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7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, WHC 222-S Laboratory, and PNL 325 Analytical Chemistry Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of auger samples from tank 241-AX-104.

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DATE

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WHC-SD-WM-TP-243  
Revision 0

# Tank 241-AX-104 Tank Characterization Plan

Prepared for the U.S. Department of Energy  
Office of Environmental Restoration  
and Waste Management

**MASTER**

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LIST OF ACRONYMS

ACL	Analytical Chemistry Laboratory
AX-104	tank 241-AX-104
CASS	Computer Automated Surveillance System
DNFSB	Defense Nuclear Facility Safety Board
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
DST	double-shell tank
FY	fiscal year
GEA	gamma energy analysis
ICP	inductively-coupled plasma
MW	metal waste
NCPLX	non-complexed waste
RSST	reactive system screening tool
SST	single-shell tank
TCP	Tank Characterization Plan
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TPA	Tri-Party Agreement
TOC	total organic carbon
TWRS	Tank Waste Remediation System
USQ	unreviewed safety question
WHC	Westinghouse Hanford Company



## TANK 241-AX-104 TANK CHARACTERIZATION PLAN

## 1.0 SPECIFIC TANK CHARACTERIZATION OBJECTIVES

The Defense Nuclear Facilities Safety Board has directed the DOE to concentrate the near-term sampling and analysis activities on identification and resolution of safety issues (Conway 1993). The data quality objective (DQO) process was chosen as a tool to be used to identify sampling and analytical needs for the resolution of safety issues. As a result, a revision in the Federal Facility Agreement and Consent Order (TPA) milestone M-44-00 has been made, which states that "A Tank Characterization Plan (TCP) will also be developed for each DST and SST using the DQO process. Development of TCPs by the DQO process is intended to allow users (e.g., Hanford Facility user groups, regulators) to ensure their needs will be met and that resources are devoted to gaining only necessary information." This document satisfies this requirement for tank 241-AX-104 (AX-104) fiscal year 1994 safety screening activity.

This Tank Characterization Plan will identify characterization objectives pertaining to sample collection, hot cell sample isolation, laboratory analytical evaluation and reporting requirements in accordance with the *Tank Safety Screening Data Quality Objective* (Redus and Babad 1994), in addition to reporting the current contents and status of the tank as projected from historical information.

## 1.1 Tank Safety Screening Data Quality Objectives

There are four Watch List tank classifications with safety issues related to public and worker health that have been associated with the Hanford Site underground storage tanks: ferrocyanide, organic, flammable gas, criticality, high heat, and vapor (Babad 1992). Of the 149 single-shell (SSTs) and 28 double-shell tanks (DSTs), 52 are currently classified on one or more of the watch lists (Hanlon 1994).

The *Tank Safety Screening Data Quality Objective* (Redus and Babad 1994) describes the sampling and analytical requirements that are used to screen waste tanks for unidentified safety issues. Both Watch List and non-Watch List tanks will be sampled and evaluated to identify safety conditions related to the ferrocyanide, organic, flammable gas, and criticality safety issues. Safety screening for high heat tanks has already been completed and the vapor safety issue will be handled by ongoing industrial hygiene programs.

With respect to sampling specifications, this DQO requires that "a vertical profile of the waste be obtained from at least two widely-spaced risers assuming the quality of the data obtained supports appropriate safety classification of the tank. Such sampling can be done by core drilling, auger sampling (for shallow tanks) and/or by obtaining liquid supernate samples at several levels" (Redus and Babad 1994). Tank AX-104 contains a shallow depth, high-solids waste that will be sampled for safety screening purposes using the auger sampling method.

The analytical requirements for the safety screening of a tank are concerned with identifying a common set of primary analytes and characteristics of the waste. These analytes are energetics, total alpha activity, moisture and flammable gas concentrations. If a specific criteria level on one of these items is exceeded further analysis of a secondary set of analytes and a possible Watch-List tank classification would be warranted. Further details of the analytical procedures and criteria to be followed for this tank AX-104 sampling and characterization event are given in Section 3.2.

This Tank Characterization Plan identifies procedures and requirements, in accordance with the safety screening DQO and the Characterization Program, for collecting and characterizing a sample from tank AX-104 by the auger sampling method. The dome-space gas flammability determination is being handled separately by the Tank Vapor Issue Resolution Program.

## 1.2 Relevant Safety Information

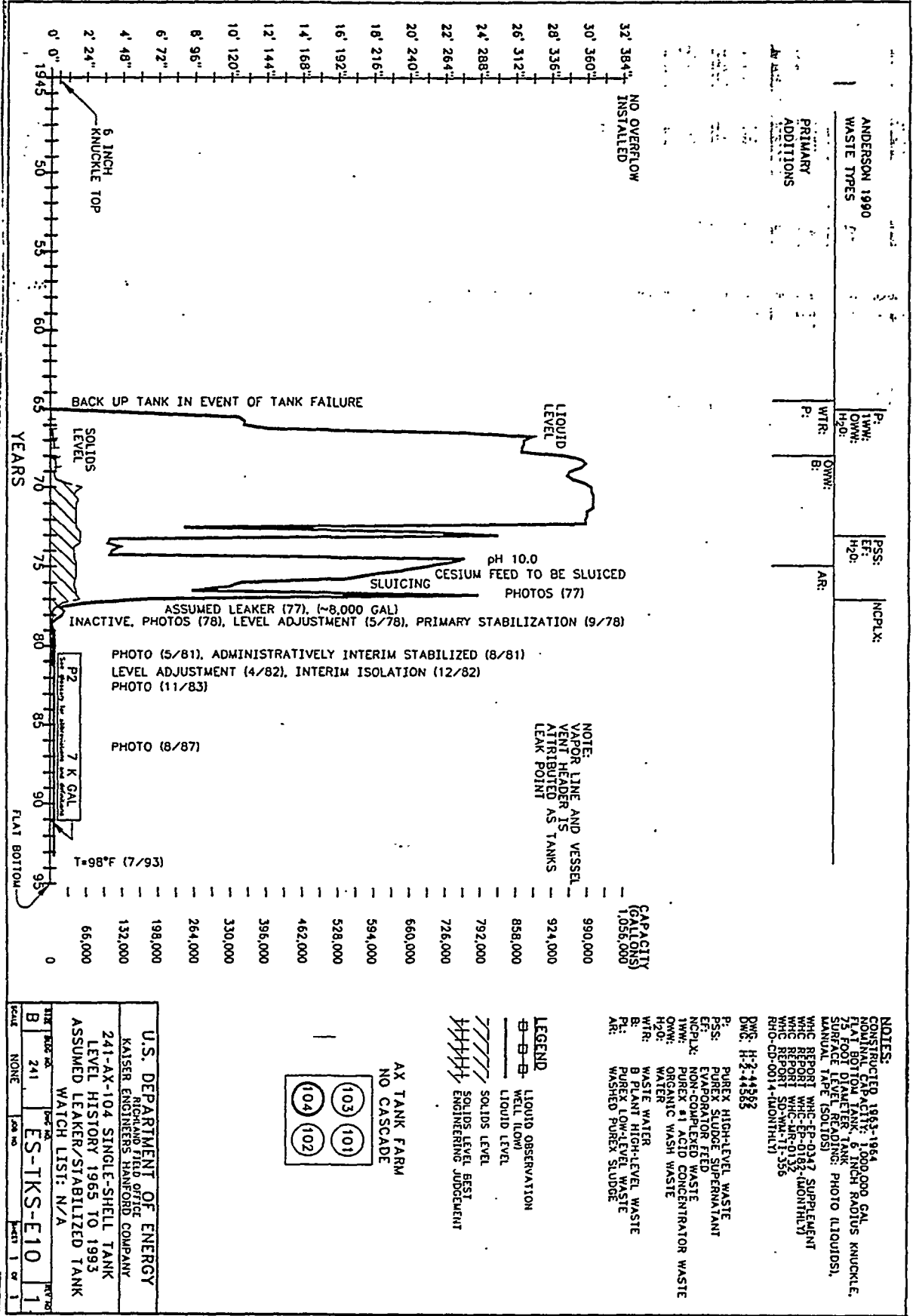
### 1.2.1 Tank Status

Single-shell Tank AX-104 is classified as a Non-Watch List low heat load tank (<40,000 Btu/hr). Tank AX-104 is an assumed leaker, interim stabilized (8/81). To prevent further waste additions to the tank, it has been isolated (1982) and designated as an Intrusion Prevention tank. Its present status is Inactive-Interim Stabilized.

Tank AX-104 is estimated to contain approximately 7 kgal of sludge waste (3 inches) and no pumpable liquid remaining; its contents are categorized as non-complexed waste material (NCPLX) (Hanlon 1994). Recent data (April 1994) obtained from the Computer Automated Surveillance System (CASS) database indicate a waste depth of 3.5 inches from the manual tape in riser 9A.

### 1.2.2 Tank Monitoring Activities

The temperature of the waste in tank AX-104 was recorded at 92°F, in January of 1994, from a thermocouple tree which resides in riser 9C. A manual tape in riser 9A monitors the waste surface level. Four out of seven active dry wells monitor radiation in surrounding soil (Hanlon 1994). An active dry well is defined as one having readings greater than the background radiation of 50 counts/sec.



**U.S. DEPARTMENT OF ENERGY**  
 KAISER ENGINEERS HANFORD COMPANY  
 RICHLAND FIELD OFFICE  
 241-AX-104 SINGLE-SHELL TANK  
 LEVEL HISTORY 1965 TO 1993  
 ASSUMED LEAKER/STABILIZED TANK  
 WATCH LIST: N/A

REVISED: 241  
 NONE  
 ES-TKS-E10  
 1

**NOTES:**  
 CONSTRUCTED 1963-1964  
 1000,000 GAL  
 75 FOOT DIAMETER TANK  
 SURFACE LEVEL READING: PHOTO (LIQUIDS),  
 MANUAL TAPE (SOLIDS)  
 WHC REPORT WHC-EP-047 SUPPLEMENT  
 WHC REPORT WHC-EP-0183 (MONTHLY)  
 WHC REPORT WHC-EP-0132  
 WHC REPORT WHC-EP-0132  
 RHO-CD-0014 (MONTHLY)  
 DWG: H-2-43583

P: PUREX HIGH-LEVEL WASTE  
 PSS: PUREX SLUDGE SUPERNATANT  
 EF: EVAPORATOR FEED  
 NCPLX: NON-COMPLEXED WASTE  
 TWW: PUREX #1 ACID CONCENTRATOR WASTE  
 OMW: ORGANIC WASH WASTE  
 H2O: WATER  
 WTR: WASTE WATER  
 B: B PLANT HIGH-LEVEL WASTE  
 PL: PUREX LOW-LEVEL WASTE  
 AR: WASHED PUREX SLUDGE

**LEGEND**  
 — LIQUID OBSERVATION  
 — LIQUID LEVEL  
 — WELL (LOW)  
 — SOLIDS LEVEL  
 — SOLIDS LEVEL BEST ENGINEERING JUDGMENT

AX TANK FARM  
 NO CASCADE

103 101  
 104 102

**NOTE:**  
 VAPOR LINE AND VESSEL  
 VENT HEADER IS  
 ATTRIBUTED AS TANKS  
 LEAK POINT

PHOTO (8/87)

T=98°F (7/93)

7 K GAL

PHOTO (5/81), ADMINISTRATIVELY INTERIM STABILIZED (8/81)  
 LEVEL ADJUSTMENT (4/82), INTERIM ISOLATION (12/82)  
 PHOTO (11/83)

ASSUMED LEAKER (77), (~8,000 GAL)  
 INACTIVE, PHOTOS (78), LEVEL ADJUSTMENT (5/78), PRIMARY STABILIZATION (9/78)

SLUICING CESIUM FEED TO BE SLUICED  
 PHOTOS (77)

PH 10.0

BACK UP TANK IN EVENT OF TANK FAILURE

6 INCH KNUCKLE TOP

NO OVERFLOW  
 INSTALLED

WTR: P  
 OWNR: H-2-B  
 AR: P  
 B: B  
 AR: P

ANDERSON 1990  
 WASTE TYPES  
 PRIMARY ADDITIONS

NCPLX: P  
 EF: H-2-B  
 PSS: H-2-B

CAPACITY (GALLONS)  
 1,055,000

990,000  
 924,000  
 856,000  
 792,000  
 726,000  
 660,000  
 594,000  
 528,000  
 462,000  
 396,000  
 330,000  
 264,000  
 198,000  
 132,000  
 66,000  
 0

0' 0" 1943  
 2' 24" 50  
 4' 48" 55  
 6' 72" 60  
 8' 96" 65  
 10' 120" 70  
 12' 144" 75  
 14' 168" 80  
 16' 192" 85  
 18' 216" 90  
 20' 240" 95  
 22' 264" 98  
 24' 288" 99  
 26' 312" 99  
 28' 336" 99  
 30' 360" 99  
 32' 384" 99

0 50 60 65 70 75 80 85 90 95

YEARS

FLAT BOTTOM

Figure 1: Process History of Tank 241-AX-104

## 2.0 SUMMARY OF HISTORICAL INFORMATION FOR TANK AX-104

Included in this section are descriptions of tank AX-104, its process history, and recorded sampling events.

### 2.1 Configuration

Tank AX-104 is a single-shell tank in the 200 East area AX Farm constructed during 1964-65. AX Farm has four tanks of 1,000,000 gallons each. Tank AX-104, is a 75 feet diameter flat bottom tank having total capacity of 1,000,000 gallons. It went into service in 1965 as a back-up tank in the event of a tank failure. Tanks in 241-AX farm do not cascade.

### 2.2 Process History

Tank AX-104 began receiving neutralized PUREX plant acid waste in the third quarter of 1965. The tank continued to receive this waste type through 1967. From 1967 through 1969 it received high level bottom waste from No. 1 acid concentrator at PUREX. In 1970 supernate was transferred to A-101 and water was added to the tank. From 1976 through 1978 the tank received evaporator feed. Cesium recovery feed was sluiced from the tank in the third quarter of 1976. The tank was sluiced for strontium and cesium recovery during the third quarter of 1977. In addition, the tank was declared an assumed leaker in 1977 after an estimated fluid loss of 8,000 gal. The leak, attributed to a vapor line and vessel vent header, was stabilized after a level adjustment was made during May 1978. The tank was primary stabilized in September 1978. Presently, the tank waste is classified as non-complexed.

A level adjustment was made in April 1984. Interim isolation and interim stabilization was complete in December 1982. In-tank photographs were taken during the following dates: several in 1977, several in 1978, May 1981, November 1983, August 1987. A pH of 10.0 was measured in May 1975.

### 2.3 Historical Sampling Events

Receipt and analysis of two supernatant samples from AX-104 between July 1974 and September 1977 were identified in historical records (Horton, 1974; J. L. Starr, 1977). However, the methods and risers used during the sample collection were not identified.

#### 2.3.1 Sample Analytical Results

Table 1 presents the physical data and analyte concentrations from the 1974 and 1977 supernatant samples. Both of these samples were taken before the primary stabilization in 1978.

Table 1a. Analytical Results for Tank AX-104 Waste Samples.

July 10, 1974	
Sample Description	Supernatant
Bulk Density	Wet= 1.609 Dry = 0.898
Chemical Analysis	
H <sub>2</sub> O	44.4%
Fe <sub>2</sub> O <sub>3</sub>	33.2%
SiO <sub>2</sub>	2.9%
Al <sub>2</sub> O <sub>3</sub>	6.0%
CuO	0.6%
MgO	0.1%
Radiological Analysis	
Sr <sup>89+90</sup>	93.5 Ci/l
Co 60	0.15 Ci/l
Ru 106, Rh106	7.83 Ci/l
Sb125	2.37 Ci/l
Cs137	2.02 Ci/l
Eu154	0.51 Ci/l
Pu239	0.14 g/l

Table 1b: Analytical Results for Tank AX-104 Waste Samples

October 3, 1977		
Composition of 241-AX-104 sample #25		
Sample Volume	0.7 ml	
Dil. Vol	265 ml	
D.F	379 ml	
Damp Bulk Density	1.8 g/ml	
Particle Density	1.7 g/ml	
H2O	41 %	
Chemical Analysis	M	ug/l
Fe	5.95	184,450
Al	3.72	55,593
Ni	0.26	8,478
Mn	0.078	2,379
Mg	0.183	2,470
Cr	0.059	1,701
Hg	1.44	160,400
Ca	0.63	14,000
Cd	0.015	936.6
Si	3.17	49,311
Ba	0.02	1,522
Bi	NR	
Na	2.96	37,657.7
NO3	<1.14	
PO4	0.110	5,805.5
SO4	<1.14	
NO2	NR	
Chromatographic method checks	M	ug/l
SO4	0.125	6,666.6
Radiological Analysis		uCi/g
Sr	69.7 Ci/l	3,392,066
U	4.25E-03 g/l	0.146
Pu	0.15 g/l	5.16
At	4.89E+07 d/m/ml	
Cs	1.60 Ci/l	118,133
Eu 155	0.68 Ci/l	61,200
Sb 125	0.53 Ci/l	35,833.8

### 3.0 SCHEDULED SAMPLING EVENT

#### 3.1 Sample Collection and Transport

In fiscal year 1994, auger samples from tank AX-104 will be taken from risers 3A and 9E. Two inches of sludge (approximately 80 g) are expected to be recovered from each auger sample. No drainable liquids are expected to be recovered; however, some liquids may be contained in the waste. A field/trip blank is not required since this sampling event is not in support of any regulatory issues (Zuroff 1994). For specific information concerning sample handling, custody, and transport refer to quality assurance/quality control requirements in Section 4.1. Documents which contain applicable operating procedures and chain of custody requirements for the AX-104 auger sampling activities are procedure T0-080-500, and work package 2E-94-00702.

#### 3.2 Tank-Specific Analytical Procedures

Any decisions, observations, or deviations to this characterization plan made during sample receipt, isolation, and analysis will be documented in the deliverable report.

##### 3.2.1 Hot Cell Sample Isolation and Analysis Scheme

Before sample removal and breakdown begins, H. Babad or another delegate of the Characterization Program should be contacted so that they may be present to observe the sample breakdown. Because of the limited turn-around time for the safety screening analyses, the laboratory will not delay sample breakdown more than 4 hours to accommodate program observers. The sample isolation and breakdown procedure will follow guidelines provided in this Tank Characterization Plan.

A flowchart showing the general safety screening sample breakdown and analysis scheme is presented as Figure 2. Both auger samples are to be prepared and analyzed in accordance with this scheme, assuming adequate sample exists to perform all the analyses. These analysis requirements are prioritized (see section 3.2.2) and should be performed accordingly if insufficient sample is recovered. If necessary, the laboratory may make modifications to this breakdown. Sample receipt, custody, preparation, and analysis shall be performed in accordance with the approved procedures.

- Step 1 Receive auger samples at the performing laboratory.
- Step 2 Isolate and break down auger samples. Record visual observations of physical properties of the sample such as sample color, texture, homogeneity, and consistency. Note the color and clarity of any drainable liquid. Take a color photograph or videotape the isolated samples.
- Step 3A If the auger contains any drainable liquid, then proceed to Step 3B. If no drainable liquid is present go to Step 4A.
- Step 3B Separate the drainable liquid from the solids by allowing the liquid to drain into a bottle. Measure and record the volume of liquid. Retain the liquids for further analyses.

- Step 3C Immediately report any visually observed potential organic layers by the early notification system (Format I). Separate and retain the potential organic layer until all analyses are complete. Proceed to Step 4A.
- Step 4A Inspect the top portion of the auger sample for a hard, dry layer. If present continue to step 4B, otherwise go to step 5.
- Step 4B Separate the hard, dry layer and retain for analysis. Go to Step 5.
- Step 5 Separate remaining auger sample into two equal subsamples.
- Step 6 Homogenize each subsample individually.
- Step 7 Filter the aqueous sample through a 0.45 micron filter. Retain liquid subsample for safety screening analyses.
- Step 8 Remove aliquots from each homogenized subsample obtained in Steps 3A through 5. Retain enough sample to perform each analysis in Table 2 except RSST adiabatic calorimetry. Secure and retain any remaining subsamples until analyses are complete.
- Step 9 Perform DSC/TGA analysis on each undried solid subsample and liquid sample in duplicate. Perform total alpha analysis on a fusion dissolution of each solid aliquot in duplicate; total alpha analysis is not required on the liquid aliquot.
- Step 10A Analyze DSC/TGA scans and total alpha results. Do the DSC/TGA results exceed the established safety criteria limits? Are the total alpha results exceed the established safety criteria limits (see Table 2, Notification Limits)? If the answer is no for either question, go to step 10B. If yes to both questions, then proceed to Step 11.
- Step 10B Report results exceeding notification limits as described in the format I reporting deliverable requirements (see Section 7.1). Proceed to the following steps (10C, etc...).
- Step 10C Perform analyses for secondary analytes.
  - If the DSC safety criteria limit was exceeded, perform micro cyanide distillation and hot persulfate TOC analyses on each solid and liquid aliquot in duplicate. In addition, perform RSST adiabatic calorimetry analysis.<sup>1</sup>
  - If the total alpha safety criteria limit was exceeded, determine radiological activity due to the presence of <sup>239/240</sup>Pu on each solid and liquid aliquot in duplicate. Also, determine Iron, Manganese, and Uranium concentrations by Inductively Coupled Plasma (ICP) analysis.

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<sup>1</sup> RSST, if necessary, shall be performed at the 222-S Laboratory from an archived subsample which shows a DSC exotherm.



- Step 10D Are the results of analyses for secondary analytes within safety criteria limits? If not go to step 10E, if the answer is yes proceed to step 11.
- Step 10E Report results for the secondary analytes exceeding notification limits as described in the format I reporting deliverable requirements (see Section 7.1).
- Step 11 Archive remaining sample. Any subsamples remaining from crust/sludge/liquid separations should be archived in separate containers (Bratzel 1994).
- Step 12 Deliver a Format III Report that characterizes the results of the safety screening for primary analytes within 45 days of receipt of the last sample at the laboratory loading dock. Follow the Format III Reporting requirements provided in Section 7.2.
- Step 13 Following the same reporting requirements, deliver a Format III Report that characterizes the results of the safety screening for secondary analytes within 90 days of receipt of the last sample at the laboratory loading dock (required only if any primary analytes exceeded notification limits).

### 3.2.2 Prioritization of Requested Analyses

A list of analyses to be performed for tank safety screening is provided in preferential order in Table 2. However, if the amount of sample recovered is found to be insufficient to perform each analyses in Table 2, Characterization Support and Analytical Services shall be notified (for points of contact, see Section 5.0, Table 3). Confirmation of the prioritization levels or revision of sample breakdown procedures will be provided based upon the sample recovery, readily observable physical property distinctions within the sample, and the requested sample breakdown steps provided in section 3.2.1.

#### PRIORITY LEVEL 1

First, DSC and TGA analyses will be performed to determine a fuel energy value and weight percent moisture. Second, total alpha activity will be determined on a fusion preparation.

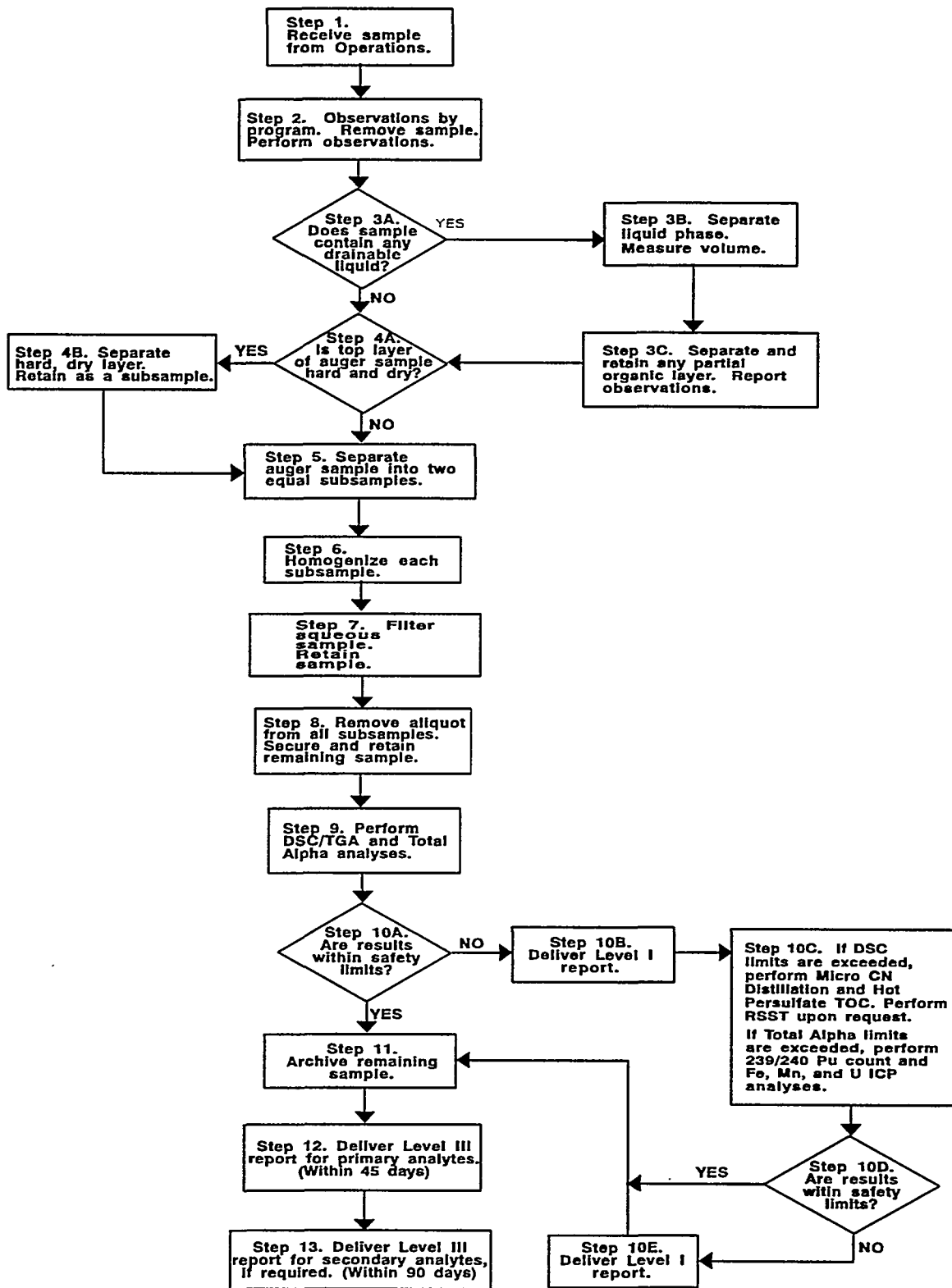
#### PRIORITY LEVEL 2

If the net energetics of the tank waste from the thermal analysis are greater than 125 cal/g on a dry weight basis, or if the alpha activity exceeds 41  $\mu\text{Ci/g}$  (see Section 6.2 for unit conversion guidelines), further analyses to determine concentration levels of total organic carbon (TOC), cyanide (CN), Plutonium 239/240 ( $^{239/240}\text{Pu}$ ), Iron (Fe), Manganese (Mn), and total uranium (U) will be done.

#### PRIORITY LEVEL 3

If the DSC shows that a net exotherm greater than or equal to 125 cal/g (on a dry weight basis) exists in tank AX-104, and adequate sample is available, personnel at the 222-S Laboratory are directed to perform an adiabatic calorimetry analysis using the Reactive System Screening Tool (RSST) method to characterize the exothermic behavior.

Figure 2: Test Plan Flowchart for Tank Safety Screening



**3.2.3 Analytical Methods**

Table 2 summarizes the analyses to be performed as well as the corresponding laboratory procedure numbers. All analyses, except RSST adiabatic calorimetry, are requested to be performed in duplicate at the performing laboratory. If the performing laboratory is not the 222-S Laboratory, arrangements shall be made by the performing laboratory for transport of one or more waste samples to the 222-S Laboratory for RSST analysis.

These analyses are based on the *Tank Safety Screening DQO* (Redus and Babad 1994). None of the analyses are being used to support regulatory compliance; therefore, regulatory methods are not required.

Any analyses prescribed by this document, but not performed, shall be identified, with justification for non-performance, in the appropriate data report. Also, in this situation, Characterization Support and Analytical Services personnel shall be contacted (Section 5.0, Table 3).

**4.0 QUALITY ASSURANCE/QUALITY CONTROL**

**4.1 Sampling Operations**

Auger samples are to be taken and shipped to the performing laboratory by Sampling Operations in accordance with the Work Package 2E-94-00702. That work package shall also initiate the chain-of-custody form. Approved operating procedures for obtaining an auger sample are provided in procedure TO-080-500 and cask loading and transport procedure TO-080-090.

When a decision is made to perform the sampling activity, Analytical Services Program Management and Integration will designate the performing laboratory based on availability and capacity. The sampling and transportation documentation shall be revised to identify the assignment of the performing laboratory.

The auger samples are to be shipped in a cask and sealed with a waste tank sample seal. All sample shipments are identified by a unique number and labeled with the following information:

**WASTE TANK SAMPLE SEAL**

Supervisor	Sample No.
Date of Sampling	Time of Sampling
Shipment No.	Serial No.

Sampling Operations shall send the samples to the laboratory within one working day, if possible, of removing the sample from the tank, but must send the samples within three working days of removal from the tank. Sampling Operations is responsible for verbally notifying the project coordinator at the 222-S Laboratory at least 24 hours in advance of an expected shipment. If the samples are sent to 325 Analytical Chemistry Laboratory (ACL), sampling operations shall notify the project manager at least 72 hours in advance to the expected shipment.

The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook in addition to recording this information in the comment section of the chain-of-custody form.

#### 4.2 Laboratory Operations

The sample receipt and control procedures used in the Pacific Northwest Laboratories 325 ACL are reported in the *Quality Assurance Plan for Activity Conducted by the Analytical Chemistry Laboratory (ACL)*, (Kuhl-Klinger 1994). Procedures used in the Westinghouse Hanford 222-S Laboratory are described in LO-090-101.

Analyses and quality assurance/quality control performed at the 325 ACL shall be guided by the 325 Laboratory Quality Assurance Project Plan (Kuhl-Klinger 1994). The 222-S Laboratory has a quality assurance program plan (Mezmarich 1994) and a quality assurance project plan (Taylor 1993) that shall provide the quality assurance/quality control requirements for analyzing the tank waste samples.

Method specific quality control such as calibrations and blanks are also found in the analytical procedures. Sample quality control (duplicates, spikes, standards) are identified in Table 2. If no criteria are provided in Table 2, the performing laboratory shall perform to its quality assurance plan(s).

WHC-SD-WM-TP-243, REV. 0

Table 2. AX-104 Auger Sample Chemical, Physical, and Radiological Analytical Requirements

Project Name	AX-104 Auger	CHARGE CODES	ORGANIZATION	COMMENTS	REPORTING FORMATS	
Plan Number	WHC-SD-WM-TP-243	ED4599 (PNL)	Pacific Northwest Laboratory	Homogenization Test - Not Required Field Blank - Not Required Hot Cell Blank - Not Required	FORMAT I	Early Notify
Tank	AX-104	TBD (WHC)	Laboratory Engineering		FORMAT II	Process Control
2 Augers	Augers No. 1 and 2	TBD (WHC)	Program Support		FORMAT III	Safety Screen
Program Contact	H. Babad	TBD (WHC)	Laboratory Data Management		FORMAT IV	Waste Management
TWRS Contact	P. Sathyanarayana	TBD (WHC)	Sample Data and Administration		FORMAT V	RCRA Compliance
Lab. Project Coordinator	S. G. McKinley (PNL) J.G. Kristofzski (WHC)	TBD (WHC)	Shift Relief/Data Handling		FORMAT VI	Special

ANALYSES					PREPARATION				QUALITY CONTROL <sup>1</sup>				CRITERIA				REPORT FORMAT
METHOD	ANALYTE	WHC PROCEDURE	PNL PROCEDURE	UNITS	ACID	FUS	H <sub>2</sub> O	OTHER	DUP	SPK/MSD <sup>2</sup>	BLANK	STD	PRECISION	ACCURACY	NOTIFICATION LIMIT <sup>3</sup>	EXPECTED RANGE	
DSC	energy	LA-514-113	PNL-ALO-508	cal/g				direct	ea smpl	N/A	N/A	ea AB	≤10%	90-110%	>net 125 cal/g (dry)	N/A	I, III
TGA	% water	LA-560-112	PNL-ALO-508	%				direct	ea smpl	N/A	N/A	ea AB	≤10%	90-110%	<17 wt%	41 wt%	I, III
Alpha Det.	Total α	LA-508-101	PNL-ALO-421 PNL-ALO-423	μCi/g		X			ea smpl	1/ mtrx	ea PB	ea AB	≤10%	90-110%	>41 μCi/g <sup>4</sup>	N/A	I, III
RSST <sup>5</sup>	energy	see <sup>4</sup>	N/A	cal/g				direct	N/A	N/A	N/A	ea AB	≤10%	90-110%	>net 125 cal/g (dry)	N/A	I, III
Microdist. <sup>6</sup>	CH	LA-695-102	PNL-ALO-270 PNL-ALO-285	μg/g				direct	ea smpl	1/ mtrx	ea AB	ea AB	≤10%	90-110%	>31,000 μg/g (dry)	N/A	I, III
Hot Persulfate <sup>7</sup>	TOC	LA-342-100	PNL-ALO-381	μg/g				direct	ea smpl	1/ mtrx	ea AB	ea AB	≤10%	90-110%	>30,000 μg/g	N/A	I, III
Sep & α count <sup>8</sup>	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422	μCi/g		X			ea smpl	1/ mtrx <sup>4</sup>	ea PB	ea AB	≤10%	90-110%	>41 μCi/g <sup>4</sup>	N/A	I, III
ICP <sup>9</sup>	Fe, Mn, U	LA-505-151	PNL-ALO-211	μg/g	X	X			ea smpl	1/ mtrx	ea PB	ea AB	≤10%	90-110%	none	1.84E+5Fe; 2.37E+3 Mn; 4.25E+3 U (all μg/g)	III

1 ea - each, DUP - duplicate, spk/msd - spike or matrix spike duplicate, PB - preparation batch, AB - analytical batch, NA - not applicable, mtrx - matrix, std - calibration standard.  
 2 These analyses are performed only if the DSC Measurement indicates an energy content greater than 125 cal/g (dry weight basis).  
 3 These analyses are performed only if total alpha exceeds 41 μCi/g.

4 The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104, Rev. 0. Tracer or carrier is used in place of a spike and results corrected for recovery.  
 5 See assumption in Section 6.2 to convert alpha detection results to curies.  
 6 Post-digestion spike, except for ICP acid digest, which shall have pre-digestion spikes.  
 7 If not specified, notification limits are on wet basis.

## 5.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank AX-104 safety screening project are listed in Table 3.

Table 3: Tank AX-104 Project Key Personnel List.

Individual	Organization	Responsibility
S. G. McKinley	325 Analytical Chemistry Laboratory	Project Manager for Tank Waste Characterization
J. G. Kristofzski	222-S Analytical Operations	Program Support Manager of Analytical Operations
P. Sathyanarayana	TWRS Characterization Support	AX-104 Tank Characterization Plan Cognizant Engineer
H. Babad	WHC Characterization Program	Characterization Program Point of Contact
J. L. Deichman	Analytical Services	Manager of Analytical Services Program Management and Integration
East Area Shift Operations Manager	Tank Farm Operations	East Tank Farm Point of Contact if Notification Limit is Exceeded (373-2689)

## 6.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

## 6.1 Exceptions to DQO Requirements

At this time no exceptions to the safety screening data quality objective, pertaining to this Tank Characterization Plan, have been identified.

## 6.2 Clarifications and Assumptions

A number of clarifications and assumptions relating to the notification limits or decision thresholds identified in the applicable DQO efforts need to be made with respect to the analyses in Table 2. Each of these issues are discussed below (Bell 1994).

- Any exotherm determined by differential scanning calorimetry (DSC) must be reported on a dry weight basis as shown in equation (1) using the weight percent water determined from thermal gravimetric analysis.

$$\text{Exotherm (dry wt)} = [\text{exotherm(wet wt)} * 100] / (100 - \% \text{ water}) \quad (1)$$

- Redus and Babad (1994) requires that additional analyses be performed if total  $\alpha$  activity measures greater than 1 g/L. Total  $\alpha$  activity is measured in  $\mu\text{Ci/mL}$  rather than g/L. To convert the notification limit for total  $\alpha$  activity into a number more readily usable by the laboratory, two assumptions and calculations are necessary. All alpha decay will be assumed to originate from Pu-239 and a sample density of 1.50 g/mL is assumed. The notification limit then may then be calculated as:

$$1 \text{ g/L limit} * 1 \text{ L/E+3 ml} * 1 \text{ mL/1.5 g} * 0.062 \text{ Ci/g} * \text{E+6 } \mu\text{Ci/Ci} = 41\mu\text{Ci/g}$$

- The safety screening DQO effort, upon which the analyses are based, does not address the analyses performed on any drainable liquid present. To adequately characterize the tank, all analyses performed on the solids will also be performed on any drainable liquids with the exception of total alpha activity. Results shall be reported by volume ( $\mu\text{g/mL}$ ).
- The DQO efforts also do not address field and hot cell blanks. Since none of the analyses are being performed for regulatory purposes or for trace amounts of analyte, no field or hot cell blanks are required.

## 7.0 DELIVERABLES

All analyses of tank waste material will be reported as format I or III, as shown in Table 2. The Characterization Program may request progress reports from the laboratory regarding the analyses. However, due to the rapid turn around time required for the safety screening analyses (the laboratories must report the results for primary analytes within 45 days), no special progress reports for this tank characterization project shall be required from the laboratory. All reports generated as part of normal operations (e.g., monthly reports) shall still be done. All data shall be reported by tank or by sample. The data shall be reported in the units given in Table 2. More information regarding reporting formats is found in (Schreiber 1994a).

### 7.1 Format I Reporting

Table 2 contains the notification limits for each analyte. Analytes that exceed the notification limits defined in the DQO processes shall be reported by calling the East Area Shift Manager of Tank Farm Operations at 373-2689 and the Characterization Program Office (Schreiber 1994b). This verbal communication must be followed within 1 working day by written communication to C. DeFigh-Price, D. R. Bratzel and H. Babad of the Characterization Program, P. Sathyanarayana and R. D. Schreiber of Characterization Support, J. L. Deichman of Analytical Services, and N. W. Kirch of Waste Tank Process Control, documenting the observation(s). A further review of the data, including quality control results and additional analyses for verification of the exceeded analyte, may be contracted between the performing laboratory and the contacts above.

## 7.2 Format III Reporting

A format III report, involving the results of the primary safety screen analysis, shall be issued to H. Babad, C. DeFigh-Price, D. R. Bratzel, R. D. Schreiber, and N. W. Kirch within 45 days of receipt of the last sample at the laboratory loading dock. Although normally raw data would not be attached to this type of report, the DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analysis for the secondary analytes are required, these results will be provided within 90 days of receipt of the last sample at the laboratory loading dock. No calibration data are requested for these reports. An electronic record of the summary tables shall also be sent to H. Babad. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

## 8.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Program. These changes shall be brought to the attention of the project manager and the Characterization Program as quickly as possible and documented accordingly. Changes may be documented through the use of analytical deviation reports or internal characterization change notices for minor, low-impact changes. All significant changes shall be documented by Characterization Support via an Engineering Change Notice to this Tank Characterization Plan. All changes shall also be clearly documented in the final data package.

Additional analysis of sample material from this characterization project at the request of the Characterization Program shall be performed according to a revision of this Tank Characterization Plan.



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