--	--
B	<497.0	7.3	<359.9	<178.6	--	--
Cd	15,000.0	1.7	10,800.5	5,358.9	--	--
Ca	3,020.0	5.3	2,175.9	1,079.6	--	--
Ce	<785.0	10.4	<568.1	<281.9	--	--
Cr	1,920.0	879.0	1,628.5	808.0	879.0[f]	3,128.9
Co	<1,470.0	7.5	<1,060.5	<526.2	--	--
Cu	350.0	2.7	252.8	125.4	--	--
Dy	<50.4	0.8	<36.5	<18.1	--	--
Fe	131,000.0	9.5	94,322.7	46,800.1	<9.1	<32.4
La	4,500.0	1.5	3,240.4	1,607.8	--	--
Pb	1,100.0	5.2	793.5	393.7	--	--
Li	<29.2	0.1	<21.0	<10.4	--	--
Mg	950.0	0.8	684.2	339.5	--	--
Mn	2,880.0	0.7	2,073.8	1,028.9	--	--
Mo	47.0	31.9	42.8	21.2	--	--
Nd	2,910.0	4.8	2,096.5	1,040.2	--	--
Ni	8,860.0	1.9	6,379.7	3,165.4	--	--
P	977.0	58.6	719.8	357.2	--	--

Table 4-2. Analytical Summary for Waste in Tank 241-AZ-102. (5 sheets)

Analyte	1989 Sludge sample				1995 Grab samples	
	Centrifuged sludge concentration	Interstitial liquid concentration	Calculated average sludge concentration[a]	Total projected sludge inventory[b]	Liquid concentration[c]	Total projected supernatant inventory[d]
Total Pu[g]	59.6	0.5	43.0	21.4	--	--
K	2,080.0	1,260.0	1,850.4	918.1	--	--
Re	<72.6	0.7	<52.5	<26.0	--	--
Rh	<422.0	11.2	<307.0	<152.3	--	--
Ru	<182.0	4.2	<132.2	<65.6	--	--
Se	<1,030.0	9.5	<744.3	<369.3	--	--
Si	4,480.0	484.0	3,361.1	1,667.7	484.0[f]	1,722.8
Ag	674.0	6.0	487.0	241.6	--	--
Na	59,100.0	42,100.0	54,340.0	26,961.9	4,836.4	17,215.6
Sr	327.0	0.2	235.5	116.8	--	--
Te	<345.0	2.3	249.0	123.6	--	--
Tl	<8,380.0	33.3	33.3[h]	16.5	--	--
Th	<487.0	<1.3	ND	ND	--	--
Ti	77.6	0.4	56.0	27.8	--	--
Total U[g]	12,600.0	1,400.0	9,464.0	4,695.8	1,400.0[f]	4,983.4
V	<32.6	1.8	<24.0	<11.9	--	--
Zn	141.0	0.3	101.6	50.4	--	--
Zr	18,100.0	3.7	13,033.0	6,466.6	--	--
Ion	µg/g	µg/g	µg/g	kg	µg/g	kg
Cl^-	147.0	59.9	122.6	60.8	<60.0	<213.6
Cr(VI)	--	105.0	29.4	14.6	--	--
F^-	460.0	739.0	538.1	267.0	913.6	3,252.1
OH^-	--	--	--	--	1,709.1	6,083.7
NO_3^-	2,520.0	5,750.0	3,424.4	1,699.1	21,636.4	77,016.9

Table 4-2. Analytical Summary for Waste in Tank 241-AZ-102. (5 sheets)

Analyte	1989 Sludge sample				1995 Grab samples	
	Centrifuged sludge concentration	Interstitial liquid concentration	Calculated average sludge concentration[a]	Total projected sludge inventory[b]	Liquid concentration[c]	Total projected supernatant inventory[d]
NO_2^-	15,400.0	2,820.0	11,877.6	5,893.3	25,090.9	89,313.6
PO_4^{3-}	416.0	129.0	335.6	166.5	<360.0	<1,281.5
SO_4^{2-}	6,540.0	11,900.0	8,040.8	3,989.6	15,727.3	55,982.9
Radchem	µCi/g	µCi/g	µCi/g	Ci	µCi/g	Ci
^{241}Am	101.0	1.3E-03	72.7	36,081.7	<4.3E-03	<15.3
^{125}Sb	576.0	<8.0	416.9	<24,204.7	--	--
^{14}C	1.8 E-03	1.1 E-03	1.6 E-03	0.8	--	--
^{144}Ce	ND	ND	ND	ND	--	--
^{137}Cs	880.0	747.0	842.8	342,801.2	945.5	3,370,000.0
^{154}Eu	ND	ND	ND	ND	--	--
^{129}I	ND	ND	ND	ND	--	--
^{237}Np	0.1	1.7 E-03	0.1	49.2	--	--
^{238}Pu	--	--	0.02[g]	169.6	<2.7 E-03	<9.6
$^{239/240}Pu$	--	--	42.7[g]	1,558.8	<2.7 E-03	<9.6
^{241}Pu	--	--	0.3[g]	13,794.0	--	--
^{106}Ru	4,030.0	<8.1	<2,903.9	<3,570.3	--	--
^{79}Se	<3.0 E-05	3.7 E-04	<1.2 E-04	<0.1	--	--
$^{89/90}Sr$	--	--	--	--	1.6	5,802.1
^{90}Sr	22,000.0	6.0	15,841.7	6,366,730.3[i]	--	--
^{99}Tc	0.6	0.1	0.4	220.3	--	--
Physical property	µg/g	µg/g	µg/g	kg	µg/g	kg
$\%H_2O$Grav	--	--	--	--	84.5	--
$\%H_2O$TGA	--	--	--	--	84.1	--

Table 4-2. Analytical Summary for Waste in Tank 241-AZ-102. (5 sheets)

Analyte	1989 Sludge sample				1995 Grab samples	
	Centrifuged sludge concentration	Interstitial liquid concentration	Calculated average sludge concentration[a]	Total projected sludge inventory[b]	Liquid concentration[c]	Total projected supernatant inventory[d]
Density (g/mL)	1.73	1.05	1.49	--	--	--
SpG	--	--	--	--	1.10	--
pH	--	11.8	--	--	12.9	--
Carbon	µgC/g	µgC/g	µgC/g	kg	µgC/g	kg
TOC	3,160.0	1,340.0	2,650.4	1,315.0	1,366.4	4,863.8
TIC	15,600.0	7,040.0	13,203.2	6,551.0	5,311.8	18,907.9

-- not measured, analyzed, or applicable.
ND = not detected.
TIC = total inorganic carbon.
TOC = total organic carbon.
[a]This column lists the average concentration of each analyte and is calculated as follows:

$$\text{(Sludge concentration) (Sludge Wt\%)} + \text{(Supernatant concentration) (1-Sludge Wt\%)}$$

Example calculation for aluminum:

$$(53{,}700\ \mu g/g)(0.72) + (89.1\ \mu g/g)(1-0.72) = 38{,}688.9\ \mu g/g$$

[b]Projected sludge inventory calculated using Agnew estimate for 1989 sludge level of 333 kL (88 kgal). This sludge level translates to a total sludge mass of 496,170 kg (as calculated in Section 4.2).

Total projected inventory is calculated using the following equation:

(Total Sludge Mass) x (Average Sludge Analyte Concentration)

Table 4-2. Analytical Summary for Waste in Tank 241-AZ-102. (5 sheets)

Analyte	1989 Sludge sample				1995 Grab samples	
	Centrifuged sludge concentration	Interstitial liquid concentration	Calculated average sludge concentration[a]	Total projected sludge inventory[b]	Liquid concentration[c]	Total projected supernatant inventory[d]

Example calculation for aluminum:

$$\frac{(496{,}170\ \text{kg})\ (1{,}000\ \text{g/kg}) \times [(38{,}688.9\ \mu\text{g/g})\ (1 \times 10^{-6}\ \text{g/}\mu\text{g})]}{1{,}000\ \text{g/kg}} = 19{,}196.3\ \text{kg}$$

[c]Concentration values in μg/mL (as listed in Appendix B) have been converted to μg/g using the supernatant density of 1.10 g/mL.

[d]Projected supernatant inventory calculated using Hanlon estimate for February 1995 supernatant level of 3,236 kL (855 kgal) and a supernatant density of 1.10 g/mL. This is calculated using the following equation:

[(Supernatant Concentration)(Supernatant Density)(Total Supernatant Volume)(Unit Conversions)] x (Unit Conversions)

Example calculation for aluminum:

$$[(140.9\ \mu\text{g/g})(1.10\ \text{g/mL})(3{,}236{,}000\ \text{L})(1{,}000\ \text{mL/L})$$

$$(1 \times 10^{-6}\ \text{g/}\mu\text{g})] \times (1 \times 10^{-3}\ \text{kg/g}) = 501.5\ \text{kg}.$$

[e]It should be noted that additional aluminum may have precipitated since the 1989 sludge sampling as a result of the OH^- depletion which results from absorption of atmospheric carbon dioxide. This would account for some of the 26 kL (7 kgal) increase in sludge volume since the 1989 sampling event.

[f]A significant quantity of this element is expected in the supernatant. Since the 1995 grab sampling analyses did not analyze for this element, the supernatant concentration is taken from the 1989 core sample analysis.

[g]See Table A-2 in Appendix A for isotopic distributions.

[h]The large less than value (<) for the centrifuged sludge was ignored.

[i]Independent computer modelling of the PUREX waste streams entering this tank account for only approximately 50% of this value. This suggests that the calculated average sludge concentration for this radionuclide, based on the measured core sample concentrations, may be excessively conservative.

When all of the results for an analyte were less than the instrument detection limit, the average analyte concentration is reported as not detected (ND). Similarly, the calculated projected inventory is also reported as ND. In the case of one detected concentration result and one value at less than the detection limit, the resulting mean is calculated between the detected concentration value and the minimum detection limit value. In this situation the calculated projected inventory is shown preceded by a less than sign (<).

4.2 DATA PRESENTATION

The chemical composition of the sludge layer and the liquid which exists above the sludge layer is reported in Table 4-2. The projected inventory calculations associated with the sludge in the tank were calculated using a sludge volume of 333 kL (88 kgal). This was the approximate amount of sludge present at the time of the 1989 sampling (Agnew 1995). According to Gray et al. (1993), the weight of centrifuged solids from the core composite comprise 72% of the sludge in tank 241-AZ-102 with the remainder of the core composite being interstitial liquids (28%). The density of the composite sludge was determined to be 1.49 g/mL (see Section 4.3.1 for discussion), so the volume of sludge can be converted from liters to kilograms by utilizing the following equation:

Volume of Sludge (L) x Determined Sludge Density

$$333{,}000 \text{ L} \times 1{,}000 \text{ mL/L} \times 1.49 \text{ g/mL} \times 1.0 \times 10^{-3} \text{ kg/g} = 496{,}170 \text{ kg}$$

The volume of supernatant liquid waste existing above the sludge layer in tank 241-AZ-102 was 3,236 kL (855 kgal) when the February 1995 sampling event occurred. This volume and appropriate conversion factors were utilized to calculate inventory estimates from the liquid grab sample data (Appendix B, column 7).

A brief narrative of each column in Table 4-2 follows. The first column, labeled Analyte, gives the abbreviation for the name of the analyte being calculated or measured. The second column, labeled Centrifuged Sludge Concentration, is the analyte concentration calculated in the centrifuged sludge sample using tabulated information from Appendix A and Gray et al. (1993). The third column, labeled Interstitial Liquid Concentration, is the analyte concentration measured in the interstitial liquid sample drained/centrifuged from the sludge (Appendix A and Gray et al. [1993]). The fourth column, labeled Calculated Average Sludge Concentration, is the analyte concentration calculated to be present in the tank sludge as it exists in the tank with interstitial liquids (not centrifuged). The fifth column, labeled Total Projected Sludge Inventory, is the total projected inventory of the analyte calculated to be present in the sludge layer. The sixth column, labeled Liquid Concentration, is the concentration of analyte found in the supernatant samples (Appendix B). The seventh column, labeled Total Projected Supernatant Inventory, is the amount/total projected inventory of analyte calculated to be in supernatant layer above the sludge in the tank.

Also, please note the activity levels for radioactive components (Table 4-1, column 5) have been decayed approximately six years (May 1989 through June 1995) from the value shown to determine the total projected activity level (Ci) in the sludge.

4.3 PHYSICAL MEASUREMENTS

Physical measurements were performed on both of the sets of samples which were used as a basis for this report. These sample sets were a characterization core sample obtained in the second quarter of 1989 (Gray et al. 1993), and three waste compatibility samples obtained in the first quarter of 1995 (Rollison 1995). The physical measurements of the core samples were extensive, while only DSC and TGA were performed on the waste compatibility samples. Physical characterization includes, as discussed in this section, measured quantities such as density, percent solids, percent water, viscosity, and energetics as well as cohesiveness, color, and texture. Table 4-3 lists the results of the physical tests conducted on the core samples obtained in 1989. A discussion of physical testing methodology and interpretation of results can be found in *The Tank Characterization Reference Guide* (De Lorenzo et al. 1994).

Table 4-3. Tank 241-AZ-102 Physical Measurements.*

Physical property	Composite solids	40% Slurry	10% Slurry	Core composite	Washed solids
Density (g/mL)	1.49	1.24	1.10	1.36	1.11
Vol% settled solids	100	76	NM	NM	67
Vol% centrifuged solids	64	26	NM	46	22
Wt% centrifuged solids	72	37	NM	58	31
Centrifuged solids density	1.69	1.78	NM	1.73	1.52
Centrifuged supernatant density	1.13	1.10	NM	1.05	0.99
Wt% dissolved solids	3.4	12	NM	NM	NM
Wt% total solids	49	32	15	37	14
Wt% oxides	37	NM	NM	28	11

*Gray, W. J., M. E. Peterson, R. D. Scheele, J. M. Tingey, 1993, *Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 102-AZ*, Pacific Northwest Laboratories, Richland, Washington.

NM = not measured.

Prior to centrifugation, composition, and dilution of the 1989 core samples, layer samples 1 cm in length were obtained from the two core segments at the following distances from the rotary valve located near the drill bit end (bottom) of the sampler: L1 at 37 cm, L2 at 17 cm, L4 at 31 cm, and L3 at 8 cm. L1 and L2 are from Segment 1, and L3 and L4 are from Segment 2. Note that the numerical order of sampling was reversed between the two samplers. L1 came from near the top of the settled solids region of the waste, L3 came from near the bottom. L2 and L4 came from locations between those of L1 and L3. Table 4-4 lists the physical properties of the layer samples. An informal evaluation of the physical data shows little or no difference between the layers.

Table 4-4. Physical Properties of Layer Samples.[a]

Physical measurement	Sample identification			
	L1	L2	L4	L3
	Segment #1		Segment #2	
Location cm[b]	37	17	31	8
Wt% total solids	47.3[c]	NM	42.8[c] 48.5[d]	48.8[d]
Mean particle size (population) μm	1.3	1.1	1.0	1.3
Mean particle size (volume) μm	9.9	4.8	4.5	14.1

[a]Gray, W. J., M. E. Peterson, R. D. Scheele, J. M. Tingey, 1993, *Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 102-AZ*, Pacific Northwest Laboratories, Richland, Washington.

[b]Distance in cm from the sampler rotary valve.

[c]Dried for 21 hours at 105 °C.

[d]Dried under a heat lamp for approximately one hour prior to fusion preparation.

NM = not measured.

4.3.1 Density, Wt% Total Solids, Vol% Solids, and Wt% Oxides

Density measurements of the 1989 core samples were performed on a composite of the two core segments (the core segment composite), a 40% and a 10% slurry solution (formed by combining centrifuged solids and centrifuged supernatant in two proportions), a core composite (material remaining after other physical tests had been performed), and on the washed solids (solid slurry remaining after washing of the core composite). The values ranged from 1.49 g/mL for the core segment composite to 1.10 g/mL for the 10% slurry solution (Table 4-3). The 1.49 g/mL density of the core segment composite (or composite solids) was determined to be the least disturbed or manipulated sample and best representative of the sludge as it exists in the tank. The density of the centrifuged solids

from the same groups of samples ranged from 1.73 g/mL for the core composite to 1.52 g/mL for the washed solids, while the density of the centrifuged supernatant ranged from 0.99 g/mL for the washed solids to 1.13 g/mL for the composite solids (Table 4-3). In the 1995 supernatant samples the SpG measurements exhibited an average of 1.10.

Wt% total solids measurements were performed on segment layers L1, L3, and L4 and yielded a range of 42.8% to 48.8%. In addition, wt% total solids measurements were performed on the core segment composite, both slurry solutions, the core composite, and the washed solids. The values ranged from 14 wt% for the washed solids to 49 wt% for the core composite.

Other measurements were performed as noted in Table 4-3. Trends in the data reflect the sample handling steps; that is, most density and percent solids values decreased upon dilution and washing steps since the addition of water to the samples caused the dissolution of solids.

4.3.2 Settling Behavior

Settling rates were conducted in preweighed, volume-graduated centrifuge tubes. The solids level in the tubes were recorded periodically. Settling behavior data should be used with caution. The settling rate is expected to be friction-dependent, and is most likely slower in the laboratory than in the waste tanks themselves.

No settling behavior data were measured on the as-received waste samples. Settling behavior was, however, measured on the 40% solid slurry, the first wash slurry. The test was run in duplicate. Both samples exhibited similar behavior. After an initial drop in volume of about 40% after 6 hours, the waste settled in nearly a linear fashion for another 24 hours, to a value of about 25% of the original volume. Very little volume was lost between 30 hours and the end of the test at 51 hours.

4.3.3 Particle Size

Particle size analysis was performed using a light scattering particle size analyzer on the composite solids, the washed solids, and the layer samples, as noted above in Section 4.3. The particle size analyzer measures the time required for a rapidly moving laser beam to traverse a glycerin solution of suspended particles. The particle size data were based on both the population and volume of the particles in the sample. When based on the population the particles were within the 1.0 - 1.3 μm range, and when based on volume the particles were within the 3.4 - 4.9 μm range, for both the composite solids and the washed solids. Although the differences between the population and volume results for the composite and washed solids are not as great as those for the layer samples, the data suggest the presence of a few large particles which skew the volume data and leave the population data relatively unchanged.

4.3.4 Rheology and Shear Strength Measurements

Shear strength was measured on samples obtained from both segments of the core. The shear strength was measured at cell temperature (31 °C) using a shear vane and the Haake RV 100 viscometer. Shear vane height was 1.58 cm, and the diameter was 0.80 cm. The sample from segment 1 was taken 35.6 cm from the top of the segment, which corresponds to approximately 68.6 cm from the bottom of the tank. The sample flowed upon placement in a jar. After transfer to a smaller jar, the shear strength was found to be about 600 dynes/cm^2. The small value was thought to be observed because the waste was disturbed prior to being measured. Additional shear strength measurements were taken on the bulk of the sludge from segment 1, which had been stored undisturbed in the laboratory for about seven months.

The shear strength for the bulk sample was measured in duplicate. The results were 15,400 dynes/cm^2 and 13,140 dynes/cm^2. Segment 2 was sampled at a point equivalent to about 10.2 cm from the bottom of the tank. The sample size was insufficient for a duplicate analysis. The result was 26,500 dynes/cm^2. The shear strength for segment 1 was about half that of segment 2. This is consistent with the observation after extrusion of the segments that segment 2 held its shape, while segment 1 flowed.

The rheological properties of the 40% slurry solution, the 10% slurry solution, and the centrifuged supernatant were measured at 65 °C and at 95 °C. The viscosity of the centrifuged supernatant, measured at 33 °C, was below the accurate viscosity range of the instrument.

The 40% slurry solution exhibited yield-pseudoplastic behavior, i.e., the sample had a significant yield stress at both sample temperatures, while the 10% slurry solution exhibited pseudoplastic behavior, or no yield stress. The data from the rheograms were fit to a non-linear yield power law model:

$$\sigma = \alpha + \beta\gamma^n$$

where

σ = shear stress in Pa (lb$_f$/ft^2)
γ = shear rate
α = yield stress (not a fit parameter)
β and n are fit parameters

Results from the power law model are presented in Table 4-5.

Table 4-5. Results From Fit to Rheological Models.

Slurry	Temperature (°C)	α	β	n	R^2
40 wt% solids	65	2.02	0.0284	0.7392	0.97
		2.12	0.0081	0.9554	0.98
	95	2.53	0.0073	0.9155	0.94
		2.48	0.0002	1.5511	0.93
10 wt% solids	65	0	0.0015	0.9416	0.36
		0	0.0019	0.9306	0.35
	95	0	0.00004	1.5051	0.42
		0	0.0017	0.8765	0.30
Washed solids	33	0.53	0.0049	0.9144	0.91
		0.49	0.0141	0.7774	0.90

Gray, W. J., M. E. Peterson, R. D. Scheele, J. M. Tingey, 1993, *Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 102-AZ*, Pacific Northwest Laboratories, Richland, Washington.

The rheological parameters along with the density of the dilutions were input into the Hank's computer model to obtain the critical Reynold's Number and the critical velocity for transferring the slurries in 3.0 in. and 2.0 in. diameter pipes. The critical velocities and Reynold's Numbers calculated for these samples are minimum values that must be maintained to assure that the solids remain suspended during transport so that they do not settle and clog the pipes. The results are summarized in Table 4-6.

4.3.5 Penetrometry

The penetration resistance of a sample taken from the bottom end of the second segment was measured using the CT-421A penetrometer to a depth of 2.54 cm. The test was conducted to determine whether the solids exhibited dilatant or cohesive properties.
A dilatant sludge would be eroded by the mixer pumps and a cohesive sludge would be broken down into large pieces of sludge by the mixer pump. A low penetration resistance (0 - 10 psi) indicates that the sludge is cohesive and a high penetration resistance (100 psi or greater) shows that the sludge is dilatant. The penetration resistance of the sample was 0.176 kg/cm^2 (2.5 psi), indicating that the sludge is cohesive.

Table 4-6. Critical Reynold's Numbers and Velocities.

Slurry	Temperature (°C)	Nominal pipe diameter (in.)	Critical velocity (m/s)	Reynold's number	Critical flow (L/min)
40 wt% Solids	65	2.0	2.77	6,500	29
			2.69	8,100	28
		3.0	2.52	7,700	58
			2.40	10,500	55
	95	2.0	2.60	10,400	27
			2.35	20,300	25
		3.0	2.33	13,200	54
			2.01	34,900	46
10 wt% Solids	65	2.0	0.16	2,100	2
			0.02	2,200	2
		3.0	0.12	2,200	3
			0.14	2,100	3
	95	2.0	0.01	6,000	0.1
			0.17	2,200	2
		3.0	0.12	2,200	3
			0.14	2,100	3
Washed solids	33	2.0	1.46	7,400	15
			1.65	5,200	17
		3.0	1.31	9,400	30
			1.49	6,200	34

Gray, W. J., M. E. Peterson, R. D. Scheele, J. M. Tingey, 1993, *Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 102-AZ*, Pacific Northwest Laboratories, Richland, Washington.

4.3.6 Thermodynamic Analyses

DSC analyses were performed on the 1995 waste compatibility grab samples. No exothermic reactions were noted (Rollison 1995). TGA was utilized to determine the percentage of water in the 1995 grab samples, and the results are reported in Appendix B. No DSC or TGA were conducted on the 1989 core sample.

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

This section evaluates sampling and analysis factors that may impact interpretation of the data. These factors are used to assess the overall quality and consistency of the data and to identify any limitations in the use of the data. It should also be noted that data generated prior to 1989 may not be considered valid for some applications under the constraints of the Hanford Federal Facility Agreement and Consent Order (Ecology et al. 1994).

5.1.1 Field Observations

The sampling, extrusion, and subsampling of the 1989 waste samples were uneventful. Sample recoveries were good, and few problems were noted in the data report (Gray et al. 1993). The analysis of a composite of both segments gives a good average value for the waste in that region of the tank, and the analysis of the layer samples gives a good idea of the vertical distribution of analytes in that region of the tank. Some problems were noted, however, as discussed below.

Insufficient sample made it impossible to perform wt% solids measurements on any of the layer samples except L4. The situation was remedied by weighing the samples from L1, L2, and L3, which were set aside for chemical and radiochemical analyses and then dried under a heat lamp for one hour prior to fusion preparation (in comparison with the 24 hour drying period required by the wt% solids analysis). The results were consistent, and therefore are probably representative of the condition of the waste.

When attempting shear strength measurements for segment 1, the sample flowed upon being placed in the jar. The sample was placed in a smaller jar and the shear strength was successfully measured. The result, however, was quite low (600 dynes/cm^2). It was thought that the disturbance of the sample when it was placed in the first jar resulted in the low value. A subsequent measurement of the shear strength was made of the bulk of the sludge from segment 1, which had been undisturbed for about seven months. The original and duplicate results of the shear strength measurement of the bulk samples (15,400 dynes/cm^2 and 13,140 dynes/cm^2) were a factor of approximately 20 to 25 higher than the first measurement. A comparison of these results with those from the shear strength of segment 2 (26,500 dynes/cm^2) corroborates the hot cell chemist's observation that the waste from segment 1 flowed upon extrusion while the waste from segment 2 held its shape.

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 a strong 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 analytical method error, matrix interferences, sample contamination, and poor instrument calibration. Error estimates are determined from the analysis of standards, spike recoveries, blank contamination, and sample duplicate variation.

Quality control information was not provided in the sludge core sample data package. Therefore, the accuracy and precision of those analytical results are unknown. The data package for the 1995 grab samples provided some quality control information, which is discussed in the following paragraphs (Rollison 1995).

Standards are used to estimate the accuracy of the analytical method, and are evaluated prior to and concurrent with sample analysis. Standards contain the analytes of interest at known concentrations. Standard solutions may or may not be independent of the standard used for calibration. The criterion for standard recovery is 100 $\pm$ 10%. If a standard is above or below the criterion, then the analytical results may be biased high or low, respectively. For the three grab samples, two standards were run for DSC, $^{89/90}$Sr, and $^{239/240}$Pu, no standards run for the ICP metals (aluminum, iron, and sodium), and one standard was run for the remainder of the analytes. The only standard that failed the criteria was for ^{241}Am (87.3% recovery), which indicated the data for this analyte may be biased slightly low.

Matrix spikes are used to estimate the bias of the analytical method due to matrix interferences. Spike samples are prepared by splitting a sample into two aliquots and adding a known amount of a particular analyte to one aliquot to calculate a percent recovery. The quality control criterion for matrix spikes is 100 $\pm$ 20% recovery. As with standards, if a spike is above or below the criterion, then the analytical results may be biased high or low, respectively. Spikes were only conducted on TOC (3 spikes), all anions except hydroxide (1 spike), and aluminum and iron (2 spikes). The only spike to fall outside the criteria was one of the two conducted for aluminum (78.3). Thus the analytical results for aluminum may be biased slightly low. The possible influence of matrix interferences on the data results of the remaining analytes cannot be assessed or estimated since no matrix spikes were conducted on them.

Method blanks document the contamination resulting from the analytical process, and are prepared by filling sample containers with deionized, distilled water. They are carried through the complete sample preparation and analytical procedure, and all reagents used in the sample processing are added in the same volumes. One blank was conducted on all of the anions, $^{89/90}$Sr, and ^{241}Am, and two blanks were conducted on ^{238}Pu and $^{239/240}$Pu. The

only one of these analytes whose blank was detected was hydroxide, whose value was four orders of magnitude below the analytical result. This indicates that contamination was not a problem for any of the analytes on which blanks were conducted.

Random analytical error can be estimated from the analytical results (variation between duplicate samples, and sample heterogeneity), which provides an indication of laboratory precision between the homogenized samples. To estimate this error, a relative percent difference (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-AZ-102 set the duplicate precision acceptance RPD criterion 20% (Schreiber 1995). The analytes in which all values were detected each had three duplicate pairs. The only violations of the criterion were for one of three duplicate pairs each for TIC (20.6%), sulfate (24.2%), nitrate (21.9%), nitrite (23.1%), and fluoride (25.4%). These violations are relatively slight, however, and should not have a large impact on the data quality.

5.1.3 Data Consistency Checks

The ability to assess the overall consistency of the data from the various tank 241-AZ-102 sampling events is limited because of the nature of the data and the limited duplication of analysis. A comparison was possible between the analytical results of the layer samples and core composite of the 1989 core sample. In addition, a mass and charge balance was performed for the 1989 core sample. Other checks on individual analytes were generally unable to be performed. The comparison of analytical results from different sampling events described in Section 5.2 also gives an indication of the data consistency for this tank.

5.1.3.1 Comparison of 1989 Core Sample Data Before and After Compositing. An internal data check was performed by comparing the analytical results obtained before and after compositing. Prior to formation of the core composite, two samples were removed at different points from each segment for layer tests. Analyses were run on these four layer samples. The composite was formed by combining solids and liquids remaining after various physical tests had been performed and the 10% and 40% slurry solutions had been formed. Table 5-1 compares the results from both analyses. Column 2 lists the mean for each analyte from the layer tests. Column 3 contains the core composite mean for each analyte. Column 4 tabulates the RPD, defined as the absolute value of the difference of the two results, divided by the mean and multiplied by 100) between the two results. An analyte was not included if either of the two means exhibited a value less than the detection limit.

Table 5-1. Comparison of Core 1 Data Before and After Homogenization. (2 sheets)

Analyte	Mean of layers 1-4	Core composite mean	RPD
Metal	(μg/g)	(μg/g)	(%)
Silver	315	363	14
Aluminum	29,900	28,600	4.4
Arsenic	518	319	48
Barium	364	296	21
Beryllium	9.23	7.26	24
Calcium	2,580	1,610	46
Cadmium	7,870	7,980	1.4
Cerium	939	423	76
Chromium	1,740	1,360	25
Copper	224	188	17
Iron	73,500	70,200	4.6
Potassium	2,370	1,590	39
Lanthanum	2,710	2,400	12
Magnesium	590	508	15
Manganese	1,400	1,540	9.5
Molybdenum	60.0	37.3	47
Sodium	67,100	47,600	34
Neodymium	1,810	1,560	15
Nickel	5,170	4,740	8.7
Phosphorus	3,650	544	148
Lead	736	587	23
Rhenium	88.5	39.1	77
Ruthenium	268	98.8	92
Silicon	3,100	2,580	18
Strontium	202	175	14
Tellurium	373	185	67
Titanium	58.7	41.5	34
Vanadium	48.4	18.1	91
Zinc	124	75.2	49
Zirconium	12,400	9,660	25

Table 5-1. Comparison of Core 1 Data Before and After Homogenization. (2 sheets)

Analyte	Mean of layers 1-4	Core composite mean	RPD
Ion	μg/g	μg/g	%
F^-	878	526	50
Cl^-	65.6	101	40
NO_2^-	28,600	9,310	102
NO_3^-	5,310	3,530	40
PO_4^{-3}	911	272	108
SO_4^{-2}	12,100	8,020	41
Carbon	μg/g	μg/g	RPD
TIC	8,330	11,000	28
TOC	8,330	2,200	116
Radionuclide	μCi/g	μCi/g	RPD
^{106}Ru	2,420	2,160	11
^{125}Sb	318	310	2.5
^{137}Cs	939	742	23

TIC = total inorganic carbon.

On first inspection of the table, 10 of the 41 analytes have RPDs greater than 50%. However, if the analytes which had results below or near the detection limit (arsenic, cerium, potassium, phosphorus, rhenium, ruthenium, tellurium, and vanadium) are excluded, only 3 analytes remain with RPDs over 50%. These analytes are NO_2^-, PO_4^{-3}, and TOC. The bottom segment, or layers 3 and 4, contained over three times as much phosphate as the upper segment, possibly skewing the layer test results. For TOC, the results were inconsistent and incomplete and should be used with caution (Gray et al. 1993). Unsatisfactory results were obtained for two out of the four layer samples, which caused an invalid comparison because the waste from layers 2 and 3 were included in the composite, but results from layers 2 and 3 were not included in the layer mean. Taking into account these factors, there was excellent correlation between the layer samples and the core composite.

5.1.3.2 Mass and Charge Balance. The principal objective in performing a mass and charge balance is to determine if the measurements are self-consistent. In calculating the balances, only sludge phase analytes listed in Table 4-2 which were detected at a concentration of 2,000 μg/g or greater were considered.

With the exception of sodium, all cations listed in Table 5-2 were assumed to be in their most common hydroxide or oxide forms, and the concentrations of the assumed species were calculated stoichiometrically. There may be some argument whether or not certain species are hydroxides or oxides, but the difference in molecular weight has a minimal effect on the overall mass balance. Although smaller concentrations of other forms of the species are probably present in the waste, they are not included in order to keep the mass-charge balance calculations simple and consistent.

Table 5-2. Cation Mass and Charge Data.

Analyte	Concentration (μg/g)	Assumed species	Concentration of assumed species (μg/g)	Charge (μmol/g)
Aluminum	38,689	$Al(OH)_3$	111,768	0
Cadmium	10,801	CdO	12,344	0
Calcium	2,176	CaO	3,046	0
Iron	94,323	FeO(OH)	149,906	0
Lanthanum	3,240	$La(OH)_3$	4,429	0
Neodymium	2,097	Nd_2O_3	3,012	0
Nickel	6,380	$Ni(OH)_2$	10,906	0
Sodium	54,340	Na^+	54,340	2,363
Uranium	9,464	U_2O_3	10,418	0
Zirconium	13,033	ZrO_2	17,616	0
Totals			377,785	2,363

Since precipitates are neutral species, all positive charge was attributed to the sodium cations. The anionic analytes listed in Table 5-3 were assumed to be present as sodium salts and were expected to balance the positive charge. The acetate and carbonate data were derived from the total organic and TIC analyses, respectively. The concentrations of the assumed species in Table 5-2, of the anionic species in Table 5-3, and the percent water were ultimately used to calculate the mass balance, shown in Table 5-4.

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

Table 5-3. Anion Mass and Charge Data.

Analyte	Concentration (μg/g)	Charge (μmol/g)
Acetate (TOC)	6,515	-110
Carbonate (TIC)	66,015	-2,202
Nitrate	3,424	-55
Nitrite	11,878	-258
Permanganate	4,487	-38
Silicate	9,122	-240
Sulfate	8,041	-167
Totals	109,482	-3,070

Table 5-4. Mass Balance Concentration Totals.

Total from Table 5-2	377,785 μg/g
Total from Table 5-3	109,482 μg/g
Water	510,000 μg/g
Grand total	997,267 μg/g

$$\text{Mass balance} = \%\ \text{Water} + 0.0001 \times \{\text{Total Analyte Concentration}\}$$

$$= \%\ \text{Water} + 0.0001 \times \{Al(OH)_3 + CdO + CaO + FeO(OH) + La(OH)_3 + Nd_2O_3 + Ni(OH)_2 + SiO_3^{2-} + Na^+ + U_2O_3 + ZrO_2 + C_2H_3O_2^- + CO_3^{2-} + NO_3^- + NO_2^- + MnO_4 + SO_4^{2-}\}$$

The total analyte concentrations calculated from the above equation was 398,426 μg/g. The weight percent of water as discussed earlier is calculated to be 51% (Table 4-3). The mass balance resulting from adding the percent water to the total analyte concentration is 0.997.

The charge balance is the ratio of cations (microequivalents) to total anions (microequivalents) with respect to the species listed below, which were assumed to be water soluble.

Total Cations (microequivalents) = $Na^+/23.0$

The total cation charge, 2,363 μg/g, is calculated in Table 5-2.

Total anions (microequivalents) = $C_2H_3O_2^-/59.0 + CO_3^{2-}/30.0 + NO_3^-/62.0 + NO_2^-/46.0 + MnO_4^-/119.0 + SiO_3^{2-}/38.0 + SO_4^{2-}/48.0$

The total anion charge, |-3,070 μg/g|, is calculated in Table 5-3.

The ratio of microequivalents of total cations to microequivalents of total anions was 0.770; a perfect charge balance would yield a ratio equivalent to 1.00.

The charge and mass balance ratios (0.770 and 0.997, respectively) demonstrate fair agreement among analyses when considering the uncertainty in the assumptions and numerous measurements used to arrive at the values. However, the mass balance shows much closer agreement to unity, being off by less than 1%. These results do not indicate that large data inconsistencies or errors are present or that major components may not have been analyzed and evaluated.

5.2 DATA COMPARISON

As discussed in Section 3, this report is based on four sampling events, two of which were used to project the estimated inventories of tank 241-AZ-102. Of additional interest is a comparison of different analyses of waste constituents. The supernatant samples from

August 1987, October 1987, and February 1995 are compared in Table 5-5, and two digestion methods for the solid samples from August 1987 are compared in Table 5-6.

The comparison of the supernatant results gives an indication of the precision of the analyses, assuming that the supernatant was relatively unchanged between 1987 and 1995. It is a good assumption, given the transfer history of the tank in that period (see Section 2.0 for a detailed discussion). Using an arbitrary criterion of 50% increase or decrease, only three analytes (nitrate, fluoride, and hydroxide) showed good agreement among all three data sets. Total beta and total organic carbon showed good agreement between two data sets.

The solid samples from August 1987 are compared in Table 5-6, in order to give personnel involved in retrieval an idea of the solubility of the analytes. The concentrations of analytes which appear in both data reports at levels above the minimum detectable are listed. Results from the August 1987 sampling event are used because that sample set has the larger number of common and detected analytes. The data comes from samples which were centrifuged, and the supernatant discarded. The remaining solids were water washed, then recentrifuged and the solids dissolved in acid. The solid sample results for the acid digestion are assumed to represent all of the analytes present in the sample, i.e., it is assumed that the acid digestion is complete. The percent solubility is a ratio of the water digestion results and the total water and acid results.

5.3 TANK WASTE INVENTORY PROFILE

Several restraints were encountered in the attempt to describe the horizontal and vertical disposition of the waste in tank 241-AZ-102. The risers from which the two samples from 1987 were taken were not identified; therefore, it is not known whether they were the same risers from which the 1989 sludge sample or the 1995 supernatant sample were taken. This precludes any effort to compare or incorporate the 1987 information with the 1989 and 1995 information for horizontal or vertical analysis.

The three 1995 supernatant samples were all taken from the same riser at evenly spaced depths: the first near the top of the supernatant layer, the second near the middle, and the third near the bottom of the supernatant layer. Considering that for a given analyte none of the samples was more than 20% different than another, and that the criterion for duplicate precision is also 20%, any differences between the supernatant samples are not large enough to be considered different from another. Thus, based on these results, the contents of the supernatant layer appear to be homogeneous.

The two sludge segments from the 1989 sample were divided into two sub-segments, giving a total of four subsegments. Table 5-7 lists the sub-segments in columns 2-5, with tank depth increasing from left to right. Samples L1 and L2 were obtained from the upper segment and samples L3 and L4 from the second segment. Note that the numerical order of sampling was reversed between the two samplers. Sample L1 came from the upper sludge layer, sample L3 came from near the bottom, and samples L2 and L4 came from near the

Table 5-5. Comparison of Supernatant Constituents.

Metal/Ion	August 1987	October 1987	February 1995
	μg/mL		
Aluminum	2,620	2,300	155
Sodium	37,000	36,100	5,320
Nitrate	21,700	21,700	23,800
Nitrite	12,400	12,400	27,600
Fluoride	703	722	1,005
Sulfate	10,800	10,600	17,300
Chloride	248	2,160	--
Hydroxide	3,400	3,060	1,880
TOC	1,520	720	1,503
TIC	12,000	16,200	5,843
Radionuclide	μCi/mL		
$^{89/90}Sr$	1.46	0.00165	1.79
^{137}Cs	936	735	1,040
Total Beta	1,370	1,360	--

-- Not detected/measured.

Table 5-6. Comparison of Sludge Constituents.

Metal/Ion	Water Digest	Acid Digest	% Solubility
	μg/mL		
Aluminum	1,120	2,970	27.4
Sodium	27,200	6,110	81.7
Uranium	157	5,120	2.98
Fluoride	589	21,500	2.67
Phosphate	190	3,520	5.12
Radionuclide	μCi/mL		
^{137}Cs	550	290	65.5
$^{89/90}Sr$	0.843	9,300	<0.01

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Table 5-7. Composition of 1989 Core from Tank 241-AZ-102 Before Compositing. (2 sheets)

Analyte	Sample identification			
	L1	L2	L4	L3
	Segment 1		Segment 2	
Metals	µg/g	µg/g	µg/g	µg/g
Ag	237	237	291	496
Al	15,200	14,900	45,900	43,500
As	<554	397	<554	<569
B	<454	<313	<454	<476
Ba	426	330	357	343
Be	12.6	9.91	10.8	3.60
Ca	2,630	2,210	3,460	2,020
Cd	16,400	11,800	3,120	146
Ce	<1,060	546	<1,060	<1,090
Co	<1,360	<943	<1,360	<1,410
Cr	1,540	1,100	1,460	2,870
Cu	280	229	241	146
Dy	<65	<33	<65	<65
Fe	1.11 E+05	78,200	61,400	43,400
K	<2,420	2,220	<2,390	<2,460
La	4,670	3,320	1,900	945
Li	<69.4	<18.7	<69.4	<76.4
Mg	357	306	445	1,250
Mn	1,750	1,240	1,320	1,270
Mo	57.6	48.0	76.8	57.6
Na	50,800	43,200	60,300	1.14 E+05
Nd	2,930	2,030	1,310	966
Ni	8,810	6,220	4,140	1,490
P	<4,340	1,280	<4,340	4,650
Pb	746	663	539	995
Re	<93.1	55.9	<93.1	<112
Rh	<628	<268	<628	<648
Ru	<263	273	<263	<273
Sb	<1,830	<2,190	<1,830	<1,830

Table 5-7. Composition of 1989 Core from Tank 241-AZ-102 Before Compositing. (2 sheets)

Analyte	Sample identification			
	L1	L2	L4	L3
	Segment 1		Segment 2	
Se	<592	<640	<592	<616
Si	2,490	3,760	2,110	4,020
Sr	263	184	263	96.4
Te	<396	293	<396	<408
Th	<766	<302	<743	<789
Ti	<52.7	47.9	62.3	71.9
Tl	<9,810	<5,520	<9,810	<10,020
U	10,000	20,000	1,200	56,000
V	<56.0	25.5	<56.0	<56.0
Zn	131	124	124	118
Zr	20,900	13,200	14,500	930
Ion	μg/g	μg/g	μg/g	μg/g
F^-	950	809	904	847
Cl^-	81.5	70.9	49.6	60.3
NO_2^-	34,200	27,600	33,400	19,200
NO_3^-	2,980	7,130	3,190	7,940
PO_4^{3-}	475	<19	1,460	1,690
SO_4^{2-}	13,100	13,100	13,100	9,100
Carbon	μg C/g	μg C/g	μg C/g	μg C/g
TIC	4,600	16,400	4,000	--
TOC	7,100	--	9,550	--
Radionuclide	μCi/g	μCi/g	μCi/g	μCi/g
^{60}Co	19.0	18.8	16.9	1.0
^{106}Ru	4,770	4,670	239	16.0
^{125}Sb	515	501	226	31.0
^{134}Cs	23.5	31.2	22.6	16.5
^{137}Cs	950	1,130	926	749
^{144}Ce	21,900	21,400	6,360	75.0
^{154}Eu	97.5	68.9	65.4	18.5
^{155}Eu	240	320	186	27.0

middle. Vertical variability within the sludge layer was then examined by looking for trends in the data as a function of tank depth. Of the analytes with concentrations over 10,000 $\mu g/g$, aluminum, sodium, and uranium generally showed an increasing concentration as a function of depth, whereas the opposite was generally true for iron, zirconium, and nitrite. Most of the radionuclides showed a decreasing concentration as a function of depth. Given these mixed results, it is difficult to make any conclusions regarding the disposition of waste within the sludge layer.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER HISTORY INFORMATION

A comparison of the transfer history of tank 241-AZ-102 and analytical data shows some correlation in terms of overall characteristics. For example, ^{241}Am, which was a constituent of NCAW, appears in a relatively insoluble form in the tank sludge. Tank layering and waste chronology, however, are difficult to establish by evaluating process history and analytical results. Most of the waste which entered the tank was composed of three types: B Plant aging waste and complexant concentrate which initially filled the tank and were subsequently removed; and NCAW, which makes up the present day tank contents. Because of the difficulty in sluicing waste below 5,000 gal, it is possible that a minute heel of B Plant waste and complexant concentrate still exists in the tank. The layer samples from the 1989 core may or may not have encountered the heel. The layer results for TOC, an indicator of complexant concentrate waste, were inconclusive in showing evidence of the waste, as results were missing for the bottom-most layer where this waste would be found. This was also the case for TIC. The other samples were not designed to describe heterogeneity.

The ^{241}Am which appears in relatively high concentrations in the 1987 and 1989 sludge samples is a component of the waste stream from PUREX in which americium was separated from other TRUs in the solvent extraction process. Strontium, a component of aging waste which forms relatively insoluble compounds, is present in high concentrations in the sludge sampled in 1987 and 1989. Cesium, a highly soluble component of aging waste, is present in the 1987 and 1989 sludge samples and in the 1995 supernatant samples.

Large quantities of iron were used in the form of ferrous sulfamate in the PUREX second solvent extraction step which reduced the valence of plutonium to the +3 state. The iron, which is assumed to exist in the hydroxide form, shows up in all of the data used in this report in the sludge analyses in relatively high concentrations and does not appear in the supernatant analyses above detectable levels.

Zirconium is a primary ingredient in the zircalloy cladding for the N-reactor fuel. Although NCAW does not come from decladding operations, enough zirconium carried over (from the decladding operation) in the NCAW waste stream for it to appear in waste samples.

Cadmium was used as a neutron poison to reduce the chance of inadvertent criticality during process operations, and appears as a major constituent in the solid portion of the waste (Schofield 1991).

Chromium and nickel appear in the NCAW waste stream as a consequence of normal corrosion of the process piping and components of PUREX, and both are major constituents of the waste in tank 241-AZ-102 (Schofield 1991).

5.5 EVALUATION OF PROGRAM REQUIREMENTS

The waste compatibility Data Quality Objectives (DQO) (Carothers 1994) outlined the requirements that must be applied to the 1995 grab sample data in order to assess the compatibility of tank 241-AZ-102 waste with that in other tanks. This compatibility assessment is presented in this section. The TCP for the 1995 grab sampling event (Schreiber 1995) specified that the obtained samples were not to be safety screened due to the fact that only one riser was sampled instead of the two required by the safety screening DQO (Babad and Redus 1994). Although DQOs were not in existence when the 1989 core sample was taken, the requirements of the safety screening DQO have been compared with the analytical results.

5.5.1 Safety Evaluation

The data criteria identified in the safety screening DQO (Babad and Redus 1994) is used to assess the safety aspect of the waste in tank 241-AZ-102. Although the supernatant wasn't specifically sampled for safety screening, data needed for a safety screening evaluation is coincidentally provided through the waste compatibility DQO. The safety screening DQO identifies several primary and secondary factors for consideration.

The waste fuel energy value is normally determined using DSC analysis of the waste material. DSC analyses were performed on the 1995 grab samples; no exotherms were found. No DSC analyses have been performed on the sludge in tank 241-AZ-102. Cyanide and TOC are other indicators of the fuel content of the waste. The TOC concentration of the sludge (~0.22% wet weight) is well below the 5% TOC (dry weight) criteria established by the organic safety program (Babad, Blacker, and Redus 1994). Even though there are no cyanide analyses for the sludge, there is no historical basis to expect cyanide. Therefore, it appears unlikely that excessive fuel is present.

Large amounts of moisture reduce the potential for propagating exothermic reactions in the wastes. Because the waste in tank 241-AZ-102 is 90% liquid by volume, the moisture content of the sludge is expected to be high. The waste is approximately 90% supernatant, and the 1989 centrifuged sludge moisture content was found to be 63% (by weight), which

more than satisfies the 17 wt% water requirement of the safety screening DQO (Babad and Redus 1994). This value more than satisfies the 17% criteria established by the safety screening DQO.

Another factor in assessing the safety of the tank waste is the heat generation and temperature of the wastes. Heat is generated in the tanks primarily from radioactive decay. The major contributors for tank 241-AZ-102 are ^{90}Sr, ^{106}Ru, and ^{137}Cs. The estimated heat generated from the isotopes in the tank is <58,441 W (<199,457 Btu/hr) as shown in Table 5-8. This heat load is high when compared to other double-shell tanks, but is expected because of the aging waste. The maximum heat limit for tank 241-AZ-102 is 4,000,000 Btu/hr (Bergmann 1989), so the heat load is only about 5% of this maximum limit. Temperature data for the previous year (December 1993 through December 1994) is displayed graphically in Figure 2-6. In that time the temperature has ranged from 78 °C (172 °F) to 83 °C (181 °F), excluding one suspect spike.

Table 5-8. Tank 241-AZ-102 Projected Heat Load.

Radionuclide	Curies* (Ci)	Watts
^{241}Am	<36,097	<1,206
^{137}Cs	3,712,801	14,554
$^{239/240}Pu$	<1,568	<49
^{106}Ru	<3,570	<0.2
$^{89/90}Sr$ (supernatant)	5,802	39
^{90}Sr (sludge)	6,366,730	42,593
Total watts		<58,441

*Analyte values from the liquid portion of the tank were added to the sludge portion. Therefore, the total tank inventory is based on 1987 sludge and 1995 liquid grab sample results.

The potential for criticality is assessed from either total alpha or plutonium analysis. Criticality specifications for double-shell storage tanks are defined in Vail (1994). The safety screening criteria is 1 g/L. This is equivalent to 41.3 μCi of $^{239/240}Pu$/g in the waste, using the sludge density of 1.49 g/mL. The 1989 core data showed that the sludge contained 43.0 μg/g of plutonium (Table 4-2), which translates to 3.14 μCi/g of $^{239/240}Pu$ when using the plutonium isotope weight percentage breakdown given in Gray et al. (1993) and tabulated in Table A-2. The concentration of $^{239/240}Pu$ in the supernatant is $<2.7 \times 10^{-3}$ μg/g, so the total waste inventory of $^{239/240}Pu$ is well below the safety screening limit. The criticality specifications also require the pH of the waste to be greater than 8.0 when the plutonium inventory exceeds 10 kg and the depth of the supernatant liquid exceeds 30 cm. The pH of

the supernatant is 12.9, the sludge pH is 11.8, and the supernatant depth exceeds 30 cm, which satisfies the criticality prevention specification since the plutonium inventory exceeds 10 kg in the tank.

The flammability of the gas in the headspace of a tank is another safety screening consideration. There is no information available concerning sampling and analysis of the vapor space composition/flammability of tank 241-AZ-102. It should be noted that because of the high radioisotope concentrations, the production of radiolytically generated hydrogen is expected to be significant. However, the build of hydrogen in the tank headspace is precluded by the active ventilation system.

The analysis of tank 241-AZ-102 does not fully meet the safety screening requirements. However, historical and analytical information does not show that the waste composition exceeds the safety criteria for water content, heat or criticality. No exotherms were found in the supernatant; even though the fuel content of the sludge has not been measured by DSC, the TOC, cyanide and historical uses of the tank indicate that excessive fuel is not present.

5.5.2 Operational Evaluations

The 1995 supernatant sampling and analysis were performed to evaluate compatibility of tank 241-AZ-102 waste with that in other tanks. Sampling and analysis requirements for assessing waste compatibility have been addressed in the waste compatibility DQO (Carothers 1994). This DQO is based on both safety and operational considerations. Operational considerations include pumpability and corrosion. Comparisons between some of the key criteria for evaluating compatibility and the 1995 supernatant results are summarized in Table 5-9. No viscosity or cooling curve analysis was required since historical information already exists which adequately addresses the potential for line plugging and precipitation of solids during the transfer of waste (Schreiber 1995).

It should be noted that all of the criteria listed in Table 5-9 are met and that the analysis indicates that the waste is compatible with other similar tank waste types. Additionally, low phosphate (<360 μg/g) indicates that the potential for insoluble phosphates forming is low and the waste is pumpable. Other operational factors, as defined and discussed in Carothers (1994), need to be considered as part of the overall assessment before the waste is transferred out of the tank.

5.5.3 Process Development Evaluation

The metal analysis of the sludge in 1989 is important for evaluating the disposal waste form (glass) formulations and identifying potential components that may affect the treatment and disposal process. Because the waste sludges may be blended, washed and treated before disposal, there are no specific criteria for the parameters measured. The 1989 physical

Table 5-9. Compatibility Assessment for Tank 241-AZ-102 Supernatant.

Parameter	Criteria	1995 Result
$^{239/240}Pu$	< 0.05 g/gal (0.8 μCi/g)[a]	< 2.7 E-03 μCi/g
SpG	< 1.41	1.10
Energetics	exotherm/endotherm < 1	0
Corrosivity[b]	If $[NO_3^-]$ and $[OH^-]$ each < 1.0 M, then $[NO_2^-] \geq 0.011$ M	$[NO_3^-] = 0.0384$ M $[OH^-] = 0.110$ M $[NO_2^-] = 0.0600$ M
TRU	< 100 nCi/g	< 7 nCi/g
TOC	< 10 g/L	1.50 g/L

[a]In converting g/gal to μCi/g, the specific activity of ^{239}Pu was used and the supernatant density assumed to be 1 g/mL.

[b]Corrosivity decision rules were not developed for the aging waste tanks in the waste compatibility DQO because aging wastes are no longer generated with the permanent shutdown of PUREX (Carothers 1994). Instead, the wastes stored in tank 241-AZ-102 must comply with the corrosion specifications listed in *Operating Specifications for Aging-Waste Operations in 241-AY and 241-AZ* (Bergmann 1989). The criteria shown in the table are from (Bergmann 1989).

SpG = specific gravity.

measurements, discussed in Section 4.3, will be important in evaluating retrieval and pretreatment process equipment needs. Solubility information was obtained from results of the analyses of water washed solids. Table 5-10 compares the results from these water washed solids with those obtained from analyzing the core composite centrifuged solids. The analytes listed in the table were chosen because of their environmental concern. All of the data was taken from Gray et al. (1993).

Specific analyses were also requested to be performed on tank 241-AZ-102 waste for process testing purposes. A sludge washing test has been developed to gain information relative to the use of tanks 241-AZ-101 and 241-AZ-102 to fully test the process, equipment, and instruments needed for in-tank processing of the waste. The test plan for this operation has just recently been completed and is contained in Herting (1995). Utilizing this test plan, the requested analyses will be performed in accordance with the requirements outlined in Schreiber (1995).

Table 5-10. Comparison of Washed Solids and Centrifuged Solids Results.

Analyte	Washed Solids (μg/g or μCi/g)	Centrifuged Solids (μg/g or μCi/g)
Ag	169	674
Ba	140	553
Cd	3,690	15,000
Cr	372	1,920
Ni	2,120	8,860
Pb	369	1,100
U	2,950	12,600
^{241}Am	25	101
^{137}Cs	146	880
^{90}Sr	5,530	22,000

6.0 CONCLUSIONS AND RECOMMENDATIONS

The sludge in tank 241-AZ-102 has been sampled and analyzed three times, twice in 1987 and most recently in 1989. The supernatant was also analyzed in the two 1987 samplings. The most recent supernatant sampling occurred in February 1995. The analysis of tank 241-AZ-102 does not fully meet the safety screening requirements. Limited samples and data from different risers and waste depths make it difficult to accurately determine the variability of the waste composition in the tank. Composition and quantity of sludge have changed since the 1989 characterization because of continuing transfers of waste into the tank and settling of that waste. Limited quality control data for the 1987 and 1989 analyses make it difficult or impossible to assess the accuracy and variability for some measurements. However, a comparison of the results from the latest sampling events of each waste phase to the safety screening criteria seem to indicate that the tank waste does not pose any immediate safety concerns. The fuel content of the sludge has not directly been measured, although TOC analyses and historical information do not indicate that excessive fuel is present. If the sludge is sampled again, it is recommended that DSC and TGA be performed. The supernatant meets the compatibility assessment criteria.

The sludge contained large quantities of iron, sodium, aluminum, uranium, SO_4^{2-}, NO_2^-, and NO_3^- as expected from the NCAW waste stream. Also found in relatively high concentrations were nickel and chromium, which entered the waste stream through corrosion of PUREX piping and process vessels.

$^{89/90}Sr$, ^{90}Sr, ^{137}Cs, $^{239/240}Pu$, ^{106}Ru, and ^{241}Am were the most abundant radionuclides found in the waste. The heat generated by these and other significant isotopes is approximately <58,441 W (<199,457 Btu/hr). The estimated heat load is far below the 4,000,000 Btu/hr design limit for the tank (Bergmann 1989).

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APPENDIX A

ANALYTICAL RESULTS — 1989 CORE SAMPLE DOUBLE-SHELL TANK 241-AZ-102

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A.1 INTRODUCTION

Table A-1 is a summary of the chemical and radiological characteristics of the sludge layer in tank 241-AZ-102. The column labeled Analyte contains the symbol for the analyte. The second and fourth columns are the centrifuged solid and centrifuged liquid results in units of μmol/g, as they were reported in Gray et al. (1993). The third and fifth columns are these same results converted to μg/g.

Table A-2 presents the division of the uranium and plutonium data into their respective isotopes by weight percentage, according to Gray et al. (1993). The third and fifth columns are these same results converted to μg/g.

Table A-1. Analytical Summary for the Sludge in Double-Shell Tank 241-AZ-102. (3 sheets)

Analyte	Centrifuged solid		Centrifuged liquid	
Metal	(μmol/g)	(μg/g)	(μmol/g)	(μg/g)
Aluminum	1,990	53,700.0	3.30	89.1
Antimony	<28	<3,410.0	<0.074	<9.0
Arsenic	<7.9	<592.0	0.050	3.8
Barium	4.03	553.0	0.005	0.7
Beryllium	1.50	13.5	0.011	0.1
Boron	<46	<497.0	0.676	7.3
Cadmium	133	15,000.0	0.015	1.7
Calcium	75.4	3,020.0	0.132	5.3
Cerium	<5.6	<785.0	0.074	10.4
Chromium	37.0	1,920.0	16.9	879.0
Cobalt	<25	<1,470.0	0.127	7.5
Copper	5.50	350.0	0.043	2.7
Dysprosium	<0.31	<50.4	0.005	0.8
Iron	2,350	131,000.0	0.170	9.5
Lanthanum	32.4	4,500.0	0.011	1.5
Lead	5.29	1,100.0	0.025	5.2
Lithium	<4.2	<29.2	0.011	0.1
Magnesium	39.1	950.0	0.032	0.8
Manganese	52.4	2,880.0	0.012	0.7
Molybdenum	0.49	47.0	0.332	31.9
Neodymium	20.2	2,910.0	0.033	4.8
Nickel	151	8,860.0	0.032	1.9
Phosphorus	31.5	977.0	1.89	58.6
Plutonium		59.6		0.5
Potassium	53.1	2,080.0	32.3	1,260.0
Rhenium	<0.39	<72.6	0.004	0.7

Table A-1. Analytical Summary for the Sludge in Double-Shell Tank 241-AZ-102. (3 sheets)

Analyte	Centrifuged solid		Centrifuged liquid	
Rhodium	<4.1	<422.0	0.109	11.2
Ruthenium	<1.8	<182.0	0.041	4.2
Selenium	<13	<1,030.0	0.120	9.5
Silicon	160	4,480.0	17.3	484.0
Silver	6.25	674.0	0.056	6.0
Sodium	2,570	59,100.0	1,830	42,100.0
Strontium	3.73	327.0	0.002	0.2
Tellurium	<2.7	<345.0	0.018	2.3
Thallium	<41	<8,380.0	0.163	33.3
Thorium	<2.1	<487.0	<0.0054	<1.3
Titanium	1.62	77.6	0.009	0.4
Uranium	12.6 mg/g	12,600.0	1.40 mg/g	1,400.0
Vanadium	<0.64	<32.6	0.035	1.8
Zinc	2.15	141.0	0.004	0.3
Zirconium	198	18,100.0	0.041	3.7
Ion	(μmol/g)	(μg/g)	(μmol/g)	(μg/g)
Cl^-	4.15	147.0	1.69	59.9
Cr(VI)	--	--	2.02	105.0
F^-	24.2	460.0	38.9	739.0
NO_3^-	40.6	2,520.0	92.7	5,750.0
NO_2^-	335	15,400.0	61.3	2,820.0
PO_4^{3-}	4.38	416.0	1.36	129.0
SO_4^{2-}	68.1	6,540.0	124	11,900.0
Carbon	μmol/g	μg C/g	μmol/g	μg C/g
TOC	263	3,160.0	112	1,340.0
TIC	1,300	15,600.0	587	7,040.0

Table A-1. Analytical Summary for the Sludge in Double-Shell Tank 241-AZ-102. (3 sheets)

Analyte	Centrifuged solid	Centrifuged liquid
Radionuclide	(μCi/g)	(μCi/g)
^{241}Am	101.0	1.3E-03
^{125}Sb	576.0	<8.0
^{14}C	1.8 E-03	1.1 E-03
^{144}Ce	ND	ND
^{137}Cs	880.0	747.0
^{154}Eu	ND	ND
^{129}I	ND	ND
^{237}Np	0.1	1.7 E-03
^{106}Ru	4,030.0	<8.1
^{79}Se	<3.0 E-05	3.7 E-04
^{90}Sr	22,000.0	6.0
^{99}Tc	0.6	0.1

Table A-2. Distribution of Uranium and Plutonium Isotopes.

Isotope	Wt %[a]	μg/g	Ci/g[d]	Ci[e]
U Isotopes[b]				
^{234}U	0.00663	0.63	6.25 E-03	1.95
^{235}U	0.782	74.00	2.16 E-06	0.08
^{236}U	0.0447	4.23	6.47 E-05	0.14
^{238}U	99.17	9,385.44	3.36 E-07	1.56
Plutonium isotopes[c]				
^{238}Pu	0.0471	0.02	1.71 E+01	169.63
^{239}Pu	92.26	39.67	6.20 E-02	1,219.90
^{240}Pu	7.00	3.01	2.27 E-01	338.90
^{241}Pu	0.624	0.27	1.03 E+02	13,794.00
^{242}Pu	0.0761	0.03	3.93 E-03	0.06

[a]Based on the weight percentage breakdowns given in Gray et al. (1993).

[b]Based on an average sludge uranium concentration of 9,464.0 μg/g (Table 4-2).

[c]Based on an average sludge plutonium concentration of 43.0 μg/g (Table 4-2).

[d]Specific activities for each of these radionuclides have been taken from N. P. Willis and G. C. Triner, 1991, *Hanford Site Solid Waste Acceptance Criteria*, WHC-EP-0063-3, Westinghouse Hanford Company, Richland, Washington.

[e]Total curies calculated using a total sludge mass of 4.96 x 10^8 g (from a sludge volume of 333 kL at 1.49 g/mL).

REFERENCE

Gray, W. J., M. E. Peterson, R. D. Scheele, J. M. Tingey, 1993, *Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 102-AZ*, Pacific Northwest Laboratories, Richland, Washington.

APPENDIX B

ANALYTICAL RESULTS — 1995 GRAB SAMPLES DOUBLE-SHELL TANK 241-AZ-102

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B.1 INTRODUCTION

Appendix B presents the chemical and radiological characteristics of the 1995 Grab Sample taken from tank 241-AZ-102, in a tabular form, in terms of the specific concentrations of metals, ions, radionuclides, physical properties, and organic constituents.

The data table for each analyte lists the laboratory sample identification number, the mean of the primary and duplicate results, the mean of all the results, the relative standard deviation (RSD) of the mean, and a projected tank inventory. The RSD of the mean is the standard deviation of the mean divided by the mean. The standard deviation of the mean was computed using analysis of variance methods.

B.2 TABLE DESCRIPTION

Tables B-1 and B-2 are divided into groups dependent on the characteristics of the analytes.

Table B-1. Location of Tables.

Analyte characteristic	Table number
Metals	Table B-1
Ions (anions and cations)	Table B-2
Radionuclides	Table B-3
Physical Properties	Table B-4
Total Carbon	Table B-5

Table B-2. Description of Analytical Methods.

Metals:	ICP -	Inductively Coupled Plasma (generic for all metals unless otherwise indicated)
Anions:	IC -	Ion Chromatograph
	Pot. Titrat. -	Potentiometric Titration
Radionuclides:	GEA -	Gamma Energy Analysis
	Extr. -	Extraction
	IonEx -	Ion Exchange
	HighLev. -	High Level Alpha and Beta Analysis
Physical Properties	TGA -	Thermo-gravimetric Analysis
	Grav. -	Gravimetric
Total Carbon	Coul. -	Coulometry

Note: Standard abbreviations are used to describe analytical methods.

B.3 COLUMN HEADINGS

The column labeled Analyte contains information about the method of measurement, in addition to the name of the analyte or physical characteristic. Additionally, where applicable, information about the method of digestion.

Digestion methods will be denoted for those analytes that were digested by more than one method. Digestion methods used are abbreviated: a is acid digestion, w is water leach, and f is potassium hydroxide fusion, followed by acid digestion. Analytes may also be measured directly on an undigested sample and these are abbreviated with d as direct.

The analyte and method are presented as follows: method.analyte, or, method.digestion.analyte (where applicable). For example, the specific concentration of sodium was determined by the ICP method which was preceded by acid digestion, and is listed as ICP.a.Na.

The column labeled Sample Number lists the laboratory sample from which the analyte was measured; this identification number is different from the number assigned to the samples at the tank farm. Sampling rationale, locations, and a description of the sampling event is contained in Section 3. The 1995 grab samples were obtained from tank 241-AZ-102 through riser 24A. Table B-3 provides the grab sample breakdown.

Table B-3. Sample Breakdown and Identification Numbers.

Grab sample number	Laboratory sample number
AZ-102-1	S95T000245 S95T000263 S95T000266
AZ-102-2	S95T000246 S95T000264 S95T000267
AZ-102-3	S95T000247 S95T000265 S95T000268

Three additional samples (S95T000743, S95T000744, and S95T000746) were taken and archived. The samples in Tables B-4 through B-24 are listed in order of increasing depth.

Result is the specific concentration of the analyte determined at different sampling points. No quality control data such as matrix spikes, serial dilutions, or duplicate analyses are listed. This information may be obtained from the tank 241-AZ-102 grab sample data package (Rollinson 1995a and 1995b).

The number listed is an average between the primary sample and its duplicate sample. Where more than one duplicate sample was performed the primary sample was averaged with all the duplicates.

The last column lists the total projected inventory of tank 241-AZ-102 (after μg/mL have been converted to μg/g using a supernatant density of 1.10 g/mL) using a total supernatant volume of 3,236 kL. This is calculated using the following equation:

[(Supernatant Concentration)(Supernatant Density)(Total Supernatant Volume)(Unit Conversions)](Unit Conversions)

Example calculation for aluminum:

$$[(140.9\ \mu g/g)(1.10\ g/mL)(3{,}236{,}000\ L)(1{,}000\ mL/L)\ 1 \times 10^{-6}\ g/\mu g)](1 \times 10^{-3}\ kg/g) = 6{,}069.1\ kg.$$

Numbers that are preceded by a less than symbol (<) indicate the analyte was noted, but was below the analytical instrument's calibrated detection limit for the sample.

Table B-4. Tank 241-AZ-102 Analytical Data: Aluminum.

Analyte	Sample Number	Result	Mean	RSD (Mean)	Projected Inventory
Metal					
Drainable Liquid		μg/mL	μg/mL	%	kg
ICP.a.Al	S95T000266	155	155	0.22	501.5
	S95T000267	154			
	S95T000268	154.5			

Table B-5. Tank 241-AZ-102 Analytical Data: Iron.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Metal					
Drainable liquid		µg/mL	µg/mL	%	kg
ICP.a.Fe	S95T000266	<10.0	<10.0	N/A	<32.4
	S95T000267	<10.0			
	S95T000268	<10.0			

Table B-6. Tank 241-AZ-102 Analytical Data: Sodium.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Metal					
Drainable liquid		µg/mL	µg/mL	%	kg
ICP.a.Na	S95T000266	5,340	5,320	0.23	17,215.6
	S95T000267	5,320			
	S95T000268	5,300			

Table B-7. Tank 241-AZ-102 Analytical Data: Chloride.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.Cl^-	S95T000263	<121	<66	N/A	<213.6
	S95T000264	<66			
	S95T000265	<66			

Table B-8. Tank 241-AZ-102 Analytical Data: Fluoride.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.F^-	S95T000263	1,080	1,005	4.69	3,252.1
	S95T000264	914			
	S95T000265	1,020			

Table B-9. Tank 241-AZ-102 Analytical Data: Hydroxide.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
Pot.Titrat.OH^-	S95T000246	1,920	1,880	2.39	6,083.7
	S95T000247	1,840			

Table B-10. Tank 241-AZ-102 Analytical Data: Nitrate.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.NO_3^-	S95T000263	25,600	23,800	5.10	77,016.9
	S95T000264	21,400			
	S95T000265	24,300			

Table B-11. Tank 241-AZ-102 Analytical Data: Nitrite.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.NO_2^-	S95T000263	29,600	27,600	4.77	89,313.6
	S95T000264	25,100			
	S95T000265	28,200			

Table B-12. Tank 241-AZ-102 Analytical Data: Phosphate.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.PO_4^{3-}	S95T000263	<726	<396	N/A	<1,281.5
	S95T000264	<396			
	S95T000265	<396			

Table B-13. Tank 241-AZ-102 Analytical Data: Sulfate.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Anion					
Drainable liquid		µg/mL	µg/mL	%	kg
IC.SO_4^{2-}	S95T000263	18,700	17,300	5.04	55,982.9
	S95T000264	15,700			
	S95T000265	17,500			

Table B-14. Tank 241-AZ-102 Analytical Data: ^{241}Am.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Radionuclide					
Drainable liquid		µCi/mL	µCi/mL	%	Ci
Extr.^{241}Am	S95T000263	<0.00429	<0.00429	N/A	<15.3
	S95T000264	<0.00439			
	S95T000265	<0.00462			

Table B-15. Tank 241-AZ-102 Analytical Data: ^{137}Cs.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Radionuclide					
Drainable liquid		µCi/mL	µCi/mL	%	Ci
GEA.^{137}Cs	S95T000263	1,040	1,040	0.47	3,370,000
	S95T000264	1,040			
	S95T000265	1,040			

Table B-16. Tank 241-AZ-102 Analytical Data: ^{238}Pu.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Radionuclide					
Drainable liquid		µCi/mL	µCi/mL	%	Ci
IonEx. ^{238}Pu	S95T000263	<0.00863	<0.00296	N/A	<9.6
	S95T000264	<0.0119			
	S95T000265	<0.00296			

Table B-17. Tank 241-AZ-102 Analytical Data: $^{239/240}Pu$.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Radionuclide					
Drainable liquid		µCi/mL	µCi/mL	%	Ci
IonEx. $^{239/240}Pu$	S95T000263	<0.00863	<0.00296	N/A	<9.6
	S95T000264	<0.0119			
	S95T000265	<0.00296			

Table B-18. Tank 241-AZ-102 Analytical Data: $^{89/90}Sr$.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Radionuclide					
Drainable liquid		µCi/mL	µCi/mL	%	Ci
HighLev. $^{89/90}Sr$	S95T000263	1.59	1.79	13.10	5,802.1
	S95T000264	1.53			
	S95T000265	2.26			

Table B-19. Tank 241-AZ-102 Analytical Data: Gravimetric Percent Water.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Physical property					
Drainable liquid		%	%	%	
Grav. $\%H_2O$	S95T000245	84.5	84.5	0.06	N/A
	S95T000246	84.6			
	S95T000247	84.5			

Table B-20. Tank 241-AZ-102 Analytical Data: Thermo-Gravimetric Analysis Percent Water.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Physical Property					
Drainable liquid		%	%	%	
TGA. $\%H_2O$	S95T000245	84.4	84.1	0.33	N/A
	S95T000246	84.4			
	S95T000247	83.5			

Table B-21. Tank 241-AZ-102 Analytical Data: Specific Gravity.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Physical Property					
Drainable liquid					
SpG	S95T000245	1.10	1.10	0.00	N/A
	S95T000246	1.10			
	S95T000247	1.10			

Table B-22. Tank 241-AZ-102 Analytical Data: pH.

Analyte	Sample Number	Result	Mean	RSD (Mean)	Projected Inventory
Physical Property					
Drainable liquid				%	
pH	S95T000245	12.9	12.9	0.07	N/A
	S95T000246	12.9			
	S95T000247	13.0			

Table B-23. Tank 241-AZ-102 Analytical Data: Total Organic Carbon.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Carbon					
Drainable liquid		µg/mL	µg/mL	%	kg
Coul.TOC	S95T000263	1,560	1,503	3.16	4,863.8
	S95T000264	1,540			
	S95T000265	1,410			

Table B-24. Tank 241-AZ-102 Analytical Data: Total Inorganic Carbon.

Analyte	Sample number	Result	Mean	RSD (mean)	Projected inventory
Carbon					
Drainable liquid		µg/mL	µg/mL	%	kg
Coul.TIC	S95T000263	5,690	5,843	3.57	18,907.9
	S95T000264	5,980			
	S95T000265	5,860			

REFERENCE

Rollison, M. D., 1995a, *Results for 241-AZ-102 Grab Samples* (internal memo 8E480-95-024, to J. M. Jones, April 11, 1995), Westinghouse Hanford Company, Richland, Washington.

Rollison, M. D., 1995b, *Results for 241-AZ-102 Grab Samples, Revision 1*, (internal memo 75970-95-039, to J. M. Jones, June 29, 1995), Westinghouse Hanford Company, Richland, Washington.

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APPENDIX C

SAMPLING RESULTS PRIOR TO 1989
DOUBLE-SHELL TANK 241-AZ-102

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C.1 INTRODUCTION

The data in Tables C-1 and C-2 was derived from *Chemical Analysis of Tank AZ-102 Samples Taken from August 1987* and *Chemical Analysis of Tank AZ-102 Samples Taken from October 1987* (Herting 1987 and Herting 1988). The sampling and subsequent analysis of these samples is discussed in Section 3. Sampling events in 1985 and earlier were not considered relevant to the current contents of tank 241-AZ-102 due to significant waste transfers in 1985 and 1986.

Table C-1. Previous Sampling Results of Tank 241-AZ-102 (October 1987). (2 sheets)

Analyte	Supernatant (μg/mL)	Centrifuged solids-water digestion (μg/mL)		Centrifuged solids-acid digestion (μg/mL)	
		Sample A	Sample B	Sample A	Sample B
Aluminum	2,620	864	1,380	2,970	ND
Sodium	37,000	31,300	23,000	920	11,300
Iron	--	--	--	68,600	72,000
Calcium	--	--	--	2,040	23,600
Chromium	--	--	--	1,820	1,870
Magnesium	--	--	--	316	802
Nickel	--	--	--	4,870	4,930
Zirconium	--	--	--	4,560	4,920
NO_3^-	21,700	15,900	12,000	--	--
NO_2^-	12,400	12,100	10,200	--	--
CO_3^{2-}	12,000	4,680	3,540	--	--
F^-	703	665	513	22,200	20,700
PO_4^{3-}	475	ND	190	ND	3,520
SO_4^{2-}	10,800	9,220	7,580	ND	ND
Cl^-	248	ND	ND	ND	ND
OH^-	3,400	--	--	--	--
TOC as carbon	1,520	1,240	1,140	7,040	6,710
Uranium	45	3.2	310	7,520	2,710

Table C-1. Previous Sampling Results of Tank 241-AZ-102 (October 1987). (2 sheets)

Radionuclides	Supernatant (μCi/mL)	Centrifuged solids-water digestion (μCi/mL)		Centrifuged solids-acid digestion (μCi/mL)	
		Sample A	Sample B	Sample A	Sample B
$^{89/90}Sr$	1.46	0.496	1.190	9,900	8,700
^{129}I	<0.002	--	--	--	--
^{137}Cs	936	603.2	496	280	300
$^{144}CePr$	--	--	--	6,500	6,000
^{241}Am	<0.032	--	--	130	96
Total beta	1,370	--	--	--	--
Total alpha	0.200	0.0053	0.003	170	160

Table C-2. Previous Sampling Results of Tank 241-AZ-102 (August 1987). (2 sheets)

Analyte	Supernatant (μg/mL)	Centrifuged solids-water digestion (μg/mL)		Centrifuged solids-acid digestion (μg/mL)	
		Sample A	Sample B	Sample A	Sample B
Aluminum	2,300	216	54	2,510	2,650
Sodium	36,100	6,210	1,150	1,610	690
Iron	--	--	--	52,500	61,400
Calcium	--	--	--	4,000	3,600
Chromium	--	--	--	1,980	2,340
Magnesium	--	--	--	1,360	1,090
Nickel	--	--	--	3,640	4,110
Zirconium	--	--	--	--	3,190
Arsenic	2.8	--	--	--	--

Table C-2. Previous Sampling Results of Tank 241-AZ-102 (August 1987). (2 sheets)

Analyte	Supernatant (μg/mL)	Centrifuged solids-		Centrifuged solids-	
		Sample A	Sample B	Sample A	Sample B
NO_3^-	21,700	ND	ND	--	--
NO_2^-	12,400	1,380	2,760	--	--
CO_3^{2-}	16,200	ND	ND	--	--
F^-	722	38	19	--	--
PO_4^{3-}	<5,230	--	--	1,900	--
SO_4^{2-}	10,600	384	ND	<182,000	<144,000
Cl^-	2,160	--	--	<21,300	<17,700
OH^-	3,060	--	--	--	--
TOC as carbon	720	3,800	5,200	10,500	7,800
Uranium	290	--	--	6,700	5,400
Radionuclides	Supernatant (μCi/mL)	Centrifuged solids-water digestion (μCi/mL)		Centrifuged solids-acid digestion (μCi/mL)	
		Sample A	Sample B	Sample A	Sample B
$^{89/90}Sr$	0.00165	--	1.53	4,500	5,400
^{99}Tc	37.2	12,100	5,400	ND	ND
$^{106}RuRh$	--	234	252	2,400	3,100
^{129}I	<2E-05	--	--	--	--
^{137}Cs	735	--	--	--	--
$^{144}CePr$	--	--	--	4,400	5,700
$^{239/240}Pu$	<0.018	--	--	0.790	0.930
^{241}Am	<0.018	--	--	85.0	95.0
Total Beta	1,360	--	--	--	--
Total Alpha	<0.088	--	--	89.4	94.0

REFERENCES

Herting, D. L., 1987, *Chemical Analysis of Tank 102-AZ Samples Taken in August 1987*, (internal memo 12221-PCL87-041 to L. A. Mihalik, December 8, 1987), Westinghouse Hanford Company, Richland, Washington.

Herting, D. L., 1988, *Chemical Analysis of Tank 101-AZ and 102-AZ Samples Taken in October 1987*, (internal memo 12221-PCL88-091 to L. A. Mihalik, March 11, 1988), Westinghouse Hanford Company, Richland, Washington.

APPENDIX D

ANALYTICAL METHODS DOUBLE-SHELL TANK 241-AZ-102

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Table D-1. Analytical Methods For Waste Compatibility and Process Control Samples (Schreiber 1995).

Analyte	Method	Procedure
OH^-	Titration	LA-211-102
TIC as carbonate	Coulometry	LA-622-102
Anions Cl^-, F^-, NO_3^-, NO_2^-, PO_4^{3-}, SO_4^{2-}	IC	LA-533-105
Net exothermic energy	DSC/TGA	LA-560-112 LA-514-113
Sodium, Aluminum, Iron	ICP	LA-505-151
TOC	Furnace oxidation	LA-344-105
^{137}Cs	Gamma energy analysis	LA-548-121
^{90}Sr	Separation and β count	LA-220-101
$^{239/240}Pu$	Separation and α count	LA-503-156
^{241}Am	Separation and α count	LA-503-156
Vol% solids	Centrifugation	LA-519-132
$[H^+]$	pH	LA-212-103
Liquid density	SpG	LA-510-112

DSC = differential scanning calorimetry.
IC = ion chromatograph.
ICP = inductively coupled plasma.
SpG = specific gravity.
TGA = thermo-gravimetric analysis.

Table D-2. Analytical Procedures For 1989 Core Samples.

Analyte	Method	Procedure
Density, vol% solids, % oxides, rheology	Mass and volume measurement, shear strength, viscosity	PNL-ALO-501
% Solids	Centrifugation	PNL-ALO-504
$[H^+]$	pH	PNL-ALO-290
OH^-	Titration	PNL-ALO-228
Total metals	Inductively coupled plasma	PNL-ALO-211
Mercury	Cold vapor atomic absorption	PNL-ALO-213
Anions	Ion chromatograph	PNL-ALO-212
Total carbon	Hot persulfate-liquids/solids coulometry-solids	PNL-ALO-381 PNL-ALO-380
Cr(VI)	Absorption	PNL-ALO-227
Total uranium	Laser fluorimetry	PNL-ALO-445
^{137}Cs, ^{60}Co, $^{154/155}Eu$, ^{125}Sb, and ^{106}Ru	Gamma energy analysis	PNL-ALO-450
$^{243/244}Cm$, ^{241}Am, $^{239/240}Pu$, and ^{237}Np	Alpha Energy Analysis	PNL-ALO-422
^{129}I	Separation, liquid scintillation counting	PNL-ALO-454/450
^{99}Tc	Separation, liquid scintillation counting	PNL-ALO-431/432
^{14}C	Separation, liquid scintillation counting	PNL-ALO-482/474
^{3}H	Separation, liquid scintillation counting	PNL-ALO-441/474
Total alpha	Evaporation, alpha proportional counting	PNL-ALO-420/421
Total beta	Evaporation, beta proportional counting	PNL-ALO-430/431

REFERENCE

Schreiber, R. D., 1995, *Tank 241-AZ-102 Tank Characterization Plan*, WHC-SD-WM-TP-228, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

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