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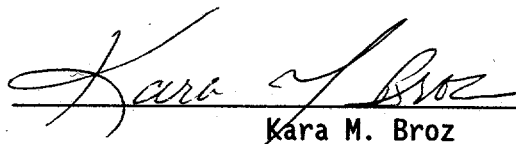
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7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, 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-C-107.

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Tank 241-C-107 Tank Characterization Plan

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LIST OF ABBREVIATIONS

IC	1st Cycle Decontamination Waste
C-107	Tank 241-C-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 objectives (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." This document satisfies that requirement for the tank 241-C-107 (C-107) sampling activities.

2.0 DATA QUALITY OBJECTIVES APPLICABLE TO TANK C-107

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 sampling of tanks to resolve safety issues. Other samples may be requested for ongoing waste processing and regulatory requirements in active tanks. As of January 1995, the DQO's that may apply to tank C-107 are discussed in the following sections.

2.1 SAFETY SCREENING DATA QUALITY OBJECTIVES

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. A tank can be removed from a Watch List if it is classified as SAFE. The Tank Safety Screening Data Quality Objectives (Babad and Redus 1994) identifies the requirements used to determine to which classification a tank belongs, based on analyses that indicate if certain measures are above or below established thresholds. The measures begin with the determination of the concentration of primary analytes which have been considered indicators of potentially unsafe conditions within a tank. The primary analytes include 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, 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.

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 C-107 HISTORICAL INFORMATION

This section gives a summary of descriptive information available on tank C-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 C-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 Northeast Quadrant of the Hanford 200 East Area* (Brevick 1994).

3.1 JANUARY 1995 TANK STATUS

Currently tank C-107 is categorized as a sound, low-heat load tank with partial isolation completed in December 1982. The tank is awaiting stabilization. It has a waste volume of 1,040 kL (275 kgal), which is equivalent to approximately 240 cm (95 in) in depth. The waste consists of 1,040 kL (275 kgal) of sludge, of which 98 kL (26 kgal) are drainable interstitial liquids (Hanlon 1994). Recent readings (January, 1995), obtained from Tank Farm Surveillance, indicate a waste depth of 233 cm (approximately 92 in) below riser #8. The highest recent waste temperature in tank C-107, taken from thermocouple #3 in riser #5, is approximately 52 C (125 f).

3.2 TANK CONFIGURATION

Tank C-107 is one of sixteen single-shell tanks in the 200 East Area C-Tank Farm. It is 23 m (75 ft) in diameter and approximately 5 m (16 ft) in operating depth. It has a concave-shaped base and an operating capacity of 2,010 kL (530 kgal). The tank has seven active dry wells monitoring radiation in the surrounding soil (Hanlon 1994).

3.3 TANK HISTORY

Tank C-107 was constructed between 1943 and 1944 as the first tank in a three-tank cascade series with tanks 241-C-108 and 241-C-109. The tank was put into service in April 1946. The cascade began filling in April 1947 with first cycle decontamination waste from the BiPO₄ process (1C waste). In September 1947, the tank was full and began cascading to tank 241-C-108. The cascade was declared full in September 1948.

In 1952, 490 kL (130 kgal) of 1C supernate was transferred to tank 241-B-106, and the tank was refilled with tributyl phosphate (TBP) waste. On December 18, 1952, the

overflow line from tank C-107 to tank 241-C-108 was discovered to be plugged. The line remained plugged until the third quarter of 1955. During October 1956, 587 kL (155 kgal) of the TBP waste was scavenged. Between the beginning of 1957 and the middle of 1961 the tank contained 1C waste and the volume was increased by several line flushes.

From the fourth quarter of 1961 until the third quarter of 1976 the tank received cladding waste from a variety of sources. These sources include the hot semi-works, CR vault, Hanford Lab Operations, strontium semi-works, Battelle Northwest, and ion exchange. Tank C-107 received waste from N-Reactor, miscellaneous lab waste, decontamination waste, and evaporator bottoms. There were several transfers to BY tanks and other C tanks.

In 1976, tank C-107 was removed from service. Saltwell pumping began in the third quarter of 1977. By the time the tank was declared inactive in 1978, it contained 1,290 kL (340 kgal) of non-complexed waste.

Figure 1 summarizes the fill history from when tank C-107 was first placed on active status to the present time (Brevick 1994).

3.4 EXPECTED TANK CONTENTS

Tank C-107 is expected to contain three primary layers of waste. The bottom layer should contain a mixture of the following wastes: ion exchange, concentrated phosphate waste from N-Reactor, Hanford Lab Operations, strontium semi-works, Battelle Northwest, 1C, TBP waste, cladding waste, and the hot semi-works. The middle layer should contain strontium recovery supernate. The upper layer should consist of non-complexed waste (Hill et al. 1991).

Although tank C-107 currently contains no supernate, the most recent and complete analytical data available were obtained from supernate analyses conducted in the first quarter of 1991. These results are summarized in Table 2 (Edrington 1991).

Figure 1: Fill History of Tank C-107



Table 1: Supernate Results for Tank C-107

Physical Property			
Analyte	Result	Analyte	Result
Density	1.14 g/mL	pH	9.6
Percent Water	82 %	TOC	1.03 g/L of C
Radiochemistry			
Analyte	Result ($\mu\text{Ci/L}$)	Analyte	Result ($\mu\text{Ci/L}$)
Americium-241	0.76	GEA Liquid (^{137}Cs)	41,200
Plutonium-239/240	462	Total Alpha	484
Strontium-89/90	1,700	Total Beta	68,300
Technetium-99	81		
Anion			
Analyte	Result ($\mu\text{g/mL}$)	Analyte	Result ($\mu\text{g/mL}$)
CO_3^{2-}	19,200	NO_2^-	69,500
OH^-	2,890	PO_4^{3-}	10,900
NO_3^-	47,900	SO_4^{2-}	12,500
Cation			
Analyte	Result ($\mu\text{g/mL}$)	Analyte	Result ($\mu\text{g/mL}$)
Aluminum	36.5	Molybdenum	26.2
Arsenic	0.5	Potassium	315
Calcium	2.28	Selenium	< DL ¹
Chromium	418	Silicon	41.0
Iron	2.28	Silver	125
Magnesium	2.28	Sodium	70,700
Manganese	0.57	Tin	5.7
Mercury	< DL ¹	Zirconium	116

¹ Less than Detection Limit

4.0 STRATEGY FOR WASTE CHARACTERIZATION AND SAFETY ISSUE RESOLUTION

In this section, the DQO requirements for sampling and analysis are integrated and compared with scheduled sampling and analysis activities.

4.1 TANK C-107 SCHEDULED SAMPLING EVENTS

The characterization objectives in fiscal year 1995 involve sampling of tanks to identify and resolve safety issues. A push-mode core sampling in March 1995 is scheduled for tank C-107. This sampling event 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). This sampling and analysis event, if successful, will also satisfy the January 1995 applicable DQO requirements. These requirements are summarized in Table 2. A more complete list of analytical requirements are given in the Sampling and Analysis Plan (SAP). It should be noted that the pretreatment DQO, at the request of the Pretreatment Program, will have limited use in is TCP (refer to the SAP). No other sampling is scheduled for tank C-107 through fiscal year 1997 (Stanton 1994).

Table 2: Integrated DQO Requirements

Sampling Event	Applicable DQO's	Sampling Requirements	Analytical Requirements
Push-mode Core Sampling	-Safety Screening DQO -Pretreatment DQO	3 core samples from risers separated radially to the maximum extent possible	Energetics, Moisture, Total Alpha, Gas Composition

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APPENDIX A

SAMPLING AND ANALYSIS PLAN

FOR PUSH-MODE CORE SAMPLING

OF TANK C-107 IN FISCAL YEAR 1995

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LIST OF ABBREVIATIONS FOR APPENDIX A

ACL	Analytical Chemistry Laboratory
C-107	Tank 241-C-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	<i>Resource Conservation and Recovery Act of 1976</i>
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 C-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

Currently, single-shell tank 241-C-107 (C-107) is classified as a low heat load, non-Watch List tank. Its integrity is categorized as sound. It contains 1,040 kL (275 kgal), or an approximate depth equivalence of 240 cm (95 in), of dilute complexed waste (Hanlon 1994). Recent readings (January, 1995) obtained from Tank Farm Surveillance indicate a waste depth of 233 cm (91.7 in) below riser #8. The recent waste temperature in tank C-107, taken from thermocouple #3 in riser #5, is approximately 51.7 °C (125 °F).

A2.2 SAMPLING INFORMATION

Tank C-107 is currently scheduled to be sampled by the push-mode core sampling method. A total of three core samples shall be collected from risers 3 and 7 of the tank. If, for some reason, it becomes necessary to change the riser(s) to be sampled, this change may be done if the alternate riser(s) meet(s) the intent of the applicable DQOs, and must be authorized by the sampling cognizant engineer and recorded in a controlled document such that traceability is preserved. 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 shall consist of five 48 cm-(19 in) segments. However, since the sampling objective is to obtain a vertical profile of the waste in tank C-107, a different number of segments may need to be obtained, depending on the accuracy of the current waste volume records. For detailed information regarding the sampling activities, refer to work packages ES-94-01267 and ES-94-01268. These documents contain operating procedures and the chain-of-custody records for this sampling event. In addition, refer to Plant Operating Procedure TO-080-055.

Hydrostatic head fluid (HHF) with lithium bromide (LiBr) as a tracer shall be used to aid in the collection of core samples. An HHF blank shall be prepared as part of the sampling procedure. The blank shall consist of a container filled with HHF (with LiBr tracer) from the same batch of HHF used during the push-mode core 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. This blank is required in addition to the field/trip blank (sampler filled with water). For specific information concerning sample

handling, custody, and transport, refer to the quality assurance/quality control requirements in Section A4.2.

A3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

It should be noted that in accordance with the Safety and Analysis Report for Packaging (SARP), samples from tank C-107 must be vented every 47 days from the time of cask sealing to allow any retained gas to escape.

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.

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.

In order to meet a request of the Pretreatment Program, an additional core sample from one of the two risers to be sampled is being obtained from tank C-107. This core sample is to be extruded, composited, and prepared for shipment to a laboratory for process development work. The Pretreatment Program will provide more specific direction to the laboratory regarding this material at a later date. The following steps are therefore applicable to only two of the three cores to be obtained (one from each riser sampled).

- Step 1 Receive push-mode 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 Separate any drainable liquid from the solids. Measure and record the volume. Retain drainable liquids for further processing.
- Step 4 Is the segment 100% drainable liquid?
 - Yes: Proceed to Step 14
 - No: Proceed to Step 5

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 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 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 insufficient 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 analysis prioritization 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).
- Step 11 Combine half-segments proportional to the sludge recovery of the segment to build the solid core composite.
- Step 12 Remove 100 mL of the solid composite as the Pretreatment solid composite archive (Bratzel 1994).
- Step 13 Remove all remaining material from the solid composite for process development work (see Section A6.2).

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 (see 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

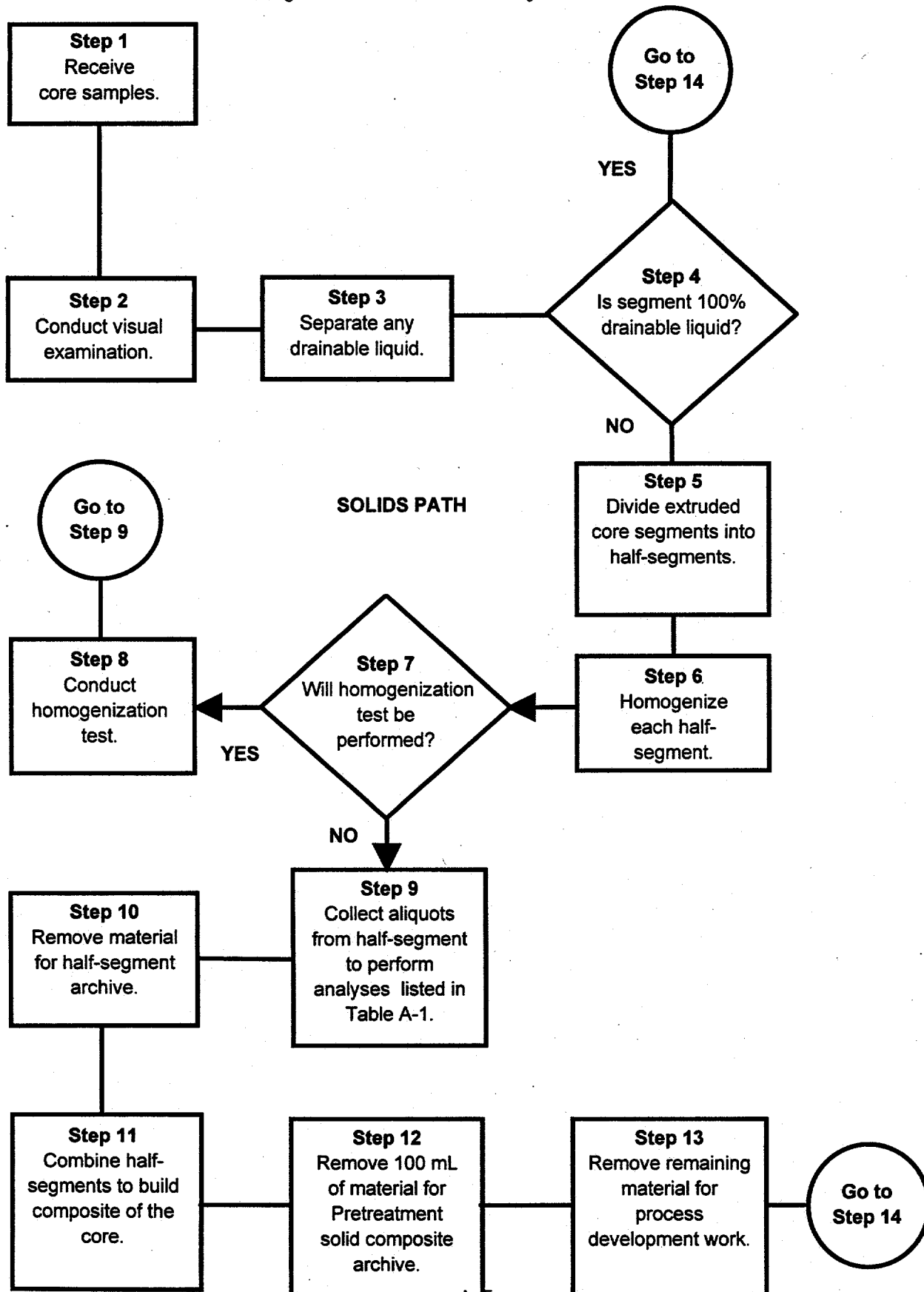


Figure A-2: Liquid Analysis Flowchart
LIQUIDS PATH

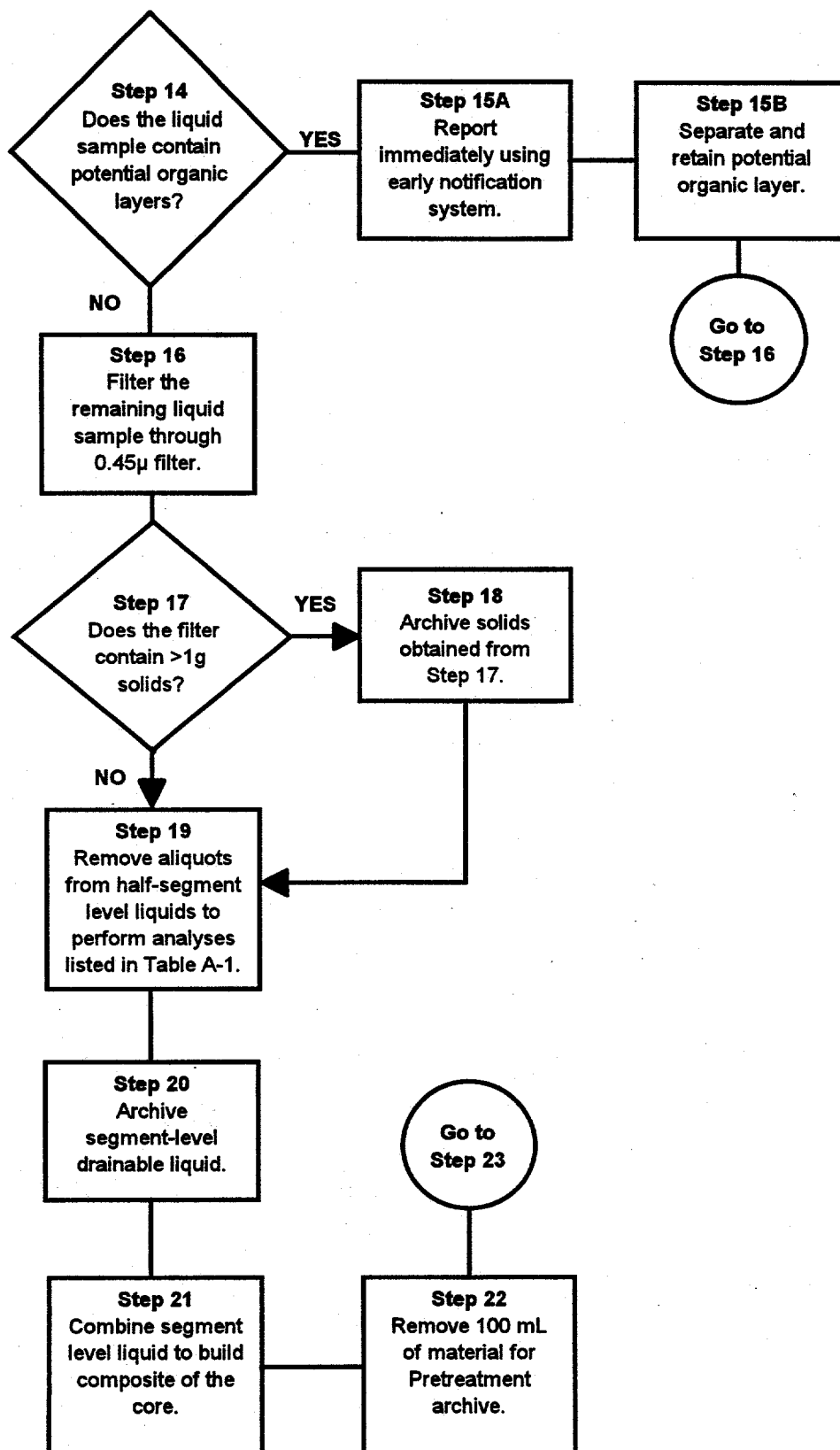
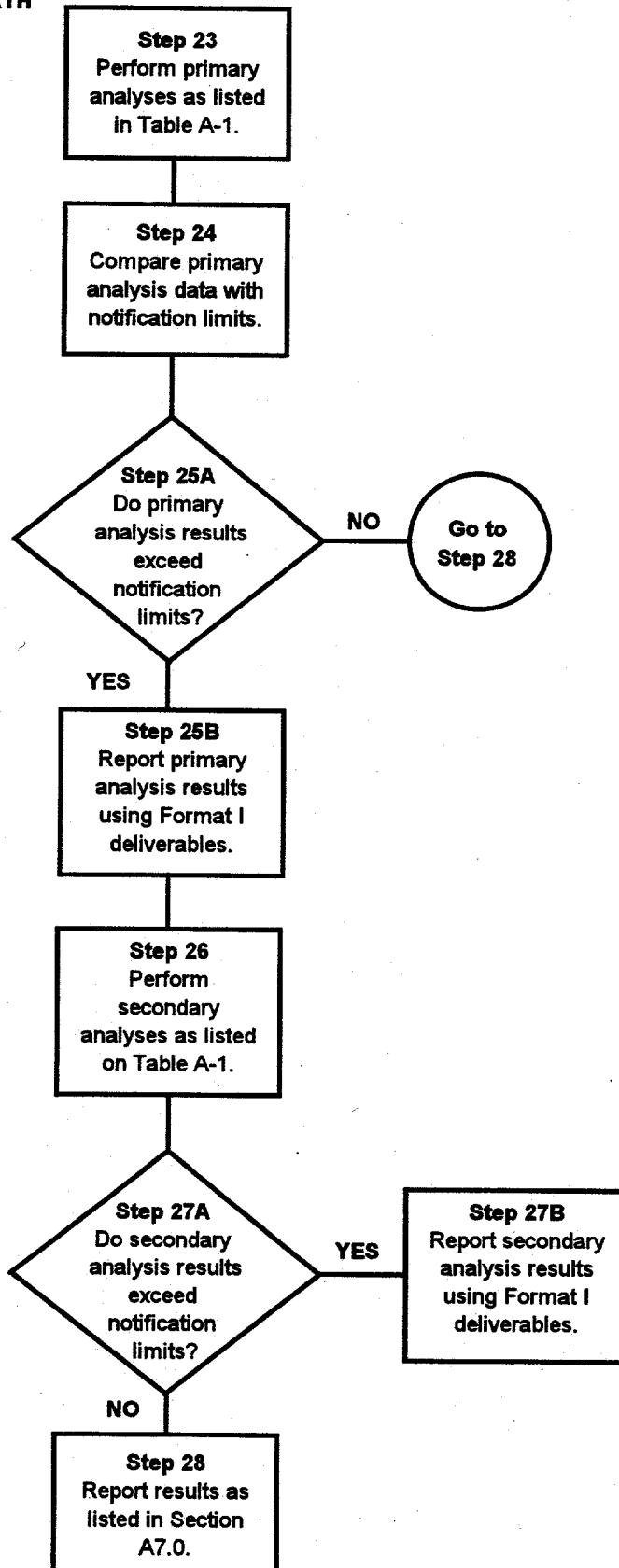


Figure A-3: Sample Analysis and Reporting Flowchart
ANALYSES PATH



A3.2 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from core samples taken from tank C-107 is insufficient to perform the analyses requested and to permit a minimum 10 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

The analyses to be performed for the tank safety screening have 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

DSC, TGA, Total Alpha (when necessary), and Li analyses shall be performed.

PRIORITY LEVEL 2

Secondaries for safety screening (TOC, cyanide, RSST, bromide, Pu-239/240, and ICP for 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 C-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 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 222-S Laboratory. If the analyses are performed at the 325 Analytical Chemistry Laboratory (ACL), they 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

Three core samples are to be taken from tank C-107 and shipped to the performing laboratory by Sampling Operations in accordance with work packages ES-94-01267 and ES-94-01268. Those work packages shall also initiate the chain-of-custody for the samples. Approved Plant Operating Procedure T0-080-055, 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 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 (373-2639), 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 packages ES-94-01267 and ES-94-01268. 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. Receipt and control of samples in the 325 ACL are described in procedure PNL-AL0-051.

Table A-1: C-107 Chemical, Radiological, Physical Analytical Requirements

SOLID ANALYSES																								
Project Name		C-107 Push Mode Core Sample				COMMENTS				REPORTING LEVELS														
Plan Number		WHC-SD-WM-TP-290, REV. 0				Homogenization Test - Not Required				FORMAT I														
						Field Blank - Required				FORMAT II														
PROGRAM		PROGRAM CONTACTS				Hot Cell Blank - Not Required				FORMAT III														
A. Safety Screening		Safety Screening				H-F Blank - Required				FORMAT IV														
		TWRS				TANK				#CORES				RISER#			FORMAT V							
		222-S Laboratory				J. G. Kristofzski				C-107				3				3 and 7			FORMAT VI			
		325 Laboratory				S. G. McKinley																		
PROGRAM	PRIMARY ANALYSES				SAMPLE ¹	PREP ²	QUALITY CONTROL ³				CRITERIA				FOR-MAT									
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE			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	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	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III								
A	Alpha	Total Alpha	LA-508-101	PNL-ALO-421	X	f or a	ea smpl	1/mtrix	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III								
A	ICP	Li	LA-505-151	PNL-ALO-211	X	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				FOR-MAT									
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE			DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴		EXPECTED RANGE ⁴								
A	Distillation ⁸	CN	LA-695-102	PNL-ALO-287	X	d	ea smpl	1/mtrix	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	PNL-ALO-423	X	f	ea smpl	1/mtrix ⁶	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III								
A	ICP ⁹	Fe	LA-505-151	PNL-ALO-211	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	PNL-ALO-211	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	PNL-ALO-211	X	f or a	ea smpl	see 7	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III								
A	IC ¹⁰	Br	LA-533-105	PNL-ALO-212	X	w	ea smpl	1/mtrix	ea PB	ea AB	±10	90-110	µg/g	1200	unknown	I, III								
A	RSST ⁸	Energy	see 8 below	N/A	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	PNL-ALO-381	X	d	ea smpl	1/mtrix	ea AB	ea AB	±10	90-110	µg C/g	> 30,000	unknown	I, III								

¹½ SEG SLDG-½ 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, mtrix-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: C-107 Chemical, Radiological, Physical Analytical Requirements

LIQUID ANALYSES															
Project Name		C-107 Push Mode Core Sample				COMMENTS				REPORTING LEVELS					
Plan Number		WHC-SD-WM-TP-290, REV. 0				Homogenization Test -Not Required				FORMAT I					
						Field Blank - Required				FORMAT II					
PROGRAM		PROGRAM CONTACTS				Hot Cell Blank - Not Required				FORMAT III					
A. Safety Screening		Safety Screening				HHF Blank - Required				FORMAT IV					
		TWRS				R. D. Schreiber				FORMAT V					
		222-S Laboratory				J. G. Kristofzski				RCRA Compliance					
		325 Laboratory				S. G. McKinley				Special					
PROGRAM	PRIMARY ANALYSES				SAMPLE ¹ FB & S-LEV LIQ	PREP ²	QUALITY CONTROL ³						CRITERIA		FOR- MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE			DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	
A	DSC	Energy	LA-514-113	PNL-ALO-508	X	d	ea smpl	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	ea smpl	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III
A	ICP	Li	LA-505-151	PNL-ALO-211	X	d ⁶	ea smpl	see 7	ea AB	±10	90-110	µg/mL	> 100	1.82 to 2.74	I, III
A	Visual	Organic Layer	LA-519-151	PNL-ALO-501	X	d	N/A	N/A	N/A	N/A	N/A		presence	unknown	I, III
PROGRAM	SECONDARY ANALYSES				SAMPLE ¹ FB & S-LEV LIQ	PREP ²	QUALITY CONTROL ³						CRITERIA		FOR- MAT
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE			DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	
A	Distillation ⁸	CN	LA-695-102	PNL-ALO-287	X	d ⁶	ea smpl	1/mtrix	ea AB	±10	90-110	µg/mL	> 39,000 ¹⁰	unknown	I, III
A	IC ⁹	Br	LA-533-105	PNL-ALO-289	X	d ⁶	ea smpl	1/mtrix	ea AB	±10	90-110	µg/mL	> 1,200	unknown	I, III
A	RSST ⁸	Energy	see 8 below	PNL-ALO-212	X	d	N/A	N/A	ea AB	±10	90-110	J/g ⁵	> 481	unknown	I, III
A	Hot Persulfate ⁸	TOC	LA-342-100	PNL-ALO-381	X	d ⁶	ea smpl	1/mtrix	ea AB	±10	90-110	µg C/mL	> 30,000 ¹⁰	824 to 1,240	I, III

A5.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank C-107 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 C-107. 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 C-107 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 Single-Shell Tank (Core Sampling) Projects
R. D. Schreiber	TWRS Characterization Support	Tank C-107 Tank Characterization Plan Cognizant Engineer
H. Babad	WHC 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, 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). However, the soon to be released revision of the safety screening DQO 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 (481 J/g) (dry weight basis). 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 (dry weight basis).

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 1994). In addition, the Pretreatment Program specifically requested a 2000 mL composite solid sample from tank C-107, resulting in the taking of an additional core sample from one of the two risers to be sampled.

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 determined by DSC (in cal/g or J/g) 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 $\mu\text{Ci/g}$ 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):

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

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 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 of sludge composite material from each of the two core samples taken from Risers 3 and 7, in addition to 2000 mL of the solid composite material from an additional core to be taken. This material will be used 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.

A7.0 DELIVERABLES

All analyses of tank C-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 C-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 C-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% 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 exceeding their notification limits shall be reported by calling the East Tank Farm Operations Shift Manager at 373-2689 and the Characterization Program (Schreiber 1994b). This verbal notification must be followed within 1 working day by written communication, documenting the observations, to Analytical Services, Characterization Support, Characterization Program Office, Safety Screening Representative, and Waste Tanks Process Engineering (Schreiber 1994c). Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above by a revision to this document, 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 Support, Characterization Program Office, Waste Tank Process Engineering, and the Los Alamos Technical Associates, Tank Characterization Resource Center, and Tank Characterization Database representatives (Schreiber 1994c) 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 records. 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 package.

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

A9.0 REFERENCES

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