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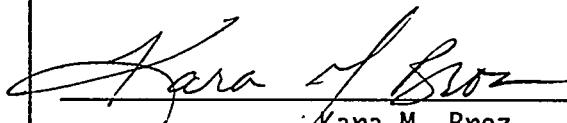
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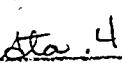
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7. Abstract

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-S-107.

8. RELEASE STAMP

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Tank 241-S-107

Tank Characterization Plan

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LIST OF ABBREVIATIONS

S-107	Tank 241-S-107
DL	Detection Limit
DNFSB	Defense Nuclear Facilities Safety Board
DOE	Department of Energy
DQO	Data Quality Objective
DST	Double-Shell Tank
GEA	Gamma Energy Analysis
RCRA	Resource Conservation and Recovery Act
SAP	Sampling and Analysis Plan
SST	Single-Shell Tank
TBP	Tributyl Phosphate
TCP	Tank Characterization Plan
TOC	Total Organic Carbon
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

The Defense Nuclear Facilities Safety Board (DNFSB) has advised the 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 the 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) milestone M-44 has been made, which states that "A Tank Characterization Plan (TCP) will also 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-S-107 (S-107) sampling activities.

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK S-107

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 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 S-107 as of January 1995 are discussed in the following paragraphs.

2.1 SAFETY SCREENING DATA QUALITY OBJECTIVES

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). The safety screening DQO identifies the guidelines used to determine to which classification a tank belongs, based on analyses that indicate if certain measurements are within established parameters. The 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 on one of these items is exceeded, further analysis of a secondary set of analytes, resulting in a possible change in tank classification, would be warranted. A tank can be removed from a Watch List if it is classified as SAFE.

This 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. 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 tank crust burn issue.

2.2 PRETREATMENT DATA QUALITY OBJECTIVES

Interim Data Quality Objectives for Waste Pretreatment and Vitrification (Kupfer et al. 1994) addresses the characterization needs for the Pretreatment, High-Level Waste Disposal, and Low-Level Waste Disposal programs. These programs are responsible for developing long-term treatment and storage processes for the Hanford Site Waste. This effort will require comprehensive physical and chemical information from waste tank samples. The pretreatment process must be able to separate the waste into feed streams that satisfy the safety issues associated with the operating requirements for the low-level and high-level vitrification facilities.

3.0 TANK AND WASTE INFORMATION

This section gives a summary of descriptive information available on tank S-107. Included are the present status and physical description of the tank, its age, process history, and expected tank contents from previous sampling and analytical data. The different types of waste, by layer, for tank S-107 will also be discussed. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990) and *Historical Tank Content Estimate for the Southwest Quadrant of the Hanford 200 West Area* (Brevick 1994).

3.1 TANK STATUS

As of December 1994, tank S-107 has been categorized as sound and was partially isolated in December 1982. It is a low-heat load tank and is awaiting stabilization. Tank S-107 has a waste volume of 1,420 kL (approximately 380 kgal), which is equivalent to approximately 359 cm (141 in) in depth. The waste consists of 260 kL (70 kgal) of saltcake, 53 kL (14 kgal) of supernate, and 1,110 kL (293 kgal) of sludge, of which 170 kL (45 kgal) are drainable interstitial liquids (Hanlon 1995).

3.2 TANK CONFIGURATION

Tank S-107 is one of twelve single-shell tanks in the 200 West Area S-Tank Farm. It is 23 m (75 ft) in diameter and 7 m (23 ft) in operating depth. It has a concave-shaped base and an operating capacity of 2,840 kL (750 kgal). Tank S-107 is the first tank in a three-tank cascade series which includes tanks 241-S-108 and 241-S-109. A cascade system consists of tanks connected in series by pipes. When the primary tank in the system became full, the waste would then flow to the secondary tank(s) in the system.

3.3 AGE AND PROCESS HISTORY OF TANK S-107

Tank S-107 was constructed between 1950 and 1951 and was filled with waste from the REDOX facility in the fourth quarter of 1952 until the fourth quarter of 1956. It was almost filled with liquids by 1953. Figure 1 summarizes the fill history from when tank S-107 was first placed on active status to the present time (Brevick 1994).

To provide more tank space, surface condensers that concentrated the wastes in the first two cascades by disposing of vapor condensate to cribs were installed in all tanks in the S-Tank Farm. Liquids levels in tank S-107 fluctuated for the next 20

years; then the tank filled rapidly with solids. The change can be attributed to the 242-S Evaporator/Concentrator, since tank S-107 was used as a receiver for the evaporator waste products. The tank was removed from service in 1980. Its level was adjusted in November 1978 and again on September 1980. The increase in the tank's liquid (supernatant) level in December 1993 is due to a comparison of 1983 and 1987 photo packages indicating a level increase. Based on this influx, an elevation was extrapolated for 1993 which correlates with the current liquid level (Brevick 1994).

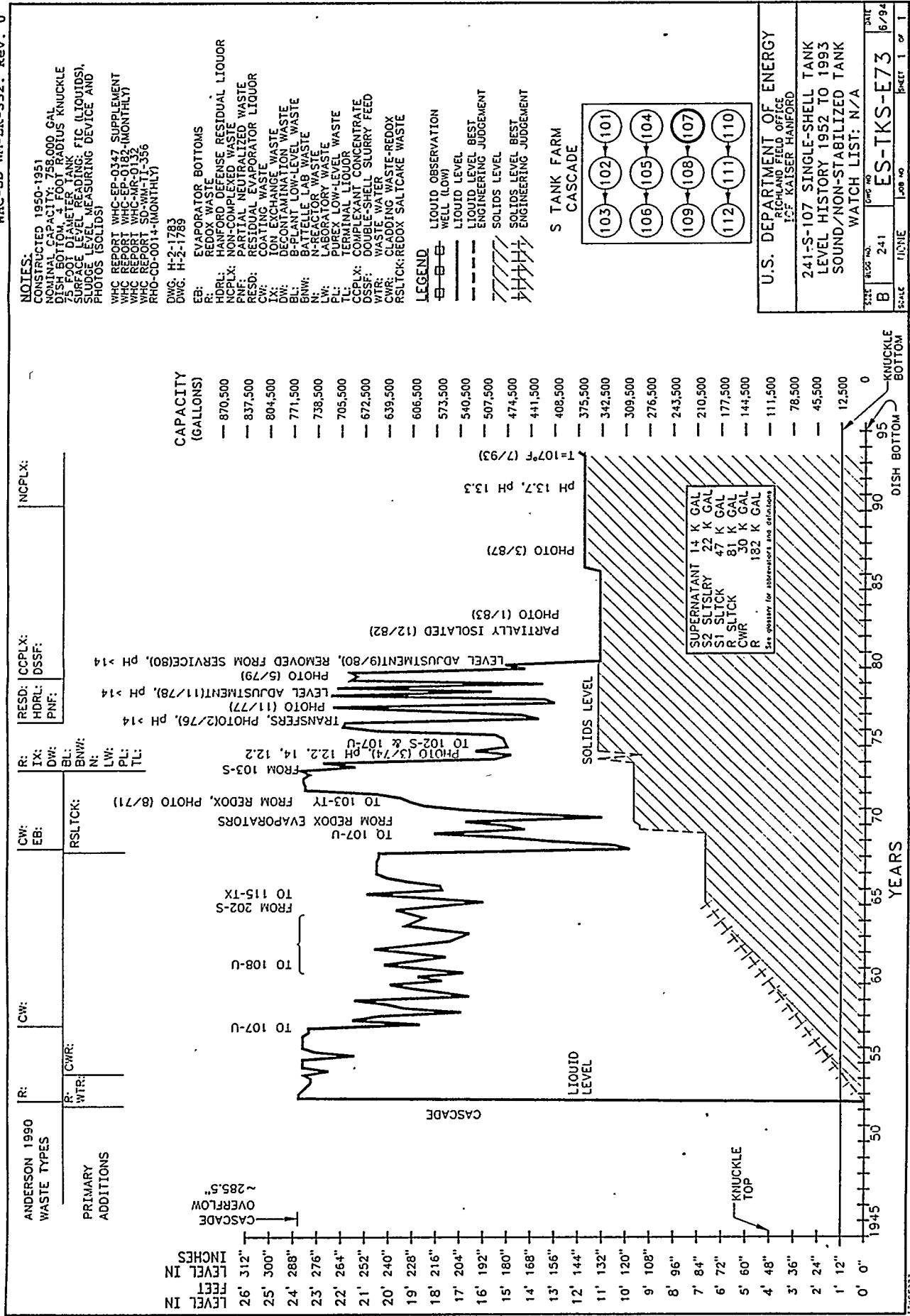
3.4 EXPECTED TANK CONTENTS

Tank S-107 is expected to contain two primary layers of waste. The bottom layer should contain a mixture of REDOX waste and REDOX cladding waste. The second layer contains S1 saltcake (waste generated from the 242-S evaporator/crystallizer from 1973 until 1976), and S2 salt slurry (waste generated from the 242-S evaporator-crystallizer from 1977 until 1980. The estimated inventory of Tank S-107 is presented below (Brevick 1994).

Table 1: Tank S-107 Solids Composite Inventory

Physical Properties			
Total Solid Waste	2.22E+06 kg (362 kgal)		
Heat Load	4.93 kW (1.68E+04 BTU/hr)		
Bulk Density	1.62 (g/cc)		
Water wt%	39.64		
TOC wt% C (wet)	0.02		
Chemical Constituents	µg/g	Chemical Constituents	µg/g
Na	6.58	PO ₄	0.10
Al	2.82	SO ₄	0.16
Fe	4.24E-02	F	9.23E-02
Cr	0.25	Cl	5.39E-03
Ni	7.58E-04	glycolate	3.66E-03
Mn	2.05E-02	acetate	2.32E-03
K	4.25E-03	U	1.97E+03
OH	12.41	Rad Constituents	µCi/g
NO ₃	4.93	Pu	0.27
NO ₂	0.15	Cs	96.71
CO ₃	0.27	Sr	2.61E+02

Figure 1: Fill History of Tank S-107



4.0 TANK S-107 SCHEDULED SAMPLING EVENTS

As of March 1995, only one sampling event for tank S-107 is currently scheduled, using the push mode core sampling system (Stanton 1995). The push mode core sampling system was chosen based on both historical information and surface photographs taken of the waste in 1987. Since the waste surface looks to be supernate and the waste material is comprised of saltcake, sludge, and pumpable liquid, tank S-107 seems a candidate for push mode core sampling. Although rotary mode core sampling could be performed, it would be substantially more expensive. Auger sampling is not appropriate due to the waste depth and supernate on the top layer. The push core shall be conducted following *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994) and *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994). Sampling and analytical requirements from these DQOs are summarized in Table 2. A more complete list of analytical requirements are given, as an appended attachment, in the appropriate Sampling and Analysis Plan (SAP).

Table 2: Integrated DQO Requirements

Sampling Event	Applicable DQO's	Sampling Requirements	Analytical Requirements*
Push-mode Core Sampling	-Safety Screening DQO -Pretreatment DQO	2 core samples from risers separated radially to the maximum extent possible	Energetics, Moisture, Total Alpha, Gas Composition

* These analyses are being requested per the Safety Screening DQO. At the present time, the Pretreatment program is asking for sample material only (refer to Section A6.1 of Appendix A, "Sampling and Analysis Plan for Push-Mode Core Sampling of Tank S-107 in Fiscal Year 1995").

5.0 REFERENCES

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APPENDIX A
SAMPLING AND ANALYSIS PLAN
FOR PUSH-MODE CORE SAMPLING
OF TANK S-107 IN FISCAL YEAR 1995

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LIST OF ABBREVIATIONS FOR APPENDIX A

ACL	Analytical Chemistry Laboratory
S-107	Tank 241-S-107
DOE	Department of Energy
DQO	Data Quality Objective
DSC	Differential Scanning Calorimetry
HHF	Hydrostatic Head Fluid
IC	Ion Chromatography
ICP	Inductively Coupled Plasma (atomic emission spectroscopy)
PNL	Pacific Northwest Laboratory
RCRA	Resource Conservation and Recovery Act of 1976
RSST	Reactive System Screening Tool
SAP	Sampling and Analysis Plan
TCP	Tank Characterization Plan
TGA	Thermogravimetric Analysis
TOC	Total Organic Carbon
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

A1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) will identify characterization objectives pertaining to sample collection, hot cell sample breakdown, and laboratory analytical evaluation and reporting requirements in accordance with the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994) and *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994). These Data Quality Objectives (DQOs) are described in the Tank Characterization Plan (TCP) for tank 241-S-107 (S-107). The pretreatment DQO, at the request of the Pretreatment Program, will have limited use in this SAP (refer to section A6.1).

A2.0 TANK STATUS AND SAMPLING INFORMATION

A2.1 TANK STATUS

As of December 1994, single-shell tank S-107 is identified as a low-heat load tank, that is passively ventilated, with partial interim isolation completed. Its integrity is categorized as sound. Tank S-107 contains 1,420 Kt (approximately 380 kgal), which is equivalent to a waste height of approximately 359 cm (141 in) of non-complexed waste (Hanlon 1995).

A2.2 SAMPLING INFORMATION

Tank S-107 is currently scheduled to be core sampled. Three core samples shall be collected from risers 16 and 6 of the tank (two from the 12 inch riser). The additional core sample is being taken to test different bit recoveries for the core sampling truck. If a different riser is capable of meeting the intent of other requirements in the DQO, it may be used if the riser number is recorded and approved in writing in advance by the sampling cognizant engineer. Risers used may be recorded on a permanent data sheet, or recorded directly in a work package.

Based on current waste volume information, each of the push-mode core samples is expected to consist of seven 48 cm (19 in) segments, and one 13 cm (5 in) segment. It should be noted that 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 current waste volume records. For detailed information regarding the sampling activities, refer to work package WS-95-00049. These documents contain operating procedures and the chain-of-custody records for this sampling event.

One field blank for this tank shall be obtained by filling a sampler with deionized water. This field blank is to accompany the samples to the laboratory hot cell. All collected samples shall be shipped to the laboratory according to procedure T0-080-090 ("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 in the chain of custody form that accompanies the sample to the laboratory, and must also provide an HHF blank for the laboratory. The HHF blank shall consist of a container filled with HHF (with LiBr tracer) from the same batch of HHF used during the sampling. It shall be analyzed for Li (and Br if the Li notification limit is exceeded) in order to determine the concentration of the tracer 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 (sampler filled with water).

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 segments 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 S-107 core samples. For tank S-107 core samples, the transport sample casks must be vented every 47 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. At this time the plans for the second core from the 12 inch riser have yet to be decided. However, this core is to be extruded with the other two cores. At the time of the extrusion, a decision regarding the purpose for this core will be made, and will be subsequently noted in the data report.

Step 1 Receive core samples at the laboratory in accordance with approved procedures.

Step 2 Conduct the following on the material from each extruded segment:

- ▶ Perform a visual examination of the segment(s).
- ▶ Record observations. This may include a sketch of the extruded core sample in addition to written documentation of pertinent descriptive information such as color, texture, homogeneity, and consistency.
- ▶ Take color photographs and/or a videotape to visually document the extruded core segments.

Step 3 Is the segment 100% drainable liquid?

Yes: Proceed to Step 14
No: Proceed to Step 4

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

SOLIDS PATH

Step 5 Divide each extruded core segment into half-segments.

Step 6 Homogenize each half-segment 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 homogenization test, 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 aliquots 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 Tank Cognizant Engineer 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 as the half-segment archive sample (Bratzel 1994).

Step 11 Combine half-segments proportional to the sludge recovery of the segment to build the solid composite of the core.

Step 12 Remove 100 mL of the solid composite as the Pretreatment solid composite archive (Bratzel 1994).

Step 13 Remove 125 mL and 1000 mL of the solid composite for process development work (see Section A6.2).

NOTE: If insufficient sample material is available to provide an archive and a sample for process development of the sizes described, divide the material remaining after Step 11 into equal portions (i.e., equal-sized portions for archive and process development work).

LIQUIDS PATH

Step 14 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 15A
No: Proceed to Step 16

Step 15A Report any visually observed immiscible (potential organic) layer immediately by the early notification system (Section A7.2).

Step 15B Separate and retain the potential organic layer for possible future analysis.

NOTE: Steps 16 through 22 shall be performed on the remaining (probable aqueous) liquid layer only.

Step 16 Filter the remaining liquid sample through a 0.45 micron filter.

Step 17 Is there greater than 1 gram of solid on the filter?

 Yes: Proceed to Step 18
 No: Proceed to Step 19

Step 18 Archive the solids for possible future analysis (Bratzel 1994).

Step 19 Remove sufficient aliquots from the segment-level liquid sample to perform the appropriate analyses listed in Table A-1 in duplicate.

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

Step 21 Combine the segment-level liquid proportional to the liquid recovery of the segment to build a liquid composite of the core.

Step 22 Remove 100 mL of the liquid composite as the Pretreatment liquid composite archive (Bratzel 1994).

PRIMARY ANALYSIS PATH

Step 23 Perform primary analyses as listed in Table A-1.

Step 24 Compare the primary analysis data with notification limits.

Step 25A Do the results exceed the notification limits (Table A-1)?

 Yes: Proceed to Step 25B.
 No: Proceed to Step 28.

Step 25B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section A7.2.

SECONDARY ANALYSIS PATH

Step 26 Perform secondary analyses according to Table A-1.

Step 27A Do the secondary analyses exceed the notification limits?

 Yes: Proceed to Step 27B
 No: Proceed to Step 28

Step 27B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section A7.2.

Step 28 Report results as listed in Section A7.0.

Figure A-1: Solid Analysis Flowchart

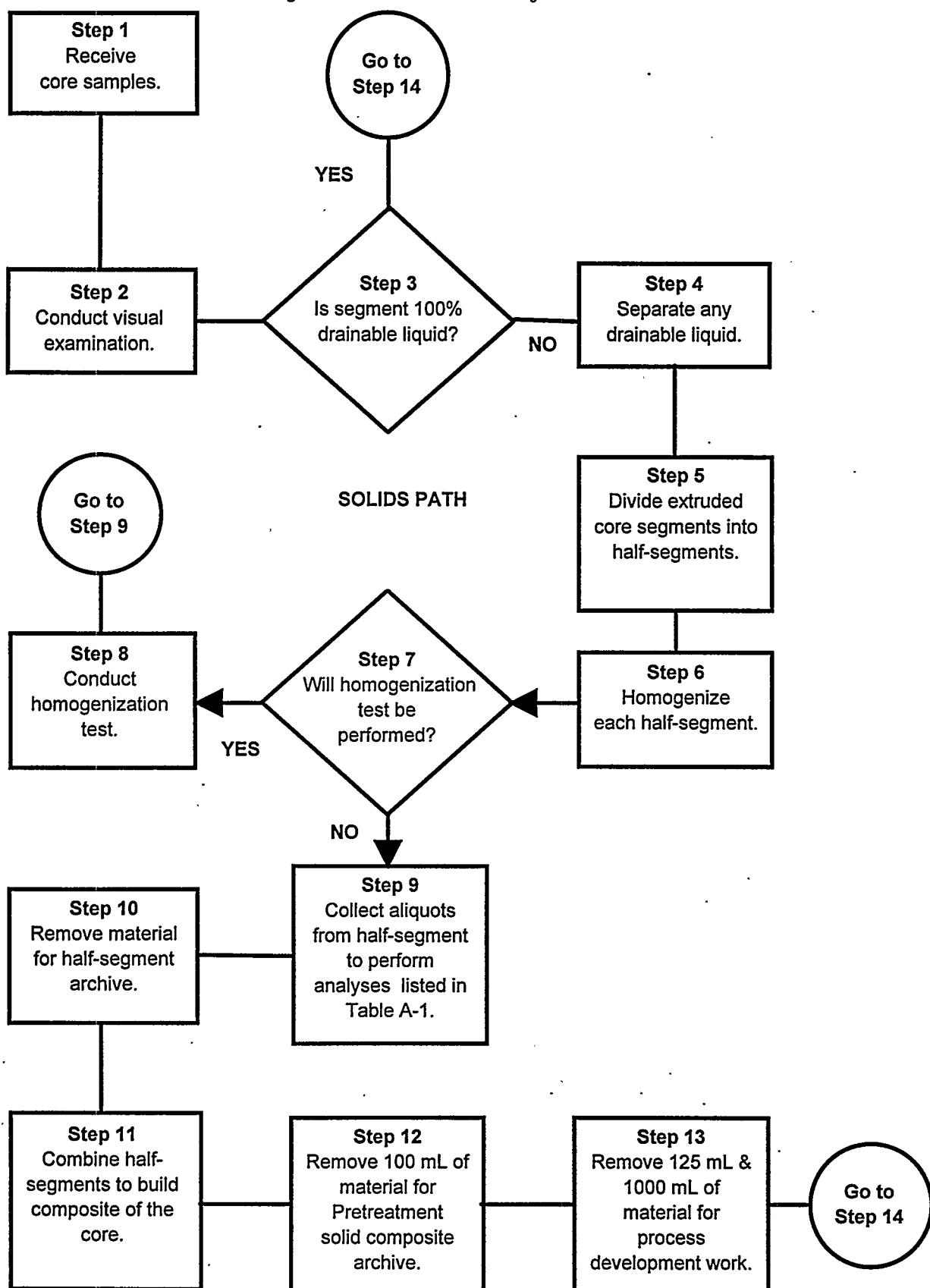


Figure A-2: Liquid Analysis Flowchart

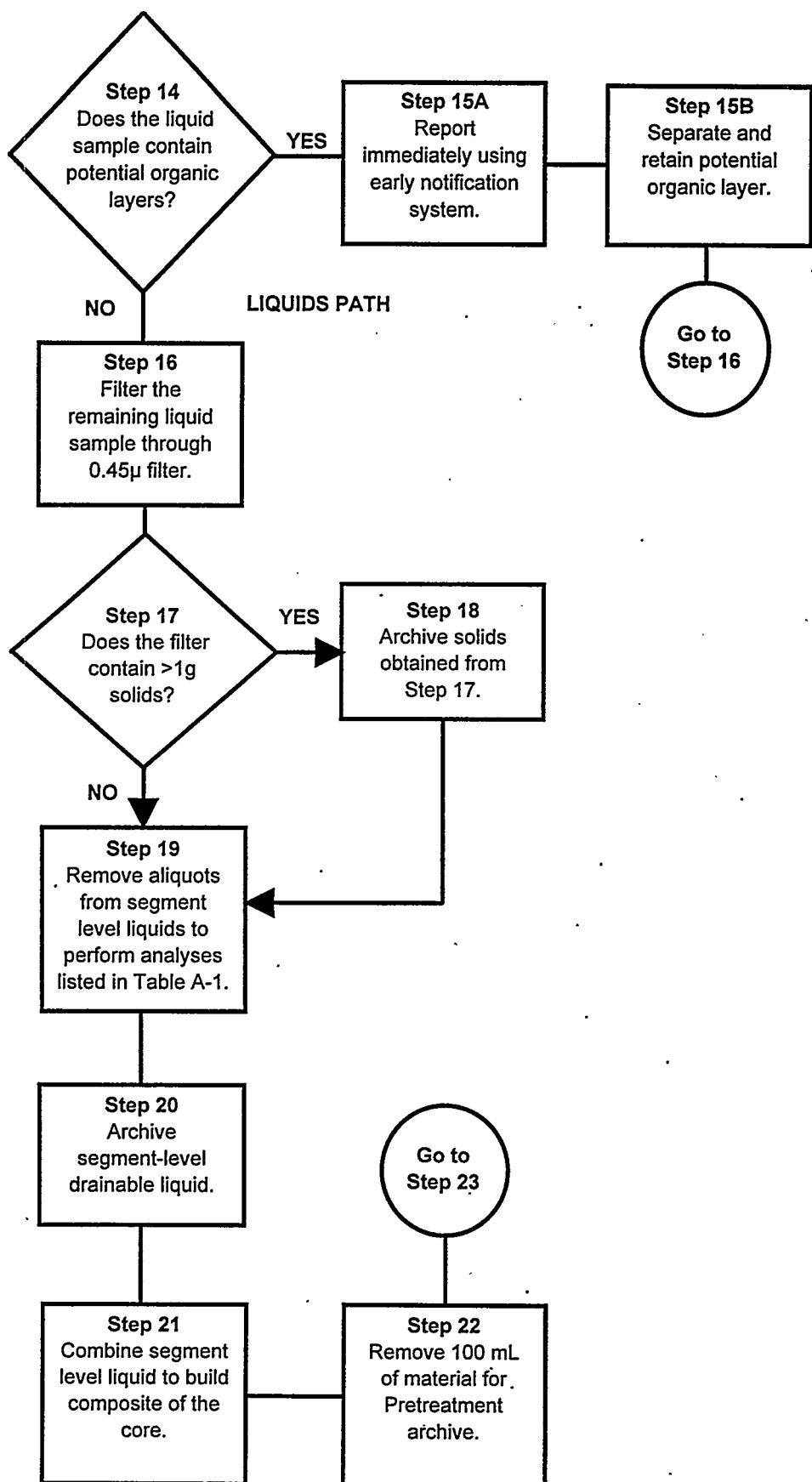
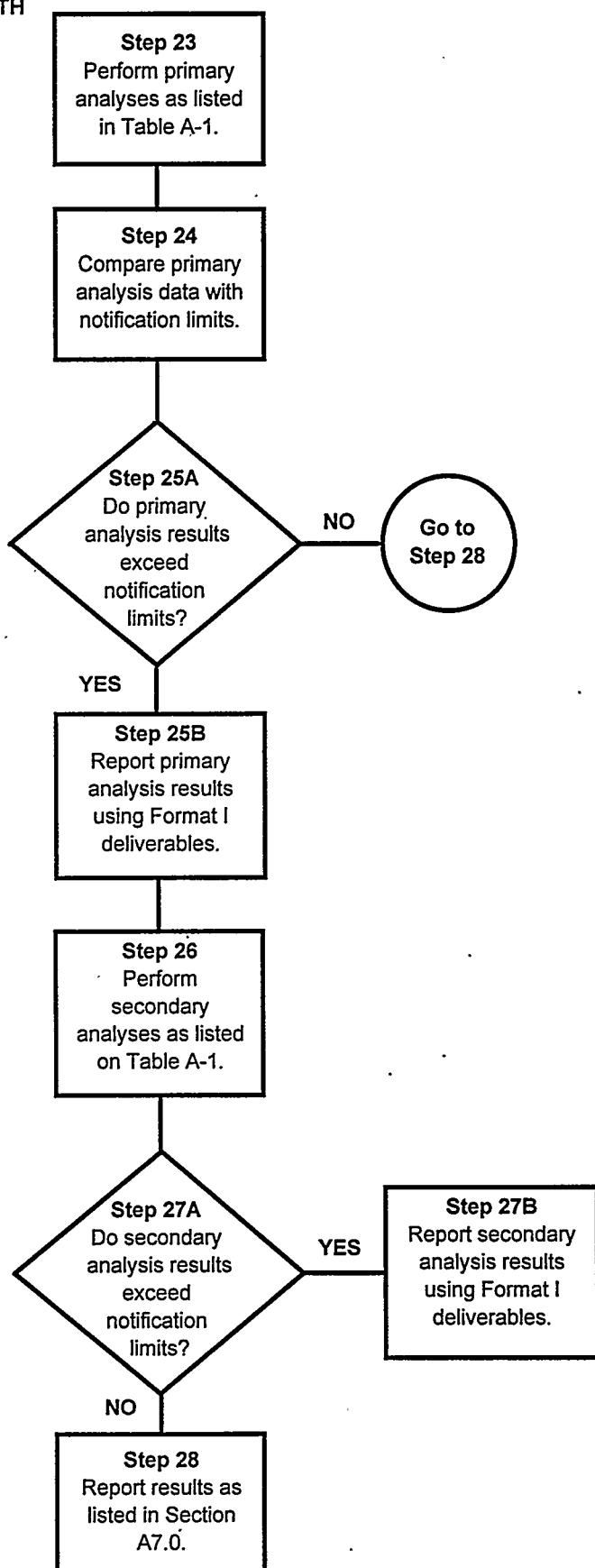


Figure A-3: Sample Analysis and Reporting Flowchart

ANALYSES PATH



A3.2 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from core samples taken from tank S-107 is insufficient to perform the analyses requested and to permit a minimum 20 mL archive per segment, the laboratory shall notify the Tank Cognizant Engineer and the manager of Analytical Services, Program Management and Integration, listed in Table A-2, within one working day. 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

Confirmation of prioritization levels, or revision of sample breakdown procedures may be provided by the Characterization Program based upon the sample recovery, readily observable physical property distinctions within the sample, and the requested breakdown procedures as provided in Section A3.1. The priority of an analysis is specified by its designation as a primary or secondary analysis. Further prioritization 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 the tank S-107 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 the quality assurance of analyzing the waste tank core samples at the WHC 222-S Laboratory. Additionally, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994), when implemented (currently scheduled for August 31, 1995), shall be used as quality assurance requirements.

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

Three core samples are to be taken from tank S-107 and shipped to the performing laboratory by Sampling Operations in accordance with the work package. That work package shall also initiate the chain-of-custody for the samples. Approved sampling procedures and procedure T0-080-090 ("Load/Transport Sample Cask(s)") are 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 segment collected to the performing laboratory within 1 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 the work package. 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 labelled 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 L0-090-101.

Table A-1: Tank S-107 Chemical, Radioological, and Physical Analytical Requirements

SOLID ANALYSES											
REPORTING LEVELS											
FORMAT I											
FORMAT II											
FORMAT III											
FORMAT IV											
FORMAT V											
FORMAT VI											
PROGRAM CONTACTS											
A. Safety Screening	Safety Screening	E. J. Lipke									
	TWRS	J. Jo									
PROGRAM ANALYSES											
PROGRAM	METHOD	ANAL.	WHC	SAMPLE ¹	PREP ²	SPK ³	QUALITY CONTROL ³	CRITERIA	FOR-MAT		
			PROCEDURE	1/2 SEG SLDG	DUP	SPK/ MSD	BLK CALIB	UNITS	NOTIFICATION	EXPECTED	RANGE ⁴
A	DSC	Energy	LA-514-113	X	d	ea smpl	N/A	ea AB	±10	90-110	J/g ⁵
A	TGA	% H ₂ O	LA-560-112	X	d	ea smpl	N/A	ea AB	±10	90-110	wt%
A	Alpha	Total	LA-508-101	X	f or a	ea smpl	1/mltr	ea PB	±10	90-110	µCi/g
A	ICP	Alpha	LA-505-151	X	f or a	ea smpl	see 7	ea PB	±10	90-110	>41
SECONDARY ANALYSES											
PROGRAM	METHOD	ANAL.	WHC	SAMPLE ¹	PREP ²	SPK ³	QUALITY CONTROL ³	CRITERIA	FOR-MAT		
			PROCEDURE	1/2 SEG SLDG	DUP	SPK/ MSD	BLK CALIB	UNITS	NOTIFICATION	EXPECTED	RANGE ⁴
A	Distillation ⁶	CN	LA-695-102	X	d	ea smpl	1/mltr	ea AB	±10	90-110	µg/g
A	Sep. & α ⁷	Pu-239/240	LA-503-156	X	f	ea smpl	1/mltr	ea PB	±10	90-110	µCi/g
A	ICP ⁹	Fe	LA-505-151	X	f or a	ea smpl	see 7	ea PB	±10	90-110	µg/g
A	ICP ⁹	Mn	LA-505-151	X	f or a	ea smpl	see 7	ea PB	±10	90-110	µg/g
A	ICP ⁹	U	LA-505-151	X	f or a	ea smpl	see 7	ea PB	±10	90-110	µg/g
A	IC ¹⁰	Br	LA-533-105	X	w	ea smpl	1/mltr	ea PB	±10	90-110	µg/g
A	RSST ⁸	Energy	see 8 below	X	d	N/A	N/A	ea AB	±10	90-110	J/g ⁵
A	Hol	TOC	LA-342-100	X	d	ea smpl	1/mltr	ea AB	±10	90-110	>481
A	Persulfate ⁶										unknown

¹1/2 SEG SLDG 1/2 segment, sludge²d-direct, f-fusion, a-acid, w-water³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, AB-analytical batch, PB-preparation batch, N/A-not applicable, mtrx-matrix⁴Units for notification limits and expected range are those listed in the "units" column.⁵Dry weight basis.⁶Tracer or carrier may be used in place of a spike and results corrected for recovery.⁷Either serial dilutions or matrix spikes will be performed.⁸These analyses required if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.⁹Performed only if total alpha exceeds notification limit.¹⁰Performed only if Li exceeds notification limit.

Table A-1: Tank S-107 Chemical, Radiological, and Physical Analytical Requirements

LIQUID ANALYSES												
Project Name		S-107 Push Mode Core Sample		WHC-SD-WM-TP-348, REV. 0		Homogenization Test - Per Laboratory Discretion		FORMAT I		REPORTING LEVELS		
Plan Number		Field Blank - Required		FORMAT II		Early Notify		FORMAT III		Process Control		
PROGRAM		PROGRAM CONTACTS		Hot Cell Blank - Not Required		FORMAT IV		Safety Screening		FORMAT V		
A. Safety Screening		Safety Screening		E. J. Lipke		Waste Management		FORMAT VI		RCRA Compliance		
TWRS		J. Jo						Special				
PRIMARY ANALYSES		SAMPLE ¹		PREP ²		QUALITY CONTROL ³		CRITERIA		FOR- MAT		
PROGRAM	METHOD	ANAL.	WHC	PROCEDURE	S-LEV LiQ	DUP	SPK/ MSD	BLK	CALIB	PR	AC	UNITS
A	DSC	Energy	LA-514-113	X	d	ea simpl	N/A	N/A	ea AB	±10	90-110	J/g ⁵
A	TGA	% H ₂ O	LA-560-112	X	d	ea simpl	N/A	N/A	ea AB	±10	90-110	wt%
A	ICP ¹¹	Li	LA-505-151	X ¹¹	d ⁶	ea simpl	see 7	ea AB	ea AB	±10	90-110	µg/mL
A	Visual	Organic Layer	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	N/A	presence
SECONDARY ANALYSES		SAMPLE ¹		PREP ²		QUALITY CONTROL ³		CRITERIA		FOR- MAT		
PROGRAM	METHOD	ANAL.	WHC	PROCEDURE	S-LEV LiQ	DUP	SPK/ MSD	BLK	CALIB	PR	AC	UNITS
A	Distillation ⁸	CN	LA-695-102	X	d ⁶	ea simpl	1/mtr ^x	ea AB	ea AB	±10	90-110	µg/mL
A	IC ⁹	Br	LA-533-105	X	d ⁶	ea simpl	1/mtr ^x	ea AB	ea AB	±10	90-110	µg/mL
A	RSST ⁸	Energy	see 8 below	X	d	N/A	N/A	N/A	N/A	N/A	N/A	J/g ⁵
A	Hot Persulfate ⁸	TOC	LA-342-100	X	d ⁶	ea simpl	1/mtr ^x	ea AB	ea AB	±10	90-110	µg C/mL
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, simpl-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 proceduralized, 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 H-HF fluid with LiBr tracer was used to obtain the segment, Li analysis is to be performed on that segment.												

1S-LEV LiQ-liquid taken from the segment level, FB-field blank

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

3PR-precision, AC-accuracy, ea-each, simpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, AB-analytical batch, PB-preparation blank, N/A-not applicable, mtrx-matrix

4Units 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.

7Either serial dilutions or matrix spikes will be performed.

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

9Performed only if Li exceeds notification limit.

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

11If the chain of custody form indicates that H-HF 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 S-107 characterization project are listed in Table A-2.

Table A-2: Tank S-107 Project Key Personnel List

Individual	Organization	Responsibility
J. Jo	TWRS Characterization Plans & Reports	Tank S-107 Tank Characterization Plan Cognizant Engineer
E. J. Lipke	WHC Safety Program	Safety Screening Point of Contact
J. T. Slankas	Pretreatment Program	Pretreatment Point of Contact
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, 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 (523 J/g). 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). The next revision to the safety screening DQO will incorporate this change. Therefore, although this Sampling and Analysis Plan uses the current safety screening DQO, this plan 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 dry weight basis (115 cal/g).

In the pretreatment DQO, a wide array of analyses has been requested. However, it has been determined by the Pretreatment Program that all of these analyses are not necessary for these samples. If necessary, the Pretreatment Program will personally contact the laboratory to run analyses on the archived composite samples. Therefore, the Pretreatment Program has directed that only a 125 mL composite solid sample for process development and a 100 mL composite sample for archive shall be obtained from this sampling event (Slankas 1995). In addition, the Pretreatment Program specifically requested a 1000 mL composite solid sample from tank S-107.

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 J/g or cal/g) determined by 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: If there is greater than 90 percent water in a sample, converting to a dry weight basis may lead to a large error in the DSC value. However, the 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 $\mu\text{Ci}/\text{g}$ 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):

$$\left(\frac{1 \text{ g}}{L} \right) \left(\frac{1 \text{ L}}{10^3 \text{ mL}} \right) \left(\frac{1 \text{ mL}}{\text{density g}} \right) \left(\frac{0.0615 \text{ Ci}}{1 \text{ g}} \right) \left(\frac{10^6 \mu\text{Ci}}{1 \text{ Ci}} \right) = \frac{61.5 \mu\text{Ci}}{\text{density g}} \quad (2)$$

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

- ▶ The safety screening DQO, upon which some of the analyses in Table A-1 are based, does not sufficiently address the analyses of any drainable liquid present. In order to characterize the tank waste adequately, 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 and on the field blank.
- ▶ The Pretreatment Program has requested 125 mL and 1000 mL of the solid composite material for process development work. A test plan (Lumetta and Rapko 1994; Temer 1994) will be used to guide this process development work. Since the Characterization Program is responsible for the taking of tank samples, the Characterization Program will need to approve the test plan. This approval will not only ensure that the DQO process has been used in the generation of the test plan and that there is justification for the samples, but also that the facility receiving the samples is in an adequate position to handle radioactive material. At such time that the test plan is approved by the Characterization Program, the Characterization Program will direct the performing laboratory, via a letter of instruction, to allow shipment of the sample material to the Process Chemistry section of PNL.

- None of the DQO efforts, upon which the analyses in Table A-1 are based, address the analyses performed on the field blank. To adequately determine if contamination of the sample material has occurred, the field blank shall be analyzed for those primary analyses done on the segment-level liquid.

A7.0 DELIVERABLES

All analyses of tank S-107 waste material shall be reported as Format 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 S-107 waste material from this core 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 S-107, 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% or \$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 exceeding their notification limits shall be reported by calling the East Tank Farm Operations Shift Manager at 373-2689 and the Characterization Program Office. This verbal notification must be followed within 1 working day by written communication, documenting the observations to Characterization Plans and Reports, Characterization Program Office, Safety Screening Representative, and Process Control (Schreiber 1995). Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above 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 screening analyses shall be issued to the Safety Screening Representative, Characterization Plans and Reports, Characterization Program Office, Los Alamos Technical Associates, Tank Characterization Resource Center, Tank Characterization Database, and Process Control

(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. 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 records. All significant changes (such as changes in scope) shall be documented by Characterization Plans & Reports via an Engineering Change Notice to this 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 plan.

A9.0 REFERENCES

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