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J. A. Poppiti X

Los Alamos Technical Associates
750 Swift, Suite 14
Richland, WA 99352

A. T. Dicenso X
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Name: R. D. SCHREIBER

R.D. Schreiber 2/1/95
Signature

Organization/Charge Code 71520/N4168

7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, PNL 325 Analytical Chemistry Laboratory, and WHC 222-S Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of samples for tank 241-B-112.

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Tank 241-B-112 Tank Characterization Plan

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

by

Los Alamos Technical Associates
8633 Gage Boulevard
Kennewick, Washington 99336

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LIST OF ABBREVIATIONS

2C	2nd cycle decontamination-BiPO ₄ process
ACL	Analytical Chemistry Laboratory
B-112	Tank 241-B-112
BL	B-Plant low level waste
DOE	Department of Energy
DQO	data quality objective
DST	double shell tank
EB	evaporator bottoms
IP	intrusion prevention
IS	interim stabilized
IX	ion exchange waste
NCPLX	noncomplexed waste
RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
SST	single shell tank
TCP	Tank Characterization Plan
TOC	total organic carbon
TWRS	Tank Waste Remediation System
USQ	unreviewed safety question
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

The Defense Nuclear Facilities Safety Board has advised 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 the analytical sampling needs for the resolution of safety issues. As a result, a revision in the Federal Facility Agreement and Consent Order (Tri-Party Agreement) milestone M-44 has been made, which states that "A Tank Characterization Plan (TCP) will be developed for each double shell tank (DST) and single-shell tank (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 that requirement for tank 241-B-112 (B-112).

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK B-112

The sampling and analytical needs associated with the 177 single-shell and double-shell underground storage tanks have been identified through the DQO process. DQOs identify information needed by a program group in the Tank Waste Remediation System (TWRS) concerned with safety issues, regulatory requirements, or the transporting and processing of tank waste. Present characterization objectives for Fiscal Year 1995 involve the sampling of tanks to resolve safety issues. Other samples may be requested for ongoing waste processing and regulatory requirements in active tanks.

Tank B-112 is currently a non-Watch List tank; therefore, the only applicable DQO as of January 1995 is the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), which is described below.

2.2 SAFETY SCREENING DATA QUALITY OBJECTIVE

The *Tank Safety Screening Data Quality Objective* (Babad and Redus 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 classify the waste tanks into one of three categories (SAFE, CONDITIONALLY SAFE, or UNSAFE) following the guidelines for the four Watch List classifications and other safety issues. A tank can be removed from a Watch List if it is classified as SAFE. The safety screening DQO identifies the requirements used to determine which classification a tank belongs based on analyses that indicate if certain measures are within established parameters. The measures begin with the determination of the primary analyte concentrations which have been determined as indicators of potentially unsafe conditions within a tank. These primary analytical requirements for the safety screening of a tank are energetics, total alpha activity, moisture content, and flammable gas concentrations. If a specific criteria level for one of these indicators is exceeded, further analysis of a secondary set of analytes, resulting in a possible change in tank classification, is required.

To meet the sampling requirements of this DQO effort, a vertical profile of the waste shall be obtained from at least two widely-spaced risers. This vertical profile may be realized using core, auger, or grab samples. The safety screening

analyses shall be applied to all core samples, DST Resource Conservation and Recovery Act (RCRA) samples, and all auger samples, except auger samples taken exclusively to assess the flammable gas tank crust burn issue.

3.0 HISTORICAL TANK INFORMATION

This section summarizes some of the available historical information for tank B-112. Discussions of the process history, recent sampling events for the tank, and general information about the tank are included. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990).

3.1 CONFIGURATION OF TANK B-112

Single-shell tank B-112 was constructed in 1944. Tank B-112 is one of twelve tanks that comprise the B-Tank Farm which embodied the original tank design with a 75-ft diameter and an operating depth of 17-ft. The tank has a design capacity of 2,010 kL (530 kgal); however, safety considerations require a maximum operating capacity of 1,890 kL (500 kgal).

3.2 AGE AND PROCESS HISTORY OF TANK B-112

Tank B-112 began being filled in April 1946 with second-cycle waste, and was at full capacity in August 1946 when it began overflowing into a crib. In late 1952 cell 5-6 waste was introduced into the tank. The cribbing came to a halt in the first quarter of 1957 when the tank was contaminated with evaporator bottoms waste. In the fourth quarter of 1957, 1,870 kL (495 kgal) of waste was scavenged and sent to tank 241-C-101. This transfer lowered the waste level to 160 kL (43 kgal). This level was maintained until the fourth quarter of 1960, when tank B-112 level instruments indicated an unexplained decrease of 42 kL (11 kgal) of waste.

Between the fourth quarter of 1963 and the third quarter of 1968, the tank contained fission product waste from B-Plant. The tank waste level fluctuated between 401 kL (106 kgal) and 2,070 kL (547 kgal) during this period as waste was cascaded from tanks 241-B-110 and 241-B-111 and transferred to tank 241-AX-101. The tank received cell 23 concentrated feed in the fourth quarter of 1967.

From the fourth quarter of 1968 until the first quarter of 1969, the tank contained B-Plant low-level waste and evaporator bottoms waste. Beginning in the second quarter of 1969 tank B-112 began receiving evaporator bottoms waste and ion exchange waste from B-Plant, via the cascade, with transfers being made to tank 241-B-103.

Between the first quarter of 1973 and the second quarter of 1976, the tank contained evaporator bottoms waste. The evaporator bottoms waste consisted of the bottoms product and recycle from the in-tank solidification process in the BY-Tank Farm. The tank contained evaporator feed concentrate in the third and fourth quarters of 1976 and the first and second quarters of 1977.

The tank was inactive in 1978 but was declared an assumed leaker after leaking approximately 30 kL (8 kgal) of waste. The tank waste was classified as non-complexed in the second quarter of 1978 and has remained so to the present time.

Interim stabilization was completed after supernate pumping, and a level adjustment was made in May 1985. Intrusion Prevention was completed in June 1985. Figure 3-1 shows the supernate and solids waste levels within tank B-112 from 1946 to the present. Solid and supernate levels were taken on a quarterly basis as part of the overall surveillance effort in the tank farms. Zero on the vertical scale is at the knuckle bottom of the tank and the dish bottom is 0.3 m (1 ft). The solids level in the tank is indicated by the shaded area and the supernate level is indicated by the thick line above the shaded area.

3.3 EXPECTED TANK CONTENTS

Tank B-112 is expected to have three primary layers. A bottom layer of sludge consisting of second-cycle waste, followed by a layer of BY saltcake and a top layer of supernate. A summary of the waste composition in tank B-112 is reported in Table 3-1 (Brevick 1994). The data are an estimate of the solids composite inventory.

Table 3-1: Solids Composite Inventory Estimate for Single-Shell Tank B-112

Physical Property			
Analyte	Result	Analyte	Result
Density	1.41 g/mL	TOC	0.03 wt % C
Percent Water	48.8%		
Radiochemistry			
Analyte	Result ($\mu\text{Ci/g}$)	Analyte	Result ($\mu\text{Ci/g}$)
Plutonium	0.0704	Strontium	1.07
Cesium	142		
Chemical Constituents			
Analyte	Result ($\mu\text{g/g}$)	Analyte	Result ($\mu\text{g/g}$)
Aluminum	3,840	Fluoride	4,890
Bismuth	10,100	Nitrate	2.11E+05
Iron	6,520	Nitrite	4,700
Potassium	38.7	Phosphate	50,400
Sodium	1.44E+05	Silicate	5,040
Acetate	774	Sulfate	17,300
Chloride	219		

* Composite inventory excludes supernate, diatomaceous earth, and cement.

4.0 TANK B-112 SCHEDULED SAMPLING EVENTS

As of January 1995, only one sampling event of tank B-112 is scheduled: an auger sample. No other sampling is scheduled through Fiscal Year 1997 (Stanton 1994). The auger sampling shall be conducted in accordance with the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). Sampling and analytical requirements from this DQO are identified in Table 4-1. A more complete list of analytical requirements are given, as an appended revision, in the appropriate Sampling and Analysis Plan.

Table 4-1: Integrated DQO Requirements

Sampling Event	Applicable DQO	Sampling Requirements	Analytical Requirements
Auger	► Tank Safety Screening	Samples from 2 risers separated radially to the maximum extent possible	Energetics, Moisture, Total Alpha, Gas Composition

5.0 REFERENCES

Anderson, J. D., 1990, *A History of the 200 Area Tank Farms*, WHC-MR-0132, Westinghouse Hanford Company, Richland, Washington.

Brevick, C. H., 1994, *Historical Tank Content Estimate for the NE Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

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Hanlon, B. M., 1994, *Waste Tank Summary for Month Ending October 31, 1994*, WHC-EP-0182-79, Westinghouse Hanford Company, Richland, Washington.

Stanton, G. A., 1994, Letter to Distribution, "Baseline Sampling Schedule, Change 94-06", 74320-94-10, dated December 14, 1994.

APPENDIX A

**SAMPLING AND ANALYSIS PLAN FOR FISCAL YEAR
1995 AUGER SAMPLING**

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LIST OF ABBREVIATIONS FOR APPENDIX A

ACL	Analytical Chemistry Laboratory
B-112	Tank 241-B-112
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
DST	double shell tank
GEA	gamma energy analysis
HPGE/MCA	high purity germanium - multi channel analysis
IC	ion chromatography
ICP	inductively coupled plasma
NCPLX	non-complexed waste
PNL	Battelle Pacific Northwest Laboratory
RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
RSST	reactive system screening tool - adiabatic calorimetry
SST	single shell tank
TCP	Tank Characterization Plan
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
TWRS	Tank Waste Remediation System
USQ	unreviewed safety question
WHC	Westinghouse Hanford Company

A1.0 SPECIFIC TANK CHARACTERIZATION OBJECTIVES

This Sampling and Analysis Plan will identify characterization objectives pertaining to sample collection, hot cell sample breakdown, and laboratory analytical requirements in accordance with the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994).

The sampling criteria for safety screening require 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 classifications of the tank. Such sampling can be done by core sampling, by auger sampling (for shallow waste depths) and/or by obtaining liquid grab samples at several levels (Babad and Redus 1994). Tank B-112 shall be sampled for safety screening purposes using the auger sampling method. Two samples shall be taken from existing installed risers that are separated radially to the maximum extent possible (Section A2.0).

The analytical requirements for the safety screening of a tank specify sample analyses to be performed on half-segments and include the identification of the content of a common set of primary analytes and waste characteristics. These analytes are energetics, total alpha, % moisture, and flammable gas concentrations. If acceptance criteria limits are exceeded for these analytes, further analysis of a second set of analytes and a possible Watch List tank classification would be warranted. This Sampling and Analysis Plan identifies procedures and requirements, in accordance with the safety screening DQO and the Characterization Program, for collecting and characterizing samples from tank B-112 by the auger sampling method.

A2.0 TANK STATUS AND SAMPLING INFORMATION

A2.1 TANK STATUS

Tank B-112 currently contains 120 kL (33 kgal) of non-complexed waste. The waste consists of 110 kL (30 kgal) of sludge and 10 kL (3 kgal) of supernatant liquid (Hanlon 1994). However, according to the *Historical Tank Content Estimate for the NE Quadrant of the Hanford 200 East Area* states that the waste is comprised of 10 kL (3 kgal) of supernatant, 61 kL (16 kgal) of saltcake, and 53 kL (14 kgal) of sludge with no pumpable liquid remaining (Brevick 1994). This volume of waste corresponds to approximately 18.9 cm (7.5 in) of waste. The tank is identified as a low-heat load tank, is passively ventilated, and is categorized as an assumed leaker with interim stabilization and intrusion prevention completed. Tank B-112 is third in the three-tank cascade flow series with tank 241-B-110 and 241-B-111. Tank B-112 is not on any Watch Lists and there are no unreviewed safety questions (USQs) associated with the tank at this time.

A2.2 SAMPLING INFORMATION

Tank B-112 is currently scheduled to be sampled by the auger sampling method. Samples are expected to be taken from risers 3 and 7. If different risers are necessary to meet sampling and analysis requirements, this change must satisfy the intent of the applicable DQO and be recorded and approved by the sampling cognizant engineer prior to sampling. Risers used may be recorded on permanent data sheets or recorded directly in the work package. Sampling shall be conducted according to

procedures and documentation included in tank B-112 work package ES-95-00075. If the sampling depth is within ± 5 inches of the current depth information, one 20 inch auger sample is expected to be obtained from each riser. However, in the event that the current depth information is incorrect, a different sized auger bit may be used. The objective of the sampling event is to reach the bottom of the tank; therefore the number of samples might change depending on the depth of the waste.

A3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

A3.1 TANK-SPECIFIC ANALYTICAL PROCEDURES

A flowchart depicting the general safety screening sample breakdown and analysis scheme is presented in Figures A-1, A-2, and A-3. These steps are described in detail to provide the hot cell and laboratory chemists with guidance for the breakdown of the samples and may be altered as appropriate by the performing laboratory. Several analyses listed in Table A-1 require a 45 day reporting time, as noted. The 45 day reporting format, Format III, is explained in Section A7.3.

As a precautionary measure, the Safety and Analysis Report for Packaging (SARP) has been reviewed for any safety issues involved with transportation of B-112 auger samples. For B-112 auger samples, the transport sample casks must be vented every fifteen days to release retained gas.

Any decisions, observations, or deviations and justifications made to this work plan or during the sample breakdown shall be documented in writing. These decisions and observations shall also be reported in the data report. The reporting formats for analyses are contained in Table A-1.

- Step 1 Receive auger samples at the laboratory in accordance with approved procedures.
- Step 2 Conduct the following on the sample material from each isolated auger sample:
 - ▶ Perform a visual examination of the sample(s)
 - ▶ Record observations. This may include a sketch of the isolated auger sample in addition to written documentation of pertinent descriptive information such as color, texture, homogeneity, and consistency.
 - ▶ Note the color and clarity of any drainable liquid.
 - ▶ Report sample recovery results to the Safety Program within one working day of sample breakdown.
 - ▶ Take color photographs and/or a videotape to visually document the isolated auger samples.
- Step 3 Does the sample contain drainable liquid?
 - Yes: Proceed to Step 4A
 - No: Proceed to Step 5A

Step 4A Separate any drainable liquid from the solids. Measure and record the volume. Retain drainable liquids for further processing.

Step 4B Is the sample 100% drainable liquid?

Yes: Proceed to Step 11

No: Proceed to Step 5A

SOLIDS PATH

Step 5A Is there a hard, dry layer on the top of the auger sample?

Yes: Proceed to Step 5B

No: Proceed to Step 5C

Step 5B Separate the hard, dry layer and retain for analysis.

Step 5C Divide each auger sample into two equal subsegments (i.e., half segments).

Step 6 Homogenize each subsample using the appropriate, approved procedure.

Step 7 Will a homogenization test be performed?

Yes: Proceed to Step 8

No: Proceed to Step 9

NOTE: One subsample per core, at a minimum, should be used if a homogenization test is to be performed. Additional tests may be performed at the laboratory's discretion.

Step 8 Conduct the homogenization test by taking 1 to 2 g aliquot from widely separated locations of the homogenized subsample. Conduct the homogenization test in accordance with Bell (1993).

Step 9 Collect sufficient aliquots from each homogenized subsample to perform the appropriate preparations and analyses listed in Table A-1 in duplicate.

NOTE: If there is an insufficient amount of sample available in any subsample to perform all required analyses on the half segment, notify the Characterization Program within one business day and follow the prioritization of analyses given in Section A3.3.

Step 10 Remove at least 20 mL and up to 40 mL of each homogenized subsample for the archive sample (Bratzel 1994).

LIQUIDS PATH

Step 11 Closely inspect the liquid sample for the presence and approximate volume of any potential organic layers. Does the sample contain any immiscible (potentially organic) layers?

Yes: Proceed to Step 12A

No: Proceed to Step 13

Step 12A Report any visually observed immiscible (potential organic) layer immediately by the early notification system (see Section A7.2).

Step 12B Separate and retain the potential organic layer for possible future analysis.

NOTE: Steps 13 through 17 shall be performed on the remaining (probable aqueous) liquid layer only.

Step 13 Filter the remaining liquid sample through a 0.45 micron filter.

Step 14 Is there greater than 1 gram of solid on the filter?

Yes: Proceed to Step 15

No: Proceed to Step 16

Step 15 Archive the solids for possible future analysis (Bratzel 1994).

Step 16 Remove sufficient aliquots from the liquid sample to perform the appropriate analyses listed in Table A-1 in duplicate.

Step 17 Archive at least 20 mL and up to 40 mL of the drainable liquid as the liquid archive (Bratzel 1994).

PRIMARY ANALYSIS PATH

Step 18 Perform primary analyses as listed in Table A-1.

Step 19 Compare the primary analysis data with notification limits.

Step 20A Do the results exceed the notification limits (Table A-1)?

Yes: Proceed to Step 20B.

No: Proceed to Step 23.

Step 20B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section A7.2.

SECONDARY ANALYSIS PATH

Step 21 Perform secondary analyses according to Table A-1.

Step 22A Do the secondary analyses exceed the notification limits?

Yes: Proceed to Step 22B

No: Proceed to Step 23

Step 22B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section A7.2.

Step 23 Report results as listed in Section A7.0.

Figure A-1: Solid Analysis Flow Chart

LIQUID PATH

Figure A-2: Liquid Analysis Flow Chart

ANALYSIS AND REPORTING PATH

Figure A-3: Sample Analysis and Reporting Flow Chart

A3.2 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from the auger samples taken from tank B-112 is insufficient to perform the analyses requested and permit a minimum 10 mL archive per sample, the laboratory shall notify the Tank Cognizant Engineer and the manager of Analytical Services, Program Management and Integration within one working day. (See Table A-2). A prioritization of the analyses requested in this document is given in Section A3.3. Any analyses prescribed by this document, but not performed, shall be identified in the appropriate data report, with justification for non-performance.

A3.3 PRIORITIES OF REQUESTED ANALYSES

The analyses to be performed for the tank safety screening program has been prioritized below. Confirmation of prioritization levels or revision of sample breakdown procedures may be provided to the laboratory based upon the sample recovery, readily observable physical property distinctions within the sample, and the requested sample breakdown procedures provided in Section A3.1.

PRIORITY LEVEL 1

The DSC, TGA, and Total Alpha analyses shall be performed.

PRIORITY LEVEL 2

The cyanide, total organic carbon (TOC) by Hot Persulfate, RSST, ^{239/240}Pu, Fe, Mn, and U analyses shall be performed.

A4.0 SPECIFIC ANALYTE, QUALITY ASSURANCE, AND DATA CRITERIA

A4.1 SPECIFIC METHODS AND ANALYSES

The analyses in Table A-1 to be performed on the tank B-112 auger samples are based on the safety screening DQO referenced in Section A1.1.2. The laboratory procedure numbers, which shall be used for the analyses, are included in the table.

A4.2 QUALITY ASSURANCE

A4.2.1 Laboratory Operations

The 222-S Laboratory has a quality assurance program plan (Meznarich 1994) and a quality assurance project plan (Taylor 1993) that shall provide the primary direction for the quality assurance for analyzing the waste tank core samples at the 222-S Laboratory. If the analyses are performed at the 325 Analytical Chemistry Laboratory (ACL), the analysis shall be guided by the 325 Quality Assurance Plan (Kuhl-Klinger 1994). Additionally, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994), when implemented (August 31, 1995), shall be used as quality assurance guidance.

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 A-1. If no criteria are provided in Table A-1, the performing laboratory shall perform to its quality assurance plan(s).

A4.2.2 Sample Collection

Two auger samples are to be taken from tank B-112 and shipped to the performing laboratory by Sampling Operations in accordance with work package ES-95-00075. That work package shall also initiate the chain-of-custody for the samples. Approved auger sampling procedure T0-080-008 and procedure T0-080-090 ("Load/Transport Sample Cask(s)") is to be used during the sampling event. Samples shall be identified by a unique number before being shipped to the performing laboratory. The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook. Sampling Operations shall enter this information in the comment section of the chain-of-custody form for addition to the data reports.

Sampling Operations should transport each sample collected to the performing laboratory within 1 working day of removing the sample from the tank, but must transport each sample within 3 calendar days. Sampling Operations is responsible for verbally notifying the 222-S Laboratory (373-2435) at least 24 hours in advance of an expected shipment. If samples are going to be delivered to 325 ACL (376-2639), the laboratory shall be notified at least 72 hours in advance of actual sample shipment so that proper shift operations can be planned.

A4.2.3 Sample Custody

The chain-of-custody form is initiated by the sampling team as described in work package ES-95-00075. Core samples are shipped in a cask and sealed with a Waste Tank Sample Seal.

WASTE TANK SAMPLE SEAL	
Supervisor:	Sample No.:
Date of Sampling:	Time of Sampling:
Shipment No.:	Serial No.:

The sealed and labeled samples are shipped to the laboratory along with the chain-of-custody form. The receipt and control of samples in the WHC 222-S Laboratory are described in laboratory procedure LO-090-101. Receipt and control of samples in the PNL 325 ACL are described in procedure PNL-ALO-051.

Table A-1: B-112 Chemical, Radiological and Physical Analytical Requirements

SOLID ANALYSES																	
Project Name		B-112 Auger Sample				COMMENTS								REPORTING LEVELS			
Plan Number		WHC-SD-WM-TP-302, REV. 0				Homogenization Test - Per Laboratory Discretion Field Blank - Not Required Hot Cell Blank - Per Laboratory Discretion								FORMAT I		Early Notify	
														FORMAT II		Process Control	
PROGRAM		PROGRAM CONTACTS												FORMAT III		Safety Screen	
A. Safety Screening		Safety Screening		H. Babad		TANK B-112				#AUGERS 2				FORMAT IV		Waste Management	
		TWRS		R. D. Schreiber										FORMAT V		RCRA Compliance	
		222-S Laboratory		J. G. Kristofzski										FORMAT VI		Special	
		325 Laboratory		S. G. McKinley													
PROGRAM	PRIMARY ANALYSES				SAMPLE ¹		PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	½ SEG SLDG	½ SEG SC		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
A	DSC	Energy	LA-514-113	PNL-ALO-508	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g ⁵	> 481	unknown	I, III
A	TGA	% H ₂ O	LA-560-112	PNL-ALO-508	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	39 to 58	I, III
A	Alpha	Total Alpha	LA-508-101	PNL-ALO-420 PNL-ALO-421	X		f or a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III
PROGRAM	SECONDARY ANALYSES				SAMPLE ¹		PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	½ SEG SLDG	½ SEG SC		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
A	RSST ¹⁰	Energy	see 10 below	N/A	X	X	d	N/A	N/A	N/A	ea AB	±20	80-120	J/g ⁵	> 481	unknown	I, III
A	Distillation ¹⁰	CN	LA-695-102	PNL-ALO-287 PNL-ALO-289	X	X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III
A	Hot Persulfate ¹⁰	TOC	LA-342-100	PNL-ALO-381	X	X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg C/g	> 30,000	240 to 360	I, III
A	Sep. & α counting ¹¹	Pu-239/240	LA-503-156	PNL-ALO-422 PNL-ALO-423	X		f	ea smpl	1/mtrx ⁸	ea PB	ea AB	±10	90-110	µCi/g	> 41	0.056 to 0.084	I, III
A	ICP ¹¹	Fe Mn U	LA-505-151	PNL-ALO-211	X		f or a	ea smpl	see ⁹	ea PB	ea AB	±10	90-110	µg/g	none none none	5,220 to 7,820 unknown unknown	III III III

¹½ SEG SLDG-½ segment, sludge; ½ SEG SC-½ segment, saltcake²d-direct, f-fusion dissolution, a-acid dissolution, w-water dissolution³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, AB-analytical batch, PB-preparation blank, N/A-not applicable, mtr⁹-matrix⁴Units for notification limits and expected range are those listed in the "units" column.⁵Dry weight basis.⁶Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.⁸Tracer or carrier may be used in place of a spike and results corrected for recovery.⁹Either serial dilutions or matrix spikes will be performed.¹⁰This analysis required if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.¹¹Performed only if total alpha exceeds notification limit.

Table A-1: B-112 Chemical, Radiological and Physical Analytical Requirements

LIQUID ANALYSES																
Project Name		B-112 Auger Sample				COMMENTS						REPORTING LEVELS				
Plan Number		WHC-SD-WM-TP-302, REV. 0				Homogenization Test - Per Laboratory Discretion Field Blank - Not Required Hot Cell Blank - Per Laboratory Discretion						FORMAT I		Early Notify		
												FORMAT II		Process Control		
PROGRAM		PROGRAM CONTACTS										FORMAT III		Safety Screen		
A. Safety Screening		Safety Screening		H. Babad		TANK B-112						FORMAT IV		Waste Management		
		TWRS		R. D. Schreiber								FORMAT V		RCRA Compliance		
		222-S Laboratory		J. G. Kristofzski								FORMAT VI		Special		
		325 Laboratory		S. G. McKinley												
PROGRAM	PRIMARY ANALYSES				SAMPLE	PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID		DUP	SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
A	DSC	Energy	LA-514-113	PNL-ALO-508	X	d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g ⁵	> 481	unknown	I, III
A	TGA	% H ₂ O	LA-560-112	PNL-ALO-508	X	d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	39 to 58	I, III
A	Visual	Organic Layer	LA-519-151	PNL-ALO-501	X	d	N/A	N/A	N/A	N/A	N/A	N/A	none	presence	unknown	I, III
PROGRAM	SECONDARY ANALYSES				SAMPLE	PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID		DUP	SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
A	RSST ¹⁰	Energy	see 10 below	N/A	X	d ⁶	N/A	N/A	N/A	ea AB	±20	80-120	J/g ⁵	> 481	unknown	I, III
A	Distillation ¹⁰	CN	LA-695-102	PNL-ALO-287 PNL-ALO-289	X	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/mL	> 39,000 ⁷	unknown	I, III
A	Hot Persulfate ¹⁰	TOC	LA-342-100	PNL-ALO-381	X	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg C/mL	> 30,000 ⁷	(240 to 360) ⁷	I, III

²d-direct, f-fusion dissolution, a-acid dissolution, w-water dissolution³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, AB-analytical batch, PB-preparation blank, N/A-not applicable, mtrx-matrix⁴Units for notification limits and expected range are those listed in the "units" column.⁵Dry weight basis.⁶Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.⁷Converted from weight basis to a volumetric basis assuming a liquid density of 1.0 g/mL.⁸Tracer or carrier may be used in place of a spike and results corrected for recovery.⁹Either serial dilutions or matrix spikes will be performed.¹⁰This analysis required if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

A5.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank B-112 characterization project are listed in Table A-2. Procedures for both the WHC 222-S Laboratory and the PNL 325 Analytical Chemistry Laboratory are given in Table A-1 since it is as yet undecided which laboratory shall receive the samples from tank B-112. Analytical Services shall make the laboratory selection two weeks prior to the sampling event. The laboratory selection will be based on the ability of the laboratory to receive the samples as well as its ability to provide the required analytical data in the requested time. Once the performing laboratory is selected, Analytical Services shall send written notification to inform Sampling Operations of the laboratory to which the samples are to be sent.

Table A-2: Tank B-112 Project Key Personnel List

Individual	Organization	Responsibility
J. G. Kristofzski	222-S Analytical Operations	Program Support Manager of Analytical Operations
S. G. McKinley	325 Analytical Chemistry Laboratory	Project Manager for Tank Waste Characterization Projects
R. D. Schreiber	TWRS Characterization Support	Tank B-112 Tank Characterization Plan Cognizant Engineer
H. Babad	Characterization Program	Safety Screening Point of Contact
J. L. Deichman	Analytical Services	Manager of Analytical Services Program Management and Integration
East Tank Farm Operations Shift Manager	Tank Farm Operations	200 East Tank Farm Point of Contact if Action Limit is Exceeded (373-2689)

A6.0 EXCEPTIONS, CLARIFICATIONS AND ASSUMPTIONS**A6.1 EXCEPTIONS TO DQO REQUIREMENTS**

In the safety screening DQO (Babad and Redus 1994), it is specified that cyanide analyses are to be run on a quarter-segment level and that the notification limit for the DSC analysis is 125 cal/g. However, the revised ferrocyanide DQO (Meacham et al. 1994) has changed the requirements such that the cyanide analysis is now to be run on a half-segment level and the DSC notification limit is 115 cal/g (dry weight basis). This change shall be made to the safety screening DQO when it is revised. Therefore, although this Sampling and Analysis Plan uses the current safety screening DQO, it specifies that cyanide is to be run on a half-segment basis and that notification shall be made if the DSC value exceeds 481 J/g (115 cal/g dry weight basis).

A6.2 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 A-1. Each of these issues are discussed below.

- Any exotherm (in cal/g or J/g) 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 Thermogravimetric Analysis.

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

NOTE: A large error in the DSC value may result when converting samples containing greater than 90% water to a dry weight basis. However, this conversion is still required.

- The safety screening DQO (Babad and Redus 1994) requires that additional analyses be performed if total alpha activity measures greater than 1 g/L. Total alpha is measured in $\mu\text{Ci/g}$ rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it was assumed that all alpha decay originates from Pu-239. The notification limit may then be calculated as shown in equation (2):

$$\left(\frac{1 \text{ g}}{\text{L}} \right) \left(\frac{1 \text{ L}}{10^3 \text{ mL}} \right) \left(\frac{1}{\text{density}} \frac{\text{mL}}{\text{g}} \right) \left(\frac{0.062 \text{ Ci}}{1 \text{ g}} \right) \left(\frac{10^6 \mu\text{Ci}}{1 \text{ Ci}} \right) = \frac{61.5}{\text{density}} \frac{\mu\text{Ci}}{\text{g}} \quad (2)$$

NOTE: If a density of 1.5 g/mL is assumed for solid material, the notification limit becomes 41 $\mu\text{Ci/g}$.

- The safety screening DQO, upon which the analyses in Table A-1 are based, does not sufficiently address the analysis of any drainable liquid present. To adequately characterize the tank, all analyses performed on the solids for the safety screening DQO, with the exception of total alpha analyses, shall also be performed on any drainable liquids.

A7.0 DELIVERABLES

All analyses of tank B-112 waste material shall be reported as Formats I and/or III as indicated in Table A-1. Additional information regarding reporting formats is given in Schreiber (1994a).

A7.1 PROGRESS REPORTS

Each laboratory performing analyses on tank B-112 waste material from this sampling project shall provide monthly status reports to the Characterization Program. This report shall contain 1) a summary of the activities on the analysis

of tank B-112, 2) preliminary results to the program, and 3) schedule and cost information on a DQO basis.

Monthly and accumulative costs will be compared to the base as part of the progress report. Monthly variances greater than 10% and \$10,000, and accumulative variances greater than \$50,000 from the estimated costs or schedule must be explained in the report. Cost reporting shall consist of the following:

1. budgeted cost of work scheduled
2. monthly cost (actual cost of work performed)
3. year-to-date costs (actual cost of work performed)

Schedule reporting shall consist of the following:

1. monthly schedule
2. year-to-date schedule

A7.2 FORMAT I REPORTING

Table A-1 contains the notification limits for each analyte. Any results that exceed the notification limits defined in the DQO processes shall be reported by calling the East Tank Farm Operations Shift Manager at 373-2689 and the Characterization Program Office (Schreiber 1994b). This verbal notification must be followed within 1 working day by written communication to Analytical Services, Characterization Support, the Characterization Program Office, the Safety Screening Representative, and Waste Tanks Process Engineering, documenting the observations. Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above either by a revision to this document, or by a letter of instruction.

A7.3 FORMAT III REPORTING

A Format III report, reporting the results of the primary safety screen analyses, shall be issued to the Safety Screening Representative, Characterization Support, Waste Tanks Process Engineering, the Characterization Program Office, the Tank Characterization Resource Center, Los Alamos Technical Associates, and the Tank Characterization Database representative (Schreiber 1994c) within 45 days of receipt of the last sample at the laboratory loading dock. The DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analyses for the safety screening secondary analytes are required, these results shall be provided within 90 days of receipt of the last sample at the laboratory loading dock. No calibration data are requested for these reports. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

A8.0 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 documented. Changes may be documented through the use of internal characterization change notices or analytical deviation reports for minor low-impact changes and documented

in applicable laboratory notebooks. All significant changes (such as changes in scope) 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 report.

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.

A9.0 REFERENCES

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- Schreiber, R. D., 1994b, Letter to J. G. Kristofzski, "Format I Reporting Requirement", 7E720-94-128, dated August 15, 1994.
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