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1	1	Cog. Mgr. J.G. KRISTOFZSKI	[signature]	4/18/95		Central Files (2)			L8-04	3	
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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, and WHC 222-S Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of samples for tank 241-BX-103.

8. RELEASE STAMP

WHC-SD-WM-TP-339
Revision 0

Tank 241-BX-103
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

BX-103	Tank 241-BX-103
cm	centimeter
DOE	U.S. Department of Energy
DNFSB	Defense Nuclear Facilities Safety Board
DQO	data quality objective
DST	double-shell tank
HEPA	high-efficiency particulate air filter
PUREX	plutonium-uranium extraction waste
RCRA	*Resource Conservation and Recovery Act of 1976*
SAP	Sampling and Analysis Plan
SST	single-shell tank
TCP	Tank Characterization Plan
TOC	total organic carbon
TPA	Federal Facility Agreement and Consent Order (Tri-Party Agreement)
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

The Defense Nuclear Facilities Safety Board (DNFSB) has advised the U.S. Department of Energy (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 (Tri-Party Agreement or TPA) 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 (Ecology et al. 1994)." This document satisfies that requirement for tank 241-BX-103 (BX-103) sampling activities.

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK BX-103

The sampling and analytical needs associated with the Hanford Site underground storage tanks on one or more of the four Watch Lists (ferrocyanide, organic, flammable gas, and high heat) and the safety screening of all 177 tanks have been identified through the DQO process. A DQO identifies the information needed by a program group concerned with safety issues, regulatory requirements, tank waste processing, or the transport of tank waste. The DQOs that have been completed and are applicable to tank BX-103 are discussed in the following paragraphs.

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 waste tanks into one of three categories (SAFE, CONDITIONALLY SAFE, or UNSAFE). The safety screening DQO identifies the guidelines to determine to which category a tank belongs based on analyses that indicate if certain measurements are within established parameters. If a specified parameter is exceeded, further analysis of a second set of properties and a possible Watch List classification would be warranted. A tank can be removed from a Watch List if it is classified as SAFE. As of February 1995, tank BX-103 was classified as a non-Watch List tank.

The safety screening DQO requires that a vertical profile of the tank waste be obtained from at least two widely spaced risers. This vertical profile may be obtained using core, auger, or grab samples. The primary analytical requirements for the safety screening of a tank are energetics, total alpha activity, moisture content, and flammable gas concentration. These analyses shall be applied to all core samples, DST Resource Conservation and Recovery Act (RCRA) samples, and all auger samples, except those taken exclusively to assess the flammable gas crust burn issue.

3.0 TANK AND WASTE INFORMATION

This section summarizes the available historical information on tank BX-103. Included are the age of the tank, process history, and the expected contents of the tank based on historical information. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990).

3.1 1995 TANK STATUS

Tank BX-103 is not on a Watch List and is classified as sound. The tank is passively ventilated, interim stabilized, and intrusion prevention measures have been completed. Surface level monitoring is through riser #8 with a Food Instrument Corporation gauge. As of January 31, 1995, approximately 250,000 liters (66,000 gallons) of non-complexed waste was contained in the tank. The waste was comprised of 15,000 liters (4,000 gallons) of supernatant and 235,000 liters (62,000 gallons) of sludge with no drainable interstitial liquid present. The total volume of waste within the tank corresponds to a depth of 48 cm (19 inches). Thermocouple data for the tank produces a median temperature of 18° C (64° F) with a minimum of 15° C (59° F) and a maximum of 33° C (91° F). Current information indicates that the thermocouple tree is now out of service and the last temperature reading was in October 1992. An occurrence report was issued in March 1993 when the surface level exceeded the increase limit and an investigation to determine the cause of the increase is underway (Brevick et al. 1994).

3.2 TANK CONFIGURATION

Single-shell tank BX-103 was constructed between 1946 and 1947 and is located in the 200 East Area. Tank BX-103 is 23 meters (75 feet) in diameter and has a capacity of 2,006,000 liters (530,000 gallons). The tank is third in a cascade system consisting of tanks 241-BX-101, 241-BX-102 and 241-BX-103. A cascade system consists of tanks connected in series by pipes. When the primary tank in the system becomes full, the waste flows to the secondary tanks in the system. Tank BX-103 has 10 risers including the 31 cm (12 inch) riser #7.

3.3 TANK HISTORY

Tank BX-103 was filled with metal waste from September 1948 to January 1949 as part of a cascade system. From 1948 to 1977, when it was declared inactive, tank BX-103 received and transferred metal waste, tributyl phosphate waste, plutonium-uranium extraction (PUREX) wastes, organic wash waste, laboratory wastes, ion exchange waste, low level waste, evaporator bottoms waste, and N reactor waste. Waste was transferred to 11 and 12 BC Ditch, BY tanks, BX tanks, S tanks, SX tanks, and C tanks. Figure 3-1 shows the supernatant and solids waste levels of tank BX-103 from 1948 to the present (Anderson 1990). The solids and supernatant 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 and the dish bottom is 30 cm (1 foot) below the knuckle bottom. The solids level in the tank is indicated by the shaded area and the supernatant level is indicated by the thick solid line above the shaded area.

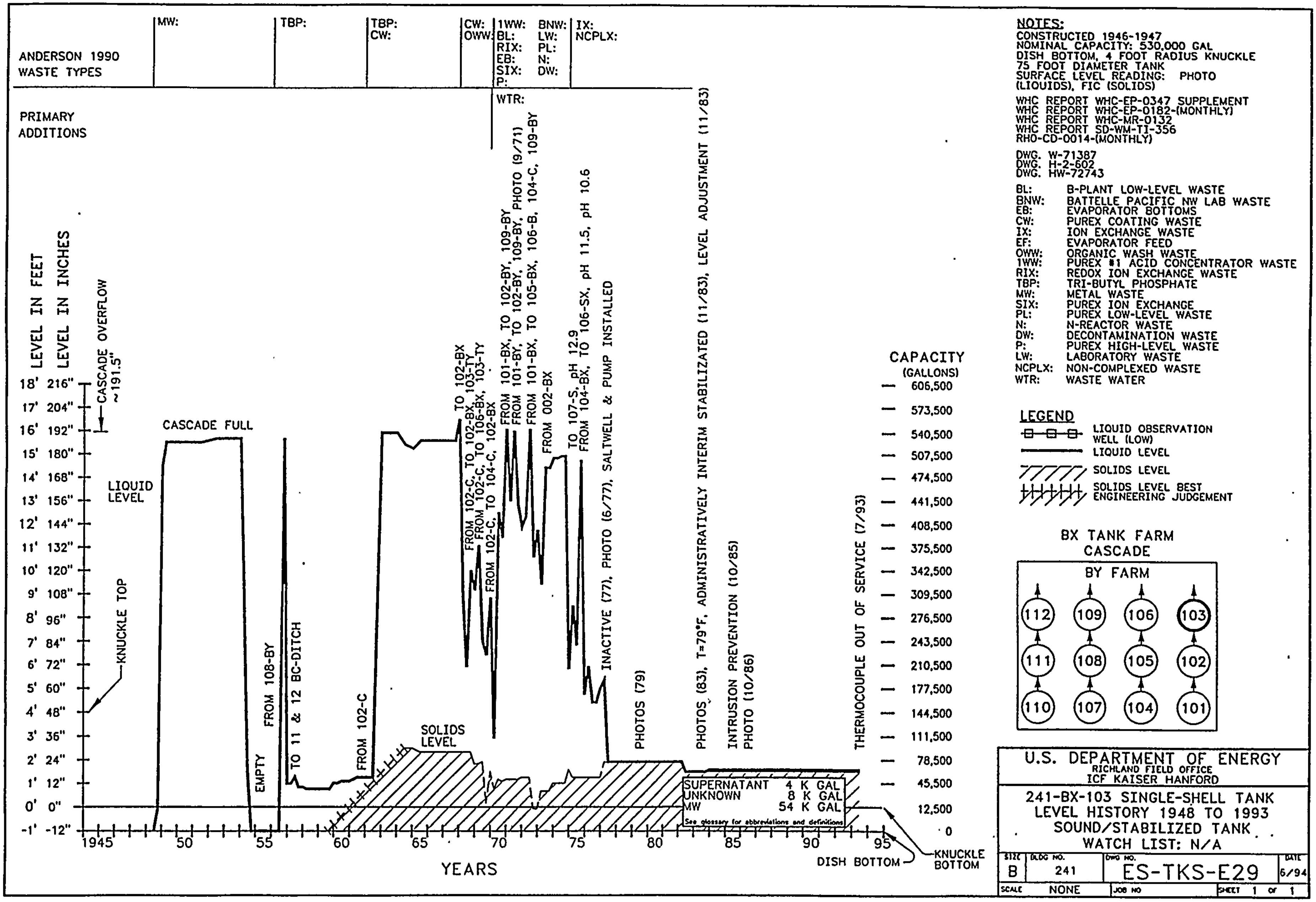
3.4 EXPECTED TANK CONTENTS

Tank BX-103 is expected to have two primary layers: a bottom sludge layer composed of metal waste and unknown waste, followed by a top layer of non-complexed supernatant. Records indicate that 57 tons of Portland cement was added to the tank (Brevick et al. 1994). The photograph shows a narrow ring of black sludge around the perimeter of the tank and most of the surface as a black liquid. Due to its poor quality, this photograph may not represent the tank's contents. An estimated inventory based on historical sample and analytical data is shown in Table 3-1 (Brevick et al. 1994). This estimate is only based on the 235,000 liters (62,000 gallons) of sludge in the tank.

Table 3-1: Solids Composite Inventory Estimate for Tank BX-103

Physical Properties			
Total Solid Waste	Mass = 3.93E+05 kg; Volume = 235 kL (62 kgal)		
Heat Load	0.45 kW (1.53E+03 BTU/hr)		
Bulk Density	1.67 (g/cm^3)		
Void Fraction	0.26		
Water wt%	48.49		
TOC wt% C (wet)	0.00		
Chemical Constituents	**M**	**μg/g**	**kg**
Na^+	1.05E+01	1.45E+05	5.68E+04
Al^{3+}	7.50E-01	1.20E+04	4.73E+03
OH^-	4.92E+00	5.00E+04	1.96E+04
NO_3^-	6.88E-02	2.55E+03	1.00E+03
NO_2^-	2.38E-02	6.55E+02	2.57E+02
CO_3^{2-}	2.05E+00	7.35E+04	2.89E+04
PO_4^{3-}	2.50E+00	1.42E+05	5.58E+04
SO_4^{2-}	3.73E-02	2.14E+03	8.41E+02
Radiological Constituents	**Ci/L**	**μCi/g**	**Ci**
Pu	n/a	4.40E-01	2.88E+00 (kg)
U	6.60E-01 (M)	9.33E+04 (μg/g)	3.67E+04 (kg)
Cs	2.73E-03	1.63E+00	6.41E+02
Sr	2.80E-01	1.69E+02	6.63E+04

Figure 3-1: Fill History of Tank 241-BX-103



4.0 TANK BX-103 SCHEDULED SAMPLING EVENT

Only one sampling event for tank BX-103 is currently scheduled: a push core sample in December 1995. However, an acceleration of, or changes to, the sampling schedule could lead to sampling at an earlier date. No other sampling is scheduled through Fiscal Year 1997 (Stanton 1995). Push core sampling shall be conducted following the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). Sampling and analytical requirements from this DQO are summarized in Table 4-1. A complete list of analytical requirements is given, as an appended attachment, in the appropriate Sampling and Analysis Plan (SAP).

Table 4-1: Integrated DQO Requirements

Sampling Event	Applicable DQO	Sampling Requirements	Analytical Requirements
Push Core Sampling	▸Safety Screening DQO	Samples from 2 risers separated radially to the maximum extent possible	▸Energetics ▸Moisture Content ▸Total Alpha

5.0 REFERENCES

Anderson, J. D., 1990, *A History of the 200 Area Tank Farms*, WHC-MR-0132, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Babad H. and K. S. Redus, 1994, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Brevick, C. H., L. A. Gaddis, and W. W. Pickett, 1994, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-WM-ER-349, Rev. 0, Kaiser Hanford Company, Richland, Washington.

Conway, J. T., 1993, Letter to H. R. O'Leary, DOE, "DNFSB Recommendation 93-5 to the Secretary of Energy," 9400070, dated July 19, 1993.

Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order, Fourth Amendment*, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

Stanton, G. A., 1995, Letter to Distribution, "Baseline Sampling Schedule, Change 95-03," 74320-95-04, dated March 24, 1995.

APPENDIX A

SAMPLING AND ANALYSIS PLAN FOR PUSH CORE SAMPLING IN FISCAL YEAR 1995

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LIST OF ABBREVIATIONS FOR APPENDIX A

ACL	Analytical Chemistry Laboratory
BX-103	Tank 241-BX-103
cm	centimeter
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
DST	double-shell tank
GEA	gamma energy analysis
HHF	hydrostatic head fluid
HPGE/MCA	high purity germanium - multi channel analysis
IC	ion chromatography
ICP	inductively coupled plasma
LiBr	lithium bromide
NCPLX	non-complexed waste
PNL	Battelle Pacific Northwest Laboratory
RCRA	*Resource Conservation and Recovery Act of 1976*
RSST	reactive system screening tool - adiabatic calorimetry
SAP	Sampling and Analysis Plan
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 TANK CHARACTERIZATION OBJECTIVES

This Sampling and Analysis Plan (SAP) will identify characterization objectives for sample collection, hot cell sample breakdown, and laboratory analytical requirements following the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994) as described in the Tank Characterization Plan (TCP) for tank 241-BX-103 (BX-103). This SAP will also identify procedures and requirements for collecting and characterizing samples from tank BX-103 by the core sampling method.

A2.0 TANK STATUS AND SAMPLING INFORMATION

A2.1 TANK STATUS

Tank BX-103 is not on a Watch List and is classified as sound. The tank is passively ventilated, interim stabilized, and intrusion prevention measures have been completed. As of January 31, 1995, approximately 250,000 liters (66,000 gallons) of non-complexed waste was contained in the tank. The waste was comprised of 15,000 liters (4,000 gallons) of supernatant and 235,000 liters (62,000 gallons) of sludge with no drainable interstitial liquid present. The total volume of waste within the tank corresponds to a depth of 48 cm (19 inches). The latest tank photograph shows a narrow ring of black sludge around the perimeter of the tank and most of the surface as a black liquid.

A2.2 SAMPLING INFORMATION

Tank BX-103 is currently scheduled to be core sampled. Two core samples are expected to be taken from risers #2 and #7. If a different riser is necessary to meet sampling and analysis requirements, this change must be recorded and approved by the cognizant engineer before sampling. The risers used may be recorded on a permanent data sheet or recorded directly in the work package.

Based on current waste volume information, each of the core samples is expected to consist of one 48 cm (19 inch) segment. The sampling objective is to obtain a vertical profile of the waste; therefore, more or less segments may need to be taken depending on the accuracy of the available waste depth information. For detailed information regarding the sampling activities, refer to tank BX-103 work package ES-95-00194. This document contains operating procedures and the chain-of-custody records for this sampling event.

One field blank for this tank shall be prepared by filling a sampler with deionized water. This field blank is to accompany the samples to the laboratory hot cell. All collected samples and the field blank shall be shipped to the laboratory following procedure TO-080-090 (*"Load/Transport Sample Cask(s)"*). Core samples shall be transported to the laboratory within three calendar days of each segment's removal from the tank.

Occasionally, hydrostatic head fluid (HHF) with lithium bromide (LiBr) may be used to aid in the collection of the core samples. If HHF is used, Sampling Operations must state this on the chain-of-custody form that accompanies the sample to the laboratory and provide a HHF blank for analysis. The HHF blank shall consist of a container filled with HHF from the same batch used during the sampling. This blank shall be analyzed for lithium and bromide in order to determine the concentration of the LiBr at the time the core was taken. Only one HHF blank per tank is required. The HHF blank is required in addition to the field/trip blank.

A3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

A3.1 TANK-SPECIFIC ANALYTICAL PROCEDURES

Flowcharts depicting the general safety screening sample breakdown and analysis scheme are 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. 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) in procedure TO-080-090 has been reviewed for any safety issues involved with transportation of core samples from tank BX-103. **For core samples from tank BX-103, the shipping containers must be vented every 47 days to release any 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 push core samples at the laboratory following approved procedures.

Step 2 Conduct the following on each extruded segment:

- Perform a visual examination of the segment(s).
- Record observations. This may include a sketch of the extruded segment in addition to written documentation of pertinent descriptive information such as color, texture, homogeneity, and consistency.
- Report the sample recovery results to the Characterization Program within one working day of sample breakdown.
- Take color photographs or a color videotape to visually document the composition of the extruded core segment.

Step 3 Does the segment contain drainable liquids?

Yes: Proceed to Step 4A
No: Proceed to Step 5

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

Step 4B Is the segment 100% drainable liquids?

Yes: Proceed to Step 11
No: Proceed to Step 5

SOLIDS PATH

Step 5 Divide each extruded segment into two equal subsegments (i.e., half-segments).

Step 6 Homogenize each half-segment following 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 a 1 to 2 gram aliquot from widely separated locations of the homogenized subsegment. Conduct the homogenization test following Bell (1993).

Step 9 Collect sufficient aliquots from each homogenized subsegment 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 subsegment 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 subsegment 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 (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 liquids as the liquid archive sample (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 requirements as listed in Section A7.2.

SECONDARY ANALYSIS PATH

Step 21 Perform secondary analyses following 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 requirements as listed in Section A7.2.

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

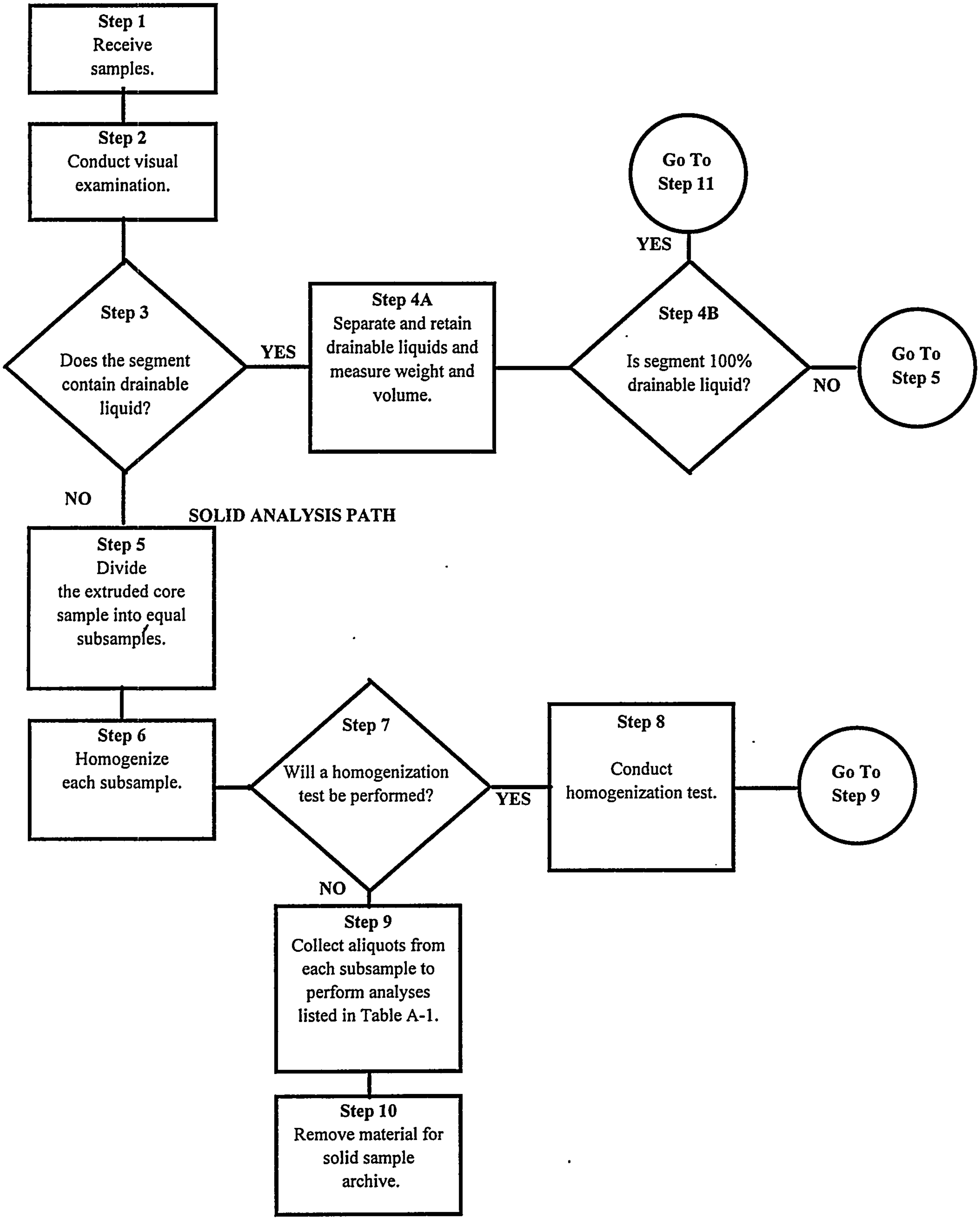

Figure A-1: Solid Analysis Flow Chart

LIQUID PATH

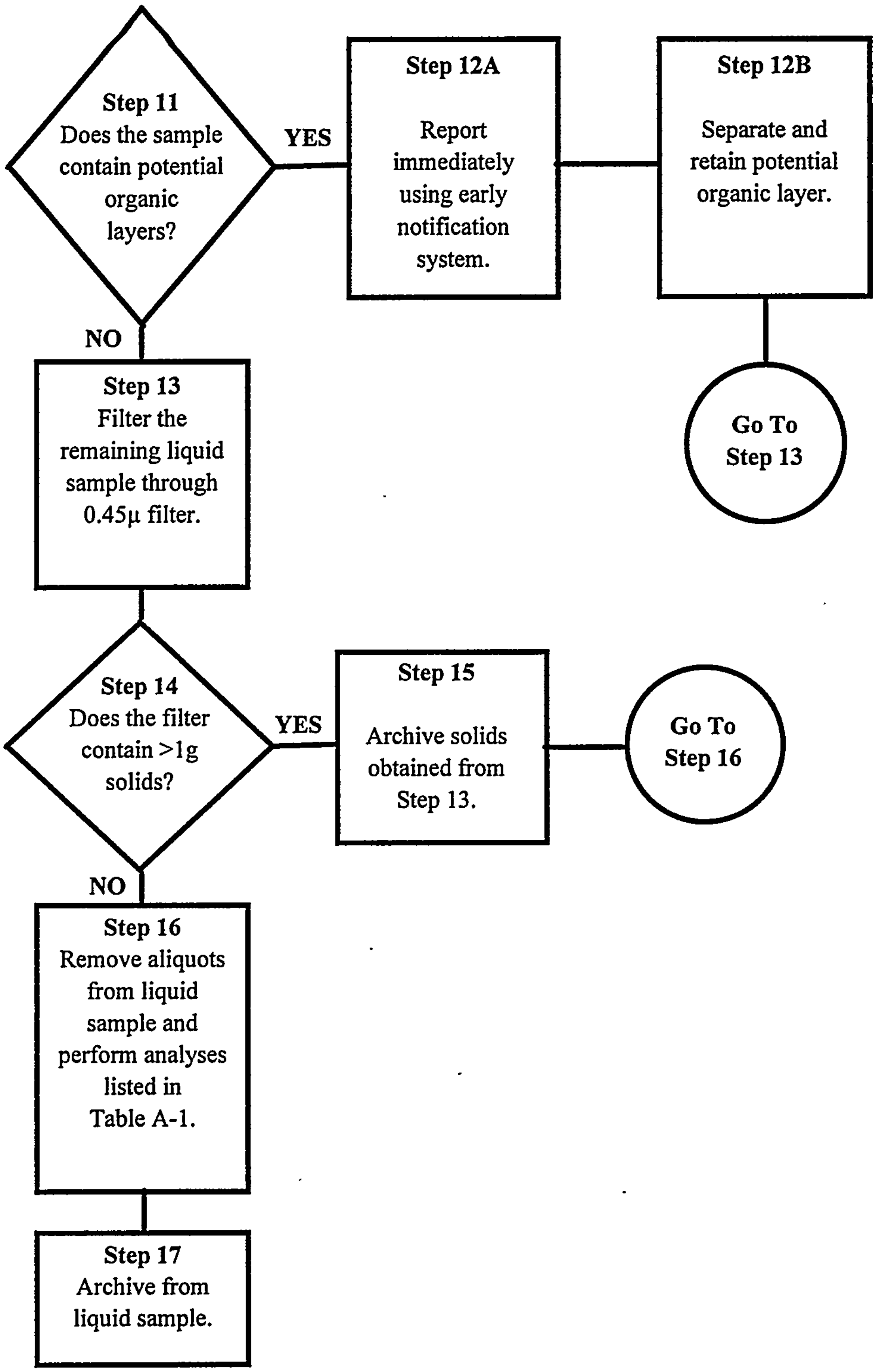

Figure A-2: Liquid Analysis Flow Chart

ANALYSIS AND REPORTING PATH

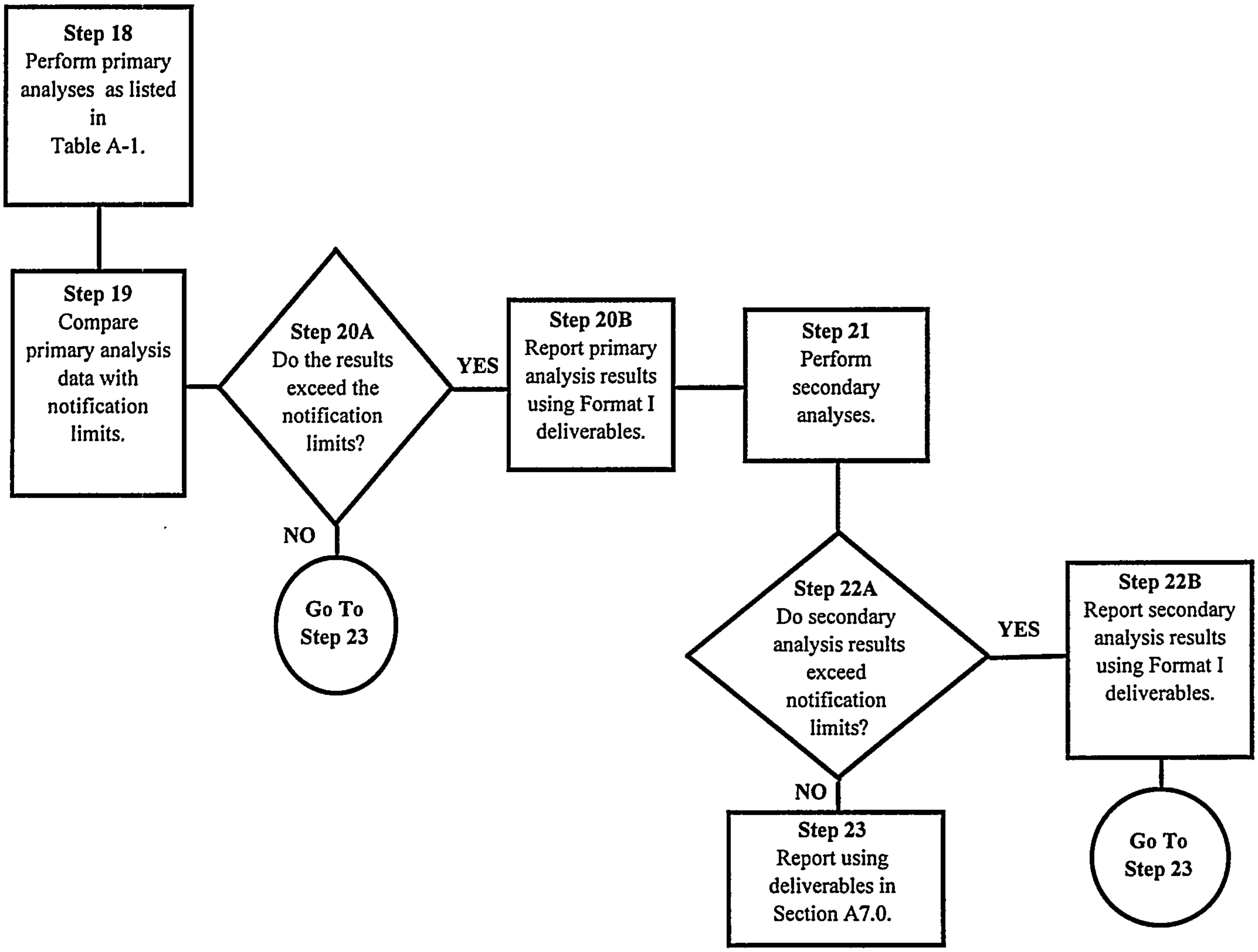

Figure A-3: Sample Analysis and Reporting Flow Chart

A3.2 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from the core samples taken from tank BX-103 is insufficient to perform the analyses requested and permit a minimum 20 mL archive per sample, the laboratory shall notify the Tank Cognizant Engineer within one working day (See Table A-2). A ranking 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

Confirmation of the priority levels or revision of sample breakdown procedures may be provided to the laboratory by the Characterization Program based upon the sample recovery, readily observable physical properties within the sample, and the requested sample breakdown procedures provided in Section A3.1. The priority of an analysis is specified by its designation as a primary or secondary analysis. Further ranking will be determined by the program on a DQO basis.

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 tank BX-103 core samples are based on the safety screening DQO referenced in Section A1.0. 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 WHC 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 quality assurance when analyzing the waste tank core samples at the WHC 222-S Laboratory. Additionally, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994), when implemented (August 31, 1995), shall be used for quality assurance guidance.

Method specific quality control such as calibrations and blanks are found in the analytical procedures. Sample quality control (duplicates, spikes, standards) is 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 core samples are to be taken from tank BX-103 and shipped to the performing laboratory by Sampling Operations following work package ES-95-00194. That work package shall also initiate the chain-of-custody for the samples. Approved procedure *Load/Transport Sample Cask(s)* (TO-080-090) 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.

Preferably, Sampling Operations should transport each segment collected to the performing laboratory within one working day of removing the segment from the tank, but must transport each segment within 3 calendar days. The field blank and HHF blank shall each count as a segment. Sampling Operations is responsible for verbally notifying the WHC 222-S Laboratory (373-2435) at least 24 hours in advance of an expected shipment.

A4.2.3 Sample Custody

The chain-of-custody form is initiated by the sampling team as described in work package ES-95-00194. 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.

Table A-1: Tank BX-103 Chemical, Radiological and Physical Analytical Requirements

SOLID ANALYSES						
Project Name	BX-103 Push Mode Core Sample		COMMENTS		REPORTING LEVELS	
Plan Number	WHC-SD-WM-TP-339, REV. 0		Homogenization Test - Per Laboratory Discretion		FORMAT I	Early Notify
			Field Blank - Required		FORMAT II	Process Control
PROGRAM	PROGRAM CONTACTS		Hot Cell Blank - Not Required		FORMAT III	Safety Screening
A. Safety Screening	Safety Screening	E. J. Lipke	HHF Blank - Required		FORMAT IV	Waste Management
	TWRS	K. E. Bell	TANK	#CORES	FORMAT V	RCRA Compliance
			BX-103	2	FORMAT VI	Special

PROGRAM	PRIMARY ANALYSES			SAMPLE[1]	PREP[2]	QUALITY CONTROL[3]						CRITERIA			FORMAT
	METHOD	ANAL.	WHC PROCEDURE	½ SEG SLDG		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
A	DSC	Energy	LA-514-113	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III
A	TGA	% H_2O	LA-560-112	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	48	I, III
A	Alpha	Total Alpha	LA-508-101	X	f or a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III
A	ICP[11]	Li	LA-505-151	X[11]	f or w	ea smpl	see 7	ea PB	ea AB	±10	90-110	µg/g	> 100	unknown	I, III
PROGRAM	**SECONDARY ANALYSES**			**SAMPLE[1]**	**PREP[2]**	**QUALITY CONTROL[3]**						**CRITERIA**			**FORMAT**
	METHOD	ANAL.	WHC PROCEDURE	½ SEG SLDG		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
A	Distillation[8]	CN	LA-695-102	X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III
A	Sep. & α counting[9]	Pu-239/240	LA-503-156	X	f	ea smpl	1/mtrx[6]	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III
A	ICP[9]	Fe	LA-505-151	X	f or a	ea smpl	see 7	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III
A	ICP[9]	Mn	LA-505-151	X	f or a	ea smpl	see 7	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III
A	ICP[9]	U	LA-505-151	X	f or a	ea smpl	see 7	ea PB	ea AB	±10	90-110	µg/g	none	93,300	III
A	IC[10]	Br	LA-533-105	X	w	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	> 1200	unknown	I, III
A	RSST[8]	Energy	see 8 below	X	d	N/A	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III
A	Hot Persulfate[8]	TOC	LA-342-100	X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg C/g	> 30,000	unknown	I, III

[1] ½ SEG SLDG-½ segment, sludge
[2] d-direct, f-fusion, a-acid, w-water
[3] PR-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-matrix
[4] Units for notification limits and expected range are those listed in the "units" column.
[5] Dry weight basis.
[6] Tracer or carrier may be used in place of a spike and results corrected for recovery.
[7] Either serial dilutions or matrix spikes will be performed.
[8] These analyses required if DSC exceeds notification limits. The RSST method, yet to be finalized, may be found in WHC-SD-WM-TP-104.
[9] Performed only if total alpha exceeds notification limit.
[10] Performed only if Li exceeds notification limit.
[11] If the chain of custody form indicates that HHF fluid with LiBr tracer was used to obtain the segment, Li analysis is to be performed on that segment.

Table A-1: Tank BX-103 Chemical, Radiological and Physical Analytical Requirements

LIQUID ANALYSES

Project Name	BX-103 Push Mode Core Sample	COMMENTS		REPORTING LEVELS	
Plan Number	WHC-SD-WM-TP-339 REV. 0	Homogenization Test - Per Laboratory Discretion		FORMAT I	Early Notify
		Field Blank - Required		FORMAT II	Process Control
PROGRAM	PROGRAM CONTACTS	Hot Cell Blank - Not Required		FORMAT III	Safety Screening
A. Safety Screening	Safety Screening E. J. Lipke	HHF Blank - Required		FORMAT IV	Waste Management
	TWRS K. E. Bell	TANK	#CORES	FORMAT V	RCRA Compliance
		BX-103	2	FORMAT VI	Special

PROGRAM	PRIMARY ANALYSES			SAMPLE[1]	PREP[2]	QUALITY CONTROL[3]						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	FB & S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
A	DSC	Energy	LA-514-113	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III
A	TGA	% H_2O	LA-560-112	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III
A	ICP[11]	Li	LA-505-151	X[11]	d[6]	ea smpl	see 7	ea AB	ea AB	±10	90-110	μg/mL	> 100[10]	unknown	I, III
A	Visual	Organic Layer	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	N/A		presence	unknown	I, III

PROGRAM	SECONDARY ANALYSES			SAMPLE[1]	PREP[2]	QUALITY CONTROL[3]						CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	FB & S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
A	Distillation[8]	CN	LA-695-102	X	d[6]	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	μg/mL	> 39,000[10]	unknown	I, III
A	IC[9]	Br	LA-533-105	X	d[6]	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	μg/mL	> 1,200[10]	unknown	I, III
A	RSST[8]	Energy	see 8 below	X	d	N/A	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III
A	Hot Persulfate[8]	TOC	LA-342-100	X	d[6]	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	μg C/mL	> 30,000[10]	unknown	I, III

[1] S-LEV LIQ-liquid taken from the segment level, FB-field blank

[2] d-direct, f-fusion, a-acid, w-water

[3] PR-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-matrix

[4] Units for notification limits and expected range are those listed in the "units" column.

[5] Dry weight basis.

[6] Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.

[7] Either serial dilutions or matrix spikes will be performed.

[8] These analyses required if DSC exceeds notification limits. The RSST method, yet to be finalized, may be found in WHC-SD-WM-TP-104.

[9] Performed only if Li exceeds notification limit.

[10] Converted from μg/g limit assuming a liquid density of 1.0 g/mL.

[11] If the chain of custody form indicates that HHF fluid with LiBr tracer was used to obtain the segment, Li analysis is to be performed on that segment.

A5.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank *BX-103 characterization* project are listed in Table A-2.

Table A-2: Tank BX-103 Project Key Personnel List

Individual	Organization	Responsibility
K. E. Bell	TWRS Characterization Plans and Reports	Tank BX-103 Tank Characterization Plan Cognizant Engineer
E.J. Lipke	Characterization Program	Safety Screening Point of Contact
East Area Shift Operations Manager	Tank Farm Operations	East Tank Farm Point of Contact if Notification Limit is Exceeded (373-2689)

A6.0 EXCEPTIONS, CLARIFICATIONS AND ASSUMPTIONS

A6.1 EXCEPTIONS TO DQO REQUIREMENTS

It is specified in the safety screening DQO (Babad and Redus 1994) 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. The revised ferrocyanide DQO (Meacham et al. 1994) has changed the requirement 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). The next revision to the safety screening DQO will incorporate this change. This Sampling and Analysis Plan specifies that cyanide analysis will be run on a half segment level and that notification shall be made if the DSC value exceeds 481 J/g dry weight basis (115 cal/g).

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 exothermic reaction (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.

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

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 is greater than 1 g/L. Total alpha is measured in µCi/g rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it is assumed that all alpha decay originates from Pu-239. The notification limit may then be calculated as shown in equation (2):

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

NOTE: If a density of 1.5 g/ml is assumed for solid material, the notification limit becomes 41 µCi/g.

- The safety screening DQO, upon which the analyses in Table A-1 are based, does not sufficiently address the analysis of the field blank or any drainable liquid present in the sample. To adequately characterize the tank, all analyses performed on the solids for the safety screening DQO, except total alpha, shall also be performed on any drainable liquids. To adequately determine if contamination of the sample material has occurred, the field blank shall be analyzed for those analyses performed on the segment-level liquid.

A7.0 DELIVERABLES

All analyses of tank BX-103 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 "Revised Interim Tank Characterization Plan Guidance" (Schreiber 1994a).

A7.1 PROGRESS REPORTS

Each laboratory performing analyses on tank BX-103 waste material from this sampling project shall provide a monthly status report to the Characterization Program. This report shall contain 1) a summary of the activities on the analyses completed or started under the work package, 2) results of preliminary analyses, and 3) schedule and cost information on a DQO basis.

Monthly and cumulative costs will be compared to the budgeted costs as part of the progress report. Monthly variances greater than 10% or $10,000, and cumulative variances greater than $50,000 from the budgeted costs 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 one working day by written communication to Analytical Services, Characterization Plans and Reports, the Characterization Program Office, the Safety Screening Representative, and Process Control documenting the observations. Points of contact within each program/project are defined in Schreiber (1995). 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, containing the results of the primary safety screen analyses shall be issued to the Safety Screening Representative, Characterization Plans and Reports, Process Control, the Characterization Program Office, Los Alamos Technical Associates, the Tank Characterization Resource Center, and the Tank Characterization Database representative (Schreiber 1995) within 45 days of receipt of the last segment of the last core 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 segment of the last core 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 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 Plans and Reports via an Engineering Change Notice to this Tank Characterization Plan. All changes shall also be clearly documented in the final data report.

At the request of the Characterization Program, additional analysis of sample material from this characterization project shall be performed following a revision of this Tank Characterization Plan.

A9.0 REFERENCES

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Schreiber, R. D., 1995, Letter to J. G. Kristofzski, "Point of Contact/Distribution List," 71520-95-108, dated March 23, 1995.

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