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7. Abstract

Tank 241-AX-102 was sampled in 1995, and laboratory analyses were conducted to support general safety screening and specific organic safety evaluation requirements. Exotherms were observed and fuel in the form of total organic carbon was identified. However, with the significant amount of moisture present and the low-tank temperatures, the tank should remain safe in its current configuration.

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Tank Characterization Report for Single-Shell Tank 241-AX-102

J. P. Sederburg
R. D. Cromar
Westinghouse Hanford Company

J. L. Stroup
ICF Kaiser Hanford Company

Date Published
August 1995

Prepared for the U.S. Department of Energy
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Westinghouse Hanford Company P.O Box 1970 Richland, Washington

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EXECUTIVE SUMMARY

This Tank Characterization Report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell underground storage tank 241-AX-102. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994),[1] Milestone M-44-08.

Tank 241-AX-102 is located in the AX Tank Farm in the 200 East Area of the Hanford Site. The tank went into service in 1966 and was initially used as a Plutonium-Uranium Extraction (Facility) (PUREX) high-level waste receiver. From 1969 through 1975, tank 241-AX-102 was used to store high-activity waste from B Plant. Other wastes routed to tank 241-AX-102 until 1980 when it was deactivated included evaporator feed, evaporator slurry, complexant, and concentrated complexant wastes.

In 1988, tank 241-AX-102 was declared an assumed leaker. Approximately 12,000 L (3,000 gal) are estimated to have leaked from the tank. The supernatant in the tank was pumped and the tank was interim stabilized by September 1988.

A description and status of the tank are summarized in Table ES-1 and Figure ES-1. Because the tank is inactive, interim stabilized, interim isolated (currently called intrusion

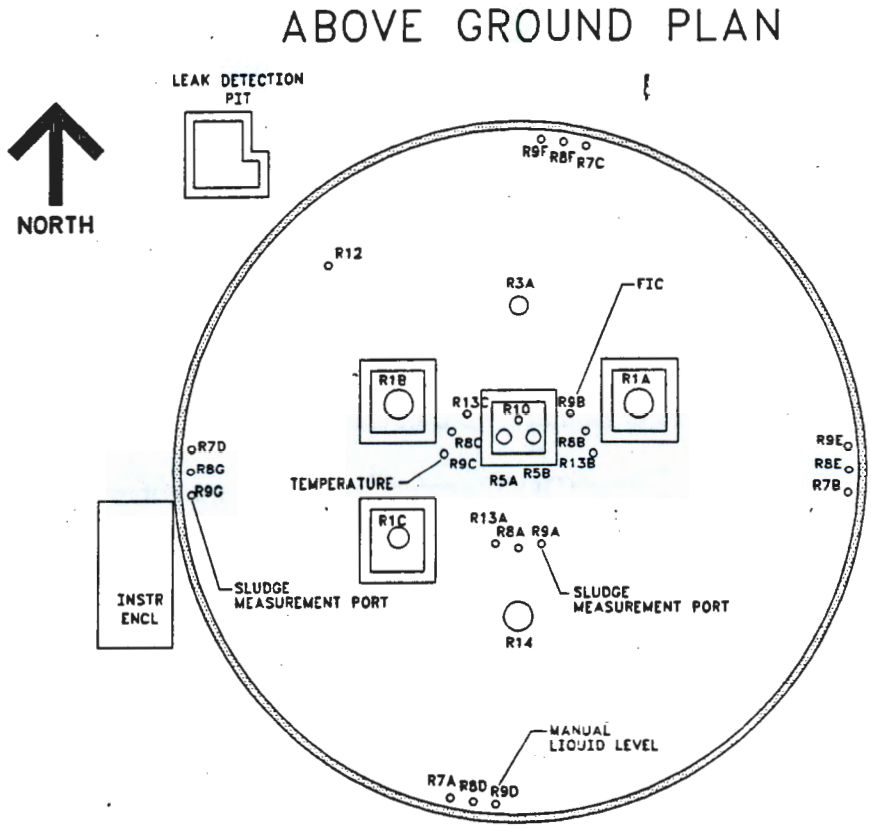
[1]Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order*, as amended, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

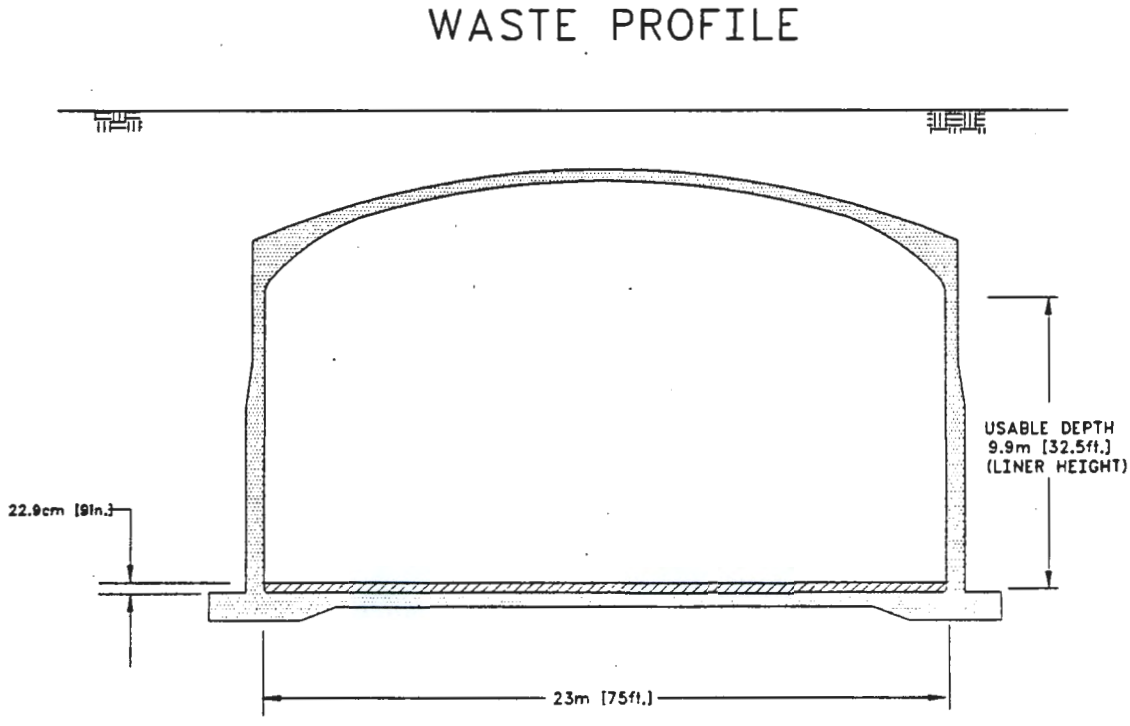
Table ES-1. Description and Status of Tank 241-AX-102.

TANK DESCRIPTION	
Type:	Single shell
Constructed:	1964-65
In service:	1966
Diameter:	23 m (75 ft)
Usable depth:	9.9 m (32.5 ft)
Capacity:	3,800 kL (1,000 kgal)
Bottom shape:	Flat
Ventilation:	Passive
TANK STATUS (as of February 1995)	
Watch List:	Organic[1]
Contents:	Concentrated complexed waste
Total waste volume:	150 kL (39 kgal)
Sludge volume:	27 kL (7 kgal)
Salt volume:	110 kL (29 kgal)
Supernatant volume:	11 kL (3 kgal)
Manual tape surface level:	22.9 cm (9 in.) (01/02/95)
Temperature:	55 °C (131 °F) to 23 °C (73 °F) from January 1990 to January 1995
Integrity category:	Assumed leaker
ISOLATION STATUS	
Interim stabilized:	1988
Intrusion prevention:	1982

[1]Tank 241-AX-102 was added to the Organic Tanks Watch List in May 1994 (Payne, M. A., 1994, *Revision of the Organic Tanks Watch List* [external letter 9453328 to R. E. Gerton, May 15], Westinghouse Hanford Company, Richland, Washington).

Figure ES-1. Tank 241-AX-102 Views.





prevention), and not receiving or transferring waste, the volume of waste will not be changing. The tank, which has an operational capacity of 3,800,000 L (1,000,000 gal), contains 150,000 L (39,000 gal) of waste existing primarily as salt cake and sludge.

This report summarizes seven sampling and analysis events. Waste composition and properties are based on auger samples taken from the tank in 1995 to support requirements of the organic safety program. The 1995 samples were obtained in accordance with the requirements of the data quality objective (DQO) process and therefore comply with the recent requirements of the Safety Screening DQO (Babad et al. 1995a)[1] and the Organic Safety DQO (Babad et al. 1995b)[2].

Only those analyses required by the two safety DQOs were performed on the 1995 samples. The analyses included thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), total alpha, cyanide, total organic carbon (TOC), hydroxide, nitrate, and nitrite.[3] The analyses revealed the following.

---

[1]Babad, H., J. W. Hunt, and K. S. Redus, 1995a, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

[2]Babad, H., S. M. Blacker, and K. S. Redus, 1995b, *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue*, WHC-SD-WM-DQO-006, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

[3]Flammable gas concentration is one of the analyses required by the Safety Screening DQO (Babad et al. 1995a), but these are not provided in this report because vapor sampling was conducted in June 1995 and the time necessary to complete the required analysis has not yet elapsed.

- Exotherms above the DQO limit (481 J/g, dry basis) from DSC were observed in the sampled waste.

- All of the total alpha results as reported in units of μCi/g when converted to g/L were below the DQO limit of 1 g/L.

- All moisture measurements made using TGA were above the DQO limit of 17 wt% H_2O.

- TOC measurements exceeded the 5 wt% (dry basis) criterion of the Organic Safety DQO (Babad et al. 1995b).

- The historical tank content estimate model of this tank's contents could not be fully substantiated.

The 1995 sampling event did not fully meet the objectives specified in the applicable DQOs; however, the intent of both DQOs was met. The data resulting from analyses of the 1995 sampling event show that tank 241-AX-102 has been properly identified and classified as an Organic Watch List (OWL) tank. Furthermore, the controls that have been in place because tank 241-AX-102 is on the OWL will be maintained as a result of the findings from the 1995 sampling event. Further requirements will not be imposed because the waste temperature is low and precludes any imminent safety hazard. However, due to limited data and inconsistencies, no summary table of concentration and inventory is presented herein.

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LIST OF TERMS

B	B Plant high-level waste from cesium/strontium recovery process at B Plant
CASS	Computer Automated Surveillance System
CW	cladding waste
DBP	dibutylphosphate
DOE	U.S. Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
EDTA	ethylenediametetraacetic acid
HEDTA	N-(hydroxyethyl)-ethylenediaminetriacetic acid
HTCE	historical tank content estimate
IX	ion exchange
LL	lower limit
NA	not available
N/A	not applicable
NPH	normal paraffin hydrocarbon
NTA	nitrilotriacetate
OWL	Organic Watch List
OWW	organic wash waste
PL	Purex low-level waste from the PUREX process
purex	plutonium uranium extraction
PUREX	Plutonium-Uranium Extraction (Facility)
RPD	relative percent difference
SU	supernatant (generic term)
TBP	tributylphosphate
TCP	Tank Characterization Plan
TCR	Tank Characterization Report
TGA	thermogravimetric analysis
TLM	tank layer model
TOC	total organic carbon
UL	upper limit
WHC	Westinghouse Hanford Company
WTR	water
1C	first-cycle decontamination

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TANK CHARACTERIZATION REPORT FOR SINGLE-SHELL TANK 241-AX-102

1.0 INTRODUCTION

This Tank Characterization Report (TCR) presents an overview of single-shell tank 241-AX-102 and its waste contents. It provides estimated concentrations and inventories for the waste components based on the latest sampling and analysis activities and background tank information. This TCR describes the results of seven sampling events as follows.

Two auger samples were taken during February 1995 to support decisions concerning safety. One liquid grab sample was taken during August 1988. One liquid grab sample was taken in approximately February 1980. One liquid grab sample was taken in approximately January 1980. Six sludge samples were taken between February 1977 and July 1977. Twelve sludge samples were taken in approximately January 1977. One sludge sample was taken in approximately August 1974.

Tank 241-AX-102 is not in active service, has been interim stabilized (September 1988) and interim isolated (currently termed intrusion prevention [1982]), and can be expected to have no additional transfers (in or out) that will alter the composition of the waste. The concentration and inventory estimates reported in this document reflect the best estimate of the composition of the waste based on historical knowledge and the most recent and available data. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994), Milestone M-44-08.

1.1 PURPOSE

The purpose of this report is to summarize the information about the use and contents of tank 241-AX-102. When possible, this information will be used to assess issues associated with safety, operations, environmental, and process development activities. This report also provides a focal point for the current historic information about tank 241-AX-102.

1.2 SCOPE

The primary intent of the 1995 sampling and analysis event was to address the Safety Screening Data Quality Objective (DQO) (Babad et al. 1995a). In doing this, the auger samples acquired in 1995 support a safety assessment of tank 241-AX-102 waste. The technical basis for this safety assessment is described in the Safety Screening DQO (Babad et al. 1995a) and in the Organic Safety DQO (Babad et al. 1995b) and only includes the analyses of thermogravimetric analysis (TGA), used to determine the concentration of

water in the waste; differential scanning calorimetry (DSC), used to measure the energy produced from any chemical reaction(s); as well as concentrations of total alpha, cyanide, total organic carbon (TOC), hydroxide (OH^-), nitrate (NO_3^-), and nitrite (NO_2^-).[1]

The liquid grab sample acquired in 1988 was taken to assist in the assessment of a potential tank leak and to determine how to disposition the fluid remaining in the tank at that time. There were no DQOs at that time.

The liquid grab sample acquired in February 1980 was taken to "determine the nucleation and crystallization point of this complexed waste at 5 °C (~40 °F) as a function of waste volume reduction (WVR)." The liquid grab sample acquired in January 1980 was taken to perform a hot boildown of the waste. The sludge samples acquired in 1974 and 1977 were taken to support the sluicing of tank 241-AX-102 solids. There were no DQOs for these sampling events.

[1]Flammable gas concentration is one of the decision criteria for the Safety Screening DQO (Babad et al. 1995a). However, this report does not include any information on vapor space sampling and analysis to determine the composition of the tank headspace gases. Vapor sampling was conducted on June 27, 1995, and there has not yet been sufficient time for the requisite analyses to be completed and reported.

2.0 HISTORICAL TANK INFORMATION

Tank 241-AX-102 is not in active service. It is expected that there will be no transfers into the tank. The volume status of the tank is provided in documentation that is routinely issued. Tank history includes tank design information, waste transfer history, and waste temperature and level surveillance information.

2.1 TANK STATUS

According to Hanlon (1995), tank 241-AX-102 contains 150,000 L (39,000 gal) of waste: 27,000 L (7,000 gal) are sludge; 110,000 L (29,000 gal) are salt cake; and 12,000 L (3,000 gal) are supernatant (65,000 L [17,000 gal] of drainable liquid). The solids volume estimate reported in Hanlon (1995) is based on a measurement obtained on September 6, 1988. It is considered to be accurate. The salt cake portion of waste represents the major tank contents. Periodic measurements using a manual tape are obtained to ensure that the surface level is not dramatically changing.

An in-tank temperature of 23 °C (73 °F) was reported for tank 241-AX-102 in February 1995. This was for the bottom thermocouple (defined as #1) and is for a point in time (i.e., not an average, maximum, or minimum). Waste levels and tank temperatures are further discussed in Section 2.4.

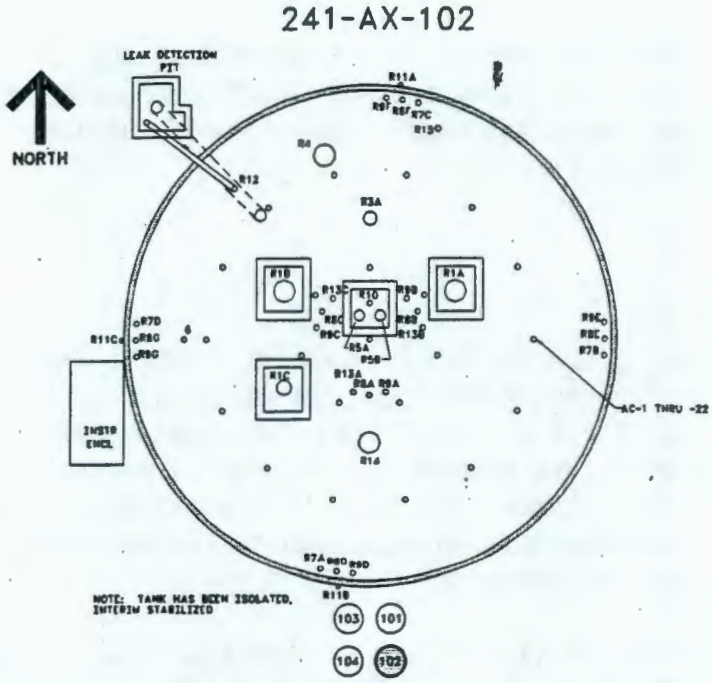
The tank is classified as an assumed leaker and is on the Organic Tank Watch List. There are no unreviewed safety questions associated with tank 241-AX-102 at this time. All tank monitoring instruments are in compliance with documented standards (Hanlon 1995).

Tank 241-AX-102 is passively ventilated and was removed from service in 1980. In 1988, the tank was declared to be an assumed leaker with an approximate leak volume of 12,000 L (3,000 gal). Removal of tank 241-AX-102 from service means that it is not allowed to continue to receive waste transfers.

2.2 TANK DESIGN AND BACKGROUND

Tank 241-AX-102 consists of a carbon steel liner surrounded by a reinforced concrete shell. The tank has a design capacity for storing 3,800,000 L (1,000,000 gal) of waste. The tank has a diameter of 23 m (75 ft) and a usable depth of 9.9 m (32.5 ft). Instruments access tank 241-AX-102 through risers and monitor the temperature, liquid level, sludge level, and other bulk tank characteristics (Bell 1994). The locations of these risers are depicted in Figure 2-1. A diagram of a single-shell tank is presented in Figure 2-2. For more information about the AX Tank Farm and single-shell tanks, refer to WHC-SD-WM-TI-648, *Tank Characterization Reference Guide* (Simpson et al. 1994).

Figure 2-1. Riser Configuration for Tank 241-AX-102.

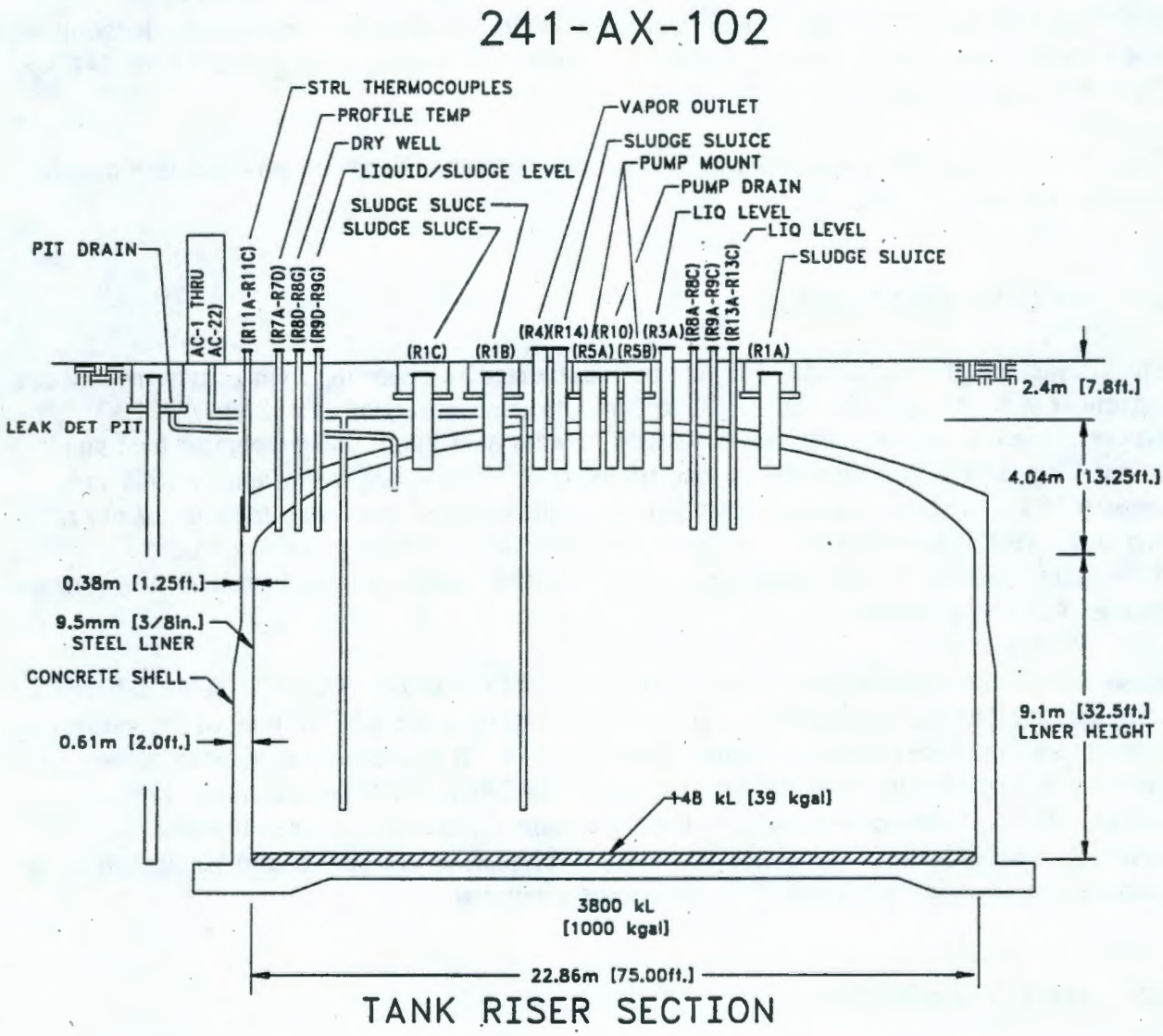


NO.	DIA. (in.)	DESCRIPTION AND COMMENTS
R1A	34	(SLUICER) WC
R1B	34	(PUMP) WC
R1C	12	WC
R3A	16	OBSV PORT, BM
R5A	12	(SALT WELL SCREEN) WC
R5B	12	(PUMP ACCESS) WC
R6	4	BG
R7A	4	FLANGE
R7B	4	TEMPERATURE
R7C	4	TEMPERATURE, BM
R7D	4	TEMPERATURE
R8A	6	PLUG
R8B	6	PLUG
R8C	6	PLUG - FLANGE WITH BALE
R8D	6	PLUG - FLANGE WITH BALE
R8E	6	PLUG
R8F	6	PLUG - FLANGE WITH BALE
R8G	6	PLUG - FLANGE WITH BALE

NO.	DIA. (in.)	DESCRIPTION AND COMMENTS
R9A	6	SMP
R9B	6	FIC
R9C	6	TEMPERATURE
R9D	6	LLR
R9E	6	BREATHER FILTER
R9F	6	FLANGE
R9G	6	SMP
R10	4	(PIT DRAIN) WC
R11A	7.75	STRUCTURAL TEMPERATURE - BG
R11B	7.75	STRUCTURAL TEMPERATURE - BG
R11C	7.75	STRUCTURAL TEMPERATURE - BG
R12	4	PIT DRAIN - BG
R13A	4	WC
R13B	4	TEMPERATURE
R13C	4	WC
R14	42	STEAM COIL
R15	4	BG
R4	20	VAPOR OUTLET - BG

WC - Weather cover
BM - Bench mark
BG - Below Grade
SMP - Sludge Measurement Port
FIC - Food Instrument Corp (Level Indicating Transmitter)
LLR - Liquid Level Reel (Manual tape)

Figure 2-2. Basic Design of a Single-Shell Tank.



Tank 241-AX-102 was constructed between 1964-65 and went into service in 1966. It is one of four tanks that comprise the 241-AX Tank Farm located on the eastern end of the 200 East Area of the Hanford Site. Figure 2-3 details the 200 East Area and the location of the 241-AX Tank Farm. Tank 241-AX-102 is located in the southeast corner of the 241-AX Tank Farm (see Figure 2-1).

Tank 241-AX-102 was constructed as a "flat bottom" tank. There are no cascading transfer lines to, or from, this tank.

2.3 PROCESS KNOWLEDGE

The fill history of tank 241-AX-102 can be summarized as collecting high-activity neutralized current acid waste generated by the Plutonium-Uranium Extraction (Facility) (PUREX) process, waste from the waste fractionization process at B Plant, and evaporator feed and slurry. Figure 2-4 illustrates the service life of tank 241-AX-102 from January 1965 to January 1995. The surface-level fluctuations are the result of the active transfer history of this tank. (Fill data before 1981 were not available from the Computer Automated Surveillance System [CASS] database and are therefore based on other historical data sources such as Anderson [1990]).

Many waste types have been transferred into and out of tank 241-AX-102. Table 2-1 uses a compilation of transaction records to present an estimate of the total volume of the various waste types that have entered the tank. Table 2-2 lists, in greater detail, some of these transfers and summarizes the transfer history of tank 241-AX-102 from January 1965 to January 1975. It should be noted that there are some differences between the data of Tables 2-1 and 2-2. It is speculated that these discrepancies are due to errors/assumptions in source documentation from which the data were generated.

2.3.1 Waste Transfer History

For the last two quarters of 1965 and the first two quarters of 1966, tank 241-AX-102 had a water heel left from construction. The tank began receiving waste from PUREX in the third quarter of 1966 and continued to receive waste until the fourth quarter of 1968.

In the last quarter of 1968, tank 241-AX-102 was deemed a "spare tank" and most of the supernatant was pumped out of the tank.

During the first quarter of 1969, the tank was flushed to remove solids. After flushing in early 1969, the tank received B Plant waste through the first quarter of 1971. The tank received some PUREX waste during the second and third quarters of 1969.

Figure 2-3. Location of the AX Tank Farm.

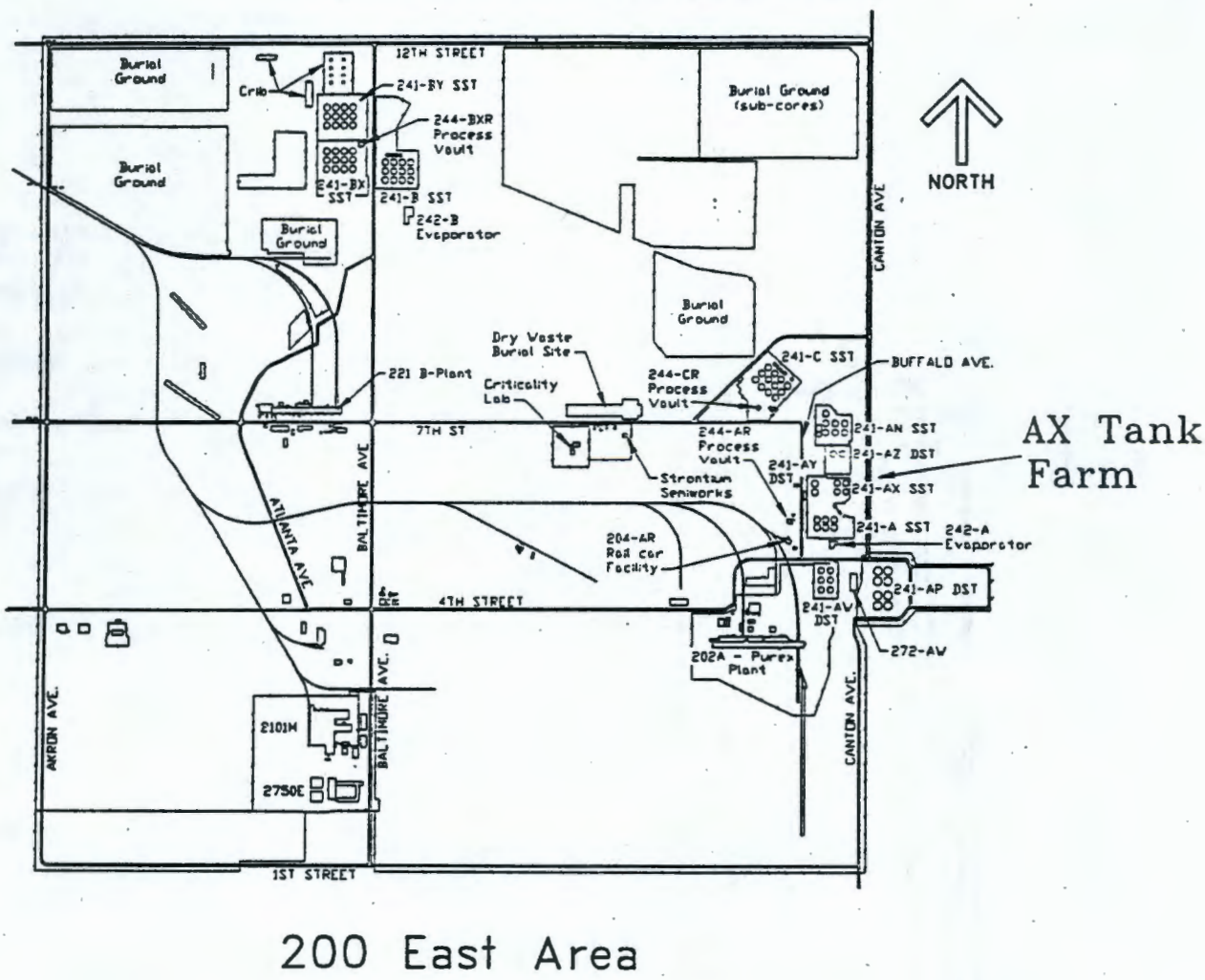


Figure 2-4. Tank 241-AX-102 Fill History.

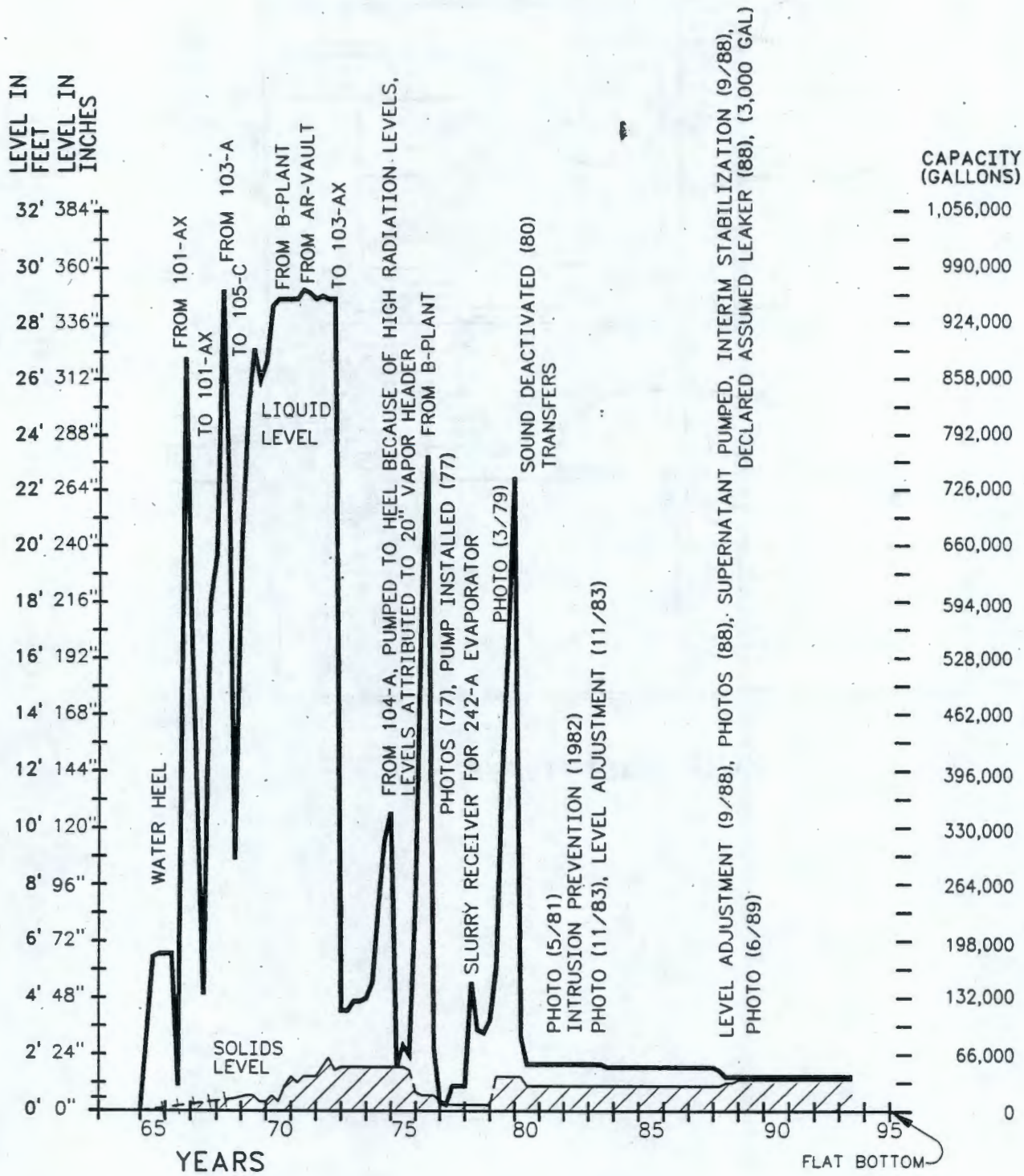


Table 2-1. Synopses of Major Waste Transferred into Tank 241-AX-102.

Transfer type	Volume kL (kgal)
Addition of WTR	1,374 (363)
Addition of PUREX wastes	1,915 (506)
Addition of UNKNOWN	2,502 (661)
Addition of SU	9,551 (2,523)
Addition of B	12,966 (3,425)

B = B Plant high-level waste from cesium/strontium recovery process at B Plant
OWW = Organic wash waste
PUREX = Plutonium-Uranium Extraction (Facility)
SU = Supernatant (generic term)
WTR = Water

Between 1971-76, numerous tank-to-tank transfers were completed. In 1976-77, the tank was sluiced and the waste was routed to B Plant for removal of the high-heat-producing strontium-90. From 1977 to 1980, tank 241-AX-102 was used in conjunction with 242-A Evaporator Operations primarily as a dilute complex receiver and complex concentrate storage tank.

Currently, the tank waste is classified as concentrated complexant waste. The tank was declared inactive in 1980, and intrusion prevention was completed in 1982. In 1988, the tank was declared an assumed leaker with a 12,000 L (3,000 gal) leak volume. A level adjustment was made before pumping in 1988 to learn if evaporation was the cause of liquid loss. It was determined that evaporation was not the cause of the liquid loss therefore the supernatant was pumped and the tank declared interim stabilized in September 1988.

The waste transfer history of tank 241-AX-102 is detailed in Table 2-2. Figure 2-4 depicts the fill history of the tank based on information from WHC-MR-0132, *A History of the 200 Area Tank Farms* (Anderson 1990) and WHC-SD-WM-TI-615, *Waste Status and Transaction Record Summary for the Northeast Quadrant* (Agnew and Brown 1994). Table 2-1 presents an estimate of the total volumes of the specific waste types that were added to the tank.

2.3.2 Historical Estimation of Tank Contents

Tank inventory estimates based on the tank layer model, the waste status and transaction record summary, and the Hanford Site defined waste types developed by Los Alamos National Laboratory are presented in WHC-SD-WM-ER-349, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Areas, ICF Kaiser Hanford*

Table 2-2. Tank 241-AX-102 Major Transfers.[1,2]

Year: Qtr	Waste received by tank 241-AX-102			Waste transferred out of tank 241-AX-102		
	Transaction source	Waste type	Waste amount kL (kgal)	Receiving tank	Waste type	Waste amount kL (kgal)
1965:3-1965:4	UNSPECIFIED	WTR	1,374 (363)	--	--	--
1966:3-1967:3	PUREX	OWW	1,620 (428)	--	--	--
1966:4	AX-101	SU	1,117 (295)	--	--	--
1967:1-1967:3	--	--	--	AX-101	SU	5,262 (1,390)
1967:2	A-103	SU	318 (84)	--	--	--
1967:4	AX-101	SU	1,738 (459)	--	--	--
1968:1	--	--	--	C-102	SU	95 (25)
1968:1	--	--	--	C-105	SU	6,492 (1,715)
1968:1-1969:1	--	--	--	TY-103	SU	2,067 (546)
1968:1-1968:2	A-103	SU	5,065 (1,338)	--	--	--
1969:2-1971:1	B Plant	B	12,966 (3,425)	--	--	--
1969:2-1969:3	PUREX	PL	295 (78)	--	--	--
1971:2	244-AR (AR Vault)	UNSPECIFIED	129 (34)	--	--	--
1973:1-1975:2	--	--	--	AX-103	SU	3,577 (945)

[1]This table does not include activities from 1975 through 1988, which are known to include solids removal due to sluicing, 242-A evaporator support, and interim stabilized pumping.

[2]Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1994b, *Supporting Document for the Northeast Quadrant Hanford Tank Content Estimate for AX Tank Farm*, WHC-SD-WM-ER-309, Rev. 0, ICF Kaiser Hanford Company, Richland, Washington.

B = B Plant high-level waste from cesium/strontium recovery process at B Plant
OWW = Organic wash waste
PL = Purex low-level waste from the PUREX process
PUREX = Plutonium-Uranium Extraction (Facility)
SU = Supernatant (generic term)
WTR = Water

Table 2-3. Historical Tank Inventory Estimate. (sheet 1 of 2)

Single-shell tank 241-AX-102			
Solids composite inventory estimate[1,2]			
Physical properties			
Total solid waste	2.07E+05 kg (36 kgal)		
Heat load	4.76 kW (1.62E+04 Btu/h)		
Bulk density	1.52 (g/cc)		
Void fraction	0.795		
Water wt%	45.9		
TOC wt% C (wet)	0.313		
Chemical constituents	**mole/L**	**ppm**	**kg**
Na^{+1}	8.87	1.34E+05	2.78E+04
Al^{+3}	1.35	2.40E+04	4.98E+03
Fe^{+3} (total Fe)	0.894	3.29E+04	6.80E+03
Cr^{+3}	2.27E-02	777	161
Bi^{+3}	7.38E-04	102	21.0
La^{+3}	0	0	0
Ce^{+3}	1.48E-06	0.137	2.83E-02
Zr [as $ZrO(OH)_2$]	1.64E-03	98.2	20.3
Pb^{+2}	1.26E-04	17.2	3.56
Ni^{+2}	1.50E-02	581	120
Sr^{+2}	3.45E-04	19.9	4.12
Mn^{+4}	1.36E-03	49.1	10.2
Ca^{+2}	0.170	4.49E+03	930
K^{+1}	3.60E-02	926	192
OH^{-1}	8.32	9.31E+04	1.93E+04
NO_3^{-1}	4.25	1.73E+05	3.59E+04
NO_2^{-1}	1.20	3.64E+04	7.54E+03
CO_3^{-2}	0.427	1.69E+04	3.49E+03
PO_4^{-3}	8.63E-02	5.39E+03	1.12E+03
SO_4^{-2}	0.394	2.49E+04	5.16E+03
Si (as SiO_3^{-2})	6.14E-02	1.13E+03	235
F^{-1}	0.108	1.35E+03	280
Cl^{-1}	9.73E-02	2.27E+03	470
$C_6H_5O_7^{-3}$	2.20E-02	2.74E+03	568
$EDTA^{-4}$	3.84E-03	728	151

Table 2-3. Historical Tank Inventory Estimate. (sheet 2 of 2)

Chemical constituents	mole/L	ppm	kg
$HEDTA^{-3}$	1.82E-05	3.28	0.680
NTA^{-3}	0	0	0
$glycolate^{-1}$	7.55E-02	3.73E+03	771
$acetate^{-1}$	2.45E-02	950	197
$oxalate^{-2}$	0	0	0
DBP	3.57E-03	625	129
NPH	0	0	0
CCL_4	0	0	0
hexone	0	0	0
$Fe(CN)_6^{-4}$	0	0	0
Radiological constituents			
Pu	–	2.02 (μCi/g)	6.96 (kg)
U	1.22E-02 (M)	1.92E+03 (μg/g)	397 (kg)
Cs	0.485 (Ci/L)	319 (μCi/g)	6.60E+04 (Ci)
Sr	4.85 (Ci/L)	3.19E+03 (μCi/g)	6.60E+05 (Ci)

[1]Composite inventory excludes supernatant, diatomaceous earth, and cement.
[2]Unknowns in tank inventory are assigned by tank layer model (TLM).

DBP = Dibutylphosphate
EDTA = Ethylenediametetraacetic acid
HEDTA = N-(hydroxyethyl)-ethylenediaminetriacetic acid
NPH = Normal paraffin hydrocarbon
NTA = Nitrilotriacetate
TOC = Total organic carbon

Company (Brevick et al. 1994a). This model combines waste type inventories and historical waste stream data to obtain an estimate of the current chemical contents of tank 241-AX-102. This estimate is included as Table 2-3.

2.4 SURVEILLANCE DATA

2.4.1 Surface-Level Readings

To determine the surface level of the waste, tank 241-AX-102 is equipped with a manual tape, which uses a conductivity probe that is lowered by a hand crank until an electric circuit is completed as the probe contacts the waste surface. Measurements of the surface level are made on a quarterly basis through riser 9D. The data obtained from field readings of the manual tape are input to the CASS on a periodic basis. Liquid waste volume is determined by the manual tape while solid waste volume is determined by a photographic evaluation and a sludge-level measurement device. The most recent solids level recorded was 33 cm (13 in.) on September 6, 1988. Figure 2-5 graphically represents the surface level from 1983 to the present. Solids consolidation after the interim stabilization efforts of 1988 are believed to account for the current lower surface-level reading.

No liquid observation well is available for establishing interstitial liquid level in the solids of tank 241-AX-102.

There were eleven dry wells associated with tank 241-AX-102. In 1975, one was capped due to interference with construction. A review of historical data from the remaining ten dry wells shows no apparent increase in activity, although one well has some anomalous readings. Tank 241-AX-102 also has a leak detection pit. This was one of the first leak detection pits ever used at the Hanford Site. Problems of false leak indications and erratic data have been noted since its construction. Assessment of tank integrity based on the available historic leak detection pit and dry well data for tank 241-AX-102 is inconclusive.

2.4.2 Internal Tank Temperatures

To measure in-tank temperatures, a probe with 18 thermocouples assembled in a pipe (termed a thermocouple tree) is in riser 9C (see Figure 2-1 for the location of this riser). The thermocouple tree monitors the waste temperatures at various levels in the tank.

Review of the tank 241-AX-102 level history indicates that thermocouple 1 is located in or near the solids level and the rest of the thermocouples are in the vapor space. The first 12 thermocouples are evenly spaced every 0.6 m (2 ft) along the tree starting at 330 mm (13 in.) from the bottom (defined as probe 1) up to probe 12. Probes 13-18 are spaced every 1.2 m (4 ft). Other risers previously used for monitoring the waste temperature were 7A-7D, 11A-11C, and 13A-13C. Temperature data from the tank in-service date to January

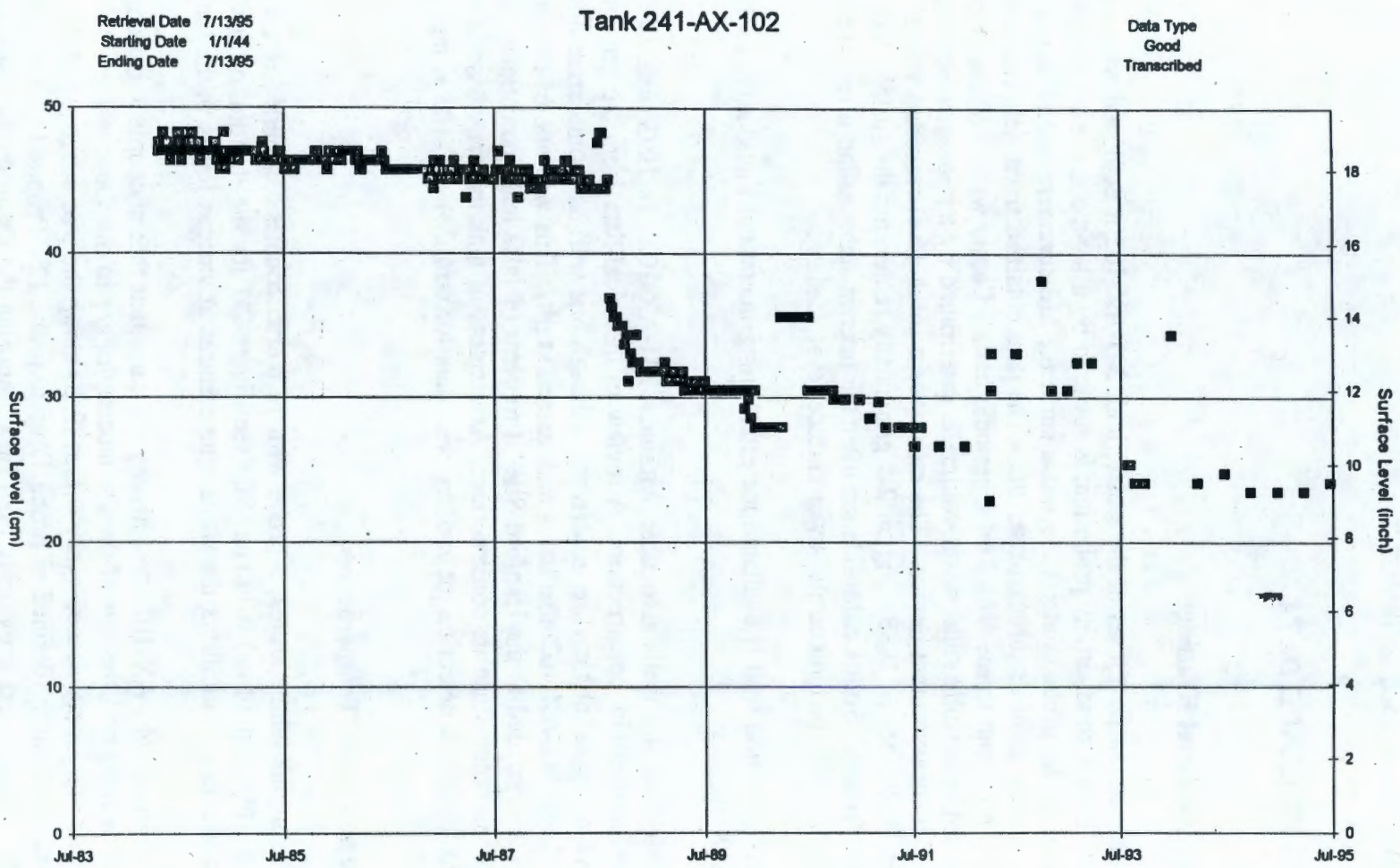


Figure 2-5. Tank 241-AX-102 Surface-Level Readings, 1983 to 1995.

1991 are sporadic. Relative to the other tanks in the AX Tank Farm, only a small amount of temperature data is available for this tank and readings have been recorded with thermocouples 1-6, 17, and 18.

The first available in-tank temperature measurement was recorded in July 1969. This data was 93 °C (200 °F) from the vapor space of the tank. From January 1990 to the present, the highest recorded in-tank temperature was 55 °C (131 °F) and the lowest temperature was 23 °C (73 °F). The median temperature of tank 241-AX-102 was 33 °C (91 °F). Based on the surface-level data and the thermocouple elevations (see above), these temperature data from 1990 to the present are most likely from the vapor space of the tank.

Tank 241-AX-102 is classified as a low heat-load tank and scheduled to have weekly in-tank temperature data taken.[1] In-tank temperature readings recorded since January 1975 are available in the documentation for the historical tank content estimate (HTCE) (Brevick et al. 1994a) along with graphs and thermocouple heights where available. Plots of available thermocouple readings for probes 1 and 2 are provided in Figure 2-6.

2.4.3 Tank Photographs

The interior of tank 241-AX-102 was last photographed on June 5, 1989. From these photographs, a "montage" was prepared and is presented in Figure 2-7. It is difficult to ascertain much about the waste surface from these photographs. The most notable objects in the photographs are the airlift circulators. There have been no changes in the tank which would affect the waste since these photographs were taken, and these pictures should represent the current tank contents.

[1]Normally, low heat-load tanks are scheduled for semiannual temperature monitoring in January and July. However, because tank 241-AX-102 is currently an Organic Watch List tank, the in-tank temperature is to be monitored weekly.

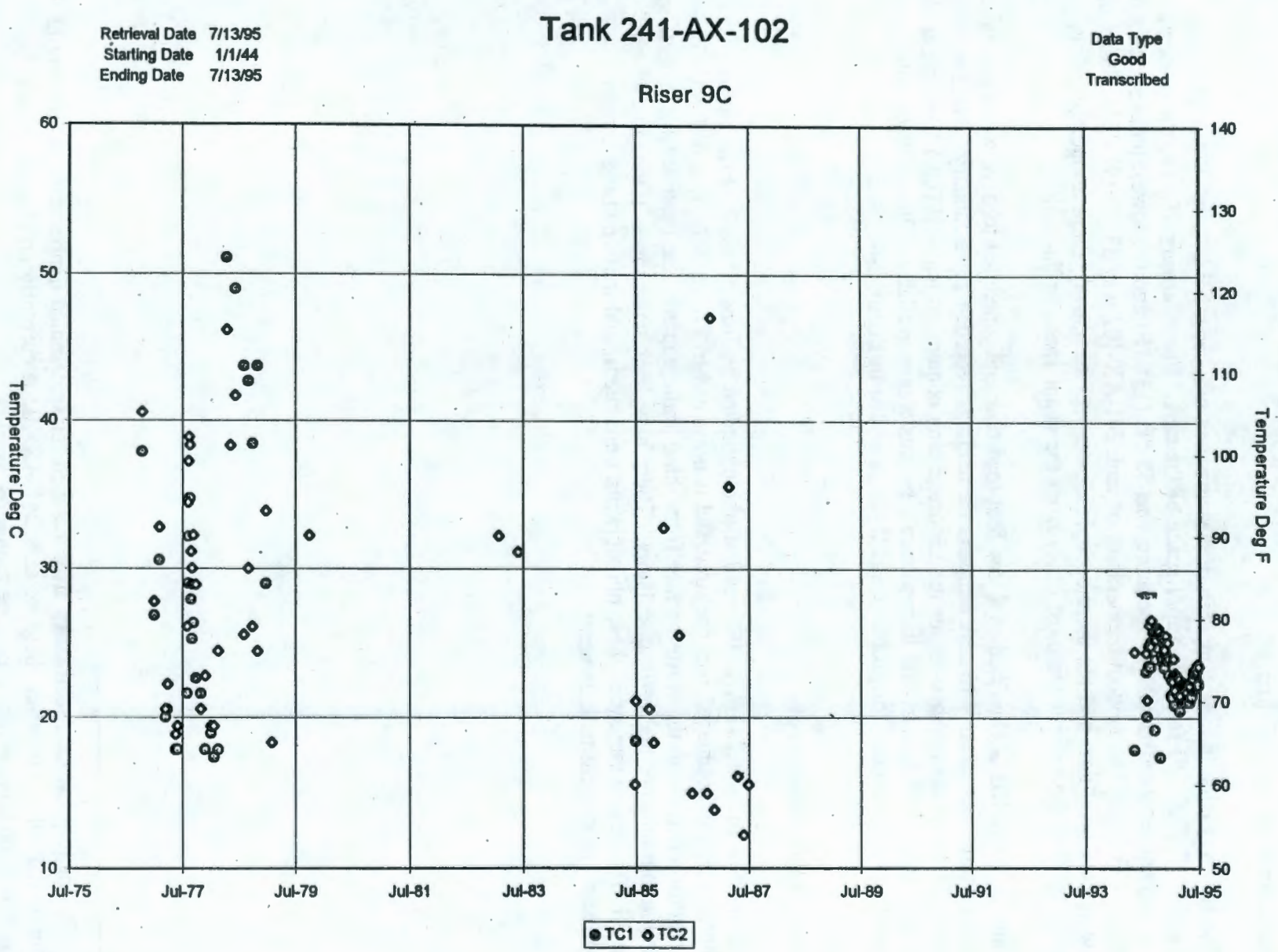


Figure 2-6. Tank 241-AX-102 Historical Temperature Data, 1975 to 1995.

9613456.0530

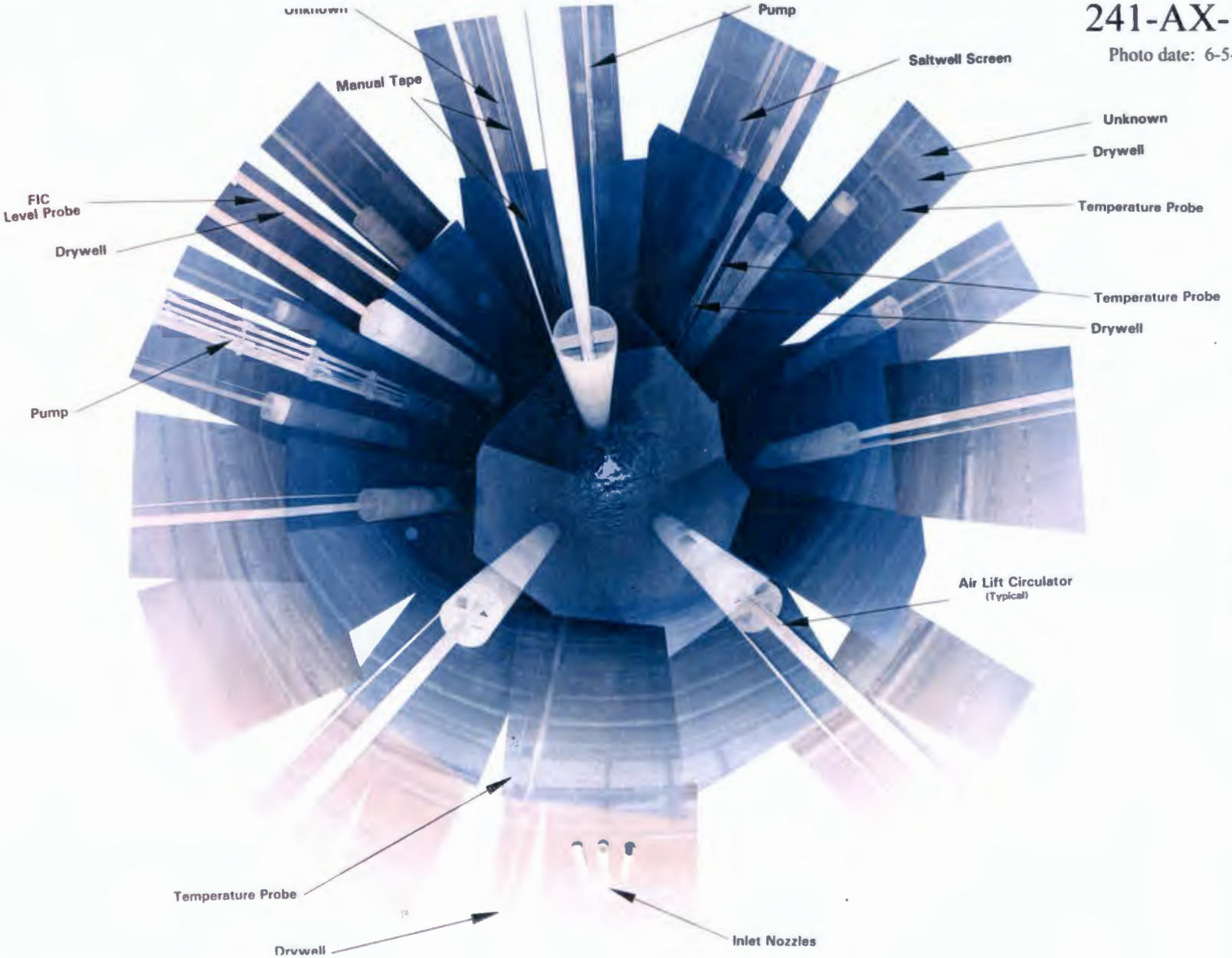


Figure 2-7. Photo Montage of the Tank 241-AX-102.

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3.0 TANK SAMPLING OVERVIEW

This section describes seven sampling and analysis events for tank 241-AX-102. These events are (1) auger sampling performed in 1995, (2) liquid grab sampling performed in 1988, (3) liquid grab sampling performed in February 1980, (4) liquid grab sampling performed in January 1980, (5) sludge sampling performed between February and July 1977, (6) sludge sampling performed in January 1977, and (7) sludge sampling performed in 1974.

The auger samples obtained from tank 241-AX-102 in 1995 were for a safety screening evaluation. The other six sampling events are useful from an historic perspective, and no attempt to determine current waste composition is made from these data. A further description of the sampling procedure may be found in WHC-SD-WM-TI-648, *Tank Characterization Reference Guide* (Simpson et al. 1994).

3.1 DESCRIPTION OF 1995 SAMPLING EVENT

In February 1995, two auger samples were obtained from tank 241-AX-102 (one sample from riser 3A and one from riser 9E). Each auger sample obtained from tank 241-AX-102 had 9 flutes--flute 1 defined as beginning at the auger shaft and flute 9 as ending at the auger tip. A description of the samples recovered is provided in Table 3-1. Rice (1995a) and Rice (1995b) are the documentation from which these data were obtained.

3.1.1 Sample Handling (1995)

The samples were extruded at the 222-S Laboratory and kept there for analysis. There was less material recovered from riser 3A than from riser 9E. Because of a "coiled up wire on the auger stem," it was "difficult to remove the sleeve" from the riser 9E auger sample (Rice 1995b).

Refer to Table 3-1 for information about extrusion dates and masses recovered for each sample.

There was little drainable liquid present in either sample. This lack of drainable liquid is not unexpected due to the nature of auger sampling. Most likely much of the liquid drained back into the tank as the auger was lifted from the waste surface to the riser flange.

3.1.2 Sample Analysis (1995)

The radionuclides are determined on fused samples of sludge. The fusions were performed in nickel crucibles with potassium hydroxide. The fused samples are dissolved in acid and analyzed by the methods identified in Appendix A of this TCR. The analytical methods used

Table 3-1. 1995 Sample Description Summary.

Riser number	Date extruded	Subsampled location	Sample mass (g)	Sample description
3A	N/A	Flute 1	N/A	N/A
	3/1/95	Flutes 2-9	1.97	Dark brown solids distributed as a thin layer or film. No drainable liquid.
9E	N/A	Flute 1	N/A	N/A
	3/1/95	Flutes 2-9	34.5	Dark brown solids distributed as a thin layer or film.
	3/1/95	N/A	2	Drainable liquid

N/A = Not applicable

for thermodynamic measurements, anions, hydroxide, cyanide, and carbon are also identified in Appendix A. The DSC and TGA analyses are performed on small (5-20 mg) quantities of the solid waste. Because of the small sample size, the reproducibility of the results are affected by the sample heterogeneity. Additional information on analytical methods can be obtained from WHC-SD-WM-TI-648, *Tank Characterization Reference Guide* (Simpson et al. 1994).

3.2 DESCRIPTION OF 1988 SAMPLING EVENT

In August 1988, a sample was taken from tank 241-AX-102 as part of a response to the indications that this tank was leaking. No information was given as to the location of the sample; however, Weiss (1988) (see Appendix B) identifies it as "a sample of the residual supernatant liquid in the tank." Most likely the sample was obtained using a bottle on a string. Aliquots of the sample were taken and submitted to Analytical Chemistry Services Laboratories for component analysis. Sample results are given in Weiss (1988) and include metal, anion, and radionuclide data.

3.3 DESCRIPTION OF FEBRUARY 1980 SAMPLING EVENT

In 1980, samples were taken from tank 241-AX-102. The exact date that the samples were taken is not clear, but results of analyses conducted on them were reported in February 1980 (Delegard 1980a) (see Appendix C). Little information is available about this sampling event, but it was most likely using a bottle on a string. Sample results include metal, anion, and physical data and are reported in Delegard (1980a).

3.4 DESCRIPTION OF JANUARY 1980 SAMPLING EVENT

In 1980, tank 241-AX-102 waste was sampled and analyzed. The exact date and reason for sampling are not clear but the data were most likely collected in support of evaporator operations and results were reported in January 1980 (Delegard 1980b) (see Appendix D). Little information is available about this sampling event, but it was most likely using a bottle on a string. Sample results include metal, anion, and physical data and are reported in Delegard (1980b).

3.5 DESCRIPTION OF FEBRUARY - JULY 1977 SAMPLING EVENT

In 1977, a series of six samples were taken from tank 241-AX-102. These samples were taken from the residual sludge that remained in tank 241-AX-102 following sluicing. Exact dates when the samples were obtained are not clear; however, the samples were obtained to provide data to estimate heat output from the sludge remaining in the tank. Little information is available about these sampling events, but most likely they were bottle on string samples. Sample results include primarily radionuclide data and are reported in Starr (1977) (see Appendix E).

3.6 DESCRIPTION OF JANUARY 1977 SAMPLING EVENT

In January 1977, two memos were issued identifying analytical data from sampling events of tank 241-AX-102 waste. These data are minimal and exact dates when the samples were drawn from the tank are not clear. However, the data are from samples taken as the tank was sluiced and transferred through the 244-AR Vault to B Plant during 1976. Only radionuclide data are presented as shown in Buckingham (1977a, 1977b) (see Appendix F).

3.7 DESCRIPTION OF AUGUST 1974 SAMPLING EVENT

In 1974, a sample was taken from the sludge of tank 241-AX-102. The exact date the sample was taken is not clear, but the data were reported in September 1974 (Horton 1974) (see Appendix G). This sample was most likely taken using a bottle on a string. Sample results include metal, radionuclide, and physical data.

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4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

4.1 OVERVIEW

The purpose of this section is to summarize the sampling and analytical results from the events described in Section 3.0. The 1995 sampling event was the most recent for tank 241-AX-102 and performed to evaluate the safety screening criteria of the Safety Screening DQO (Babad et al. 1995a) and the Organic Safety DQO (Babad et al. 1995b). This section describes analysis results based on the 45-day report (Rice 1995a) and the 90-day report (Rice 1995b). Table 4-1 summarizes where the data can be found in this document. The other (older) sample data (presented in Appendixes B through G) are included for historical reference purposes only. (No attempt was made to use the older data to characterize the current waste contained in tank 241-AX-102.)

The results based on the analysis of two auger samples taken in 1995 are reported in the following sections. Duplicate measurements were made for each determination for each subsample. The mean for most of the tests on the solids is based on the average of two auger sample results.

Because very little data are available, it is appropriate to provide a measure of uncertainty for the means. For this reason, 95% confidence intervals on the mean were computed.

The 95% confidence interval, lower limit (LL), on the mean for TGA is

$$\hat{\mu} - t_{(a-1)} * \sqrt{\hat{\sigma}^2_{\hat{\mu}}}$$

where $\hat{\mu}$ is the ordinary sample mean and $\hat{\sigma}^2_{\hat{\mu}}$ is the variance of the sample mean.

The 95% confidence interval, upper limit (UL), on the mean for DSC, TOC, total alpha, and cyanide is

$$\hat{\mu} + t_{(a-1)} * \sqrt{\hat{\sigma}^2_{\hat{\mu}}} \ .$$

The 95% confidence interval, upper and lower limits, on the mean for NO_2 is

$$\hat{\mu} \pm t_{(a-1)} * \sqrt{\hat{\sigma}^2_{\hat{\mu}}}$$

where $t_{(a-1)}$ is a quantile from "Student's t" distribution with a-1 degrees of freedom, and

$$\hat{\sigma}^2_{\hat{\mu}} = \frac{\hat{\sigma}^2(\text{spatial})}{a} + \frac{\hat{\sigma}^2(\text{analytical})}{an}$$

where:

a = number of auger samples

n = number of replicate measurements

$t_{(1)}$ = 6.314 for a one-sided 95% confidence interval

$t_{(1)}$ = 12.706 for a two-sided 95% confidence interval.

For TGA, DSC, TOC, and total alpha, there was a result and duplicate in augers 6 and 7. Therefore, the confidence limits for these analytes are based on spatial and analytical variability. For these analytes, estimates of the spatial and analytical variances were computed using restricted maximum likelihood methods (Harville 1977). The computations were made using the computer program S-Plus.[1] For NO_2 and cyanide, there were only a result and duplicate from auger 7. For these two analytes, the variance of the mean, used in the confidence limits, is based only on the analytical variability. In this case, the analytical variance is computed using the ordinary formula for a variance.

Table 4-1. Analytical Data Presentation Tables.

Analysis	Tabulated result location
Total alpha	Table 4-2
Thermogravimetric analysis	Table 4-8
Differential scanning calorimetry	Table 4-9
Total organic carbon	Table 4-3
Cyanide	Table 4-4
Hydroxide	Table 4-5
Nitrite	Table 4-6
Nitrate	Table 4-7

4.2 DATA PRESENTATION

Total alpha, TOC, cyanide, hydroxide, nitrite, and nitrate analyses were performed on the auger samples obtained from tank 241-AX-102 in 1995. The data resulting from these analyses are presented in Tables 4-2 through 4-7.

[1]S-Plus is a trademark of Stat Sci, a division of Math Soft Inc., Seattle, Washington.

No inventory or volume-based results are presented for tank 241-AX-102 because the waste density was not measured.

4.2.1 Total Alpha

Analyses for total alpha in 1995 were performed using procedure LA-508-101, *Alpha and Beta in Liquid Samples* (Fitzgerald 1994). These data were obtained to assist in determining how safe the waste contained in tank 241-AX-102 is relative to the 1 g/L ^{239}Pu criticality safety limit[1] (Vail 1994). The total alpha data resulting from these analyses are presented in Table 4-2 in which the UL to the 95% confidence interval on total alpha is given. The interpretation is that there is a 95% confidence level that the mean total alpha in the waste would be no >1.5 μCi/g.

4.2.2 TOC

Analyses for TOC were performed because some of the DSC results exceeded the Tank Characterization Plan (TCP) (Schreiber 1995) notification limit (481 J/g, dry basis) (see Section 4.3.2). TOC analyses of tank 241-AX-102 waste samples were conducted using procedure LA-342-100, *Determination of Carbon by Hot Persulfate Oxidation and Coulometric Detection* (Schroeder 1994). The TOC data results are presented in Table 4-3 in which the UL to the 95% confidence interval on TOC is given. This means that one would be 95% confident that the mean TOC concentration would be no $>79{,}000$ μg C/g on a wet basis.

4.2.3 Cyanide

Analyses for cyanide were performed because some of the DSC results exceeded the Tank Characterization Plan (Schreiber 1995) notification limit (481 J/g, dry basis) (see Section 4.3.2). Cyanide analyses of tank 241-AX-102 waste samples were conducted using procedure LA-695-102, *Determination of Cyanide by Microdistillation and Spectrophotometric Analysis* (Schroeder 1995). The cyanide data results are presented

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[1]Although the actual decision criteria listed in the DQO is 1 g/L, total alpha is measured in μCi/g. To convert between the notification units and laboratory reported units, it is assumed that all alpha decay originates from ^{239}Pu. Therefore, the only remaining information needed to convert units is the density of waste. The conversion then becomes:

$$\text{g/L} = \frac{\text{density}}{61.5}\ \mu\text{Ci/g},$$

as shown in Section 5.5.

Table 4-2. Total Alpha Results for Tank 241-AX-102 (Rice 1995a, Rice 1995b).[1,2]

Sample location	Run	Result (μCi/g)	Riser $\hat{\mu}$ (μCi/g)	Tank		
				$\hat{\mu}$ (μCi/g)	$\hat{\sigma}^2_{\hat{\mu}}$	UL (μCi/L)
Riser 3A: flutes 2-9	1	1.2	1.27	1.2	0.00189	1.5
	2	1.35				
Riser 9E: flutes 2-9	1	1.27	1.21			
	2	1.15				

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

UL = Upper limit

in Table 4-4 in which the UL to the 95% confidence interval on cyanide is given. This means that one would be 95% confident that the mean cyanide concentration would be no >29.7 μg CN/g.

4.2.4 Hydroxide

Analyses for hydroxide were performed because some of the DSC results exceeded the Tank Characterization Plan (Schreiber 1995) notification limit (481 J/g, dry basis) (see Section 4.3.2). Hydroxide analyses of tank 241-AX-102 waste samples were conducted using procedure LA-211-102, *Determination of Acid/Base/pH Using Metrohm 682 Titroprocessor* (Lachut 1993). The hydroxide data results are presented in Table 4-5. No UL 95% confidence interval on hydroxide is given because the result and duplicate were both equal values and less than the detection limit.

Table 4-3. Total Organic Carbon Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Sample location	Run	Result (μg C/g)		Riser average (μg C/g)		Tank					
						$\hat{\mu}$ (μg C/g)		$\hat{\sigma}^2_{\hat{\mu}}$		UL (μg C/g)	
		Wet basis	Dry basis[3]	Wet basis	Dry basis	Wet basis	Dry basis	Wet basis	Dry basis	Wet basis	Dry basis
Riser 3A: flutes 2-9	1	61,200	88,100	57,300	82,400	56,600	81,400	12,600,000	26,100,000	79,000	114,000
	2	53,400	76,800								
Riser 9E: flutes 2-9	1	63,500	91,400	55,800	80,300						
	2	48,100	69,200								

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]Conversion from wet basis to dry basis requires moisture data (refer to Section 4.3.1 for determination of tank 241-AX-102 as 30.5 wt% H_2O) where:

$$\text{wet basis} \div (1 - (\text{wt\% } H_2O)/100) = \text{dry basis},$$

and for tank 241-AX-102 from the 1995 sample analysis:

$$\text{wet basis}/0.695 = \text{dry basis}.$$

UL = Upper limit

9613456.0535

Table 4-4. Cyanide Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Subsample location[3]	Run	Result (μg/g)	$\hat{\mu}$ (μg/g)	$\hat{\sigma}^2_{\mu}$	UL (μg/g)
Riser 9E: flutes 2-9	1	26.8	26.3	0.303	29.7[4]
	2	25.7			

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]Cyanide analyses were not conducted on material from riser 3A because insufficient sample remained after alpha, thermogravimetric analysis, differential scanning calorimetry, and total organic carbon analyses were completed.

[4]95% upper confidence interval limit, $t_{(1)} = 6.314$.

UL = Upper limit

4.2.5 Anions - Nitrite and Nitrate

Analyses for nitrite and nitrate anions were performed because some of the DSC results exceeded the Tank Characterization Plan (Schreiber 1995) notification limit (481 J/g, dry basis) (see Section 4.3.2). Anion analyses of tank 241-AX-102 waste samples were conducted using procedure LA-533-105, *Anion Analysis on DIONEX Model 4000i and 4500i* (Frye 1994a).[1] The nitrite and nitrate anion concentration data results are presented in Tables 4-6 and 4-7, respectively. In Table 4-6, a UL and an LL to the 95% confidence interval on nitrite are given. The interpretation is that one would be 95% confident that the mean nitrite concentration in the waste would be no >48,300 μg/g and no <33,100 μg/g. In Table 4-7, no UL or LL of the 95% confidence interval on nitrate is given because the result and duplicate were the same.

[1]The DIONEX (a trademark of DIONEX Corporation) Model 4000i was used in this case.

Table 4-5. Hydroxide Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Subsample location[3]	Run	Result (μg/g)	$\hat{\mu}$ (μg/g)	$\hat{\sigma}^2_{\hat{\mu}}$	UL (μg/g)
Riser 9E: flutes 2-9	1	<1,660[4]	N/A	N/A	N/A
	2	<1,660[4]			

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]Hydroxide analyses were not conducted on material from riser 3A because insufficient sample remained after alpha, thermogravimetric analysis, differential scanning calorimetry, and total organic carbon analyses were completed.

[4]The detection limit for hydroxide is 1,660 μg OH^-/g.

N/A = Not applicable
UL = Upper limit

4.3 PHYSICAL MEASUREMENTS

The only physical measurements of the samples obtained in 1995 were DSC and TGA. The TGA provides an estimate of the chemical property, weight percent water, but also is used to interpret and understand the DSC results. The results of both tests are summarized in the following sections.

4.3.1 TGA

Weight percent water by TGA was performed under a nitrogen atmosphere using procedure LA-560-112, *Determination of Weight Loss as Percent Water by Thermogravimetric Analysis (TGA) - Mettler[1] TG 50* (Frye 1994b). The results of the TGA analyses that were performed are summarized in Table 4-8.

The first transition in each sample began at the lower temperature limit of the analysis (30 °C [86 °F]) and was complete at approximately 120 °C (248 °F). In this region, the observed decreases in weight are mainly due to the loss of bulk and interstitial water in the samples. The second transition occurred between 200 and 490 °C (392 and 914 °F). The phenomena demonstrated in this region could be attributed to the loss of covalently bound water molecules or the dehydration of compounds such as aluminum hydroxide. The weight loss represents the percent water in the specimen. Examples of typical TGA scans for the samples from risers 3A and 9E are provided in Appendix H.

Table 4-6. Nitrite Anion Concentration Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Subsample location[3]	Run	Result (μg/g)	$\hat{\mu}$ (μg/g)	$\hat{\sigma}^2_{\hat{\mu}}$	UL[4]	LL[4]
Riser 9E: flutes 2-9	1	40,100	40,700	360,000	48,300	33,100
	2	41,300				

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]Nitrite anion analyses were not conducted on material from riser 3A because insufficient sample remained after alpha, thermogravimetric analysis, differential scanning calorimetry, and total organic carbon analyses were completed.

[4]95% two-tailed confidence interval limits, $t_{(1)} = 12.706$.

LL = Lower limit
UL = Upper limit

In Table 4-8, the LL to the 95% confidence interval on the percent water (TGA) for tank 241-AX-102 is given. If the LL is greater than 17%, then the null hypothesis that the percent water is $\leq$17% is rejected. Because the LL is equal to 19.7%, there is a 95% confidence level that the mean water concentration is >17%.

4.3.2 DSC

Analyses for DSC were performed under a nitrogen atmosphere using procedure LA-514-113, *Differential Scanning Calorimetry (DSC)* (Frye 1994c). The DSC data results are presented in Table 4-9. Examples of the DSC scans for the samples from risers 3A and 9E are provided in Appendix H.

Endothermic and exothermic behavior was noted in all of the DSC runs conducted of the waste samples for tank 241-AX-102. Endotherms are represented in Table 4-9 by positive enthalpy values and exotherms are denoted by negative enthalpy values in the table. In Table 4-9, the UL to the 95% confidence interval on exotherms from DSC is given. For example, there is a 95% confidence level that the mean exothermic data on a wet basis would be <465.9 J of energy produced/g of wet waste.

Table 4-7. Nitrate Anion Concentration Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Subsample location[3]	Run	Result (μg/g)	$\hat{\mu}$ (μg/g)	$\hat{\sigma}^2_{\hat{\mu}}$	UL (μg/g)	LL (μg/g)
Riser 9E: flutes 2-9	1	172,000	172,000	0	N/A	N/A
	2	172,000				

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]Nitrate anion analyses were not conducted on material from riser 3A because insufficient sample remained after alpha, thermogravimetric analysis, differential scanning calorimetry, and total organic carbon analyses were completed.

LL = Lower limit
N/A = Not applicable
UL = Upper limit

Table 4-8. Thermogravimetric Analysis Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1,2]

Subsample location	Run	Transition 1 and 2 wt% loss							
		1 (30-120 °C)	Riser mean 1 wt% loss	Tank			2 (200-490 °C)	Riser mean 2 wt% loss	Total weight loss (wt%)
				$\hat{\mu}$	$\hat{\sigma}^2_{\hat{\mu}}$	LL			
Riser 3A: flutes 2-9	1	29.6	28.8	30.5	2.9	19.7	17.0	18.8	47.6
	2	28.0					20.5		
Riser 9E: flutes 2-9	1	31.1	32.2				20.1	20.3	52.5
	2	33.3					20.5		

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

LL = Lower limit

Table 4-9. Differential Scanning Calorimetry Energetic Results for Tank 241-AX-102 (Rice 1995a, 1995b).[1, 2]

Subsample location	Run	Peak °C	ΔH[3] J/g (wet basis)	Peak °C	ΔH[3] J/g		$\hat{\mu}$ (J/g)		$\hat{\sigma}^2_{\hat{\mu}}$		UL (J/g)	
					Wet basis	Dry basis[4]	Wet basis	Dry basis	Wet basis	Dry basis	Wet basis	Dry basis
Riser 3A: flutes 2-9	1	131.5	851.4	390.6	-352.1	-506.6	-328.1	-472.1	476.3	984.4	-465.9	-670.2
	2	134.2	828.3	391.0	-347.8	-500.4						
Riser 9E: flutes 2-9	1	128.2	794.7	390.5	-282.3	-406.2						
	2	119.1	991.2	390.3	-330.3	-475.3						

[1]Rice, A. D., 1995a, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

[2]Rice, A. D., 1995b, *90-Day Final Report for Tank 241-AX-102, Auger Samples 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

[3]For enthalpy values given, a positive value signifies endotherm and a negative value signifies exotherm.

[4]Conversion from wet basis to dry basis requires moisture data (refer to Section 4.3.1 for determination of tank 241-AX-102 as 30.5 wt% H_2O) where:

wet basis ÷ (1 - (wt% H_2O)/100) = dry basis,

and for tank 241-AX-102 from the 1995 sample analysis:

wet basis/0.695 = dry basis.

UL = Upper limit

9613456.0538

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

The following topics will be discussed in this section:

- The assessment of the sampling and analytical results
- The tank waste profile
- Comparison of historical and analytical information
- The evaluation of program requirements.

Furthermore, sampling and analysis factors that may impact interpretation of the data are evaluated. These factors are used to assess the overall quality and consistency of the data and to identify any limitations in the use of the data.

5.1.1 Field Observations

The most notable observation regarding the auger samples obtained from tank 241-AX-102 during the 1995 sampling event was the "low recovery." Only a thin coating was obtained on both the auger from riser 3A and the auger from riser 9E. This "low recovery" led to only a limited number of analyses on the sample obtained from riser 3A. A greater quantity of material was available from the auger taken from riser 9E and this comprised the bulk of the secondary analyses for this tank. Ultimately, the effect of "low recovery" is to make it difficult to draw conclusions about the relationship between the analytical results and the bulk tank contents.

During extrusion of the sample taken from riser 9E, a "difficult time [was had in] removing the sleeve from the auger. [A] coiled up wire on the auger stem made it difficult to remove the sleeve" (Rice 1995b). This observation was made and is noted here, but it is difficult to ascertain any effects it may have had.

5.1.2 Quality Control Assessment of Analytical Data

An attempt is usually made to quantify the different sources of variability during the chemical analysis of a sample. When these variabilities are summarized, they give a strong indication of data reliability. If one or more of the variability estimates is outside acceptable limits, the precision of the concentration estimate is questioned. Possible sources of variability include poor instrument calibration, matrix interferences, sample contamination, and analytical variability. Systematic variability is estimated from analyses of reference

standards or spike[1] recoveries. Analytical variability is estimated from the differences between the primary and duplicate samples.

Reference standards are used to estimate the systematic variability of the analytical method, and they are evaluated prior to and concurrent with sample analysis. Standards contain the analytes of interest or carriers at known concentrations. Standard solutions may or may not be independent of the standard used for calibration. The criterion for standard recovery is 100 $\pm$ 10 percent. If a standard is above or below the criterion, the analytical result may be biased high or low, respectively. Only one standard was found to not be acceptable (i.e., 100 $\pm$ 10 percent). This one exception was the cyanide result from the auger sample of riser 9E. A potential biasing of the cyanide result from riser 9E is signified by a 121.3% standard recovery. However, this high standard recovery is of little consequence as the analytical results were very low ($\hat{\mu}_{CN^-}$ = 26.3 μg/g, with UL_{CN^-} = 29.7 μg/g, and the cyanide limit = 39,000 μg/g). The results were approximately three orders of magnitude below the limit.

Evaluation of analytical preparation blanks for the sampling event of 1995 applies to total alpha activity, TOC analyses, cyanide, and anion analyses. Contamination was not indicated to be a problem because all preparation blanks were below the respective detection limits.

Duplicate analyses were performed on each of the auger samples to provide an indication of the reproducibility for these analyses and also an indication of the sample homogeneity. Precision requirements are evaluated by the relative percent difference (RPD) between the primary and duplicate samples. In general, the analytical results agree well with most[2] of the analytes within the 10% RPD criterion specified in the Safety Screening DQO (Babad et al. 1995a). The implication is that for this set of data, the analytical precision qualifies the data as being reliable.

Extremely large systematic variability is concluded based on the poor spike recovery results. (In virtually all cases, the spike recoveries were outside the TCP recommended range of 80 - 120%). The quality control data for the 1995 sample analyses event are summarized in Table 5-1.

---

[1]Spikes do not apply to DSC and TGA methods.

[2]The major exception was the TOC analyses, with the RPD of the riser 9E data being 27.6. The RPD of the riser 3A data was 13.6, which was also above the 10% limit but not as severe. Further compounding this issue are the problems that were noted during TOC analyses--that there were "sample incompatibilities with this [the] method." This leads one to question the TOC method used on this particular waste type.

Table 5-1. Quality Control Summary for Tank 241-AX-102.

Riser/ subsample	Analyte	Relative percent difference %[1]	Standard %[2]	Spike recovery %[3]
3A/ flutes 2-9	% water (TGA)	5.42	97.26	NA
	DSC	1.23	98.42	NA
	Alpha	11.8	93.89	72.20
	TOC	13.6[4]	90.33	196.0[4]
9E/ flutes 2-9	% water (TGA)	6.77	97.26	NA
	DSC	15.7	98.42	NA
	Alpha	9.92	93.89	67.00
	TOC	27.6[4]	90.33	NA[4]
	Cyanide	4.19	121.3	103.0
	OH^-	NA[5]	100.2	NA[5]
	Nitrite	2.95	99.62	87.80
	Nitrate	0	100.9	48.40

[1]10% limit.
[2]Range = 90-110%.
[3]Range = 80-120%.
[4]TOC method incompatibilities with samples.
[5]OH^- below detection limit.

DSC = Differential scanning calorimetry
NA = Not available
TGA = Thermogravimetric analysis
TOC = Total organic carbon

5.1.3 Data Consistency Checks

The ability to assess the overall consistency or trends of the data between auger samples is limited for the tank 241-AX-102 sampling and analysis events of 1995 because of the limited quantity of sample material recovered. If a greater amount of waste material had been recovered in auger samples 95-AUG-006 and 95-AUG-007 and/or additional samples had been successfully obtained, further analyses would have been possible to subsequently check the consistency of these data. The comparison of results from different sampling events is described in Section 5.4 of this report and provides added indication of the data consistency for this tank. Checks can be made among individual analytes when the same aliquot of

sample is analyzed by two comparable methods. Two of these checks are made and described for the data of the 1995 analysis event in the following sections.

5.1.3.1 Comparison of DSC and TOC Analyses. This evaluation can be used to check the accounting of fuel (as organic carbon) and energy production due to an exothermic chemical reaction. The DSC data for the 1995 sampling event are described in Section 4.3.2. The TOC data for the 1995 sampling event are described in Section 4.2.2. Therefore, using these data and the TGA results from Section 4.3.1 the equivalent dry basis values are shown in Table 5-2.

If the exotherms detected by the DSC analyses were produced exclusively from the oxidation of the organic carbon present in the samples, then knowing the form of the molecule(s) in which the organic carbon is/are contained allows for the calculation of the exothermic heat of reaction for that compound(s). Organic compounds known to have been transferred to Hanford Site wastes are presented in Table 5-3. The resultant heats of reaction (assuming reaction to completion) for each of these compounds is presented in Table 5-3.

Table 5-2. Combination of Total Organic Carbon, Differential Scanning Calorimetry, and Thermogravimetric Analysis Data.

Analyte	Direct analytical results	Equivalent values[1]
TOC	56,600 μg C/g (wet basis)	81,400 μg C/g (dry basis)
DSC	328.1 J/g (wet basis)	472.1 J/g (dry basis)[2]
TGA	30.5 wt% H_2O	NA

[1]To convert from a "wet basis" to a "dry basis" use:

wet basis ÷ (1 - (wt% H_2O)/100) = dry basis

Therefore for TOC:

[56,600 μgC (wet basis)/g] ÷ (1 - (30.5 wt% H_2O)/100) = 81,400 μgC/g (dry basis)

[2]Even though the mean DSC value is below the 481 J/g (dry basis) action limit specified in the DQOs (Babad, H., J. W. Hunt, and K. S. Redus, 1995a, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 1, Westinghouse Hanford Company, Richland, Washington; Babad, H., S. M. Blacker, and K. S. Redus, 1995b, *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue*, WHC-SD-WM-DQO-006, Rev. 1, Westinghouse Hanford Company, Richland, Washington), the majority of the data were above 481. Refer to Table 4-9.

DSC = Differential scanning calorimetry
TGA = Thermogravimetric analysis
TOC = Total organic carbon
NA = Not available

Table 5-3. Theoretical Energy from Oxidation of Organic Carbon by $NaNO_3$.

Molecular form containing organic carbon[1]				Energy that would be produced in tank 241-AX-102 from 56,600 μg C/g of waste (J/g) (wet basis)[4]
Fuel	MW (g/mole)	C atoms/ molecule	Enthalpy (J/g)[2,3]	
Na_3 HEDTA	340	10	-11,000	-1,760
Na_4 EDTA	380	10	-8,800	-1,580
Na_3 Citrate	258	6	-6,840	-1,390
$NaCH_3C00$	82	2	-7,940	-1,540
TBP	266	12	-17,200	-1,800
Na DBP	200	8	-21,100	-2,490
$Na_2NiFE(CN)_6$	N/A	N/A	-9,510	N/A

[1]Theoretical data from PNL-8557, *Calculation of Reaction Energies and Adiabatic Temperatures for Waste Tank Reactions*, 1993, L. L. Burger, Pacific Northwest Laboratory, Richland, Washington.

[2]Negative enthalpy values signify exotherms.

[3]Values are energy per gram of fuel.

[4]Values are energy per gram of "wet" waste and calculated assuming all carbon detected is in the indicated fuel form. Therefore, for the case of Na_3 HEDTA:

$$\frac{56{,}600\ \mu gC}{g\ \text{"wet" waste}} \cdot \frac{-11{,}000\ J}{g\ \text{sodium HEDTA}} \cdot \frac{1\ \text{mole C}}{12\ g\ C} \cdot \frac{1\ \text{mole sodium HEDTA}}{10\ \text{mole C}} \cdot \frac{340\ g\ \text{sodium HEDTA}}{1\ \text{mole sodium HEDTA}} \cdot \frac{1\ g}{1\times10^6\ \mu g} =$$

-1,760 J/g "wet" waste if fuel from sodium HEDTA.

DBP = Dibutylphosphate
EDTA = Ethylenediametetraacetic acid
HEDTA = N-(hydroxyethyl)-ethylenediaminetriacetic acid
N/A = Not applicable
TBP = Tributylphosphate

Most compounds that were originally transferred to storage in the waste tanks at the Hanford Site no longer exist in their original form. Further, most chemical reactions will not proceed to completion. Therefore, some fraction of the theoretical energy production from a fuel is typically observed in experimental settings. Based on experimentation performed by R. D. Scheele, some of these reactions have been measured in the laboratory and it was found that from 10 to 50% of the theoretical energy is the most energy that has been observed.

Using this 50% efficiency and the data from Table 5-3, it is not possible to discern if the DSC results are from TOC. Furthermore, it is possible that the DSC results from the 1995 analysis event are produced from combustion of $NaNO_3$ with a less energetic form of TOC than shown in Table 5-3, or $NaNO_3$ is not the only oxidant.

5.1.3.2 Comparison of TGA Data and Extrusion Photographs. When auger samples 95-AUG-006 and 95-AUG-007 were extruded in the hot cell, color photographs were obtained. These photographs revealed small pools of liquid forming on the trays below the extruded augers. The TGA results in Table 4-8 (30.5% H_2O) are believable based on the evidence in these photographs.

5.2 COMPARISON OF RESULTS FROM DIFFERENT SAMPLING EVENTS

The analyses performed for the seven sampling and analyses events identified in this report were extremely limited, and the overlap between analyses is insufficient to make a comparison. However, a comparison of the results from these analyses to the analytes predicted for the transfer history of tank 241-AX-102 is provided in Section 5.4.

5.3 TANK WASTE PROFILE

With the limited number of analyses performed for the 1995 analysis project, it is not possible to gain an understanding of the waste profile for tank 241-AX-102.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER HISTORY INFORMATION

There are several observations and comparisons that can be made with the available historical model results, past samples, and contemporary analytical data. Because tank 241-AX-102 was emptied of most of the high-level waste that was originally routed to it, and subsequent transfers indicate the likely presence of organic-bearing waste and evaporator slurries, notable TOC and/or high exotherms are expected. A comparison of the analytes from the HTCE reported in Section 2.3.2 with the 1995 results is presented in Table 5-4.

5.5 EVALUATION OF PROGRAM REQUIREMENTS

Tank 241-AX-102 is currently classified as an Organic Watch List Tank, and the 1995 auger sampling and analysis event was performed to meet the requirements of the Safety Screening DQO (Babad et al. 1995a) coupled with the Organic Safety DQO (Babad et al. 1995b). Therefore, only the safety program requirements can be assessed. No requirements were identified for evaluating operational, environmental, and/or process development program needs. Only the 1995 auger sampling event will be discussed in this section as data generated before 1989 may not be considered valid for some applications under the constraints of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1994).

Table 5-4. Comparison of Analytes.

Analyte	HTCE	1995 auger sample data
H_2O (%)	45.9	30.5 (from Table 4-8)
TOC (μg/g)	3,100	56,550 (from Table 4-3)
NO_3^- (μg/g)	173,000[1]	172,000 (from Table 4-7)
NO_2^- (μg/g)	36,300[2]	40,700 (from Table 4-6)
OH^- (μg/g)	93,000[3]	<1,660 (from Table 4-5)
Density (g/mL)	1.52	Not reported

[1](4.25 mol NO_3^-/L)(1/1.52 g/mL)(1L/1,000 mL)(62 g/mol NO_3^-)(1,000,000 μg/g) = 173,000 μg NO_3^-/g.
[2](1.20 mol NO_2^-/L)(1/1.52 g/mL)(1L/1,000 mL)(46 g/mol NO_2^-)(1,000,000 μg/g) = 36,300 μg NO_2^-/g.
[3](8.32 mol OH^-/L)(1/1.52 g/mL)(1L/1,000 mL)(17 g/mol OH^-)(1,000,000 μg/g) = 93,000 μg OH^-/g.

HTCE = Historical tank content estimate
TOC = Total organic carbon

The Safety Screening DQO (Babad et al. 1995a) establishes decision criteria or notification limits for concentrations of analytes of concern. The decision criteria are used to determine if a tank has been appropriately categorized concerning safety issues, or if further investigation into the tank's safety is warranted. If results from the primary analyses exceed the decision criteria, the tank is classified as "not safe" and further analyses are conducted to ensure the safety of the tank. The primary analytical requirements identified in the Safety Screening DQO (Babad et al. 1995a) are fuel energy content, percent moisture content, total alpha activity, and flammable gas concentration. Table 5-5 lists the applicable analytes, the notification limits for each, and the pertinent 1995 auger sampling analytical results.

The waste fuel energy value of tank 241-AX-102 was determined using DSC analysis of the sampled material. For both auger samples, results were obtained as greater than the DQO limit of 481 J/g (dry weight basis). This indicates that some fuel content is present in the waste material of tank 241-AX-102.

Large amounts of moisture reduce the potential for propagating exothermic reactions in the wastes, and it has been found that sufficient moisture content can ensure exothermic reactions will not occur regardless of the fuel energy concentration. It is specified in the Safety Screening DQO (Babad et al. 1995a) that a percent moisture content above 17 wt% is necessary for safe storage. For both auger samples, results obtained were within the specifications of the DQO. In fact, statistical analysis of the data indicated 95% confidence

that the mean water concentration is >19.7%. This indicates that sufficient moisture is present in the waste matrix of tank 241-AX-102 to reduce the potential for propagating any exothermic reaction(s).

Because the DSC notification limit was exceeded for the tank 241-AX-102 auger samples, TOC secondary analysis was performed to identify the source of the fuel. Both samples produced results that exceeded the TOC notification limit of 5 wt% (dry weight basis) suggesting that tank 241-AX-102 may contain organic species. This hypothesis is consistent with the tank fill history, which indicates that complexants and complex concentrate wastes were added to the tank. Further, the limited cyanide concentration detected (26 μg CN/g) was far below the 39,000 μg/g limit, indicating little evidence that ferrocyanide is present in the waste.

Another factor in assessing the safety of Hanford Site tanks is the heat generation and temperature of the wastes stored in them. Heat is generated in the tanks primarily from radioactive decay. For the tank 241-AX-102 auger samples, analyses for heat-generating radionuclides such as ^{137}Cs and ^{90}Sr were not performed. However, the recorded tank temperature over the past several years has been relatively stable, at or below 25 °C (77 °F). Because these data may be from thermocouple(s) located in the vapor space of the tank, a more detailed analysis would be necessary to fully understand the heat load of the waste in the tank; however, because a dramatic increase has not been observed in the temperature data collected, classification of this tank as a low heat-load tank is supported by the observed data from in-tank temperature monitoring.

The potential for criticality is assessed from either total alpha analysis or plutonium analysis. The safety screening criterion for criticality is 1 g/L. With 95% confidence, the mean total alpha activity for the auger samples obtained during the 1995 sampling event was <1.5 μCi/g. In order to compare these values, a density of the waste is needed.[1] However, even if a density is approximated (1.5 g/mL), the analytical data are well within the 1 g/L limit as shown in Table 5-5.

The flammability of the gas in the headspace of a tank is another safety screening consideration. Analysis of the tank headspace is not yet complete for tank 241-AX-102 as it was sampled on June 27, 1995.

---

[1]Although the actual decision criteria listed in the DQO is 1 g/L, total alpha is measured in μCi/g. To convert between the notification units and laboratory reported units, it is assumed that all alpha decay originates from ^{239}Pu. Therefore, the only remaining information needed is the density of waste (units of g/mL). The conversion then becomes:

$$(1.5\ \mu\text{Ci/g})\left(\frac{1}{61.5}\right)(1.5\ \text{g/mL}) = 0.04\ \text{g/L}$$

Table 5-5. Safety Screening Data Quality Objectives Decision Variables and Criteria for Tank 241-AX-102.

Safety issue	Primary decision variable	TCP criteria threshold	Observed values	
			Mean	95% confidence analytical
Ferrocyanide/ organic	Total fuel content	481 J/g (dry basis)	472.1	<596 J/g, dry
Organic	Percent moisture	17 wt% H_2O	30.5	>19.7 wt% H_2O
Criticality	Total alpha analysis	1 g/L	0.03	<0.04 g/L
Flammable gas	Flammable gas concentration	Not applicable	Not yet available	
Organic	Total organic carbon	5 wt% (dry basis)	8.1 wt%[1] TOC, dry	<11.4 wt%[1] TOC, dry

[1]wt% = μg/g ÷ 10,000

TCP = Tank Characterization Plan
TOC = Total organic carbon

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6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-AX-102 was sampled and analyzed in 1995. Limited samples and data from different risers and different waste depths make it difficult to determine the variability of the waste composition in the tank. However, the 1995 sampling and analysis event did meet the requirements of the DQOs (Babad et al. 1995a, 1995b).

Based on the data presented in Table 5-5, tank 241-AX-102 is being managed in a conditionally safe manner. This means that even though there is a high concentration of TOC, and exothermic behavior is exhibited, sufficient moisture exists to reduce the potential propagation of an exothermic reaction. Because TOC is present in concentrations above the notification limit of the DQO (>5 wt% dry basis TOC), tank 241-AX-102 should remain on the Organic Tank Watch List.

In the future, consideration may be given to resampling tank 241-AX-102 for process development. This resampling should only be done when (and if) more precise criteria can be identified to justify this action; until then, the waste should be maintained in the current conditionally safe configuration.

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APPENDIX A

ANALYTICAL PROCEDURES

SINGLE-SHELL TANK 241-AX-102

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APPENDIX A

ANALYTICAL PROCEDURES
SINGLE-SHELL TANK 241-AX-102

Table A-1 contains the procedure numbers for each of the analytical methods performed on the tank 241-AX-102 waste during the 1995 auger sampling and analysis event. All analyses were performed at the Westinghouse Hanford Company 222-S Laboratory.

Table A-1. Analytical Methods.

Analyte	Digestion	Procedure number
Thermogravimetric analysis	N/A	LA-560-112[1]
Differential scanning calorimetry	N/A	LA-514-113[2]
Total alpha	Fusion	LA-508-101[3]
Total organic carbon	N/A	LA-342-100[4]
Hydroxide	N/A	LA-211-102[5]
Cyanide	N/A	LA-695-102[6]
Ion chromotography	Water	LA-533-105[7]

[1]Frye, J. M., 1994b, *Determination of Weight Loss as Percent Water by Thermogravimetric Analysis (TGA) - Mettler[1] TG 50*, LA-560-112, Rev. A-2, Westinghouse Hanford Company, Richland, Washington.

[2]Frye, J. M., 1994c, *Differential Scanning Calorimetry (DSC)*, LA-514-113, Rev. B-1, Westinghouse Hanford Company, Richland, Washington.

[3]Fitzgerald, S. L., 1994, *Alpha and Beta in Liquid Samples*, LA-508-101, Rev. D-2, Westinghouse Hanford Company, Richland, Washington.

[4]Schroeder, R. W., 1994, *Determination of Carbon by Hot Persulfate Oxidation and Coulometric Detection*, LA-342-100, Rev. A-0, Westinghouse Hanford Company, Richland, Washington.

[5]Lachut, J. S., 1993, *Determination of Acid/Base/pH Using Metrohm 682 Titroprocessor*, LA-211-102, Rev. B-1, Westinghouse Hanford Company, Richland, Washington.

[6]Schroeder, R. W., 1995, *Determination of Cyanide by Microdistillation and Spectrophotometric Analysis*, LA-695-102, Rev. C-0, Westinghouse Hanford Company, Richland, Washington.

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N/A = Not applicable

APPENDIX B

ANALYSIS OF TANK 241-AX-102 SAMPLE

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AX-102

Westinghouse Hanford Company

Internal Memo

From: Process Chemistry Laboratories
Phone: 3-1972 MO-037/200W T6-50
Date: November 14, 1988
Subject: ANALYSIS OF TANK 241-AX-102 SAMPLE

12712-PCL88-018

xc JWDC LWB DGB VCB RCR ATA

To: J. A. Eacker R1-51

cc: J. E. Bramson T1-30
D. R. Bratzel DRB T6-50
K. G. Carothers R1-51
A. J. DiLiberto R2-05
G. L. Dunford R1-51
RLW File/LB

During August 1988, there were indications that the single-shell tank (SST) ~~241-SX-104~~ AX-102 was no longer sound. As part of the response to these indications, a sample of the residual supernatant liquid in the tank was taken and shipped to 222S for analysis. This report summarizes the results of the analysis.

The sample was received as a dark, coffee-brown liquid with no indication of solids present. No sample preparation was needed, an aliquot of the solution was transferred to the Analytical Chemistry Services Laboratories (ACSL) for component analysis.

A representative composition for the material recovered from tank 241-AX-102 is shown in Table 1. The error range for these values is approximately ± 10%. Values prefaced with the less than symbol (<) were not quantifiable and the value reported is the analytical procedure limit of confirmation value.

The liquid recovered from tank 241-AX-102 is consistent with an expected composition for supernate from a salt cake waste tank. The major radionuclides present are cesium-137 and strontium-90. Based on available results, the material is complexed (>10 g/L TOC) and transuranic (>100 nCi/gm Pu + Am) waste.

R. L. Weiss
Chemist

cse

Attachment (1)

RECEIVED
G.L. DUNFORD
NOV 18 '88
ACTION
COPIES
ROUTE

Hanford Operations and Engineering Contractor for the US Department of Energy

TABLE 1
COMPOSITION OF 241-AX-102 SAMPLE

Component	Value
pH	11.3
NO_3	3.7 M
NO_2	1.4 M
CO_3	0.98 M
PO_4	<.056 M
NH_4	0.028 M
TOC	36.8 g/l
Ag	<0.004 M
Al	0.006 M
B	0.002 M
Ba	<0.0001 M
Bi	<0.0004 M
Ca	0.014 M
Ce	0.0009 M
Cd	<0.0004 M
Co	<0.0006 M
Cr	0.004 M
Cu	0.0006 M
Fe	0.033 M
K	0.002 M
La	0.0004 M
Li	<0.002 M
Mg	0.0005 M
Mn	0.011 M
Mo	0.0004 M
Na	7.32 M
Nd	0.001 M
Ni	0.009 M
Pb	0.002 M
Pd	0.0006M
P	0.023 M
Si	0.0009 M
Sn	<0.001 M
Sr	<0.0003 M
Ta	<0.0003 M
Ti	0.00007 M
Zn	0.0013 M
Zr	0.0016 M
Am	1000 µCi/l
Pu	97 µCi/l
Cs^{137}	3.5×10^5 µCi/l
Co^{60}	7.1×10^2 µCi/l
Eu^{154}	3.5×10^3 µCi/l
Eu^{155}	4.7×10^3 µCi/l
Sr^{90}	1.7×10^5 µCi/l

APPENDIX C

HOT BOILDOWN OF TANK 102-AX LIQUOR

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Internal Letter

Rockwell International

Date. February 22, 1980

No . 65124-80-093

TO: (Name, Organization, Internal Address)
. R. B. Bendixsen
. Tank Farm Process Control
. 2750E, 200 East

FROM: (Name, Organization, Internal Address, Phone)
. C. H. Delegard
. Waste Chemistry Unit
. 222-S, 200 West
. 2-1571

Subject: . Hot Boildown of Tank 102-AX Liquor

References: 1) Letter, February 4, 1980, R. B. Bendixsen to H. J. Eding, "Hot Boildown of Complexed Waste in Tank 102-AX"

2) Letter, January 23, 1980, C. H. Delegard to R. B. Bendixsen, "Hot Boildown of Tank 102-AX Waste Liquor"

In response to your referenced letter, a hot boildown of Tank 102-AX waste liquor was done. The boildown of a similar Tank 102-AX dilute complexed B-Plant waste was reported in a recent letter (Ref. 2). The purpose of the present study is to determine the nucleation and crystallization point of this complexed waste at 5°C (~40°F) as a function of waste volume reduction (WVR). This point would serve as the WVR limit for evaporator operations. Precipitation of complexed waste in the tanks is undesirable since the wastes' high viscosity precludes their retrieval for further processing. Five degrees Celsius is the estimated winter surface temperature of the tanked waste.

The routine boiling temperature and pressure data were gathered as a function of WVR. Note the flatness of this response as shown in Table I. Aliquots of the waste liquor were taken at 50, 60, 65 and 70 percent WVR and cooled to 5°C. No crystals were observed although the samples were slightly turbid. Viscosity data gathered for these aliquots are given in Table II.

The boildown was conducted at 40 Torr. Nucleation occurred during boildown, not during 5°C cooling, at 74 percent WVR. Typical of a complexed waste, crystallization was rapid producing ~90 volume percent settled solids at the boiling temperature. The waste's density was ~ 1.14 g/ml at 50 percent WVR and ~ 1.28 g/ml at nucleation (74 percent WVR).

R. B. Bendixsen
Page 2
February 22, 1980

The compositions of the two Tank 102-AX samples are given in Table III. The boildown was done on a 50/50 v/v composite of these samples. No analysis of the resultant slurry was done. Due to the high concentration of sulfate and carbonate in the feed and the limited solubilities of these salts, the solids are probably sodium sulfate dodecahydrate and sodium carbonate mnohydrate.

Please call me should you have questions regarding this work.

C. H. Delegard
Advanced Chemist

CHD:met

Information:

D. E. Bowers
J. S. Buckingham
G. T. Dukelow
M. J. Eding
D. L. Herting
M. T. Jansky
F. M. Jungfleisch
D. L. Merrick
S. A. Olsen
R. D. Prosser
T. B. Veneziano
Process Aids (8)
File KF55E

TABLE I

TEMPERATURE AND PRESSURE AT BOILING
VERSUS WVR FOR TANK 102-AX WASTE LIQUOR

	Boiling Point (°C)		
Percent WVR	40 Torr	60 Torr	80 Torr
0	35.6	43.0	49.0
10	35.8	43.2	48.8
20	36.0	42.8	48.8
30	36.2	43.6	49.2
40	36.2	43.8	49.4
50	36.6	44.2	50.2
60	37.8	45.4	51.2
65	38.6	47.4	53.8
70	40.8	48.0	54.6
74	42.6	51.2	56.8

TABLE II

VISCOSITY VERSUS WVR FOR
TANK 102-AX WASTE LIQUOR

	Viscosity (centipoise) at Spindle Speed		
Percent WVR	12 RPM	30 RPM	60 RPM
50	4.60	4.29	4.22
60	6.95	6.61	6.61
65	19.74	19.28	>19.1
70	22.99	22.80	>19.1

TABLE III

COMPOSITION OF TANK 102-AX
WASTE LIQUOR SAMPLES

	Sample # 7700		Sample # 7701	
Component	Molarity	Wt. Percent	Molarity	Wt. Percent
$NaAlO_2$	0.070	0.53	0.147	1.13
NaOH	0.329	1.21	0.172	0.64
$NaNO_3$	0.725	5.66	0.717	5.69
$NaNO_2$	0.267	1.69	0.268	1.73
Na_3PO_4	0.0142	0.22	0.0138	0.21
Na_2SO_4	0.187	2.44	0.164	2.17
NaF	0.0074	0.03	0.0074	0.03
Na_2CO_3	0.490	4.77	0.52	5.15
TOC (g/l)	-	-	9.75	0.91
H_2O		87.62		87.25
Total		104.17		104.91
Sp. G. (g/ml)	1.088		1.071	

APPENDIX D

HOT BOILDOWN OF TANK 102-AX WASTE LIQUOR

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Internal Letter

Rockwell International

Date January 23, 1980

No. 651240-80-064

TO: Name Organization (Internal Address)
. R. B. Bendixsen
. Tank Farm Process Control
. 2750-E, 200 East

FROM: (Name, Organization (Internal Address, Phone)
. C. H. Delegard
. Chemical Sciences Group
. 222-S, 200 West
. 2-1571

Subject . Hot Boildown of Tank 102-AX Waste Liquor

Ref: Letter, November 21, 1979, R. B. Bendixsen to H. J. Eding, "Hot" Boildown of Complexed Waste in Tank 102-AX

In response to your referenced letter, a hot boildown of Tank 102-AX waste liquor was recently done. This B-Plant waste contained significant organic carbon and carbonate, and was colored brown due to the presence of iron.

Boiling temperature and pressure data were gathered as a function of waste volume reduction (WVR). These data are presented in Table I. The waste nucleated at about 77 percent WVR. The boildown was stopped there, producing, upon cooling, about 90 volume percent solids which centrifuged to about 55 volume percent solids. The rapid crystallization and sticky character of the waste solids were indicative of complexed waste. At 50 percent WVR, the waste liquor had a density of ~1.24 g/ml. At nucleation, the slurry's density was about 1.57 g/ml.

Chemical analyses of the feed and product slurry material are presented in Table II. Aside from the erroneous NaOH assay, the slurry solids are primarily sodium carbonate and sodium nitrate.

Please call me if you should have any questions regarding this work.

C. H. Delegard, Advanced Chemist
Waste Chemistry Unit

CHD:jkr

Attachment

Information: J. S. Buckingham
G. T. Dukelow
H. J. Eding
D. L. Herting
F. M. Jungfleisch
T. A. Lane
D. L. Merrick
R. D. Prosser
T. B. Veneziano
Process Aids (8)
File Code: KF55E

Table I

TEMPERATURE AND PRESSURE AT BOILING
VERSUS WVR FOR TANK 102-AX WASTE LIQUOR

	Boiling Point (°C) at		
Percent WVR	40 Torr	60 Torr	80 Torr
0	34.2	42.4	48.2
10	35.0	43.2	48.8
20	35.8	43.6	49.2
30	36.2	43.8	49.6
40	36.6	44.4	50.2
50	37.0	45.0	50.8
60	38.6	46.4	52.2
70	41.2	48.6	54.8
77	44.2	51.8	57.8

Table II

ANALYSES OF FEED AND PRODUCT SLURRY - TANK 102-AX

	Feed Liquor		Product Liquor		Product Solid
Component	Molarity	Wt Percent	Molarity	Wt Percent	Wt Percent
$NaAlO_2$	0.0371	0.27	0.215	1.26	-0-
NaOH	0.572	2.06	<0.5*	<1.42*	>31.4*
$NaNO_2$	0.443	2.75	1.98	9.73	3.5
$NaNO_3$	0.865	6.61	4.12	24.94	11.2
Na_2CO_3	0.490	4.67	0.95	7.17	40.9
Na_3PO_4	0.100	1.47	0.0488	0.57	3.7
Fe_2O_3	0.0060	0.09	0.0308	0.35	2.4
TOC (g/l)	16.1	1.45	68.5	4.88	-0-
H_2O	--	84.4	--	53.62	-0-
Total	--	103.8	--	103.94	93.1
SpG (g/ml)	1.113	--	1.404	--	--

*Comparison of feed and product liquor analyses, especially aluminum, show a concentration factor of about five. This is consistant with the 77 percent WVR. Since it is unlikely that NaOH precipitated through concentration, the product liquor hydroxide analysis is undoubtedly low, while the product solid NaOH percentage is high.

APPENDIX E

ANALYSIS OF TANK 102-AX SLUDGE

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Internal Letter Rockwell International AX-102

Date: July 20, 1977

TO: (Name Organization Address)
J. W. Bailey

FROM (Name Organization Address Phone)
J. L. Starr
Chemical Sciences
222-S Bldg., 200 West
2-2529

Subject: Analysis of Tank 102-AX Sludge

Tank 102-AX has been sluiced and now contains some residual sludge. Before tank may be used for storage of evaporator slurry waste, an estimate of heat output from the remaining sludge must be made. Analysis of radionuclides contained in the sludge has been conducted to facilitate making such an estimate.

A series of six samples have been received and analyzed since February of this year. With the single exception of the last sample (#5403), all samples were dark, reddish-brown, viscous, and sticky. Sample #5403 was a dark brown, dry, hard, solid. All analyses were, apparently, done on solids only, except for #4095. As received, sample #4095 contained 89 percent solids. Analysis was conducted on a portion of the sample containing only 35 percent solids.

Analytical results are presented in the attachment. It has been suggested that these data points might best be evaluated by assuming a log-normal distribution curve. Therefore, average values and standard deviations have been omitted.

If you have questions, please feel free to call me.

J. L. Starr
Chemist

JLS:jf

Att.

cc: w/att.
JS Buckingham
RG Geier
GW Grimes
JE Horton
DC Lini
OR Rasmussen
ME Weems

R-07044

	#1	#2	#3039	#3040	$4095	#5403	
Pu	7.0×10^{-2}	7.2×10^{-2}	6.0×10^{-2}	1.9×10^{-1}	5.5×10^{-2}	7.9×10^{-2}	g/ℓ
$^{89+90}Sr$	6.5×10^{6}	1.5×10^{7}	1.4×10^{7}	2.2×10^{7}	2.7×10^{7}	1.5×10^{7}	μCi/ℓ
^{137}Cs	1.6×10^{6}	3.3×10^{5}	5.5×10^{3}	4.9×10^{5}	4.8×10^{5}	1.0×10^{6}	μCi/ℓ
^{60}Co	1.4×10^{5}	nf	nf	nf	nf	nf	μCi/ℓ
^{125}Sb	9.3×10^{5}	6.7×10^{5}	4.8×10^{5}	6.8×10^{5}	3.6×10^{5}	7.3×10^{7}	μCi/ℓ
^{144}Ce	1.3×10^{6}	1.8×10^{5}	9.0×10^{5}	2.1×10^{6}	nf	2.3×10^{6}	μCi/ℓ
^{155}Eu	5.4×10^{5}	8.9×10^{5}	4.6×10^{5}	8.8×10^{5}	nf	nf	μCi/ℓ
^{154}Eu	nf	nf	nf	nf	nf	3.9×10^{-1}	μCi/ℓ
U	na	1.7×10^{-6}	3.0×10^{-4}	8.1×10^{-4}	1.3×10^{-2}	1.0×10^{-2}	#/gal
Si	na	na	na	na	na	0.86M	

nf - not found
na - not analyzed

APPENDIX F

ACID INSOLUBLE SOLIDS IN PAS

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TCRC-0 Atlantic Richfield Hanford Company AX-102 1

Date: January 20, 1977

To: J. C. Womack

From: J. S. Buckingham - 2-2487 J. S. Buckingham

Subject: ACID INSOLUBLE SOLIDS IN PAS

References:
(1) Letter, September 25, 1974, J. E. Horton to O.R.H. Rasmussen, "analysis and Characterization of Sludge Samples Received from Tank 102-AX"

(2) Letter, November 12, 1974, J. E. Horton to O.R.H. Rasmussen, "Analysis and Characterization of Sludge Sample Received from Tank 103-AX"

(3) Letter, August 4, 1975, J. E. Horton to O.R.H. Rasmussen, "Analysis and Characterization of 101-AX Tank Sludge and Supernatant Liquid"

After our conversation Tuesday morning, January 18, 1977, concerning insoluble or "terminal" solids, I calculated the amount of strontium or solids in AR-001P (PAS) samples as reported by analytical services.

TABLE I

STRONTIUM ANALYSIS OF SOLIDS IN PAS

Sample "T" No.	Date Sampled	$^{89+90}Sr$, Ci/l
4617	4/27/76	11.3
6190	8/11/76	0.4
6272	8/16/76	21.9
6653	8/30/76	9.9
6725	9/01/76	0.2
6841	9/04/76	0.2
6957	9/07/76	20.8
7167	9/14/76	21.2
7326	9/18/76	19.9
7483	9/22/76	23.6
7631	9/25/76	22.8
7734	9/28/76	22.8
7734	9/28/76	19.7
7980	10/05/76	8.3
8097	10/08/76	24.8
8710	10/29/76	10/7
9506	11/25/76	2.6
9877	12/04/76	141.0
71	12/09/76	7.5
Average		20.4

54-6000-030 (10-68)

The data are quite scattered; however, quite a few seem to fall near the 20.8 Ci/liter average. The $^{89+90}Sr$ analysis of sludge from 102-AX, reported in reference 1, was 9.31 Ci/liter, which is lower than the average solid analyses from PAS samples. The $^{89+90}Sr$ analyses of sludges from Tanks 103-AX and 101-AX, as reported in references 2 and 3, were 22.4 Ci/liter and 28.2 Ci/liter, respectively.

If we can assume the sludges in Tanks 101-AX, 102-AX, and 103-AX are similar, then it looks like our analysis of the 102-AX sludge is low by a factor of around 2. We are going to try to rerun the analysis on the 102-AX sludge. Further, if my assumption is correct, the 102-AX sludge should contain around 20 Ci/liter $^{89+90}Sr$ which is the average we are seeing on PAS solids.

In conclusion, this scattered data seems to indicate that the strontium content of the solids being sent to B Plant are identical to what was in the original sludge.

Please let me know if I can be of any further help on this problem.

JSB:ms

cc: GD Campbell
DC Lini
OR Rasmussen
RW Spencer
RE Van der Cook
Process Aids (7)
File
LB

54-6000-031 (10-68)
AEC RL RICHLAND WASH

Atlantic Richfield Hanford Company

3

Date: January 28, 1977

To: G. D. Campbell

From: J. S. Buckingham - 2-2487 JSBuckingham

Subject: ACID INSOLUBLE SOLIDS IN PAS

Reference: Letter, January 20, 1977, J. S. Buckingham to J. C. Womack, same subject

Below are listed some additional $^{89+90}Sr$ concentrations on PAS solids. Hopefully these PAS samples were all from sluicing Tank 102-AX. Throwing out the one high number on sample number 1535, the average is 2.25. Assuming the sludge analysis reported in reference 1 of the above reference is correct at 9.31 Ci $^{89+90}Sr$/liter, it appears that around 75 percent of the $^{89+90}Sr$ in the 102-AX sludge is soluble in nitric acid.

TABLE I

STRONTIUM ANALYSIS OF SOLIDS IN PAS

Sample No.	$^{89+90}Sr$ Ci/ℓ
976	3.75
1209	2.67
1293	1.68
1354	2.48
1446	1.22
1535	37.9*
1624	2.26
1697	1.76
1915	3.33
2018	2.33
2126	1.31
2356	1.94
Average	2.25

*Not used in average

54-6000-030 (10-68)

Atlantic Richfield Hanford Company

4

G. D. Campbell
Page 2
January 28, 1977

Please let me know if I can be of any further help with this problem.

JSB:ms

cc: DC Lini
OR Rasmussen
RW Spencer
RE Van der Cook
JC Womack
Process Aids (7)
File
LB

94-6000-031 (10-68)

APPENDIX G

ANALYSES AND CHARACTERIZATION OF SLUDGE SAMPLES RECEIVED FROM TANK 102-AX

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Date: September 25, 1974

To: O. R. H. Rasmussen

From: J. E. Horton

Subject: ANALYSES AND CHARACTERIZATION OF SLUDGE SAMPLES RECEIVED FROM TANK 102-AX

A sample of sludge from 102-AX showed that it was a very soft material, and radish brown in color. The $^{89+90}Sr$ content of the 102-AX sludge was 9.31 Curies/liter. Particle size distribution showed that 99 weight percent of the sludge was between 10 and 50 µm. Purex Acid Sludge (PAS) was made from a 30 percent solids and 70 percent supernatant. It contained 80 percent of the $^{89+90}Sr$. The PAS gelled in two days due to high silica content.

The "as received" 102-AX sludge was analyzed by fusing 1.5 milliliters with KOH, and dissolving the melt with HCl and dilution with H_2O to a volume of 265 milliliters. Analyses, shown in Table I (attached), are per liter of "as received" sludge.

Particle size distribution of the 102-AX sludge was determined on a HIAC particle counter. Results are shown in Table II (attached).

The PAS was made up from a 30 volume percent sludge slurry. Ten milliliters of the 30 percent slurry was added to 1.5 milliliters of 12M HNO_3. Severe foaming accompanied by the evolution of copious amounts of NO_x fumes occurred during acidification. The foaming was very stable. The 102-AX is comparable to the foaming in samples 104-A and 104-AX. Analyses of PAS solution are shown in Table III (attached).

Acidification did not dissolve all the sludge. The remaining sludge was treated with equal volumes of 50 percent NaOH, then acidified with 12M HNO_3; this removes additional strontium.

The remaining two milliliters of solids from the PAS and NaOH strikes were fused with KOH. The melt

51-4000-030 (10-78)
AEC-GL RICHLAND WASH

O. R. H. Rasmussen
Page 2
September 25, 1974

was dissolved with con HCl, and diluted to a volume of 450 milliliters with H_2O. The amount of strontium in the PAS, removed by the caustic-acid treatment, and left on the undissolved solids are shown in Table IV (attached).

Please call me if you have any questions regarding this work.

JEH:jf

Att. (2)

cc: w/att.
H Babad
JS Buckingham
WR Christensen
NL Harms
GJ Raab
RL Walser
JC Womack
Process Aids (7)

54-6000-031 (10-68)
AEC-RL RICHLAND, WASH.

TABLE I

ANALYSIS OF 102-AX SLUDGE "AS RECEIVED"

		wt.%
Wet Density	1.57	
Dry Density	0.9025	
% H_2O		42.5
Si	1.26 moles/ℓ	2.2
Fe	2.54 moles/ℓ	9.05 wt.%
Mg	0.044 moles/ℓ	.07
Mn	0.217 moles/ℓ	0.77
Ca	0.199 moles/ℓ	0.52
Ba	0.011 moles/ℓ	0.01
Sr, M	0.057 moles/ℓ	0.3
Pu	6.71×10^{-2} g/ℓ	
$^{89+90}Sr$	9.31×10^{6} µCi/ℓ	
^{137}Cs	1.26×10^{6} µCi/ℓ	
^{60}Co	1.31×10^{4} µCi/ℓ	
^{125}Sb	1.51×10^{5} µCi/ℓ	
$^{144}Ce^{144}Pr$	2.38×10^{7} µCi/ℓ	
^{154}Eu	3.51×10^{5} µCi/ℓ	

TABLE II

PARTICLE SIZE DISTRIBUTION

Particle Size µm	Average Dia., µm	Wt. Percent
10	16.5	35.0
20	25.9	45.7
30	35.7	14.3
40	45.5	3.1
50	55.4	1.1
60	65.4	0.4
70	75.3	0.3
80	85.3	0.2
90	95.3	0.1

Avel = 23µ

TABLE III

ANALYSIS OF 102-AX PAS

Si	0.121M
Fe	0.253M
Al	0.494M
Ca	1.33×10^{-2}M
Sr	1.50×10^{-4}M
H	1.56M
$^{89+90}Sr$	1.61×10^{6} µCi/ℓ
^{137}Cs	4.78×10^{2} µCi/ℓ
^{60}Co	16 µCi/ℓ
^{125}Sb	1.22×10^{2} µCi/ℓ
$^{144}Ce^{144}Pr$	2.23×10^{3} µCi/ℓ
^{154}Eu	48.5 µCi/ℓ

TABLE IV

PERCENT STRONTIUM IN PAS AND ON SOLIDS

Material	Volume ml.	Volume Vol%	$^{39+90}Sr$ µCi/ℓ	$^{39+90}Sr$ Total µCi	Percent
riginal Sludge	3	100	9.31×10^{6}	2.79×10^{4}	100
AS (liquid)	14		1.61×10^{6}	2.25×10^{4}	81
(solid)	2	67	4.68×10^{5}	1.87×10^{3}	7
austic-Acid (liquid)	4				
sol. Solid (fusion)	2	67	3.04×10^{3}	1.37×10^{3}	5
	450				

APPENDIX H

DIFFERENTIAL SCANNING CALORIMETRY AND THERMOGRAVIMETRIC ANALYSIS SCANS FOR 1995 SAMPLE ANALYSES OF TANK 241-AX-102

(The data contained in this Appendix were excerpted from WHC-SD-WM-DP-100[1])

[1]Rice, A. D., 1995, *45-Day Safety Screen Results for Tank 241-AX-102, Augers 95-AUG-006 and 95-AUG-007*, WHC-SD-WM-DP-100, Westinghouse Hanford Company, Richland, Washington.

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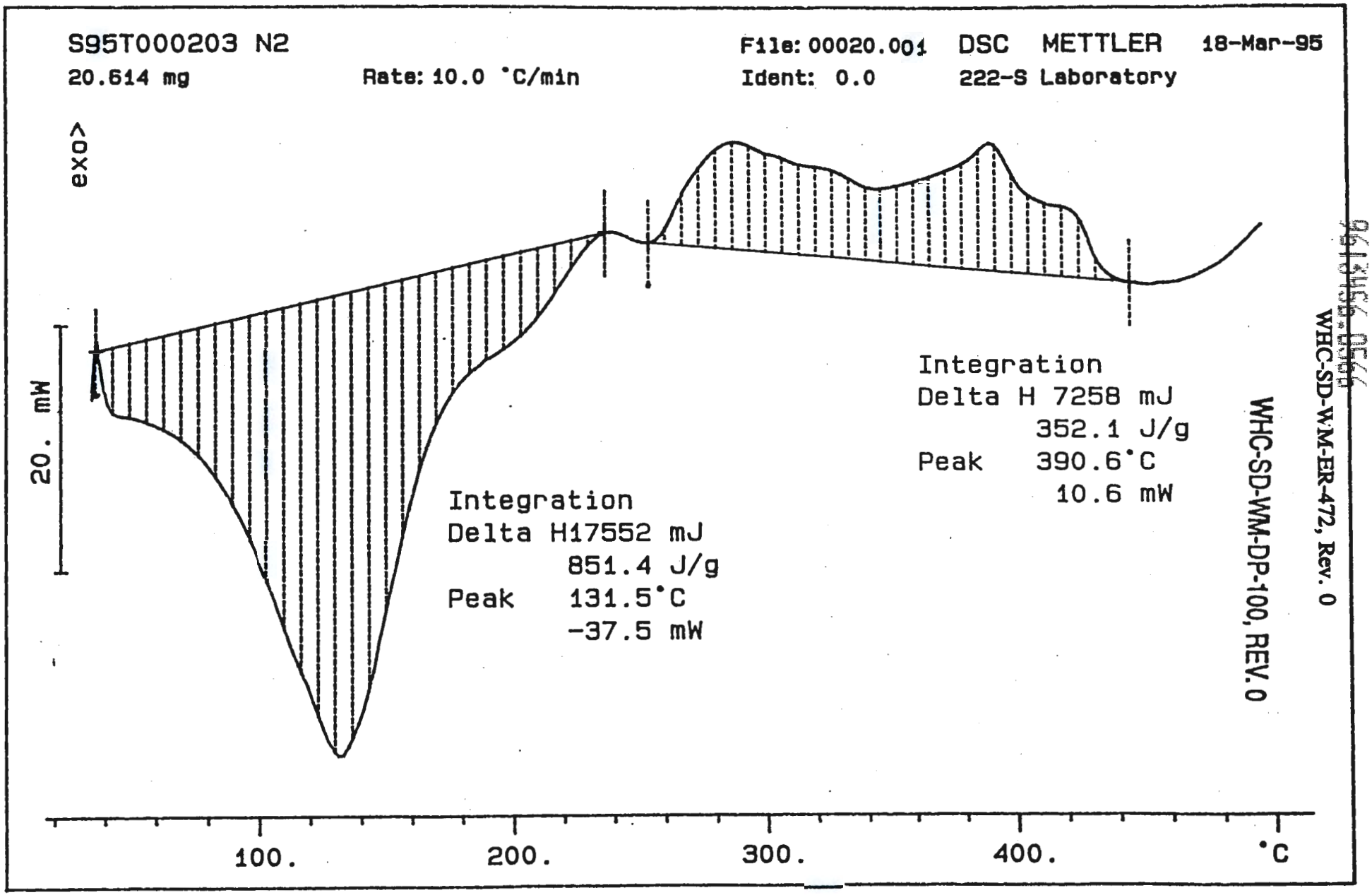
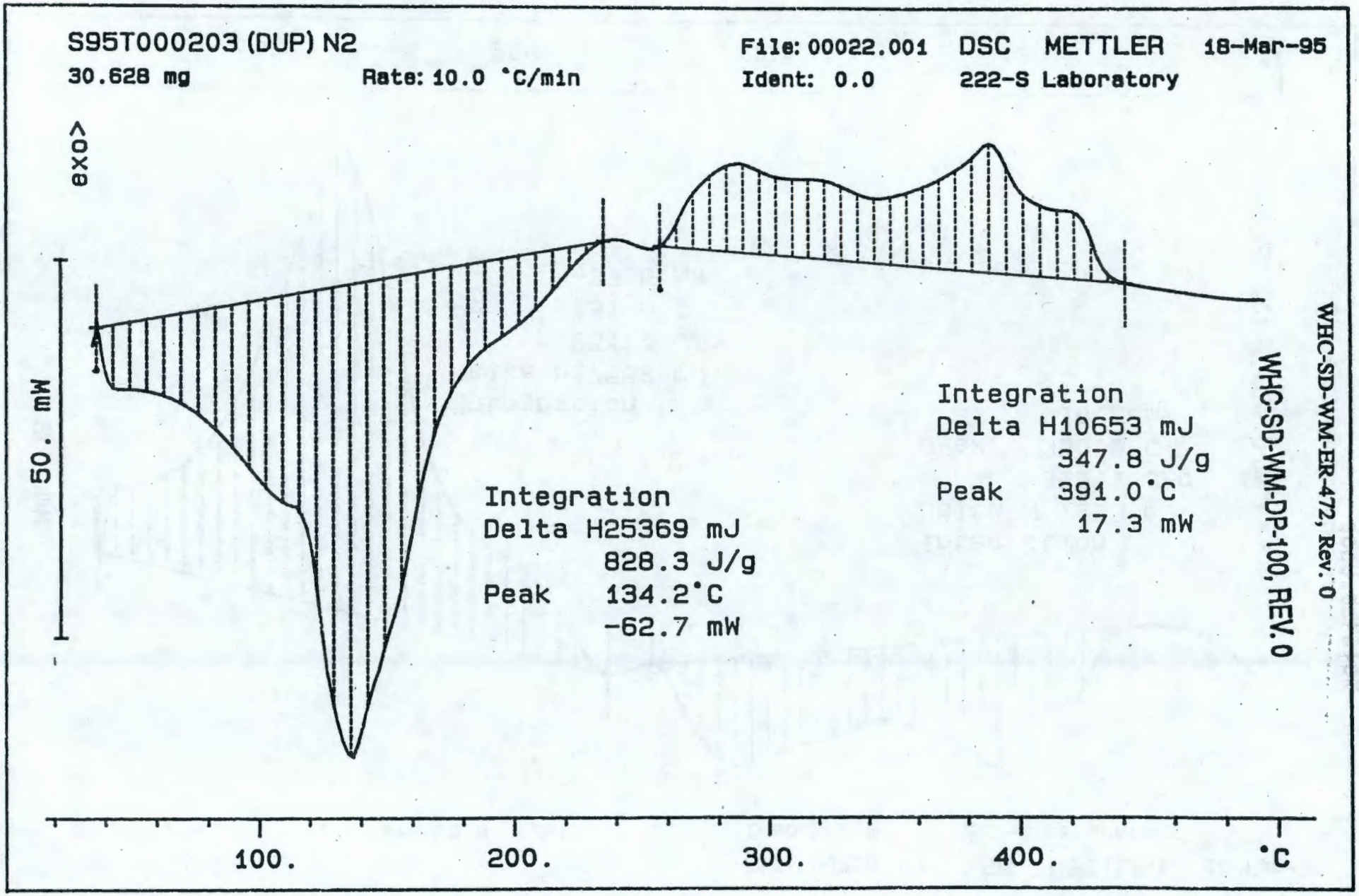


WHC-SD-WM-DP-100, REV. 0

WHC-SD-WM-ER-472, Rev. 0



WHC-SD-WM-ER-472, Rev. 0

WHC-SD-WM-DP-100, REV. 0

DSC STD 12N14-A

6.438 mg

Rate: 10.0 °C/min

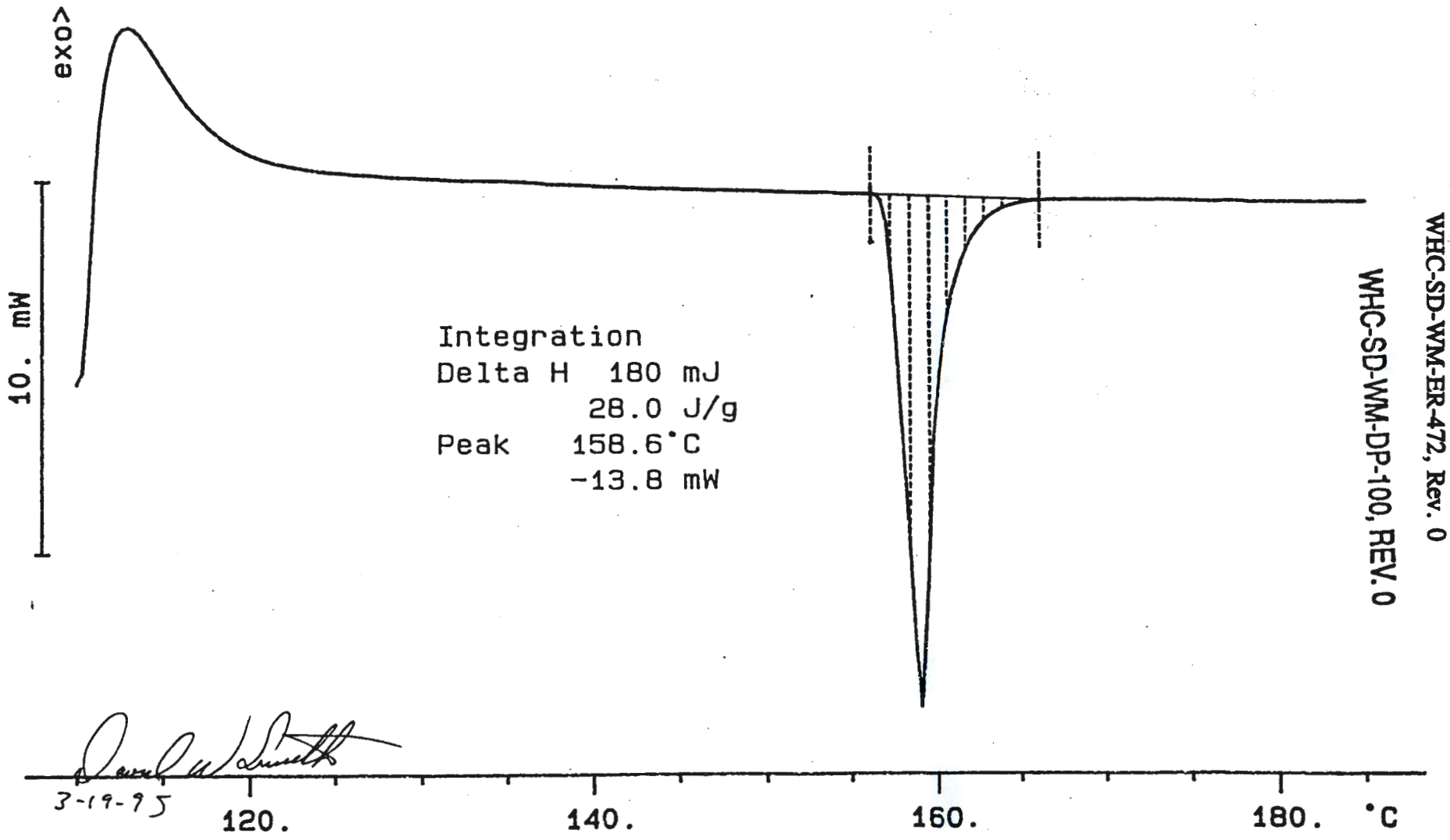
File: 00026.001

Ident: 0.0

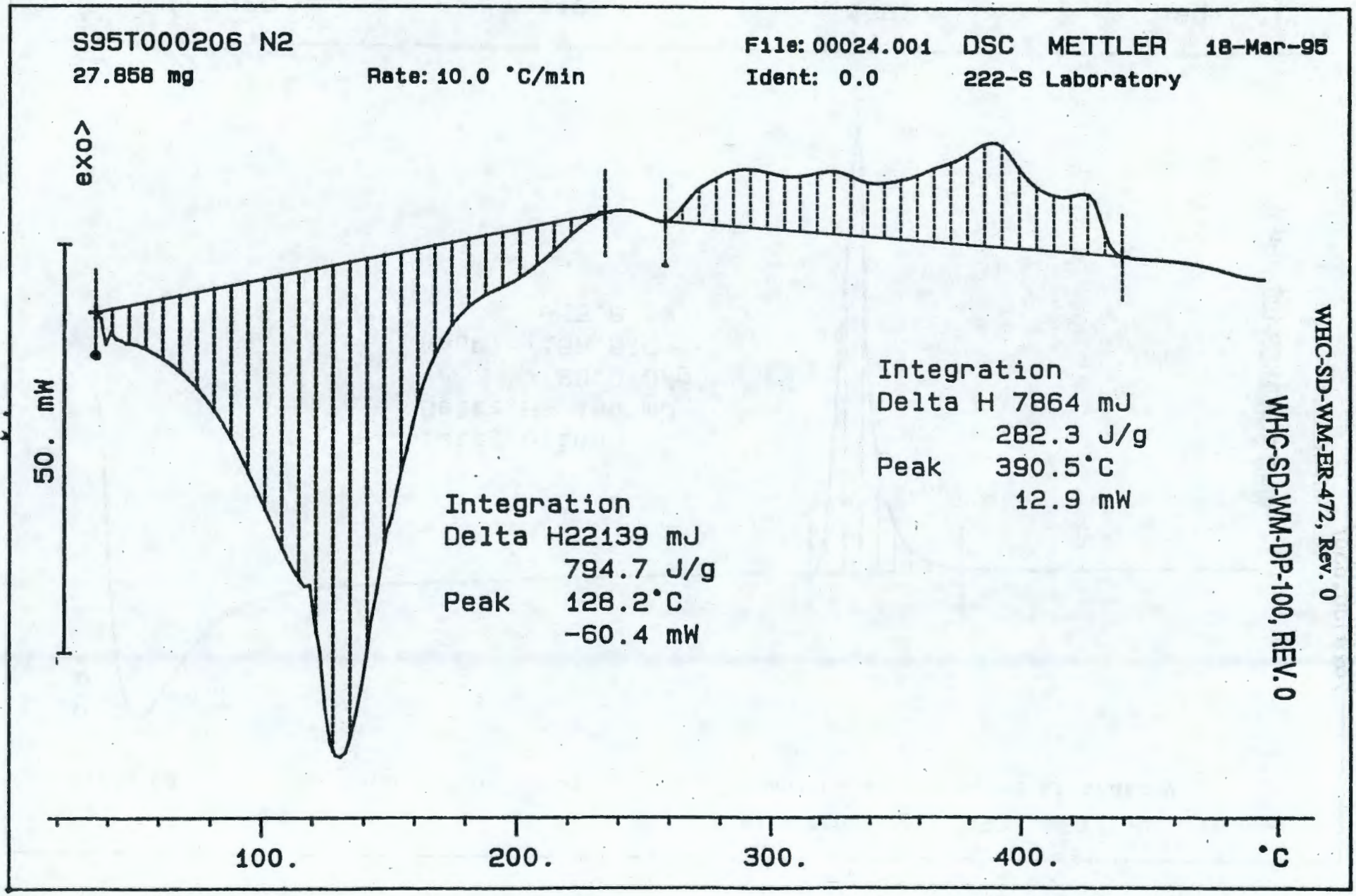
DSC METTLER

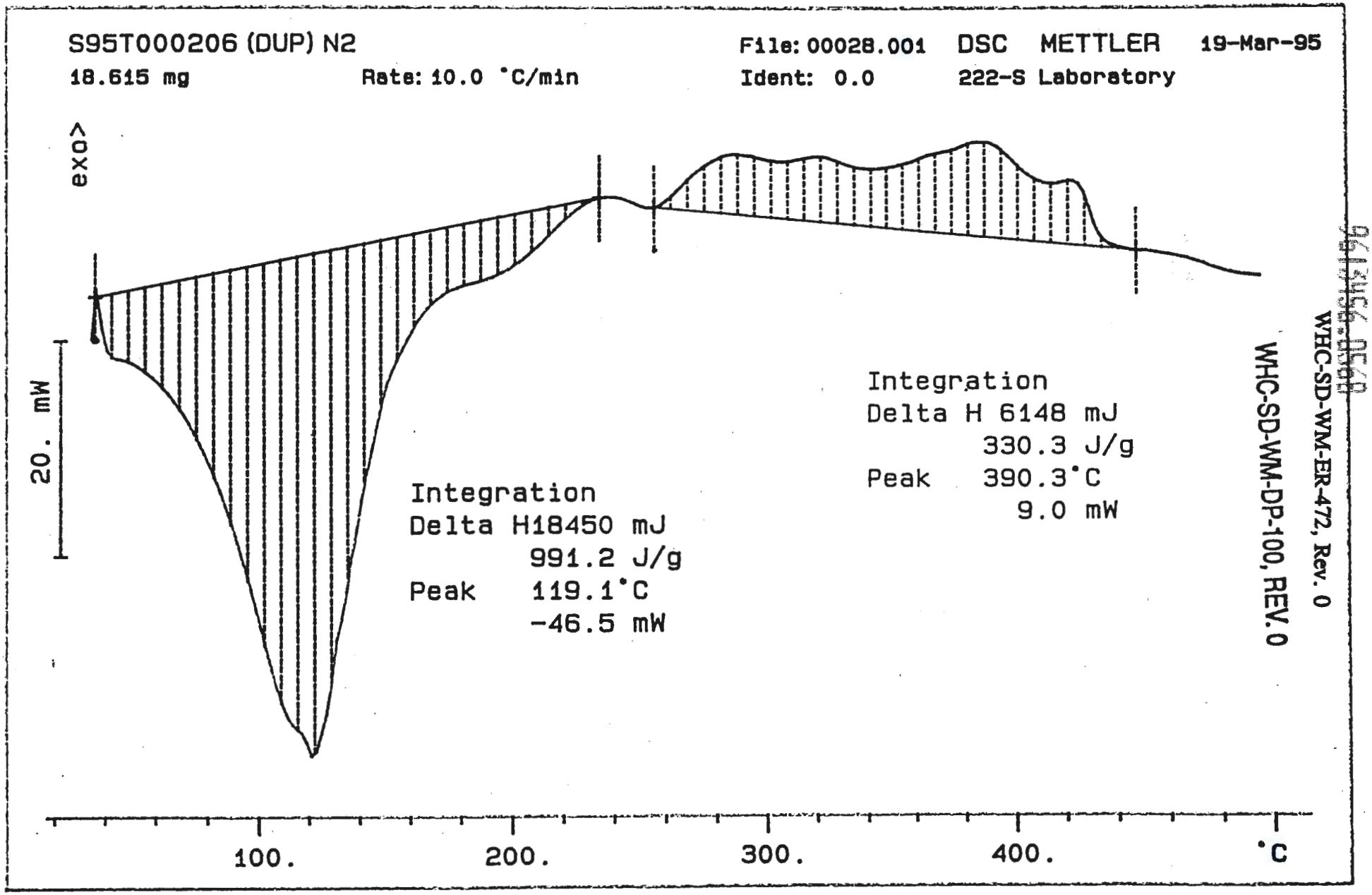
222-S Laboratory

19-Mar-95



3-19-95


S95T000206 N2
27.858 mg
Rate: 10.0 °C/min
File: 00024.001
Ident: 0.0
DSC METTLER
222-S Laboratory
18-Mar-95
exo>
50. mW
Integration
Delta H22139 mJ
794.7 J/g
Peak 128.2°C
-60.4 mW
Integration
Delta H 7864 mJ
282.3 J/g
Peak 390.5°C
12.9 mW
100.
200.
300.
400.
°C


S95T000206 (DUP) N2
18.615 mg
Rate: 10.0 °C/min
File: 00028.001
Ident: 0.0
DSC METTLER
222-S Laboratory
19-Mar-95
exo>
20. mW
Integration
Delta H18450 mJ
991.2 J/g
Peak 119.1°C
-46.5 mW
Integration
Delta H 6148 mJ
330.3 J/g
Peak 390.3°C
9.0 mW
100.
200.
300.
400.
°C

LABCORE Data Entry Template for Worklist# 779

Analyst: BDV Instrument: DSC01 _____ Book # _____

Method: LA-514-113 Rev/Mod B-1 WHC-SD-WM-DP-100, REV. 0

Worklist Comment: Calculated Dry DSC for AX-102

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-------TEST------	MATRIX	~~ACTUAL~~ 3/22/95 BDV (DSC)	~~FOUND~~ 3/22/95 BDV (TGA)	~~DL~~ 3/22/95 BDV (Dry DSC)	UNIT
95000014	AX-102	1 SAMPLE	S95T000203	0	DSC-02	SOLID	3/22/95 BDV ~~N/A~~ 352.1	28.79	494	Joules/g Dry
95000014	AX-102	2 DUP	S95T000203	0	DSC-02	SOLID	347.8	28.79	488 3/22/95 BDV ~~N/A~~	Joules/g Dry
95000015	AX-102	3 SAMPLE	S95T000206	0	DSC-02	SOLID	3/22/95 BDV ~~N/A~~ 282.3	32.21	416	Joules/g Dry
95000015	AX-102	4 DUP	S95T000206	0	DSC-02	SOLID	330.3	32.21	487 3/22/95 BDV ~~N/A~~	Joules/g Dry

Final page for worklist # 779

Blandina Valenzuela 3/22/95
Analyst Signature Date

Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

LABCORE Data Entry Template for Worklist# 647

Analyst: DWS **Instrument:** TGA01 **Book #** 42N8-A

Method: LA-560-112 Rev/Mod A-2 WHC-SD-WM-DP-100, REV. 0

Worklist Comment: Please run under N2. AX-102 JMF

GROUP	PROJECT	S	TYPE	SAMPLE#	R	A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1	STD				TGA-01	SOLID	59.19	57.44	N/A	%
95000014	AX-102	2	SAMPLE	S95T000203	0		TGA-01	SOLID	N/A	29.57		%
95000014	AX-102	3	DUP	S95T000203	0		TGA-01	SOLID	29.57	28.01	N/A	%
95000015	AX-102	4	SAMPLE	S95T000206	0		TGA-01	SOLID	N/A	31.12		%
		5	STD				TGA-01	SOLID	59.19	57.57	N/A	%
95000015	AX-102	6	DUP	S95T000206	0		TGA-01	SOLID	31.12	33.30	N/A	%

Final page for worklist # 647

See attached for signatures 3/21/95
Analyst Signature **Date**

Analyst Signature **Date**

Data entered and verified by
Blandina Valenzuela 3/22/95

Data Entry Comments: S95T000203 produced a second weight loss step of 17% after the initial water loss. S95T000206 produced a second weight loss step of 20.11% after the initial water loss.

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

worklistrpt Version 2.0 02/21/95
03/03/95 16:14

LABCORE Data Entry Template for Worklist# 647

Analyst: DWS **Instrument:** TGA01 **Book #** 42WB-A

Method: LA-560-112 Rev/Mod A-2

WHC-SD-WM-DP-100, REV. 0

Worklist Comment: Please run under N2. AX-102 JMF

GROUP	PROJECT	S TYPE	SAMPLE#	R	A	-------TEST------	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD				TGA-01	SOLID	~~59.19~~	~~57.44~~	3/21/95 BDV N/A	%
95000014	AX-102	2 SAMPLE	S95T000203	0		TGA-01	SOLID	N/A	~~29.57~~	3/21/95 BDV	%
95000014	AX-102	3 DUP	S95T000203	0		TGA-01	SOLID	~~29.57~~	~~28.01~~	3/21/95 BDV N/A	%
95000015	AX-102	4 SAMPLE	S95T000206	0		TGA-01	SOLID	N/A			%
95000015	AX-102	5 DUP	S95T000206	0		TGA-01	SOLID			N/A	%

Final page for worklist # 647

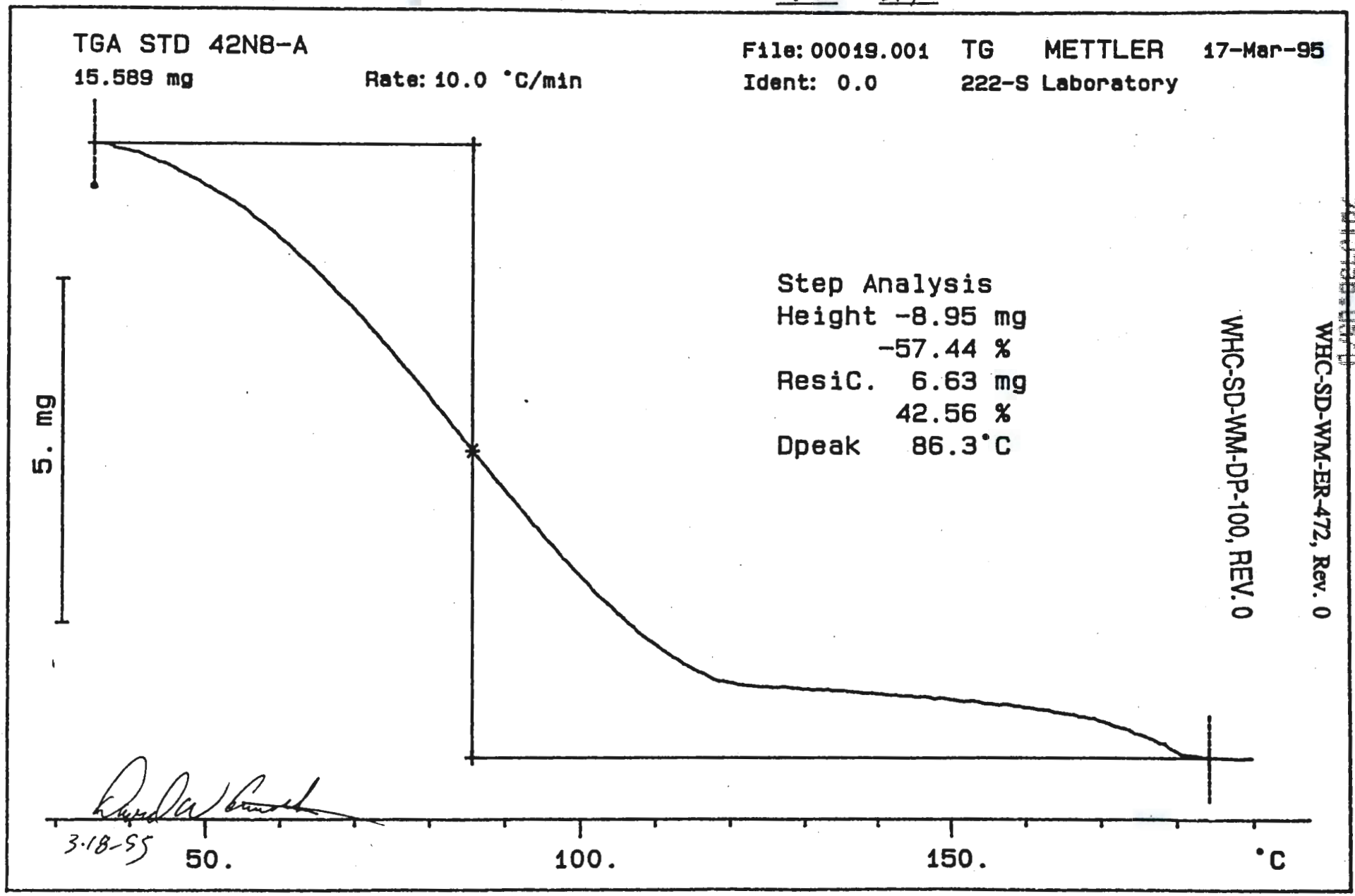
Analyst Signature Date 3-18-95

Analyst Signature Date

Data Entry Comments: ~~S95T000203 produced a second weight loss step~~ 3/22/95 BDV

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

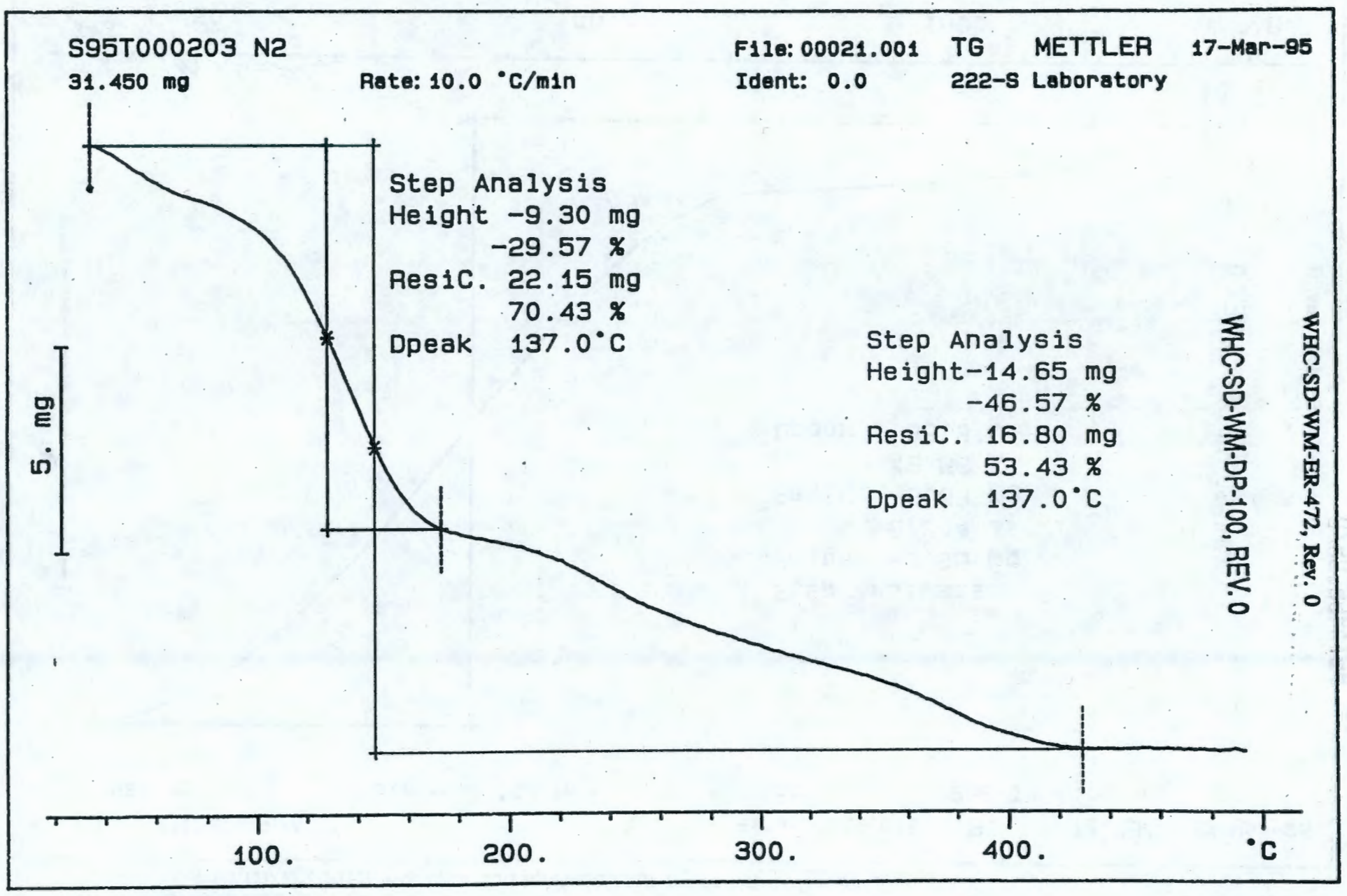
SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 19 TO 24.

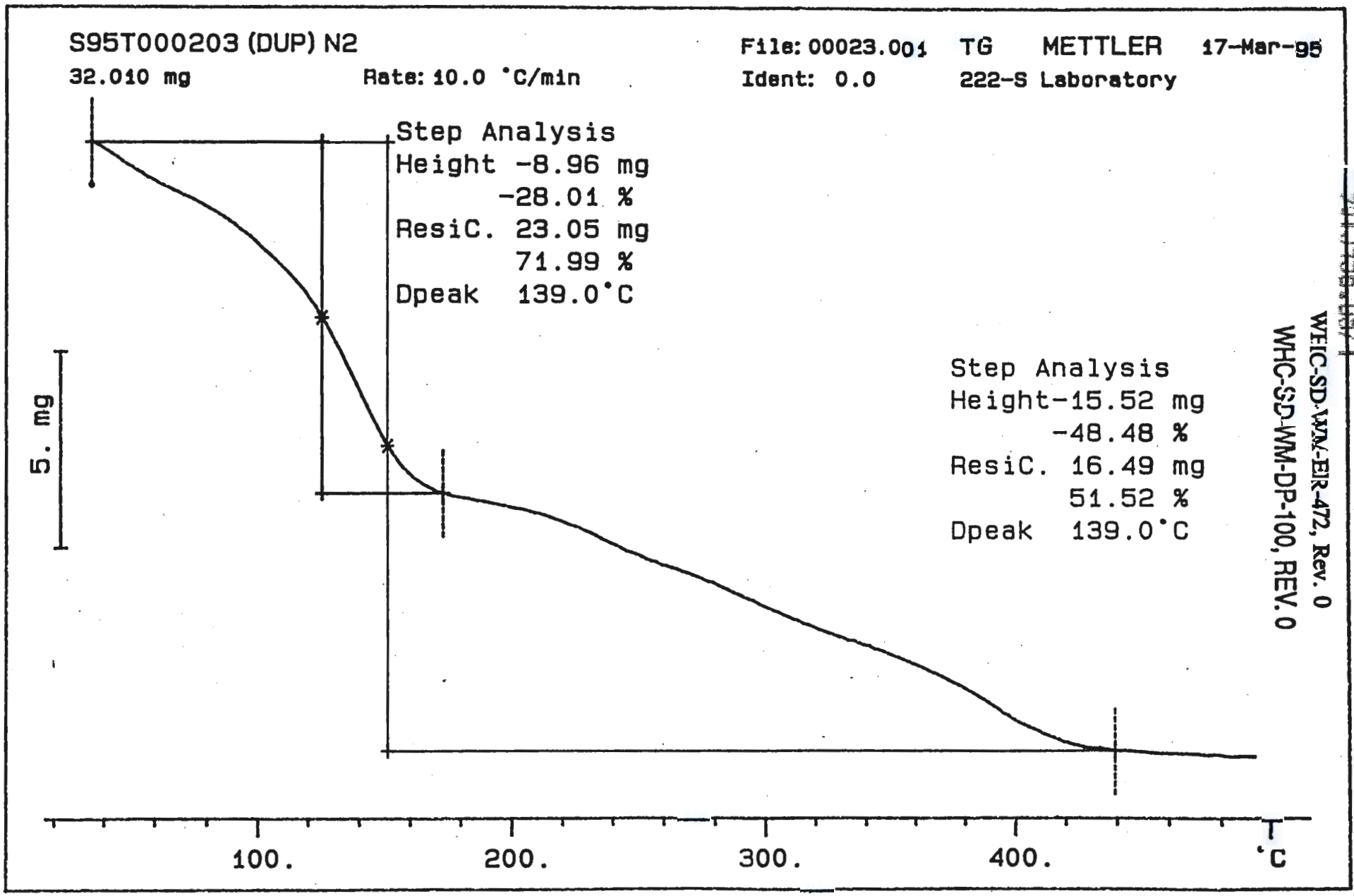


3-18-95

WHC-SD-WM-DP-100, REV. 0

WHC-SD-WM-ER-472, Rev. 0


S95T000203 N2
31.450 mg
Rate: 10.0 °C/min
File: 00021.001
TG METTLER
17-Mar-95
Ident: 0.0
222-S Laboratory
Step Analysis
Height -9.30 mg
-29.57 %
ResiC. 22.15 mg
70.43 %
Dpeak 137.0°C
Step Analysis
Height-14.65 mg
-46.57 %
ResiC. 16.80 mg
53.43 %
Dpeak 137.0°C
5. mg
100.
200.
300.
400.
°C



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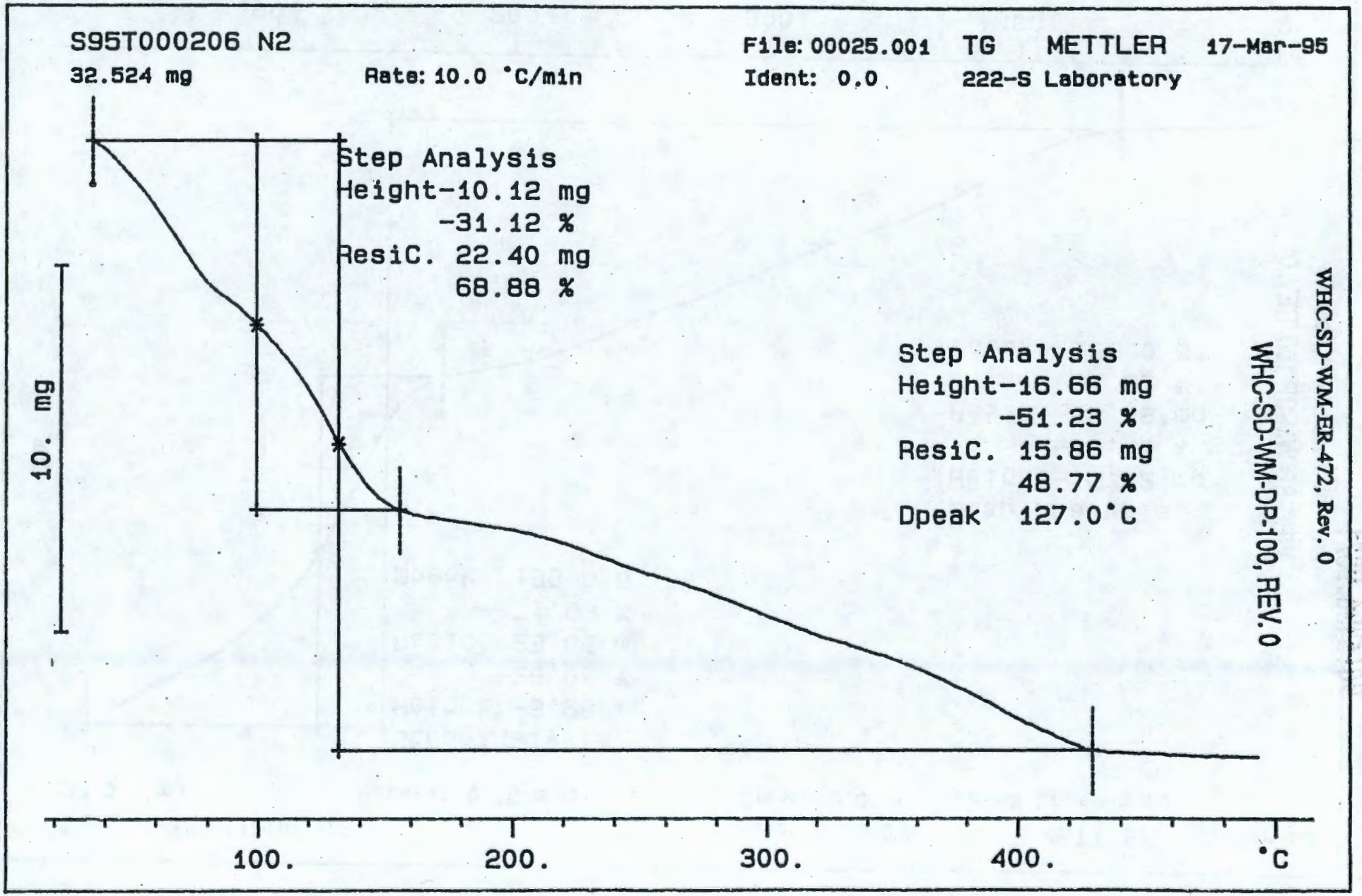
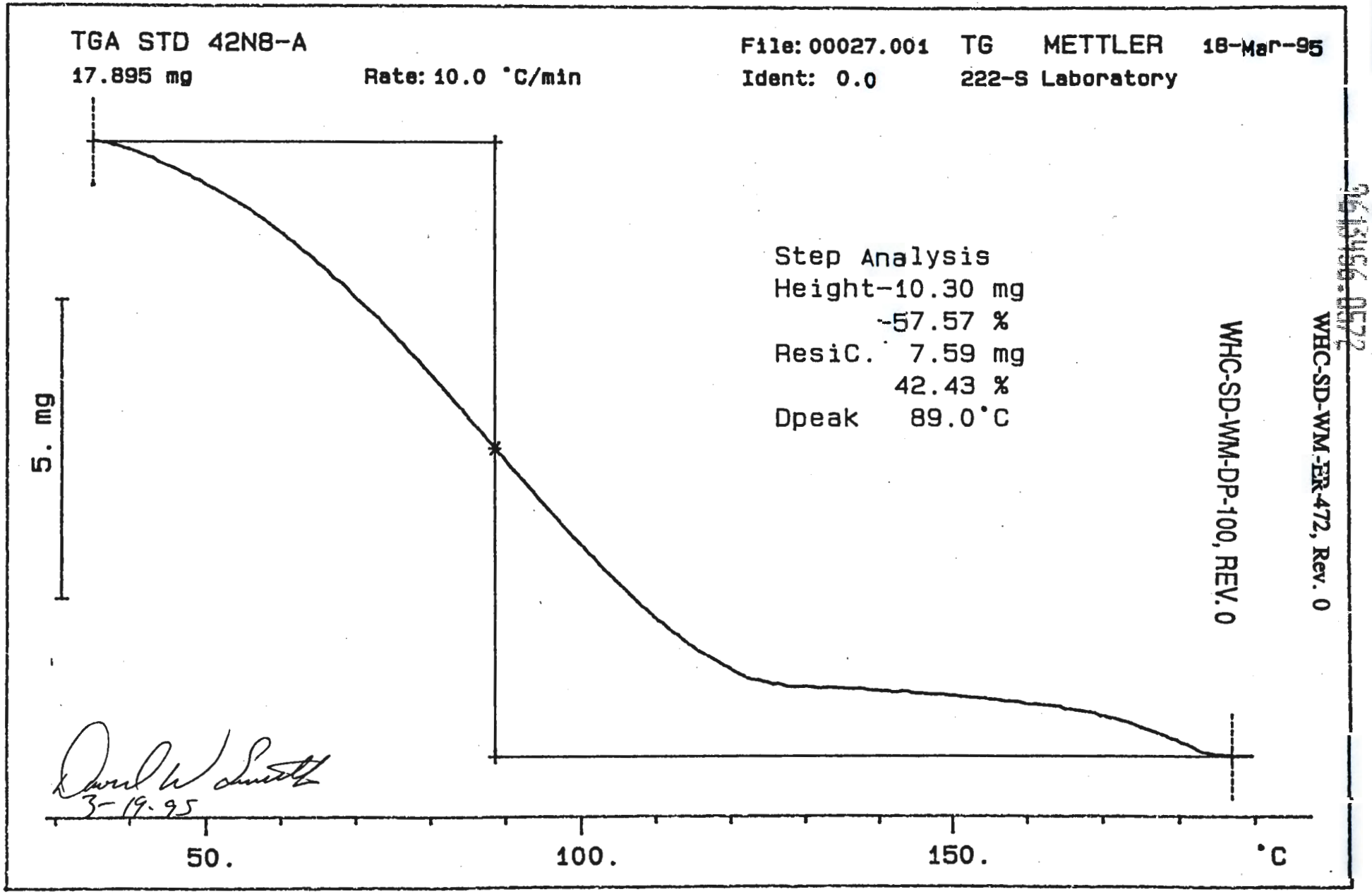
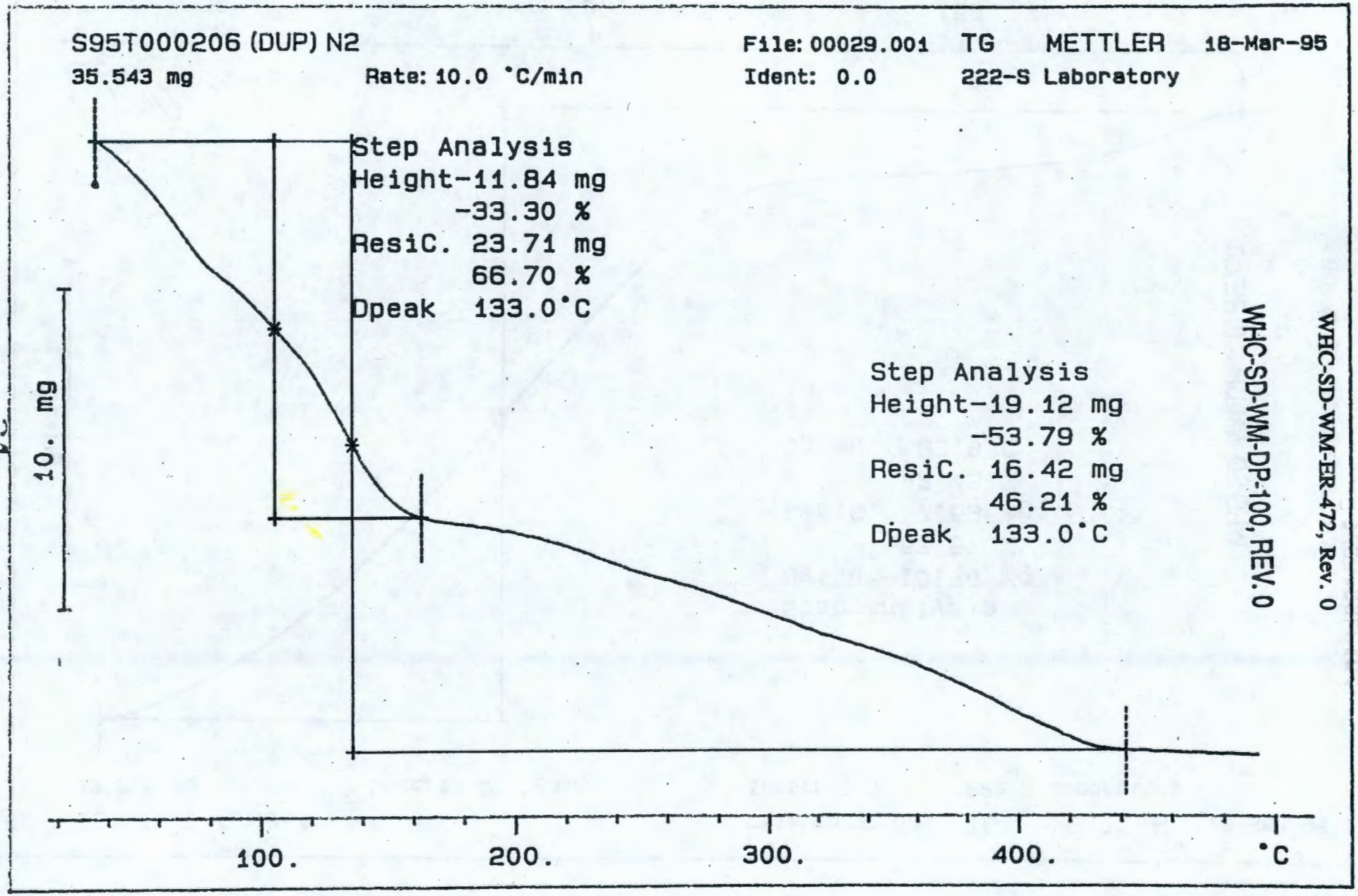
WHC-SD-WM-DP-100, REV. 0


S95T000206 N2
32.524 mg
Rate: 10.0 °C/min
File: 00025.001
TG
METTLER
17-Mar-95
Ident: 0.0
222-S Laboratory
Step Analysis
Height-10.12 mg
-31.12 %
ResiC. 22.40 mg
68.88 %
Step Analysis
Height-16.66 mg
-51.23 %
ResiC. 15.86 mg
48.77 %
Dpeak 127.0°C
10. mg
100.
200.
300.
400.
°C


TGA STD 42N8-A
17.895 mg
Rate: 10.0 °C/min
File: 00027.001
TG METTLER
18-Mar-95
Ident: 0.0
222-S Laboratory
Step Analysis
Height-10.30 mg
-57.57 %
ResiC. 7.59 mg
42.43 %
Dpeak 89.0°C
5. mg
50.
100.
150.
°C
3-19-95



WHC-SD-WM-DP-100, REV. 0

WHC-SD-WM-ER-472, Rev. 0

9613456.0573

DISTRIBUTION SHEET

To	From	Page 1 of 4
Distribution	J. P. Sederburg	Date August 8, 1995
Project Title/Work Order		EDT No. 612678
Tank Characterization Report for Single-Shell Tank 241-AX-102		ECN No. N/A

Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
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Sandia National Laboratory P.O. Box 5800 MS-0744, Dept. 6404 Albuquerque, New Mexico 87815					
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Nuclear Consulting Services Inc. P.O. Box 29151 Columbus, Ohio 43229-01051					
J. L. Kovach		X			
Chemical Reaction Sub-TAP 202 Northridge Court Lindsborg, Kansas 67456					
B. C. Hudson		X			
Tank Characterization Panel Senior Technical Consultant Contech 6301 Indian School Road, N.W., Suite 614 Albuquerque, New Mexico 87110					
J. Arvisu		X			
U.S. Department of Energy - Headquarters Office of Environmental Restoration and Waste Management (EM-563) 12800 Middlebrook Road Germantown, Maryland 20874					
K. T. Lang		X			
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