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Tank Characterization Report for Single-Shell Tank 241-BY-110

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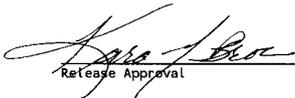
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Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-BY-110. This report supports the requirements of Tri-Party Agreement Milestone M-44-09.

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Tank Characterization Report for Single-Shell Tank 241-BY-110

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EXECUTIVE SUMMARY

This tank characterization report summarizes information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell underground tank 241-BY-110. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996) and the Ferrocyanide Safety Program, Milestone T22-96-020 (WHC 1995b). As a result of the analyses addressed by this report, the tank meets the "safe" category based on the decision limits of the ferrocyanide data quality objective (DQO) (Schreiber 1995, 1996b). However, tank 241-BY-110 meets the "conditionally safe" category as defined by the organic DQO.

Tank 241-BY-110 is one of 12 single-shell underground waste storage tanks located in the 200 East Area BY Tank Farm on the Hanford Site. It is the first tank in a three-tank cascade. The tank went into service in March 1951 and received first-cycle decontamination waste from the bismuth phosphate process. An active process history followed the initial waste receipts. The tank received ferrocyanide-scavenged waste (1954 to 1957), cladding waste (1957 to 1958), in-tank solidification waste (1969 to 1976), evaporator bottoms (1970 and 1974 to 1975), and noncomplexed evaporator feed (1977 to 1979). The ferrocyanide-scavenged waste was allowed to settle in the tank prior to discharging the supernatant to various cribs (Agnew et al. 1995).

A description and the status of tank 241-BY-110 are summarized in Table ES-1 and Figure ES-1. The tank has an operating capacity of 2,870 kL (758 kgal) and presently

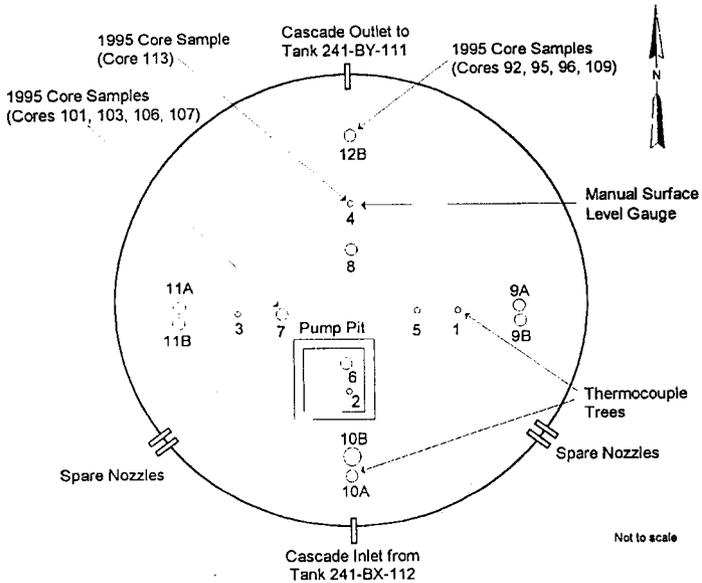
Table ES-1. Description and Status of Tank 241-BY-110.

TANK DESCRIPTION	
Type	Single-shell
Constructed	1948 to 1949
In service	1951
Diameter	23 m (75 ft)
Maximum operating depth	7 m (23 ft)
Capacity	2,870 kL (758 kgal)
Bottom shape	Dish
Ventilation	Passive
TANK STATUS	
Waste classification	Noncomplexed
Total waste volume	1,507 kL (398 kgal)
Sludge volume ¹	390 kL (103 kgal)
Drainable interstitial liquid	34 kL (9 kgal)
Saltcake volume ¹	1,120 kL (295 kgal)
Waste surface level (1992 to 1995)	385 cm (152 in.)
Temperature (1974 to present)	20 °C (68 °F) to 62 °C (143 °F)
Integrity	Sound
Watch List	Ferrocyanide
SAMPLING DATES	
Vapor sample	November 1994
Rotary mode core sample	July 11 to October 28, 1995
SERVICE STATUS	
Intrusion prevention	October 1989
Interim stabilized	January 1985

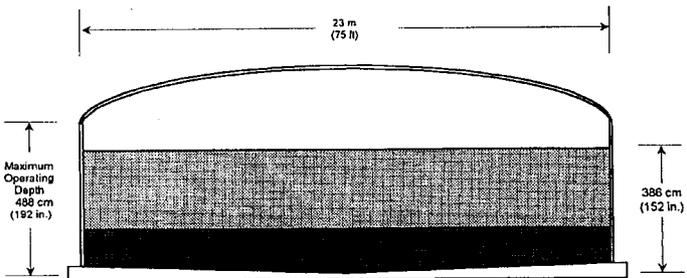
Notes:

¹Interpretation of other sampling and surveillance data suggests a range of values. Section 4.1 elaborates further on the volumes used in calculating inventories.

Figure ES-1. Profile of Tank 241-BY-110.



Not to scale



Not to scale

Total Tank Volume: 2,870 kL (758 kgal)
 Waste Volume: 1,510 kL (398 kgal)
 Sludge Volume: 390 kL (103 kgal)
 Saltcake Volume: 1,120 kL (295 kgal)
 Supernate Volume: 0 kL (0 kgal)
 (Hanton 1996)

contains 1,507 kL (398 kgal) of waste. The total amount is approximately 390 kL (103 kgal) of sludge and 1,120 kL (295 kgal) of saltcake; no supernatant liquid remains. The sludge contains approximately 34 kL (9 kgal) of drainable interstitial liquid. These volumes are based on surveillance information and sampling observations. Interpretation of other surveillance and sampling data suggests minor variations from these values. Waste surface levels have remained constant at 385 cm (152 in.) over the past three years (Hanlon 1996). Tank 241-BY-110 is on the Ferrocyanide Watch List and has operating controls for tanks on the Organic Watch List.

The characterization of tank 241-BY-110 is based on a core sampling event that took place from July 11 through October 28, 1995 and a 1994 vapor sampling event. During the 1995 sampling event, cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 were taken from tank 241-BY-110 using the rotary and push mode core sampling methods. The cores were extruded and analyzed at the Westinghouse Hanford Company 222-S Laboratory in accordance with the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994), the *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994), the *Test Plan for Samples From Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-106, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109* (Meacham 1995), the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995), and the *Historical*

Model Evaluation Data Requirements (Simpson and McCain 1995). Analyses for all cores included energetics, moisture content, total alpha activity, density, metals, cyanide, anions, total organic carbon, and an organic screen analysis (Schreiber 1996a).

Total alpha activity results for all cores were well below the safety screening limit of 29.7 $\mu\text{Ci/g}$; the highest average subsegment result was 0.238 $\mu\text{Ci/g}$, indicating the potential for a criticality event is low.

Thermogravimetric analysis showed that the tank is drier than predicted by Agnew et al. (1996a), but still contains between 20 and 30 percent water. Because of the various physical processes acting in this tank, it does not have the moisture distribution observed in other waste tanks, which had much simpler process histories. Based on the data, the moisture distribution is highly irregular.

No sample obtained from tank 241-BY-110 had mean exothermic reactions (on a dry-weight basis) exceeding the safety screening, organic, and ferrocyanide DQOs enthalpy change limit of -480 J/g (dry weight). The computation of a one-sided 95 percent confidence interval (required by the safety screening DQO) indicated the calculated limit for exothermic change in enthalpy was -475 J/g (dry). Further, the majority of the samples that demonstrated exothermic activity contained at least 25 percent water, which reduces the potential for a propagating reaction.

The observed cyanide concentrations were less than 1,000 $\mu\text{g/g}$, well below the ferrocyanide DQO limit of 39,000 $\mu\text{g/g}$. Analytical nickel concentrations are reasonably close to historical estimates (Borsheim and Simpson 1991) and indicate that ferrocyanide has substantially (> 90 percent) decomposed. The data suggest that this tank should be removed from the Ferricyanide Watch List.

The organic screen analysis revealed that minor amounts of normal paraffin hydrocarbons were present in the solid phase (Schreiber 1996b), but no separable organic layer was observed in the liquids. Organic vapors were detected in small quantities in the headspace and breather filter. Most total organic carbon results were below the notification limit of 30,000 $\mu\text{g/g}$. However, two samples had results above the notification limit: (S95T002207 and S95T002093). Three samples had a one-sided 95 percent confidence interval with values greater than the limit (S95T001755, S95T002217, and S95T001745). The sample analyses are inconclusive regarding the degradation of organic compounds.

Historical data do not indicate that tank 241-BY-110 received substantial amounts of organic complexants (Agnew et al. 1996b). No data were obtained regarding the solubility of organic complexants. The observed values for TOC are not sufficient to account for the observed exotherms in some cases.

The heat load in the tank produced by radioactive decay is estimated at 2.59 kW. This value is reasonably close to the 2.02 kW estimated from the headspace temperature (Kummerer 1994). Surveillance data show tank temperatures ranged from 62 °C (143 °F) to 20 °C

(68 °F) from 1974 to the present. However, temperatures have remained between 33 and 41 °C (91 and 106 °F) for the past three years. The heat load and thermal history indicate that the waste cannot generate radiolytic temperatures high enough to initiate an exothermic reaction.

Before samples were removed from the tank, combustible gas meter readings were taken from inside the vapor space as required by the safety screening DQO (Dukelow et al. 1995). No result was greater than one percent of the lower flammability limit, satisfying the DQO requirement that the results be less than 25 percent of the lower flammability limit. These measurements are consistent with the findings of a previous headspace sampling effort (Huckaby and Bratzel 1995). However, measurements in the drill string indicate that radiologically generated gases accumulate in the waste. The gases are rapidly dispersed when encountered during sampling. Present operational measures (application of flammable gas controls during sampling) appear to be prudent and appropriate when performing intrusive in-tank activities, because H₂ concentrations of 24 percent have been observed in the drill string vapor space.

Remaining material from the sampling event has been set aside for pretreatment studies as identified in the pretreatment DQO.

Table ES-2 shows concentration and inventory estimates for the most prevalent analytes and analytes of concern. These concentrations are based on the 1995 analytical results.

Table ES-2. Major Analytes and Analytes of Concern.

Metals	Saltcake (Mean)		Sludge (Mean)		Projected Inventory ¹
	µg/g	RSD (%)	µg/g	RSD (%)	kg
Aluminum	14,100	13.4	28,300	4.20	40,900
Calcium	400	17.8	14,200	17.4	9,360
Chromium	2,900	11.4	2,220	27.0	6,200
Iron	924	23.9	20,000	22.7	13,700
Nickel	193	23.3	6,670	20.7	4,400
Sodium	237,000	2.99	161,000	5.47	495,000
Strontium	58.1	16.0	6,840	31.7	4,290
Uranium	697	16.7	20,900	22.4	14,000
Anions	µg/g	RSD (%)	µg/g	RSD (%)	kg
Chloride	2,250	20.5	3,570	26.0	5,940
Fluoride	5,420	21.7	4,220	18.1	11,600
Nitrate	184,000	37.2	111,000	5.62	375,000
Nitrite	30,600	18.6	43,200	4.23	77,500
Carbonate (TIC)	159,000	24.5	32,200	17.6	285,000
Oxalate	13,600	28.3	5,870	24.9	26,300
Phosphate (ICP)	14,200	21.6	32,100	17.5	43,400
Sulfate	18,400	32.3	18,400	14.9	42,000
Radionuclides	µCi/g	RSD (%)	µCi/g	RSD (%)	Ci
¹³⁷ Cs	60.0	13.2	140	9.01	186,000
⁹⁰ Sr	22.5	24.7	348	18.0	251,000
Total Alpha/ ^{239/240} Pu	0.0192	21.9	0.061	62.8	69.3
Carbon	µg C/g dry	RSD (%)	µg C/g dry	RSD (%)	kg C dry
Total Organic Carbon	5,920	16.3	11,100	14.3	16,700
Physical Properties	g/mL	RSD (%)	g/mL	RSD (%)	
Density	1.44	3.0	1.78	2.2	
	Wt%	RSD (%)	Wt%	RSD (%)	kg
Percent water	23.2	7.72	30.5	7.59	574,100

Note:

¹Inventory values are based on 1.67E+09 grams saltcake and 6.12E+08 grams sludge.

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LIST OF TERMS

1C	first-cycle contamination waste
AES	atomic emission spectrometry
ANOVA	analysis of variance
C	Celsius
Ci	curies
cm	centimeter
cm ³	cubic centimeters
DL	detection limit
DLiq	drainable liquid
DQO	data quality objective
DSC	differential scanning calorimetry
F	Fahrenheit
ft	feet
g	gram
HDW	Hanford Defined Waste
HTCE	Historical Tank Content Estimate
IC	ion chromatography
ICP	inductively coupled plasma
in.	inches
ITS	in-tank solidification
J	joule
kg	kilogram
kgal	kilogallon
kL	kiloliter
kW	kilowatt
L	liter
LFL	lower flammability limit
m	meter
mg	milligram
mL	milliliter
mR/hr	milliroentgens per hour
ORNL	Oak Ridge National Laboratory
PFeCN1	type 1 in-plant ferrocyanide
PFeCN2	type 2 in-plant ferrocyanide
ppm	parts per million
ppmv	parts per million by volume
REML	restricted estimated maximum likelihood
RPD	relative percent difference (the absolute value of the difference between two numbers divided by their mean)
RSD	relative standard deviation
SAP	sampling and analysis plan
TIC	total inorganic carbon

LIST OF TERMS (Continued)

TGA	thermogravimetric analysis
TLM	Tank Layer Model
TOC	total organic carbon
W	watts
WSTRS	Waste Status and Transaction Record Summary
wt%	weight percent
μ	micro
μ Ci	microcurie
μ eq	microequivalent
μ g	microgram
μ g/g	microgram per gram

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1.0 INTRODUCTION

This tank characterization report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell tank 241-BY-110. The tank was sampled in 1995 to satisfy the requirements of the following documents: the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994), the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995), the *Test Plan for Samples From Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109* (Meacham 1995), the *Historical Model Evaluation Data Requirements* (Simpson and McCain 1995), and the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994). Tank 241-BY-110 is on the Ferrocyanide Watch List. It also has been identified as potentially containing organic complexants (WHC 1995a). This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996) and the Ferrocyanide Safety Program, Milestone T-22-96-020 (WHC 1995b).

Tank 241-BY-110 has been removed from service and interim stabilized. Interim stabilization was completed in January 1985, and intrusion prevention was completed in October 1989. Consequently, it is unlikely that waste removals or additions will occur until pretreatment and retrieval activities commence. The concentration estimates reported are based on the current sampling data.

1.1 PURPOSE

This report summarizes information about the use and contents of tank 241-BY-110. When possible, this information will be used to assess issues associated with safety, operations, environmental, and process activities.

1.2 SCOPE

Nine rotary and push mode core samples were taken from July to October 1995 to acquire sample material for analysis and to test various operational parameters of the rotary waste sampling system. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 were analyzed in accordance with the requirements of the safety screening, ferrocyanide, organic, historical, and pretreatment data quality objectives (DQOs) and the safety program test plan. The primary safety objectives of the sampling event were to determine whether the ferrocyanide content was low enough to classify the tank as safe (Postma et al. 1994) and whether the organic content was high enough to place the tank on the Organic Watch List. A further objective was to gather additional information and sample for future retrieval and vitrification activities.

The primary analyses of the core sampling event included the following: differential scanning calorimetry (DSC) to evaluate fuel level and energetics, thermogravimetric analysis (TGA) to determine moisture content, total alpha activity analysis to evaluate criticality potential, ion chromatography (IC) to determine anion concentrations, inductively coupled plasma/atomic emission spectrometry (ICP/AES) to determine metal concentrations, specific assays to determine total cyanide concentration, gamma energy analysis to obtain radiochemical activities, persulfate oxidation to determine the total organic carbon (TOC) concentration, and gas chromatography to determine organic species and concentrations.

Nine core samples were taken during this sampling event to test the rotary mode core sampling system capabilities (in push and rotary mode) and procedures. Although analysis of only four cores was required to satisfy the applicable DQOs and the test plan, all nine cores were analyzed as an opportunistic venture.

In 1994, a full vapor characterization of the tank headspace gases was performed. The data from this sampling and analysis event is in the *Tank 241-BY-110 Headspace Gas and Vapor Characterization Results for Samples Collected in November 1994* (Huckaby and Bratzel 1995). These samples were taken and analyzed for organic vapors and inorganic gases to resolve outstanding tank safety and industrial health concerns.

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-BY-110 based on historical information. It details the current documented condition of the tank. Information on the tank's design and transfer history, and the process sources that contributed to the tank waste are included. An estimate of the current contents based on the process history is also provided. The section describes events that may be related to tank safety issues, such as potentially hazardous tank contents or off-normal operating temperatures, and it summarizes available surveillance data for the tank. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the solid layers of the tank. Temperature data are provided to evaluate the heat generating characteristics of the waste.

2.1 TANK STATUS

As of February 29, 1996, tank 241-BY-110 contained an estimated 1,507 kL (398 kgal) of waste classified as noncomplexed (Hanlon 1996). The liquid waste volume was estimated using a manual tape. The solid waste volume, last updated on September 10, 1979, was estimated using a sludge-level measurement device. Table 2-1 shows the estimated tank contents.

Table 2-1. Estimated Tank Contents.¹

Waste Form	Estimated Volume	
	Kiloliters	Kilogallons
Total waste	1,507	398
Supernatant liquid	0	0
Sludge ²	390	103
Saltcake ²	1,117	295
Drainable interstitial liquid ³	34	9
Drainable liquid remaining ³	34	9
Pumpable liquid remaining	0	0

Notes:

¹Hanlon (1996)

²For saltcake and sludge phase designation, see Table 4-2.

³In this case, the drainable interstitial liquid and the drainable liquid remaining are the same.

Tank 241-BY-110 is on the Ferrocyanide Watch List. It was declared inactive in 1979 and removed from service. This tank is categorized as sound. Interim stabilization was completed in January 1985 and intrusion prevention was completed in October 1989. The tank is passively ventilated. All monitoring systems were in compliance with documented standards as of February 29, 1996 (Hanlon 1996).

2.2 TANK DESIGN AND BACKGROUND

The 241-BY Tank Farm was constructed from 1948 to 1949 in the 200 East Area of the Hanford Site. It contains 12, type II 100-series tanks. The tanks have a capacity of 2,870 kL (758 kgal), a diameter of 23 m (75 ft), and an operating depth of 7 m (23 ft) (Leach and Stahl 1993). Tank 241-BY-110 began receiving waste in the fourth quarter of 1951 (Agnew et al. 1995). Built according to the second generation design, the 241-BY Tank Farm was designed for nonboiling waste with a maximum fluid temperature of 104 °C (220 °F). A cascade overflow line 7.5 cm (3 in.) in diameter connects tank 241-BY-110, the first tank in a cascade of three tanks. Tanks 241-BY-111 and 241-BY-112 are the other tanks in the cascade (Brevick et al. 1994). An overflow line connects tank 241-BY-110 with tank 241-BX-112. However, transfer records do not indicate that cascading occurred between 241-BX-112 and 241-BY-110 (Agnew 1995). Each tank in the cascade is one foot lower in elevation than the preceding tank. The cascade overflow height is approximately 6.9 m (22.7 ft) from the tank bottom and 38 cm (1.2 ft) below the top of the steel liner.

The tank has a dished bottom with a 1.2 m (4 ft) radius knuckle, and is covered with approximately 2.6 m (8.6 ft) of overburden.

According to drawings and engineering change notices tank 241-BY-110 has 19 risers. The risers range in diameter from 10 cm (4 in.) to 1.1 m (3.5 ft). Table 2-2 lists each riser number and diameter and a description of its use. Figure 2-1 shows the riser configuration. Four risers are available for intrusive tank work: risers 5, 7, 10B, and 12B (Lipnicki 1995). Figure 2-2 shows the tank cross-section with the approximate waste level and a schematic of the tank equipment.

Table 2-2. Tank 241-BY-110 Risers.^{1, 2, 3}

Riser Number	Diameter (in.)	Description and Comments
1	4	Thermocouple tree
2	4	Pit drain, weather covered, plugged
3	4	B-436 liquid observation well
4	4	Manual tape
5	4	Spare, flange
6	12	Saltwell screen and pump
7	12	Flange, below grade
8	12	Thermocouple tree
9	42	Adapter plate, below grade
9A	12	Airlift circulator, cut off, below grade
9B	12	Flange, below grade
10	42	Adapter plate, below grade
10A	12	Thermocouple tree/breather filter
10B	18	Observation port
11	42	Adapter plate, below grade
11A	12	Airlift circulator, cut off, below grade
11B	12	Flange, below grade
12	42	Adapter plate, bench mark, below grade
12B	12	Blind flange & heated vapor probe on 12 in. to 4 in. adapter
Nozzle Number	Diameter (in.)	Description and Comments
N1	3	Spare
N2	3	Spare
N3	3	Spare
N4	3	Spare
N5	3	Inlet
N6	3	Outlet

Notes:

¹Alstad (1993)

²WHC (1976)

³WHC (1986)

Figure 2-1. Riser Configuration for Tank 241-BY-110 (not to scale).

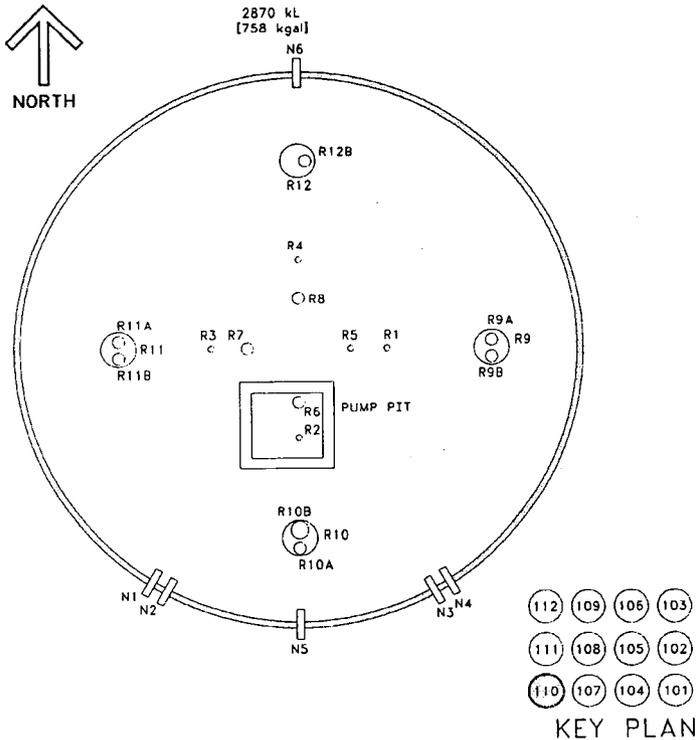
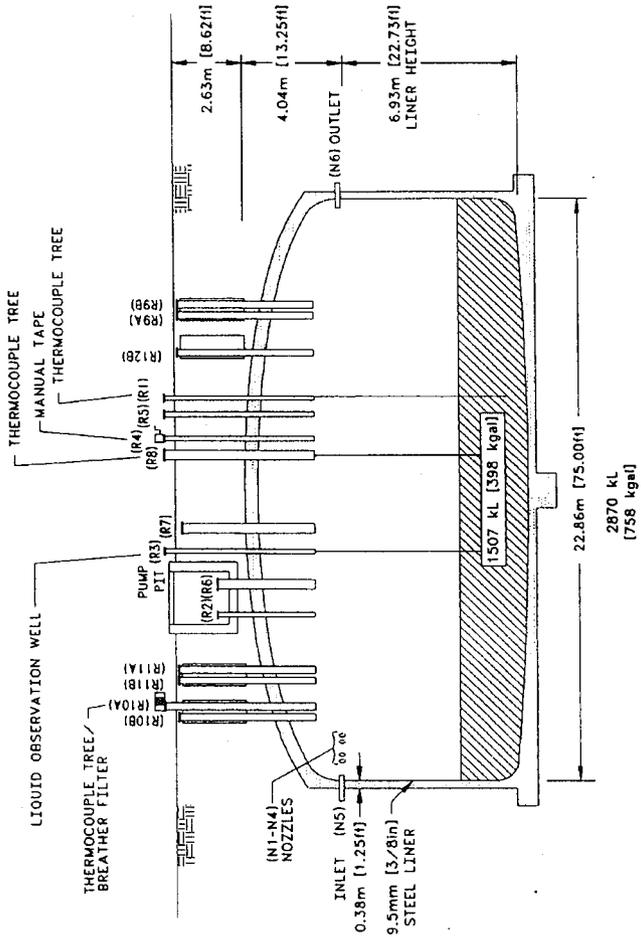


Figure 2-2. Tank 241-BY-110 Configuration (not to scale).



2.3 PROCESS KNOWLEDGE

This section describes the transfer history of tank 241-BY-110 and the process wastes that made up the transfers. This is followed by an estimate of current tank contents based on transfer history. Table 2-3 summarizes the waste transfers to tank 241-BY-110.

2.3.1 Waste Transfer History

Tank 241-BY-110 received first-cycle decontamination waste from the fourth quarter of 1951 to 1952. This waste originated during the bismuth phosphate (BiPO_4) process in the initial purification processing of plutonium. In 1952, the tank received first-cycle decontamination waste from an unknown source; likely from B Plant. In the third quarter of 1954, 95 percent of the waste (2,623 kL [693 kgal]) was transferred to Crib B-038. From the fourth quarter of 1954 to the second quarter of 1957, the tank received in-plant ferrocyanide scavenged waste from the uranium recovery process. Tank 241-BY-110 was a primary settling tank. During this process, ferrocyanide $[\text{Fe}(\text{CN})_6]^{4-}$ and nickel sulfate were added to the uranium recovery waste to precipitate cesium. After settling, the scavenged ferrocyanide supernatant waste, which was relatively free of cesium, was transferred from tank 241-BY-110 to various cribs. The precipitation of cesium and the subsequent disposal of supernatant to the cribs substantially reduced the volume of the stored tank waste. Cladding waste was reportedly added to the tank from tank 241-C-105 during 1957 and 1958. In the third quarter of 1958, cladding waste was transferred into the tank from tank 241-C-106 (Agnew et al. 1995).

From 1969 to 1976, tank 241-BY-110 received in-tank solidification waste from tank 241-BY-112. In 1970, 1974, and 1975, evaporator bottoms waste was added from tanks 241-BY-109, 241-BY-102, and 241-BY-107, respectively. In 1975, waste was sent to tank 241-BX-106. In 1976, tank 241-BY-110 received evaporator bottoms from tank 241-BY-107. Finally, from 1977 to 1979, the tank received evaporator feed waste and noncomplexed waste from tank 241-A-102. At this time waste was sent to tanks 241-BX-105 and 241-AW-102. Two separate additions of 49 kL (13 kgal) of water occurred in 1957 and 1968. Because water is not a considered a waste addition, the corresponding transactions are not shown in Table 2-3. Approximately 1,507 kL (398 kgal) of waste were left in the tank after the final transfer from it in 1982 (Agnew et al. 1995).

2.3.2 Historical Estimation of Tank Contents

The following is an estimate of the contents of tank 241-BY-110 based on historical transfer data. The historical data is from the *Waste Status and Transaction Record Summary (WSTRS) for the Northeast Quadrant* (Agnew et al. 1995) and the *Hanford Tank Chemical and Radionuclide Inventories, HDW Model Rev. 3* (Agnew et al. 1996a).

Table 2-3. Summary of Tank 241-BY-110 Waste Input History.^{1,2}

Transfer Source	Waste Type Received	Time Period	Estimated Waste Volume	
			kiloliters	(kilogallons)
B Plant	First-cycle decontamination	1951 to 1952	2,457	(649)
Unknown	Decontamination	1952	299	(79)
U Plant	In-plant ferrocyanide scavenged from uranium recovery	1954 to 1957	33,580	(8,871)
241-C-105	Cladding	1957 to 1958	1,791	(473)
241-C-106	Cladding	1958	27	(7)
241-BY-112	In-tank solidification	1969 to 1976	1,813	(479)
241-BY-109	Evaporator bottoms (supernate)	1970	2,347	(620)
241-BY-102	Evaporator bottoms (supernate)	1974	45	(12)
241-BY-107	Evaporator bottoms (supernate)	1975	375	(99)
241-A-102	Evaporator feed/noncomplexed	1977 to 1979	416	(110)

Notes:

¹Agnew et al. (1995b)

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

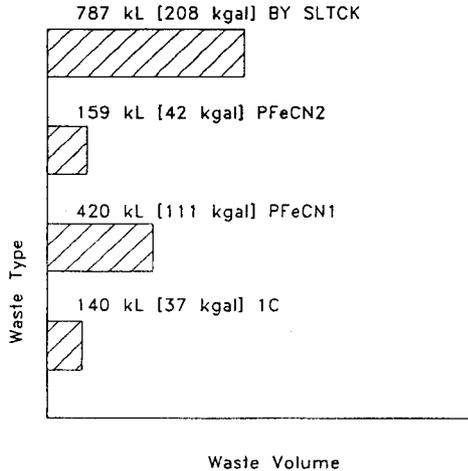
The waste status and transaction record summary (WSTRS) is a compilation of available waste transfer and volume status data. The Hanford Defined Wastes (HDW), a subsection of Agnew et al. (1996a), lists the assumed typical compositions for 50 separate wastes types.

The available data from the above documents are incomplete. This reduces the reliability of the transfer data and the modeling results derived from it. The Tank Layer Model (TLM), a separate section of Agnew et al. (1996a), uses theWSTRS data, models the waste deposition processes and, using additional data from the HDW, generates an estimate of the tank contents. Several errors are introduced as the model elements are combined to create the estimates; therefore, these model predictions can only be considered estimates that require further evaluation using analytical data.

The *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area* (Brevick et al. 1995) states that tank 241-BY-110 contains 140 kL (37 gal) of

first-cycle decontamination (1C) waste, 420 kL (111 kgal) of type 1 in-plant ferrocyanide (PFeCN1) waste, 159 kL (42 kgal) of type 2 in-plant ferrocyanide (PFeCN2) waste, and 787 kL (208 kgal) of BY saltcake waste. The PFeCN1 and PFeCN2 waste used 0.005 M and 0.0025 M ferrocyanide, respectively, to precipitate ¹³⁷Cs. Figure 2-3 is a graph of the estimated waste types and volumes for the tank layers.

Figure 2-3. Tank Layer Model for Tank 241-BY-110.



The 1C waste was the first reported waste added to the tank. It is predicted by Agnew et al. (1996a) to contain very large amounts (greater than three weight percent) of sodium, hydroxide, and phosphate; large amounts (between one and three weight percent) of iron, bismuth, aluminum, and nitrate; and smaller amounts (less than 0.5 weight percent) of calcium, silicate, sulfate, and carbonate. Trace amounts of ¹³⁷Cs and ⁹⁰Sr give this waste a very modest activity.

The ferrocyanide scavenging (PFeCN1 and PFeCN2) wastes were the next waste types added to the tank. These waste types are predicted to be similar in composition. They contain a very large amount of sodium and nitrate and large quantities of iron, bismuth, uranium, sulfate, phosphate, calcium, nitrite, carbonate, and fluoride, which are typical components for early plutonium separation wastes. Unique species to these wastes include ferrocyanide (or ferrocyanide degradation products), a significant quantity of nickel, and elevated levels of ¹³⁷Cs and ⁹⁰Sr. Because of the nature of the scavenging process (deliberate precipitation and concentration of radionuclides), this waste may contain substantially higher concentrations of ¹³⁷Cs and ⁹⁰Sr than predicted by Agnew et al. (1996a) and possess a high activity.

The last waste added was BY saltcake. This waste is predicted by Agnew et al. (1996a) to contain very large quantities of sodium and nitrate and large quantities of aluminum, nitrite, and sulfate. Moderate to small quantities of potassium, iron, chromium, zirconium, lead, and bismuth may also be present. Because of the nature of this waste type (evaporation/concentration of the supernatant), the soluble (sodium) forms of these species are expected to be in the majority. The waste should be water soluble, unlike the 1C and PFeCN wastes. Cesium-137 should be found in significant quantities. BY saltcake waste can be further distinguished from the 1C and PFeCN wastes because BY saltcake waste contains aluminum, lead, and chromium (all not predicted in the PFeCN wastes) and does not contain substantial nickel or iron (found in the PFeCN wastes). Physically, the wastes should be quite distinct, and visual observation should provide cues for distinguishing one from another. Table 2-4 shows an historical estimate of the expected waste constituents and their concentrations.

2.4 SURVEILLANCE DATA

Tank 241-BY-110 surveillance consists of surface level measurements (liquid and solid) and temperature monitoring (waste and headspace). These data provide the basis for determining tank integrity.

Liquid level measurements may indicate if there is a major leak from a tank. Solid surface level measurements may indicate physical changes (such as gas accumulation and retention) and solid layers' consistency. Tank 241-BY-110 has one liquid observation well in riser 3 to measure interstitial liquid levels and has drywells around the tank perimeter to monitor for increased radiation caused by leaks.

2.4.1 Surface Level

The surface level of the waste is monitored with a manual tape through riser 4. The allowable deviation from the tank's baseline of 3.85 m (12.64 ft) is a 7.5 cm (3 in.) increase; no limitation is established for level decreases (Boyles 1992). Because of faulty equipment, all readings since April 2, 1995 have been suspect. Figure 2-4 shows the level history of the tank.

2.4.2 Drywells

Tank 241-BY-110 has four drywells. None have had readings higher than the 50 counts per second background radiation.

Table 2-4. Tank 241-BY-110 Historical Inventory Estimate^{1,2,3}. (2 sheets)

Solids Composite Inventory Estimate			
Physical Properties			
Total solid waste	2.24E+06 kg	(398 kgal)	
Heat load	1.55 kW	(5.30E+03 BTU/hr)	
Bulk density	1.49 (g/cm ³)		
Void fraction	0.839		
Water wt%	48.2		
Total organic carbon wt% carbon (wet)	0.479		
Chemical Constituents	Mole/L	ppm	kg
Na ¹⁺	8.32	1.29E+05	2.88E+05
Al ³⁺	1.2	21,800	48,800
Fe ³⁺ (total Fe)	0.375	14,100	31,600
Cr ³⁺	2.8E-02	981	2,200
Bi ³⁺	0.104	14,600	32,600
La ³⁺	1.69E-06	0.158	0.353
Ce ³⁺	0	0	0
Zr (as ZrO(OH) ₂)	8.07E-04	49.5	111
Pb ²⁺	2.96E-03	413	925
Ni ²⁺	5.90E-02	2,330	5,220
Sr ²⁺	1.88E-06	0.111	0.248
Mn ⁴⁺	1.70E-03	62.8	141
Ca ²⁺	0.148	4,000	8,950
K ¹⁺	2.64E-02	695	1,560
OH ⁻	6.28	71,900	1.61E+05
NO ₃ ⁻	4.33	1.81E+05	4.04E+05
NO ₂ ⁻	0.994	30,800	68,900
CO ₃ ²⁻	0.372	15,000	33,600
PO ₄ ³⁻	0.263	16,800	37,700
SO ₄ ²⁻	0.170	11,000	24,700
Si (as SiO ₃ ²⁻)	6.95E-02	1,310	2,940

Table 2-4. Tank 241-BY-110 Historical Inventory Estimate^{1,2,3}. (2 sheets)

Chemical Constituents (Cont'd)	Mole/L	ppm	kg
F ⁻	0.124	1,590	3,560
Cl ⁻	9.74E-02	2,320	5,200
C ₆ H ₅ O ₇ ³⁻	1.25E-02	1,590	3,570
EDTA ⁴⁻	2.81E-03	545	1,220
HEDTA ³⁻	3.80E-04	70.1	157
NTA ³⁻	0	0	0
glycolate	8.83E-03	446	998
acetate	1.67E-02	665	1,490
oxalate	1.44E-06	0.0855	0.191
DBP	1.33E-02	2,380	5,340
NPH	0	0	0
CCl ₄	0	0	0
hexone	0	0	0
Fe(CN) ₆ ⁴⁻	4.59E-02	8,360	18,700
Radiological Constituents (Decayed to January 1, 1994)			
Pu		6.27E-02 (μCi/g)	2.34 (kg)
U	5.63E-02 (M)	643 (μg/g)	2.02E+04 (kg)
Cs	0.121 (Ci/L)	81.2 (μCi/g)	1.82E+05 (Ci)
Sr	6.91E-02 (Ci/L)	46.5 (μCi/g)	1.04E+04 (Ci)

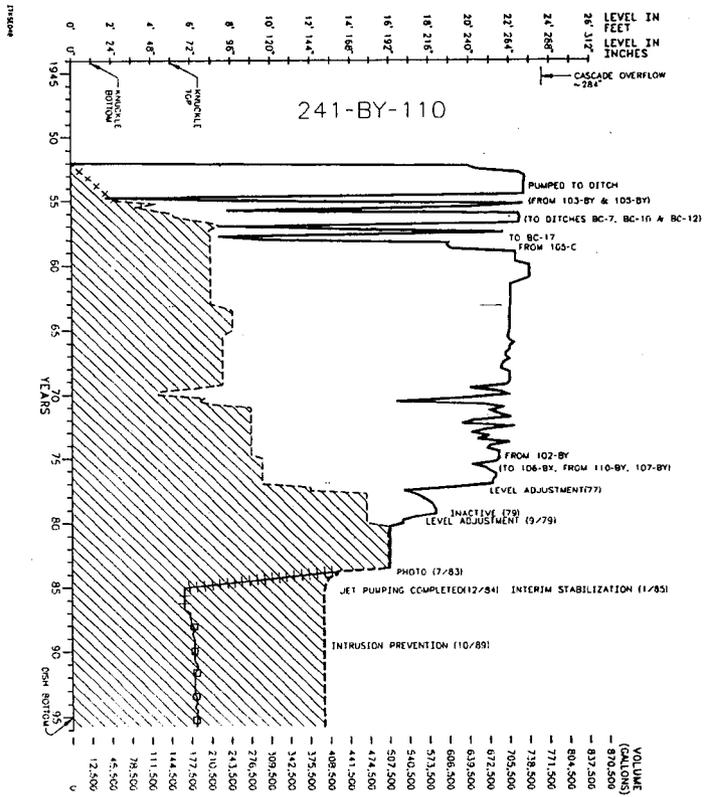
Notes:

¹Agnew et al. (1996a)

²The HDW predictions have not been validated and should be used with caution.

³Small differences appear to exist among the inventory above and the inventories calculated from the two sets of concentration estimates in the table. These differences are being evaluated.

Figure 2-4. Tank 241-BY-110 Level History.



2.4.3 Internal Tank Temperatures

Currently tank 241-BY-110 has two active thermocouple trees, each with six thermocouples to monitor waste temperature. The thermocouple trees, located in risers 1 and 10A, monitor the waste temperature. Elevations are available for the thermocouples. Table 2-5 shows selected temperature data.

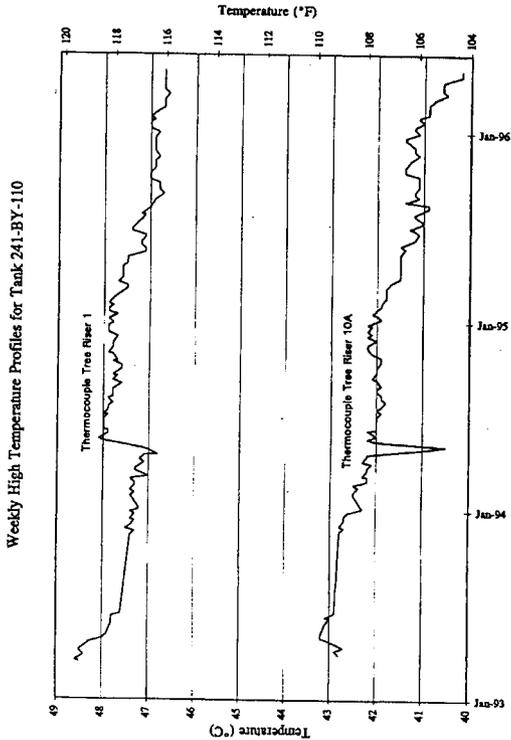
Table 2-5. Tank 241-BY-110 Selected Temperature Data.

Past Temperature Data (1975 to 1996)			
Riser/Thermocouple	Mean (°C)	Maximum (°C)	Minimum (°C)
Riser 1, Thermocouple 6	40.6	62.0	21.0
Riser 10A, Thermocouple 6	33.0	43.2	20.3
Recent Temperature Data (May 8, 1996)			
Riser 1, Thermocouple (TC)		46.7 (TC1)	22.3 (TC6)
Riser 10A, Thermocouple (TC)		40.2 (TC1)	22.7 (TC6)

Although historical temperature data are available between August 1974 and March 1994, they are not assigned to one of the two currently active thermocouple trees. Temperature data are identified for the thermocouple tree in riser 1 for February 1, 1975; December 3, 1986; and from March 1993 to May 1996. The mean temperature for the data is 40.6 °C (105.1 °F) with a minimum of 21 °C (69.8 °F) and a maximum of 62 °C (144 °F). Temperature data exist for the thermocouple tree in riser 10A from March 1993 to May 1996. The mean temperature for this data is 33 °C (91 °F) with a minimum of 20.3 °C (68.5 °F) and a maximum of 43.2 °C (109.76 °F).

Figure 2-5 shows a graph of the weekly high temperature data from the two thermocouple trees from March 1993 to May 1996. Tank 241-BY-110 is monitored weekly because it is on the Ferrocyanide Watch List. On May 8, 1996, the minimum temperatures recorded were 22.3 °C (72.14 °F) from riser 1 on thermocouple 6 and 22.7 °C (72.86 °F) from riser 10A on thermocouple 6. The maximum temperature recordings were 46.7 °C (116.06 °F) from riser 1 on thermocouple 1 and 40.2 °C (104.36 °F) from riser 10A on thermocouple 1. Plots of the individual thermocouple readings are in the HTCE supporting document for the BY Tank Farm (Brevick et al. 1994). The consistently observed difference between the temperature measurements (approximately 8.3 °C [15 °F]) obtained from the risers is probably attributable to heat transfer boundary effects. Riser 1 is at the center of the tank, and riser 10A is located nearer to the tank wall.

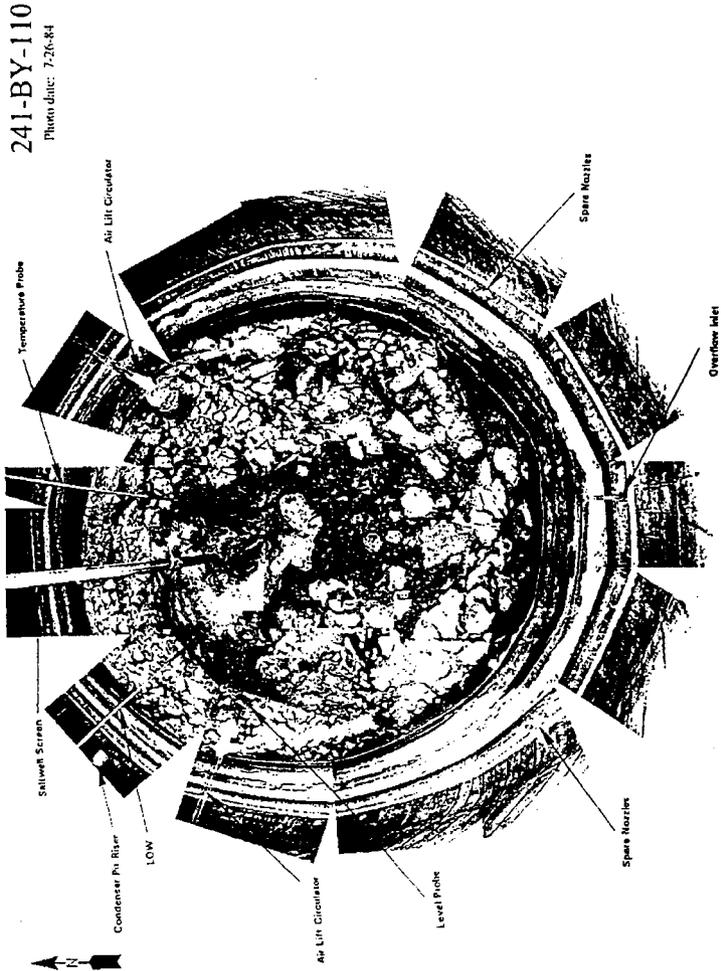
Figure 2-5. Tank 241-BY-110 Weekly High Temperature Plot.



2.4.4 Tank 241-BY-110 Photograph

Figure 2-6 shows a 1984 photographic montage of the tank interior. It indicates that white chunks of waste are distributed throughout a green matrix. At the time of the photograph, the tank contained approximately 1,514 kL (400 kgal) of waste which equals approximately 3.89 m (12.75 ft) of depth. The photograph should adequately represent the current tank interior.

Figure
2-6. Photographic Montage of Tank 241-BY-110.



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3.0 TANK SAMPLING OVERVIEW

This section describes the July through October 1995 sampling and analysis event for tank 241-BY-110. Rotary mode and push mode core samples were taken to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994), the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995), the *Historical Model Evaluation Data Requirements* (Simpson and McCain 1995), the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994), and the *Test Plan for Samples from Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-106, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109* (Meacham 1995). The sampling and analyses were performed in accordance with the *Tank 241-BY-110 Rotary Mode Core Sampling and Analysis Plan* (Schreiber 1996a). This section also describes the 1994 vapor sampling event and a 1991 gamma and neutron scan taken through the liquid observation well. For further discussions of the sampling and analysis procedures, refer to the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

3.1 DESCRIPTION OF THE 1995 CORE SAMPLING EVENT

Nine core samples were collected from tank 241-BY-110 between July 11 and October 28, 1995. Push and rotary mode were used in taking samples. Cores 92, 95, 96, and 109 were collected from riser 12B; cores 101, 103, 106, and 107 from riser 7; and core 113 from riser 4. Between seven and nine segments per core were anticipated. The cores were sent to the Westinghouse Hanford Company 222-S Laboratory for analysis. Table 3-1 summarizes the applicable DQOs and their respective sampling and analysis requirements.

Nitrogen gas was used to maintain hydrostatic head pressure during sampler changeout. Water was used to wash the drill string between core sampling operations. A tracer (lithium bromide) was added to the wash water to gauge contamination of the segments by the wash water. A field blank obtained during the sampling operation and a lithium bromide wash water blank were sent to the 222-S Laboratory for analysis. Table 3-2 summarizes segment numbering prior to subsampling and dose rate information for the 1995 core sampling event.

3.2 1995 CORE SAMPLE HANDLING

Nine cores were received by the Westinghouse Hanford Company 222-S Laboratory between July 12 and October 30, 1995, and were extruded between July 13 and November 6, 1995. All subsamples were homogenized with a spatula prior to analysis by the 222-S Laboratory.

Table 3-1. Integrated Requirements for Tank 241-BY-110.¹

Sampling Event	Sampling Requirements	Applicable References and Analytical Requirements
Cores 92, 95, 96, 101, 103, 106, 107, 109 and 113		
Core sampling	Core samples from a minimum of two risers separated radially to the maximum extent possible.	<p><i>Tank Safety Screening Data Quality Objective:</i> moisture content, total alpha activity, energetics, density, separable organic layer.</p> <p><i>Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objective Process:</i> moisture content, energetics, total organic carbon, cyanide, nickel.</p> <p><i>Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue:</i> total organic carbon, moisture content, energetics.</p> <p><i>Historical Model Evaluation Requirements:</i> energetics, moisture content, ICP, IC, ⁹⁰Sr, gamma energy analysis (¹³⁷Cs), total alpha activity, density.</p> <p><i>Interim Data Quality Objectives for Waste Pretreatment and Vitrification:</i> collection of sample for process development work.</p> <p><i>Test Plan for Samples from Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-106, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109:</i> energetics, gas chromatography.</p> <p><i>Tank 241-BY-110 Rotary Mode Core Sampling and Analysis Plan:</i> lithium, bromide.</p>

Note:

¹Schreiber (1996a)

Table 3-2. Rotary Mode Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Sample Information. (3 sheets)

Core/Segment	Sample Number	Segment Labcore Number ¹	Dose Rate through Drill String (mR/hr) ²
Riser 12B			
92/1	95-103	S95T001250	1
92/2	95-104	S95T001251	< 1
92/3	95-105	S95T001253	< 0.5
92/4	95-106	S95T001254	100
92/5	95-107	S95T001255	130
92/6	95-108	S95T001256	1,000
92/7	95-109	S95T001257	1,500
95/1	95-114	S95T001315	10
95/2	95-115	S95T001316	1
95/3	95-116	S95T001317	150
95/4	95-117	S95T001384	60
95/5	95-118	S95T001385	50
95/6	95-119	S95T001386	60
95/7	95-120	S95T001387	200
109/1R	95-205R	S95T002398	2
109/1AR	95-205AR	S95T002397	2
109/2	95-206	S95T002399	< 0.5
109/2A	95-206A	S95T002401	2
109/3	95-207	S95T002402	800
109/4	95-208	S95T002439	150
96/1	95-121	S95T002581	70
96/2	95-122	S95T002582	110
96/3	95-123	S95T002583	500
96/4	95-124	S95T002919	80
Riser 7			
101/1	95-142	S95T001447	2
101/2	95-143	S95T001448	10
101/3	95-144	S95T001449	16
101/4	95-145	S95T001452	5

Table 3-2. Rotary Mode Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Sample Information. (3 sheets)

Core/Segment	Sample Number	Segment Labcore Number ¹	Dose Rate through Drill String (mR/hr) ²
Riser 7 (Cont'd)			
101/5	95-146	S95T001453	150
101/6	95-147	S95T001454	200
101/6A	95-147A	S95T001864	450
101/7	95-148	S95T001865	1,000
101/8	95-149	S95T001866	1,500
101/9	95-150	S95T001867	1,500
103/1	95-157	S95T001514	45
103/2	95-158	S95T001515	60
103/3	95-159	S95T001516	80
103/4	95-160	S95T001517	150
103/5	95-161	S95T001518	nr
103/6	95-162	S95T001519	1,300
103/7	95-163	S95T001521	1,500
103/8	95-164	S95T001522	1,500
103/9	95-165	S95T001520	1,500
106/1	95-179	S95T001590	40
106/1A	95-179A	S95T001591	15
106/2	95-180	S95T001592	3
107/1	95-188	S95T001593	6
107/1A	95-188A	S95T001594	25
107/2	95-189	S95T001595	45
107/2A	95-189A	S95T001839	45
107/3	95-190	S95T001840	90
107/4	95-191	S95T001841	120
107/5	95-192	S95T001842	480
107/6	95-193	S95T001843	800
107/7	95-194	S95T001844	1,000
107/8	95-195	S95T001845	1,000
107/9	95-196	S95T001846	1,400

Table 3-2. Rotary Mode Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Sample Information. (3 sheets)

Core/Segment	Sample Number	Segment Labcore Number ¹	Dose Rate through Drill String (mR/hr) ²
Riser 4			
113/1	95-239	S95T003149	47
113/2	95-240	S95T003150	10
113/3	95-241	S95T003151	100
113/4	95-242	S95T003138	120
113/4A	95-242A	S95T003141	1,000
113/5	95-243	S95T003144	1,400
113/5A	95-243A	S95T003145	900
113/6	95-244	S95T003146	1,500
113/7	95-245	S95T003128	1,000
113/8	95-260	S95T003129	1,000

Notes:

nr = not recorded

¹Segment Labcore number is parent. Individual sample aliquot numbers for specific assays are traceable from parent number.

²mR/hr = milliroentgens per hour

Each segment's subdivision depended on the waste matrix present: saltcake was subdivided at the half-segment level, and sludge was subdivided at the quarter-segment level. Sample recovery of the bottom portion of the tank (predominately sludge material) was generally good (≥ 90 percent). Recovery of the saltcake material at the top of the tank varied between 0 percent to 80 percent. For solid material, half segments were identified as upper or lower halves, and quarter segments were identified by the letters A, B, C, or D. Drainable liquids were identified as such. Quarter segment identifiers A, B, C, and D denote the position of the quarter segment in the core sampler ("A" denotes the top of the sampler and "D" denotes the bottom).

Solid core composite samples were made from saltcake and sludge material. Three sludge composites and five saltcake composites were formed.

Table 3-3 describes cores 92, 95, 96, 101, 103, 106, 107, 109, and 113. It lists, for each riser, the segment number, subsamples, total liquid and solid weights, and a description of each segment. The identification of material as "saltcake" or "sludge" was based on visual cues at the time of extrusion.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 92 - Riser 12B				
1	n/a	0	0	No solid or liquid sample material observed or collected.
2	n/a	0	0	No solid or liquid sample material observed or collected.
3	n/a	0	0	No solid or liquid sample material observed or collected.
4	WS	35.5	0	Extruded approximately 2 in. of grayish black, crystalline solids that crumbled out of the sampler onto the sample tray. Material was subsampled into jar #7085.
5	WS, LL	111.1	17.2	No drainable liquid was observed or collected, but liner liquid was recovered and placed in jar #7088. Extruded approximately 5 in. of grayish yellow, crystalline solids which crumbled out of the sampler onto the sample tray. Solid material was subsampled into jar #7089. It was noted that the sampler valve was partially open before and after cutting cables.
6	UH, LH, DLiq	230.5	129.1	Drainable liquid was turbid and yellow green in color and was subsampled into jar #7090. Extruded approximately 9 in. of yellow, crystalline solids. Upper half solids (6 in) resembled crushed ice and were subsampled into jar #7352. Lower half solids (3 in.) looked like shaved ice and were placed into jar #7091.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 92 - Riser 12B (Cont'd)				
7	WS, DLiq	137.9	320.7	Drainable liquid was turbid and yellow green in color, and was subsampled into jar #7353. Also extruded slurry material (considered solids for analysis purposes), which was yellow green in color and was comprised of very fine crystals. Slurry was subsampled into jar #7092.
Core 95 - Riser 12B				
1	WS	8.8	0	Extruded less than 1 in. of solid material which was subsampled into jar #7371. Sample was dry, crumbly saltcake and was a mixture of beige and black in color.
2	n/a	0	0	No solid or liquid sample material observed or collected.
3	DLiq	0	8.7	Drainable liquid collected into jar #7337 was opaque and yellow in color. No solids were extruded, and piston appeared clean when pulled from the sampler.
4	WS	62.7	0	Extruded approximately 2.5 in. of brown solid material. Sample was crystalline, crumbly, and very hard, and was subsampled into jar #7479.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 95 - Riser 12B (Cont'd)				
5	UH, LH	236.4	0	Extruded approximately 10 in. of crystalline, dry, crumbly, brownish-gray solids using the hydraulic extruder because the push mode extruder could only push the piston half way through the sampler (noted that sampler valve was open before and after cutting cables). Material was subsampled as half segments into jar #7480 (lower half) and jar #7481 (upper half).
6	WS	72.3	0	Extruded approximately 4 in. of solid material into jar #7484. Sample was crystalline, dry, and light yellow to white in color.
7	WS	44.8	0	Extruded 3 in. of solid material into jar #7380. Sample was crystalline, wet, and light yellow in color with brown dirt-like material mixed throughout.
Core 96 - Riser 12B				
1	WS	76.0	0	Extruded approximately 3 in. of yellow to brown solids which were collected into jar #7739. Material was dry, crumbly, and resembled saltcake.
2	WS	193.4	0	Extruded approximately 10 in. of crystalline saltcake solids. The sample was moist, did not retain the shape of the sampler, and was yellow to brown in color. This material was collected into jar #7955.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 96 - Riser 12B (Cont'd)				
3	Q/A, Q/B	208.6	0	Extruded approximately 10 in. of material. Upper 6 in. was light yellow to medium brown in color and was dry and crumbly resembling saltcake. This material was identified as quarter-segment A and was collected into jar #7742. The lower 4 in. of sample appeared to be a very moist mixture of sludge and saltcake; it was dark brown in color. This material was designated as quarter-segment B and was collected into jar #7740.
4	n/a	0	0	No solid or liquid sample material observed or collected.
Core 109 - Riser 12B				
1	UH	97.9	0	Obtained in two sections (1R and 1AR). Extruded approximately 3 in. of dry, crumbly, yellow and white sand-like particles intermixed with gray to black sand-like material as upper half solids, which was collected into jar #7643. No drainable liquid or solid sample was observed or collected from the second section. These sections are labeled R because they are a resample of this segment. The first attempt yielded no sample, and the samplers were reused.
2	n/a	0	0	No solid or liquid sample material observed or collected in either section.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 109 - Riser 12B (Cont'd)				
3	WS, DLiq	56.6	37.8	Extruded both solids and liquids. About 20 to 25 mLs of greenish-brown drainable liquid was collected into jar #7732, and 2 to 3 in. of solids were also obtained. The solids appeared grayish-green in color, did not retain the shape of the sampler, and had the consistency of a sludge/saltcake mixture. The solids were also damp and grainy, and were collected into jar #7733.
4	n/a	0	0	The piston was lodged at the valve head, preventing extrusion. A small puddle of dark brown liquid near the bottom end of the extrusion tray with a few drops of liquid along the length of the extrusion tray were observed, but this was insufficient sample to recover for analysis.
Core 101 - Riser 7				
1	n/a	0	0	No solid or liquid sample material observed or collected.
2	WS	16.3	0	Extruded approximately 2 in. of light brown, dry, crystalline solid material. Texture of material was hard and resembled saltcake. Solids were subsampled into jar #7508.
3	WS	39.6	0	Extruded approximately 2 in. of tan, dry, powdery solid material that resembled sand. Four large, hard chunks of material (about 0.5 in. in diameter) were recovered as well. All material was subsampled into jar #7509. It was also noted that the sampler valve was halfway open before and after cutting cables.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 101 - Riser 7 (Cont'd)				
4	WS, DL	1.8	24.8	Drainable liquid was brown and opaque and was collected into jar #7427. Extruded approximately 2 in. of dark brown, relatively hard, granular, saltcake material which was subsampled into jar #7339.
5	WS	115.8	0	Sample was observed on the outside of the sampler near the valve housing because the valve was about halfway open before and after cutting cables. Extruded approximately 4 in. of dry, olive green/brown sludge which was subsampled into jar #7510. During the extrusion process, the piston got lodged in the valve housing.
6	UH, LH	146.4	0	Obtained in two sections (6 and 6A). Extruded a small amount of wet, brown, saltcake material as upper half solids, which was subsampled into jar #7452. During the extrusion process for this section, the piston got lodged in the valve housing. Extruded approximately 4 in. of damp, smooth, grainy, dark brown sludge from section 6A. This lower half solids material retained its shape, although the sample broke into 1.5 to 2 in. sections during extrusion. The lower half solids were subsampled into jar #7632.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 101 - Riser 7 (Cont'd)				
7	Q/A, Q/B, Q/C, Q/D, WS	437.8	0	Extruded approximately 18 in. of dark gray/brown, grainy sludge, which broke into sections upon extrusion but retained their shape. Slurry material with small, discrete, pieces of solid dispensed throughout was also recovered. Slurry material was subsampled separately into jar #7728, and the sludge was subdivided into quarter-segments jars. A through D (jars 7726, 7722, 7721, and 7619, respectively).
8	Q/A, Q/B, Q/C, Q/D	419.5	0	Extruded approximately 18.5 in. of sludge material which ranged from a gray/black sludge and slurry mixture (upper 11 in.) to a medium dark brown sludge (lower 7.5 in.). Solids were subdivided into quarter-segments A through D (jars #7725, 7723, 7617, and 7724, respectively).
9	Q/A, Q/B, Q/C, Q/D	523.2	0	Extruded approximately 18 in. of medium brown sludge, which retained its shape during extrusion. Solids were subdivided into quarter-segments A through D (jars #7618, 7636, 7727, and 7633, respectively).
Core 103 - Riser 7				
1	UH, LH	182.8	0	Extruded approximately 10 in. of light yellow to white, dry, crystalline material. Texture was hard and resembled saltcake. Light yellow material was designated lower half solids and was subsampled into jar #7511; white material was labeled upper half solids and was collected into jar #7485.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109,
and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 103 - Riser 7 (Cont'd)				
2	WS	130.1	0	Extruded approximately 11 in. of light brown, dry, crystalline material which was subsampled into jar #7486. Texture of material was hard and resembled saltcake.
3	WS	92.9	0	Extruded approximately 10 in. of light brown, dry, crystalline material which was subsampled into jar #7487. Texture was hard and resembled saltcake.
4	UH, LH	155.6	0	Extruded approximately 14 in. of light brown, dry, crystalline material. Texture of material was hard and resembled saltcake. Sample was collected into half-segments; the upper half was placed into jar #7488, and the lower half solids were stored into jar #7512.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 103 - Riser 7 (Cont'd)				
5	WS	163.4	0	Extruded approximately 10 in. of brown, crumbly, dry, granular, crystalline material resembling saltcake. Sample was collected into jar #7489.
6	UH, LH	313.3	0	Extruded 16 to 19 in. of gray/black to medium brown material. This color change occurred half way through the segment, so the gray/black material was designated as lower half solids while the brown material was classified as upper half solids. The texture of the material next to the piston (4-5 in.) resembled a brown sludge, but in general the sample appears similar in texture to previous samples from this core, except that the entire sample is wet. The lower half solids were subsampled into jar #7490; the upper half solids were collected into jar #7492.
7	UH, LH, WS	242.9	169.0	Extruded approximately 100 mL of slurry liquid into jar #7513. In addition, 3 to 4 in. of gray/black solids were extruded, followed by a 6 in. gap, followed by 7 in. of gray/black, hard, granular, wet solids. The first 3 to 4 in. were designated as lower half solids and were subsampled into jar #7491; the remaining 7 in. was subsampled as upper half solids into jar #7493.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 103 - Riser 7				
8	Q/A, Q/B, Q/C, Q/D	402.4	0	Extruded 19 in. of wet material. The upper 14 to 15 in. were gray/black, hard, granular solids which resembled the previous segments. The bottom 4 to 5 in. appeared to be dark brown sludge. This bottom facie was collected into jar #7514 as quarter-segment D, and the remaining material was divided into quarter-segments A and B and was subsampled into jars #7495 and 7515, respectively.
9	Q/A, Q/B, Q/C, Q/D	473.8	0	Extruded 19 in. of dark brown sludge, which retained its shape. The surface of the lower half of the segment was smooth; the upper half was less smooth. Sample material was subsampled into quarter-segments A through D and was collected into jars #7519, 7518, 7517, and 7516, respectively.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 106 - Riser 7				
1	UH, LH	107.1	0	Obtained in two sections (1 and 1A). Extruded approximately 4 in. of dry, crumbly, heterogeneous saltcake material as upper half solids, which was subsampled into jar #7623. This material ranged in color from tan to almost white. Extruded approximately 2 in. of dry, light brown saltcake material from section 1A. This lower half solids material was crumbly and ranged from a fine texture to particles 0.25 to 0.5 in. in diameter. The lower half solids were subsampled into jar #7602. The piston did not push out of the sampler, the valve was difficult to open during extrusion of section 1A (lower half solids).
2	WS	28.1	0	Extruded approximately 2 in. of solids which were light to medium brown in color. The solids appeared to be dry, crystalline saltcake, and the size of the particles ranged from fine to 0.5 in. in diameter. All material from this segment was collected into jar #7605.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 107 - Riser 7				
1	UH, LH	41.3	0	Obtained in two sections (1 and 1A). Extruded less than 1 in. of dry, white, crystalline saltcake material as upper half solids, which was collected into jar #7580. Extruded approximately 2 in. of slightly damp, crumbly, light tan to brown saltcake material from section 1A. This lower half solids material was composed of particles which ranged from 1/8 to 3/4 in. in diameter. The lower half solids were subsampled into jar #7603. Prior to the valve opening for section 1A, yellow crystals were noted on the end of the sampler.
2	UH, LH	152.9	0	Obtained in two sections (2 and 2A). Extruded approximately 6 in. of dry, crumbly, crystalline solids as upper half solids, which was collected into jar #7608. The sample appeared light to medium brown in color, although the upper 2 in. appeared slightly more yellow. Extruded approximately 7 in. of fairly dry, crumbly, crystalline solids from section 2A. This material appeared olive green and light to medium brown in color; it was collected into jar #7523.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 107 - Riser 7 (Cont'd)				
3	WS	138.6	0	Extruded approximately 7 in. of fairly dry, crumbly, crystalline saltcake material. The sample appeared medium brown in color; it was collected into jar #7624. During the extrusion process the piston got lodged in the sampler, and the final 1 to 2 in. was extruded using the hydraulic extruder.
4	WS	130.9	0	Extruded approximately 7 in. of fairly dry, crumbly, crystalline saltcake material. The sample appeared medium brown in color; it was collected into jar #7625.
5	Q/A, Q/B	199.6	0	Extruded 10 in. of material. The lower 7 in. were medium brown, damp, and resembled sludge. This material was quarter-segment B; it was collected into jar #7626. The upper 3 in. were yellow brown in color and resembled saltcake. This material was quarter-segment A; it was collected into jar #7615.
6	UH, LH	353.9	0	Extruded approximately 16 in. of material. The lower 11 in. of solids were dark brown in color, damp, and had a texture which resembled damp saltcake. This material was the lower half solids; it was collected into jar #7627. The upper 5 in. of sample appeared to be light brown in color with a saltcake texture. This material was the upper half solids; it was collected into jar #7524.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 107 - Riser 7 (Cont'd)				
7	Q/A, Q/B, Q/D	346.8	0	Extruded 10 in. of solids in slurry. Solids were brown, homogeneous, and consisted of sludge and hard, chunky material. This material was subsampled into quarter-segments A, B, and D; it was collected into jars #7606, 7613, and 7525, respectively.
8	Q/A, Q/B	266.4	0	Extruded 10 to 11 in. of material, including about 8 in. of solids and some slurry material. The solids were dark brown (lower portion) and grayish-brown (upper portion) in color and resembled sludge. The lower half of the solids retained its shape and was subsampled as quarter-segment B into jar #7601. The upper half solids, which were not a dark in color or as smooth, made up as quarter-segment A. Upon subsampling it was noted that the upper portion was a mixture of sludge and hard saltcake. The slurry material was subsampled with the quarter-segment A material into jar #7628.
9	Q/A, Q/B, Q/C, Q/D	460.5	0	Extruded 18 in. of solids. Material was dark brown, homogeneous, retained its shape, and resembled sludge. The surface of the sample was smooth. Upon subsampling, it was found the material was sticky. The solids were subsampled into quarter-segments A through D; they were collected into jars #7634, 7629, 7630, and 7631, respectively.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 113 - Riser 4				
1	WS	60.4	0	Extruded approximately 6 in. of light brown to yellow solids. The material was dry and crumbly and did not retain the shape of the sampler; it was collected into jar #8479.
2	WS	22.6	0	Extruded approximately 2 to 3 in. of yellow solids. The material was hard, dry, and crystalline in texture, it was collected into jar #7758.
3	WS	103.6	0	Extruded approximately 8 in. of yellowish-brown solids. The material was hard, dry, crumbly, and crystalline in texture; it was collected into jar #8477.
4	UH, Q/C, Q/D	205	0	Obtained in two sections (4 and 4A). Extruded approximately 3 in. of dry, crumbly, crystalline material as upper half solids; it was collected into jar #7760. This material was yellowish-brown in color. Second section (lower half) had 2 in. of solids followed by a 7 in. gap, followed by 7 in. of solids. All solids in this section were brown, granular, wet, hard, and crystalline. This section was subsampled into two parts quarter-segments C and D and were collected into jars #7762 and 7765, respectively.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109,
and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 113 - Riser 4 (Cont'd)				
5	Q/A, Q/B, LH	397.5	0	Obtained in two sections (5 and 5A). Extruded approximately 10 in. of solids and slurry in the upper half of the sampler labeled sampler 95-243. The first 7 in. of material quarter-segment B; it was collected into jar #7767. This sample resembled a wet, granular, greenish-black saltcake. The uppermost 3 in. was quarter-segment A; it was collected into jar #7766. This material was a smooth, soft, greenish-black sludge. The second section (95-243A) of segment 5 contained 8 in. of greenish-black, very wet sludge; it was collected into jar #8464 as segment 5, lower half solids.
6	Q/A, Q/B, Q/C, Q/D	333.8	0	Extruded 17 in. of solids. The material was a mixture of sludge, slurry, and saltcake; it was greenish-brown in color. The sample did not retain its shape. This material was subdivided into quarter-segments A through D; it was collected into jars #7755, 7761, 7764, and 8177, respectively.

Table 3-3. Cores 92, 95, 96, 101, 103, 106, 107, 109, and 113 Rotary Sample Description.¹ (17 sheets)

Segment Number	Subsamples	Total Solid Weight (g)	Total Liquid Weight (g)	Segment Description
Core 113 - Riser 4 (Cont'd)				
7	Q/A, Q/B, Q/C, Q/D	413.7	31.4	Extruded solid and liquid material. About 30 grams of dark brown drainable liquid was collected into jar #7683, and 19 in. of solids were observed. This material was brown and resembled sludge although some saltcake was noted at the top and bottom of the segment. The solids were subdivided into quarter-segments A through D; they were collected into jars #7753, 7759, 7754, and 7751, respectively.
8	Q/A, Q/B, Q/C, Q/D	449.2	0	Extruded 19 in. of brown sludge. Solids were firmer in texture than the sludge in segment 7 of this core and retained the shape of the sampler. No saltcake material was observed. The sludge material was subdivided into quarter-segments A through D; it was collected into jars #7756, 7757, 7752, and 7763, respectively.

Notes:

- DLiq = drainable liquid
- LL = liner liquid
- LH = lower half segment
- UH = upper half segment
- Q/A = upper quarter segment
- Q/B = second quarter segment from top of core
- Q/C = third quarter from top of core
- Q/D = bottom quarter segment of core
- WS = whole segment
- n/a = not applicable

¹Schreiber (1995)

3.3 SAMPLE ANALYSIS

The analytical data provided by the analysis of the nine cores were not limited to the required by the safety program test plan and safety screening, ferrocyanide, organic, and historical DQOs. Additional analytical results, such as total inorganic carbon, were obtained on an opportunistic basis in the process of meeting these DQO requirements (Kristofzski 1995).

The analyses of solid waste in tank 241-BY-110 were performed on half-segments and quarter-segments as discussed above. The exceptions were the analyses of segments that recovered insufficient solid sample for subdivision or were slurry samples. In these cases, all material was combined and was identified as a "whole segment." Vapor sampling using a combustible gas analyzer was performed in the field prior to and during sampling. A brief discussion of the analyses follows.

All solid sample analyses were performed by the laboratory on homogenized samples. Weight percent water was determined directly by thermogravimetric analysis (TGA). The fuel content of the waste was determined directly by differential scanning calorimetry (DSC).

Metals were measured on acid or fusion prepared samples using inductively coupled plasma/atomic emission spectroscopy (ICP). Total alpha activity, gamma energy analysis, total beta activity measurement, and analyses for plutonium, strontium, and uranium were performed on fusion-digested samples.

Anions were measured on water-leached samples using ion chromatography (IC). Total inorganic and organic carbon were measured using hot persulfate oxidation and coulometry. The samples were prepared for cyanide analysis using microdistillation and were measured using a colorimetric method. Composite samples were also analyzed by ICP on a water-leached aliquot.

Organic compounds, specifically normal paraffin hydrocarbons and tributyl phosphate, were measured using gas chromatography with flame ionization detection, and mass spectrometry. Visual inspection was used to determine the presence of a separable organic layer. Density was measured using weight and volume measurements. Table 3-4 provides further information regarding the various laboratory procedures used in the analysis of these samples.

Section 4.0 discusses the results of the analyses, and Section 5.0 discusses the results of the quality control tests, the implications for data quality, and data interpretation. For a summary of the cores, segments, segment portions, and individual sample numbers, and the analyses performed on each sample, refer to Schreiber (1995, 1996b).

Table 3-4. Analytical Procedures. (2 sheets)

Analysis	Instrument	Preparation Procedure	Procedure Number
Energetics by DSC	Mettler ¹ Perkin-Elmer ²	not applicable	LA-514-113, Revs. C-0 and C-1 LA-514-114, Revs. C-0 and C-1
Percent water by TGA	Mettler ¹ Perkin-Elmer ²	not applicable	LA-560-112, Rev. B-1 LA-514-114, Revs. C-0 and C-1
Total alpha activity	Alpha proportional counter	LA-549-141, Revs. D-0 and E-0	LA-508-101, Rev. D-2
Total cyanide	Distillation	not applicable	LA-695-102, Rev. D-0 LA-695-103, Rev. A-0
Total organic carbon	Direct persulfate oxidation	not applicable	LA-342-100 Revs. A-0, B-0, and C-0
Metals and cations	Inductively coupled plasma spectrometer (ICP)	LA-549-141 Revs. D-0 and E-0 (fusion digestion) LA-505-158, Rev. A-4 (acid dilution liquids) LA-505-159, Revs. C-0 and D-0 (acid digestion solids) LA-504-101, Revs. D-0 and E-0 (water digestion)	LA-505-151 Rev. D-3 LA-505-161 Rev. B-0
Anions	Ion chromatography (IC)	LA-504-101, Revs. D-0 and E-0	LA-533-105, Rev. D-1
^{239/240} Pu	Separation and alpha counting	LA-549-141 Revs. D-0 and E-0	LA-943-127, Revs. B-0 and B-1
⁹⁰ Sr	Separation and beta counting	LA-549-141 Revs. D-0 and E-0	LA-220-101, Rev. D-1
GEA	High purity germanium-multi channel analysis	LA-549-141 Revs. D-0 and E-0	LA-548-121, Rev. D-1
Organic screen	Flame ionization detector/gas chromatography	not applicable	LA-523-437 Revs. A-0 and B-0

Table 3-4. Analytical Procedures. (2 sheets)

Analysis	Instrument	Preparation Procedure	Procedure Number
Specific gravity	not applicable	not applicable	LA-510-112, Rev. C-3
Bulk density	not applicable	not applicable	LO-160-103, Rev. A-7
Flammability	Combustible gas analyzer ³	not applicable	WHC-IP-0030, (WHC 1992) IH 1.4, IH 2.1

Notes:

¹Mettler is registered trademark of Mettler Electronics, Anaheim, California.

²Perkin-Elmer is a registered trademark of Perkins Research and Manufacturing Company, Inc., Canoga Park, California.

³The safety screening DQO notification limit for flammable gas concentration is 25 percent of the lower flammability limit. The combustible gas meter that sampled the tank headspace reports results as a percent of the lower explosive limit. Because the National Fire Protection Association defines lower flammability limit and lower explosive limit identically, the terms are used interchangeably (NFPA 1995).

3.4 PRIOR SAMPLING EVENTS

No historical records indicating that the tank liquids or solids had been previously sampled and analyzed were available for tank 241-BY-110. Gamma and neutron scans of the waste taken through a liquid observation well are presented in Grigsby et al. (1992). A previous vapor sampling event was documented in Huckaby and Bratzel (1995). Data from that report are summarized in Section 4.3.4 and Appendix C.

3.4.1.1 Description of Sampling Event - Vapor. Tank 241-BY-110 headspace gas and vapor samples were collected and analyzed to help determine the potential risks to tank farm workers caused by fugitive emissions from the tank. The drivers and objectives of waste tank headspace sampling and analysis are discussed in *Program Plan for the Resolution of Tank Vapor Issues* (Osborne and Huckaby 1994). Tank 241-BY-110 was vapor sampled in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution* (Osborne et al. 1994).

Headspace gas and vapor samples were collected from tank 241-BY-110 using the vapor sampling system on November 11, 1994 by the Westinghouse Hanford Company Sampling and Mobile Laboratories (WHC 1995b). Sample collection and analysis were performed as directed by the *Tank 241-BY-110 Tank Characterization Plan* (Carpenter 1994). The tank headspace temperature was determined to be 27 °C. Air from the tank 241-BY-110

headspace was withdrawn through a 7.9 m-long heated sampling probe mounted in riser 12B and transferred through heated tubing to the vapor sampling system sampling manifold. All heated zones of the vapor sampling system were maintained at approximately 50 °C.

3.4.1.2 Sample Handling. Sampling media were prepared and analyzed by Westinghouse Hanford Company, Oak Ridge National Laboratory (ORNL), and Pacific Northwest National Laboratory.

A general description of vapor sampling and sample analysis methods is given by Huckaby et al. (1995). The sampling equipment, sample collection sequence, sorbent trap sample air flow rates and flow times, chain-of-custody information, and a discussion of the sampling event are given in WHC (1995b).

3.4.1.3 Sample Analysis.

Inorganic

Analytical results of sorbent trap and SUMMA¹ canister tank air samples for selected inorganic gases and vapors are given in Appendix C in parts per million by volume (ppmv) in dry air. Inorganic analyte sorbent traps and SUMMATM canisters were prepared and analyzed by Pacific Northwest National Laboratory at quality assurance impact level 2. Claus et al. (1995) describes sample preparation and analyses.

Analyses of the inorganic vapor sorbent traps were performed within 39 days of sample collection, analyses of SUMMATM canisters for inorganic compounds were performed 70 days after sample collection (Ligotke 1995). Thus the 60-day holding time requirement of the WHC quality assurance project plan (Keller 1994) was satisfied for analyses of vapor sampling system sorbent traps, but not for SUMMATM sample analysis.

It should be noted that these inorganic compounds (that is, the permanent gases) are expected to be very stable in the SUMMATM canisters, and the results may not have been affected even though the administratively chosen 60-day holding time requirements were exceeded. No holding time study has been performed to determine the stability of the inorganic analytes in SUMMATM canisters in the chemical matrix of the tank samples.

Organic

Organic vapors in the tank 241-BY-110 headspace were sampled using the vapor sampling system SUMMATM canisters, which were analyzed at Pacific Northwest National Laboratory, and triple sorbent traps, which were analyzed by ORNL. Both laboratories used a gas

¹SUMMA is a trademark of Moletics Inc., Cleveland, Ohio

chromatograph equipped with a mass spectrometer to separate, identify, and quantify the analytes. Descriptions of sample device cleaning, sample preparations, and analyses are given by Jenkins et al. (1995) and Clauss et al. (1995).

SUMMA™ sample results are considered the primary organic vapor data for tank 241-BY-110. These results were produced at Pacific Northwest National Laboratory using quality assurance impact level 2. However, analyses of organic vapors in SUMMA™ canisters were not completed until 76 days after sample collection (Ligotke 1995), and exceeded the administratively chosen 60-day holding time specified by the Westinghouse Hanford Company quality assurance project plan (Keller 1994). No holding time study has been performed to determine the stability of the organic analytes in SUMMA™ canisters in the chemical matrix of the tank air samples.

ORNL analyses of triple sorbent trap samples from this and other waste tanks generally support the SUMMA™ sample results. However, because certain Westinghouse Hanford Company quality assurance requirements were not satisfied by ORNL, the quality assurance assessment of ORNL by Hendrickson (1995) should be reviewed before results unique to the triple sorbent trap samples are used for decision making.

All triple sorbent traps prepared by ORNL had three surrogate compounds added to evaluate sample matrix effects, potential handling, storage, shipment problems, analyte trapping efficiencies and analytical instrumentation performance (Jenkins et al. 1995). ORNL evaluated the surrogate recoveries using a statistical approach similar to that prescribed by *Test Methods for Evaluating Solid Waste*, SW 846 (EPA 1992). Using this approach, ORNL reported that all surrogates had standard deviation values within the 95 percent confidence interval for variance, indicating that no bias was introduced in measurement of analyte quantities (Jenkins 1995).

3.4.2.1 Description of Sampling Event--Neutron and Gamma Scans. Neutron and gross gamma scans were made in 12 ferrocyanide waste tanks to identify interstitial liquid levels. Tank 241-BY-110 was one tank sampled in this manner. Traces of the neutron and gross gamma scans obtained in 1985 and 1991 are in Grigsby et al. (1992). Gross gamma scans are used to identify liquid levels in the waste tanks and to observe movements of ¹³⁷Cs over time. A spectral gamma scanning cadmium-telluride detector was used to measure the concentrations of ¹³⁷Cs, ^{154,155}Eu, and ⁶⁰Co.

3.4.2.2 Sampling Handling. There was no sampling handling in the conventional sense because assays were performed remotely, in-situ, and nondestructively.

3.4.2.3 Sample Analysis. Probes were lowered through the liquid observation well located in riser 3, and data was collected as a function of depth. The signals were processed and analyzed to provide quantitative results and to calculate an inventory. These results are reported in Grigsby et al. (1992). The ¹³⁷Cs inventory estimate corresponds closely with results from contemporary analytical data (see Section 5.2). In general, the neutron and

gamma scans are in agreement regarding the interstitial liquid level. The water content of the waste remained essentially constant in the six years between the two observations. Currently, the data suggest that contemporary water loss over time from the tank is negligible.

4.0 ANALYTICAL RESULTS

This section discusses the analytical results associated with the July to October 1995 sampling of tank 241-BY-110. The sampling and analysis were performed as directed in the *Tank 241-BY-110 Rotary Mode Core Sampling and Analysis Plan (SAP)* (Schreiber 1996a). The SAP integrated all documents related to sampling and analytical requirements including applicable DQOs. The SAP sampling and analytical requirements were taken from the safety screening DQO (Dukelow et al. 1995), the ferrocyanide DQO (Meacham et al. 1994), the historical model evaluation data requirements (Simpson and McCain 1995), the organic DQO (Turner et al. 1995), and the safety program test plan (Meacham 1995). Nine core samples were taken. Analysis of the cores was performed at the Westinghouse Hanford Company 222-S Laboratory.

Table 4-1 lists the locations of the analytical results. Because of the large size of the data set, appendices containing the individual analytical results are not in this report but may be found in Schreiber (1995 and 1996b). Because the solid waste in the tank contains saltcake and sludge, the analyte means are reported as two tables in Section 4.0. A combined tank inventory table is in Section 5.0. The classification of segments was determined by process knowledge, visual cues, and the iron concentration of the subsegment. Qualitative and quantitative analyses were performed on the segregated data and on the entire data set. Appendix B contains data for the analytes (lithium and bromide) evaluated to measure the amount of contamination by the drill string wash water.

Table 4-1. Analytical Data Tables.

Data Type	Location
Sample phase designation	Table 4-2
Chemical data summary	Table 4-3, 4-4
Thermogravimetric analysis results	Table 4-5
Differential scanning calorimetry results	Table 4-6
Density results	Table 4-7
Statistical analysis	Appendix A
Drill string wash water contamination check data	Appendix B
Headspace analytical data	Appendix C

4.1 DATA PRESENTATION

Section 4.2 summarizes chemical data. Section 4.3 summarizes physical data and also includes information about thermogravimetric analyses, differential scanning calorimetry, density, drill string water wash contamination check results, density, and vapor data. Data from the analysis of all cores were reported in Schreiber (1995 and 1996b) and can also be found in the Tank Characterization Database.²

4.2 CHEMICAL DATA SUMMARY

Data from the nine cores were segregated initially by phase (saltcake or sludge), then results from each phase were combined in a statistical model to derive an overall concentration mean and uncertainty for each analyte. Table 4-2 summarizes the assignments made in the statistical model. The assignments may not correspond exactly to the observations made in Table 3-3, because they incorporate additional information.

Table 4-2. Sample Phase Designation.

Core	Saltcake	Sludge
92	All segments	
95	All segments	
96	All segments	
101	Segments 1-8B	8C, 8D, 9A-9D
103	Segments 1-8C	8D, 9A-9D
106	All segments	
107	Segments 1-7D	8A, 8B, 9A-9D
109	All segments	
113	Segments 1-6	7A-7D, 8A-8D

Tables 4-3 and 4-4 list the analytes present in saltcake and in sludge, respectively. For each analyte, the following information is provided: the restricted estimated maximum likelihood (REML) mean from Appendix A, the REML-derived relative standard deviation (RSD) of the mean, and the projected inventory in kilograms. The RSD is defined as the standard deviation of the mean divided by the mean, multiplied by 100. The REML means and uncertainties are derived using analysis of variance (ANOVA) methods (see Section 5.3). For further information about the structure, assumptions, and methodology for calculating the REML means and the RSDs, see Appendix A.

²<http://twins.pnl.gov:8001/refmain.htm>

Table 4-3. Chemical Data Summary for Tank 241-BY-110 Saltcake. (2 sheets)

Analyte	REML Mean Concentration	Relative Standard Deviation (Mean)	Projected Inventory ¹
METALS	µg/g	%	kg
Aluminum	14,100	13.4	23,600
Antimony ²	< DL	n/a	Not calculated
Arsenic ²	< DL	n/a	Not calculated
Barium	< DL	n/a	Not calculated
Beryllium	< DL	n/a	Not calculated
Bismuth	< DL	n/a	Not calculated
Boron	92.3	13.4	154
Cadmium	21.1	9.35	35.2
Calcium	400	17.8	670
Cerium	< DL	n/a	Not calculated
Chromium	2,900	11.4	4,840
Cobalt	<DL	n/a	Not calculated
Copper	< DL	n/a	Not calculated
Iron	924	23.9	1,540
Lanthanum	< DL	n/a	Not calculated
Lead	130	18.0	217
Magnesium	<DL	n/a	Not calculated
Manganese	52.8	19.5	88.2
Molybdenum	< DL	n/a	Not calculated
Neodymium	< DL	n/a	Not calculated
Nickel	193	23.3	322
Phosphorus	4,650	21.6	7,770
Potassium	1,930	10.9	3,220
Samarium	< DL	n/a	Not calculated
Selenium ²	< DL	n/a	Not calculated
Silicon	451	18.8	753
Silver	17.5	18.5	29.2
Sodium	2.37E+05	2.99	3.96E+05
Strontium	58.1	16.0	97.0
Sulfur	5,950	24.5	9,940
Thallium ²	< DL	n/a	Not calculated
Titanium	<DL	n/a	Not calculated
Uranium	697	16.7	1,160
Vanadium	< DL	n/a	Not calculated
Zinc	32.8	13.3	4.8

Table 4-3. Chemical Data Summary for Tank 241-BY-110 Saltcake. (2 sheets)

Analyte	REML Mean Concentration	Relative Standard Deviation (Mean)	Projected Inventory ¹
METALS (Cont'd)	$\mu\text{g/g}$	%	kg
Zirconium	14.4	14.6	24.0
ANIONS	$\mu\text{g/g}$	%	kg
Chloride	2,250	20.5	3,760
Cyanide	16.3	46.8	27.2
Fluoride	5,420	21.7	9,050
Carbonate ³ (total inorganic carbon)	1.59E+05	24.5	2.65E+05
Nitrate	1.84E+05	37.2	3.07E+05
Nitrite	30,600	18.6	51,100
Oxalate	13,600	28.3	22,700
Phosphate (total)	14,200	21.6	23,700
Phosphate (soluble)	11,500	20.9	19,200
Sulfate	18,400	32.3	30,700
RADIONUCLIDES	$\mu\text{Ci/g}$	%	Ci
¹³⁷ Cs	60.0	13.2	1.00E+05
^{239/240} Pu	0.0192	21.9	32.1
⁹⁰ Sr	22.5	24.7	37,600
Total alpha	0.0434	24.9	72.5
CARBON	$\mu\text{g C/g}$	%	kg C
Total organic carbon	5,920	16.3	9,890
ORGANICS	$\mu\text{g/g}$	%	kg
Hexadecane	473	3.65	790
PHYSICAL PROPERTIES			
Percent water	23.2	7.72	3.87E+05

Notes:

n/a = not applicable

<DL = below analytical detection limits

¹Saltcake mass basis = 1.67E+09 g²High < DL values for certain analytes are because of the lower sensitivity for these analytes by ICP/AES. Also, high uranium concentration can cause interference effects on some trace metals.³Carbonate calculated from 31,800 $\mu\text{gC/g}$ (TIC).

Table 4-4. Chemical Data Summary for Tank 241-BY-110 Sludge. (2 sheets)

Analyte	REML Mean Concentration	Relative Standard Deviation (Mean)	Projected Inventory ¹
METALS	µg/g	%	kg
Aluminum	28,300	4.20	17,300
Antimony ²	< DL	n/a	Not calculated
Arsenic ²	< DL	n/a	Not calculated
Barium	< DL	n/a	Not calculated
Beryllium	< DL	n/a	Not calculated
Bismuth	< DL	n/a	Not calculated
Boron	39.8	26.1	24.3
Cadmium	7.40	20.5	45.3
Calcium	14,200	17.4	8,690
Cerium	< DL	n/a	Not calculated
Chromium	2,220	27.0	1,360
Cobalt	< DL	n/a	Not calculated
Copper	< DL	n/a	Not calculated
Iron	20,000	22.7	12,200
Lanthanum	< DL	n/a	Not calculated
Lead	1,880	54.5	1,150
Magnesium	< DL	n/a	Not calculated
Manganese	228	22.9	140
Molybdenum	< DL	n/a	Not calculated
Neodymium	< DL	n/a	Not calculated
Nickel	6,670	20.7	4,080
Phosphorus	10,500	17.5	6,430
Potassium	2,930	8.43	1,790
Samarium	< DL	n/a	Not calculated
Selenium ²	< DL	n/a	Not calculated
Silicon	1,190	18.4	728
Silver	10.2	14.0	6.24
Sodium	1.61E+05	5.47	98,500
Strontium	6,840	31.7	4,190
Sulfur	5,360	13.5	3,280
Thallium ²	< DL	n/a	Not calculated
Titanium	< DL	n/a	Not calculated
Uranium	20,900	22.4	12,800

Table 4-4. Chemical Data Summary for Tank 241-BY-110 Sludge. (2 sheets)

Analyte	REML Mean Concentration	Relative Standard Deviation (Mean)	Projected Inventory ¹
METALS (Cont'd)	µg/g	%	kg
Vanadium	< DL	n/a	Not calculated
Zinc	91.6	21.0	56.1
Zirconium	19.7	14.6	12.0
ANIONS	µg/g	%	kg
Chloride	3,570	26.0	2,180
Cyanide	98.5	57.6	60.2
Fluoride	4,220	18.1	2,580
Carbonate ³ (total inorganic carbon)	32,200	17.6	19,700
Nitrate	1.11E+05	5.62	67,900
Nitrite	43,200	4.23	26,400
Oxalate	5,870	24.9	3,590
Phosphate (total)	32,100	17.5	19,600
Phosphate (soluble)	11,200	29.9	6,850
Sulfate	18,400	14.9	11,300
RADIONUCLIDES	µCi/g	%	Ci
¹³⁷ Cs	140	9.01	85,700
^{239/240} Pu	0.061	62.8	37.3
⁹⁰ Sr	348	18.0	213,000
CARBON	µg C/g	%	kg C
Total organic carbon	11,100	14.3	6,790
ORGANICS	µg/g	%	kg
Hexadecane	581	9.61	356
PHYSICAL PROPERTIES			
Percent water	30.5	7.59	187,000

Notes:

- n/a = not applicable
- < DL = below analytical detection limits

¹Sludge mass basis = 6.12E+08 g.

²High < DL values for certain analytes are because of the lower sensitivity for these analytes by ICP/AES. Also, high uranium concentration can cause interference effects on some trace metals.

³Carbonate calculated from 6,430 µg C/g (TIC).

Analyte inventories were calculated using phase designation information and knowledge about tank geometry to estimate the volume of each phase. When more than half of the subsegments had detected results, the mean was reported as a detected value. When more than half the subsegments were not detected, the mean was reported as a less-than detection limit (< DL) value. Tables 4-3 and 4-4 show these overall means. The original subsegment analytical data used to calculate those means are in Schreiber (1995 and 1996b). The drainable liquid results were combined with the saltcake results because the waste composition of the liquid more closely resembles the saltcake on a physical and chemical basis.

The projected inventories were derived by multiplying the mean ($\mu\text{g/g}$ or $\mu\text{Ci/g}$) by the estimated waste phase volumes (1,162 kL [307.1 kgals] saltcake and 344 kL [90.9 kgals] sludge) and their respective densities. These volumes were estimated from sampling observations. Although they differ slightly from the Hanlon (1996) estimates, the agreement is close (approximately 4 percent for the saltcake volume, and 16 percent for the sludge volume).

To maintain consistency in the calculation, the sampling-derived volume estimates were used with analytical data. Density measurements were available as a function of depth for most cores, and a mean was determined for each phase. The average saltcake density was 1.44 g/mL, and the average sludge density was 1.78 g/mL.

4.3 PHYSICAL DATA SUMMARY

Thermal analyses were performed on tank 241-BY-110 core samples to satisfy the requirements of the safety screening DQO (Dukelow et al. 1995) and the organic DQO (Turner et al. 1995). These DQOs dictated that thermal analyses be performed on solid and liquid phases of the waste samples. The ferrocyanide DQO (Meacham et al. 1994) and the historical DQO (Simpson and McCain 1995) required thermal analyses to be performed on the solid phase only.

4.3.1 Thermogravimetric Analysis

In a TGA, the mass of a sample is measured while its temperature is increased at a constant rate. A gas, such as nitrogen or air, is passed over the sample during the heating to remove any evolved gaseous matter. Any decrease in the mass of a sample represents a loss of gaseous matter from the sample through evaporation or through a reaction that forms gas phase products. The change in mass is assumed to be from the loss of moisture. Weight percent water by TGA was performed under a nitrogen purge using procedures LA-560-112 Rev. B-1 and LA-514-114 Revs. C-0 and C-1.

Table 4-5 shows the TGA percent water data for tank 241-BY-110. Twenty-five of 110 sample means (23 percent) exhibited percent water means below the organic DQO

notification limit of 17 weight percent. Several others were just fractionally above the 17 weight percent threshold. Each core had samples that were below the notification limit.

The fact that several samples were below 17 weight percent does not constitute an unsafe condition. The energetics values for these samples must also exceed the safety screening and safety resolution notification limits for the tank to be considered unsafe.

The shaded rows in Table 4-5 indicate samples that were potentially contaminated with hydrostatic head fluid. Section 4.3.4 briefly discusses the tracer used to determine whether a sample has been contaminated and tabulates the corrected results for the suspect samples. For more information regarding the correction method and comprehensive results, refer to Winkleman (1996) and Appendix B, respectively.

Table 4-5. Thermogravimetric Analysis Results for Tank 241-BY-110. (4 sheets)

Sample Number ¹	Sample Location Segment (Portion)	Temperature Range	Result	Duplicate	Mean
		°C	% H ₂ O	% H ₂ O	% H ₂ O
Core 92, Riser 12B					
1732	4 (whole)	25-235	15.48	18.00	16.74
1752	5 (whole)	35-365	14.49	14.36	14.43
1816	5 (liner liquid) ²	35-105	89.32	89.22	89.27
1795	6 (upper half)	35-500	25.80	33.74	29.77
1794	6 (lower half)	35-500	15.20	15.69	15.45
1817	6 (drainable liquid)	35-280	38.41	36.15	37.28
1818	7 (drainable liquid)	35-275	38.07	37.90	37.98
1799	7 (whole)	35-240	40.44	37.35	38.90
Core 95, Riser 12B					
1740	1 (whole)	35-460	7.42	1.76	4.59
1789	3 (drainable liquid)	35-295	36.88	36.95	36.92
1741	4 (whole)	35-460	11.89	12.13	12.01
1743	5 (upper half)	35-175	18.60	16.73	17.66
1742	5 (lower half)	30-150	15.65	14.26	14.96
1744	6 (whole)	35-160	4.17	4.01	4.09
1744	6 (whole) R ³	35-260	3.96	19.76	11.86
1745	7 (whole)	35-220	11.14	8.9	10.02
1745	7 (whole) R	35-230	0.76	10.72	5.74
Core 96, Riser 12B					
2872	1 (whole)	35-130	16.28	14.34	15.31
2880	2 (whole)	35-130	10.33	6.99	8.66
2887	3 (top quarter)	35-480	12.28	13.43	12.86
2894	3 (second quarter)	35-275	45.67	44.38	45.03
Core 101, Riser 7					
1730	2 (whole)	35-125	13.40	13.60	13.50
1737	3 (whole)	35-125	11.55	13.05	12.30
1751	4 (whole)	35-135	37.69	32.05	34.87
1791	4 (drainable liquid)	35-500	70.92	70.64	70.78
1755	5 (whole)	35-445	17.05	17.38	17.21
1772	6 (upper half)	35-165	21.23	20.39	20.81
2150	6 (lower half)	35-160	24.38	23.34	23.86
2205	7 (top quarter)	35-210	30.84	26.90	28.87
2206	7 (second quarter)	35-215	30.52	27.86	29.19

Table 4-5. Thermogravimetric Analysis Results for Tank 241-BY-110. (4 sheets)

Sample Number ¹	Sample Location Segment (Portion)	Temperature Range	Result	Duplicate	Mean
		°C	% H ₂ O	% H ₂ O	% H ₂ O
Core 101, Riser 7 (Cont'd)					
2207	7 (third quarter)	35-225	26.84	25.88	26.36
2208	7 (bottom quarter)	35-500	29.11	30.78	29.95
2209	7 (whole)	35-270	43.46	44.02	43.74
2210	8 (top quarter)	35-215	24.65	26.26	25.45
2211	8 (second quarter)	35-235	37.51	36.03	36.77
2212	8 (third quarter)	35-225	32.37	32.45	32.41
2213	8 (bottom quarter)	35-500	22.19	37.11	29.65
2214	9 (top quarter)	35-500	25.29	0.00	12.64
2215	9 (second quarter)	35-265	27.21	25.44	26.33
2216	9 (third quarter)	35-275	28.41	31.37	29.89
2217	9 (bottom quarter)	35-500	29.90	15.89	22.90
Core 103, Riser 7					
1628	1 (upper half)	35-130	26.16	19.43	22.80
1614	1 (lower half)	35-140	15.52	13.21	14.37
1632	2 (whole)	35-125	16.56	16.12	16.34
1633	3 (whole)	35-110	17.51	17.95	17.73
1634	4 (upper half)	35-140	16.99	17.05	17.02
1635	4 (lower half)	35-135	17.42	17.81	17.62
1635	4 (lower half) R	35-410	15.11	18.52	16.81
1636	5 (whole)	35-145	17.45	17.45	17.45
1636	5 (whole) R	35-155	15.56	17.34	16.45
1667	6 (upper half)	35-210	33.02	34.21	33.62
1637	6 (lower half)	35-195	25.28	29.77	27.52
1669	7 (upper half)	35-260	25.07	33.36	29.21
1669	7 (upper half) R	35-215	31.11	43.71	37.41
1668	7 (lower half)	35-225	30.42	33.82	32.12
1670	7 (whole)	35-275	43.53	39.43	41.48
1671	8 (top quarter)	35-240	39.79	34.68	37.23
1672	8 (second quarter)	35-240	34.06	32.96	33.83
1672	8 (second quarter) R	35-245	34.68	33.63	34.16
1673	8 (bottom quarter)	35-240	31.09	31.38	31.24

Table 4-5. Thermogravimetric Analysis Results for Tank 241-BY-110. (4 sheets)

Sample Number ¹	Sample Location Segment (Portion)	Temperature Range	Result	Duplicate	Mean
		°C	% H ₂ O	% H ₂ O	% H ₂ O
Core 103, Riser 7 (Cont'd)					
1674	9 (top quarter)	35-225	33.71	32.75	33.23
1675	9 (second quarter)	35-235	28.13	36.24	32.19
1676	9 (third quarter)	35-455	31.44	28.60	30.02
1677	9 (bottom quarter)	35-455	26.44	24.17	25.30
Core 106, Riser 7					
1834	1 (upper half)	35-500	9.61	9.17	9.39
1909	1 (lower half)	35-115	17.71	19.52	18.62
1929	2 (whole)	35-140	15.33	16.18	15.75
Core 107, Riser 7					
1948	1 (upper half)	35-400	0.88	1.76	1.32
1950	1 (lower half)	35-165	24.10	26.43	25.27
1956	2 (upper half)	35-155	23.19	23.24	23.21
1964	2 (lower half)	35-145	22.05	20.82	21.44
2058	3 (whole)	35-500	20.71	15.95	18.33
2059	4 (whole)	35-500	18.77	18.76	18.77
2060	5 (top quarter)	35-185	18.78	18.26	18.52
2088	5 (second quarter)	35-265	40.65	40.27	40.46
2061	6 (upper half)	35-230	31.50	29.73	30.62
2062	6 (lower half)	35-205	32.36	28.20	30.28
2089	7 (top quarter)	35-255	32.56	33.67	33.12
2090	7 (second quarter)	35-275	26.18	29.16	27.67
2091	7 (bottom quarter)	35-500	26.16	25.61	25.88
2092	8 (top quarter)	35-500	34.72	30.77	32.74
2093	8 (second quarter)	35-500	30.09	10.32	20.21
2094	9 (top quarter)	35-500	29.69	33.98	31.84
2095	9 (second quarter)	35-250	29.22	30.83	30.02
2096	9 (third quarter)	35-500	31.21	30.02	30.62
2097	9 (bottom quarter)	35-500	29.55	30.65	30.10
Core 109, Riser 12B					
2417	1 (upper half)	35-370	13.26	19.81	16.54
2430	3 (whole)	35-215	33.64	32.23	32.94
2427	3 (drainable liquid)	35-500	47.70	47.38	47.54

Table 4-5. Thermogravimetric Analysis Results for Tank 241-BY-110. (4 sheets)

Sample Number ¹	Sample Location Segment (Portion)	Temperature Range	Result	Duplicate	Mean
		°C	% H ₂ O	% H ₂ O	% H ₂ O
Core 113, Riser 4					
3166	1 (whole)	35-485	18.31	17.69	18.00
3351	2 (whole)	35-120	24.89	26.18	25.54
3359	3 (whole)	35-500	16.21	16.11	16.16
3367	4 (upper half)	35-190	0.63	1.72	1.173
3542	4 (third quarter)	35-245	29.37	34.31	31.84
3550	4 (bottom quarter)	35-255	46.76	41.14	43.95
3558	5 (top quarter)	35-340	30.57	35.53	33.05
3566	5 (second quarter)	35-245	36.53	37.96	37.25
3430	5 (lower half)	35-245	40.86	41.10	40.98
3438	6 (top quarter)	35-225	43.24	36.02	39.63
3446	6 (second quarter)	35-245	44.93	41.32	43.13
3454	6 (third quarter)	35-275	43.49	33.26	38.38
3462	6 (bottom quarter)	35-265	27.28	28.97	28.12
3470	7 (top quarter)	35-245	34.17	31.62	32.90
3478	7 (second quarter)	35-215	38.62	36.84	37.73
3486	7 (third quarter)	35-220	33.42	32.73	33.08
3494	7 (bottom quarter)	35-235	38.26	31.69	34.98
3574	7 (drainable liquid)	35-255	46.59	48.00	47.30
3176	8 (top quarter)	35-265	31.91	31.83	31.87
3186	8 (second quarter)	35-265	31.03	32.37	31.70
3187	8 (third quarter)	35-500	29.94	31.89	30.91
3188	8 (bottom quarter)	35-500	31.24	32.67	31.96

Notes:

¹All sample numbers are preceded by "S95T00"

²Shaded rows indicate samples potentially contaminated with hydrostatic head fluid (see Section 5.1.4)

³Sample results designated with an "R" are rerun results.

4.3.2 Differential Scanning Calorimetry

In a DSC analysis, heat absorbed or emitted by a substance is measured while the substance is heated at a constant rate. The onset temperature for an endothermic event (characterized by or causing the absorption of heat) or exothermic event (characterized by or causing the release of heat) is determined graphically.

The DSC analyses were performed under a nitrogen atmosphere using procedure LA-514-113, Revs. C-0 and C-1 and a Mettler™ Model 20 differential scanning calorimeter and procedure LA-514-114, Revs. C-0 and C-1 and Perkin-Elmer™ equipment. No exotherms were observed in excess of the safety screening, organic, or ferrocyanide notification limits. However, modest exotherms were observed in some subsegments of most cores examined.

Table 4-6 shows the DSC results. For each transition, the table lists the sample number, sample location, sample weight, temperature at maximum enthalpy change, and the magnitude of the enthalpy change. Convention dictates that a negative enthalpy change indicates an energy release or an exotherm, and positive enthalpy change indicates energy absorbed or an endotherm.

The first transition, which ranged from ambient to approximately 150 °C, represented the endothermic reaction associated with the evaporation of free and interstitial water. The endotherms were quite large, usually greater than 400 J/g. In the second transition, which took place between 180 and 330 °C, both exotherms and endotherms were observed. The endotherms were substantial, but they were not as large as in the first transition (most often between 200 and 350 J/g) and probably represented the energy (heat) required to remove bound water from hydrated compounds such as aluminum hydroxide or to melt salts such as sodium nitrate.

The threshold of -75 J/g describing the significance of enthalpy changes was selected because the observed enthalpy changes were generally either very much lower or very much higher than that value, making it a convenient reference value. Such results are usually found in the upper (saltcake) portions of the core sample. The exotherms ranged from modest (enthalpy changes of -4 to -75 J/g) to substantial (enthalpy changes of greater than -75 J/g) and were probably caused by the fuel components of the sample reacting with the nitrate salts. Exotherms were observed more often in the lower (sludge) portions of the core sample. However, in every case, the net energy of the samples indicated by the DSC was endothermic, which suggests an inert-rich, fuel-poor environment. These two transitions contained most of the observed responses.

Additional DSC responses were observed infrequently at higher temperatures (above 330 °C) as well (see Transitions 3 and 4 in Table 4-6). It is hypothesized that these responses were caused by one or more factors. Generally, the results were modestly exothermic; therefore, some explanations are that they could represent additional reactions or a carryover of a reaction that started at a lower temperature and was masked by a concurrent endotherm.

However, for DSC results of this type and magnitude, a more likely cause was a shift in the instrument baseline at higher temperatures. In these cases, the DSC may have indicated a response where none occurred, and observations could be artifacts of the analytical method. Endotherms, when observed at higher temperatures, were usually much more substantial in absolute magnitude than the exotherms. A likely explanation of these results is that compounds other than nitrates and hydroxides may be melting.

The DSC results are reported on a wet weight basis. The safety screening DQO, however, requires that the exothermic reactions be evaluated on a dry weight basis in order to make a decision about tank safety. The dry weight value is obtained from the wet weight value by dividing the reported exothermic value for a subsegment by the solid fraction of the subsegment (that is, 1 minus the fractional percent water value for that subsegment). The maximum observed sample value was -434.2 J/g (dry).

The results for cores 92 and 95 were mostly endothermic. Modest exotherms were observed occasionally, but no exotherms of consequence were observed. In core 92 segment 7, small exotherms were observed in the drainable liquids, but none in the solids. This supports a general hypothesis that states that higher energy organics are mostly soluble and found in liquid, while lower energy organics are found in the solid waste matrices.

Cores 96, 106, and 109 behaved similarly to cores 92 and 95. This similarity was expected because these cores were from the upper (saltcake) portion of the tank. Segment 3 from core 109 exhibited modest exothermic responses of approximately the same magnitude in the solid and the liquid phases.

Cores 101, 103, 107, and 113 were similar. The first four segments behaved like those previously described and were mostly endothermic. Some small exotherms were observed intermittently at higher temperatures. Exotherms were observed routinely beginning at segment 5 of each core and increased in magnitude as a function of depth. In segments 7, 8, and 9 of cores 101, 103, and 107, transition 2 exotherms were observed routinely and were substantial (enthalpy changes greater than -75 J/g). However, no core exceeded the safety screening criteria, and the net energy for the samples was always endothermic. Core 107 had slight exotherms in transition 3. These appear to be carryovers or extended reactions that began at lower temperatures.

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 92, Riser 12B											
1732	4 (whole)	1	19.83	115.9	502.5	293.4	45.3	411.7	-4.3		
		2	22.52	119.6	647.9	295.1	57.8	409.7	-4.9		
1752	5 (whole)	1	31.30	115.3	455.7	297.1	34.9				
		2	41.91	112.4	608.4	317.6	3.13				
1816	5 (liner liquid)	1	16.80	124.3	476.5						
		2	15.20	119.7	550.7						
1795	6 (upper half)	1	23.10	108.9	847.8						
		2	25.60	111.6	589.6						
1794	6 (lower half)	1	44.22	154.7	287.6	288.0	55.6				
		2	53.45	149.5	437.8	288.7	39.8				
1817	6 (drainable liquid)	1	27.75	140.6	1058.5	241.7	40.0				
		2	24.25	135.7	1162.2						
1818	7 (drainable liquid)	1	16.10	133.2	995.5	251.4	-57.0				
		2	23.35	139.9	1136.2	245.4	-43.3				
1799	7 (whole)	1	16.60	135.6	966.0	249.6	-17.1				
		2	30.16	146.3	1018.9	302.4	-14.8				

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Run	Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)				Peak (°C)	ΔH (J/g)						
Core 95, Riser 12B												
1740	1 (whole)		1	15.53	97.2	176.4	301.7	95.1				
			2	19.63	104.9	275.3	299.0	99.4	402.1	5.1		
1741	1 (whole)		1	18.37	108.4	338.6	290.7	63.7				
			2	22.47	108.9	341.8	284.6	83.5				
1789	3 (drainable liquid)		1	17.80	137.8	988.0						
			2	23.0	129.1	564.5						
1743	5 (upper half)		1	18.10	117.1	442.9						
			2	20.68	116.4	431.9						
1742	5 (lower half)		1	29.55	94.9	586.8						
			2	13.60	119.0	584.9						
1744	6 (whole)		1	20.29	302.7	144.5						
			2	20.29	104.4	49.5	187.1	8.8	304.6	210.7		
1745	7 (whole)		1	22.54	105.4	72.6	185.1	18.6	297.0	115.3		
			2	41.7	102.1	144.8	220.6	82.1	292.7	308.8		
Core 96, Riser 12B												
2872	1 (whole)		1	16.72	124.6	453.9	310.6	39.2				
			2	49.28	132.8	389.9	311.1	75.2				

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 96, Riser 12B (Cont'd).											
2880	2 (whole)	1	19.28	120.9	336.2	299.5	45.3				
		2	36.75	115.6	240.2	296.8	80.9	405.5	-10.87		
2887	3 (top quarter)	1	32.29	110.8	344.6	297.6	19.3				
		2	35.05	111.2	262.8	300.3	43.6				
2894	3 (second quarter)	1	67.65	142.3	417.2						
		2	6.68	129.1	2355.6	297.5	84.9				
Core 101, Riser 7											
1730	2 (whole)	1	8.70	84.87	335.9						
		2	14.84	109.7	295.6	303.8	78.9				
1737	3 (whole)	1	14.70	107.0	319.3						
		2	15.20	105.7	298.6						
1751	4 (whole)	1	10.50	123.0	841.5						
		2	9.00	112.6	851.2						
1791	4 (drainable liquid)	1	12.32	130.2	1648.2	288.3	15.0				
		2	13.38	127.0	1656.8	287.7	14.3				
1755	5 (whole)	1	42.06	122.3	325.4	302.4	5.5	419.3	-29.6	481.2	-8.6
		2	24.01	124.9	552.5	298.6	20.1	413.4	-53.6		

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 101, Riser 7 (Cont'd)											
1772	6 (upper half)	1	47.18	121.3	538.0	329.2	-54.5				
		2	43.20	119.3	569.7	327.2	-50.0				
2150	6 (lower half)	1	15.83	109.4	497.0						
		2	20.04	108.0	489.8						
2205	7 (top quarter)	1	20.24	108.8	401.4						
		2	19.35	105.5	523.1						
2206	7 (second quarter)	1	11.20	106.0	579.9						
		2	14.21	106.4	414.8						
2207	7 (third quarter)	1	44.00	135.3	749.1	414.8	-146.1				
		2	30.92	142.8	630.5	412.8	-142.7				
2208	7 (bottom quarter)	1	62.00	144.8	646.2						
		2	33.05	141.5	687.4						
2209	7 (whole)	1	14.73	122.7	442.9						
		2	30.13	109.3	361.2						
2210	8 (top quarter)	1	21.99	107.5	324.2						
		2	35.70	108.9	392.4						

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
2211	8 (second quarter)	1	30.13	140.8	696.9	321.7	-81.8				
		2	18.11	125.7	893.3	317.7	-88.8				
2212	8 (third quarter)	1	22.67	141.4	875.4	329.8	-112.0				
		2	31.48	144.6	701.6	319.8	-91.1				
2213	8 (bottom quarter)	1	49.88	147.8	883.9	338.3	-43.0				
		2	36.01	142.8	911.7	335.6	-45.4				
2214	9 (top quarter)	1	47.45	145.6	728.5	370.2	12.8				
		2	42.38	154.7	699.6						
2215	9 (second quarter)	1	22.13	147.9	794.3	404.1	-252.0				
		2	31.24	131.2	850.0	408.4	-273.1				
2216	9 (third quarter)	1	15.77	138.2	717.1	329.8	-304.0				
		2	33.35	144.5	820.3	332.3	-304.4				
2217	9 (bottom quarter)	1	24.9	147.2	532.6						
		2	25.79	146.2	577.3						

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 103, Riser 7											
1628	1 (upper half)		22.10	136.1	1375.4						
	2		37.2	135.1	917.8	311.1	38.3				
1614	1 (lower half)		15.50	115.1	507.6	296.7	46.0			411.4	10.5
	2		19.90	110.8	472.6	298.3	58.0			413.4	10.4
1632	1 (whole)		13.30	111.3	597.8	299.0	3.7	407.2	-10.9		
	2		39.25	131.4	591.3	300.7	2.1	412.9	-11.4		
1633	1 (whole)		16.47	107.3	531.7						
	2		15.77	109.7	455.1						
1634	1 (upper half)		37.80	127.4	616.1	328.8	-25.7	422.8	-12.3		
	2		35.78	126.2	559.4	323.0	-28.9	429.0	-11.7		
1635	1 (lower half)		40.80	125.5	500.1	327.0	-32.7	419.0	-13.5		
	2		47.46	125.3	559.9	333.1	-31.3	421.1	-16.4		
1636	1 (whole)		9.36	114.7	563.4						
	2		13.57	111.9	417.2						
1667	1 (upper half)		13.88	129.4	832.1	317.3	92.3				
	2		13.63	125.7	737.7	311.4	88.9				
1637	1 (lower half)		10.96	107.9	967.4	317.3	97.1				
	2		9.68	124.1	851.1	319.4	96.9				

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 103, Riser 7 (Cont'd)											
1669	7 (upper half)	1	37.50	139.8	801.0	323.1	64.6				
		2	42.60	129.3	799.1	331.5	61.0				
1668	7 (lower half)	1	26.77	138.2	930.1	325.1	70.2				
		2	52.80	129.3	629.5	333.1	66.2				
1670	7 (whole)	1	39.80	129.3	1107.9	321.0	-65.9				
		2	28.70	133.3	1346.3	293.0	-25.3				
1671	8 (top quarter)	1	28.43	131.1	811.9	319.2	-38.6				
		2	15.35	125.4	1073.2	315.2	-45.4				
1672	8 (second quarter)	1	23.70	132.4	576.2						
		2	39.50	151.2	508.8						
1673	8 (bottom quarter)	1	28.60	149.5	480.7						
		2	16.40	130.7	488.7						
1674	9 (top quarter)	1	30.62	133.2	781.0	315.7	-59.7				
		2	26.26	144.7	975.3	321.8	-64.4				
1675	9 (second quarter)	1	22.17	108.2	798.7	351.6	-68.5				
		2	16.75	122.2	1061.2	321.9	-68.0				
1676	9 (third quarter)	1	38.41	141.9	793.8	336.3	-230.9				
		2	47.10	133.3	757.0	372.4	-258.2				

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 103, Riser 7 (Cont'd)											
1677	9 (bottom quarter)		35.16	142.0	770.4	376.2	-266.8				
			42.60	145.6	808.4	408.4	-241.0				
Core 106, Riser 7											
1834	1 (upper half)		15.07	110.4	339.4	300.1	169.7	417.6	-6.5		
	2		8.78	85.0	239.9	298.9	171.5		415.7	-6.3	
1909	1 (lower half)		19.62	110.7	531.6						
	2		21.01	110.4	523.1						
1929	2 (whole)		10.93	95.2	194.6						
			12.61	105.7	349.5						
Core 107, Riser 7											
1948	1 (upper half)		16.45	276.7	26.1	307.3	158.5				
	2		22.51	142.9	2.1	276.5	33.6	305.3	138.3		
1950	1 (lower half)		27.23	153.9	784.9						
	2		15.02	135.7	690.2						
1956	2 (upper half)		19.94	120.3	447.7						
	2		24.57	139.7	345.9						
1964	2 (lower half)		21.66	116.7	512.3						
			18.59	125.5	520.5						

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
2058	3 (whole)	1	31.50	118.0	639.7						
		2	37.1	125.4	630.8						
2059	4 (whole)	1	39.50	114.9	522.1						
		2	37.90	134.8	544.9						
2060	5 (top quarter)	1	28.75	122.6	458.2	317.4	-25.1				
		2	42.80	129.8	690.8	327.4	-29.9	427.2	-4.3		
2088	5 (second quarter)	1	48.37	135.3	855.6	330.3	-81.7				
		2	21.8	137.1	934.2	330.0	-86.1				
2061	6 (upper half)	1	42.64	145.5	888.3	325.5	-82.5				
		2	34.7	133.3	955.4	325.8	-89.8				
2062	6 (lower half)	1	36.00	143.1	634.7						
		2	32.28	142.7	698.2						
2089	7 (top quarter)	1	59.12	137.3	757.5	265.0	-2.3	339.8	-47.9		
		2	33.40	144.5	718.1	259.5	-4.4	335.9	-41.8		
2090	7 (second quarter)	1	34.70	140.4	703.6	323.8	-77.3				
		2	20.57	108.0	666.2	327.8	-68.4				

Core 107, Riser 7 (Cont'd)

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
2091	7 (bottom quarter)	1	14.62	106.5	359.1						
		2	12.94	101.8	556.9						
2092	8 (top quarter)	1	19.06	129.4	628.9						
		2	14.7	102.9	380.1						
2093	8 (second quarter)	1	12.57	107.0	380.3	171.2	190.6				
		2	18.2	153.8	415.5						
2094	9 (top quarter)	1	14.25	122.1	380.0						
		2	14.25	145.2	1615.2						
2095	9 (second quarter)	1	25.02	139.3	986.7	396.2	-239.9				
		2	23.18	137.5	911.8	398.4	-242.9				
2096	9 (third quarter)	1	30.19	151.48	601.4						
		2	36.71	145.1	643.5						
2097	9 (bottom quarter)	1	38.87	143.1	708.4						
		2	39.05	148.6	610.8						

Core 107, Riser 7 (Cont'd)

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number	Sample Location		Transition 1		Transition 2		Transition 3		Transition 4	
	Run	Segment (Portion)	Peak (°C)	ΔH (J/g)						
Core 109, Riser 12B										
2417	1	1 (upper half)	110.3	108.4	307.6	182.6	405.4	-14.4		
	2		117.0	471.6	300.5	105.0	415.8	-24.8		
	3		123.3	528.5	298.8	105.6	417.9	-25.6		
2430	1	3 (whole)	131.4	949.3	251.8	-52.7				
	2		144.4	1206.8	249.7	-53.0				
2427	1	3 (drainable liquid)	136.3	1133.9	247.6	-18.6				
	2		130.5	1040.2	249.6	-12.4				
Core 113, Riser 4										
3166	1	1 (whole)	113.1	419.7	255.1	59.7	439.7	-6.7		
	2		127.0	470.8	266.5	194.3	425.8	-6.9		
3351	1	2 (whole)	124.2	420.4	316.7	119.3	421.5	-12.8		
	2		121.0	657.3	302.7	58.0	425.7	-8.1		
3359	1	3 (whole)	123.7	464.4	302.6	14.3	407.6	-14.7		
	2		117.1	520.0	301.5	1.3	419.6	-12.2		
3367	1	4 (upper half)	130.0	571.7	237.3	6.2	325.5	-29.0	419.5	-8.8
	2		112.5	598.4	237.3	-7.4	323.5	-28.9	417.5	-8.0

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
3542	4 (third quarter)	1	32.03	137.6	958.9	242.7	24.2	289.7	-20.4		
		2	35.94	133.3	1062.3	295.7	-22.2				
3550	4 (bottom quarter)	1	47.66	127.3	731.2	240.5	20.8	287.5	-16.0		
		2	30.15	144.2	912.8	241.0	19.2	315.8	-47.4		
3558	5 (top quarter)	1	39.56	142.3	820.2	320.9	-32.7				
		2	32.66	130.4	903.0	319.1	-57.4				
3566	5 (second quarter)	1	17.88	139.4	1121	313.6	-63.3				
		2	48.09	141.9	915.6	326.3	-59.2				
3430	5 (lower half)	1	28.95	136.2	1158.8	317.2	-66.6				
		2	34.38	147.2	1170.8	240.8	9.6	252.7	-67.8		
3438	6 (top quarter)	1	13.47	122.2	1122.3	286.1	-24.7	429.1	-16.7		
		2	10.58	120.5	1202.6	252.9	-21.9	427.2	-65.0		
3446	6 (second quarter)	1	41.67	143.7	1136.3	254.6	-61.1				
		2	39.48	145.9	1142.6	250.9	-41.7				
3454	6 (third quarter)	1	19.36	119.8	966.2	278.9	44.4				
		2	26.37	137.6	1012.2						

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 113, Riser 4 (Cont'd)											
3462	6 (bottom quarter)	1	34.94	144.6	981.4	276.3	25.7	360.8	4.6		
		2	27.57	141.6	919.8	289.2	33.8	378.1	5.6		
3470	7 (top quarter)	1	25.37	128.3	1132.4	462.8	104.4				
		2	37.57	149.4	1206.7	425.0	64.2				
3478	7 (second quarter)	1	11.20	122.6	1052.9	319.2	-29.8				
		2	25.83	149.9	1255.2	330.4	-90.6				
		3	54.80	149.3	563.6	326.7	-29.0				
3486	7 (third quarter)	1	11.92	121.5	997.0	432.9	135.1				
		2	30.39	144.1	1135.6	417.9	60.6				
3494	7 (bottom quarter)	1	16.12	142.3	1091.0	324.9	-51.8				
		2	13.21	122.4	1080.0	324.6	-42.0				
3574	7 (drainable liquid)	1	25.68	129.0	807.3	471.5	-25.6				
		2	13.41	136.1	1526.5						
3176	8 (top quarter)	1	34.88	142.8	550.2						
		2	39.64	139.9	538.4	365.9	13.4				

Table 4-6. Differential Scanning Calorimetry Results for Tank 241-BY-110. (14 sheets)

Sample Number ¹	Sample Location		Sample Weight (mg)	Transition 1		Transition 2		Transition 3		Transition 4	
	Segment (Portion)	Run		Peak (°C)	ΔH (J/g)						
Core 113, Riser 4 (Cont'd)											
3186	8 (second quarter)	1	37.82	143.9	645.8	296.2	78.3				
		2	44.60	141.4	570.1	387.6	20.5				
3187	8 (third quarter)	1	36.11	148.35	497.8						
		2	27.69	143.0	508.2						
3188	8 (bottom quarter)	1	25.89	139.69	551.1						
		2	23.35	136.6	545.6						

Note: ¹All sample numbers are preceded by *S95T00*

4.3.3 Density

Density measurements were performed on most segments or subsegments of all cores. In some cases, no measurement was possible because of the limited material available. Where measurements were taken, only a single measurement was made. Schreiber (1995) provides the entire data set for these measurements. A general increase in density as a function of depth was observed for both saltcake and sludge samples. A possible explanation for this behavior is overburden compression.

Table 4-7 summarizes density data. To obtain overall saltcake and sludge density values, mean estimates and RSDs of the mean were calculated. No overall tank density was calculated because the phases have distinctly different properties. Inventory and other density-dependent calculations were performed using sludge and saltcake-specific values.

Table 4-7. Tank 241-BY-110 Density Data Summary.

	Salt Cake				Sludge			
	Core	Segment/ Subsegment	Sample No.	Result (g/mL)	Core	Segment/ Subsegment	Sample No.	Result (g/mL)
Min.	107	Segment 1	1955	1.02	107	Segment 8A	2142	1.60
Max.	101	Segment 7C	2272	1.90	103	Segment 9D	1729	2.07
Mean	All saltcake segments			1.44	All sludge segments			1.78
RSD				3.0%				2.2%

4.3.4 Vapor Data Summary

The safety screening DQO has established a notification limit that states that the headspace vapor results be less than 25 percent of the lower flammability limit. Prior to removing core samples, tank vapors were measured using a combustible gas meter and an organic vapor meter. Results of less than 0.5 percent of the lower flammability limit were observed in riser 12B for hydrogen and ammonia. This result satisfied the safety screening notification limit of < 25 percent of the lower flammability limit. Table 4-8 provides the results from the flammability vapor survey. The most abundant organic analytes detected in the 1994 vapor sample event were propanone, ethanenitrile, and 2-butanone (Huckaby and Bratzel 1995). However, they do not represent a flammability hazard individually or collectively.

Table 4-8. Flammability Testing Vapor Survey Results for Tank 241-BY-110.

Riser	Location	Measurement	Result
12B	Breathing zone	Total organic vapor	0 ppm
12B	Headspace	Total organic vapor	3.5 ppm
12B	Breather filter	Total organic vapor	15 ppm
		Ammonia gas	250 ppm
		Hydrogen cyanide gas	0 ppm
		Flammable vapor concentration as percent of LFL	0%
10A	Breather filter	Volume percent oxygen	10.9%
		Flammable vapor concentration as percent of LFL	0%
		Total organic vapor	8.0 ppm
		Ammonia gas	300 ppm
		Hydrogen cyanide gas	0 ppm
4	Headspace	Flammable vapor concentration as percent of LFL	0%
		Volume percent oxygen	10.9%
		Total organic vapor	5.0 ppm
7	Exhauster	Flammable vapor concentrations as percent of LFL	0%
		Volume percent oxygen	20.9%
		Total organic vapor	5.3 ppm
		Ammonia gas	150 ppm

As part of field operations for the tank sampling event, grab samples were taken of the headspace inside the drill string. On one occasion, the atmosphere changed substantially from a pure nitrogen atmosphere. Nitrogen was used as the hydrostatic balance for this core sample event. Table 4-9 shows the analysis of the drill string results as performed by the Pacific Northwest National Laboratory.

Table 4-9. Analysis of Tank 241-BY-110 Drill String Vapor Grab Sample.

Analyte	Concentration (%)
Nitrogen (N ₂)	57
Hydrogen (H ₂)	24
Nitrogen oxide (N ₂ O)	15
Oxygen (O ₂)	1
C ₂ H _x	1
Methane (CH ₄)	0.83
Other hydrocarbons	0.6
Ammonia (NH ₃)	0.28

A full vapor characterization was performed on the tank headspace gases in 1994. The data from this sampling and analysis is in *Tank 241-BY-110 Headspace Gas and Vapor Characterization Results for Samples Collected in November 1994*, Rev. 2A (Huckaby and Bratzel 1995). These results did not indicate that the elevated H₂ and N₂O levels, which were observed in the drill string, were present in the headspace. A calculation of the lower flammability limit using the vapor characterization data showed that the headspace gases were less than 1.0 percent of the lower flammability limit.

4.3.5 Separable Organic Layer

Each sample was visually inspected for the presence of a separable organic layer. None was found in any of the samples taken from tank 241-BY-110.

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

Section 5.0 evaluates the overall quality and consistency of the available results for tank 241-BY-110. These results are assessed and compared against historical information and program requirements.

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

The subsections below evaluate sampling and analysis factors that may impact data use or interpretation. These factors are used to assess the overall data quality and consistency and to identify limitations in data use.

5.1.1 Field Observations

The analytical data from the 1995 sampling event was obtained from nine cores: cores 92, 95, 96, and 109 from riser 12B, near the outer edge of the tank; cores 101, 103, 106, and 107 from riser 7, near the center of the tank; and core 113 from riser 4, also near the center of the tank. The position of these risers met the sampling requirements of the historical, safety screening, and ferrocyanide DQOs (Simpson and McCain 1995, Dukelow et al. 1995, and Meacham et al. 1995, respectively).

Sample recoveries were generally less than expected for all segments from the cores obtained from riser 12B (92, 95, 96, and 109). Better sample recovery was obtained from risers 4 and 7. Sampler performance improved when taking sludge samples. The cohesion of the sludge material appeared to be much higher than that of the saltcake. These results agree with observations from other tanks, that is, high cohesion sludges are easily sampled, whereas granular, low cohesion materials such as saltcake are not.

Multiple samples taken from a single riser may have contributed to recovery difficulties after the initial core sample was taken in riser 12B. Variation in recovery may have resulted from a combination of two factors: the heterogeneity of the waste and the wide variety of sampling parameters (bit speed, downforce, bit type, and sampling crews) attempted during the sampling. These parameters were varied to understand their impact on sampling operations. The experience gained by the sampling crews from this effort has been formalized in the current set of sampling protocols.

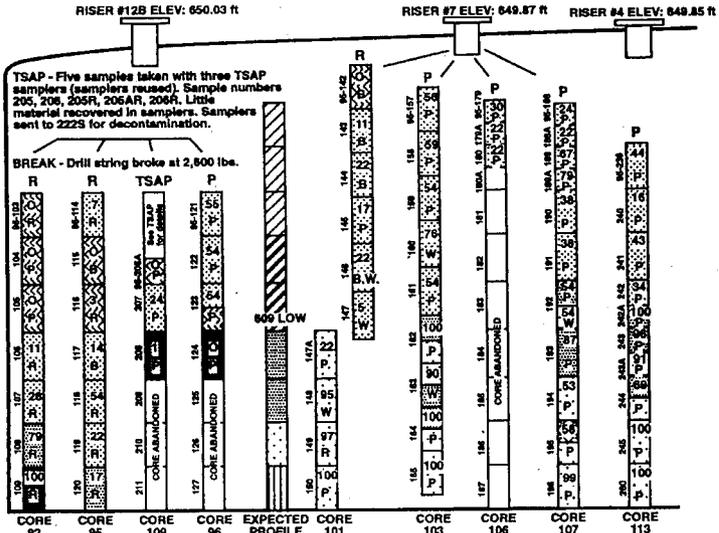
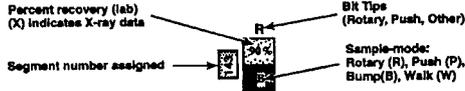
Figure 5-1 shows the cores and the degree of recovery from each riser sampled. It was noted upon extrusion that in many cores there were gaps in the samples. The first three segments of core 92 were empty. In core 95, an initial capture of some material occurred followed by two empty samplers. The core 95 gaps are not easily explained because core 96 recovered material at approximately the same depth. Sample acquisition from riser 12B was irregular in all four cores. A hard object/layer or the tank bottom prevented

Figure 5-1. Tank 241-BY-110 Physical Core Profile.

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Profiles below indicate primary constituent in sampler and are intended to reflect tank waste conditions. In some instances, extrusion information may be adjusted knowing other key information such as previous water additions.

STANDARD LABELING CONVENTION



2090000147.3

recovery of a top-to-bottom profile of the waste. Recoveries were much better in the cores from risers 4 and 7. Full depth waste profiles are believed to have been recovered from both risers.

5.1.2 Quality Control Assessment

The quality control assessment included an evaluation of the four quality control checks (blanks, duplicates, spikes, and standards) performed in conjunction with the chemical analyses. Because of the large amount of data collected for tank 241-BY-110, only a general evaluation and summary of some key safety and characterization areas are provided here. For more detailed quality control information on the data refer to Schreiber (1995 and 1996b) and DOE (1995) for specific laboratory control protocol. The SAP (Schreiber 1996a) establishes specific accuracy and precision criteria for the four quality control checks. Samples with one or more quality control results outside of the criteria are identified in Schreiber (1996b).

Precision criteria between several of the principal inorganic sample/duplicate pairs was greater than the quality control limits set by the laboratory. The precision is estimated by the relative percent difference, defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times one hundred. The variability in the results does not necessarily reflect on the laboratory procedures or equipment, but it may be intrinsic to the sample. Large differences may result from the relatively small samples (from 10 to 20 mg for DSC/TGA to 1g for ICP and IC results) used in these analyses. The requisite degree of homogeneity may not have been achievable with the procedures and equipment in place at the time of analysis. Difficulties in producing a homogeneous subsample are probably responsible for most of the precision problems observed for these and other analytes.

Tables 5-1 and 5-2 illustrate which ICP/IC analytes in which cores exhibited high RPDs at concentration levels greater than 1 weight percent (10,000 $\mu\text{g/g}$). Although many of the sample results exceed the specified RPD criteria, reruns were not performed because the results were substantially below any established threshold criteria, or no rerun criteria were specified.

The analytes identified in Tables 5-1 and 5-2 also exhibited RPDs greater than 20 percent most frequently at concentration levels less than the 10,000 $\mu\text{g/g}$ specified. Sample preparation appears to influence reproducibility. Analytes determined by water digestion have 41 of 73 responses meeting the above criteria, and there were notable differences in the same analytes determined by different methods (that is sulfate determined by IC and derived from the ICP sulfur value [8 vs 2]).

In summary, the samples often had precision quality control results outside the SAP boundaries. However, these precision results do not impact the usefulness of the data. The vast majority of blank, spike, and standard results, which relate to sample contamination,

were within the SAP-specified boundaries. The broad variation in the data can be attributed primarily to the spatial variability intrinsic to the Hanford tank wastes. An evaluation of quality control discrepancies has been made, and no impact to the validity or use of the data has been found.

Table 5-1. Riser 12B Cores--Analytes Greater than 10,000 $\mu\text{g/g}$ and RPDs > 20 Percent.

Analyte	Core 92	Core 95	Core 96	Core 109	Totals
Nitrate	X	X	X		3
Nitrite	X	X	X	X	4
Sodium				X	1
Aluminum	X	X		X	3
Phosphate	X		X	X	3
Phosphorous	X			X	2
Oxalate		X		X	2
Sulfate	X		X	X	3
Sulfur					0
Fluoride	X				1
Uranium					0
Chloride					0
Strontium					0
Nickel					0
Calcium					0
Totals	7	4	4	7	22

Note:

One or more segment/subsegment responses met the specified criteria.

Table 5-2. Risers 4 and 7 Cores--Analytes Greater Than 10,000 µg/g and RPDs Greater Than 20 Percent.

Analyte	Riser 7				Riser 4	Total
	Core 101	Core 103	Core 106	Core 107	Core 113	
Nitrate		X			X	2
Nitrite	X	X	X	X		4
Sodium	X		X	X	X	4
Aluminum	X	X	X	X	X	5
Phosphate	X	X		X	X	4
Phosphorous	X	X	X	X	X	5
Oxalate	X		X	X	X	4
Sulfate	X	X	X	X	X	5
Sulfur	X			X		2
Fluoride		X	X	X	X	4
Uranium	X	X		X	X	4
Iron						0
Chloride	X			X	X	3
Strontium	X			X		2
Nickel	X	X		X		3
Calcium		X		X	X	3
Total	12	10	7	14	11	54

Note:

One or more segment/subsegment responses met the specified criteria.

5.1.3 Data Consistency Checks

Comparing results for the same analytes using different analytical methods helps in assessing data consistency and quality. Several comparisons are provided by the nine core samples are discussed below. They include comparing phosphorus and sulfur as analyzed by ICP with phosphate and sulfate as analyzed by IC, comparing total alpha and total beta with the sum of alpha and beta emitters, and calculating a mass and charge balance to help assess the overall data consistency.

5.1.3.1 Comparison of Results from Different Analytical Methods. The following data consistency checks compare the results from two or more analytical methods for a given analyte. Close agreement between the two methods can strengthen the credibility of both results. Poor agreement may bring the reliability of the data or the assumptions about the waste into question. All segment analytical mean results were taken from Tables 4-3 and 4-4; composite analytical results are in Schreiber (1996b).

Table 5-3. Comparison of Phosphate/Phosphorous and Sulfate/Sulfur Concentrations by Different Methods.

Saltcake						
Analyte	ICP:A ($\mu\text{g/g}$)	RSD (%)	IC ($\mu\text{g/g}$)	RSD (%)	Solubility	RPD ¹ (%)
PO_4^{3-}	14,200	21.6	11,500	20.9	81.0%	21.0
SO_4^{2-}	17,900	24.5	18,400	32.3	102.8% (100%)	2.8
Sludge						
PO_4^{3-}	32,100	17.5	11,200	29.9	34.9%	96.5
SO_4^{2-}	16,100	13.5	18,400	14.9	114.3% (100%)	13.3

Notes:

- ICP:A = Inductively coupled plasma - acid prepared sample result
- IC = Ion chromatography result

¹RPD is calculated between ICP:A and IC result

The analytical phosphorus mean result in the saltcake, as determined by ICP:A, was 4,650 $\mu\text{g/g}$, which converts to 14,200 $\mu\text{g/g}$ of phosphate (assuming that all the phosphorous is present as phosphate). This compares relatively well with the IC phosphate mean result of 11,500 $\mu\text{g/g}$.

The ratio of IC to ICP results indicates the degree of solubility for the analyte, because insoluble phosphates are often present, and the IC does not detect them. In the saltcake, the

phosphate was approximately 81.0 percent soluble. The RPD between these two phosphate estimates is 21.0 percent and the RSD of the ratio is approximately 30 percent. Taken with the uncertainties, these results indicate the phosphate potentially could be even more soluble.

For the sludge, the results indicated that the phosphate was not as soluble as the saltcake. The analytical phosphorus mean result in the sludge, as determined by ICP:A, was 10,500 $\mu\text{g/g}$ which converts to 32,100 $\mu\text{g/g}$ of phosphate. This does not compare well with the IC phosphate mean result of 11,200 $\mu\text{g/g}$, and suggests that most phosphate in this waste is insoluble. This observation will alter some of the assumptions used in performing the mass and charge balance calculations.

The ICP sulfur value in the saltcake of 5,950 $\mu\text{g/g}$ converts to 17,900 $\mu\text{g/g}$ of sulfate (assuming all the sulfur is present as sulfate). This is reasonably close to the IC sulfate result of 18,400 $\mu\text{g/g}$. The RPD between the two sulfate estimates was a low 2.8 percent, and the RSD of the saltcake solubility ratio was approximately 40 percent. The most plausible explanation for these observations is that almost all of the sulfur/sulfate in the saltcake was soluble.

The ICP sulfur value in the sludge of 5,360 $\mu\text{g/g}$ converts to 16,100 $\mu\text{g/g}$ of sulfate. This compared closely with the IC sulfate result of 19,000 $\mu\text{g/g}$. The RPD between these two sulfate estimates was 13.3 percent, and the RSD of the sludge solubility ratio was approximately 20 percent. In this case, the sulfur/sulfate in the sludge is also considered entirely soluble.

Because of differences in sensitivity between the test methods and uncertainty associated with the measurements, the values did not precisely agree (that is, the sulfate appeared to be present and soluble at levels greater than 100 percent). The ion chromatography results were the results used in the mass and charge balance equations, and sulfate solubility was considered 100 percent.

A limited comparison was made between the gross beta and gross alpha activities with the sum of the individual beta and alpha emitters. Because of the sampling and analytical direction, total alpha and total beta measurements were not gathered on each sample. However, some composite results are available, and Tables 5-4 and 5-5 show selected comparisons for the segment and composite level results.

The sum of the activities of the individual alpha emitters is usually determined by adding ^{241}Am and $^{239/240}\text{Pu}$ plutonium isotope activities. However, because ^{241}Am was not detected, and ^{238}Pu was not measured, they were not included in the calculation. The activity sum was therefore derived by the following equation:

$$\text{Sum of alpha emitters} = {}^{239/240}\text{Pu}$$

The sum of activities of the individual beta emitters are as follows:

$$\text{Sum of beta emitters} = (2 * {}^{90}\text{Sr}) + {}^{137}\text{Cs}$$

Since ${}^{90}\text{Sr}$ is in equilibrium with its daughter product ${}^{90}\text{Y}$, the radiochemically measured value for ${}^{90}\text{Sr}$ alone must be multiplied by 2 to obtain numbers comparable with total beta.

Comparisons of total alpha results with plutonium can be done only with the saltcake samples, because Dukelow et al. (1995) specified that plutonium assays be performed instead of total alpha on the sludge samples. The comparison of total alpha with observed ${}^{239/240}\text{Pu}$ is poor, with an RPD of 77.3 percent. However, self-absorption may impose a substantial bias given the very low levels of total alpha and plutonium in the samples. Furthermore, although americium was not observed in the GEA scans, it may be present but below the detection limit for that method.

Table 5-4. Tank 241-BY-110 Comparison of Gross Alpha Activities With the Total of the Individual Activities.

Analyte	Half-Life (years)	Saltcake Overall Mean ($\mu\text{Ci/g}$)	Sludge Overall Mean ($\mu\text{Ci/g}$)
${}^{241}\text{Am}$	432	0	0
${}^{239/240}\text{Pu}$	24,100 (${}^{239}\text{Pu}$)	0.0192	0.0608
Sum of alpha emitters		0.0192	0.0608
Gross alpha		0.0434	nm
Relative percent difference		77.3%	nm

Note:

nm = no measurement

Table 5-5. Tank 241-BY-110 Selected Comparison of Gross Beta Activities With the Total of the Individual Activities.

Analyte	Composite Tank Mean ($\mu\text{Ci/g}$)
${}^{90}\text{Sr}$	84.7
${}^{137}\text{Cs}$	62.4
Sum of beta emitters	231.8
Gross beta	245
Relative percent difference	5.9%

Comparison of total beta results with ^{90}Sr and ^{137}Cs can be done only on composite data (see Appendix A). Comparison of the sum of the beta emitters with the total beta on the composites was quite favorable; with an RPD of 5.9 percent. Thus, the composite level results for beta emitters appear to be internally consistent.

5.1.3.3 Mass and Charge Balance. The principal objective in performing a mass and charge balance was to determine whether the measurements were consistent. In calculating the balances, only analytes which were detected at a concentration of 1,500 $\mu\text{g/g}$ or greater were considered.

Sludge Mass and Charge Balance

For sludges, except for sodium and potassium, the cations listed in Table 5-6 were assumed to be in their most common oxide/hydroxide form or an insoluble phosphate. The concentrations of the assumed species were calculated stoichiometrically. Because precipitates are neutral, all positive charge was attributed to sodium and potassium cations.

Acetate and carbonate were the species assumed; they were derived from the total organic carbon and total inorganic carbon analyses, respectively. Acetate was converted to a wet basis concentration. The other anionic analytes listed in Table 5-7 were assumed to be present as sodium salts and were expected to balance the positive charge exhibited by the cations. Sulfur, present as the sulfate ion, was assumed to be completely water soluble, and appeared only in the anion mass and charge calculations (see Section 5.1.3.1). The water soluble phosphate was included in the anion mass and charge data and was subtracted from the total phosphate calculated from the ICP. The insoluble phosphate was combined stoichiometrically with the calcium and strontium. The concentrations of the cationic species, the anionic species, and the percent water were ultimately used to calculate the mass balance. The uncertainty estimates (RSDs) associated with each analyte, and the uncertainty for the cation and anion totals also are shown in the tables.

The mass balance was calculated from the formula below and is evaluated in several steps. The conversion factor from $\mu\text{g/g}$ to weight percent is 0.0001.

$$\begin{aligned} \text{Mass balance} &= \% \text{ water} + 0.0001 \times \{ \text{total analyte concentration} \} \\ &= \% \text{ water} + 0.0001 \times \{ \text{Al(OH)}_3 + \text{FeO(OH)} + \text{Ca}_3(\text{PO}_4)_2 + \text{Ca(OH)}_2 + \text{Cr(OH)}_3 \\ &+ \text{Pb(OH)}_2 + \text{Ni(OH)}_2 + \text{K}^+ + \text{Sr}_3(\text{PO}_4)_2 + \text{Na}^+ + \text{U}_3\text{O}_8 + \text{C}_2\text{H}_3\text{O}_2^- + \text{CO}_3^{2-} + \text{F}^- + \text{Cl}^- \\ &\text{NO}_3^- + \text{NO}_2^- + (\text{COO})_2^{2-} + \text{PO}_4^{3-} + \text{SO}_4^{2-} \} \end{aligned}$$

The total analyte concentrations calculated from the above equation was 697,000 $\mu\text{g/g}$. The mean weight percent water obtained from thermogravimetric analysis was 30.5 percent (see Table 4-2). The mass balance resulting from adding the percent water to the total analyte concentration is 100 percent (see Table 5-8). One RSD for the mass balance is approximately 3.4 percent.

Table 5-6. Sludge Cation Mass and Charge Data.

Analyte	Concentration (µg/g)	RSD (Mean) (%)	Assumed Species	Concentration of Assumed Species (µg/g)	Charge (µeq/g)
Aluminum	28,300	4.20	Al(OH) ₃	81,800	0
Calcium	14,200	17.4	Ca ₃ (PO ₄) ₂ Ca(OH) ₂	78,100 7,600	0
Chromium	2,220	27.0	Cr(OH) ₃	4,400	0
Iron	20,000	22.7	FeO(OH)	31,800	0
Lead	1,880	54.5	Pb(OH) ₂	2,200	0
Nickel	6,670	20.7	Ni(OH) ₂	10,500	0
Potassium	2,930	8.43	K ⁺	2,930	75
Phosphorous (net)	6,840	17.5		with Ca and Sr	
Strontium	6,840	31.7	Sr ₃ (PO ₄) ₂	35,400	0
Sodium	161,000	5.47	Na ⁺	161,000	7,000
Uranium	20,900	22.4	U ₃ O ₈	32,100	0
Totals		5.41 ¹		448,000	7,075

¹RSD is for charge balance only.

Table 5-7. Sludge Anion Mass and Charge Data.

Analyte	Concentration (µg/g)	RSD (Mean) (%)	Charge (µeq/g)
Acetate (TOC)	19,000	14.3	-322
Carbonate (TIC)	32,200	17.6	-1,073
Fluoride	4,220	18.1	-222
Chloride	3,570	26.0	-101
Nitrate	111,000	5.62	-1,790
Nitrite	43,200	4.23	-939
Oxalate	5,870	24.9	-133
Phosphate	11,200	29.9	-353
Sulfate	18,400	14.9	-383
Totals	249,000	4.88 ¹	-5,316

¹RSD is for charge balance only.

Table 5-8. Sludge Mass Balance Totals.

	Concentrations ($\mu\text{g/g}$)	RSD (%)	Charge ($\mu\text{eq/g}$)	RSD (%)
Total from Table 5-6 (cations)	4.48E+05	--	7,075	5.41
Total from Table 5-7 (anions)	2.49E+05	--	-5,316	4.88
Water	3.05E+05	7.59	0	--
Subtotal	1.00E+05	3.39	1,759	7.3
Mass from assumed free OH ⁻	2.99E+04	--	-1,759	--
Grand Total	1.03E+06	--	0	--

The following equations are the derivation of total cations and total anions, the mass balance, and the charge balance. Relative standard deviations are based on propagation of error techniques and on an assumption of independence of the terms in the mass balance equation.

Total cations (microequivalents) = $\text{Na}^+/23.0 + \text{K}^+/39.1 = 7,075$ microequivalents

Total anions (microequivalents) = $\text{C}_2\text{H}_3\text{O}_2/59.0 + \text{CO}_3^{2-}/30.0 + \text{F}/19.0 + \text{Cl}/35.5 + \text{NO}_3/62.0 + \text{NO}_2/46.0 + (\text{COO})_2^{2-}/44.0 + \text{PO}_4^{3-}/31.7 + \text{SO}_4^{2-}/48.1 = -5,316$ microequivalents

The charge balance was 1.33 with an RSD of approximately 7.3 percent. The charge balance is the absolute value of the ratio of total cations to total anions. The net positive charge was 1,759 microequivalents. Boundary conditions for this system are 1,000,000 $\mu\text{g/g}$ (100 percent) for the mass balance and 1.00 for the charge balance with no net charge remaining.

The results indicate the mean concentrations for the tank were a biased description of the tank contents, or that some cations and/or anions have not been accounted for. Additional assumptions regarding the species present are needed.

The simplest assumption is to set the net charge equal to zero. Assuming the free positive charge is balanced by sufficient hydroxide to make the sludge neutral (-1,759 $\mu\text{eq/g}$ OH⁻), an additional 2.99E+04 $\mu\text{g/g}$ of hydroxide is added bringing the total to 1.03E+06 $\mu\text{g/g}$ (103 percent). This assumption is reasonable given the history of the wastes and the uncertainty in the measurements, and it provides sufficient charge to acceptably account for the individual analytes that comprise the sludge.

With this assumption and the uncertainty regarding these measurements, the mass and charge balance calculations were considered to be consistent. However, this calculation does

illustrate that a substantial contributor (that is, hydroxide) to the waste matrix is not directly measured.

Saltcake Mass and Charge Balance

For saltcake, sodium and potassium were assumed to be the cations present (see Table 5-9). All positive charge was attributed to these cations. The acetate and carbonate concentrations were derived from the total organic carbon and total inorganic carbon analyses, respectively. Acetate was converted to a wet basis concentration. Other anionic analytes were assumed to be present as sodium salts and were expected to balance the positive charge exhibited by the cations (see Table 5-10).

Table 5-9. Saltcake Cation Mass and Charge Data.

Analyte	Concentration (µg/g)	RSD (Mean) (%)	Assumed Species	Concentration of Assumed Species (µg/g)	Charge (µeq/g)
Potassium	1,930	10.9	K ⁺	1,930	50
Sodium	237,000	2.99	Na ⁺	237,000	10,300
Totals		2.99 ¹		239,000	10,350

Note:

¹RSD is for charge balance only.

Table 5-10. Saltcake Anion Mass and Charge Data.

Analyte	Concentration (µg/g)	RSD (Mean) (%)	Assumed Species	Concentration of Assumed Species (µg/g)	Charge (µeq/g)
Aluminum	14,100	13.4	Al(OH) ₄ ⁻	49,600	-522
Chromium	2,900	11.4	CrO ₄ ²⁻	6,470	-112
TOC	5,920	16.3	C ₂ H ₃ O ₂ ⁻	11,200	-190
TIC	31,800	24.5	CO ₃ ²⁻	159,000	-5,300
Fluoride	5,420	21.7	F ⁻	5,420	-285
Chloride	2,250	20.5	Cl ⁻	2,250	-63
Nitrate	184,000	37.2	NO ₃ ⁻	184,000	-2,968
Nitrite	30,600	18.6	NO ₂ ⁻	30,600	-665
Oxalate	13,600	28.3	C ₂ O ₄ ²⁻	13,600	-309
Phosphate	14,200	21.6	PO ₄ ³⁻	14,200	-448
Sulfate	18,400	32.3	SO ₄ ²⁻	18,400	-383
Totals		15.3 ¹		495,000	-11,245

Note:

¹RSD is for charge balance only.

Sulfur was assumed to be present as the sulfate ion and phosphorus as the phosphate ion. Both species were assumed to be completely water soluble and appeared only in the anion mass and charge calculations. This is a simplification for phosphate. The solubility of aluminum and chromium suggests that they were present primarily as anions in the saltcake and are represented as aluminate and chromate. This assumption is also supported by process history (that is, saltcake formation is caused by evaporation of supernatants, and soluble species are expected). The concentrations of the cations, the anions, and the percent water were used to calculate the mass balance. The uncertainty estimates (RSDs) associated with each analyte and the uncertainty for cation and anion totals are also shown in the tables.

The mass balance was calculated from the formula below. The factor 0.0001 is the conversion factor from $\mu\text{g/g}$ to weight percent.

$$\begin{aligned} \text{Mass balance} &= \% \text{ water} + 0.0001 \times \{\text{total analyte concentration}\} \\ &= \% \text{ water} + 0.0001 \times \{\text{Al(OH)}_4^- + \text{CrO}_4^{2-} + \text{Na}^+ + \text{K}^+ + \text{C}_2\text{H}_3\text{O}_2^- + \text{CO}_3^{2-} + \text{F}^- + \text{Cl}^- + \\ &\text{NO}_3^- + \text{NO}_2^- + (\text{COO})_2^{2-} + \text{PO}_4^{3-} + \text{SO}_4^{2-}\} \end{aligned}$$

The total analyte concentrations was 734,000 $\mu\text{g/g}$. The mean weight percent water obtained from thermogravimetric analysis is 23.2 percent. The mass balance resulting from adding the percent water to the total analyte concentration was 96.6 percent with and RSD of 8.5 percent (see Table 5-11).

Table 5-11. Saltcake Mass Balance Totals.

	Concentrations ($\mu\text{g/g}$)	RSD (%)	Charge ($\mu\text{eq/g}$)	RSD (%)
Total from Table 5-9 (cations)	2.39E+05	--	10,350	2.99
Total from Table 5-10 (anions)	4.95E+05	--	-11,245	15.3
Water	2.32E+05	7.72	0	--
Total	9.66E+05	8.50	-895	--

The following equations are the derivation of total cations and total anions, the mass balance and the charge balance. Relative standard deviations are based on propagation of error techniques, and an assumption of independence of the terms in the mass balance equation.

$$\text{Total cations (microequivalents)} = \text{Na}^+/23.0 + \text{K}^+/39.1 = 10,350 \text{ microequivalents}$$

$$\begin{aligned} \text{Total anions (microequivalents)} &= \text{Al(OH)}_4^-/95 + \text{CrO}_4^{2-}/58 + \text{C}_2\text{H}_3\text{O}_2^-/59.0 + \text{CO}_3^{2-}/30.0 \\ &+ \text{Cl}^-/35.5 + \text{F}^-/19.0 + \text{NO}_3^-/62.0 + \text{NO}_2^-/46.0 + (\text{COO})_2^{2-}/44.0 + \text{PO}_4^{3-}/31.7 + \\ &\text{SO}_4^{2-}/48.1 = -11,245 \text{ microequivalents} \end{aligned}$$

The charge balance was 0.92 with an RSD of approximately 15.6 percent. The charge balance is the absolute value of the ratio of total cations to total anions. The net charge is -895 microequivalents. Boundary conditions for this system are 1,000,000 $\mu\text{g/g}$ (100 percent) for the mass balance and 1.00 for the charge balance with no net charge remaining.

The results indicate that the mean concentrations for the tank were a relatively complete description of the tank contents. The small net negative charge is likely to be the result of assuming all aluminum and chromium ions as soluble when they are likely partitioned between soluble and insoluble forms.

Together with the assumptions and the uncertainty regarding these measurements, the mass and charge balance calculations are considered consistent.

5.1.4 Drill String Wash Water Contamination Check

During sampling, wash water was used to clean the drill string after each core was removed. Lithium bromide was added to the wash water as a tracer, and its presence in core samples indicates contamination by the wash water. This check, through analyses for lithium and bromide, was prescribed by the SAP (Schreiber 1996a). The SAP established notification limits of 100 $\mu\text{g/g}$ for lithium and 1,200 $\mu\text{g/g}$ for bromide.

Appendix B contains tables with lithium and bromide contamination data and the estimated corrections. Winkelman (1996) describes the correction method. Lithium can precipitate with components in the tank waste, so that the Li concentration in the wash water is biased low. Similarly, a high wash water intrusion can cause the bromide based correction to be negative. Therefore, both sets of corrected results are presented.

Fourteen of 104 subsegment samples and 2 composite samples are believed to be contaminated with wash water. Table 5-12 provides a summary of these potentially contaminated samples. Most of the corrections are within one RSD of the mean, suggesting that wash water intrusion was not a significant problem. Where corrections were greater than one RSD of the mean, the outcome with regards to safety remained unchanged: no exotherms were observed that approached the safety screening threshold. No samples were found to exceed the safety screening threshold for energetics although several of them were apparently below 17 weight percent water. Because most of the corrections are within one RSD of the mean, the impact to the overall mean is negligible.

Table 5-12. Samples Potentially Contaminated With Wash Water.

Sample Number ¹	Sample Description	Initial Mean Wt% H ₂ O	Li Corrected Wt% H ₂ O	Br Corrected Wt% H ₂ O
1789	Core 92 and 95, segment 3, drainable liquid	36.92	36.65	34.05
1732	Core 92 and 95, segment 4	16.74	13.86	10.41
1816	Core 92 and 95, segment 5, liner liquid	89.27	77.57	n/a
2872	Core 96, segment 1	15.31	13.17	7.60
1791	Core 101, segment 4, drainable liquid	70.78	57.01	n/a
1755	Core 101, segment 5	17.21	9.94	9.36
2150	Core 101, segment 6, lower half	23.86	21.98	9.83
2205	Core 101, segment 7, top quarter	28.87	28.24	16.77
1950	Core 107, segment 1, lower half	25.27	4.20	17.96
1956	Core 107, segment 2, upper half	23.21	16.94	15.88
1964	Core 107, segment 2, lower half	21.44	18.45	16.53
2417	Core 109, segment 1, upper half	16.54	12.48	9.61
2430	Core 109, segment 3	32.94	27.36	28.52
2427	Core 109, segment 3, drainable liquid	47.54	47.37	37.92
436 ²	Core 92 and 95, composite	12.56	11.68	n/a
479 ²	Core 107, composite	16.74	12.38	n/a

Note:

n/a = not applicable

¹Sample number preceded by "S95T00"

²Sample number preceded by "S96T00"

5.2 COMPARISON OF HISTORICAL AND ANALYTICAL RESULTS

There are no documented solid or liquid sampling/analysis events, other than the 1995 event for this tank. Therefore, no comparisons are possible.

However, ¹³⁷Cs inventory derived from spectral gamma scans compares quite well with the analytically derived inventory.

1.8E+05 Ci (Grigsby et al. 1992), 1.86E+05 Ci (see Tables 4-2 and 4-3)

5.3 TANK WASTE PROFILE

One objective of the 1995 sampling event was to obtain a vertical profile of the waste from two or more widely-spaced risers (Schreiber 1996a). Full vertical profiles were obtained from risers 4 and 7, and a partial profile was obtained from riser 12B. This enabled a statistical assessment of the vertical and horizontal distribution of the tank waste for many analytes.

Although the sample recovery from riser 12B was incomplete, the samples obtained were incorporated in the statistical evaluation of the data. Because this tank has two phases (saltcake and sludge), and these phases are not expected to vary substantially with horizontal position, the data were pooled according to phase designation. Therefore, saltcake and sludge means and RSDs of the means were calculated separately. Because there were no sludge samples obtained from the edge of the tank, the variability for that waste phase may be biased. The sample recovery was incomplete for some segments, consequently the analyte concentration and inventory estimates for the tank may be biased. The magnitude of the bias cannot be estimated.

Prior information on the vertical disposition of the waste was also available from the TLM (see Figure 2-3). According to the TLM, the waste is composed of three layers: a heel of IC waste, PFeCN1 and PFeCN2 waste on the bottom third of the tank, and BY saltcake in the upper two-thirds. These layers indicate that tank contents may be vertically heterogeneous. The visual descriptions of the extruded cores and segments also imply that tank contents are somewhat heterogeneous vertically and horizontally (see Table 3-2) although these differences may not be statistically significant.

5.3.1 Analysis of Variance Results

Random effects statistical ANOVA (analysis of variance) models were fit to concentration data from the core/segment samples. The results from these models can be used, on an analyte by analyte basis, to judge the vertical and horizontal variability in mean analyte concentration. Statistical results from these models are univariate results. Multivariate statistical results are outlined in the next section.

Two types of nested random effects ANOVA models were used. One random effects model was fit to core composite data; another was fit to core/segment data. The latter random effects model was fit to three subsets of the data: saltcake data, sludge data, and combined saltcake and sludge data. The ANOVA models were fit to analyte concentration data, provided, at least 50 percent of the measurements were above the detection limits. For these analytes, the detection limit was used as the measured concentration. Consequently, the summary statistics for these analytes are biased. The magnitude of the bias cannot be estimated.

For each analyte, estimates of mean concentrations and RSDs of the mean were obtained using restricted maximum likelihood (REML) methods. Appendix A contains the details and the results from the ANOVA. In addition to estimates of the mean and RSDs of the mean, Appendix A contains estimates of the standard deviation of the mean, 95 percent confidence intervals on the mean, estimates of the variance components used to measure horizontal and vertical variability and the p-values associated with the F-tests.

The p-values, associated with the F-tests from the ANOVA, are compared to a standard significance level ($\alpha = 0.05$). If it is less than 0.05, the analyte means are significantly different from each other. If a p-value is greater than 0.05, the analyte means are not significantly different from each other. The p-value is used to determine the significance of horizontal and vertical variability within the waste. The results are on an analyte by analyte basis.

The results from the ANOVA were mixed. There were 41 analytes in the combined saltcake and sludge data. There were significant differences in the mean concentrations between risers for 9 (22 percent) of the analytes and significant differences in mean concentrations between core samples for 3 (7 percent) of the analytes. There were significant differences in the mean concentrations between segments for 41 analytes. This suggests that, based on combined saltcake and sludge data, there is vertical variability within the waste. However, the evidence for horizontal variability is not conclusive.

Similar results were obtained from the individual sets of saltcake and sludge data. For the saltcake data (41 analytes), there were significant differences in mean concentrations between risers for 5 (12 percent) of the analytes and significant differences in mean concentrations between core samples for 6 (15 percent) of the analytes. There were significant differences in the mean concentrations between segments for 33 (80 percent) of the analytes.

For the sludge data (40 analytes), there were significant differences in mean concentrations between risers for 6 (15 percent) of the analytes and significant differences in mean concentrations between core samples for 2 (5 percent) of the analytes. There were significant differences in the mean concentrations between segments for 28 (70 percent) of the analytes. Consequently, based on the saltcake data and the sludge data, there is vertical variability within the waste, but little evidence for horizontal variability.

For the core composite data (40 analytes), there were significant differences in the mean concentrations between core samples for 6 (15 percent) of the analytes. That is, based on core composite data, there is little evidence for horizontal variability within the waste.

The general conclusion from the ANOVA models is that there is vertical variability within the waste. There is little evidence for horizontal variability.

5.3.2 Multivariate Clustering

The results given in Section 5.3.1 are based on ANOVA models applied to data from individual analytes. Alternatively, the analyte concentrations from the subsegments can be statistically analyzed using multivariate statistical methods. This section gives the results obtained from a multivariate technique known as cluster analysis. The results from the cluster analysis are given in the PNNL report in Appendix A.

Analytes were selected that would provide a characteristic description of the tank and a means of differentiating discrete waste types. In the analysis performed on the samples from tank 241-BY-110, 188 individual sample and duplicate results (from 94 segments/subsegments) in a reduced data set are 188 points in 14-dimensional space. The 14 dimensions are defined by the concentrations of aluminum, calcium, chromium, iron, lead, nickel, phosphorous, strontium, uranium, oxalate, ^{137}Cs , ^{90}Sr , total inorganic carbon, and total organic carbon. A quantitative summary of the cluster (number of samples, 10th percentile, median, and 90th percentile) were calculated from the measurements on the cluster members.

The cluster analysis partitioned the data into six clusters. There were two principal clusters. One major cluster has four distinct subsets (one major, three minor), and the other has two distinct subsets (one major, one minor). Figure 5-2A shows a cluster tree and the linkages among samples and clusters and the relative distances between them. The symbols at the bottom of the cluster tree in Figure 5-2A correspond to the individual segment or subsegment in the cluster analysis. The key to the notation is as follows:

Segment number/subsegment designation/core designation/primary or duplicate.

The segment number and subsegment designation are assigned as follows:

(W = whole, U = upper, L = lower, A-D = subsegment).

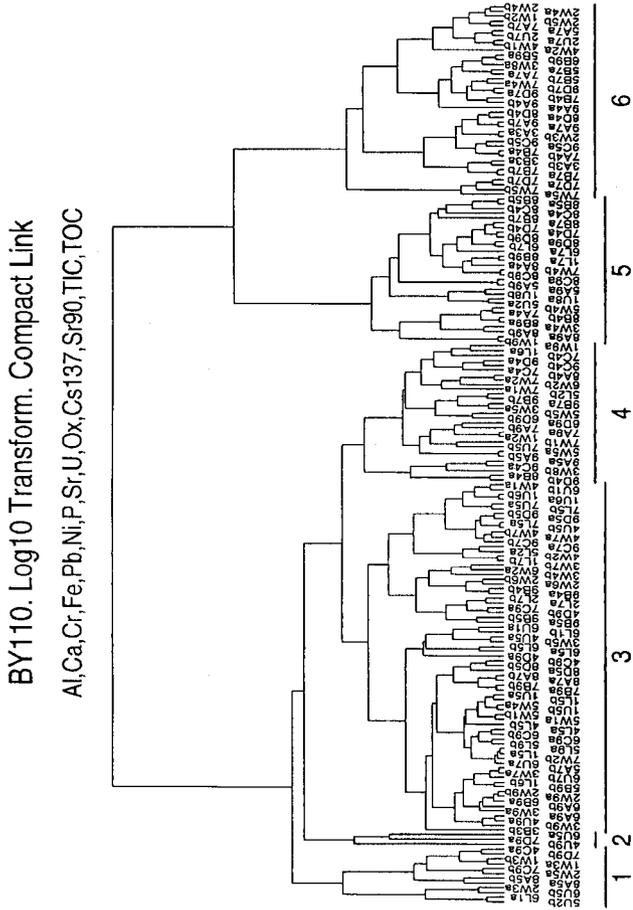
Core designation is as follows:

92 = 1, 95 = 2, 96 = 3, 109 = 4, 105 = 5, 103 = 6, 106 = 7, 107 = 8,
113 = 9. Primary or duplicate were denoted with a or b.

The two major groups consist of samples from the top and bottom of tank 241-BY-110, respectively.

The representation in Figure 5-2B is useful in interpreting the results of the cluster analysis. The figure contains three types of information. First, it gives the relative locations of the subsegments and core samples from the tank. Second, the boxes next to the subsegment contains the cluster number for the primary/duplicate result for that subsegment, as defined in Figure 5-2A. Third, the right hand column lists the appropriate position of the waste type as given by conventional interpretation of the Tank Layering Model (Agnew 1996a).

Figure 5-2A. Tank 241-BY-110 Log10 Transformation Compact Link
(Relationship of Sample Groups).



A quantitative summary of the six clusters is in Appendix A. In this appendix, the clusters are identified in the same manner as given by Figure 5-2A and 5-2B. From a visual comparison across clusters, it is evident that there are differences between the two principal clusters for iron, nickel, uranium, TIC, oxalate, and calcium. Appendix A also contains the technical details associated with the cluster analysis method.

The cluster analysis findings correspond to historical and process knowledge associated with the waste. The findings help with the interpretation of the historical behavior of the waste in the tank. One group (consisting of clusters 1 to 4) corresponds with saltcake, and the other group (consisting of clusters 5 and 6) corresponds with sludge. The subclusters in the larger group appear to represent differences that occurred during the in-tank solidification process, where differences in solubility, temperature, and particle size were captured in the strata sampled. The cluster 5 samples appear to be a transition layer where some sludge was captured with saltcake. The cluster 6 samples appear to be ferrocyanide sludge.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER DATA

5.4.1 Gateway Historical Evaluation

In addition to the safety screening DQO, cores from tank 241-BY-110 were analyzed in accordance with the historical DQO (Simpson and McCain 1995). The historical DQO strives to quantify errors associated with the tank waste composition predictions based on waste transaction history, input waste compositions, and assumptions regarding waste and process behavior. The DQO identifies key components and their characteristic concentrations for certain waste types.

Tank 241-BY-110 was selected as a tank for historical evaluation because it was expected to contain a thick saltcake layer and a thick ferrocyanide waste layer (Agnew et al. 1995). The first step in the evaluation was to compare the analytical results with DQO-defined concentration levels for a selected number of analytes. This comparison ensures that a predicted waste type may be in the tank and at the predicted location. If the analytical results are ≥ 10 percent of the DQO levels (ratio of 0.1), the waste type and layer identification are considered acceptable for further investigation (Simpson and McCain 1995).

BY saltcake was predicted to comprise the top two thirds of the tank waste (Agnew et al. 1996a). Table 5-12 presents a comparison between the mean results of the saltcake segments (see Table 4-2) and the historical DQO levels. The historical levels in many cases are minimum values. The comparison for BY saltcake appeared to support the historical results; therefore, further comparisons on an analyte basis are warranted.

Ferrocyanide waste was predicted to comprise approximately the bottom 38 to 55 in. of tank waste (Agnew et al. 1996a). The sludge segments from cores 101, 103, 107, and 113 should be of this waste type. Table 5-13 compares the concentration levels for ferrocyanide waste

from the historical DQO and the mean analytical results from those segments. All analytes except bismuth had analytical results at least 10 percent of the DQO-specified level.

Table 5-13. Comparison of BY Saltcake Fingerprint Analytes with Analytical Results.

Fingerprint Analyte	Mean Analytical Results	Historical DQO Concentration Level ^{1,2}	Ratio
Sodium	237,000 µg/g	> 165,000 µg/g	1.44
Aluminum	14,100 µg/g	18,600 µg/g	0.76
Nitrate	184,000 µg/g	> 266,000 µg/g	0.69
Sulfate	18,400 µg/g	33,700 µg/g	0.55
Percent water	23.2	35.9	0.65

Note:

¹Simpson and McCain (1995)

²These concentrations are the expected values.

Table 5-14. Comparison of Ferrocyanide Fingerprint Analytes with Analytical Results.

Fingerprint Analyte	Mean Analytical Result	Historical DQO Concentration Level ^{1,2}	Ratio
Bismuth	<DL	>25,000 µg/g	0
Nickel	6,670 µg/g	>4,000 µg/g	1.67
Sodium	161,000 µg/g	60,000 - 1.50E+05 µg/g	1.07 to 2.68
¹³⁷ Cs	140 µCi/g	> 8 µCi/g	17.5
⁹⁰ Sr	348 µCi/g	> 4 µCi/g	87
Percent water	30.5	28 to 81	0.38 to 1.09

Note:

¹Simpson and McCain (1995)

²These concentrations are the expected values.

The historical tank content estimate of tank 241-BY-110 is shown in Table 5-14 along with the inventory estimates (in kg) derived from the 1995 analytical results. The comparison is for information only. The HTCE values are generated from a combination of inputs from

Table 5-15. Comparison of Historical Inventory With 1995 Analytically-Derived Inventory for Tank 241-BY-110 (Saltcake and Sludge). (2 sheets)

Analyte	1995 Analytical Result	HTCE ¹ Estimate	Relative Percent Difference	Ratio Analytical/HTCE
METALS	kg	kg	%	
Aluminum	40,900	48,800	17.6	0.84
Bismuth	< DL	32,600	200	0
Calcium	9,360	8,950	4.48	1.05
Chromium	6,200	2,200	95.4	2.82
Iron	13,700	31,600	78.5	0.44
Lead	1,370	925	38.8	1.48
Manganese	228	141	47.2	1.62
Nickel	4,400	5,220	17.0	0.84
Potassium	5,020	1,560	105	3.22
Silicon	1,490	2,940	65.5	0.50
Sodium	4.95E+05	2.88E+05	52.9	1.72
Strontium	4,290	0.3	200	14,300
Uranium	14,000	20,200	36.5	0.69
Zirconium	36	111	102	0.32
IONS	kg	kg	%	
Cl ⁻	5,950	5,200	13.3	1.14
CO ₃ ⁻²	2.85E+05	33,600	160	9.11
F ⁻	11,600	3,560	106	3.29
NO ₃ ⁻	3.75E+05	4.04E+05	7.18	0.93
NO ₂ ⁻	77,500	68,900	11.7	1.13
PO ₄ ⁻³	43,400	37,700	14.1	1.15
SO ₄ ⁻²	42,000	24,700	52.0	1.70
Oxalate	26,300	0.2	200	132,000
RADIONUCLIDES	Ci	Ci	%	
¹³⁷ Cs	186,000	182,000	2.17	1.02
²³⁸ Pu + ^{239/240} Pu	69.3	140	67.6	0.50
⁹⁰ Sr	251,000	104,000	82.8	2.41

Table 5-15. Comparison of Historical Inventory With 1995 Analytically-Derived Inventory for Tank 241-BY-110 (Saltcake and Sludge). (2 sheets)

Analyte	1995 Analytical Result	HTCE ¹ Estimate	Relative Percent Difference	Ratio Analytical/HTCE
PHYSICAL PROPERTIES	%	%	%	
Percent Water ²	25.2	48.2	62.7	0.52
ORGANICS	kg	kg	%	
Acetate	n/a	1,490	n/a	0
CARBON	µg C/g	µg C/g	%	
Total Organic Carbon	16,700	4,790	111	3.49
Fe(CN) ₆ ⁴⁻	120	18,700	197	0.01

Notes:

n/a = not applicable

¹Agnew et al. (1996)²Calculated

the WSTRS (Agnew et al. 1995) and the HDW (Agnew 1996a). Each input contains assumptions and/or other factors that may impact the HTCE numbers (such as transfers of an unknown waste type into the tank). Because the HTCE values have not been validated, they should be used with caution.

Comparison of the predicted values with the analytical values produced varied results. A total of 29 analytes were compared. Eleven analytes (aluminum, calcium, lead, manganese, nickel, uranium, chloride, nitrate, nitrite, phosphate, and ¹³⁷Cs) exhibited RPDs less than 50 percent. Of these, seven analytes (aluminum, calcium, nickel, chloride, nitrate, nitrite, and ¹³⁷Cs) exhibited RPDs less than 20 percent. Eight analytes (bismuth, potassium, strontium, zirconium, carbonate, fluoride, oxalate, and ferrocyanide) exhibited RPDs greater than 100 percent. The RPDs for the remaining analytes were in between these two extremes. In several cases, the analytes with poor agreement were trace constituents in the tank waste. Large RPDs are often observed for analytes that are in trace quantities or near their detection limits. Using ratios to evaluate the results demonstrated that most analytes were within a factor of three of their predicted value.

5.4.2 Further Observations Between Predicted and Observed Properties

Other observations can be made by generally comparing analytical results with predicted waste type constituents. Sodium was predicted to be in higher quantities in the BY saltcake than in the ferrocyanide waste. In reviewing the subsegment analytical results in Appendix A, sodium was in higher concentrations in the upper segments. Analytes (nickel and iron) characteristic of PFeCN1 and PFeCN2 were in higher concentrations in the lower segments. No evidence of first cycle waste, specifically bismuth and low levels of radionuclides, was observed in any samples.

No strong trends were observed in the TGA data. The tank saltcake and sludge are substantially drier than predicted by Agnew et al. (1996). As expected, the surface material was very dry, and the saltcake material was drier than the sludge. However, the data from this tank indicated that assumptions about moisture distribution based on simple physical processes are not applicable. Moisture did not increase as a function of depth. In several cases, moisture content changed abruptly and irregularly (within one segment) and drier samples often were obtained from deep in the tank. The reason for this condition is not known although it was previously observed in tank 241-BY-108.

Currently, there is no way of determining whether there was a physical mechanism in the waste that promoted this type of stratification or whether the process of generating and distributing tank wastes caused the waste to be deposited in that configuration. A plausible explanation could be that the boiling performed as part of the in-tank solidification campaign could have contributed to this situation. Therefore, other tanks in BY farm, which were part of that process, or the self-boiling tanks in S and SX farm, may exhibit the same unexpected behavior.

Tables 5-15 and 5-16 show the mean analyte concentration and 95 percent confidence intervals on the mean based on the analytical data. The tables also show the corresponding HDW estimates. Table 5-15 gives the values for PFeCN1 sludge; Table 5-16 gives values for BY saltcake. A measure of agreement between the two estimates is whether the HDW estimate is in the confidence interval.

For the ferrocyanide sludge, the comparison can be made for 25 analytes. Twenty of the 25 HDW estimates (80 percent) were in the 95 percent confidence interval on the mean. For the BY saltcake, only 10 out of 24 HDW estimates (42 percent) were in the 95 percent confidence interval on the mean. If the saltcake and sludge results are combined, 30 of 49 HDW estimates (61 percent) are in the 95 percent confidence interval on the mean. Based on these results, there are no definite conclusions regarding the agreement of the predicted concentrations with the observed analytical concentrations. However, several of the discrepancies can be explained.

The disagreement in carbonate content is probably a result of the absorption of CO₂ from the air during ITS. Conditions for carbonate formation were quite favorable then, (hot caustic liquid with large contact area). The proposed aging phenomena (Lilga et al. 1993; Lilga et al. 1994; Lilga et al. 1995) accounts for the substantial difference in ferrocyanide estimates. Ferrocyanide breaks down in the presence of elevated temperatures, high caustic, and radiation. Because the tank has been at elevated temperatures and interim stabilized and still generates a modest heat load, it is drier than predicted.

Several source term errors in the Agnew et al. (1996a) model are apparent as well, with substantial differences between predicted and observed values for bismuth, strontium, fluoride, and oxalate. Some of the discrepancies of the highly soluble analytes (for example, sodium, potassium, and sulfate) may be a result of an increase in their relative concentration because of tank drying. The HDW model does not factor in water losses from evaporation in its predictions; however, radionuclides are decayed to January 1, 1994. The predictive capability of this model is also generally weak for trace analyses because of the scarcity and quality of the inputs.

Table 5-16. Comparison of Hanford Defined Waste--PFcN1 With 1995 Analytical Results for Tank 241-BY-110 Sludge. (2 sheets)

Analyte	1995 Mean Analytical Result	HDW ¹ Estimate	95% Confidence Interval on the Mean	HDW in 95% Confidence Interval
METALS	µg/g	µg/g		
Aluminum	28,300	0	13,200 - 43,400	No
Bismuth	< DL	37,700	Not calculated	No
Calcium	14,200	7,720	0 - 45,500	Yes
Chromium	2,220	114	0 - 9,820	Yes
Iron	20,000	36,400	0 - 77,800	Yes
Lead	1,880	0	0 - 14,900	Yes
Nickel	6,670	5,860	0 - 24,200	Yes
Potassium	2,930	483	0 - 6,070	Yes
Silicon	1,190	1,250	0 - 3,990	Yes
Sodium	1.61E+05	66,600	49,000 - 2.72E+05	Yes
Strontium	6,840	0	0 - 34,400	Yes
Uranium	20,900	18,500	0 - 80,600	Yes

Table 5-16. Comparison of Hanford Defined Waste--PFecN1 With 1995 Analytical Results for Tank 241-BY-110 Sludge. (2 sheets)

Analyte	1995 Mean Analytical Result	HDW ¹ Estimate	95% Confidence Interval on the Mean	HDW in 95% Confidence Interval
IONS	$\mu\text{g/g}$	$\mu\text{g/g}$		
Cl ⁻	3,570	2,010	0 - 15,400	Yes
CO ₃ ⁻²	32,200	11,600	0 - 20,800	No
F ⁻	4,220	2,730	0 - 14,000	Yes
NO ₃ ⁻	1.11E+05	1.11E+05	31,600 - 1.89E+05	Yes
NO ₂ ⁻	43,200	5,200	20,000 - 66,400	No
PO ₄ ⁻³ (total)	32,100	25,600	0 - 1.04E+05	Yes
SO ₄ ⁻²	18,400	12,000	0 - 53,400	Yes
Oxalate	5,870	0	0 - 24,400	Yes
RADIONUCLIDES	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$		%
¹³⁷ Cs	140	9.72	0 - 299	Yes
^{239/240} Pu	0.0608	0.0015	0 - 0.546	Yes
⁹⁰ Sr	348	2.36	0 - 1,140	Yes
PHYSICAL PROPERTIES	%	%		%
Percent water	30.5	62.0	10.8 - 59.9	No
CARBON	$\mu\text{g C/g}$	$\mu\text{g C/g}$		%
Total organic carbon	11,100	7,100	0 - 31,200	Yes

Note:

¹Agnew et al. (1996a)

Table 5-17. Comparison of Hanford Defined Waste--BY Saltcake With 1995 Analytical Results for Tank 241-BY-110 Saltcake. (2 sheets)

Analyte	1995 Mean Analytical Result	HDW ¹ Estimate	95% Confidence Interval on the Mean	HDW in 95% C.I.
METALS	$\mu\text{g/g}$	$\mu\text{g/g}$		%
Aluminum	14,100	32,100	5,950 to 22,200	No
Calcium	400	2,930	93 to 706	No
Chromium	2,900	1,490	1,480 to 4,320	Yes
Iron	924	3,720	0 to 1,880	No
Lead	130	13	29 to 232	No
Nickel	193	860	0 to 386	No
Potassium	1,930	1,400	1,020 to 2,840	Yes
Silicon	451	2,210	87 to 816	No
Sodium	2.37E+05	1.72E+05	2.06E+05 to 2.67E+05	No
Strontium	58	0	17 to 99	No
Uranium	697	4,740	198 to 1,200	No
IONS	$\mu\text{g/g}$	$\mu\text{g/g}$		%
Cl ⁻	2,250	2,700	265 to 4,230	Yes
CO ₃ ⁻²	159,000	17,600	0 to 65,300	No
F ⁻	5,420	660	368 to 10,500	Yes
NO ₃ ⁻	1.84E+05	2.51E+05	0 to 4.78E+05	Yes
NO ₂ ⁻	30,600	45,000	6,120 to 53,100	Yes
PO ₄ ⁻³	11,500	4,000	1,140 to 21,900	Yes
SO ₄ ⁻²	18,400	9,750	0 to 44,000	Yes
Oxalate	13,600	0.6	0 to 30,100	Yes
RADIONUCLIDES	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$		%
¹³⁷ Cs	60	131	26 to 94	No
^{239/240} Pu	0.019	0.139	0 to 0.0427	No
⁹⁰ Sr	22.5	76	1.3 to 44	No

Table 5-17. Comparison of Hanford Defined Waste--BY Saltcake With 1995 Analytical Results for Tank 241-BY-110 Saltcake. (2 sheets)

Analyte	1995 Mean Analytical Result	HDW ¹ Estimate	95% Confidence Interval on the Mean	HDW in 95% C.I.
PHYSICAL PROPERTIES	g/mL	g/mL		%
Percent water	23.2	39.7	15.5 to 30.9	No
CARBON	μg C/g	μg C/g		%
Total organic carbon	5,920	2,980	1,760 to 10,100	Yes

Note:

¹Agnew et al. (1996a)

5.5 SAFETY EVALUATION

Data criteria identified in the safety screening DQO (Dukelow et al. 1995) were used to assess the waste safety. The DQO requires samples from two widely-spaced risers. This requirement was met. The three primary analyses required by the safety screening DQO included the following: DSC to evaluate energetics, TGA to measure weight percent water, and a determination of total alpha activity. For each required analysis, the DQO notification limit was established which, if exceeded, could warrant further investigation to ensure tank safety. A final requirement of the safety screening DQO was to determine the flammability of tank headspace vapors. These measurements were taken prior to removing core samples.

5.5.1 Flammability Evaluation

The highest measured result for the headspace was less than 1 percent of the lower flammability limit, well below the safety screening limit of 25 percent (Dukelow et al. 1995). The flammability of tank 241-BY-110 headspace gas was also calculated on results from the 1994 vapor sampling and analysis event (Huckaby and Bratzel 1995). As shown in Table 5-17, the hydrogen, organic vapor, and ammonia fuel represented a combined total of less than 1.0 percent of the LFL, well below the safety screening limit of 25 percent of the LFL. At the reported concentrations, hydrogen, organic vapor, and ammonia do not individually or collectively represent a flammability hazard in the vapor space.

Table 5-18. Vapor Flammability Results from 1994 Vapor Sampling and Analysis Event.¹

Analyte	LFL	Average Concentration	Concentration as Percent of LFL
H ₂	40,000 ppmv	< 160 ppmv	< 0.4 percent
Total organics	46,000 mg/m ³	29 mg/m ³	< 0.1 percent
NH ₃	150,000 ppmv	401 ppmv	< 0.3 percent
		Total	< 0.8 percent

Note:

¹Huckaby and Bratzel (1995)

The data suggests that the drill string vapor space may change during sampling operations. A possible explanation for the observation made in the drill string vapor space (see Table 4-9) is that these wastes harbor voids containing gas mixtures that evolved from the chemical and radiolytic activity of the waste. The gases formed in the waste were unable to escape (for reasons such as surface tension, particle packing, and overburden compression) and collected in a pocket. Core sampling has the potential to release these gases to the surrounding atmosphere. The gas mixtures are often flammable or above the flammability notification limit before they are vented and dispersed. Gas generation and retention has been observed in other Hanford waste tanks, usually with episodic releases. Although gas generation and long-term retention in the waste without episodic releases has been theorized, evidence supporting this hypothesis had not been observed until this event.

Because no means of detection for this particular situation currently exist, care should be exercised when performing intrusive tank operations. Until the conditions that define this phenomenon are better defined, implementation of flammable gas tank controls and vapor space monitoring of the drill string on all tanks during this type of work appears to be prudent.

5.5.2 Criticality Evaluation

The safety screening DQO limit for criticality is 29.7 $\mu\text{Ci/g}$. It is assessed from the total alpha activity. All results obtained were well below this limit; the largest single result was 0.272 $\mu\text{Ci/g}$ in the sludge and 0.142 $\mu\text{Ci/g}$ in the saltcake. The upper 95 percent confidence interval limit on the mean in the samples exhibiting the highest values was 0.455 $\mu\text{Ci/g}$. This result is from a particular sample pair. No individual result from any sample approached the sludge limit.

5.5.3 Energetics Evaluation

The safety screening DQO has established a notification limit of -480 J/g (dry weight basis) for the DSC analysis. Of the observations from cores 92, 95, 96, 101, 103, 106, 107, 109, and 113, no sample duplicate pair had an average exceeding the limit. Several had substantial exotherms, especially the lower segments (7, 8, and 9) from cores 101, 103, and 107. However, for all observations, the individual DSC results were below the limit.

The DQO also requires that the exothermic upper limit (that is, the negative value of greatest magnitude) to one-sided 95 percent confidence interval on the mean be calculated for each primary/duplicate pair. No exothermic upper limit exceeded the -480 J/g (dry) notification limit (Schreiber 1996). The exothermic upper limit of the one-sided 95 percent confidence interval on the mean for this data set was -475 J/g (dry). This result was from the data pair from core 103 subsegment 9C. The largest individual result was -434.2 J/g, the primary result from core 101 subsegment 9C.

In addition, the upper exothermic limit to a one-sided 95 percent confidence interval on the mean using all the results from the sludge samples was also computed. The upper exothermic limit, in this case -507 J/g (dry), is slightly above the safety screening threshold (see Table A-1-3). The difference is a result of the spatial variability. Although the upper limit exceeds the safety screening threshold, there is no impact on the conclusion that the tank can be categorized as safe.

As a consistency check, the DSC analyses exceeding -200 J/g were compared to the energy equivalents of the cyanide and TOC analytical results for a given subsegment. The mean cyanide and TOC values were converted to a dry weight basis (see footnote 1, Table 5-18) using the mean percent water result for the subsegment. The cyanide fuel content was assumed to exist as disodium nickel ferrocyanide, $\text{Na}_2\text{NiFe}(\text{CN})_6$ (Meacham et al. 1994). The cyanide analytical result (dry weight) was converted to the weight percent of the assumed species using by the following equation:

$$\text{Na}_2\text{NiFe}(\text{CN})_6 \text{ wt\%} = \left[\frac{X \mu\text{g CN}}{\text{g}} \right] * \left[\frac{1 \mu\text{mol CN}}{26 \mu\text{g CN}} \right] * \left[\frac{1 \mu\text{mol Na}_2\text{NiFe}(\text{CN})_6}{6 \mu\text{mol CN}} \right] * \left[\frac{316.5 \mu\text{g Na}_2\text{NiFe}(\text{CN})_6}{1 \mu\text{mol Na}_2\text{NiFe}(\text{CN})_6} \right] * \frac{1 \text{ g}}{1 \times 10^6 \mu\text{g}} * 100 \text{ wt\%}$$

The calculated weight percent ferrocyanide was converted to the energy equivalent ferrocyanide (J/g) using the equation:

$$(\text{weight\% Na}_2\text{NiFe}(\text{CN})_6) * \left[\frac{6,000 \text{ J/g Na}_2\text{NiFe}(\text{CN})_6}{100 \text{ weight\%}} \right] = X \text{ J/g waste}$$

The energy equivalent conversion for TOC (based on a sodium acetate average energetics standard) is calculated by converting the analytical results from $\mu\text{g/g}$ to weight percent (dividing by 10,000). The equation (Babad et al. 1994) is:

$$\left(\frac{X \mu\text{g TOC}}{\text{g}} \right) * \left(\frac{1}{10,000} \right) * \left(\frac{30.2 \text{ cal}}{\text{g}} \right) \left[\frac{4.18 \text{ J}}{\text{cal}} \right] = \left(\frac{X \text{ J}}{\text{g}} \right)$$

For a given subsegment, the energy equivalent values for cyanide and TOC were added in column 6 of Table 5-19; the DSC analytical result is in column 7. The sum of the two energy equivalents were roughly 43 to 51 percent of the DSC results for all subsegments. A potential reason for difference may be attributed to a bias in the analytical method. If insoluble organic compounds are contributing to the DSC response, energy values derived from the TOC measurement will not agree with the observed enthalpy change. Alternately, a higher energy content could be considered for the organics.

The total organic carbon content notification limit is at 30,000 $\mu\text{g/g}$ (dry); a mean TOC concentration of 5,920 $\mu\text{g/g}$ (dry) was found in the salt cake, and a mean TOC concentration of 11,100 $\mu\text{g/g}$ (dry) was found in the sludge. No sample exceeded the notification limit.

Table 5-19. Selected Comparison of Differential Scanning Calorimetry Analytical Results With Total Organic Carbon and Cyanide Energy Equivalents. (2 sheets)

Core	Quarter Segment	Analyte	Mean Analytical Results ($\mu\text{g/g}$)	Energy Equivalent (J/g) ¹	Sum (J/g)	Mean DSC Analytical Result (J/g)
101	9B	TOC	9,000	-154	-155	-357
		CN ⁻	40.8	-0.7		
101	9C	TOC	10,900	-195	-196	-434
		CN ⁻	42	-0.7		
103	9C	TOC	9,430	-176	-176	-350
		CN ⁻	16.5	-0.3		
103	9D	TOC	9,650	-174	-178	-340
		CN ⁻	248	-4		

Table 5-19. Selected Comparison of Differential Scanning Calorimetry Analytical Results With Total Organic Carbon and Cyanide Energy Equivalents. (2 sheets)

Core	Quarter Segment	Analyte	Mean Analytical Results (µg/g)	Energy Equivalent (J/g) ¹	Sum (J/g)	Mean DSC Analytical Result (J/g)
107	9B	TOC	8,740	-157	-163	-344
		CN ⁻	369	-6.4		

Notes:

¹Fuel values are averaged and converted to a dry weight basis using the following equation:

$$\frac{X \mu\text{g/g (Avg. wet weight)}}{1 - (\text{Avg. \% water}/100)} = X \mu\text{g/g (Avg. dry weight)}$$

Similarly, DSC values are averaged and converted to dry basis.

According to the SAP, the safety screening DQO limit of 17 weight percent water was superseded by the ferrocyanide DQO water content requirement. The action of saltwell pumping, active ventilation (earlier in its process history), participation in the in-tank solidification program, and its current heat load have all contributed to water loss over the tank's service life. Thus, the tank is drier than predicted by Agnew et al. (1996a). An inspection of the TGA data revealed that 25 subsegments had means below 17 percent, however, none of the samples had DSC results greater than -480 J/g.

Table 5-19 shows the result of a correlation analysis between TOC, TGA, and DSC (dry). The correlations were computed using 50 means. Knowledge regarding the relationships between these analyses is useful in interpreting data.

Table 5-20. Correlation Between Differential Scanning Calorimetry, Thermogravimetric Analysis, and Total Organic Carbon.

Analyte	TOC	TGA	DSC
TOC: Coefficient	1		
TOC: p-value	0.0000		
TGA: Coefficient	0.2612	1	
TGA: p-value	0.0669	0.0000	
DSC: Coefficient	0.3823	0.4893	1
DSC: P-value	0.0061	0.0003	0.0000

A correlation is a linear measure of agreement between two sets of observations. In these three cases, the correlations are significantly different from zero, as indicated by the p-values in the table being less than 0.05 (the TOC/TGA relationship is marginal). A correlation coefficient is a number between 1 and -1. A positive correlation means that as one analyte increases, the other increases. A negative correlation means that as one analyte increases, the other decreases. Consequently, because the correlations are significantly different from zero, the data indicates that there are linear relationships between TOC, TGA, and DSC (dry). For example, TOC and TGA are related because the TOC provides the fuel content measured in the waste. DSC and TGA are related because the fuel and oxidizer in the tank wastes are soluble.

These analytes should be positively correlated with each other. However, the correlations, are not as large as expected given the nature of physical and chemical relationships between them. The DSC/TGA relationship is expected to be the weakest because it is the most subject to confounding from external factors.

There are three obvious reasons for this. The correlations were computed based on mean values instead of individual results, there is substantial spatial variability in the data, and there are confounding analytes in the data that are not included in the analysis. Given the complexity of the tank wastes, there may be other reasons for the lack of large correlations between TOC, TGA, and DSC (dry).

5.5.4 Ferrocyanide Evaluation

Because the ferrocyanide DQO requires analyses on a quarter segment basis, the notification limits were applied to the data on a quarter segment level. Only individual limit excursions are discussed. Primary analyses required by the ferrocyanide DQO include DSC and TGA and measurements of the cyanide, nickel, and total organic carbon concentrations. The DSC requirements were the same as those discussed previously for the safety screening DQO. The actual TGA notification limit listed in the ferrocyanide DQO is as follows:

$$\text{weight percent water} \geq 4/3 (\text{weight percent fuel} - 8 \text{ weight percent})$$

The SAP has converted this limit into an equation more readily usable by the laboratory. The applicable assumptions and calculations for this conversion are listed in Schreiber (1996a). The modified decision limit is as follows:

$$\text{weight percent water} > (0.0223 * \text{DSC exotherm [dry weight]}) - 10.7$$

Using this equation, the minimum water contents required for samples with DSC results above -480 J/g, can be calculated (according to the ferrocyanide DQO). However, based on the DSC results, no further calculation is necessary. No DSC result exceeded the safety screening threshold.

Recent aging studies of ferrocyanide waste show that the combined effects of temperature, radiation, and pH during 38 years or more of storage would have destroyed most of the ferrocyanide originally added to the tanks (Babad et al. 1993; Lilga et al. 1993, 1994, and 1995). This prediction has been confirmed by the tank samples analyzed. To determine the extent of ferrocyanide degradation, estimates of the total amount of $\text{Na}_2\text{NiFe}(\text{CN})_6$, originally present in the tank, and recent analytical cyanide concentrations are needed. According to Borsheim and Simpson (1991), 22,400 kg of $\text{Na}_2\text{NiFe}(\text{CN})_6$ were expected to remain in the tank at the end of the ferrocyanide waste transfer activity in 1957. This number is reasonably close to the $\text{Na}_2\text{NiFe}(\text{CN})_6$ inventory of 27,900 kg calculated based on the HTCE concentration for $\text{Fe}(\text{CN})_6^{4-}$ of 8,360 $\mu\text{g/g}$ and the HTCE total weight for the waste of 2.24E+06 kg.

Any comparison between current analytical data to the original estimates of $\text{Na}_2\text{NiFe}(\text{CN})_6$ in the tank should be based only on the mean analytical values from the designated sludge segments of cores 101, 103, 107, and 113 because ferrocyanide waste has been predicted to comprise approximately the bottom 37 to 56 in. of the tank content (Agnew et al. 1995; Hanlon 1996). Another method of estimating the amount of $\text{Na}_2\text{NiFe}(\text{CN})_6$ originally present would be to assume that all the nickel currently found in the tank originated from $\text{Na}_2\text{NiFe}(\text{CN})_6$. The mean nickel concentration based on the sludge segments of cores 101, 103, 107, 113 was 6,670 $\mu\text{g/g}$. The estimated sludge mass is 612,000 kg. If all the nickel originated from $\text{Na}_2\text{NiFe}(\text{CN})_6$, then the observed nickel concentration would indicate that 36,000 $\mu\text{g/g}$ of $\text{Na}_2\text{NiFe}(\text{CN})_6$ (22,000 kg of $\text{Na}_2\text{NiFe}(\text{CN})_6$) existed in the tank before degradation. Therefore, these three sources of information place the original $\text{Na}_2\text{NiFe}(\text{CN})_6$ inventory approximately within the 22,000 to 27,900 kg range. Thus the analytical estimate supports the historical estimates of ferrocyanide inventory prior to degradation.

The recent total cyanide analytical mean based on the sludge segments of cores 101, 103, 107, and 113 is 98.5 $\mu\text{g/g}$ (60.3 kg of cyanide). This is equivalent to 122 kg of $\text{Na}_2\text{NiFe}(\text{CN})_6$. Consequently, it appears that greater than 99 percent of the ferrocyanide complex has decomposed. Even when the highest cyanide analytical mean from the bottom three segments were used (800 $\mu\text{g/g}$ for core 103 subsegment 9A which is equivalent to 2,100 kg of $\text{Na}_2\text{NiFe}(\text{CN})_6$), a ferrocyanide degradation of at least 90 percent is obtained. All quarter segment results and the overall mean cyanide analytical result were lower than the 39,000 $\mu\text{g/g}$ ferrocyanide DQO limit. No decision limit was specified for the nickel concentration in the ferrocyanide DQO which was applied to the 1995 sampling and analysis event (Meacham et al. 1994).

5.5.5 Organic Screening Evaluation

The safety program test plan specified two primary analyses: TOC and an organic screen. The organic screen targets determined the presence of normal paraffin hydrocarbons. No decision limits were established for any analytes. Minor amounts of hexadecane were found (see Section 4.1 for the overall means) and very low combinations of TOC vapor were found

in the tank headspace or breather filter. No separable organic layer was visible in any of the samples.

5.5.6 Analyte Criteria Summary

Table 5-19 displays the analyte criteria limits for the safety related DQOs and the test plan. The number of results, which exceeded each criterion, are also included. Because the samples exceeding the limit have been discussed in the text, they are not addressed in Table 5-19.

Table 5-21. Data Quality Objective and Test Plan Decision Variables and Criteria.

Applicable DQO or Test Plan	Applicable Core(s)	Primary Decision Variable	Decision Criteria Threshold	Number Outside Threshold ¹
Safety screening, Ferrocyanide	All	Total fuel content	-480 J/g	None
Ferrocyanide	All	Percent water	(0.0223 * DSC exotherm [dry weight] - 10.7)	None
Organic	All	Percent water	17 weight percent	25
Safety screening	All	Total alpha	29.7 $\mu\text{Ci/g}$ (1 g/L) ²	None
Ferrocyanide	All	Cyanide	39,000 $\mu\text{g/g}$	None
Ferrocyanide, organic	All	TOC	30,000 $\mu\text{g/g}$	2
Safety screening	All	Organic layer	Present/not present	None present

Notes:

¹The number of sample means outside the limit. Limit excursions for individual samples have been discussed in detail in the accompanying text.

²Although the actual decision criterion listed in the DQO is 1 g/L, total alpha is measured in $\mu\text{Ci/g}$ rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it was assumed that all alpha decay originates from ²³⁹Pu. Assuming a tank density of 2.07 (the extreme value obtained from sampling) and using the specific activity of ²³⁹Pu (0.0615 Ci/g), the decision criterion may be converted to 29.7 $\mu\text{Ci/g}$ as follows (Schreiber 1996b):

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

5.5.7 Heat Load Calculation

Another factor in assessing tank waste safety is heat generation and waste temperature. Heat is generated in the tanks from radioactive decay. An estimate of the tank heat load was calculated from the 1995 radionuclide data. Only radionuclides present in detected quantities were used in the heat load calculation. Table 5-21 displays the calculated heat load. The estimated heat load is 2,560 W (8,740 Btu/hr). The corresponding value, estimated from the headspace temperature is 2,020 W (6,900 Btu/hr) (Kummerer 1994). It is below the 11,700 W (40,000 Btu/hr) threshold differentiating high-heat from low-heat tanks (Bergmann 1991). This value does not compare well with the HTCE estimate of 1,550 W (5,330 Btu/hr) because of the difference in ^{90}Sr content in the tank. Because an upper temperature limit is exhibited (see Section 2.4), it may be concluded that any heat generated from radioactive sources throughout the year is dissipated.

Table 5-22. Tank 241-BY-110 Projected Heat Load.

Radionuclide	Curies	Watts
^{137}Cs	1.86E+05	878
^{90}Sr	2.51E+05	1,682
Total	4.37E+05	2,560

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-BY-110 was core sampled using the rotary and push mode from July to October 1995. Three DQOs and a test plan governed the sampling and analysis of cores 92, 95, 96, 101, 103, 106, 107, 109, and 113: the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), the *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994), the *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994), the *Test Plan for Samples from Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109* (Meacham 1995), and the *Historical Model Evaluation Data Requirements* (Simpson and McCain 1995). Although not addressed by the SAP, the analyses required by the *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner et al. 1995) were met while meeting the requirements of the other DQOs and the test plan. In addition, an internal letter (Kristofzski 1995) directed the laboratories to perform all feasible analyses of the waste samples on an opportunistic basis, according to the work load in the laboratory.

Analytical results show that the waste contains compounds in a few selected tank areas capable of exothermic reactions. However, no sample exceeded the -480 J/g notification limit in the safety screening DQO. The percent water result from all samples exhibiting changes in enthalpy above -200 J/g were greater than 25 weight percent water. Comparisons were made between the DSC results and the energy equivalents of the TOC and cyanide results for selected quarter segments which had changes in enthalpy greater than -200 J/g. For the selected samples, 43 to 51 percent of the DSC result was accounted for by the TOC energy equivalent, while the cyanide energy equivalent accounted for only a negligible fraction of the observed exotherm. This suggests that there may be insoluble TOC or ferrocyanide degradation products contributing to the observed enthalpy changes.

There was evidence of gas generation and long-term retention in the sludge. During the course of sampling, a vapor sample obtained from the drill string had a much different composition than the surrounding headspace, suggesting that a void or gas bubble had been disturbed by sampling. This type of structure has long been suspected as being possible in the wastes, but none has been encountered until now. The gas in the headspace itself is not flammable, but has elevated ammonia concentration (150-300 ppm).

Total alpha activities were approximately one percent of the safety screening limit. Total organic carbon and cyanide concentrations, required by Meacham et al. (1994), were well below their respective limits. No separable organic layer was detected, and organic vapor levels in the headspace were low. Analytical nickel concentrations compare favorably with historical estimates and indicate that the ferrocyanide has decomposed.

The estimated tank heat load of 2,560 W was well below the 11,700 W limit that separates high-heat from low-heat load tanks. The highest LFL measured from the tank headspace was less than 1 percent, which is significantly lower than the 25 percent notification limit (Dukelow et al. 1995).

Analytical data confirm several assumptions regarding the composition and structure of the tank waste. Several other assumptions were not supported by the data. The degree of spatial variability observed in this tank is high, as indicated from physical observation and statistical analysis. Two distinct phases, with very different chemical and physical properties exist.

The saltcake phase, which accounts for approximately 77 percent of the tank waste (by volume), is made up of mostly soluble species and contains modest concentrations of radionuclides. It did not agree well with several predictions regarding its physical and chemical composition. This saltcake is drier than predicted (not cohesive) and contains several species that were not predicted (oxalate, fluoride, soluble aluminum and chromium ions). Predictions for only 10 of 24 analytes fall within a 95 percent confidence interval on the mean. The saltcake also possesses a more complicated structure than expected. Moisture does not increase as a function of depth and does not display any consistent trend. A likely reason for this behavior are the processes and boundary conditions that formed the saltcake (that is, saltwell pumping, active heating and ventilation during the in-tank solidification program, and the present heat load). It is suspected in other tanks that experienced similar conditions (for example other in-tank solidification campaign tanks in BY farm and the self-boiling tanks in S and SX farm).

The sludge phase, which accounts for approximately 23 percent of the tank waste (by volume) and lies beneath the saltcake. Its composition agrees well with several qualitative predictions regarding its physical and chemical properties, and predictions for 21 of 25 analytes fall within a 95 percent confidence interval on the mean. It is largely insoluble, very cohesive, and contains most of the radionuclides. However, measurements of strontium, aluminum, and bismuth did not agree with historical estimates. The sludge was also much drier than expected.

In summary, the analytical results from the 1995 rotary core sampling show that tank 241-BY-110 is safe when compared to the safety screening and ferrocyanide data quality objectives (Schreiber 1995 and 1996b). Although some exothermic activity was observed, the fuel content estimate based on the TOC and cyanide samples do not indicate excessive fuel sources are present. In addition, adequate moisture is present for samples exhibiting exothermic behavior, thereby reducing the potential for reaction propagation. The tank heat load and headspace flammability were both well below their established limits.

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APPENDIX A

STATISTICAL ANALYSIS RESULTS FROM 1995 CORE SAMPLING

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**Westinghouse
Hanford Company**
**Internal
Memo**

From: Process Chemistry and Statistics 75764-PCS96-062
 Phone: 373-4034T6-07
 Date: June 24, 1996
 Subject: STATISTICAL ANALYSIS FOR SINGLE-SHELL TANK 241-BY-110

To: R. D. Schreiber R2-12

cc: B. C. Simpson R2-12
 S. J. Eberlein R2-12
 J. G. Kristofzski R2-12
 J. W. Hunt R2-12
 J. R. Jewett T6-09
 L. Jensen T6-07
 RDC File/LB

- References: (1) Book, Statistical Sciences, Inc. S-PLUS Reference Manual, Version 3.2, Seattle: StatSci, a division of MathSoft, Inc., 1993.
- (2) Book, Snedecor, G. W., and W. G. Cochran, 1980, Statistical Methods, 7th Edition, Iowa State University Press, Ames, Iowa.
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Summary

This memo per your request provides mean concentration estimates, relative standard deviations (RSDs), and 95% confidence intervals (CIs) on the mean for each of the selected analytes. These statistical analysis were done for segment data and composite data from three riser samples obtained from single-shell tank 241-BY-110 (BY-110) The core segment data and core composite data were obtained from LABCORE.

The statistical analyses for the segment data were first done on combined salt cake and sludge results, then on salt cake results, and then on sludge results. The following segments were sludge samples and the remaining segments were salt cake samples:

Riser 4, Core 113, Segment 7 A-D and Segment 8 A-D,
 Riser 7, Core 101, Segment 8 C-D and Segment 8 A-D,
 Riser 7, Core 103, Segment 8 D and Segment 9 A-D,
 Riser 7, Core 107, Segment 8 A-B and Segment 9 A-D.

Hanford Operations and Engineering Contractor for the US Department of Energy

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This memo also provides estimates of spatial variability and estimates of the measurement variability from both the segment data and the composite data.

Results and Conclusions

The lower limit (LL) to a two-sided 95% confidence interval for the mean is

$$LL = \hat{\mu} - t_{(df,0.95)} * \hat{\sigma}_{\bar{\mu}},$$

and the upper limit (UL) to a two-sided 95% confidence interval for the mean is

$$UL = \hat{\mu} + t_{(df,0.95)} * \hat{\sigma}_{\bar{\mu}}.$$

In these equations, $\hat{\mu}$ is the estimate of the mean concentration, $\hat{\sigma}_{\bar{\mu}}$ is estimate of the standard deviation of the mean, and $t_{(df,0.95)}$ is the quantile from Student's t distribution with df degrees of freedom corresponding to the two-sided 95% confidence level. How $\hat{\mu}$, $\hat{\sigma}_{\bar{\mu}}$, and df were obtained are explained in detail in Attachment 2.

For the analytes of interest, Tables 1-4 in Attachment 1 lists the mean ($\hat{\mu}$), standard deviation associated with the mean ($\hat{\sigma}_{\bar{\mu}}$), the relative standard deviation of the mean (RSD) ($\hat{\sigma}_{\bar{\mu}}/\hat{\mu}$), the degrees of freedom (df), and the limits to a two-sided 95% confidence interval on the mean (95% LL and 95% UL).

Table 1, Attachment 1, lists the statistics for the combined salt cake and sludge segment data; Table 2, Attachment 1, lists the statistics for the salt cake segment data; Table 3, Attachment 1, lists the statistics for the sludge segment data; and Table 4, Attachment 1, lists the statistics for the composite data.

In order to obtain the statistics listed above, a statistical model is needed to account for the spatial and measurement variability. Estimates of variance components were determined from the analysis of variance (ANOVA). Also, a statistical test (F-Test) was performed to determine if each of the variance components were significantly different from zero. The p-values are the attained level of significance of the statistical test. A p-value less than 0.05 indicates that the variance component is significantly different from zero at the 0.05 level of significance. Statistical modeling is explained in more detail in Attachment 2.

For the combined salt cake and sludge segment data, the p-values from the test on riser-to-riser variability [$\sigma^2(R)$] were less than 0.05 for nine out of 41 analytes; the p-values from the test on the core within riser variability [$\sigma^2(C)$] were less than 0.05 for three of the 41 analytes; and the p-values from the test on the segment within core within riser variability [$\sigma^2(S)$] was less than 0.05 for 41 of the 41 analytes. The variance component estimates and p-values from the F-Tests are given in Table 1, Attachment 2.

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For the salt cake segment data, the p-values from the test on riser-to-riser variability [$\sigma^2(R)$] were less than 0.05 for five out of 41 analytes; the p-values from the test on the core within riser variability [$\sigma^2(C)$] were less than 0.05 for six of the 41 analytes; and the p-values from the test on the segment within core within riser variability [$\sigma^2(S)$] was less than 0.05 for 33 of the 41 analytes. The variance component estimates and p-values from the F-Tests are given in Table 2, Attachment 2.

For the sludge segment data, the p-values from the test on riser-to-riser variability [$\sigma^2(R)$] were less than 0.05 for six out of 40 analytes; the p-values from the test on the core within riser variability [$\sigma^2(C)$] were less than 0.05 for two of the 40 analytes; and the p-values from the test on the segment within core within riser variability [$\sigma^2(S)$] was less than 0.05 for 29 of the 40 analytes. The variance component estimates and p-values from the F-Tests are given in Table 3, Attachment 2.

For the composite data, the p-values from the test on riser-to-riser variability [$\sigma^2(R)$] were less than 0.05 for five out of 40 analytes; and the p-values from the test on the core within riser variability [$\sigma^2(C)$] were less than 0.05 for six of the 40 analytes. The variance component estimates and p-values from the F-Tests are given in Table 4, Attachment 2.

The data for this analysis is listed in the data package for BY-110.

If there are questions, call Ryan Cromar at 373-4034.

R. D. Cromar, Statistician
Process Chemistry and Statistics

rdc
Attachment 2

Table A-1-1. Summary Statistics and 95% Confidence Limits of the Mean (Combined Salt Cake and Sludge Segment Data). (2 sheets)

Analyte	Units	$\hat{\mu}$	$\hat{\sigma}_{\mu}$	RSD	df	95% LL	95% UL
% Water	%	2.39E+01	2.18E+00	9.12%	2	1.45E+01	3.33E+01
CN	µg/g	4.68E+01	2.23E+01	47.65%	2	0.00E+00	1.43E+02
Cs-137	µCi/g	7.08E+01	1.03E+01	14.49%	2	2.67E+01	1.15E+02
Density	g/mL	1.25E+00	7.63E-02	6.12%	2	9.19E-01	1.58E+00
DSC	Joules/g	3.47E+01	1.29E+01	37.24%	2	-2.09E+01	9.03E+01
Hexadecane	µg/g	4.93E+02	1.69E+01	3.44%	2	4.20E+02	5.66E+02
IC.Cl	µg/g	2.49E+03	2.99E+02	12.02%	2	1.20E+03	3.78E+03
IC.F	µg/g	5.26E+03	9.74E+02	18.51%	2	1.07E+03	9.45E+03
IC.NO2	µg/g	3.00E+04	3.80E+03	12.65%	2	1.37E+04	4.64E+04
IC.NO3	µg/g	1.82E+05	6.86E+04	37.63%	2	0.00E+00	4.78E+05
IC.Oxalate	µg/g	1.25E+04	3.05E+03	24.45%	2	0.00E+00	2.56E+04
IC.PO4	µg/g	1.16E+04	2.16E+03	18.60%	2	2.31E+03	2.09E+04
IC.SO4	µg/g	1.85E+04	5.15E+03	27.84%	2	0.00E+00	4.07E+04
ICP.a.Ag	µg/g	1.68E+01	3.26E+00	19.45%	2	2.73E+00	3.08E+01
ICP.a.Al	µg/g	1.57E+04	1.92E+03	12.21%	2	7.46E+03	2.40E+04
ICP.a.B	µg/g	8.69E+01	1.15E+01	13.26%	2	3.73E+01	1.37E+02
ICP.a.Ca	µg/g	2.39E+03	1.02E+03	42.77%	2	0.00E+00	6.79E+03
ICP.a.Cd	µg/g	1.93E+01	1.73E+00	8.96%	2	1.19E+01	2.68E+01
ICP.a.Cr	µg/g	2.75E+03	2.53E+02	9.20%	2	1.66E+03	3.83E+03
ICP.a.Fe	µg/g	3.66E+03	1.57E+03	42.99%	2	0.00E+00	1.04E+04
ICP.a.K	µg/g	2.04E+03	1.92E+02	9.39%	2	1.22E+03	2.87E+03
ICP.a.Mn	µg/g	7.77E+01	2.05E+01	26.37%	2	0.00E+00	1.66E+02
ICP.a.Na	µg/g	2.28E+05	6.67E+03	2.93%	2	1.99E+05	2.57E+05
ICP.a.Ni	µg/g	1.12E+03	4.86E+02	43.52%	2	0.00E+00	3.21E+03
ICP.a.P	µg/g	5.55E+03	9.59E+02	17.27%	2	1.43E+03	9.68E+03
ICP.a.Pb	µg/g	4.09E+02	1.68E+02	41.06%	2	0.00E+00	1.13E+03
ICP.a.S	µg/g	5.70E+03	1.31E+03	22.90%	2	8.27E+01	1.13E+04
ICP.a.Si	µg/g	5.58E+02	1.36E+02	24.40%	2	0.00E+00	1.14E+03
ICP.a.Sr	µg/g	1.14E+03	5.09E+02	44.53%	2	0.00E+00	3.33E+03

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 ATTACHMENT 1
 (8 sheets)

Table A-1-1. Summary Statistics and 95% Confidence Limits of the Mean (Combined Salt Cake and Sludge Segment Data). (2 sheets)

Analyte	Units	$\hat{\mu}$	$\hat{\sigma}_s$	RSD	df	95% LL	95% UL
ICP.a.U	μg/g	3.70E+03	1.38E+03	37.32%	2	0.00E+00	9.63E+03
ICP.a.Zn	μg/g	4.25E+01	8.42E+00	19.84%	2	6.21E+00	7.87E+01
ICP.a.Zr	μg/g	1.53E+01	1.89E+00	12.36%	2	7.17E+00	2.34E+01
ICP.f.Al	μg/g	1.86E+04	3.12E+03	16.83%	2	5.12E+03	3.20E+04
ICP.f.Fe	μg/g	3.43E+03	1.69E+03	49.31%	2	0.00E+00	1.07E+04
ICP.f.Na	μg/g	2.41E+05	7.53E+03	3.13%	2	2.08E+05	2.73E+05
ICP.f.Ni	μg/g	1.29E+03	5.41E+02	42.09%	2	0.00E+00	3.61E+03
Pu-239	μCi/g	3.50E-02	1.32E-02	37.78%	2	0.00E+00	9.19E-02
Sr-90	μCi/g	7.08E+01	2.81E+01	39.69%	2	0.00E+00	1.92E+02
Total Alpha	μCi/g	4.61E-02	1.08E-02	23.47%	2	0.00E+00	9.26E-02
TIC	μg/g	2.81E+04	6.19E+03	22.01%	2	1.49E+03	5.48E+04
TOC-Dry	μg/g Dry	6.66E+03	1.30E+03	19.50%	2	1.07E+03	1.22E+04

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ATTACHMENT 1
(8 sheets)

Table A-1-2. Summary Statistics and 95% Confidence Limits of the Mean
(Salt Cake Segment Data). (2 sheets)

Analyte	Units	$\hat{\mu}$	$\hat{\sigma}_x$	RSD	df	95% LL	95% UL
% Water	%	2.32E+01	1.79E+00	7.72%	2	1.55E+01	3.09E+01
CN	$\mu\text{g/g}$	1.63E+01	7.61E+00	46.83%	2	0.00E+00	4.90E+01
Cs-137	$\mu\text{Ci/g}$	6.00E+01	7.89E+00	13.15%	2	2.60E+01	9.39E+01
DSC	Joules/g	2.71E+01	8.22E+00	30.34%	2	-8.30E+00	6.25E+01
Density	g/mL	1.44E+00	4.28E-02	2.98%	2	1.25E+00	1.62E+00
Hexadecane	$\mu\text{g/g}$	4.73E+02	1.72E+01	3.65%	2	3.99E+02	5.47E+02
IC.Cl	$\mu\text{g/g}$	2.25E+03	4.60E+02	20.50%	2	2.65E+02	4.23E+03
IC.F	$\mu\text{g/g}$	5.42E+03	1.17E+03	21.66%	2	3.68E+02	1.05E+04
IC.NO2	$\mu\text{g/g}$	3.06E+04	5.69E+03	18.59%	2	6.12E+03	5.51E+04
IC.NO3	$\mu\text{g/g}$	1.84E+05	6.84E+04	37.24%	2	0.00E+00	4.78E+05
IC.Oxalate	$\mu\text{g/g}$	1.36E+04	3.84E+03	28.28%	2	0.00E+00	3.01E+04
IC.PO4	$\mu\text{g/g}$	1.15E+04	2.41E+03	20.94%	2	1.14E+03	2.19E+04
IC.SO4	$\mu\text{g/g}$	1.84E+04	5.94E+03	32.26%	2	0.00E+00	4.40E+04
ICP.a.Ag	$\mu\text{g/g}$	1.75E+01	3.24E+00	18.54%	2	3.54E+00	3.15E+01
ICP.a.Al	$\mu\text{g/g}$	1.41E+04	1.89E+03	13.42%	2	5.95E+03	2.22E+04
ICP.a.B	$\mu\text{g/g}$	9.23E+01	1.24E+01	13.41%	2	3.90E+01	1.46E+02
ICP.a.Ca	$\mu\text{g/g}$	4.00E+02	7.13E+01	17.82%	2	9.33E+01	7.06E+02
ICP.a.Cd	$\mu\text{g/g}$	2.11E+01	1.97E+00	9.35%	2	1.26E+01	2.96E+01
ICP.a.Cr	$\mu\text{g/g}$	2.90E+03	3.30E+02	11.39%	2	1.48E+03	4.32E+03
ICP.a.Fe	$\mu\text{g/g}$	9.24E+02	2.21E+02	23.93%	2	0.00E+00	1.88E+03
ICP.a.K	$\mu\text{g/g}$	1.93E+03	2.11E+02	10.93%	2	1.02E+03	2.84E+03
ICP.a.Mn	$\mu\text{g/g}$	5.28E+01	1.03E+01	19.52%	2	8.46E+00	9.72E+01
ICP.a.Na	$\mu\text{g/g}$	2.37E+05	7.09E+03	2.99%	2	2.06E+05	2.67E+05
ICP.a.Ni	$\mu\text{g/g}$	1.93E+02	4.49E+01	23.31%	2	0.00E+00	3.86E+02
ICP.a.P	$\mu\text{g/g}$	4.65E+03	1.00E+03	21.58%	2	3.33E+02	8.96E+03
ICP.a.Pb	$\mu\text{g/g}$	1.30E+02	2.35E+01	18.01%	2	2.94E+01	2.32E+02
ICP.a.S	$\mu\text{g/g}$	5.95E+03	1.46E+03	24.54%	2	0.00E+00	1.22E+04
ICP.a.Si	$\mu\text{g/g}$	4.51E+02	8.47E+01	18.78%	2	8.67E+01	8.16E+02
ICP.a.Sr	$\mu\text{g/g}$	5.81E+01	9.30E+00	15.99%	2	1.81E+01	9.81E+01

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Table A-1-2. Summary Statistics and 95% Confidence Limits of the Mean
 (Salt Cake Segment Data). (2 sheets)

Analyte	Units	$\hat{\mu}$	$\hat{\sigma}_{\mu}$	RSD	df	95% LL	95% UL
ICP.a.U	$\mu\text{g/g}$	6.97E+02	1.16E+02	16.65%	2	1.98E+02	1.20E+03
ICP.a.Zn	$\mu\text{g/g}$	3.28E+01	4.37E+00	13.32%	2	1.40E+01	5.16E+01
ICP.a.Zr	$\mu\text{g/g}$	1.44E+01	2.09E+00	14.55%	2	5.38E+00	2.34E+01
ICP.f.Al	$\mu\text{g/g}$	1.63E+04	2.73E+03	16.74%	2	4.55E+03	2.80E+04
ICP.f.Fe	$\mu\text{g/g}$	1.28E+03	2.57E+02	20.10%	2	1.72E+02	2.38E+03
ICP.f.Na	$\mu\text{g/g}$	2.53E+05	6.76E+03	2.67%	2	2.24E+05	2.82E+05
ICP.f.Ni	$\mu\text{g/g}$	5.47E+02	9.07E+01	16.60%	2	1.56E+02	9.37E+02
Pu-239	$\mu\text{Ci/g}$	1.92E-02	5.45E-03	28.33%	2	0.00E+00	4.27E-02
Sr-90	$\mu\text{Ci/g}$	2.25E+01	4.93E+00	21.93%	2	1.27E+00	4.37E+01
Total Alpha	$\mu\text{Ci/g}$	4.34E-02	1.08E-02	24.86%	2	0.00E+00	8.98E-02
TIC	$\mu\text{g/g}$	3.18E+04	7.79E+03	24.49%	2	0.00E+00	6.53E+04
TOC-Dry	$\mu\text{g/g Dry}$	5.92E+03	9.66E+02	16.32%	2	1.76E+03	1.01E+04

Table A-1-3. Summary Statistics and 95% Confidence Limits of the Mean
(Sludge Segment Data). (2 sheets)

Analyte	Units	$\bar{\mu}$	$\hat{\sigma}_s$	RSD	df	95% LL	95% UL
% Water	%	3.05E+01	2.31E+00	7.59%	1	1.08E+00	5.99E+01
CN	$\mu\text{g/g}$	9.85E+01	5.67E+01	57.62%	1	0.00E+00	8.19E+02
Cs-137	$\mu\text{Ci/g}$	1.40E+02	1.26E+01	9.01%	1	0.00E+00	2.99E+02
DSC	Joules/g	7.88E+01	4.61E+01	58.51%	1	-5.07E+02	6.65E+02
Density	g/mL	1.78E+00	3.95E-02	2.22%	1	1.28E+00	2.28E+00
Hexadecane	$\mu\text{g/g}$	5.81E+02	4.28E+01	7.38%	1	3.65E+01	1.13E+03
IC.Cl	$\mu\text{g/g}$	3.57E+03	9.28E+02	25.98%	1	0.00E+00	1.54E+04
IC.F	$\mu\text{g/g}$	4.22E+03	7.66E+02	18.13%	1	0.00E+00	1.40E+04
IC.NO2	$\mu\text{g/g}$	4.32E+04	1.83E+03	4.23%	1	2.00E+04	6.64E+04
IC.NO3	$\mu\text{g/g}$	1.11E+05	6.21E+03	5.62%	1	3.16E+04	1.89E+05
IC.Oxalate	$\mu\text{g/g}$	5.87E+03	1.46E+03	24.88%	1	0.00E+00	2.44E+04
IC.PO4	$\mu\text{g/g}$	1.12E+04	3.34E+03	29.88%	1	0.00E+00	5.37E+04
IC.SO4	$\mu\text{g/g}$	1.84E+04	2.75E+03	14.93%	1	0.00E+00	5.34E+04
ICP.a.Ag	$\mu\text{g/g}$	1.02E+01	1.43E+00	14.03%	1	0.00E+00	2.84E+01
ICP.a.Al	$\mu\text{g/g}$	2.83E+04	1.19E+03	4.20%	1	1.32E+04	4.34E+04
ICP.a.B	$\mu\text{g/g}$	3.98E+01	1.04E+01	26.05%	1	0.00E+00	1.71E+02
ICP.a.Ca	$\mu\text{g/g}$	1.42E+04	2.46E+03	17.39%	1	0.00E+00	4.55E+04
ICP.a.Cd	$\mu\text{g/g}$	7.40E+00	1.52E+00	20.52%	1	0.00E+00	2.67E+01
ICP.a.Cr	$\mu\text{g/g}$	2.22E+03	5.98E+02	26.97%	1	0.00E+00	9.82E+03
ICP.a.Fe	$\mu\text{g/g}$	2.00E+04	4.54E+03	22.66%	1	0.00E+00	7.78E+04
ICP.a.K	$\mu\text{g/g}$	2.93E+03	2.47E+02	8.43%	1	0.00E+00	6.07E+03
ICP.a.Mn	$\mu\text{g/g}$	2.28E+02	5.22E+01	22.90%	1	0.00E+00	8.91E+02
ICP.a.Na	$\mu\text{g/g}$	1.61E+05	8.79E+03	5.47%	1	4.90E+04	2.72E+05
ICP.a.Ni	$\mu\text{g/g}$	6.67E+03	1.38E+03	20.65%	1	0.00E+00	2.42E+04
ICP.a.P	$\mu\text{g/g}$	1.05E+04	1.84E+03	17.50%	1	0.00E+00	3.39E+04
ICP.a.Pb	$\mu\text{g/g}$	1.88E+03	1.02E+03	54.45%	1	0.00E+00	1.49E+04
ICP.a.S	$\mu\text{g/g}$	5.36E+03	7.22E+02	13.48%	1	0.00E+00	1.45E+04
ICP.a.Si	$\mu\text{g/g}$	1.19E+03	2.20E+02	18.41%	1	0.00E+00	3.99E+03
ICP.a.Sr	$\mu\text{g/g}$	6.84E+03	2.17E+03	31.71%	1	0.00E+00	3.44E+04

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Table A-1-3. Summary Statistics and 95% Confidence Limits of the Mean
 (Sludge Segment Data). (2 sheets)

Analyte	Units	$\hat{\mu}$	$\hat{\sigma}_{\mu}$	RSD	df	95% LL	95% UL
ICP.a.U	$\mu\text{g/g}$	2.09E+04	4.69E+03	22.40%	1	0.00E+00	8.06E+04
ICP.a.Zn	$\mu\text{g/g}$	9.16E+01	1.92E+01	20.96%	1	0.00E+00	3.35E+02
ICP.a.Zr	$\mu\text{g/g}$	1.97E+01	2.87E+00	14.57%	1	0.00E+00	5.62E+01
ICP.f.Al	$\mu\text{g/g}$	3.17E+04	3.26E+03	10.27%	1	0.00E+00	7.31E+04
ICP.f.Fe	$\mu\text{g/g}$	1.55E+04	9.10E+03	58.70%	1	0.00E+00	1.31E+05
ICP.f.Na	$\mu\text{g/g}$	1.63E+05	9.57E+03	5.86%	1	4.18E+04	2.85E+05
ICP.f.Ni	$\mu\text{g/g}$	5.44E+03	2.73E+03	50.19%	1	0.00E+00	4.01E+04
Pu-239	$\mu\text{Ci/g}$	6.08E-02	3.82E-02	62.77%	1	0.00E+00	5.46E-01
Sr-90	$\mu\text{Ci/g}$	3.48E+02	6.26E+01	17.99%	1	0.00E+00	1.14E+03
TIC	$\mu\text{g/g}$	6.43E+03	1.13E+03	17.63%	1	0.00E+00	2.08E+04
TOC-Dry	$\mu\text{g/g Dry}$	1.11E+04	1.58E+03	14.27%	1	0.00E+00	3.12E+04

Table 1-1-4. Summary Statistics and 95% Confidence Limits of the Mean
(Composite Data). (sheet 1 of 2)

Analyte	Units	μ	$\hat{\sigma}_x$	RSD	df	95% LL	95% UL
% Water	%	2.05E+01	5.02E+00	24.44%	2	0.00E+00	4.21E+01
Cs-137	$\mu\text{Ci/g}$	6.24E+01	1.38E+01	22.15%	2	2.93E+00	1.22E+02
DSC	Joules/g	5.98E+01	2.41E+01	40.30%	2	-1.04E+02	1.64E+02
Density	g/mL	1.49E+00	9.35E-02	6.27%	2	1.09E+00	1.89E+00
IC.Cl	$\mu\text{g/g}$	1.74E+03	2.75E+02	15.74%	2	5.63E+02	2.93E+03
IC.F	$\mu\text{g/g}$	5.37E+03	1.31E+03	24.47%	2	0.00E+00	1.10E+04
IC.NO2	$\mu\text{g/g}$	2.47E+04	4.71E+03	19.05%	2	4.45E+03	4.50E+04
IC.NO3	$\mu\text{g/g}$	1.98E+05	1.02E+05	51.81%	2	0.00E+00	6.39E+05
IC.Oxalate	$\mu\text{g/g}$	1.39E+04	3.84E+03	27.70%	2	0.00E+00	3.04E+04
IC.PO4	$\mu\text{g/g}$	9.48E+03	1.13E+03	11.93%	2	4.61E+03	1.43E+04
IC.SO4	$\mu\text{g/g}$	2.21E+04	5.98E+03	27.07%	2	0.00E+00	4.78E+04
ICP.a.Ag	$\mu\text{g/g}$	2.19E+01	2.11E+00	9.66%	2	1.28E+01	3.10E+01
ICP.a.Al	$\mu\text{g/g}$	1.89E+04	4.36E+03	23.01%	2	1.85E+02	3.77E+04
ICP.a.B	$\mu\text{g/g}$	1.11E+02	1.20E+01	10.80%	2	5.96E+01	1.63E+02
ICP.a.Ca	$\mu\text{g/g}$	2.42E+03	8.41E+02	34.77%	2	0.00E+00	6.04E+03
ICP.a.Cd	$\mu\text{g/g}$	1.68E+01	5.16E+00	30.75%	2	0.00E+00	3.90E+01
ICP.a.Cr	$\mu\text{g/g}$	2.44E+03	6.84E+02	28.00%	2	0.00E+00	5.39E+03
ICP.a.Fe	$\mu\text{g/g}$	4.19E+03	2.16E+03	51.45%	2	0.00E+00	1.35E+04
ICP.a.K	$\mu\text{g/g}$	1.70E+03	3.61E+02	21.31%	2	1.41E+02	3.25E+03
ICP.a.Mn	$\mu\text{g/g}$	6.85E+01	2.85E+01	41.64%	2	0.00E+00	1.91E+02
ICP.a.Na	$\mu\text{g/g}$	2.36E+05	1.19E+04	5.04%	2	1.85E+05	2.87E+05
ICP.a.Ni	$\mu\text{g/g}$	1.08E+03	4.36E+02	40.37%	2	0.00E+00	2.95E+03
ICP.a.P	$\mu\text{g/g}$	3.32E+03	1.11E+03	33.62%	2	0.00E+00	8.11E+03
ICP.a.Pb	$\mu\text{g/g}$	1.74E+02	5.55E+01	31.84%	2	0.00E+00	4.13E+02
ICP.a.S	$\mu\text{g/g}$	5.84E+03	2.02E+03	34.54%	2	0.00E+00	1.45E+04
ICP.a.Si	$\mu\text{g/g}$	5.45E+02	1.87E+02	34.27%	2	0.00E+00	1.35E+03
ICP.a.Sr	$\mu\text{g/g}$	8.50E+02	3.35E+02	39.38%	2	0.00E+00	2.29E+03
ICP.a.U	$\mu\text{g/g}$	3.15E+03	1.23E+03	39.07%	2	0.00E+00	8.44E+03
ICP.a.Zn	$\mu\text{g/g}$	3.26E+01	6.10E+00	18.71%	2	6.35E+00	5.89E+01

Table 1-1-4. Summary Statistics and 95% Confidence Limits of the Mean
 (Composite Data). (sheet 1 of 2)

Analyte	Units	μ	σ_x	RSD	df	95% LL	95% UL
ICP. a. Zr	$\mu\text{g/g}$	1.19E+01	3.88E+00	32.49%	2	0.00E+00	2.87E+01
ICP. f. Al	$\mu\text{g/g}$	1.93E+04	4.43E+03	22.96%	2	2.28E+02	3.83E+04
ICP. f. Fe	$\mu\text{g/g}$	4.87E+03	1.80E+03	37.02%	2	0.00E+00	1.26E+04
ICP. f. Na	$\mu\text{g/g}$	2.53E+05	1.04E+04	4.13%	2	2.08E+05	2.97E+05
ICP. f. Ni	$\mu\text{g/g}$	1.72E+03	5.74E+02	33.39%	2	0.00E+00	4.19E+03
Sr-90	$\mu\text{Ci/g}$	8.47E+01	3.65E+01	43.12%	2	0.00E+00	2.42E+02
Total Alpha	$\mu\text{Ci/g}$	6.37E-02	2.47E-02	38.81%	2	0.00E+00	1.70E-01
Total Beta	$\mu\text{Ci/g}$	2.45E+02	9.17E+01	37.40%	2	0.00E+00	6.40E+02
TIC	$\mu\text{g/g}$	3.13E+04	7.62E+03	24.33%	2	0.00E+00	6.41E+04
TOC-Dry	$\mu\text{g/g Dry}$	6.46E+03	2.52E+03	39.06%	2	0.00E+00	1.73E+04
U-Phosphorescence	$\mu\text{Ci/g}$	3.20E+03	1.34E+03	41.81%	2	0.00E+00	8.95E+03

Statistical Modeling

A statistical model is needed to account for the spatial and measurement variability in $\hat{\sigma}_\mu$. This cannot be done using an ordinary standard deviation of the data (see Reference 2, Chapter 13).

The statistical model used to describe the structure of the segment data is

$$Y_{ijkl} = \mu + R_i + C_{ij} + S_{ijk} + A_{ijkl},$$

$$i = 1, \dots, a, j = 1, \dots, b_i, k = 1, \dots, c_{ij}, l = 1, \dots, n_{ijk},$$

where

Y_{ijkl}	=	concentration from the l^{th} analytical result of the k^{th} segment of the j^{th} core sample from the i^{th} riser
μ	=	the grand mean
R_i	=	the effect of the i^{th} riser
C_{ij}	=	the effect of the j^{th} core from the i^{th} riser
S_{ijk}	=	the effect of the k^{th} segment in the j^{th} core from the i^{th} riser
A_{ijkl}	=	the effect of the l^{th} analytical result from the k^{th} segment in the j^{th} core from the i^{th} riser
a	=	the number of risers sampled
b_i	=	the number of cores taken from the i^{th} riser
c_{ij}	=	the number of segments in the j^{th} core from the i^{th} riser
n_{ijk}	=	the number of analytical results in the k^{th} segment in the j^{th} core from the i^{th} riser

The variables R_i , C_{ij} , and S_{ijk} are assumed to be random effects. These variables, as well as A_{ijkl} , are assumed to be uncorrelated and normally distributed with means zero and

variances $\sigma^2(R)$, $\sigma^2(C)$, $\sigma^2(S)$, and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(R)$, $\sigma^2(C)$, $\sigma^2(S)$, and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML) techniques. This method applied to variance component estimation is described in Reference 3. The results using the REML techniques were obtained using the statistical analysis package S-PLUS (see Reference 1).

For BY-110, there are three risers ($a = 3$). For riser 4, there was one core ($b_1 = 1$) and for risers 7 and 12 B, there were four cores ($b_2 = b_3 = 4$). The number of segments varied within each of the cores and the number of sub-segments varied within each segment. However, since the sub-segments were in different sample sizes (e.g. whole segments, half segments, and quarter segments), all of the sub-segments were pooled within each segment. Thus, the number of analytical results varied within each segment.

The statistical model used to describe the structure of composite data is

$$Y_{ijk} = \mu + R_i + C_{ij} + A_{ijk},$$

$$i = 1, \dots, a, j = 1, \dots, b_i, k = 1, \dots, n_{ij},$$

where

- Y_{ijk} = concentration from the i^{th} analytical result of the j^{th} core sample from the i^{th} riser
- μ = the grand mean
- R_i = the effect of the i^{th} riser
- C_{ij} = the effect of the j^{th} core from the i^{th} riser
- A_{ijk} = the effect of the k^{th} analytical result in the j^{th} core from the i^{th} riser
- a = the number of risers sampled
- b_i = the number of cores taken from the i^{th} riser
- n_{ij} = the number of analytical results in the j^{th} core from the i^{th} riser

The variables R_i and C_{ij} are assumed to be random effects. These variables, as well as A_{ijk} , are assumed to be uncorrelated and normally distributed with means zero and variances $\sigma^2(R)$, $\sigma^2(C)$, and $\sigma^2(A)$, respectively. Estimates of $\sigma^2(R)$, $\sigma^2(C)$, and $\sigma^2(A)$ were obtained using Restricted Maximum Likelihood Estimation (REML) techniques. This method applied to variance component estimation is described in Reference 3. The results using the REML techniques were obtained using the statistical analysis package S-PLUS (see Reference 1).

For BY-110, there are three risers ($a = 3$). For riser 4, there was one core ($b_1 = 1$) and for risers 7 and 12 B, there were four cores ($b_2 = b_3 = 4$). The number of analytical results varied within each core.

The following paragraphs describe how the mean ($\hat{\mu}$), standard deviation of the mean ($\hat{\sigma}_{\hat{\mu}}$), and the degrees of freedom (df) were determined using the results from the statistical models fit to the data.

Mean

The estimator of $\hat{\mu}$ is the maximum likelihood estimate of the mean. This estimator was determined by the structure of the data reflected by the statistical model. The estimate of $\hat{\mu}$ was obtained using Restricted Maximum Likelihood Estimation (REML) techniques in S-PLUS.

Standard Deviation of the Mean

The estimated standard deviation of the mean, $\hat{\sigma}_{\hat{\mu}}$, is the square root of a linear combination of the variance estimates $\sigma^2(R)$, $\sigma^2(C)$, $\sigma^2(S)$, and $\sigma^2(A)$ for segment data and $\sigma^2(R)$, $\sigma^2(C)$, and $\sigma^2(A)$ for core composite data. These estimates were obtained using the Restricted Maximum Likelihood Estimation (REML) techniques. For unbalanced data, $\hat{\sigma}_{\hat{\mu}}$ is a more complicated linear combination of these variances.

$\hat{\sigma}_{\hat{\mu}}$ is the standard deviation of the mean associated with the maximum likelihood estimate of the mean.

Degrees of Freedom

The degrees of freedom (df) are dependent on the data structure or the statistical model used. The df for 241-BY-110 are number of risers minus one.

Table A-2-1. Variance Component Estimates
(Combined Salt Cake and Sludge Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
% Water	7.53E+00	0.3084	6.49E-12	0.3906	8.13E+01	0.0001	6.18E+01
CN	5.43E-20	0.5071	6.40E-15	0.6842	7.19E+03	0.0001	1.32E+04
Cs-137	1.07E+02	0.2539	1.23E-16	0.5530	3.03E+03	0.0001	4.68E+02
Density	2.72E-23	0.6531	4.05E-25	0.7838	2.63E-01	0.0001	2.44E-02
DSC	2.88E+02	0.3009	1.75E-21	0.8230	1.75E+03	0.0001	4.86E+03
Hexadecane	3.90E-24	0.5140	1.99E-22	0.8895	7.51E+03	0.0002	2.17E+04
IC.Cl	1.79E+04	0.1746	6.00E-26	0.7740	1.84E+06	0.0025	8.12E+06
IC.F	2.10E+06	0.1286	5.13E+05	0.1671	5.55E+06	0.0001	5.01E+06
IC.NO2	2.14E+06	0.3101	1.76E-24	0.4806	5.97E+08	0.0001	2.89E+08
IC.NO3	1.26E+10	0.0199	1.36E+09	0.0970	1.02E+10	0.0001	2.49E+09
IC.Oxalate	2.10E+07	0.1006	3.79E-21	0.9368	7.00E+07	0.0001	8.62E+07
IC.PO4	7.19E-28	0.7211	5.49E-22	0.3657	2.11E+08	0.0001	8.10E+07
IC.SO4	7.18E+07	0.0141	1.32E-20	0.5764	8.40E+07	0.0001	6.70E+07
ICP.a.Ag	1.15E+01	0.3429	3.85E+01	0.0065	5.45E+01	0.0001	6.81E+01
ICP.a.Al	2.13E-14	0.3260	1.05E+07	0.1327	1.03E+08	0.0001	2.82E+07
ICP.a.B	2.61E+01	0.5991	5.37E+02	0.0251	2.03E+03	0.0001	1.79E+03
ICP.a.Ca	1.04E+06	0.0000	9.47E-53	0.9961	2.83E+07	0.0001	9.66E+06
ICP.a.Cd	5.27E-26	0.8853	1.12E-13	0.1737	1.17E+02	0.0001	8.90E+01
ICP.a.Cr	2.25E-14	0.8047	3.61E-14	0.5811	2.43E+06	0.0001	2.15E+06
ICP.a.Fe	2.59E+06	0.9271	4.95E-75	0.9894	6.60E+07	0.0001	1.82E+07
ICP.a.K	2.18E-22	0.2229	3.80E-21	0.6295	1.66E+06	0.0001	4.15E+05
ICP.a.Mn	6.15E+02	0.3048	1.09E-15	0.8314	7.20E+03	0.0001	6.55E+03
ICP.a.Na	1.85E+07	0.2592	2.46E-27	0.9673	1.48E+09	0.0001	1.03E+09
ICP.a.Ni	2.13E+05	0.0000	4.80E-53	0.9951	6.93E+06	0.0001	1.80E+06
ICP.a.P	2.71E-14	0.7373	3.65E-22	0.8004	4.07E+07	0.0001	1.29E+07
ICP.a.Pb	1.74E-11	0.5769	2.27E+04	0.8019	1.10E+06	0.0001	3.97E+05
ICP.a.S	4.61E+06	0.0212	3.17E-26	0.7144	4.92E+06	0.0001	5.29E+06

Table A-2-1. Variance Component Estimates
(Combined Salt Cake and Sludge Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\hat{\sigma}^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\hat{\sigma}^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\hat{\sigma}^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.a.Si	3.05E+04	0.3786	3.02E+04	0.2680	1.53E+05	0.0001	5.54E+04
ICP.a.Sr	7.66E-06	0.0000	1.39E-54	0.9950	1.14E+07	0.0001	3.90E+06
ICP.a.U	6.52E+05	0.0000	2.13E-49	0.9956	7.47E+07	0.0001	1.69E+07
ICP.a.Zn	9.03E+01	0.2665	1.83E-18	0.8773	1.24E+03	0.0001	1.74E+03
ICP.a.Zr	1.61E-16	0.9171	1.71E+01	0.0418	3.74E+01	0.0001	9.43E+01
ICP.f.Al	1.78E+07	0.1438	9.83E-05	0.2416	1.50E+08	0.0001	3.79E+07
ICP.f.Fe	4.11E+06	0.4750	3.43E-71	0.9874	5.60E+07	0.0001	2.96E+07
ICP.f.Na	1.15E-13	0.3243	3.19E-23	0.8270	2.68E+09	0.0001	4.71E+08
ICP.f.Ni	3.95E+05	0.0000	9.26E-55	0.9963	5.95E+06	0.0001	3.65E+06
Pu-239	2.26E-04	0.7923	3.87E-24	0.9157	1.45E-03	0.0001	1.15E-03
Sr-90	1.03E+03	0.0000	8.38E-50	0.9970	1.53E+04	0.0001	1.43E+04
Total Alpha	1.58E-04	0.2632	1.02E-04	0.4413	1.69E-03	0.0001	2.50E-04
TIC	7.84E+07	0.0510	4.74E-18	0.7988	4.78E+08	0.0001	1.03E+08
TOC-Dry	4.06E+06	0.2274	7.07E-15	0.3814	7.01E+06	0.0012	2.32E+07

Table A-2-2. Variance Component Estimates
(Salt Cake Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
% Water	7.41E-11	0.2858	5.47E+00	0.5064	9.21E+01	0.0001	7.03E+01
CN	1.89E-16	0.3859	2.33E-25	0.8163	8.42E+02	0.0001	7.70E+01
Cs-137	1.45E-17	0.3844	5.47E-25	0.6537	2.56E+03	0.0001	5.01E+02
DSC	1.15E+02	0.3396	4.66E-11	0.6170	7.09E+02	0.0001	1.41E+03
Density	9.27E-17	0.4858	1.00E-25	0.4046	5.91E-02	0.0001	1.25E-02
Hexadecane	1.96E-18	0.6023	8.20E-21	0.9696	7.13E+03	0.0005	1.70E+04
IC.Cl	2.72E+05	0.0760	2.21E-30	0.8874	4.40E+06	0.0001	1.84E+06
IC.F	3.09E+06	0.0858	1.09E+06	0.0741	5.48E+06	0.0001	3.81E+06
IC.NO2	3.53E+07	0.1427	4.99E-21	0.6872	7.55E+08	0.0001	3.26E+08
IC.NO3	1.23E+10	0.0232	1.62E+09	0.1774	1.12E+10	0.0001	3.21E+09
IC.Oxalate	3.76E+07	0.0341	1.72E-28	0.7245	4.51E+07	0.0003	1.13E+08
IC.PO4	1.99E-17	0.6489	5.61E-18	0.4898	2.55E+08	0.0001	3.15E+07
IC.SO4	9.64E+07	0.0085	1.65E+06	0.4392	8.64E+07	0.0001	5.25E+07
ICP.a.Ag	8.80E+00	0.3214	4.31E+01	0.0122	4.99E+01	0.0001	9.52E+01
ICP.a.Al	3.33E-16	0.4071	9.03E+06	0.1833	9.14E+07	0.0001	3.34E+07
ICP.a.B	1.76E-17	0.6049	7.88E+02	0.0146	1.56E+03	0.0001	2.49E+03
ICP.a.Ca	3.97E+03	0.4871	1.14E+04	0.0530	8.12E+04	0.0001	2.47E+04
ICP.a.Cd	7.32E-13	0.8145	6.16E+00	0.1305	8.94E+01	0.0001	1.21E+02
ICP.a.Cr	4.13E-20	0.7002	3.18E+05	0.2993	2.04E+06	0.0001	2.55E+06
ICP.a.Fe	6.67E+04	0.2407	5.72E+04	0.4824	2.09E+05	0.0919	1.50E+06
ICP.a.K	3.35E-18	0.1882	9.08E-28	0.7859	1.72E+06	0.0001	5.08E+05
ICP.a.Mn	2.67E-11	0.5529	4.59E+02	0.2287	2.18E+02	0.9888	5.76E+03
ICP.a.Na	7.66E+07	0.0699	1.19E-23	0.8416	5.08E+08	0.0004	1.22E+09
ICP.a.Ni	1.77E+03	0.3950	7.40E+03	0.0651	1.20E+03	0.9898	5.31E+04
ICP.a.P	5.38E-27	0.8941	8.47E-28	0.7166	4.19E+07	0.0001	3.68E+06
ICP.a.Pb	1.50E-18	0.7501	3.33E+03	0.0074	3.26E+03	0.0010	9.31E+03
ICP.a.S	5.57E+06	0.0193	6.34E+05	0.2233	4.10E+06	0.0001	4.57E+06
ICP.a.Si	5.75E+03	0.5172	2.97E+04	0.0041	4.88E+04	0.0001	3.06E+04

Table A-2-2. Variance Component Estimates
 (Salt Cake Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\hat{\sigma}^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\hat{\sigma}^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\hat{\sigma}^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.a.Sr	1.56E+02	0.2276	4.55E+01	0.2664	5.35E+02	0.0002	1.37E+03
ICP.a.U	6.00E-19	0.7778	8.88E+04	0.0059	1.10E+04	0.1964	3.39E+05
ICP.a.Zn	8.52E-01	0.4912	1.01E-03	0.5600	1.50E+02	0.1975	2.07E+03
ICP.a.Zr	1.46E-14	0.7973	2.16E+01	0.0428	4.61E+01	0.0001	7.98E+01
ICP.f.Al	1.01E+07	0.2030	5.39E-15	0.3278	1.40E+08	0.0001	4.69E+07
ICP.f.Fe	8.95E+04	0.2485	1.22E+05	0.2275	1.12E+04	0.9499	2.31E+06
ICP.f.Na	1.84E-23	0.3360	6.27E-16	0.6233	1.90E+09	0.0001	2.97E+08
ICP.f.Ni	7.53E-10	0.4536	7.35E-18	0.8931	2.96E+05	0.0001	1.84E+05
Pu-239	3.43E-31	0.8543	2.65E-05	0.0771	2.92E-04	0.0020	2.61E-04
Sr-90	2.44E+01	0.2546	7.52E+01	0.1406	6.04E+01	0.3051	5.62E+02
Total Alpha	2.63E-04	0.0763	5.95E-28	0.6824	8.12E-04	0.0001	2.14E-04
TIC	1.44E+08	0.0142	9.87E-31	0.8970	3.85E+08	0.0001	1.43E+08
TOC-Dry	1.50E+06	0.3112	1.09E+06	0.3490	4.18E+06	0.0686	2.03E+07

Table A-2-3. Variance Component Estimates
(Sludge Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
% Water	7.35E+00	0.2852	2.10E-15	0.6518	5.91E+00	0.1259	3.34E+01
CN	3.35E-20	0.5957	5.17E+03	0.2640	1.01E+04	0.1419	2.88E+04
Cs-137	2.44E+02	0.1473	3.97E-16	0.8065	1.80E+02	0.0007	3.12E+02
DSC	2.26E+03	0.3727	1.02E+02	0.3976	4.28E+03	0.0959	1.51E+04
Density	9.95E-31	0.5770	4.95E-04	0.6236	2.58E-03	0.3836	1.09E-02
Hexadecane	1.62E-22	0.8012	1.33E+03	0.2903	6.48E+03	0.0810	3.12E+04
IC.Cl	4.02E-21	0.8856	1.84E+06	0.0014	4.34E-23	0.9724	2.06E+07
IC.F	5.60E-19	0.0000	1.72E-23	0.9808	3.58E+06	0.0008	6.05E+06
IC.NO2	9.18E-20	0.6901	1.70E-22	0.6224	2.13E+07	0.0002	2.91E+07
IC.NO3	2.98E-13	0.0000	6.93E-24	0.9272	1.82E+08	0.0119	7.21E+08
IC.Oxalate	1.91E-17	0.9188	5.51E-24	0.8443	1.30E+07	0.0001	2.19E+07
IC.PO4	1.31E-23	0.5386	1.71E-17	0.6523	5.46E+07	0.1488	1.99E+08
IC.SO4	3.98E+06	0.0000	5.55E-28	0.9042	2.74E+07	0.0216	8.41E+07
ICP.a.Ag	6.51E-32	0.5228	3.50E-01	0.4125	1.51E+01	0.0001	3.02E+00
ICP.a.Al	1.10E+06	0.4276	1.57E-11	0.4373	4.02E+06	0.0626	1.28E+07
ICP.a.B	2.05E+02	0.1314	4.16E-23	0.3815	1.39E-21	0.3898	2.15E+02
ICP.a.Ca	1.05E-20	0.9959	6.02E-25	0.9805	4.39E+07	0.0001	2.38E+07
ICP.a.Cd	7.84E-12	0.8197	8.74E-13	0.8355	1.53E+01	0.0001	1.64E+01
ICP.a.Cr	1.06E-21	0.3470	3.13E-27	0.9693	2.68E+06	0.0001	9.34E+05
ICP.a.Fe	8.28E-12	0.2419	1.01E-20	0.9168	1.57E+08	0.0001	4.19E+07
ICP.a.K	6.18E-23	0.0000	1.62E-25	0.9982	4.52E+05	0.0001	1.90E+05
ICP.a.Mn	7.57E-17	0.2511	2.09E-21	0.9291	2.08E+04	0.0001	5.12E+03
ICP.a.Na	1.20E-28	0.3220	1.39E-27	0.9099	5.23E+08	0.0001	4.95E+08
ICP.a.Ni	5.90E-25	0.0000	2.09E-33	0.9990	1.44E+07	0.0001	3.87E+06
ICP.a.P	3.62E-19	0.0000	3.83E-26	0.9741	2.08E+07	0.0001	3.34E+07
ICP.a.Pb	2.95E-18	0.5767	3.02E+06	0.0881	2.05E+06	0.0001	1.36E+06
ICP.a.S	4.21E-26	0.7256	1.96E-19	0.5706	2.86E+06	0.0258	7.13E+06
ICP.a.Si	4.06E-23	0.8936	2.07E-23	0.7484	3.64E+05	0.0001	1.15E+05

Table A-2-3. Variance Component Estimates
 (Sludge Segment Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(S)$	Test: $\sigma^2(S)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.a.Sr	1.03E-30	0.6911	2.44E-30	0.8881	3.50E+07	0.0001	1.29E+07
ICP.a.U	2.31E-16	0.2800	7.43E-25	0.9585	1.68E+08	0.0001	4.00E+07
ICP.a.Zn	2.75E-26	0.3079	3.17E-29	0.8840	2.75E+03	0.0001	9.62E+02
ICP.a.Zr	2.25E-09	0.9973	2.46E+01	0.0143	1.95E-10	0.7572	1.03E+02
ICP.f.Al	1.87E+07	0.1734	1.88E+05	0.3750	5.15E+06	0.0259	1.61E+07
ICP.f.Fe	1.28E+08	0.1818	3.55E-16	0.4708	1.06E+08	0.0001	8.12E+07
ICP.f.Na	7.20E-10	0.8585	4.59E-21	0.9552	6.00E+08	0.0001	6.99E+08
ICP.f.Ni	1.20E+07	0.1567	6.49E-21	0.5608	7.15E+06	0.0030	1.20E+07
Pu-239	2.39E-03	0.1320	1.86E-26	0.6264	1.37E-03	0.0008	1.90E-03
Sr-90	2.49E-19	0.3549	1.08E-31	0.8623	2.30E+04	0.0013	4.49E+04
TIC	1.90E-18	0.4079	1.83E-26	0.9240	8.88E+06	0.0001	7.91E+06
TOC-Dry	3.13E+06	0.3315	1.68E-24	0.5854	1.83E+06	0.1766	3.07E+07

Table A-2-4. Variance Component Estimates (Composite Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(A)$
% Water	4.05E+01	0.3219	3.79E+01	0.1184	6.66E+01
Cs-137	2.13E+02	0.3468	1.45E-13	0.4851	1.84E+03
DSC	2.05E+02	0.5634	2.04E+03	0.0612	2.42E+03
Density	1.24E-27	0.9649	1.18E-23	0.7027	6.99E-02
IC.Cl	1.51E-11	0.4123	5.24E+04	0.4231	1.05E+06
IC.F	4.69E+06	0.0776	2.78E+05	0.2691	1.48E+06
IC.NO2	1.44E-07	0.4433	2.23E+07	0.3599	2.90E+08
IC.NO3	3.10E+10	0.0158	2.37E+07	0.4330	2.26E+09
IC.Oxalate	3.93E+07	0.1244	1.65E-32	0.7031	2.35E+07
IC.PO4	4.30E-29	0.7277	2.81E+06	0.1814	1.24E+07
IC.SO4	9.90E+07	0.0404	1.13E+07	0.0338	7.31E+06
ICP.a.Ag	5.69E+00	0.3949	7.33E+00	0.1229	1.81E+01
ICP.a.Al	4.45E+07	0.1554	4.44E+06	0.2912	4.73E+07
ICP.a.B	3.16E+02	0.2135	7.69E-07	0.6480	5.80E+02
ICP.a.Ca	2.85E+05	0.3625	6.03E-22	0.6628	9.63E+06
ICP.a.Cd	5.63E+01	0.2216	5.34E-14	0.3004	1.16E+02
ICP.a.Cr	1.30E+06	0.0627	6.69E+04	0.6135	3.29E+05
ICP.a.Fe	1.07E+07	0.1823	5.81E-24	0.7124	1.59E+07
ICP.a.K	2.16E+05	0.2659	2.59E-16	0.0831	8.79E+05
ICP.a.Mn	1.88E+03	0.1535	6.76E+02	0.7275	7.62E+02
ICP.a.Na	7.60E+07	0.3334	3.36E-32	0.6479	1.81E+09
ICP.a.Ni	1.80E+05	0.3332	3.97E-24	0.0152	2.00E+06
ICP.a.P	2.05E+06	0.3143	2.54E+06	0.4078	1.25E+06
ICP.a.Pb	2.96E+03	0.4185	1.59E-13	0.9666	3.22E+04
ICP.a.S	1.18E+07	0.0000	1.18E-33	0.0319	1.71E+06
ICP.a.Si	1.02E+05	0.0091	3.84E+03	0.6350	2.48E+03
ICP.a.Sr	3.95E+04	0.3762	2.47E-17	0.6626	1.56E+06
ICP.a.U	1.25E+06	0.3337	1.89E-17	0.3140	1.70E+07
ICP.a.Zn	8.50E+01	0.2132	6.28E+00	0.4621	1.12E+02

Table A-2-4. Variance Component Estimates (Composite Data). (2 sheets)

Analyte	$\hat{\sigma}^2(R)$	Test: $\sigma^2(R)=0$ p-value	$\hat{\sigma}^2(C)$	Test: $\sigma^2(C)=0$ p-value	$\hat{\sigma}^2(A)$
ICP.a.Zr	4.38E+01	0.0363	1.03E-20	0.0138	6.96E+00
ICP.f.Al	2.60E-17	0.6854	9.37E+07	0.1761	5.85E+07
ICP.f.Fe	2.17E+06	0.4631	6.76E+06	0.3301	2.00E+07
ICP.f.Na	2.17E-26	0.8309	5.64E-13	0.2641	1.74E+09
ICP.f.Ni	2.42E+05	0.4724	2.86E+05	0.1705	3.02E+06
Sr-90	1.77E+03	0.3714	2.06E+03	0.0374	5.43E+03
Total Alpha	1.46E-03	0.1295	5.14E-04	0.2358	3.50E-04
Total Beta	8.60E+03	0.4225	1.56E+04	0.1632	4.07E+04
TIC	9.00E+07	0.4106	6.30E+06	0.0001	4.51E+08
TOC-Dry	1.29E+07	0.1791	1.07E+07	0.4362	4.14E+05
U-Phosphorescence	2.72E+06	0.3434	2.00E-12	0.7355	1.32E+07

**Statistical Support of the Tank
Characterization Report (TCR):
BY-110**

DW Engel
DS Daly

July 1996

Pacific Northwest National Laboratory
Richland, Washington 99352

CLUSTER ANALYSIS

The results from the ANOVA analyses indicate that there is a strong probability of a vertical heterogeneity effect, indicated by the low p-values ($V\text{-pval} < 0.01$) for most of the analytes. Defining the vertical structure of this heterogeneity (i.e., layers) is not an easy task using the ANOVA analysis, the ANOVA analysis is performed on one analyte at a time (univariate). Thus, a clustering analysis has been performed on samples from BY-110 to identify the layering structure. The results of this analysis and a description of the method is described in this section.

The BY-110 core sample dataset contains 188 multivariate observations. The observations may be cross-classified by core number (92, 95, 96, 109, 101, 103, 109, 107, and 113), sample size (whole, half, or quarter segment), and sample replicate (primary or duplicate).

A multivariate observation describing one analytical sample may include concentration measurements determined by IC (Cl, F, NO₂, NO₃, Ox, PO₄, SO₄), ICP:A (Al, Ba, B, Ca, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, P, K, Si, Ag, Na, Sr, S, Ti, U, Zn, Zr) and GEA (Cs-137, Co-60, Sr-89/90). Measurements for many of these analytes, however, were not made, or were below detection limits. Therefore, the analytes available for clustering were reduced to Al, Ca, Cr, Fe, Ni, P, Pb, Sr, Sr-90, U, Ox, Cs-137, TIC, and TOC.

The objectives of the multivariate statistical analysis of the reduced dataset were:

- to describe qualitatively potential spatial structure within the wastes by identifying groups (clusters) of similar observations,
- to describe quantitatively each layer based on the numerical characteristics of the associated cluster of observations.

The application of multivariate methods to the dataset proceeded through several steps: data screening, data transformation, sample clustering, analysis of the correlation structure, and summarization of the results. Several iterations through the steps were necessary to uncover anomalies and reveal consistencies. The results reported here are a composite of the results of several iterations.

Data Screening

The dataset was screened prior to clustering to identify variables and measurements that may have significant influence in the multivariate analysis and to identify possible

corrective actions (variable transformations or exclusions). Screening was also used to gain a better understanding of the variables and measurements that drive the clustering.

The reduction of the set of analytes because of missing values and below detection limit measurements was described in the previous section. Screening also revealed a few measurements that eluded quality control efforts, such as measurements in excess of a million parts per million and reported below detection limit values in excess of the reported detection limit were identified and addressed.

Disparities in the spread of measurement distributions significantly influence clustering results. Boxplots, which display the measurement distributions, show the disparities in the spread of measurement distributions across analytes (Figure A-3-1). Because of the large spread of measurement distribution, it is possible to say that Al and TIC would dominate the clustering analysis if performed on the original measuring scale (while, perhaps, important analytes based on physical principles would have minimal influence on the analysis). A \log_{10} transformation reduced the influence of both Al and TIC and increased the effect of others such as Sr and Fe on clustering results. With respect to the new differences in spread of analyte measurements, this transformation may be extreme, but was used since the data tends to be skewed, as shown in the boxplots (Figure A-3-1).

Multivariate Clustering

A multivariate observation on one sample can be thought of as defining the coordinates of a point in multi-dimensional space. In this analysis, the 188 samples in the analyte-reduced dataset identifies 188 points in a 14-dimensional space (one dimension for each analyte in the analysis dataset). A group of similar samples were identified by finding a cluster of points that were, in some sense, close together in the 14-dimension space. A quantitative summary of the cluster (number of samples, 10th percentile, median, and 90th percentile) was then calculated from measurements on the cluster members.

Hierarchical clustering (used here) begins with each point as its own cluster and proceeds iteratively until all points belong to one cluster. Each iteration begins with the calculation of distances between all possible pairings of points (or clusters). The two nearest points (or clusters) are then joined into one cluster. The process is repeated until all points belong to one cluster. The values representing distances between points (or clusters) depend upon the chosen linkage: compact, average, or single. Under compact linkage, points or clusters are joined whose farthest members are closest together. Under average linkage, points or clusters are joined whose centroids are closest, While using single linkage, points or clusters are joined whose nearest members are closest.

In general, compact linkage produces tight, spherical clusters while single linkage produces loose stringy clusters, with average linkage somewhere in between. Clustering results are usually visualized using a dendrogram showing the linkages among points and clusters, and the relative distances among them (Figure A-3-2).

Figure A-3-1. Boxplots of Original, Log₁₀ Transformed Data.

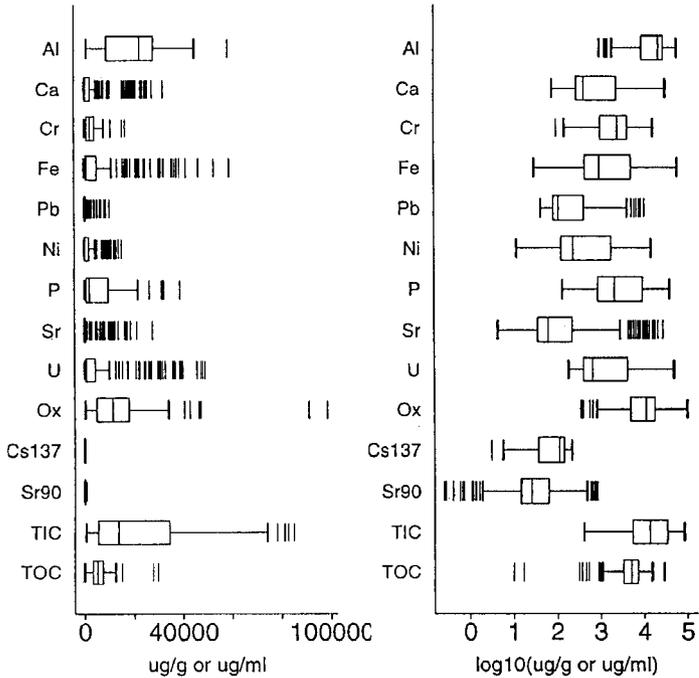
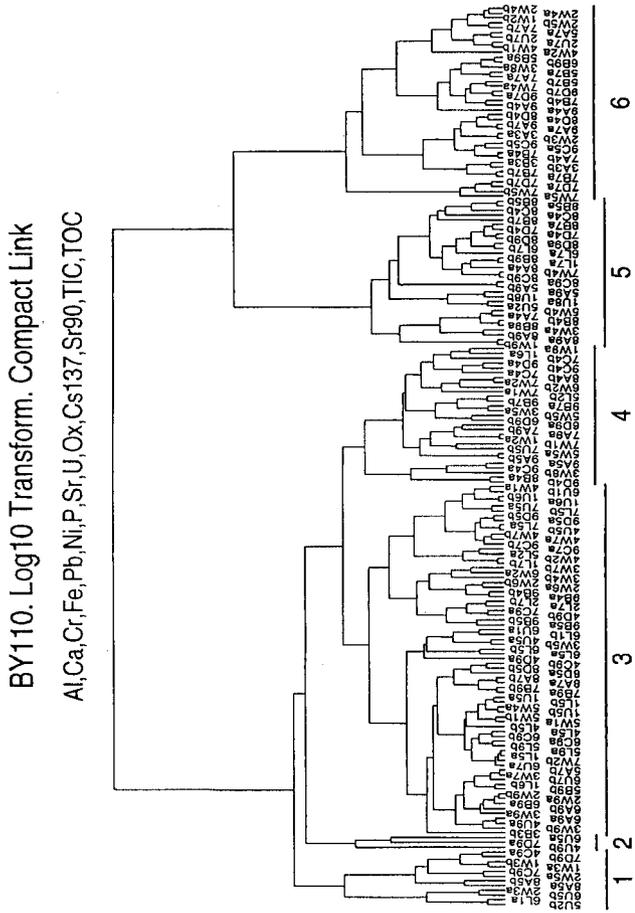


Figure A-3-2. Dendrogram of Clustering on Log₁₀ Transformed Data .



For the BY-110 dataset, groups of similar samples were identified by comparing the results of several clustering analyses. Analyses were performed on both the original-scaled and \log_{10} transformed data. For both datasets two cluster joining rules were used ("average" and "complete" linkage). Two major clusters emerged, with distinct, but minor, sample subsets in the major clusters. Sample membership in the major clusters remained stable across all data transformation and linkage combinations. Membership in the minor groups varied slightly. Figure A-3-2 presents a dendrogram (cluster tree) showing the linkages among samples and clusters, and the relative distances among them. The first major cluster is formed by combining the minor groups 1, 2, 3, and 4, while the minor groups 5 and 6 combine to form the second major cluster.

In general, the second major cluster consists of samples from the bottom of the tank, while the first major cluster tends more towards the top of the tank. The minor groups partition the samples into spatially-coherent subsets. The separation among samples in these subgroups, however, may not be physically significant. A useful visualization of the clustering results that suggests spatial structure for the waste is shown in Figure A-3-3. Here, the groups/clusters are distinguished by numbers (1-6), which correspond to the numbers featured on the dendrogram. Color versions of the plots are available, however, reproducibility of color plots is not an easy task and thus have been left out of this write-up. However, it is much easier to distinguish the groups with color plots, and thus the authors suggest that the reader color the different groups in Figure A-3-3 (suggested color scheme: 1 --> yellow green, 2 --> blue, 3 --> light green, 4 --> dark green, 5 --> light brown, and 6 --> salmon).

A quantitative summary of the five clusters can be found in Table 1. The clusters are identified by number and color in reference to the dendrogram and core plot. The number of members (samples) in each group is listed on the same line with the group number (*e.g.*, $n = 12$ for group 1). The groups are listed in the table in the same order as they occur on the dendrogram, with the left most group (1, yellow green) being first in the table and the right most group (6, salmon) being last in the table. For each analyte and cluster, three values labeled 'low', 'mid' and 'hi' are listed. These correspond to the 10th percentile, median and 90th percentile of the sample distribution for the cluster members. These provide a sense of the distribution of measurements within a cluster. Some of the percentiles in Table 1 are listed as <DL, which stands for less than detection limit, and occur because less than detection limit values are included in the analysis.

From visual comparisons of the summaries across the clusters in Table 1, we find distinct differences in many of the analytes such as U, Ca, Ni, and TIC, particularly between the salmon group (6) and the others. Less distinct differences between clusters for each analyte can also be observed.

Table 2 shows results for BY-110 using historical records for predicting the waste layers (Agnew 1996). This table contains concentrations for certain analytes for each predicted waste type. The layers predicted using the Tank Layering Model (TLM) are shown in Figure A-3-3.

Figure A-3-3. Overlay of Clustering Results onto each Core, Numbers Denote Groupings Identified from the Clustering Dendrogram.

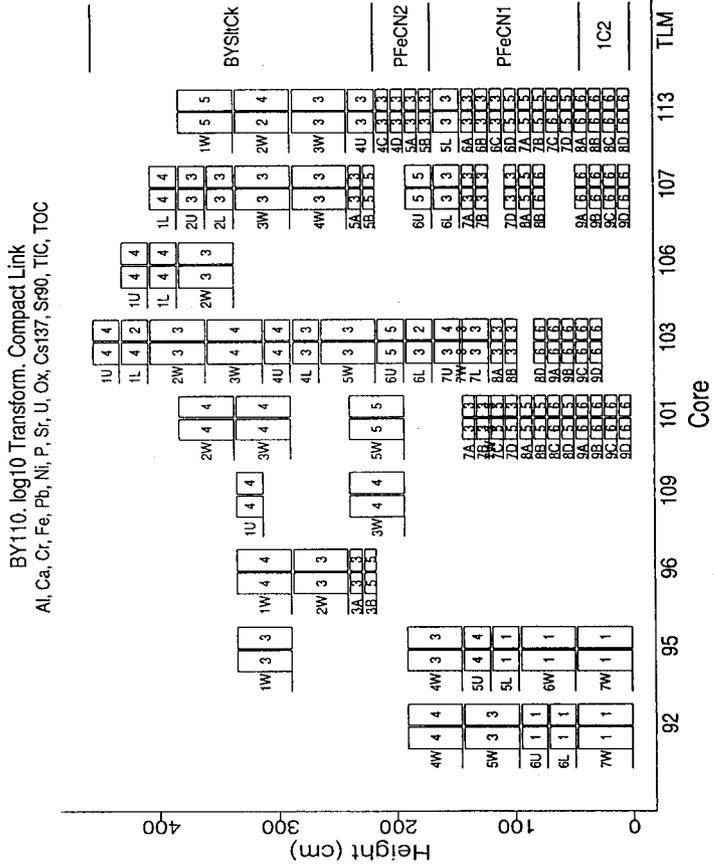


Table A-3-1. Summary of Clusters: "low", "mid", and "hi" Denote the Sample 10th Percentile, Median, and 90th Percentile. Groups Correspond to the Clusters in Figure A-3-3, *n* Denotes Number of Samples in the Cluster. (4 sheets)

Analyte	Low	Mid	High
Group 1 (Yellow Green)			n = 12
Al	9082	18475	34330
Ca	<DL	<DL	294
Cr	351	998	2974
Fe	<DL	<DL	277
Pb	<DL	<DL	88
Ni	<DL	<DL	98
P	<DL	900	2523
Sr	<DL	<DL	85
U	<DL	<DL	<DL
Ox	<DL	1240	3219
Cs137	19	55	147
Sr90	0	1	5
TIC	565	1610	6960
TOC	473	966	1900
Group 2 (Blue)			n = 3
Al	1644	4260	23604
Ca	142	211	413
Cr	999	2320	3156
Fe	387	538	1006
Pb	<DL	74	88
Ni	95	144	164
P	4195	18050	20890
Sr	28	62	108
U	<DL	447	568
Ox	7682	12410	13042
Cs137	8	11	103
Sr90	14	15	18
TIC	7416	30600	37240
TOC	<DL	17	298

Table A-3-1. Summary of Clusters: "low", "mid", and "hi" Denote the Sample 10th Percentile, Median, and 90th Percentile. Groups Correspond to the Clusters in Figure A-3-3, *n* Denotes Number of Samples in the Cluster. (4 sheets)

Analyte	Low	Mid	High
Group 3 (Light Green)		n = 73	
Al	2580	20800	32160
Ca	197	310	461
Cr	1792	3510	6148
Fe	344	608	1106
Pb	<DL	78	119
Ni	97	186	284
P	279	804	1889
Sr	18	37	64
U	<DL	460	745
Ox	7404	16100	26500
Cs137	20	112	159
Sr90	10	23	37
TIC	6422	22700	70320
TOC	2270	4560	6560
Group 4 (Dark Green)		n = 29	
Al	1798	4850	21488
Ca	206	330	600
Cr	638	1150	3856
Fe	362	680	2662
Pb	<DL	<DL	167
Ni	85	116	224
P	3478	7440	21900
Sr	41	63	126
U	<DL	421	878
Ox	5714	9900	28680
Cs137	6	16	57
Sr90	1	16	35
TIC	10620	31800	67060
TOC	2210	3420	6738

Table A-3-1. Summary of Clusters: "low", "mid", and "hi" Denote the Sample 10th Percentile, Median, and 90th Percentile. Groups Correspond to the Clusters in Figure A-3-3, *n* Denotes Number of Samples in the Cluster. (4 sheets)

Analyte	Low	Mid	High
Group 5 (Light Brown)			n = 31
Al	17900	26400	32900
Ca	520	614	4580
Cr	1860	4380	6000
Fe	1020	1580	5930
Pb	111	216	672
Ni	270	387	1960
P	1065	2200	4440
Sr	66	125	914
U	726	924	4810
Ox	7140	17100	40490
Cs137	38	123	162
Sr90	15	39	112
TIC	7160	15800	49000
TOC	3740	6160	11600
Group 6 (Salmon)			n = 40
Al	21239	27770	32010
Ca	9264	18070	24630
Cr	175	656	3862
Fe	10832	24100	39050
Pb	538	942	6232
Ni	4067	9174	12120
P	6757	12300	18023
Sr	2406	8862	17500
U	12369	28400	40390
Ox	< DL	2065	7010
Cs137	111	142	165
Sr90	151	432	743
TIC	2185	3995	8914
TOC	5117	8440	10230

Table A-3-2. Agnew's Waste Types for BY-110. (2 sheets)

pred. sludge ppm	1C2	PFeCN1	PFeCN2	BY-SltCk	MW
Na	66664.0	66551.6	63671.2	172188.6	23.0
Al	19031.1	0.0	0.0	32080.1	27.0
Fe	8170.2	36435.7	38801.4	3719.5	55.8
Cr	184.9	113.8	112.0	1490.2	52.0
Bi	7553.8	37715.4	43367.3	104.5	209.0
La	0.0	0.0	0.0	0.3	138.9
Hg	12.6	0.0	0.0	4.5	200.6
ZrO(OH)2	523.5	0.0	0.0	11.4	91.2
Pb	0.0	0.0	0.0	13.2	207.2
Ni	64.2	5862.9	5546.1	860.1	58.7
Sr	0.0	0.0	0.0	0.6	87.6
Mn	0.0	0.0	0.0	1706.7	54.9
Ca	1491.3	7717.7	8851.7	2926.3	40.1
K	89.7	482.7	473.9	1402.8	39.1
density	1.2	1.4	1.4	1.6	
vol % solids	24.9	3.7	3.2	49.0	
void frac	0.8	0.9	0.9	0.8	
wt. % H2O	78.3	62.0	61.2	39.7	
TOC wt. % C	0.0	0.7	0.4	0.4	
free OH-	316.6	389.6	269.1	4496.3	17.0
OH-	44216.6	36230.1	42810.0	93541.2	17.0
NO3-	19753.2	111458.1	109042.8	251233.2	62.0
NO2-	9304.4	5158.1	5578.9	45043.3	46.0
CO3--	2232.9	11555.4	13253.2	17552.0	60.0
PO4---	68038.3	25581.2	28018.2	3995.8	95.0
SO4--	4047.1	12019.5	11832.4	9572.6	96.1
SiO3--	1149.9	1254.4	1336.6	2206.9	28.1
F-	2976.7	2728.1	2685.7	661.1	19.0
Cl-	374.1	2012.1	1975.3	2702.0	35.4
C6H5O7---	0.0	0.0	0.0	2829.3	189.1
EDTA----	0.0	0.0	0.0	897.1	288.1
HEDTA---	0.0	0.0	0.0	4.9	274.1
glycolate-	0.0	0.0	0.0	707.0	75.0

Table A-3-2. Agnew's Waste Types for BY-110. (2 sheets)

pred. sludge ppm	1C2	PFeCN1	PFeCN2	BY-SltCk	MW
acetate-	0.0	0.0	0.0	1171.6	59.0
oxalate--	0.0	0.0	0.0	0.7	88.0
DBP	0.0	0.0	0.0	3406.8	210.0
butanol	0.0	0.0	0.0	1202.6	74.1
NH3	7.7	3.8	4.6	91.7	17.0
NiFe(CN)6--	0.0	26692.2	15379.9	0.0	270.6
Pu (μ Ci/g)	0.0	0.0	0.0	0.1	
U (μ g/g)	121.8	18541.3	21258.1	4735.0	238.0
Cs (μ Ci/g)	24.0	9.7	11.2	130.8	
Sr (μ Ci/g)	0.2	2.4	2.3	76.0	

REFERENCES

Agnew, S. F. 1996. *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev 3*. LA-UR-96-858, Rev. 2, Los Alamos National Laboratory, Los Alamos, New Mexico.

APPENDIX B

DRILL STRING WASH WATER CONTAMINATION CHECK DATA

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**Westinghouse
Hanford Company****Internal
Memo**

From: Evaluation and Planning 79200-96-014
Phone: 373-3339 R2-12
Date: June 13, 1996
Subject: LiBr ANALYSIS OF BY-110 WASH WATER CONTAMINATION

To: B. C. Simpson R2-12
cc: J. W. Hunt R2-12
J. G. Kristofzski R2-12
JWH:WDW File/LB R2-12

- References: (1) WHC-SD-WM-CSWD-081, Revision 0, "Technical Basis and Spreadsheet Documentation for Correcting Waste Tank Core Samples for Water Intrusion Based on LiBr Tracer," June 1996.
- (2) Internal memo, J. G. Kristofzski, WHC, to R. S. Popielarczyk, WHC, "Letter of Instruction for Measuring Intrusion of Hydrostatic Head Fluid in Waste and Correcting for Head Fluid Effect, dated August 25, 1995.

Several of the samples taken from waste tank BY-110 contained measurable quantities of Li and Br. These results indicate that these samples have been contaminated with the 0.3 Molar LiBr water used by sampling operations. Since this sampling event used one of the Rotary Mode Core Sampling trucks, the contamination is assumed to be from wash water used to clean or unplug the sampling bits.

Attachment 1 shows a summary of the core number, sample number, measured weight percent water, and then an estimate of how much of that water was from wash water contamination.

This data was taken from the detailed analysis performed and included as Attachment 2. The Excel spreadsheet "TABLIBR.XLS" was used to perform the analysis and produce the data outputs. The inner working and assumptions associated with this program is documented in Reference 1.

It has been shown that the Li in the wash water tracer can precipitate out of solution and join with the salts of the waste. Br on the other hand is expected to be a more stable tracer. It is, therefore, recommended that the Br analysis be used primarily to make corrections to the amount of actual water found in the waste. The Li analysis is provided also in that it can sometimes give some insights into the phenomena of the sampling event.

Based on the Br analysis, there were four samples where all of the water present appears to be from wash water contamination. These are marked "ALL" for the amount of water present which is from wash water laced with LiBr. Core 101 Segment 6 lower half, indicated that approximately 65% of the water present in this sample was from wash water. Based on the guidance in Reference 2, these samples are so contaminated with wash water such that the TGA results must be clearly marked as unusable.

The rest of the samples all indicated that at least 10% of the water present was from wash water but were less than 50%. These samples can be corrected for the correct moisture concentration. There are some cautions however. Core 96 and Core 107 both indicated wash water contamination in segment 1. This is most likely from cross contamination from previous sampling events and the sample analysis may accurately reflect tank contents. For the segments with drainable liquid, the differences between the Li and Br analysis are not unexpected because of the known Li precipitation issues.

Several of the samples have corrected weight percent values that are negative. This result is due to the errors associated with the sampling methods and measurement techniques. Also the correction function becomes very sensitive to the measured tracer concentration as the amount of water in the waste approaches zero. In particular, sample number S95T001818 had a higher bromine concentration than the measured amount in the wash water. While this gives an apparent erroneous result, one can clearly conclude that either all of the water present is from wash water or that a past sampling event has contaminated the sample. If the latter is true, very little can be determined from the analysis of tracer amounts in the sample.

Attachment 3 shows the correction curve for Sample S95T001791. The correction calculation gave an answer of -25% water that would be found in the waste. For a sample with a measure moisture concentration of 70.8%, a maximum concentration of Br of around 20,000 ug/mL should be found. This amount would indicate that all of the water present was from wash water. The Br analysis for this sample was 21,900. Considering that the method error is at least 10%, the measurement is in within the error bounds.

W. D. Winkelman, Principal Engineer
Evaluation and Planning
TWRS Technical Basis

klh

Attachments (3)

Evaluation and Planning

79200-06-014
 ATTACHMENT 1
 (1 sheet)

Table B-1-1. Summary of Wash Water Contamination for Tank 241-BY-110.

Core	Segment	Sample Number	TGA H ₂ O%	% of Water from wash water	
				Li Anal	Br Annual
92/95	composite	451	11.68	7.88	All
	4 whole	1,732	16.74	20.02	42.35
	3 drainable liquid	1,789	36.92	1.17	11.93
	5 liner liquid	1,816	39.69	46.74	All
96	1	2,872	15.31	16.14	54.62
101	4 drainable liquid	1,791	39.69	46.74	All
	5	1,755	17.21	47.03	50.48
	6 lower half	2,150	23.86	10.16	65.4
	7 top quarter	2,205	28.87	3.06	50.63
107	composite	481	12.38	29.82	All
	1	1,950	25.27	87.13	35.44
	2 upper half	1,956	23.21	32.7	37.72
	2 lower half	1,964	21.44	17.19	27.54
109	1 upper half	2,417	16.54	28.1	46.45
	3 lower half	2,430	32.94	23.52	18.92
	3 drainable liquid	2,427	47.54	.71	33.1

Evaluation and Planning

79200-96-014
 ATTACHMENT 2
 (7 sheets)

Table B-2-1. Li and Br Analysis for HHF Blank
 Tank 241-BY-110 Sample Number S95T003378.

Laboratory Analysis			Statistical Analysis			
Primary Analysis	Duplicate Analysis	Triplicate Analysis	Average Value	Difference	% Relative Difference	Standard Deviation of Average
HHF Blank Data						
Lithium Conc (gm/gm)	1900	1890	1,895	10	0.53%	5
Bromine Conc (gm/gm)	22400	22800	22,600	400	1.77%	200
HHF Density Calculation	Based on Li	Based on Br				
Density of HHF (gm/mL)	1.016	1.016	1.016	5.89E-04	0.06%	2.94E-04
Conc of H2O in HHF(gm/mL)	0.992	0.992	0.992	2.56E-04	0.03%	1.28E-04
Molarity LiBr	0.273	0.260				

Table B-2-2. Tank 241-BY-110 Density Analysis for Liquids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001816	1.05	1.04		1.045	0.01	0.96%	0.01
S95T001789	1.450	1.460		1.455	0.01	0.69%	0.00
S95T001791	1.260	1.250		1.255	0.01	0.80%	0.01
S95T002427	1.420	1.420		1.420	0.00	0.00%	0.00

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79200-96-014
ATTACHMENT 2
(7 sheets)

Table B-2-3. Tank 241-BY-110 Li Analysis for Solids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001868	75.78	52.23		64.01	23.55	36.79%	11.78
S95T000452	18.80	19.00		18.90	0.20	1.06%	0.10
S95T002875	37.40	57.00		47.20	19.60	41.53%	9.80
S95T001882	150.00	159.30		154.65	9.30	6.01%	4.65
S95T002153	28.90	63.70		46.30	34.80	75.16%	17.40
S95T002244	16.30	17.70		17.00	1.40	8.24%	0.70
S95T000482	95.50	95.20		95.35	0.30	0.31%	0.15
S95T001953	428.00	413.00		420.50	15.00	3.57%	7.50
S95T001959	158.00	132.00		145.00	26.00	17.93%	13.00
S95T001980	70.30	70.50		70.40	0.20	0.28%	0.10
S95T002419	105.00	72.50		88.75	32.50	36.62%	16.25
S95T002433	148.00	148.00		148.00	0.00	0.00%	0.00

Table B-2-4. Tank 241-BY-110 Li Analysis for Liquids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001816	1120.0	1150.0		1125.0	30.0	2.64%	15.0
S95T001789	12.0	12.0		12.0	0.0	0.00%	0.0
S95T001791	789.0	797.2	793.1	8.2	8.2	1.03%	4.1
S95T002427	10.0	8.4		9.2	1.6	17.16%	0.8

Evaluation and Planning

79200-96-014
 ATTACHMENT 2
 (7 sheets)

Table B-2-5. Tank 241-BY-110 Br Analysis for Solids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001738	1	770		1	460		1
S95T000451	3	860		3	720		3
S95T002874	980	2		830		1	905
S95T001763	2	270		1	69		1
S95T002152	3	560		3	550		3
S95T002231	3	160		3	500		3
S95T000481	4	170		4	200		4
S95T001952	2	320		1	760		2
S95T001958	2	020		1	970		1
S95T001979	1	270		1	420		1
S95T002420	1	700		1	800		1
S95T002432	1	280		1	560		1

Table B-2-6. Tank 241-BY-110 Br Analysis for Liquids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001816	22,900	23,000		22,950	100	0.44%	50
S95T001789	1,490	1,430		1,460	60	4.11%	30
S95T001791	21,900	21,800		21,850	100	0.46%	50
S95T002427	5,060	5,120		5,090	60	1.18%	30

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Table B-2-7. Tank 241-BY-110 TGA Weight Percent Water Analysis for Solids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001732	15.48%	18.00%		16.74%	2.52%	15.05%	1.26%
S95T000436	11.90%	13.21%		12.56%	1.31%	10.43%	0.66%
S95T002872	16.28%	14.34%		15.31%	1.94%	12.67%	0.97%
S95T001755	17.05%	17.38%		17.22%	0.33%	1.92%	0.16%
S95T002150	24.38%	23.34%		23.86%	1.04%	4.36%	0.52%
S95T002231	30.84%	26.90%		28.87%	3.94%	13.65%	1.97%
S95T000479	16.34%	17.14%		16.74%	0.80%	4.78%	0.40%
S95T001950	24.10%	26.43%		25.27%	2.33%	9.22%	1.17%
S95T001956	23.19%	23.24%		23.22%	0.05%	0.22%	0.03%
S95T001964	22.05%	20.82%		21.44%	1.23%	5.74%	0.62%
S95T002417	13.26%	19.81%		16.54%	6.55%	39.61%	3.28%
S95T002430	33.64%	32.23%		32.94%	1.41%	4.28%	0.70%

Table B-2-8. Tank 241-BY-110 TGA Weight Percent Wagter for Liquids Portion of Sample.

Sample number	Laboratory Analysis			Statistical Analysis			
	Primary analysis $\mu\text{g/mL}$	Duplicate analysis $\mu\text{g/mL}$	Triplicate analysis $\mu\text{g/mL}$	Average value $\mu\text{g/mL}$	Difference	% Relative difference	Standard deviation of average
S95T001816	89.32%	89.22%		89.27%	0.10%	0.11%	0.05%
S95T001789	36.88%	36.95%		36.92%	0.07%	0.19%	0.04%
S95T001791	70.92%	70.64%		70.78%	0.28%	0.40%	0.14%
S95T002427	47.70%	47.38%		47.54%	0.32%	0.67%	0.16%

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Table B-2-9. Tank 241-BY-110 Corrected Solids. (2 sheets)

Sample Number	Solids Wt.% corrected with Li Analysis						Solids Wt.% corrected with BR Analysis					
	%H ₂ O in tank	Corrected Wt% H ₂ O in tank	95% conf. Interval Low	Wt% difference	Variance of result	Std. of result	H ₂ O from HHF	Corrected Wt% H ₂ O	95% Conf. interval low	Wt% difference	Variance of result	Std. of result
S95T001732												
S95T001868	20.02%	13.86%	4.93%	-2.88%	2.00%	1.42%	42.35%	10.41%	0.88%	-6.33%	2.28%	1.51%
S95T001738												
S95T000436												
S95T000452	7.88%	11.68%	7.51%	-0.87%	0.44%	0.66%	132.50%	-4.92%	-10.60%	-17.47%	0.81%	0.90%
S95T000451												
S95T002872												
S95T002875	16.14%	13.17%	6.26%	-2.14%	1.20%	1.09%	54.62%	7.60%	-19.11%	-7.71%	17.90%	4.23%
S95T002874												
S95T001755												
S95T001882	47.03%	9.94%	8.05%	-7.27%	0.09%	0.30%	50.48%	9.36%	1.29%	-7.86%	1.63%	1.28%
S95T001763												
S95T002150												
S95T002153	10.16%	21.98%	16.31%	-1.88%	0.81%	0.90%	65.40%	9.83%	5.81%	-14.03%	0.41%	0.64%
S95T002152												
S95T002231												
S95T002244	3.08%	28.24%	15.68%	-0.63%	3.95%	1.99%	50.63%	16.77%	1.41%	-12.10%	5.91%	2.43%
S95T002231												

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Table B-2-9. Tank 241-BY-110 Corrected Solids. (2 sheets)

	Solids Wt% corrected with Li Analysis							Solids Wt% corrected with BR Analysis								
S95T000479																
S95T000482	29.82%	12.38%	9.72%	4.36%	0.18%	0.42%	109.73%	-2.00%	-5.41%	-18.74%	0.29%	0.54%				
S95T000481																
S95T001930																
S95T001953																
S95T001952	87.13%	4.20%	-5.79%	-21.07%	2.50%	1.58%	35.44%	17.96%	7.26%	-7.31%	2.87%	1.69%				
S95T001956																
S95T001959																
S95T001958	32.70%	16.94%	13.08%	-6.27%	0.37%	0.61%	37.72%	15.88%	15.09%	-7.33%	0.02%	0.13%				
S95T001964																
S95T001980																
S95T001979	17.19%	18.45%	14.41%	-2.99%	0.41%	0.64%	27.54%	16.53%	12.00%	-4.90%	0.52%	0.72%				
S95T002417	28.10%	12.48%	-9.78%	-4.05%	12.43%	3.53%	46.45%	9.61%	-12.88%	-6.92%	12.69%	3.56%				
S95T002419																
S95T002420																
S95T002430																
S95T002433																
S95T002432	23.52%	27.36%	22.52%	-5.58%	0.59%	0.77%	18.92%	28.52%	22.93%	-4.41%	0.78%	0.89%				

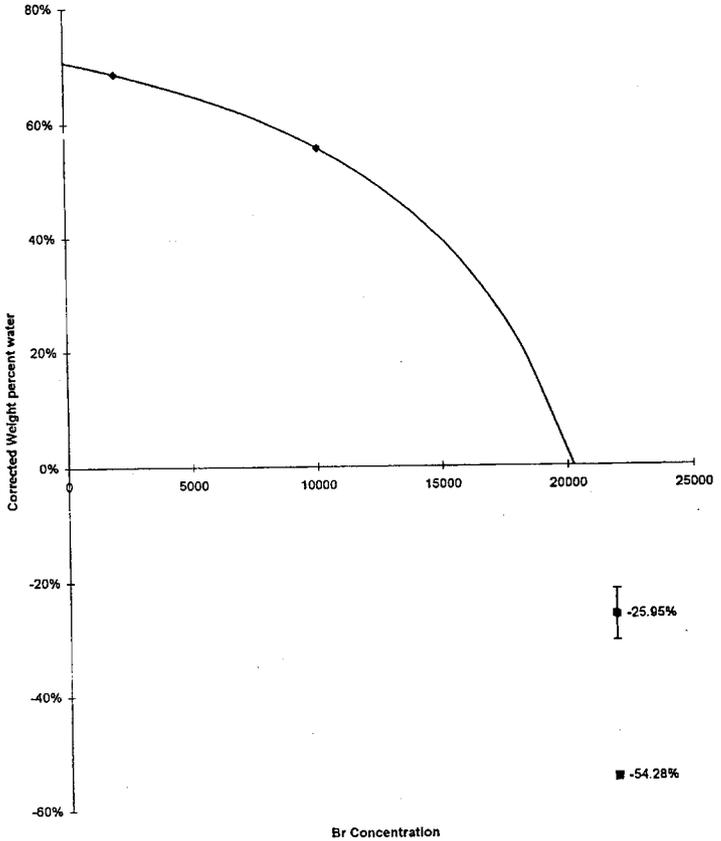
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Table B-2-9. Tank 241-BY-110 Corrected Solids. (2 sheets)

Sample Number	Solids Wt% corrected with Li Analysis					Solids Wt% corrected with BR Analysis						
	%H ₂ O in tank	Corrected Wt% H ₂ O in tank	95% conf. Interval Low	Wt% difference	Variance of result	Sd. of result	H ₂ O from HHF	Corrected Wt% H ₂ O	95% Conf. interval low	Wt% difference	Variance of result	Sd. of result
S95T001816	63.69%	77.57%	74.94%	-11.70%	0.17%	0.42%	107.98%	-579.20%	-3972.71%	668.47%	288859.78%	537.46%
S95T001789	1.17%	36.65%	36.36%	-0.27%	0.00%	0.05%	11.93%	34.05%	33.59%	-2.87%	0.01%	0.07%
S95T001791	46.74%	57.01%	55.42%	-13.77%	0.06%	0.25%	107.97%	-25.95%	-39.49%	-96.73%	4.60%	2.15%
S95T002427	0.71%	47.37%	46.35%	-0.17%	0.03%	0.16%	33.10%	37.92%	36.64%	-9.62%	0.04%	0.20%

Figure B-3-1. Correction Curve for Sample Number S95T001791
Liquids Portion Br Analysis.



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APPENDIX C

1994 VAPOR CHARACTERIZATION DATA

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**Tank 241-BY-110 Headspace Gas and Vapor Characterization Results
for Samples Collected in November 1994**

October 7, 1995

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Acronyms and Abbreviations

CES	consensus exposure standard
EPA	Environmental Protection Agency
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
LFL	lower flammability limit
MS	mass spectrometer
NFPA	National Fire Protection Association
NPH	normal paraffinic hydrocarbon
ORNL	Oak Ridge National Laboratory
PNL	Pacific Northwest Laboratory
ppmv	parts per million by volume, 1 ppmv = 10 ⁻⁴ vol%
TST	triple sorbent trap
vol%	percent by volume, 1 vol% = 10,000 ppmv
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

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**Tank 241-BY-110 Headspace Gas and Vapor Characterization Results
for Samples Collected in November 1994**

1.0 INTRODUCTION

1.1 Characterization Objectives

Tank BY-110 headspace gas and vapor samples were collected and analyzed to help determine the potential risks to tank farm workers due to fugitive emissions from the tank. The drivers and objectives of waste tank headspace sampling and analysis are discussed in *Program Plan for the Resolution of Tank Vapor Issues* (Osborne and Huckaby 1994). Tank BY-110 was vapor sampled in accordance with *Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution* (Osborne et al. 1994).

1.2 Characterization Data Criteria

Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution describes parameters for data collection to ensure appropriate conclusions can be drawn from the data. Tank headspace characterization data were collected to help in the evaluation of 1) headspace flammability, and 2) identification and quantification of compounds of toxicological concern.

Single Shell Tank Interim Operational Safety Requirements (Dougherty 1995) specifies that combustible constituents in tank headspaces be maintained below 25 % of the lower flammability limit (LFL). This essentially agrees with National Fire Protection Association requirements that combustible concentrations be maintained at or below 25 % of the LFL (NFPA 1992). Current governing operating specifications for Watchlist tanks, such as tank BY-110, specify that combustible constituents be maintained at or below 20 % of the LFL (WHC 1995a).

Headspace characterization data are used by Westinghouse Hanford Company (WHC) Tank Waste Remediation Systems Industrial Hygiene as source term data in the industrial hygiene strategy to protect workers from tank fugitive emissions. Because selection of worker protective equipment must be based on industrial hygiene monitoring of the work place and not on source term data (29 CFR 1910.120), tank headspace characterization data can not be used for this purpose. Furthermore, because there are mechanisms by which headspace constituents can be either diluted or concentrated as they are released to the atmosphere, the headspace characterization data should not be considered to be representative of emissions at the point of emission.

These statements notwithstanding, the data quality objectives document specifies that the industrial hygiene group be advised if constituents with toxicological properties exceed 50 % of the appropriate consensus exposure standard (CES) for non-carcinogens, or 10 % of the appropriate CES for carcinogens. A CES is defined as the most stringent of known regulatory or recommended toxicological values for the workplace (Osborne et al. 1994).

1.3 Sampling Overview

Tank headspace characterization data presented here are from a single sampling event. Samples collected are thought to have been representative of the tank BY-110 headspace when the tank was sampled, and sample analyses were designed to provide a reasonably accurate and complete characterization of the significant headspace constituents (Meacham et al. 1995). No assessment has been made of how the tank BY-110 headspace composition changes with time, though studies of tank C-103 suggest that composition changes probably occur very slowly in the passively ventilated tanks (Huckaby and Story 1994).

1.4 Tank Headspace Dynamics

Tank BY-110 is the first tank in a 3-tank cascade with tanks BY-111 and BY-112. It is connected to tank BY-111 via a 7.4-cm (2.9-in.) inside diameter, 7.6-m (25-ft) long cascade line. Tanks BY-111 and BY-112 are also connected by a similar line. Since these cascade lines connect the headspaces of these tanks, gases and vapors originating from the wastes in tank BY-111 or BY-112 may be transferred to tank BY-110 (unless the cascade lines are obstructed).

The cascade of tanks BY-110, BY-111, and BY-112 is passively ventilated, which means that the tanks are allowed to exhale air, waste gases, and vapors as the barometric pressure falls, and inhale ambient air as the barometric pressure rises. Each of these tanks has its own filtered breather riser. Barometric pressure typically rises and falls on a diurnal cycle, producing an average daily exchange of air equal to about 0.46 % of each tank headspace (Huckaby 1994). Changes in the concentrations of tank headspace constituents due to barometric pressure changes are consequently very slow.

2.0 SAMPLING EVENT

Headspace gas and vapor samples were collected from tank BY-110 using the vapor sampling system (VSS) on November 11, 1994 by WHC Sampling and Mobile Laboratories, (WHC 1995b). Sample collection and analysis were performed as directed by *Tank 241-BY-110 Tank Characterization Plan* (Carpenter 1994). The tank headspace temperature was determined to be 27 °C. Air from the tank BY-110 headspace was withdrawn via a 7.9 m-long heated sampling probe mounted in riser 12B, and transferred via heated tubing to the VSS sampling manifold. All heated zones of the VSS were maintained at approximately 50 °C.

Sampling media were prepared and analyzed by WHC, Oak Ridge National Laboratories (ORNL), and Pacific Northwest Laboratories (PNL). The 40 tank air samples and 2 ambient air control samples collected are listed in Table 2-1 by analytical laboratory. Table 2-1 also lists the 14 trip blanks and 2 field blanks that accompanied the samples.

A general description of vapor sampling and sample analysis methods is given by Huckaby et al. (1995). The sampling equipment, sample collection sequence, sorbent trap sample air flow rates and flow times, chain of custody information, and a discussion of the sampling event itself are given in WHC (1995b).

3.0 INORGANIC GASES AND VAPORS

Analytical results of sorbent trap and SUMMA^{TM,1} canister tank air samples for selected inorganic gases and vapors are given in Table 3-1 in parts per million by volume (ppmv) in dry air. The concentration of water vapor given in Table 3-1 has been adjusted to tank conditions as given in Section 3.3. Inorganic analyte sorbent traps and SUMMATM canisters were prepared and analyzed by PNL at PNL quality assurance impact level 2. Clauss et al. (1995) describe sample preparation and analyses.

Analyses of the VSS event inorganic vapor sorbent traps were performed within 39 days of sample collection, analyses of VSS event SUMMATM canisters for inorganic compounds were performed 70 days after sample collection (Ligotke 1995). Thus the 60-day holding time requirement of the WHC quality assurance project plan (Keller 1994) was satisfied for analyses of VSS sorbent traps, but not for SUMMATM sample analyses. It should be noted that these inorganic compounds (i.e., the permanent gases) would be expected to be very stable in the SUMMATM canisters, and the results may not have been affected even though the administratively chosen 60-day holding time requirements were exceeded. No holding time study has been performed to determine the stability of the inorganic analytes in SUMMATM canisters in the chemical matrix of the tank samples.

3.1 Ammonia, Hydrogen, and Nitrous Oxide

Ammonia concentration in the headspace of tank BY-110 was measured to be 401 ppmv. Ammonia is thought to be a product of chemical and radiolytic waste degradation processes. It has been observed in virtually all of the waste tanks sampled to date, at concentrations ranging from about 3 ppmv in C-108 (Lucke et al. 1995), to 1040 ppmv in tank BY-108 (McVeety et al. 1995). Given the LFL of ammonia in air is about 15 % by volume (vol%), the measured 401 ppmv corresponds to less than 0.3 % of the LFL, and does not contribute appreciably to the flammability of the headspace.

Hydrogen was not detected in the tank BY-110 samples, being determined to be below the limit of detection of the analytical method, 160 ppmv. In general, hydrogen is of concern as a fuel. The 160 ppmv detection limit corresponds to about 0.4 % of the lower LFL for hydrogen in air.

The average nitrous oxide concentration measured in the 3 SUMMATM canister samples was 103 ppmv. Nitrous oxide is commonly found in the passively ventilated waste tanks, and the 103 ppmv concentration in tank BY-110 is about average for the tanks that have been sampled. Under proper conditions, nitrous oxide can serve as an oxidizer to support combustion. However, Cashdollar et al. (1992) found that nitrous oxide had no significant effect on the flammability of hydrogen and air mixtures for hydrogen concentrations less than 20 vol%, and that "small amounts of nitrous oxide (relative to air) do not appear to have much effect on the flammability". Their results suggest the measured nitrous oxide concentration is much too low to have a significant effect on the flammability of the tank BY-110 headspace.

¹ SUMMA is a trademark of Molectrics, Inc., Cleveland, Ohio.

3.2 Carbon Dioxide and Carbon Monoxide

The average measured headspace carbon dioxide concentration, 229 ppmv, is significantly less than the normal ambient air concentration of 350 to 400 ppmv. Carbon dioxide introduced by air exchange with the atmosphere is readily absorbed by caustic supernatant and interstitial liquids of the waste tanks, and converted to carbonate in solution. It is reasonable to expect the level of carbon dioxide in a tank headspace will therefore depend on the tank's breathing rate, and the pH and surface area of aqueous waste (i.e., supernate, interstitial liquid, and condensate) in the tank. The 229 ppmv carbon dioxide concentration measured in tank BY-110 is typical of other tanks that have been sampled.

Carbon monoxide in the tank BY-110 headspace, measured to be < 76 ppmv, is not well characterized. The method quantitation limit, 76 ppmv, is above the highest waste tank carbon monoxide concentration measured to date (26.7 ppmv) in tank C-103², Huckaby and Story 1994).

3.3 Nitric Oxide, Nitrogen Dioxide, Water and Tritium

Nitric oxide and nitrogen dioxide concentrations in the tank BY-110 headspace were determined to be ≤ 0.09 ppmv and ≤ 0.05 ppmv, respectively. These are both acid gases that would have very low equilibrium concentrations above the high pH waste in tank BY-110. Nitric oxide is commonly found at trace concentrations, presumably due to its formation from oxygen and nitrogen in the radiation field of the headspace. These constituents could potentially serve as oxidizers to support combustion, but at the measured concentrations would have a negligible effect on the flammability of the tank BY-110 headspace.

The water vapor concentration was measured by gravimetric analysis of 5 sorbent trap systems by PNL (Clauss et al. 1995). The water vapor concentration of tank BY-110 was determined to be about 8.0 mg/L, at the tank headspace temperature of 27 °C and pressure of 985 mbar (739.2 torr). This corresponds to a water vapor partial pressure of 11.1 mbar (8.3 torr), to a dew point of 8.5 °C, and to a relative humidity of 31%.

Silica gel sorbent traps were used to test for tritium. It is assumed that tritium produced by the waste combines with hydroxide ions to form tritium-substituted water. Evaporation of the tritium-substituted water would then result in airborne radioactive contamination. Silica gel sorbent traps adsorb virtually all (normal and tritium-substituted) water vapor from the sampled tank air, and are analyzed at the WHC 222-S laboratory. Radiochemical analysis of the silica gel trap indicated the total activity of the headspace to be less than 50 pCi/L (WHC 1995b).

3.4 Discussion of Inorganic Gases and Vapors

Aside from water vapor and carbon dioxide, the most abundant waste constituents in the tank BY-110 headspace are ammonia and nitrous oxide. These have been detected in most tank

² The carbon monoxide measurement in tank C-103 was made by Oregon Graduate Institute of Science and Technology, and placed in brackets to emphasize it should be considered secondary data.

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headspaces that have been sampled, and along with hydrogen, are usually the dominate waste species.

The relative standard deviations of the results, given in the last column in Table 3-1, are reasonable for the analytical methods used. Relative standard deviations range from 2 % for water vapor, to 23 % for carbon dioxide results. The precision reported depends both on sampling parameters (e.g., sample flow rate and flow time for sorbent traps) and analytical parameters (e.g., sample preparation, dilutions, etc.), and the relative standard deviations suggest there were no significant problems in the field or in the laboratories.

As discussed briefly in Section 1.4, it is possible that gases and vapors generated by the waste in tanks BY-111 and BY-112 could be transferred to tank BY-110 via the cascade lines. If significant exchange of selected inorganic gases and vapors were taking place between adjacent tanks, either their headspace compositions would be very similar, or all constituents detected in one tank would be at or equal or higher concentrations in the other tank. The data in Table 3-2 are consistent with the premise that air, gases, and vapors could be transferring from tank BY-111 to tank BY-110 (waste species are at higher concentrations in tank BY-110 than in BY-111), but do not constitute proof that this is occurring.

4.0 ORGANIC VAPORS

Organic vapors in the tank BY-110 headspace were sampled using SUMMA™ canisters, which were analyzed at PNL, and triple sorbent traps (TSTs), which were analyzed by ORNL. Both laboratories used a gas chromatograph (GC) equipped with a mass spectrometer (MS) to separate, identify, and quantitate the analytes. Descriptions of sample device cleaning, sample preparations, and analyses are given by Jenkins et al. (1995a) and Clauss et al. (1995).

SUMMA™ sample results should be considered to be the primary organic vapor data for tank BY-110. These results were produced at PNL quality assurance impact level 2. However, PNL analyses of organic vapors in SUMMA™ canisters were not completed until 76 days after sample collection (Ligotke 1995), and exceeded the administratively chosen 60-day holding time specified by the WHC quality assurance project plan (Keller 1994). No holding time study has been performed to determine the stability of the organic analytes in SUMMA™ in the chemical matrix of the tank air samples.

ORNL analyses of TST samples from this and other waste tanks generally agree with, support, and augment the SUMMA™ sample results. However, because certain WHC quality assurance requirements were not satisfied by ORNL, the quality assurance assessment of ORNL by Hendrickson (1995) should be reviewed before results unique to the TST samples are used for decision making.

All TSTs prepared by ORNL had 3 surrogate compounds added to evaluate sample matrix effects, potential handling, storage, and shipment problems, and analytical instrumentation performance (Jenkins et al. 1995a). ORNL evaluated the surrogate recoveries using a statistical approach similar to that prescribed by *SW 846 Method 8260A Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique* (EPA 1992). Using this approach, ORNL reported that all surrogates had standard deviation values within the 95 % confidence interval for variance, indicating that no bias was introduced in the measurement of analyte quantities (Jenkins 1995a).

4.1 Positively Identified Organic Analytes

Positive identification of organic analytes using the methods employed by PNL and ORNL involves matching the GC retention times and MS data from a sample with that obtained by analysis of standards. The concentration of an analyte in the sample is said to be quantitatively measured if the response of the GC/MS has been established at several known concentrations of that analyte (i.e., the GC/MS has been calibrated for that analyte), and the MS response to the analyte in the sample is between the lowest and highest responses to the known concentrations (i.e., the analyte is within the calibration range).

ORNL and PNL were assigned different lists of organic compounds, or target analytes, to positively identify and measure quantitatively. The ORNL target analyte list was derived from a review of the tank C-103 headspace constituents by a panel of toxicology experts (Mahlum et al. 1994). The PNL target analyte list included 39 compounds in the Environmental Protection Agency (EPA) task order 14 (TO-14) method, which are primarily halocarbons and common industrial solvents (EPA 1988), plus 14 analytes selected mainly from the toxicology panel's review of vapor data on tank C-103.

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Given in Table 4-1 are 13 organic compounds positively identified and quantitated in SUMMA™ canister samples by PNL. Averages reported are from analyses of 3 SUMMA™ canister samples. PNL performed analyses according to the EPA task order 14 (TO-14) methodology, but expanded the number of target analytes from 40 to 55 to include waste tank analytes of particular interest (EPA 1988, Clauss et al. 1995). Of the original 40 TO-14 analytes, only trichlorofluoromethane and 1,2,4-trichlorobenzene were determined to be above the 0.002 ppmv quantitation limit of the analyses (Clauss et al. 1995 provide the complete TO-14 analyte list). Five of the 15 additional target analytes (1-propanol, acetonitrile, butanal, propanenitrile, and butanenitrile) were below the 0.005 ppmv method quantitation limit.

ORNL positively identified 23 of 27 target analytes selected by WHC. Four target analytes (vinylidene chloride, dichloromethane, tributyl phosphate and dibutyl butylphosphonate) were below detection limits. Five detected and quantitated analytes, and their average concentrations from the analysis of 3 TSTs, are given in Table 4-2. Despite calibration of the instrument over about a 20-fold concentration range, 16 of the 23 compounds were outside of the calibration range in at least 1 of the TST samples. Table 4-3 lists 13 of the positively identified analytes which were not quantitated. Table 4-4 lists the remaining positively identified analytes, 2 of which were quantitated and 3 of which were not quantitated, that exceeded the ORNL analyte-specific practical holding times. Data in Tables 4-3 and 4-4 may not be accurate to within $\pm 30\%$ as specified by Burnum (1995).

Based on a practical holding time study performed by ORNL the reported concentrations of propanone, butanal, 1-butanol, 2-pentanone, and n-pentanenitrile may have been affected by the 60-day period between sample collection and analysis (Jenkins 1995b). The concentration of these five analytes are reported in Table 4-4. The practical holding time is defined as the holding time for which there is a 15% risk that the concentration of an analyte in the sample will be below its initial concentration. Jenkins et al (1995b) describe the ORNL practical holding time study, and report practical holding times for these compounds. It should be noted, however, that the contractual holding time for the TST samples was 60-days.

Twelve target analytes that were common to both the ORNL and PNL analyses are listed in Table 4-5. Two of these, dichloromethane and vinylidene chloride, were not detected by either laboratory. Comparison of the results for the other 10 analytes from the 2 laboratories indicates the following:

- 1) The methods agree well on the concentrations of n-heptane and n-decane;
- 2) the methods are in fair agreement on the concentration of acetone, with TST analysis indicating 3.8 ppmv, and SUMMA™ analysis indicating 9.9 ppmv;
- 3) n-hexane was detected by both methods, but the agreement is poor, with TST analysis indicating 0.29 ppmv, and SUMMA™ analysis indicating 0.017 ppmv; and
- 4) ethanenitrile, propanenitrile, butanal, benzene, n-butanenitrile, and toluene were reported to be below SUMMA™ analysis method quantitation limits, yet each of these were measured in TST samples to be significantly above the SUMMA™ method quantitation limits.

Though the discrepancies between the TST and SUMMA™ sample results exceed accuracy requirements specified in *Tank 241-BY-110 Tank Characterization Plan* (Carpenter 1994), benzene is the only analyte in question that exceeds the action limit specified by *Data Quality Objectives for Generic In-Tank Health and Safety Issue Resolution* (Osborne et al. 1994).

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The most abundant analytes in Tables 4-1 through 4-4 are propanone, ethanenitrile, and 2-butanone. At the reported concentrations, these analytes do not individually or cumulatively represent a flammability hazard.

4.2 Tentatively Identified Organic Analytes

In addition to the target analytes, the ORNL and PNL analytical procedures allow the tentative identification of other organic compounds. Tentative identification of analytes was performed by comparing the MS molecular fragmentation patterns with a library of known MS fragmentation patterns. This method allows an organic analyte to be identified (with reasonable certainty) as an alkane, a ketone, an aldehyde, etc., and may also determine its molecular weight. The method usually does not, however, allow the unambiguous identification of structural isomers, and this ambiguity increases with analyte molecular weight. Using this method, many analytes can be tentatively identified with reasonable confidence without having to inject each into the GC/MS to determine their GC retention times or specific MS patterns.

By the nature of the sampling devices, virtually all organic vapors present in the tank headspace are collected by both TST and SUMMA™ samples. Analyses of the samples are designed to recover, separate, and identify the organic vapors in the samples. TSTs are not good for collecting highly volatile compounds (i.e., molecules more volatile than propane), but are quite good for most others. In contrast, the recovery of very low volatility compounds (i.e., molecules with more than about 15 carbon atoms) and some polar compounds with moderate volatility (i.e., butanal) from SUMMA™ samples has been problematic.

The PNL list of tentatively identified compounds, with estimated concentrations, is given in Table 4-6, and the ORNL list of tentatively identified compounds, and their estimated concentrations, is given in Table 4-7. Estimated concentrations are in mg/m^3 , based on dry air at 0 °C and 1.01 bar.

Because the list of tentatively identified organic compounds in TST samples is especially long and locating any given compound may be difficult, the list has been sorted alphanumerically by compound name in Table 4-8, and also in order of decreasing estimated concentrations in Table 4-9. Numbers in the first columns of Tables 4-8 and 4-9 (Cmpd #) identify the elution order of the compound in Table 4-7.

The ORNL and PNL methods used to tentatively identify and estimate concentrations are described by Jenkins et al. (1995a) and Claus et al. (1995), respectively, and should be reviewed before this data are used for decision making. Results in Tables 4-6 through 4-9 are presented in terms of observed peaks, and are not adjusted for the occurrence of split chromatographic peaks, or the assignment of the same identity to different peaks (e.g., Cmpd # 22 and 41 in Table 4-7). In these instances, the estimated concentration of a compound appearing as a doublet or triplet is simply the sum of the individual peak estimates.

Concentrations given in Tables 4-6 through 4-9 should be considered rough estimates. The proper quantitation of all observed analytes is outside the scope and budget of these analyses, and the estimation of concentrations involves several important assumptions. The validity of each assumption depends on the analyte, and such factors as the specific configuration of the analytical instrumentation.

4.4 Discussion of Organic Analytes

Some of the compounds listed in Tables 4-1 through 4-9 were introduced to the tank with process waste streams, and are detected in the headspace because the original inventory has not been completely evaporated or degraded. Examples of these in tank BY-110 are the semivolatile normal paraffinic hydrocarbons (NPHs), (i.e., n-dodecane, n-tridecane, n-tetradecane, n-pentadecane) and methyl-substituted decahydronaphthalenes that were used as diluents for tributyl phosphate.

Though there is no toxicological or flammability hazard associated with the 0.34 ppmv of trichlorofluoromethane measured in SUMMA™ canister samples from tank BY-110, its presence warrants an explanation. The origin of trichlorofluoromethane in the waste tanks has not been established, however, it is possible that it was introduced to the tank and the concrete pits of the tank through its use in urethane sealing foam.

Most of the compounds in Tables 4-1 through 4-9 are believed to be chemical reaction and radiolytic reaction products of the semivolatile or nonvolatile organic waste stored in the tank. For example, 1-butanol is known to be formed by the hydrolysis of tributyl phosphate, and it has been suggested that the alcohols, aldehydes, ketones, nitriles, alkenes, and short chain alkanes are all degradation products of NPHs.

Neither TST nor SUMMA™ methods detected tributyl phosphate as a headspace constituent. The relatively high concentration of 1-butanol, however, is a strong indication that tributyl phosphate does exist in tank BY-110. That tributyl phosphate was not observed in the TST samples may be due to the facts that tributyl phosphate has a very low vapor pressure and is consequently present at a very low concentration, and it has a tendency to adsorb on to the high efficiency particulate air (HEPA) filters used during sampling to protect the samples from radiological particulate contamination.

Volatile alkanes and alkenes are more prominent in the tank BY-110 headspace than is typical of NPH-rich tanks. For example, n-butane has the highest estimated concentration of any the tentatively identified analyte in both SUMMA™ samples (Table 4-6) and TST samples (Table 4-7). Also, the other tentatively identified analytes in SUMMA™ samples with concentrations above 1 mg/m³ are propene, propane, n-pentane, and 2-methylpentane.

In the semivolatile region of Table 4-7, subjectively defined here as those compounds eluting after n-decane (Cmpd # 56 through 111), there are many branched alkanes. The abundance of these, as well as the methyl-substituted decahydronaphthalenes, has also been noted in other 241-BY farm tanks. ORNL estimated the total organic vapor concentration of tank BY-110, based on quantitative and estimated MS data, to be 29 mg/m³.

5.0 SUMMARY

The tank BY-110 headspace was sampled in November 1994 for gases and vapors to address flammability and industrial hygiene concerns. Collection and analysis of samples has been reported. It was determined that no headspace constituents exceeded the flammability notification limits, but ammonia, measured to be present at 401 ppmv, exceeded the industrial hygiene notification limit of 150 ppmv specified in the current *Vapor Sampling and Analysis Plan* (Homi 1995).

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Table 2-1
Tank BY-110 Gas and Vapor Sample Type and Number

Laboratory	Sampling Device	Nominal Sample Volume (L)	Target Analytes	Number of Samples
Oak Ridge National Laboratories	Triple Sorbent Trap	0.05	Organic vapors	4 tank air samples
		0.25	Organic vapors	4 tank air samples
		0.50	Organic vapors	4 tank air samples + 2 trip blank + 2 field blank
Pacific Northwest Laboratories	Acidified Carbon Sorbent Trap	3.0	Ammonia	6 tank air samples + 3 trip blanks
	Triethanolamine Sorbent Trap	3.0	Nitrogen Dioxide	6 tank air samples + 3 trip blanks
	Oxidation bed + Triethanolamine Sorbent Trap	3.0	Nitric Oxide	6 tank air samples + 3 trip blanks
	Silica Gel Sorbent Trap	3.0	Water vapor	6 tank air samples + 3 trip blanks
	SUMMA™ canister	6.0	Organic vapors	3 tank air samples + 2 ambient air samples
WHC 222-S Laboratory	Silica Gel Sorbent Trap	1.0	Tritium-Substituted Water Vapor	1 tank air sample

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Table 3-1
 Tank BY-110 Inorganic Gas and Vapor Concentrations
 Analyses by Pacific Northwest Laboratory

Compound	CAS ¹ Number	Sample Type	Number of Samples	Average (ppmv)	Standard Deviation (ppmv)	RSD ² (%)
Ammonia, NH ₃	7664-41-7	Sorbent Trap	6	401	15	4
Carbon Dioxide ³ , CO ₂	124-38-9	SUMMA™	3	229	53	23
Carbon Monoxide ³ , CO	630-08-0	SUMMA™	3	< 76	--	--
Hydrogen ³ , H ₂	1333-74-0	SUMMA™	3	< 160	--	--
Nitric Oxide, NO	10102-43-9	Sorbent Trap	6	≤ 0.09	--	--
Nitrogen Dioxide, NO ₂	10102-44-0	Sorbent Trap	6	≤ 0.05	--	--
Nitrous Oxide ³ , N ₂ O	10024-97-2	SUMMA™	3	103	22	21
Water Vapor, H ₂ O	7732-18-5	Sorbent Trap	6	11,300 (8.0 mg/L)	250 (0.2 mg/L)	2

1. CAS = Chemical Abstracts Service.
2. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.
3. PNL SUMMA™ analyses for inorganic compounds were not completed until 70 days after sample collection, and exceeded the 60-day administratively chosen holding time (Keller 1994).

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Table 3-2
Comparison of Tank BY-110, BY-111, and BY-112 Headspace Constituents⁴

Tank:	BY-110	BY-111 ⁵	BY-112 ⁶
Date sampled, (mo/day/yr)	11/11/94	5/11/94 11/16/94	11/18/94
Headspace temperature, (°C)	27	27	23.2
Ammonia, (ppmv)	401	59	63
Hydrogen, (ppmv)	< 160	< 160	< 94
Carbon dioxide, (ppmv)	229	219	121
Carbon monoxide, (ppmv)	< 76	< 76	< 12
Nitric oxide, (ppmv)	≤ 0.09	≤ 0.15	0.18
Nitrogen dioxide, (ppmv)	≤ 0.05	≤ 0.05	≤ 0.02
Nitrous oxide, (ppmv)	103	< 67	40
Water vapor, (mg/m ³)	8.0	6.9	11.2
Water vapor, (% relative humidity)	31	27	53
Ethanenitrile (acetonitrile), (ppmv)	0.81	0.050	0.10
Propanone (acetone), (ppmv)	(3.8)	(0.48)	(1.0)
1-Butanol, (ppmv)	0.30	< 0.0011	0.059
n-Dodecane, (ppmv)	0.079	< 0.00046	0.0097
n-Tridecane, (ppmv)	0.13	(0.0015)	0.020
Total organic compounds ⁷ , (mg/m ³)	29	2.2	5.8

1. CAS = Chemical Abstracts Service.

2. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

3. PNL SUMMA™ analyses for inorganic compounds were not completed until 70 days after sample collection, and exceeded the 60-day administratively chosen holding time (Keller 1994).

4. Inorganic gas or vapor results are from PNL; organic vapor results are from ORNL, with semiquantitative values in parentheses and quantitative values not in parentheses.

5. Data are from Huckaby and Bratzel 1995a.

6. Data are from Huckaby and Bratzel 1995b.

7. Total organic compound concentration was estimated from TST sample results.

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Table 4-1
 Tank BY-110 Quantitatively Measured Organic Compounds in SUMMA™ Samples¹ --
 Analyses by Pacific Northwest Laboratory

Cmpd #	Compound	CAS ² Number	Average (ppmv)	Standard Deviation (ppmv)	RSD ³ (%)
1	Trichlorofluoromethane	75-69-4	0.34	0.06	18
2	Propanone (acetone)	67-64-1	9.9	2.5	25
3	2-Butanone	78-93-3	0.48	0.01	3
4	n-Hexane	110-54-3	0.017	0.017	96
5	4-Methyl-2-pentanone	108-10-1	0.0097	0.0006	7
6	Cyclohexane	110-82-7	0.021	0.003	13
7	n-Heptane	142-82-5	0.11	0.003	2
8	Tetrahydrofuran	109-99-9	0.15	0.002	1
9	Cyclohexanone ⁴	108-94-1	0.057	--	--
10	1,2,4-Trichlorobenzene	120-82-1	0.0159	0.0002	1
11	Pyridine	110-86-1	0.043	0.002	5
12	n-Decane	124-18-5	0.032	0.003	9
13	Methane ⁵	74-82-8	< 61	--	--

1. Analyses were not completed until 76 days after sample collection, and exceeded the 60-day administratively chosen holding time (Keller 1994).

2. CAS = Chemical Abstract Service.

3. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

4. Detected in only 1 sample.

5. PNL SUMMA™ analyses for methane were not completed until 70 days after sample collection, and exceeded the 60-day administratively chosen holding time (Keller 1994).

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Table 4-2
Tank BY-110 Quantitatively Measured Organic Compounds in TST Samples --
Analyses by Oak Ridge National Laboratory¹

Cmpd#	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Ethanenitrile (acetonitrile)	75-05-8	0.81	0.04	5
2	n-Hexane	110-54-3	0.29	0.05	18
3	n-Heptane	142-82-5	0.15	0.03	20
4	n-Dodecane	112-40-3	0.079	0.012	15
5	n-Tridecane	629-50-5	0.13	0.004	3

1. Results in this table are quantitative (as defined in Section 4.1).
2. CAS = Chemical Abstract Service.
3. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml.
4. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

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Table 4-3
 Tank BY-110 Positively Identified Organic Compounds in TST Samples --
 Analyses by Oak Ridge National Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Propanenitrile	107-12-0	0.025	0.001	4
2	Benzene	71-43-2	0.034	0.01	7
3	n-Butanenitrile	109-74-0	0.028	0.008	28
4	Toluene	108-88-3	0.029	0.006	20
5	2-Hexanone	591-78-6	0.063	0.012	19
6	n-Octane	111-65-9	0.061	0.008	14
7	n-Hexanenitrile	628-73-9	0.0058	0.0009	16
8	2-Heptanone	110-43-0	0.056	0.008	14
9	n-Nonane	111-84-2	0.030	0.005	15
10	n-Heptanenitrile	629-08-3	0.0057	0.0013	23
11	2-Octanone	111-13-7	0.010	0.001	10
12	n-Decane	124-18-5	0.024	0.005	21
13	n-Undecane	1120-21-4	0.042	0.008	19

1. Results in this table are not quantitative (as defined in Section 4.1) because measured values in at least 1 of the samples are outside instrument calibration limits.

2. CAS = Chemical Abstract Service.

3. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml.

4. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.

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Table 4-4
 Tank BY-110 Positively Identified Organic Compounds in TST Samples
 for which Practical Holding Times were Exceeded --
 Analyses by Oak Ridge National Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (ppmv)	Standard Deviation (ppmv)	RSD ⁴ (%)
1	Propanone (acetone) ⁵	67-64-1	3.8	0.3	8
2	Butanal ⁶	123-72-8	0.036	0.007	19
3	1-Butanol ⁶	71-36-3	0.30	0.02	8
4	2-Pentanone ⁶	107-87-9	0.14	0.03	20
5	n-Pentanenitrile ⁵	110-59-8	0.0068	0.0011	16

1. Practical holding times are defined and discussed in Section 4.1.
2. CAS = Chemical Abstract Service.
3. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml.
4. RSD = relative standard deviation. Burnum (1995) specifies the RSD should be less than 25 %.
5. The concentration of this analyte was not quantitatively measured (as defined in Section 4.1), because the measured concentration was outside of the instrumental calibration limits.
6. The concentration of this analyte is quantitatively measured (as defined in Section 4.1).

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Table 4-5
Tank BY-110 Comparison of Organic Compounds in TST and SUMMA™ Samples --
Analyses by Pacific Northwest Laboratory
and Oak Ridge National Laboratory

Compound	CAS ¹ Number	TST Average ² (ppmv)	SUMMA™ Average ³ (ppmv)	PRD ⁴ (%)
Ethanenitrile (acetonitrile)	75-05-8	0.81	< 0.005	> 198
Propanone (acetone)	67-64-1	3.8	9.9	89
Propanenitrile	107-12-0	0.025	< 0.005	> 133
Butanal	123-72-8	0.036	< 0.005	> 151
n-Hexane	110-54-3	0.29	0.017	178
Benzene	71-43-2	0.034	< 0.005	> 149
n-Butanenitrile	109-74-0	0.028	< 0.005	> 139
n-Heptane	142-82-5	0.15	0.11	31
Toluene	108-88-3	0.029	< 0.005	> 141
n-Decane	124-18-5	0.024	0.032	28

1. CAS = Chemical Abstract Service.

2. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml.

3. Average of 3 samples by PNL.

4. PRD = percent relative difference. Keller (1994) requires the PRD to be less than 20 %.

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Table 4-6
 Tank BY-110 Tentatively Identified Organic Compounds
 Analyses by Pacific Northwest Laboratory¹

Cmpd #	Compound	CAS ² Number	Average ³ (mg/m ³)	Standard Deviation (mg/m ³)
1	Propene	115-07-1	1.70	0.58
2	Propane	74-98-6	1.12	0.44
3	Cyclopropane	75-19-4	0.26	0.08
4	Isobutane	75-28-5	0.54	0.17
5	1-Butene	106-98-9	0.95	0.29
6	Butane	106-97-8	1.75	0.57
7	2-Methyl-1-Propene	115-11-7	0.26	0.08
8	Ethanol	64-17-5	0.19	0.06
9	2-Methyl-1-Butene	563-46-2	0.11	0.05
10	Isopropyl Alcohol ⁴	67-63-0	0.17	--
11	1-Pentene	109-67-1	0.59	0.33
12	Pentane	109-66-0	1.57	0.66
13	2-methyl-2-Propanol	75-65-0	0.10	0.03
14	4-Methyl-1-Pentene	691-37-2	0.16	0.04
15	2-Methylpentane	107-83-5	1.19	0.30
16	Butanal	123-72-8	0.13	0.04
17	3-Methylpentane	96-14-0	0.24	0.06
18	1-Hexene	592-41-6	0.20	0.05
19	Methylcyclopentane	96-37-7	0.15	0.04
20	Unknown Ketone		0.14	0.01
21	1-Butanol	71-36-3	0.59	0.04
22	2-Pentanone	107-87-9	0.81	0.06
23	Unknown C7 Alkane		0.48	0.01
24	1-Heptene	592-76-7	0.13	0.00

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Cmpd #	Compound	CAS ² Number	Average ³ (mg/m ³)	Standard Deviation (mg/m ³)
25	Unknown Alcohol ²		0.08	--
26	Unknown		0.07	0.00
27	Unknown C8 Alkane		0.29	0.00
28	2-Hexanone	591-78-6	0.29	0.01
29	Octane	111-65-9	0.27	0.00
30	Unknown C9 Alkane		0.10	0.00
31	Unknown C9 Alkene/Cycloalkane		0.06	0.00
32	3-Heptanone	106-35-4	0.42	0.01
33	2-Heptanone	110-43-0	0.23	0.00
34	Nonane	111-84-2	0.20	0.00
35	Unknown Ketone		0.24	0.01
36	2-Octanone	111-13-7	0.07	0.01
37	Unknown C11 Alkane		0.13	0.01
38	Unknown C10 Alkene/Cycloalkane		0.09	0.02
39	Undecane	1120-21-4	0.37	0.03
40	Unknown C11 Alkene/Cycloalkane ³		0.07	--
41	Unknown C12 Alkane ³		0.07	--
42	Dodecane	112-40-3	0.54	0.04
43	Undecane, 2,6-dimethyl-	17301-23-4	0.27	0.02
44	Unknown C13 Alkene/Cycloalkane		0.15	0.02
45	Unknown C14 Alkane		0.30	0.02
46	Tridecane	629-50-5	0.48	0.04
47	Unknown C14 Alkane		0.07	0.00
48	Unknown C13 Alkene/Cycloalkane		0.07	0.00

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Cmpd #	Compound	CAS ² Number	Average ³ (mg/m ³)	Standard Deviation (mg/m ³)
49	Unknown C15 Alkane		0.21	0.02
50	Tetradecane	629-59-4	0.25	0.02
Sum of tentatively identified compounds:			18.92	

1. Analyses were not completed until 76 days after sample collection, and exceeded the 60-day administratively chosen holding time (Keller 1994).

2. CAS = Chemical Abstract Service.

3. Average of three samples, values listed are estimates.

4. Detected in only 1 sample.

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Table 4-7
 Tank BY-110 Tentatively Identified Organic Compounds in TST Samples
 in the Order of Chromatographic Elution --
 Analyses by Oak Ridge National Laboratory

Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
1	Isobutane	75-28-5	0.24	0.09
2	1-Propene, 2-methyl-	115-11-7	0.44	0.05
3	Butane	106-97-8	1.4	0.4
4	1-Propene, 2-methyl-	115-11-7	0.11	0.03
5	1-Butene	106-98-9	0.053	0.054
6	2-Butene	107-01-7	0.042	0.072
7	1-Butene	106-98-9	0.015	0.026
8	Cyclopropane, 1,2-dimethyl-, trans-	2402-06-4	0.032	0.028
9	1-Butene, 2-methyl-	563-46-2	0.020	0.034
10	Butane, 2-methyl-	78-78-4	0.61	0.003
11	Trichloromonofluoromethane	75-69-4	0.13	0.006
12	1-Pentene	109-67-1	0.22	0.009
13	1,3-Pentadiene, (Z)	1574-41-0	0.084	0.015
14	Isopropyl Alcohol	67-63-0	0.17	0.09
15	1-Pentene	109-67-1	0.042	0.008
16	1-Hexene	592-41-6	0.070	0.012
17	Pentane, 2-methyl-	107-83-5	0.53	0.05
18	1-Propanol	71-23-8	0.18	0.04
19	Pentane, 3-methyl-	96-14-0	0.095	0.009
20	1-Hexene	592-41-6	0.10	0.002
21	2-Butanone	78-93-3	0.22	0.10
22	1-Hexanol	111-27-3	0.029	0.050
23	Cyclohexane	110-82-7	0.019	0.033
24	Furan, tetrahydro-	109-99-9	0.19	0.13
25	Hexane, 2-methyl-	591-76-4	0.10	0.01
26	Hexane, 3-methyl-	589-34-4	0.31	0.03
27	1-Heptene	592-76-7	0.030	0.026

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
28	3-Pentanone	96-22-0	0.042	0.003
29	2-Butanone, 3,3-dimethyl-	75-97-8	0.051	0.004
30	Cyclohexane, methyl-	108-87-2	0.079	0.006
31	Methyl Isobutyl Ketone	108-10-1	0.074	0.003
32	Pentane, 2-cyclopropyl-	5458-16-2	0.010	0.017
33	Heptane, 3-methyl-	589-81-1	0.055	0.048
34	Cyclohexane, 1,3-dimethyl-, cis-	638-04-0	0.022	0.038
35	1-Pentanol	71-41-0	0.033	0.057
36	3-Hexanone	589-38-8	0.037	0.004
37	Methanamine, N-(1-methylbutylidene)-	22431-09-0	0.015	0.026
38	Heptane, 2,6-dimethyl-	1072-05-5	0.040	0.005
39	Cyclohexane, 1,1,3-trimethyl-	3073-66-3	0.041	0.004
40	2-Hexanone, 4-methyl-	105-42-0	0.027	0.024
41	1-Hexanol	111-27-3	0.088	0.004
42	4-Heptanone & C2 Benzene		0.085	0.005
43	3-Heptanone	106-35-4	0.41	0.02
44	2-Heptanone, 6-methyl-	928-68-7	0.036	0.002
45	1H-Indene, octahydro-, cis-	4551-51-3	0.037	0.010
46	Octane, 2,6-dimethyl-	2051-30-1	0.029	0.025
47	Cyclohexane, propyl-	1678-92-8	0.045	0.002
48	1,4-Pentadien-3-ol	922-65-6	0.009	0.016
49	2-Heptanone, 6-methyl-	928-68-7	0.18	0.01
50	Nonane, 4-methyl-	17301-94-9	0.038	0.002
51	Cyclohexane, 1,1,2,3-tetramethyl-	6783-92-2	0.10	0.01
52	1-Heptanol	111-70-6	0.048	0.006
53	Cyclotetrasiloxane, octamethyl-	556-67-2	0.011	0.019
54	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.042	0.009
55	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.017	0.016
56	2-Nonenal, (E)-	18829-56-6	0.006	0.011

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
57	Nonane, 2,6-dimethyl-	17302-28-2	0.083	0.004
58	Limonene	138-86-3	0.006	0.011
59	Cyclohexane, butyl-	1678-93-9	0.027	0.001
60	Cyclopropane, 1,2-dibutyl-	41977-32-6	0.042	0.001
61	Heptane, 2,6-dimethyl-	1072-05-5	0.026	0.001
62	2-Heptanone, 6-methyl-	928-68-7	0.013	0.012
63	Undecane, 3,4-dimethyl-	17312-78-6	0.020	0.001
64	Naphthalene, decahydro-, trans-	493-02-7	0.027	0.0003
65	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.077	0.003
66	C5-cyclohexane		0.058	0.003
67	Undecane, 3,7-dimethyl-	17301-29-0	0.042	0.002
68	methyl-decahydronaphthalene		0.016	0.028
69	Undecane, 4,8-dimethyl-	17301-33-6	0.016	0.028
70	Undecane, 4,7-dimethyl-	17301-32-5	0.015	0.026
71	Decane, 2,3,8-trimethyl-	62238-14-6	0.017	0.029
72	Naphthalene, decahydro-2-methyl-	2958-76-1	0.035	0.031
73	Cyclohexane, pentyl-	4292-92-6	0.072	0.003
74	Naphthalene, decahydro-2-methyl-	2958-76-1	0.059	0.002
75	Decane, 2,5-dimethyl-	17312-50-4	0.051	0.003
76	Decane, 3-methyl-	13151-34-3	0.032	0.003
77	Nonane, 5-(2-methylpropyl)-	62185-53-9	0.057	0.002
78	Decane, 3,8-dimethyl-	17312-55-9	0.042	0.0004
79	Naphthalene, decahydro-2,3-dimethyl-	1008-80-6	0.028	0.004
80	3-Dodecene, (E)-	7206-14-6	0.015	0.013
81	Naphthalene, decahydro-2,6-dimethyl	1618-22-0	0.031	0.010
82	Naphthalene, decahydro-1,5-dimethyl	66552-62-3	0.029	0.003
83	C6-cyclohexane		0.029	0.004
84	Undecane, 2,6-dimethyl-	17301-23-4	0.25	0.01
85	Dodecane, 5-methyl- & others		0.036	0.004

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
86	Naphthalene, decahydro-1,6-dimethyl-	1750-51-2	0.042	0.004
87	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.075	0.002
88	Cyclohexane, hexyl-	4292-75-5	0.18	0.01
89	Dodecane, 4-methyl-	6117-97-1	0.042	0.004
90	Decane, 2,3,7-trimethyl-	62238-13-5	0.057	0.004
91	Cyclohexane, (2,2-dimethylcyclopentyl)-	61142-23-2	0.029	0.002
92	Tridecane, 7-methyl-	26730-14-3	0.30	0.02
93	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.028	0.003
94	Cyclopentene, 5-hexyl-3,3-dimethyl-	61142-66-3	0.027	0.001
95	Undecane, 3,9-dimethyl-	17301-31-4	0.088	0.001
96	Tridecane, 6-methyl-	13287-21-3	0.007	0.012
97	Cyclohexane, octyl-	1795-15-9	0.081	0.003
98	Tridecane, 2-methyl-	1560-96-9	0.047	0.002
99	Decane, 3,8-dimethyl-	17312-55-9	0.037	0.003
100	Dodecane, 2,6,11-trimethyl-	31295-56-4	0.33	0.02
101	Tetradecane	629-59-4	0.40	0.02
102	Tridecane, 4,8-dimethyl-	55030-62-1	0.047	0.004
103	C9-cyclohexane		0.043	0.002
104	Hexadecane	544-76-3	0.20	0.01
105	Pentadecane	629-62-9	0.16	0.01
106	Pentadecane, 2-methyl-	1560-93-6	0.006	0.011
107	Hexadecane	544-76-3	0.016	0.014
108	Tetradecanoic acid	544-63-8	0.043	0.022
109	Benzenesulfonamide, N-butyl-	3622-84-2	0.17	0.02
110	1-Heptadecanol	1454-85-9	0.008	0.014
111	Hexadecanoic acid	57-10-3	0.043	0.046
Sum of tentatively identified compounds:			10.91	

1. CAS = Chemical Abstract Service

2. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml, values listed are estimates.

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Table 4-8
 Tank BY-110 Tentatively Identified Organic Compounds in TST Samples
 Sorted Alphanumerically --
 Analyses by Oak Ridge National Laboratory

Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
22	1-Hexanol	111-27-3	0.029	0.050
2	1-Propene, 2-methyl-	115-11-7	0.44	0.05
52	1-Heptanol	111-70-6	0.048	0.006
4	1-Propene, 2-methyl-	115-11-7	0.11	0.03
5	1-Butene	106-98-9	0.053	0.054
20	1-Hexene	592-41-6	0.10	0.002
7	1-Butene	106-98-9	0.015	0.026
18	1-Propanol	71-23-8	0.18	0.04
9	1-Butene, 2-methyl	563-46-2	0.020	0.034
27	1-Heptene	592-76-7	0.030	0.026
41	1-Hexanol	111-27-3	0.088	0.004
12	1-Pentene	109-67-1	0.22	0.009
35	1-Pentanol	71-41-0	0.033	0.057
110	1-Heptadecanol	1454-85-9	0.008	0.014
15	1-Pentene	109-67-1	0.042	0.008
16	1-Hexene	592-41-6	0.070	0.012
13	1,3-Pentadiene, (Z)	1574-41-0	0.084	0.015
48	1,4-Pentadien-3-ol	922-65-6	0.009	0.016
45	1H-Indene, octahydro-, cis-	4551-51-3	0.037	0.010
6	2-Butene	107-01-7	0.042	0.072
21	2-Butanone	78-93-3	0.22	0.10
62	2-Heptanone, 6-methyl-	928-68-7	0.013	0.012
40	2-Hexanone, 4-methyl-	105-42-0	0.027	0.024
29	2-Butanone, 3,3-dimethyl-	75-97-8	0.051	0.004
56	2-Nonenal, (E)-	18829-56-6	0.006	0.011
44	2-Heptanone, 6-methyl-	928-68-7	0.036	0.002
49	2-Heptanone, 6-methyl-	928-68-7	0.18	0.01
28	3-Pentanone	96-22-0	0.042	0.003

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
80	3-Dodecene, (E)-	7206-14-6	0.015	0.013
43	3-Heptanone	106-35-4	0.41	0.02
36	3-Hexanone	589-38-8	0.037	0.004
42	4-Heptanone & C2 Benzene		0.085	0.005
109	Benzenesulfonamide, N-butyl-	3622-84-2	0.17	0.02
3	Butane	106-97-8	1.4	0.4
10	Butane, 2-methyl-	78-78-4	0.61	0.003
66	C5-cyclohexane		0.058	0.003
83	C6-cyclohexane		0.029	0.004
103	C9-cyclohexane		0.043	0.002
23	Cyclohexane	110-82-7	0.019	0.033
30	Cyclohexane, methyl-	108-87-2	0.079	0.006
34	Cyclohexane, 1,3-dimethyl-, cis-	638-04-0	0.022	0.038
39	Cyclohexane, 1,1,3-trimethyl-	3073-66-3	0.041	0.004
47	Cyclohexane, propyl-	1678-92-8	0.045	0.002
51	Cyclohexane, 1,1,2,3-tetramethyl-	6783-92-2	0.10	0.01
54	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.042	0.009
55	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.017	0.016
59	Cyclohexane, butyl-	1678-93-9	0.027	0.001
65	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.077	0.003
73	Cyclohexane, pentyl-	4292-92-6	0.072	0.003
87	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.075	0.002
88	Cyclohexane, hexyl-	4292-75-5	0.18	0.01
91	Cyclohexane, (2,2-dimethylcyclopentyl)-	61142-23-2	0.029	0.002
93	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.028	0.003
97	Cyclohexane, octyl-	1795-15-9	0.081	0.003
94	Cyclopentene, 5-hexyl-3,3-dimethyl-	61142-66-3	0.027	0.001
8	Cyclopropane, 1,2-dimethyl-, trans-	2402-06-4	0.032	0.028
60	Cyclopropane, 1,2-dibutyl-	41977-32-6	0.042	0.001
53	Cyclotetrasiloxane, octamethyl-	556-67-2	0.011	0.019
71	Decane, 2,3,8-trimethyl-	62238-14-6	0.017	0.029

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
75	Decane, 2,5-dimethyl-	17312-50-4	0.051	0.003
76	Decane, 3-methyl-	13151-34-3	0.032	0.003
78	Decane, 3,8-dimethyl-	17312-55-9	0.042	0.0004
90	Decane, 2,3,7-trimethyl-	62238-13-5	0.057	0.004
99	Decane, 3,8-dimethyl-	17312-55-9	0.037	0.003
85	Dodecane, 5-methyl- & others		0.036	0.004
89	Dodecane, 4-methyl-	6117-97-1	0.042	0.004
100	Dodecane, 2,6,11-trimethyl-	31295-56-4	0.33	0.02
24	Furan, tetrahydro-	109-99-9	0.19	0.13
33	Heptane, 3-methyl-	589-81-1	0.055	0.048
38	Heptane, 2,6-dimethyl-	1072-05-5	0.040	0.005
61	Heptane, 2,6-dimethyl-	1072-05-5	0.026	0.001
104	Hexadecane	544-76-3	0.20	0.01
107	Hexadecane	544-76-3	0.016	0.014
111	Hexadecanoic acid	57-10-3	0.043	0.046
25	Hexane, 2-methyl-	591-76-4	0.10	0.01
26	Hexane, 3-methyl-	589-34-4	0.31	0.03
1	Isobutane	75-28-5	0.24	0.09
14	Isopropyl Alcohol	67-63-0	0.17	0.09
58	Limonene	138-86-3	0.006	0.011
37	Methanamine, N-(1-methylbutylidene)-	22431-09-0	0.015	0.026
68	methyl-decahydronaphthalene		0.016	0.028
31	Methyl Isobutyl Ketone	108-10-1	0.074	0.003
64	Naphthalene, decahydro-, trans-	493-02-7	0.027	0.0003
72	Naphthalene, decahydro-2-methyl-	2958-76-1	0.035	0.031
74	Naphthalene, decahydro-2-methyl-	2958-76-1	0.059	0.002
79	Naphthalene, decahydro-2,3-dimethyl-	1008-80-6	0.028	0.004
81	Naphthalene, decahydro-2,6-dimethyl-	1618-22-0	0.031	0.010
82	Naphthalene, decahydro-1,5-dimethyl-	66552-62-3	0.029	0.003
86	Naphthalene, decahydro-1,6-dimethyl-	1750-51-2	0.042	0.004

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
50	Nonane, 4-methyl-	17301-94-9	0.038	0.002
57	Nonane, 2,6-dimethyl-	17302-28-2	0.083	0.004
77	Nonane, 5-(2-methylpropyl)-	62185-53-9	0.057	0.002
46	Octane, 2,6-dimethyl-	2051-30-1	0.029	0.025
105	Pentadecane	629-62-9	0.16	0.01
106	Pentadecane, 2-methyl	1560-93-6	0.006	0.011
17	Pentane, 2-methyl-	107-83-5	0.53	0.05
19	Pentane, 3-methyl-	96-14-0	0.095	0.009
32	Pentane, 2-cyclopropyl-	5458-16-2	0.010	0.017
101	Tetradecane	629-59-4	0.40	0.02
108	Tetradecanoic acid	544-63-8	0.043	0.022
11	Trichloromonofluoromethane	75-69-4	0.13	0.006
92	Tridecane, 7-methyl-	26730-14-3	0.30	0.02
96	Tridecane, 6-methyl-	13287-21-3	0.007	0.012
98	Tridecane, 2-methyl-	1560-96-9	0.047	0.002
102	Tridecane, 4,8-dimethyl-	55030-62-1	0.047	0.004
63	Undecane, 3,4-dimethyl-	17312-78-6	0.020	0.001
67	Undecane, 3,7-dimethyl-	17301-29-0	0.042	0.002
69	Undecane, 4,8-dimethyl-	17301-33-6	0.016	0.028
70	Undecane, 4,7-dimethyl-	17301-32-5	0.015	0.026
84	Undecane, 2,6-dimethyl-	17301-23-4	0.25	0.01
95	Undecane, 3,9-dimethyl-	17301-31-4	0.088	0.001
Sum of tentatively identified compounds:			10.91	

1. CAS = Chemical Abstract Service

2. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml, values listed are estimates.

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Table 4-9
 Tank BY-110 Tentatively Identified Organic Compounds in TST Samples
 Sorted by Estimated Concentration --
 Analyses by Oak Ridge National Laboratory

Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
3	Butane	106-97-8	1.4	0.4
10	Butane, 2-methyl-	78-78-4	0.61	0.003
17	Pentane, 2-methyl-	107-83-5	0.53	0.05
2	1-Propene, 2-methyl-	115-11-7	0.44	0.05
43	3-Heptanone	106-35-4	0.41	0.02
101	Tetradecane	629-59-4	0.40	0.02
100	Dodecane, 2,6,11-trimethyl-	31295-56-4	0.33	0.02
26	Hexane, 3-methyl-	589-34-4	0.31	0.03
92	Tridecane, 7-methyl-	26730-14-3	0.30	0.02
84	Undecane, 2,6-dimethyl-	17301-23-4	0.25	0.01
1	Isobutane	75-28-5	0.24	0.09
12	1-Pentene	109-67-1	0.22	0.009
21	2-Butanone	78-93-3	0.22	0.10
104	Hexadecane	544-76-3	0.20	0.01
24	Furan, tetrahydro-	109-99-9	0.19	0.13
49	2-Heptanone, 6-methyl-	928-68-7	0.18	0.01
88	Cyclohexane, hexyl-	4292-75-5	0.18	0.01
18	1-Propanol	71-23-8	0.18	0.04
109	Benzenesulfonamide, N-butyl-	3622-84-2	0.17	0.02
14	Isopropyl Alcohol	67-63-0	0.17	0.09
105	Pentadecane	629-62-9	0.16	0.01
11	Trichloromonofluoromethane	75-69-4	0.13	0.006
4	1-Propene, 2-methyl-	115-11-7	0.11	0.03
20	1-Hexene	592-41-6	0.10	0.002
25	Hexane, 2-methyl-	591-76-4	0.10	0.01
51	Cyclohexane, 1,1,2,3-tetramethyl-	6783-92-2	0.10	0.01
19	Pentane, 3-methyl-	96-14-0	0.095	0.009

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Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
95	Undecane, 3,9-dimethyl-	17301-31-4	0.088	0.001
41	1-Hexanol	111-27-3	0.088	0.004
42	4-Heptanone & C2 Benzene		0.085	0.005
13	1,3-Pentadiene, (Z)	1574-41-0	0.084	0.015
57	Nonane, 2,6-dimethyl-	17302-28-2	0.083	0.004
97	Cyclohexane, octyl-	1795-15-9	0.081	0.003
30	Cyclohexane, methyl-	108-87-2	0.079	0.006
65	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.077	0.003
87	Cyclohexane, 2-butyl-1,1,3-trimethyl-	54676-39-0	0.075	0.002
31	Methyl Isobutyl Ketone	108-10-1	0.074	0.003
73	Cyclohexane, pentyl-	4292-92-6	0.072	0.003
16	1-Hexene	592-41-6	0.070	0.012
74	Naphthalene, decahydro-2-methyl-	2958-76-1	0.059	0.002
66	C5-cyclohexane		0.058	0.003
77	Nonane, 5-(2-methylpropyl)-	62185-53-9	0.057	0.002
90	Decane, 2,3,7-trimethyl-	62238-13-5	0.057	0.004
33	Heptane, 3-methyl-	589-81-1	0.055	0.048
5	1-Butene	106-98-9	0.053	0.054
75	Decane, 2,5-dimethyl-	17312-50-4	0.051	0.003
29	2-Butanone, 3,3-dimethyl-	75-97-8	0.051	0.004
52	1-Heptanol	111-70-6	0.048	0.006
98	Tridecane, 2-methyl-	1560-96-9	0.047	0.002
102	Tridecane, 4,8-dimethyl-	55030-62-1	0.047	0.004
47	Cyclohexane, propyl-	1678-92-8	0.045	0.002
111	Hexadecanoic acid	57-10-3	0.043	0.046
108	Tetradecanoic acid	544-63-8	0.043	0.022
103	C9-cyclohexane		0.043	0.002
54	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.042	0.009
15	1-Pentene	109-67-1	0.042	0.008

WHC-SD-WM-ER-429 Rev. 2A

Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
86	Naphthalene, decahydro-1,6-dimethyl-	1750-51-2	0.042	0.004
6	2-Butene	107-01-7	0.042	0.072
89	Dodecane, 4-methyl-	6117-97-1	0.042	0.004
60	Cyclopropane, 1,2-dibutyl-	41977-32-6	0.042	0.001
78	Decane, 3,8-dimethyl-	17312-55-9	0.042	0.0004
67	Undecane, 3,7-dimethyl-	17301-29-0	0.042	0.002
28	3-Pentanone	96-22-0	0.042	0.003
39	Cyclohexane, 1,1,3-trimethyl-	3073-66-3	0.041	0.004
38	Heptane, 2,6-dimethyl-	1072-05-5	0.040	0.005
50	Nonane, 4-methyl-	17301-94-9	0.038	0.002
99	Decane, 3,8-dimethyl-	17312-55-9	0.037	0.003
36	3-Hexanone	589-38-8	0.037	0.004
45	1H-Indene, octahydro-, cis-	4551-51-3	0.037	0.010
85	Dodecane, 5-methyl- & others		0.036	0.004
44	2-Heptanone, 6-methyl-	928-68-7	0.036	0.002
72	Naphthalene, decahydro-2-methyl-	2958-76-1	0.035	0.031
35	1-Pentanol	71-41-0	0.033	0.057
8	Cyclopropane, 1,2-dimethyl-, trans-	2402-06-4	0.032	0.028
76	Decane, 3-methyl-	13151-34-3	0.032	0.003
81	Naphthalene, decahydro-2,6-dimethyl-	1618-22-0	0.031	0.010
27	1-Heptene	592-76-7	0.030	0.026
91	Cyclohexane, (2,2-dimethylcyclopentyl)-	61142-23-2	0.029	0.002
22	1-Hexanol	111-27-3	0.029	0.050
83	C6-cyclohexane		0.029	0.004
46	Octane, 2,6-dimethyl-	2051-30-1	0.029	0.025
82	Naphthalene, decahydro-1,5-dimethyl-	66552-62-3	0.029	0.003
93	Cyclohexane, 2,4-diethyl-1-methyl-	61142-70-9	0.028	0.003
79	Naphthalene, decahydro-2,3-dimethyl-	1008-80-6	0.028	0.004
59	Cyclohexane, butyl-	1678-93-9	0.027	0.001

WHC-SD-WM-ER-429 Rev. 2A

Cmpd #	Compound	CAS ¹ Number	Average ² (mg/m ³)	Standard Deviation (mg/m ³)
64	Naphthalene, decahydro-, trans-	493-02-7	0.027	0.0003
40	2-Hexanone, 4-methyl-	105-42-0	0.027	0.024
94	Cyclopentene, 5-hexyl-3,3-dimethyl-	61142-66-3	0.027	0.001
61	Heptane, 2,6-dimethyl-	1072-05-5	0.026	0.001
34	Cyclohexane, 1,3-dimethyl-, cis-	638-04-0	0.022	0.038
9	1-Butene, 2-methyl-	563-46-2	0.020	0.034
63	Undecane, 3,4-dimethyl-	17312-78-6	0.020	0.001
23	Cyclohexane	110-82-7	0.019	0.033
71	Decane, 2,3,8-trimethyl-	62238-14-6	0.017	0.029
55	Cyclohexane, 1-methyl-2-propyl-	4291-79-6	0.017	0.016
107	Hexadecane	544-76-3	0.016	0.014
68	methyl-decahydronaphthalene		0.016	0.028
69	Undecane, 4,8-dimethyl-	17301-33-6	0.016	0.028
80	3-Dodecene, (E)-	7206-14-6	0.015	0.013
7	1-Butene	106-98-9	0.015	0.026
70	Undecane, 4,7-dimethyl-	17301-32-5	0.015	0.026
37	Methanamine, N-(1-methylbutylidene)-	22431-09-0	0.015	0.026
62	2-Heptanone, 6-methyl-	928-68-7	0.013	0.012
53	Cyclotetrasiloxane, octamethyl-	556-67-2	0.011	0.019
32	Pentane, 2-cyclopropyl-	5458-16-2	0.010	0.017
48	1,4-Pentadien-3-ol	922-65-6	0.009	0.016
110	1-Heptadecanol	1454-85-9	0.008	0.014
96	Tridecane, 6-methyl-	13287-21-3	0.007	0.012
56	2-Nonenal, (E)-	18829-56-6	0.006	0.011
58	Limonene	138-86-3	0.006	0.011
106	Pentadecane, 2-methyl-	1560-93-6	0.006	0.011
Sum of tentatively identified compounds:			10.91	

1. CAS = Chemical Abstract Service

2. Average of 4 TST samples by ORNL: 3 were 250 ml and 1 was 50 ml, values listed are estimates.

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		ECN No. N/A

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EDMC	H6-08	X			
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TCRC (10)	R2-12	X			