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2. ECN Category (mark one)		3. Originator's Name, Organization, MSIN, and Telephone No. R. D. SCHREIBER, 71520, R2-12, 373-5589		4. Date 1-23-95
Supplemental Direct Revision Change ECN Temporary Standby Supersedure Cancel/Void		5. Project Title/No./Work Order No. TANK 241-BY-106 TANK CHARACTERIZATION PLAN	6. Bldg./Sys./Fac. No. 241-BY	7. Approval Designator Q
		8. Document Numbers Changed by this ECN (includes sheet no. and rev.) WHC-SD-WM-TP-279, REV. 0D	9. Related ECN No(s). N/A	10. Related PO No. N/A
11a. Modification Work <input type="checkbox"/> Yes (fill out Blk. 11b) <input checked="" type="checkbox"/> No (NA Blks. 11b, 11c, 11d)		11b. Work Package No. N/A	11c. Modification Work Complete N/A	11d. Restored to Original Condition (Temp. or Standby ECN only) N/A
		Cog. Engineer Signature & Date		Cog. Engineer Signature & Date
12. Description of Change Complete revision due to changes in Ferrocyanide Safety Program requirements and addition of sampling and analysis requirements for additional core sample to be taken out of riser 10B.				
13a. Justification (mark one) As-Found		Criteria Change <input checked="" type="checkbox"/> <input type="checkbox"/> Facilitate Const.	Design Improvement <input type="checkbox"/> <input type="checkbox"/> Const. Error/Omission	Environmental <input type="checkbox"/> <input type="checkbox"/> Design Error/Omission
13b. Justification Details Changes were necessary due to a revision of the Ferrocyanide Program requirements.				
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1. ECN (use no. from pg. 1)

617832

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Functional Design Criteria	<input type="checkbox"/>	Stress/Design Report	<input type="checkbox"/>	Health Physics Procedure	<input type="checkbox"/>
Operating Specification	<input type="checkbox"/>	Interface Control Drawing	<input type="checkbox"/>	Spares Multiple Unit Listing	<input type="checkbox"/>
Criticality Specification	<input type="checkbox"/>	Calibration Procedure	<input type="checkbox"/>	Test Procedures/Specification	<input type="checkbox"/>
Conceptual Design Report	<input type="checkbox"/>	Installation Procedure	<input type="checkbox"/>	Component Index	<input type="checkbox"/>
Equipment Spec.	<input type="checkbox"/>	Maintenance Procedure	<input type="checkbox"/>	ASME Coded Item	<input type="checkbox"/>
Const. Spec.	<input type="checkbox"/>	Engineering Procedure	<input type="checkbox"/>	Human Factor Consideration	<input type="checkbox"/>
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OM Manual	<input type="checkbox"/>	Operational Safety Requirement	<input type="checkbox"/>	ICRS Procedure	<input type="checkbox"/>
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Safety Equipment List	<input type="checkbox"/>	Cell Arrangement Drawing	<input type="checkbox"/>	Process Flow Chart	<input type="checkbox"/>
Radiation Work Permit	<input type="checkbox"/>	Essential Material Specification	<input type="checkbox"/>	Purchase Requisition	<input type="checkbox"/>
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Document Number/Revision	Document Number/Revision	Document Number Revision
N/A	N/A	N/A

20. Approvals

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		<u>ADDITIONAL</u>	

RELEASE AUTHORIZATION

Document Number: WHC-SD-WM-TP-217, REV.1

Document Title: TANK 241-BY-106 TANK CHARACTERIZATION PLAN

Release Date: January 26, 1995

**This document was reviewed following the
procedures described in WHC-CM-3-4 and is:**

APPROVED FOR PUBLIC RELEASE

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SUPPORTING DOCUMENT1. Total Pages **2/59**

2. Title TANK 241-BY-106 TANK CHARACTERIZATION PLAN	3. Number WHC-SD-WM-TP-217	4. Rev No. 1
5. Key Words CHARACTERIZATION, FERROCYANIDE, COMPATIBILITY, SAFETY SCREENING, SINGLE-SHELL TANK, SAMPLING, ANALYSIS, TANK CHARACTERIZATION PLAN, WATCH LIST	6. Author Name: R. D. SCHREIBER <i>R.D. Schreiber</i> Signature	
		Organization/Charge Code 71520/N4168

7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, PNL 325 Analytical Chemistry Laboratory, and WHC 222-S Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of samples for tank 241-BY-106.

8. RELEASE STAMP

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RECORD OF REVISION

(1) Document Number

WHD-SD-WM-TP-217

Page 1

(2) Title

TANK 241-BY-106 TANK CHARACTERIZATION PLAN

CHANGE CONTROL RECORD

Tank 241-BY-106

Tank Characterization Plan

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

by
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8633 Gage Boulevard
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LIST OF ABBREVIATIONS

ACL	Analytical Chemistry Laboratory
CW	waste from dissolution of zircaloy (after 1964) or aluminum cladding at the PUREX plant
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
DST	double-shell tank
EB	evaporator bottoms
EVAP	post-1976 designation for evaporator feed
IC	ion chromatography
ICP	inductively coupled plasma - atomic emission spectroscopy
IX	ion exchange waste from cesium recovery process at B plant
NCPLX	noncomplexed waste
RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
SST	single-shell tank
BY-106	tank 241-BY-106
TGA	thermogravimetric analysis
TOC	total organic carbon
TWRS	Tank Waste Remediation System
USQ	unreviewed safety question
WHC	Westinghouse Hanford Company

1.0 SPECIFIC TANK OBJECTIVES

1.1 RELEVANT SAFETY ISSUES

The Double-Shell Tank (DST) System currently receives waste from the Single-Shell Tank (SST) System in support of SST stabilization efforts or from other on-site facilities which generate or store waste. Waste is also transferred between individual DSTs. The mixing or commingling of potentially incompatible waste types at the Hanford Site must be addressed prior to any waste transfers into the DSTs. The primary goal is to prevent the formation of an Unresolved Safety Question (USQ) as a result of improper waste management.

Tank 241-BY-106 (BY-106) is an SST currently on the Ferrocyanide Watch List and which shall undergo stabilization operations in the near future. Two issues related to the overall problem of waste compatibility must be evaluated:

- Assurance of continued operability during waste transfer and waste concentration.
- Assurance that we shall not create safety problems as a result of commingling wastes under interim storage.

The results of the grab sampling activity prescribed by this Tank Characterization Plan shall help determine whether tank BY-106 waste can be transferred without creating a USQ or creating operational problems. The potential for four kinds of safety problems shall be addressed: criticality, flammable gas accumulation, energetics, and corrosion and leakage. Operational problems include plugged pipelines and equipment, exceeding the heat load limits of the receiving tank, and transuranic segregation.

1.1.1 BY-106 Characterization Objectives

The characterization efforts applicable to this Tank Characterization Plan are focused on the preparation of future tank stabilization activities, including the resolution of the waste compatibility issue of tank BY-106. To identify a potential receiver tank for the liquid transferred from tank BY-106, analyses shall be performed on the grab samples obtained from tank BY-106. These analyses are discussed in section 4.0. Only decisions based on sampling and analysis of waste from tank BY-106 shall be addressed within this document; issues such as plugged pipelines and equipment problems are not within the scope of this Tank Characterization Plan. Once the characterization of tank BY-106 has been performed, the waste compatibility assessment shall be conducted. This effort is discussed in *Tank Farm Waste Compatibility Program* (Carothers 1991).

1.1.2 Waste Compatibility Program Data Quality Objective

The document, *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994) describes the process used to develop a data quality objective (DQO) for the waste compatibility issue, as well as the analytical requirements for determining waste compatibility. Since samples shall be taken from only one riser which shall not provide an adequate representation of the waste, tank BY-106 is not being safety screened. Since only one riser is being sampled and the Ferrocyanide DQO (Buck et al. 1993) requires that two risers be sampled, the Ferrocyanide Safety Program's DQO is not applicable. Therefore, the waste compatibility program DQO (Carothers 1994) is the only applicable DQO for this sampling event.

2.0 TANK, WASTE, AND SAMPLING INFORMATION

This section summarizes some of the available information for tank BY-106. Discussions of the process history of the tank, as well as general information about the tank are included.

2.1 AGE AND PROCESS HISTORY OF TANK BY-106

SST BY-106 was constructed between 1948 and 1949, and began to receive waste in the second quarter of 1953. The tank capacity of BY-106 is 750,000 gallons. Table 1 summarizes the fill history from when BY-106 was first placed on active status to the present time.

2.2 EXPECTED TANK CONTENTS

Tank BY-106 should have two primary layers of waste. The bottom layer stored in BY-106 should be ferrocyanide sludge from the uranium recovery process (TBP). This waste type has very high concentrations of sodium, nitrate, and aluminum, while having a very low concentration of plutonium. The upper layer should be saltcake, formed from the precipitation of the slurry product from the evaporators, called evaporator bottoms (EB). EB waste is mainly sodium nitrate (73.8%) and water (12.8%) (Anderson 1992).

Table 1: Fill History of Tank BY-106^a

Qtr:Year	Waste Type and Description	Total Volume (kgals)
2:1953-3:1954	Tank on active status, received 1C waste	756
4:1954	Pumped to trench for TBP scavenge sludge	15
1:1955-3:1957	Multiple liquid transfers to and from WR vault, BC ditch, #10 ditch, and 241-WR; liquid waste composed of TBP	739
4:1957-2:1961	Transfers to BC-2 crib; liquid waste composed of TBP	249
4:1971-4:1968	Multiple liquids transfer from C and BY farms; liquid waste composed of TBP and CW waste	253
1:1959-2:1970	Received waste from BY-105; liquid waste composed of CW and EB	743
3:1970-2:1976	Multiple liquid transfers from ITS; liquid was composed of EB waste	681
1:1977-2:1977	Salt Well Pumping; waste categorized as EVAP	593
3:1977	Inactive	626
1:1994	Waste categorized as NCPLX waste	642

^a From (Anderson 1992 & Hanlon 1994)

2.3 SAMPLING INFORMATION

Tank BY-106 is a Ferrocyanide Watch List single-shell tank scheduled to be grab sampled to prepare for stabilization. These samples shall be obtained using a typical weighted-bottle sampler. Three 100 mL samples shall be taken from saltwell riser 13A. Table 2 presents the applicable sampling information:

Table 2: Tank BY-106 Sampling Information

Sample Number	Sample Type	Sample Location	Sample Depth
6BY-0694-1	Supernatant	Riser 13A (saltwell screen)	Within 1 foot of the surface
6BY-0694-2	Supernatant	Riser 13A (saltwell screen)	Within 2 feet of the middle
6BY-0694-3	Supernatant	Riser 13A (saltwell screen)	Within 1 foot of the bottom

For detailed information regarding the tank BY-106 grab sampling activities, refer to work package 2E-94-00684. This work package shall contain all the applicable operating procedures and the chain of custody records for this sampling event.

With respect to sampling quality control, no field/trip blank shall be taken during this sampling event. Due to the order of magnitude of the analyte results expected, contamination shall not have a significant effect (Sutey 1994).

Current records indicate that there are 642 kgals of waste in tank BY-106. The waste consists of 95 kgals of ferrocyanide sludge and 547 kgals of saltcake. There are 235 kgals of drainable liquids, of which 213 kgals are pumpable liquids (Hanlon 1994). This volume of waste corresponds to 241 inches of waste. However, the most current manual tape readings and the interstitial liquid level corresponds to a waste height of 243.75 inches. Tank BY-106 has been partially interim isolated and is classified as an assumed leaker with respect to tank integrity (Hanlon 1994).

3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

A flowchart showing the general analysis scheme for tank BY-106 is presented as Figure 1. Each step in the flowchart shall be performed on all three grab samples. The steps are described in detail to provide the laboratory chemist with specific sample analysis instructions. Grab sample analyses may not need to be performed in the hot cell (based on radioactivity). If the sample must be analyzed in the hot cell, a hot cell blank shall be required and would be analyzed for the same analytical suite as the samples; otherwise, no blank is necessary. The reporting levels for analyses are contained in Table 3 and are detailed in Section 7.0 of this document.

The following steps shall be taken by the performing laboratory for the sample receipt and analysis of the BY-106 grab samples:

- Step 1 — Transport and receive three grab samples at the laboratory. The receipt of the samples is discussed in Section 4.2.3, "Sample Custody" of this document.

- Step 2 — Record any visual observations such as color and clarity of the liquid, and the presence of any solid particles in the liquid sample.
- Step 3 — Closely inspect the liquid sample for the presence and approximate volume of any solids. If no solids exist, proceed to Step 5. However, if solids are recovered from the liquid, go to Step 4A.
- Step 4A — Remove a portion of the liquid sample and determine the volume percent solids by centrifugation.
- Step 4B — If greater than 1 gram of solid sample is recovered, archive these solids for possible future analyses (Strong 1992).
- Step 5 — Closely inspect the liquid sample for the presence and approximate volume of any separable liquid layers. If no separable liquid layers exist, proceed to Step 7. However, if separable layers exist, go to Step 6.
- Step 6 — Any visual floating layer shall be reported immediately to the East Tank Farm Operation Shift Manager at 373-2689. The floating layer shall be separated and retained in a jar. The next steps shall be performed on the aqueous layer alone.
- Step 7 — Remove sufficient aliquots and then perform those analyses shown in Table 3.
- Step 8 — Retain a 40 mL composite of any remaining liquid sample as the liquid archive (Strong 1992).

3.1 INSUFFICIENT GRAB SAMPLE

In the unlikely event that the sample volume from tank BY-106 is found to be insufficient to perform the requested analyses in Table 3, Characterization Support and Analytical Services shall be notified (for points of contact, see Section 5.0, Table 4). A prioritization of the analyses required in this Tank Characterization Plan is given in Section 6.2. Any analyses prescribed by this document, but not performed, shall be identified in the appropriate data report.

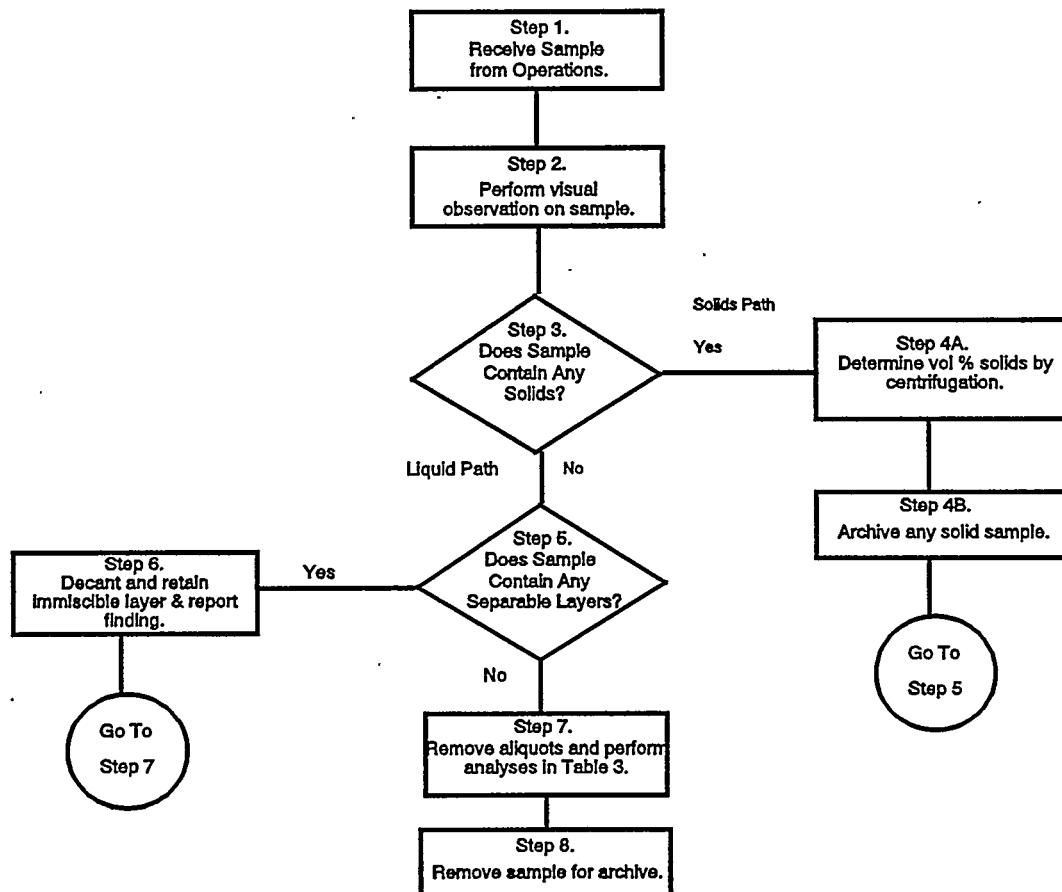


Figure 1: Test Plan Flowchart for Tank BY-106.

4.0 SPECIFIC ANALYTE, QUALITY CONTROL, AND DATA CRITERIA

4.1 SPECIFIC METHODS AND ANALYSES

Table 3 summarizes the analyses to be performed on the tank BY-106 grab samples. The procedure numbers which shall be used in the analyses are included in the table. These analyses are based on the Waste Compatibility Program's DQO (Carothers 1994). None of the analyses are being used to support regulatory compliance; therefore, there are no required regulatory determinations.

4.2 QUALITY ASSURANCE/QUALITY CONTROL

4.2.1 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/quality control involved in analyzing the BY-106 tank waste samples. In the event that analyses must be performed at the 325 ACL, the analysis shall be guided by their quality assurance plan (Kuhl-Klinger 1994).

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 3. If no criteria are provided in Table 3, the performing laboratory shall perform to its quality assurance plan(s).

4.2.2 Sample Collection

Three grab samples from tank BY-106 are to be taken and shipped to the laboratory by Sampling Operations in accordance with work package 2E-94-00684. That work package shall initiate the chain-of-custody for the samples. The following documents shall be used as guidance in the handling and shipment of the tank BY-106 liquid grab samples:

- T0-100-052, "Segregate, Package, and Inventory Radioactive Waste"
- WHC-CM-2-14, "Responsibilities and Procedures for all Hazardous Material Shipments"
- WHC-SD-TP-SARP-001, "Sample Pig Transport System Safety Analysis Report for Packaging (onsite)"
- WHC-SD-WM-HSP-002, "Tank Farm Health and Safety Plan"

Samples shall be identified by a unique number before being shipped to the 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 shall send the samples to the laboratory within 72 hours of removing the sample from tank BY-106. Sampling Operations is responsible for verbally notifying the laboratory (373-2435 for 222-S Laboratory; 373-6704 for 325 ACL) at least 24 hours in advance of an expected shipment. If samples are going to be delivered after 3:00 pm, the laboratory shall be notified at least four hours in advance of actual sample shipment so that proper shift operations can be planned.

4.2.3 Sample Custody

The chain-of-custody form is initiated by the sampling team as described in the work package. Grab samples are shipped in a bottle and sealed with a Waste Tank Sample Seal. All sample shipments are to be labeled with the following information:

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 Westinghouse Hanford 222-S Laboratory is described in procedure LO-090-101. Receipt and control of samples in the 325 ACL is described in procedure PNL-ALO-051.

Table 3. BY-106 Grab Samples Chemical,

Project Name	BY-106 Grab		TANK	# GRABS	GRAB #							
Plan Number	WHC-SD-WM-TP-217, REV 1				BY-106	3	1,2, and 3					
Charge Code	WHC - N54D2; PNL - TBD											
Program Contact	J. M. Jones											
TWRS Contact	R. D. Schreiber											
Lab. Project Coordinator	R. E. Vogel											
ANALYSES					PREPARATION							
METHOD	ANALYTE	WHC PROCEDURE	PNL PROCEDURE	UNITS	ACID	FUS	H ₂ O	OTHER				
Titration	OH ⁻ ⁴	LA-211-102	PNL-ALO-228	M				direct ⁵				
TIC	CO ₃ ²⁻	LA-622-102	PNL-ALO-381	μg/mL				direct ⁵				
Ion Chromatography	SO ₄ ²⁻ , NO ₃ ⁻ , NO ₂ ⁻ , Cl ⁻ , F ⁻ , PO ₄ ³⁻	LA-533-105	PNL-ALO-212	μg/mL				direct ⁵				
DSC/TGA	net exo. energy	LA-514-113 LA-560-112	PNL-ALO-508	cal/g				direct ⁵				
ICP	Na, Al, Fe	LA-505-151	PNL-ALO-211	μg/mL				direct ⁵				
Furnace Oxidation	TOC	LA-344-105	PNL-ALO-381	μg/mL				direct ⁵				
GEA	Cs-137	LA-548-121	PNL-ALO-450	μCi/mL				direct ⁵				
Sep. & β count	Sr-90	LA-220-101	PNL-ALO-431 PNL-ALO-433	μCi/mL				direct ⁵				
Sep. & α count	Pu-239/240	LA-503-156	PNL-ALO-421 PNL-ALO-423	μCi/mL				direct ⁵				
Sep. & α count	Am-241	LA-503-156	PNL-ALO-426	μCi/mL				direct ⁵				
Centrifugation	vol % solids	LA-519-132	PNL-ALO-504	%				direct ⁵				
pH	[H ⁺]	LA-212-103	PNL-ALO-225	none				direct ⁵				
SpG	liquid density	LA-510-112	PNL-ALO-501	none				direct ⁵				

¹ ea - each, DUP - duplicate, spk/msd - spike or matrix spike duplicate, mtrix - matrix, AB - analytical batch, N/A - not applicable.

² Serial dilutions rather than matrix spikes shall be performed to assess accuracy.

³ Tracer or carrier is used in place of a spike and results corrected for recovery.

⁴ OH⁻ shall

⁵ Direct liqu

⁶ Action lim
laboratory

⁷ A hot cell
shall be ar

Radiological and Physical Analytical Requirements

COMMENTS				REPORTING LEVELS				
nogenization Test - Not Required d Blank - Not Required Cell Blank - Not Required ⁷				FORMAT I		Early Notify		
				FORMAT II		Process Control		
				FORMAT III		Safety Screen		
				FORMAT IV		Waste Management		
				FORMAT V		RCRA Compliance		
				FORMAT VI		Special		
QUALITY CONTROL ¹			CRITERIA				REPORT FORMAT	
UP	SPK/MSD	BLANK	STD	DUP PRECN	SPK RECVRY	ACTION LIMIT	EXPECTED RANGE	
mpl	N/A	ea PB	ea AB	± 20%	N/A	$[\text{OH}^-] \leq 0.01\text{M}$ or $\geq 8\text{M}$	not available	I, II
mpl	N/A	ea PB	ea AB	± 20%	N/A	none	770-990 $\mu\text{g/mL}$	II
mpl	ea mpl	ea PB	ea AB	± 20%	80 - 120%	$[\text{NO}_2] \leq 460\mu\text{g/mL}$ or $\geq 253000\mu\text{g/mL}$; $[\text{NO}_3] \geq 6200\mu\text{g/mL}$	not available	I, II
mpl	N/A	N/A	ea AB	± 20%	N/A	net exo. energy $> 0^{\circ}$	not available	II
mpl	N/A ²	ea AB	ea AB	± 20%	N/A	none	Na: 33,000-44,000 $\mu\text{g/mL}$ Al: 1.1-110 $\mu\text{g/mL}$	II
mpl	1/mtr ^x	ea AB	ea AB	± 20%	80 - 120%	$> 10,000\mu\text{g/mL}$	3850-4400 $\mu\text{g/mL}$	II
mpl	N/A	ea AB	ea AB	± 20%	N/A	none	.11-.275 $\mu\text{Ci/mL}$	II
mpl	1/mtr ^x ³	ea AB	ea AB	± 20%	N/A	none	3.3-8.8 $\mu\text{Ci/mL}$	II
mpl	1/mtr ^x ³	ea AB	ea AB	± 20%	N/A	$> 0.8\mu\text{Ci/mL}$.143 -.165 $\mu\text{Ci/mL}$	II
mpl	1/mtr ^x ³	ea AB	ea AB	± 20%	N/A	$> 0.1\mu\text{Ci/g}$.044 -.055 $\mu\text{Ci/mL}$	II
mpl	N/A	N/A	N/A	± 20%	N/A	none	not available	II
mpl	N/A	N/A	ea AB	± 20%	N/A	none	9.5 - 10.5	I, II
mpl	N/A	N/A	ea AB	± 20%	N/A	$> 1.3\text{ g/mL}$	1.07 - 1.10 g/mL	II

be run if pH < 12.

samples may be diluted in acid or water to adjust to proper sample size.

as applicable up to 500 °C. If the energetics action limit is exceeded,

ersonnel and J. M. Jones shall decide if adiabatic calorimetry shall be performed.

nk shall be required if the sample is processed in the hot cell rather than a hood. If a hot cell blank is required, it
zed by the same analysis suite as the samples.

5.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank BY-106 characterization project are listed in Table 4.

Table 4: Tank BY-106 Project Key Personnel List.

Individual	Organization	Responsibility
J. G. Kristofzski	222-S Analytical Operations	Program Support Manager of Analytical Operations
K. L. Silvers	325 Analytical Chemistry Laboratory	Project Manager for Double Shell Tank (Grab Sampling) Projects
R. D. Schreiber	TWRS Characterization Support	BY-106 Tank Characterization Plan Cognizant Engineer
J. M. Jones	East Systems Engineering	Sampling and Compatibility Cognizant Engineer
J. L. Deichman	Analytical Services	Manager of Analytical Services Program Management and Integration
East Tank Farm Operation Shift Manager	Tank Farm Operations	200 East Tank Farm Point of Contact if Action Limit is Exceeded (373-2689)

6.0 EXCEPTIONS AND PRIORITIES

6.1 EXCEPTIONS FOR TANK BY-106

In the Waste Compatibility DQO, several necessary specifications concerning sampling and analysis were omitted. These clarifications are addressed in (Sutey 1994).

Since historical information exists to adequately address the potential for line plugging and precipitation of solids during the transfer of waste, no viscosity and cooling curve analyses shall be required during this analysis activity (Jones 1994).

6.2 PRIORITIES FOR TANK BY-106

In order to complete the compatibility assessment for tank BY-106, results from all of the analyses in Table 3 must be received. Therefore, if insufficient sample is retrieved, the tank shall need to be resampled at a later date. However,

analyses are still requested on any sample obtained, and should be performed in the following order:

- (1) Criticality Analyses: Pu-239/240, Am-241, ICP, and volume percent solids
- (2) Flammable Gas Accumulation: Density
- (3) Energetics: DSC/TGA
- (4) Corrosion Purposes: OH⁻, IC, and pH
- (5) TOC
- (6) Heat Generation: Sr-90 and Cs-137
- (7) Other analyses listed in Table 3

7.0 DELIVERABLES

All analyses of tank BY-106 waste material shall be reported as Formats I and II as shown in Table 3. The Waste Compatibility Program may have requested progress reports from the laboratory regarding the analyses via this Tank Characterization Plan. However, due to the rapid turn around time required for the BY-106 analyses (see Section 7.2), no reports shall be required from the laboratory. The data shall be reported in the units given by Table 3, and all procedure and revision numbers used in the analyses shall be included in the report. Additional information regarding reporting formats is given in (Schreiber 1994).

7.1 FORMAT I REPORTING

Table 3 contains the notification limits for each analyte. OH⁻, NO₃⁻, and NO₂⁻ results that exceed the notification limits defined in the DQO processes, as well as any observed separable floating layers, shall be reported immediately by calling the East Tank Farm Operations Shift Manager at 373-2689. This verbal communication must be followed within 24 hours by a formal letter to J. M. Jones of the East Systems Engineering Program, J. L. Deichman of Analytical Services, and R. D. Schreiber of Characterization Support, documenting the observations. Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above either by a revision to this document or by letter of instruction.

If a notification limit exists for an analysis, but no Format I reporting is required, the results of these analyses which exceed the notification limits shall be flagged in the data report.

7.2 FORMAT II REPORTING

The data found from these analyses shall determine whether or not waste from tank BY-106 is compatible with the waste in the DST receiving tanks. Due to the immediate necessity of the data, the 222-S Laboratory has agreed to have the analysis completed and results reported within 60 days of receipt of the sample at the laboratory loading dock. If the samples are sent to 325 ACL, the results shall be reported within 90 days of receipt of the last sample from tank BY-106 by the laboratory. The results shall be reported using a Laboratory Information Systems (LIMS) report or electronically to J. M. Jones. Although no data validation, supporting raw data, or quality control results are to be included, the results still require review and approval by the cognizant scientist or manager of the laboratory operation. In addition to the LIMS/electronic report, a letter report shall be sent to R. D. Schreiber and J. M. Jones summarizing the results. Any observations taken during the receipt and analysis of the grab samples should be included in this letter report.

8.0 CHANGE CONTROL

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

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

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APPENDIX A

SAMPLING EVENT B: ROTARY CORE SAMPLE IN FISCAL YEAR 1994

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SAMPLE EVENT B: ROTARY SAMPLING IN FISCAL YEAR 1994

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LIST OF ABBREVIATIONS

ACL	Analytical Chemistry Laboratory
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
DST	double-shell tank
GEA	Gamma Energy Analysis
HPGE/MCA	High Purity Germanium - multi channel analysis
IC	ion chromatography
ICP	inductively coupled plasma - atomic emission spectroscopy
PNL	Battelle Pacific Northwest Laboratory
PUREX	Plutonium-Uranium Extraction plant
QC	quality control
RSST	Reactive System Screening Tool
SST	single-shell tank
TGA	thermogravimetric analysis
TOC	total organic carbon
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

A1.0 SPECIFIC TANK CHARACTERIZATION OBJECTIVES

Process records indicate that the underground storage tank 241-BY-106 (BY-106) on the Hanford Site contains ferrocyanide from in-tank processing. Consequently, tank BY-106 is one of 20 tanks currently on the ferrocyanide Watch List. The characterization effort described in this Tank Characterization Plan will aid in the resolution of the Ferrocyanide Safety Issue. Further, the results of the sampling activity prescribed by this Tank Characterization Plan shall help determine if there are any as yet unidentified safety issues associated with this tank. In addition, the results shall help determine to which double shell tank (DST) liquid waste from tank BY-106 may be transferred without creating a safety or operational problem.

A1.1 RELEVANT SAFETY ISSUES

The relevant safety issues with tanks on the Ferrocyanide Watch List are as follows:

- 1) The potential for an uncontrolled exothermic reaction occurring between complexes of ferrocyanide and nitrate or nitrite that could result in a release of radioactive material.
- 2) The possibility that other, as yet unidentified, safety issues exist for the tank.

Two issues related to the overall problem of waste compatibility must also be evaluated:

- 1) Assurance of continued operability during waste transfer and waste concentration.
- 2) Assurance that safety problems are not created as a result of commingling wastes under interim storage.

A1.1.1 Tank BY-106 Characterization Objectives

This characterization effort of tank BY-106 is focused on the relevant safety issues above. Resolution of the Ferrocyanide Safety Issue is the ultimate goal of the Ferrocyanide Safety Program. That program has identified two key safety questions that should be answered from analytical data on the tank's waste (Postma, et al 1994):

- (1) "Is a significant exothermic reaction possible during interim storage?"
- (2) "Is a significant exothermic reaction possible under present conditions of waste moisture content?"

Each tank on the Ferrocyanide Watch List has been conservatively categorized as either Safe or Conditionally Safe by the Ferrocyanide Safety Program. The answers to the above questions will allow each tank's categorization to be revised.

To satisfy other objectives of this sampling and analysis effort, the tank contents shall be safety screened in order to identify any other potential safety issues associated with tank BY-106, and to ensure that the tank should not be placed on an additional Watch List.

In addition, this characterization effort is applicable to the preparation of future tank stabilization activities, including resolution of the waste compatibility issue of tank BY-106. To identify a potential receiver tank for the liquid to be transferred from tank BY-106, analyses shall be performed on any liquid obtained from the rotary mode core samples.

Finally, the Pretreatment Program has requested sample material for process development purposes. The Project focuses on the development of an enhanced sludge washing process, and will be guided by a test plan (Lumetta 1994; Temer 1994).

A1.1.2 Ferrocyanide, Safety Screening, Compatibility, and Pretreatment Data Quality Objectives

The sampling and analytical needs associated with ferrocyanide tanks, pretreatment, compatibility, as well as the safety screening of all tanks have been identified through the Data Quality Objective (DQO) process. Additional data needs associated with tank BY-106 may be identified in subsequent DQO efforts, which may then be incorporated into future sampling events.

Pertinent documents to this effort include the following:

- (1) *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994), which describes the sampling and analytical requirements for tanks on the Ferrocyanide Watch List, including tank BY-106.
- (2) *Safety Screening Data Quality Objective* (Babad and Redus 1994), which describes the sampling and analytical requirements for screening waste tanks for unidentified safety issues. The criteria for placing a tank on a particular Watch List are enumerated in that document.
- (3) *Data Quality Objectives for the Waste Compatibility Program* (Carothers 1994), which describes the process used to develop a DQO for the waste compatibility issue, as well as the analytical requirements for determining waste compatibility.
- (4) *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994), which describes the sampling and analytical requirements to support the TWRS technical strategy by identifying the chemical and physical composition of the waste in the tank. In addition, the DQO works to guide development efforts to define waste pretreatment processes, which will in turn define high-level and low-level waste feed to vitrification processes. This DQO, at the request of the pretreatment program, will have a limited use in this sampling and analysis plan. Refer to Section A6.1.

A1.1.2.1 Data Quality Objectives Integration

The ferrocyanide, pretreatment, and safety screening DQO efforts have compatible sampling and sample breakdown requirements. Two core samples taken from risers located approximately 180° apart and near the outer edge of the tank meet the DQO requirements for sampling. For the tank BY-106 sampling event, two risers are available. From each riser one core sample shall be taken. Thirteen segments are expected from each core.

For sample breakdown, both the safety screening DQO and the ferrocyanide DQO request primary analyses (DSC and TGA) on a quarter segment level; however, the safety screening DQO requires analyses on a half segment level as well. In addition, the pretreatment DQO requires composites for the Pretreatment Program's specific requests.

The compatibility DQO addresses only liquid samples, and has an alternate set of analyses that need to be implemented to satisfy its requirements. Therefore, analyses that satisfy the waste compatibility DQO, are listed in the liquids section of Table A-3.

It should be noted that for this Tank Characterization Plan, the Pretreatment Program has requested that only a sample for archive and a sample for process development be obtained. For further information, refer to Section A6.1.

If any notification limits for immediate reporting of analytes identified in the DQO efforts were conflicting, the most stringent limits of the DQO efforts were used in this Tank Characterization Plan.

A2.0 TANK, WASTE, AND SAMPLING INFORMATION

This section gives a summary of information describing tank BY-106. Included is the age of the tank, process history, and the risers which will be used for sampling. The number of cores, segments, and potential layers for tank BY-106 will also be discussed.

Previous characterization data on ferrocyanide tanks are available in *Compendium of Characterization Data on Ferrocyanide Tanks* (Sathyanarayana 1993) and references therein. The fill history information is available in *A History of the 200 Area Tank Farms* (Anderson 1990).

A2.1 AGE AND HISTORY OF TANK BY-106

Single Shell Tank BY-106 was constructed between 1948 and 1949, and began to receive waste in the second quarter of 1953. The capacity of tank BY-106 is 750,000 gallons. Table A-1 summarizes the fill history from when tank BY-106 was first placed on active status to the present time.

Table A-1: Tank BY-106 Fill History¹

Qtr:Year	Waste type and Description	Total Volume (kgals)
2:1953-3:1954	Tank on active status receive 1C waste	756
4:1954	Pumped to trench for TBP scavenge sludge	15
1:1955-3:1957	Multiple liquid transfers to and from WR vault, BC ditch, # 10 ditch, and 241-WR; liquid wastes composed of TBP	739
4:1957-2:1961	Transfers to BC-2 crib; liquid waste composed of TBP	249
4:1961-4:1968	Multiple liquid transfer to and from C and BY farms; liquid waste composed of TBP and CW waste	253
1:1959-2:1970	Received waste from BY-105; liquid waste composed of CW and EB waste	743
3:1970-2-1976	Multiple liquid transfers ITS; liquid composed of EB wastes	681
1:1977-2:1977	Salt Well Pumping; waste categorized as EVAP	593
3:1977	Inactive	626
1:1994	Waste categorized as NCPLX waste	642

¹(Anderson 1990 and Hanlon 1994)

A2.2 EXPECTED TANK CONTENTS

Tank BY-106 is expected to have two primary layers of waste. The bottom layer should be ferrocyanide sludge from the uranium recovery process (TBP). This

waste type has high concentrations of sodium, nitrate, and aluminum, and a very low concentration of plutonium is present. The upper layer is expected to be saltcake formed from precipitation of slurry product from the evaporator, called evaporator bottoms (EB) waste. EB waste is mainly sodium nitrate (73.8%) and water (12.8%) (Anderson 1990).

Analyses of the supernate in tank BY-106 were conducted in the first quarter of 1991. A summary of the analytical data from those analyses is reported in Table A-2 (Edrington 1991).

Table A-2: Tank BY-106 Liquid Analysis Results

DENSITY (g/ml)	1.46		
ANALYTE	µg/mL	µg/g	
Ag	55	38	
Al	29400	20137	
As	2.00	1.37	
Ba	21	14	
Bi	315	216	
Ca	14	9.4	
Ce	822	563	
CO ₃	17400	11918	
Cr	404	277	
Cu	50	34	
Fe	53	37	
K	5750	3938	
Mg	158	108	
Mo	82	56	
Na	144000	98630	
NO ₂	66400	45479	
NO ₃	118000	80822	
Ni	50	34	
OH	49300	33767	
Se	0.35	0.24	
Si	288	197	
Sn	95	65	
SO ₄	32900	22534	
TOC	3280	2247	
Ta	137	94	
Ti	34	23	
ANALYTE	Bq/L	µCi/mL	µCi/g
^{89/90} Sr	4.07E+06	1.10E-01	1.26E-03
⁹⁹ Tc	3.22E+06	8.70E-02	1.00E-03
¹³⁷ Cs	1.15E+10	3.11E+02	3.57E+00

A2.3 SAMPLING INFORMATION

The volume of solid waste in tank BY-106 is 642 kgals, which equals approximately 241 inches. However, the most recent reading indicates a waste level of 243.75 inches from the bottom of the tank. The waste consists of 95 kgals of ferrocyanide sludge and 547 kgals of saltcake. There are 235 kgals of drainable liquids, of which 213 kgals are pumpable liquids (Hanlon 1994).

One core sample from Riser #10B and one core sample from Riser #5 shall be collected from tank BY-106. Each core is expected to consist of thirteen segments. Segments 2 to 13 should be 19 inches in length. The first segment from the core is expected to be approximately 15.5 inches. It should be noted that the sampling objective is to obtain a vertical profile of the waste; therefore, more or less segments may be taken depending on the accuracy of the volume estimates above. The exhauster necessary to perform rotary mode sampling will be placed in Riser 7. If, for some reason, it becomes necessary to change the riser(s) for sampling or exhauster placement, this change must be made by the sampling cognizant engineer and kept in a controlled document such that traceability is preserved.

Sampling shall be conducted according to procedures and documentation included in the tank BY-106 Work Package 2E-94-0808 using rotary mode core sampling equipment.

Occasionally, the bit used for the rotary core sampling method may become plugged; therefore, 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) discussed below.

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 POP-080-090 (*Load/Transport Sample Casks*). Core samples shall be transported to the laboratory within three working days of the removal of each segment from the tank.

A3.0 SAMPLE EXTRUSION AND BREAKDOWN INSTRUCTIONS

A3.1 Tank-Specific Analytical Procedures

A flowchart depicting the general sample breakdown and analysis scheme is presented in Figures A-2, A-3, A-4, and A-5. Each step in the flowchart will be performed on each segment from tank BY-106. 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-3 require a 60 day reporting time, as noted. The 60 day reporting format, Format VI, is explained in section A7.4.

Any decisions, observations, or deviations made to this work plan or the flowchart during the sample removal 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-3.

It should be noted that in accordance with the Safety and Analysis Report for Packaging (SARP), samples from tank BY-106 must be vented every 47 days from the time of cask sealing to allow any retained gas to escape.

Step 1 Receive rotary 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 sample(s).
- Record observations. This may include a sketch of the extruded core sample and written documentation of pertinent, descriptive information, such as color, texture, homogeneity, consistency, and type (sludge or saltcake)
- Note the color and clarity of any drainable liquid.
- Take color photographs and/or a videotape to visually document the extruded core segments or auger samples.
- Note sludge/saltcake interface.

Step 3 Does the segment contain drainable liquid?

YES: Proceed to Step 4A

NO: Proceed to Step 5

Step 4A Separate any drainable liquid from the solids by allowing the liquid to drain into a bottle. Measure the weight and volume. Retain drainable liquids for further processing.

Step 4B Is the segment 100% drainable liquid?

Yes: Proceed to Step 20

No: Proceed to Step 5

SOLIDS PATH

Step 5 Does the core segment contain any saltcake?

Yes: Proceed to Step 6A

No: Proceed to Step 7

Step 6A Is there both sludge and saltcake in the segment?

Yes: Proceed to Step 6B

No: Proceed to Step 6C

Step 6B Separate segment at the sludge/saltcake interface and keep separate for all analyses. Retain sludge for further breakdown in Step 7. For remaining saltcake, proceed to step 6C.

Step 6C Is there more than 20 g of saltcake?

Yes: Proceed to Step 6D

No: Proceed to Step 6E

Step 6D Divide the saltcake into two equal subsegments (i.e., half segments).

Step 6E Homogenize each saltcake subsegment from Step 6D using the appropriate procedure.

Step 6F Collect sufficient aliquots from each saltcake subsegment to perform the appropriate analyses listed in Table A-3 in duplicate.

Step 6G Archive at least 10 mL and up to 20 mL of each homogenized saltcake subsegment (Bratzel 1994). If insufficient material is available to perform the required analyses and meet archiving needs, notification should be given as directed in Section A3.2.

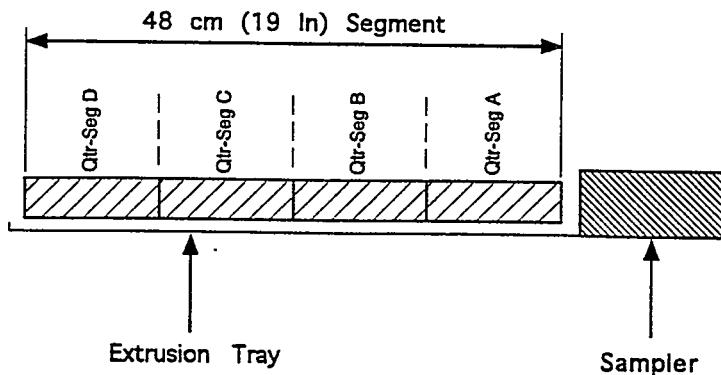
Step 6H Was the solid material entirely composed of saltcake?

Yes: Proceed to Step 20 (Steps 7-19 are to be performed on the sludge material only.)

No: Proceed to Step 7

Step 7 Divide the remaining sludge material from the segment into four 12 cm quarter segments. Figure 1 illustrates how a typical segment sample will be extruded and divided into quarter segments. The first 12 cm segment extruded from the sampler originates from the lowest portion of the tank relative to the other segments and therefore shall be assigned to the fourth quarter segment and shall be uniquely identified as (Tank - Core No. - Segment No. - D). The following three 12 cm sections of the extruded segment shall be labeled as C, B, and A, respectively. An example of this labeling protocol for the third quarter segment from the first segment of the first core would be (BY-106 - Core 1 - Segment 1 - B). If the extruded segment is less than 48 cm long, then the same labeling convention shall still apply until no solid material is left to make a complete 12 cm quarter segment. The first 12 cm extruded (i.e., bottom portion of the core) shall still be assigned to the D quarter segment, etc.

Figure A-1: Tank BY-106 Segment Breakdown



Step 8 Homogenize each quarter segment of sludge from Step 7 using the appropriate, approved procedure.

Step 9 Will a homogenization test be performed?

YES: Proceed to Step 10

NO: Proceed to Step 11

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

Step 10 Conduct the homogenization test by taking a 1 to 2 g subsample from widely separated locations of the homogenized quarter segment. Conduct the homogenization test in accordance with Bell (1993).

Step 11 Collect sufficient aliquots from each homogenized quarter segment to perform the appropriate preparations and analyses listed in Table A-3 in duplicate.

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

Step 12 Remove at least 10 mL and up to 20 mL of each homogenized quarter segment for the archive sample (Bratzel 1994).

Step 13 Combine the remainder of quarter segments A and B to create a half segment. Do the same with quarter segments C and D.

Step 14 Homogenize each sludge half segment from Step 13 using the appropriate, approved procedure.

Step 15 Collect sufficient aliquots from each homogenized half segment to perform the appropriate preparations and analyses listed in Table A-3 in duplicate.

Step 16 Combine portions of each half segment proportional to the sludge recovery of each segment to build a core composite. This composite must be large enough to provide sample for the appropriate composite analyses in Table A-3, and include 125 mL of material for process development and 100 mL of archive.

Step 17 Remove 125 mL of the solid composite for process development work (see Section A6.2).

Step 18 Remove 100 mL of the solid composite as the Pretreatment solid composite archive (Bratzel 1994).

LIQUIDS PATH

Step 19 Closely inspect the liquid sample obtained in Step 4 for the presence and approximate volume of any potential organic layers. Does the sample contain any immiscible (potentially organic) layers?

YES: Proceed to Step 20
NO: Proceed to Step 22

Step 20 Report any visually observed immiscible (potential organic) layer immediately by the early notification system.

Step 21 Separate and retain the potential organic layer for possible future analysis.

NOTE: Steps 22 through 29 shall be performed on the remaining (probable aqueous) liquid layer only.

Step 22 Filter the remaining liquid sample through a 0.45 micron filter.

Step 23 Is there greater than 1 gram of solid on the filter?

YES: Proceed to Step 24
NO: Proceed to Step 25

Step 24 Archive the solids for possible future analysis (Bratzel 1994).

Step 25 Remove sufficient aliquots from the segment-level liquid sample to perform the appropriate analyses listed in Table A-3 in duplicate.

Step 26 Archive at least 10 mL and up to 20 mL of the segment-level drainable liquid as the segment level liquid archive (Bratzel 1994).

Step 27 Combine portions of each segment-level liquid sample to build a liquid composite.

Step 28 Remove 100 mL of the liquid composite as the pretreatment liquid composite archive (Bratzel 1994).

PRIMARY ANALYSIS PATH

Step 29 Perform primary analyses as listed in Table A-3.

Step 30 Compare the primary analysis data with notification limits.

Step 31A Do the results exceed the notification limits (Table A-3)?

YES: If the results exceed the notification limits, proceed to step 31B.
NO: If results do not exceed the notification limits, proceed to Step 34.

Step 31B Report results exceeding notification limits using Format I reporting deliverable requirements (Section A7.2).

SECONDARY ANALYSIS PATH

Step 32 Perform secondary analyses according to Table A-3.

Step 33A Do the results of secondary analyses exceed the notification limits?

 YES: Proceed to Step 33B
 NO: Proceed to Step 34

Step 33B Report results for the secondary analyses which exceed the notification limits using Format I reporting deliverable requirements (Section A7.2).

REPORTING PATH

Step 34 Report and deliver data obtained using reporting requirements (Section A7.0).

AMPLE EXTRUSION AND BREAKDOWN

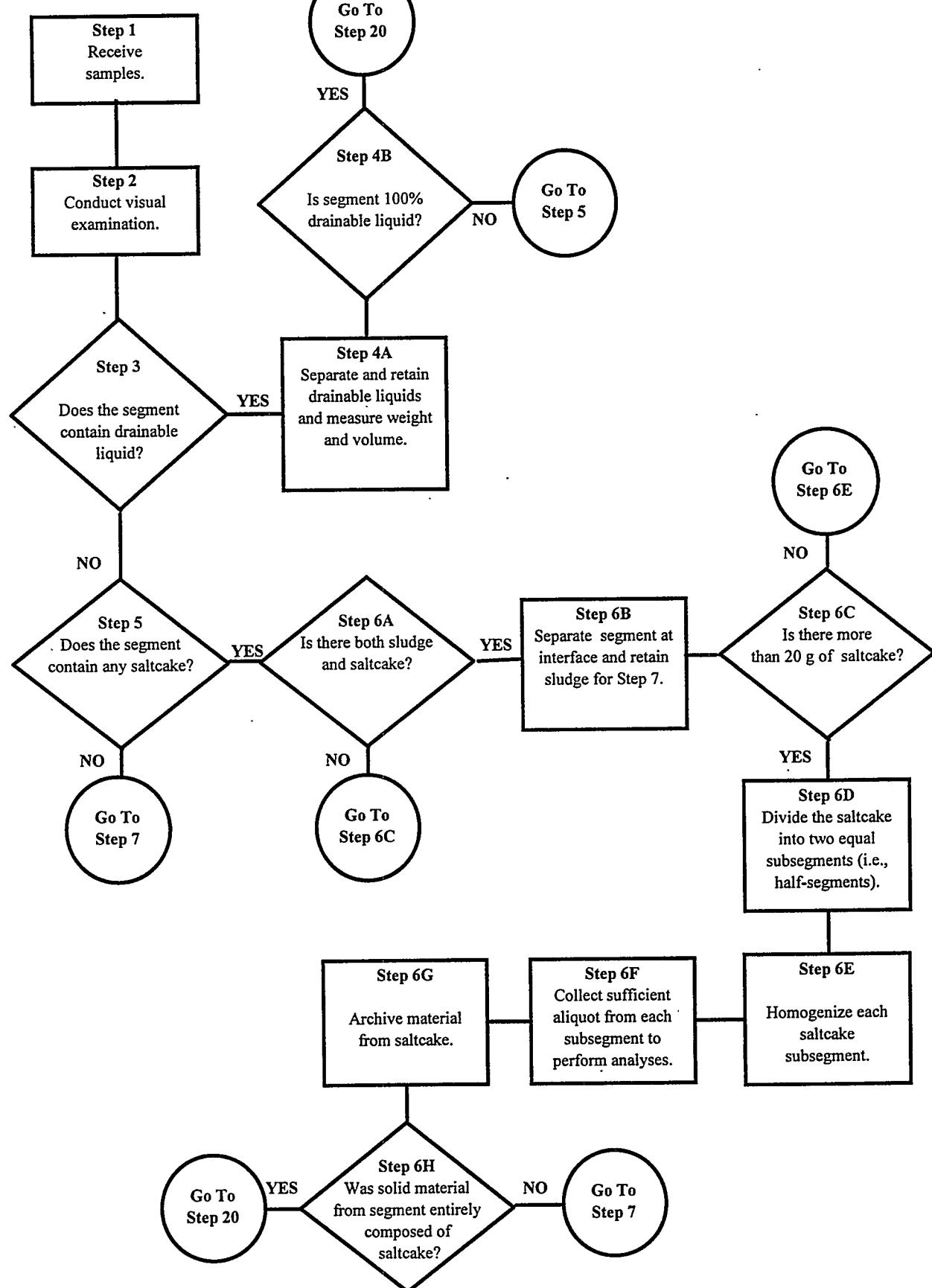


Figure A-2: Sample Extrusion and Breakdown Flow Chart

SLUDGE PATH

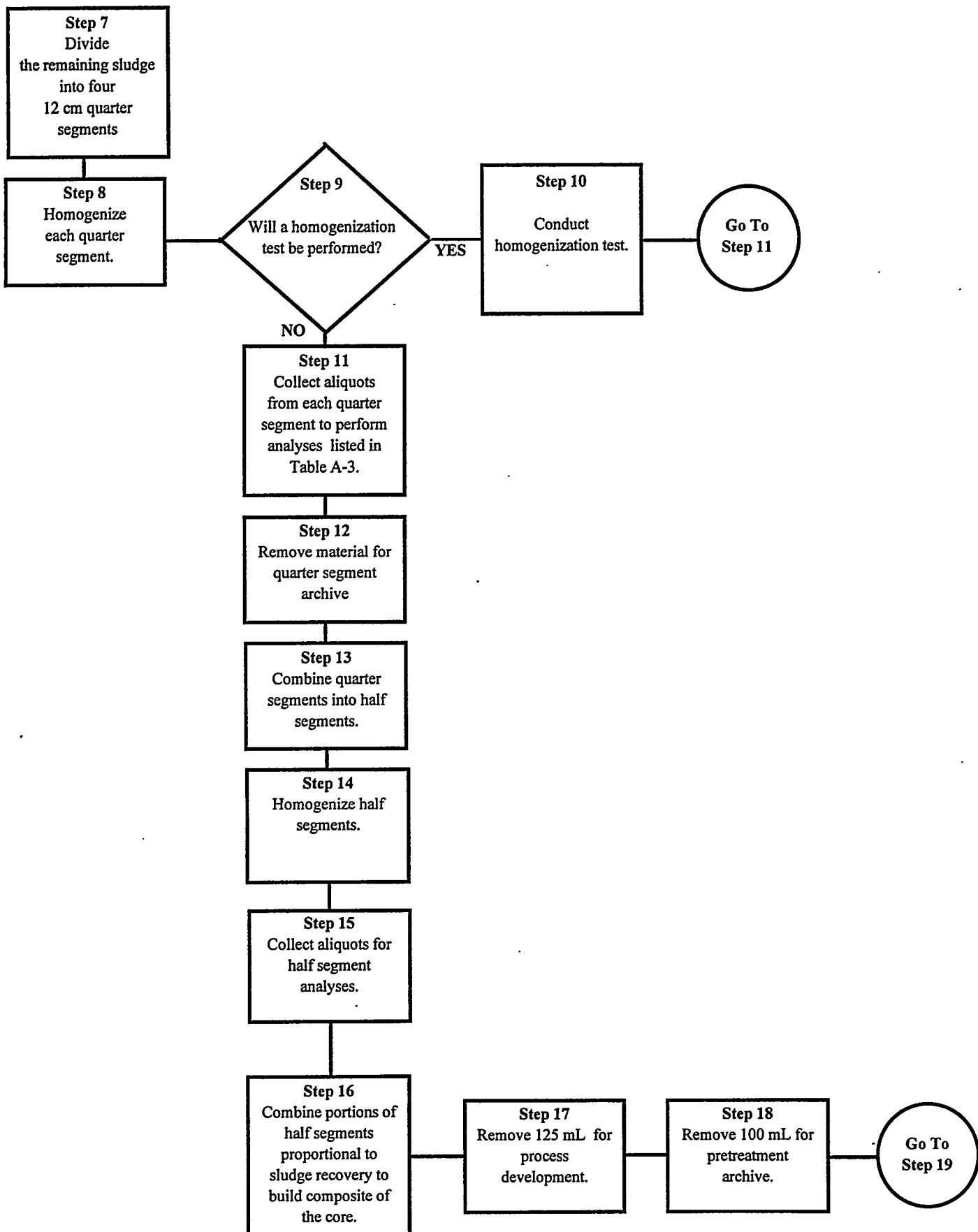


Figure A-3: Sludge Analysis Flow Chart

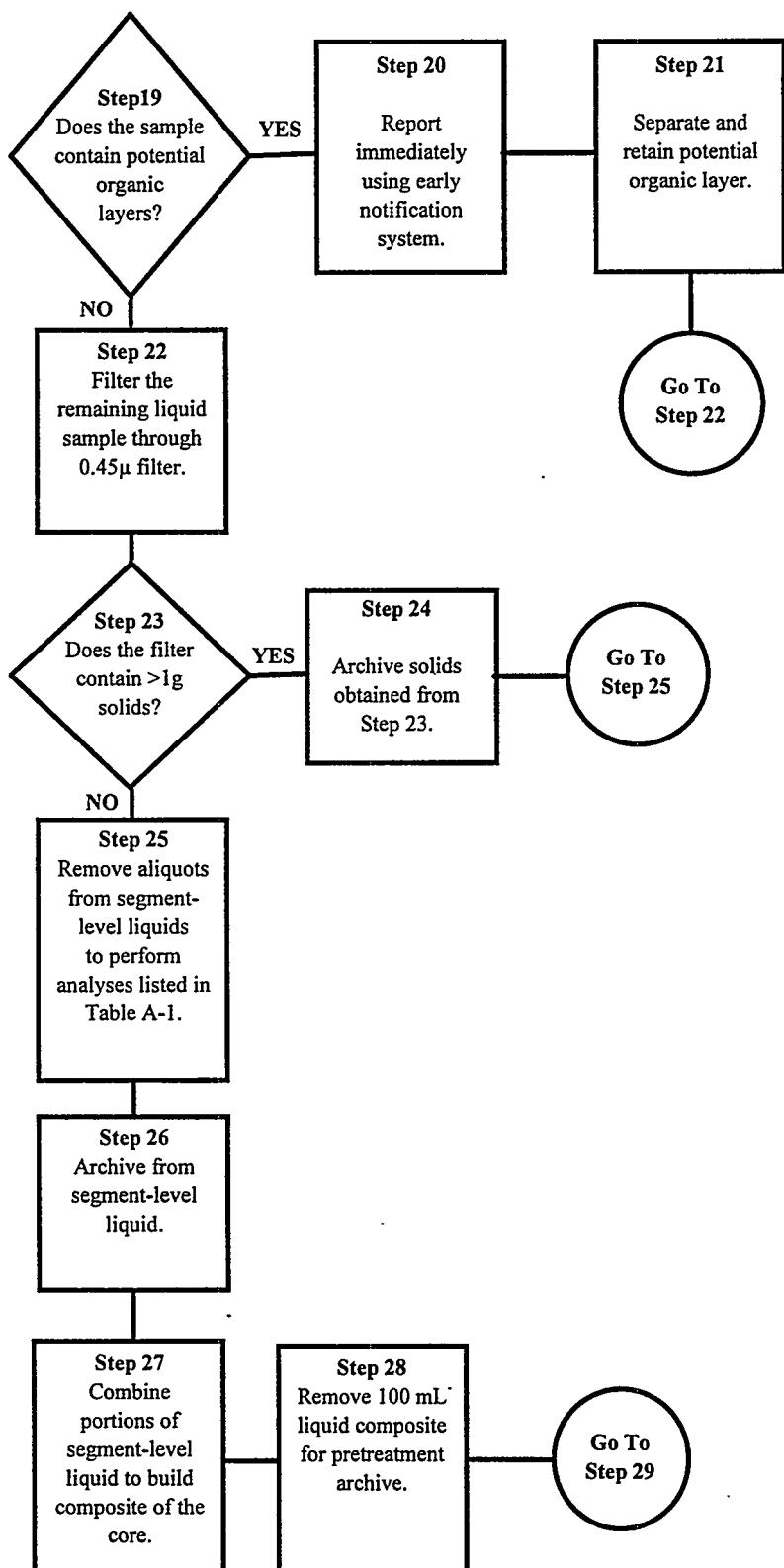


Figure A-4: Liquid Analysis Flow Chart

ANALYSIS AND REPORTING PATH

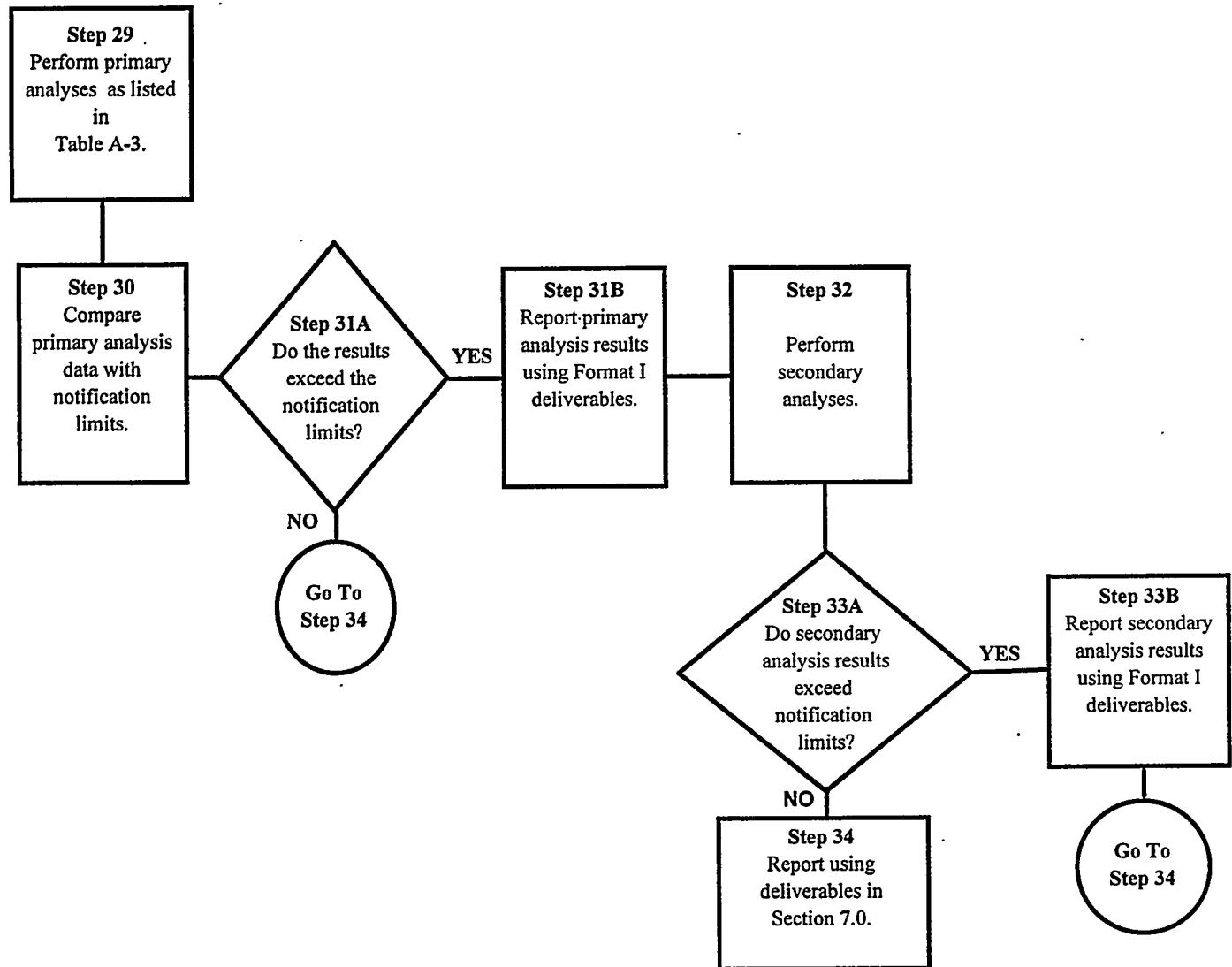


Figure A-5: Sample Analysis and Reporting Flow Chart

A3.2 Insufficient Segment Recovery

If the amount of material recovered from the core samples taken from tank BY-106 is insufficient to perform the analyses requested and permit a minimum 20 gram archive per segment, the laboratory project coordinator or project manager shall notify the Tank Cognizant Engineer and the manager of Analytical Services, Program Management and Integration 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 sample breakdown procedures as provided in section A3.1. The priority levels have been categorized to account for all three matrices and are listed below. Refer to Table A-3 for the method of analysis.

Sludge Matrix

PRIORITY LEVEL 1

The DSC, TGA, total organic carbon by direct persulfate, cyanide, nickel, and total alpha analyses on subsegments shall be performed.

PRIORITY LEVEL 2

The secondary analyses on subsegments shall be performed:

Saltcake Matrix

PRIORITY LEVEL 1

DSC and TGA analyses on the subsegments shall be performed.

PRIORITY LEVEL 2

Secondary analyses on the subsegments shall be performed.

Liquid Matrix

PRIORITY LEVEL 1

All safety screening and compatibility analyses listed on Table A-3 for segment-level liquids shall be performed.

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

A4.1 SPECIFIC METHODS AND ANALYSES

The analyses in Table A-3 to be performed on waste from tank BY-106 are based on the Ferrocyanide, Safety Screening, and Compatibility DQOs referenced in Section A1.1.2 with the exception of the clarifications made in Section A6.1.

A4.2 QUALITY ASSURANCE/QUALITY CONTROL

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/quality control of analyzing the waste tank core samples at the 222-S Laboratory. If the PNL 325 Laboratory is the performing laboratory, the analyses shall be guided by the 325 Laboratory Quality Assurance Project 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-3. If no criteria are provided in Table A-3, the performing laboratory shall perform to its quality assurance plan.

A4.2.2 Sample Collection

Two core samples with an expected thirteen segments each are to be taken and shipped to the performing laboratory by Sampling Operations in accordance with Work Package 2E-94-0808. That work package shall also initiate the chain-of-custody for the samples. Approved rotary mode sampling procedures WTWP-94-034 and WTWP-94-035, and procedure T0-080-090 ("Load/Transport Sample Cask[s]") are to be used during this 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 inclusion into 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 shall count as a segment. Sampling Operations is responsible for verbally notifying the 222-S shift manager at the laboratory (373-2435) at least 24 hours in advance of an expected shipment. If the samples are going to be delivered to PNL 325 Laboratories (376-2639), the laboratory shall be notified at least 72 hours in advance of the actual sample shipment so that proper shift operations can be planned.

A4.2.3 Sample Custody

The chain-of-custody form is initiated by the sampling team as described in Work Package 2E-94-0808. Core samples are shipped in a cask and sealed with the Waste Tank Sample Seal. All sample shipments are to be labeled with the following information:

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 is described in laboratory procedure L0-090-101. Receipt and control of samples in the 325 Analytical Chemistry Laboratory (ACL) is described in procedure PNL-AL0-051.

Table A-3: BY-106 Chemical, Radiolo

Project Name		BY-106 Rotary Mode Core Sample					
Plan Number		WHC-SD-WM-TP-217, REV. 1					
PROGRAM CONTACTS							
A. Compatibility		J. M. Jones					
B. Ferrocyanide		J. E. Meacham					
C. Safety Screening		H. Babad					
TWRS		R. D. Schreiber					
222-S Laboratory		J. G. Kristofzski					
325 Laboratory		S. G. McKinley					
PRIMARY ANALYSES				SAMPLE ¹			PREP ²
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	1/4 SEG SLDG	1/2 SEG SLDG	1/2 SEG SC	
DSC	Energy	LA-514-113	PNL-ALO-508	X		X	d
TGA	% H ₂ O	LA-560-112	PNL-ALO-508	X		X	d
Alpha	Total Alpha	LA-508-101	PNL-ALO-421 PNL-ALO-420		X		f
Distillation	CN	LA-695-102	PNL-ALO-285	X ¹²			d
ICP	Ni	LA-505-151	PNL-ALO-211	X ¹⁶			f or a
ICP ¹⁸	Li	LA-505-151	PNL-ALO-211		X		f or w
Direct Persulfate	TOC	LA-342-100	PNL-ALO-381	X ¹²			d
SECONDARY ANALYSES				SAMPLE ¹			PREP ²
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	1/4 SEG SLDG	1/2 SEG SLDG	1/2 SEG SC	
Distillation	CN	LA-695-102	PNL-ALO-285	X ¹³		X ¹⁷	d
IC ⁷	Br	LA-533-105	PNL-ALO-212		X		w
Direct Persulfate	TOC	LA-342-100	PNL-ALO-381		X ¹³	X ¹⁷	d
Alpha ¹⁵	Total Alpha	LA-508-101	PNL-ALO-421 PNL-ALO-420	X			f
Sep. & α counting ¹¹	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422		X		f
ICP ¹¹	Fe Mn U	LA-505-151	PNL-ALO-211		X		f or a
RSST ¹⁰	Energy	see 10 below	N/A	X ¹³		X ¹⁷	d
HPGE/MCA	GEA	Cs-137	LA-548-121	PNL-ALO-450	X		f
Sep. & β counting ¹⁵	Sr-90	LA-220-101	PNL-ALO-431 PNL-ALO-430	X			f or a
ICP ¹⁵	Al Ca Fe Na P	LA-505-151	PNL-ALO-211	X			f or a

Chemical and Physical Analytical Requirements

SOLID ANALYSES						REPORTING LEVELS		
Comments						FORMAT I	Early Notify	
Organization Test - Per Laboratory Discretion						FORMAT II	Process Control	
Blank - Required						FORMAT III	Safety Screen	
Blank - Per Laboratory Discretion						FORMAT IV	Waste Management	
						FORMAT V	RCRA Compliance	
						FORMAT VI	Special	
TANK			#RISERS					
BY-106			2					
QUALITY CONTROL ³					CRITERIA		FOR-MAT	PRO-GRAM
SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
N/A	N/A	ea AB	±10	90-110	J/g ⁵	> 481	unknown	I, VI B,C
N/A	N/A	ea AB	±10	90-110	wt%	<((0.0932*DSC)-10.7)	unknown	I, VI B,C
1/mtr ^x	ea PB	ea AB	±20	80-120	µCi/g ⁵	> 41	unknown	I, VI C
1/mtr ^x	ea AB	ea AB	±10	90-110	µg/g ⁵	> 39,000	unknown	I, VI B
see ⁹	ea PB	ea AB	±10	90-110	µg/g ⁵	none	unknown	VI B
see ⁹	ea PB	ea AB	±10	90-110	µg/g	> 100	unknown	I, VI B, C
1/mtr ^x	ea AB	ea AB	±20	80-120	µg/g ⁵	> 30,000	unknown	I, VI B
QUALITY CONTROL ³					CRITERIA		FOR-MAT	PRO-GRAM
SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	
1/mtr ^x	ea AB	ea AB	±10	90-110	µg/g ⁵	> 39,000	unknown	I, VI C
1/mtr ^x	ea PB	ea AB	±10	90-110	µg/g	> 1200 ¹⁰	unknown	I, VI, IV B, C
1/mtr ^x	ea AB	ea AB	±20	80-120	µg/g ⁵	> 30,000	unknown	I, VI C
1/mtr ^x	ea PB	ea AB	±20	80-120	µCi/g ⁵	> 41	unknown	I, IV B
1/mtr ^x ⁸	ea PB	ea AB	±15	85-115	µCi/g ⁵	> 41	unknown	I, VI C
see ⁹	ea PB	ea AB	±10	90-110	µg/g ⁵	none	unknown	VI C
N/A	N/A	ea AB	±20	80-120	J/g ⁵	> 481	unknown	I, VI C
N/A	ea PB	ea AB	±10	90-110	µCi/g ⁵	none	unknown	IV B
1/mtr ^x ⁸	ea PB	ea AB	±10	90-110	µCi/g ⁵	none	unknown	IV B
see ⁹	ea PB	ea AB	±10	90-110	µg/g ⁵	none	unknown	IV B

Table A-3: BY-106 Chemical, Radiolog

SECONDARY ANALYSES				SAMPLE ¹			PREP ²		
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	1/4 SEG SLDG	1/2 SEG SLDG	1/2 SEG SC		DUP	SM
IC ¹⁵	Cl ⁻ F ⁻ NO ₂ ⁻ NO ₃ ⁻ PO ₄ ³⁻	LA-533-105	PNL-ALO-212	X			w	ea smpl	1/
Direct Persulfate ¹⁵	TC	LA-342-100	PNL-ALO-381	X			d	ea smpl	1/
Direct Persulfate ¹⁵	CO ₃ ²⁻	LA-342-100	PNL-ALO-381	X			d	ea smpl	1/
Beta ¹⁵	Total Beta	LA-508-101	PNL-ALO-430 PNL-ALO-431	X			a	ea smpl	1/
Germanium Detector ¹⁵	GEA ¹⁴	LA-548-121	PNL-ALO-450	X			a	ea smpl	1/
Sep. & α counting ¹⁵	Pu-238 Pu-239/240	LA-503-156	PNL-ALO-422 PNL-ALO-423	X			f	ea smpl	1/
Sep. & α counting ¹⁵	Am-241	LA-503-156	PNL-ALO-422 PNL-ALO-489	X			f	ea smpl	1/
Phosphorescence ¹⁵	U, total	LA-925-009	PNL-ALO-445	X			f	ea smpl	1/
Bulk Density ¹⁵	Density	LA-510-112	PNL-ALO-501	X			d	ea smpl	1/

¹1/4 SEG SLDG-1/4 segment, sludge; 1/2 SEG SLDG-1/2 segment, sludge; 1/2 SEG SC-1/2 segment, saltcake²d-direct, f-fusion, a-acid, w-water³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike or matrix spike duplicate, AB-anal⁴Units for notification limits and expected range are those listed in the "units" column.⁵Dry weight basis (to be calculated by Ferrocyanide Safety Program, with exception of exothermic energy).⁶Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.⁷This analysis required if Li exceeds notification limit.⁸Tracer or carrier may be used in place of a spike and results corrected for recovery.⁹Either serial dilutions or matrix spikes will be performed.¹⁰If DSC exotherm exceeds 75 cal/g (dry wt. basis), laboratory must notify Ferrocyanide Program to determine if RSST RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.¹¹Performed only if total alpha on 1/2 segment exceeds notification limit.¹²Analyses performed on one 1/4 segment per riser (1/4 segment with the highest DSC exotherm).¹³Analyses performed if DSC exotherm exceeds notification limit (for sludge sample).¹⁴GEA analytes to be reported: Co-60, Eu-154/155¹⁵If a sludge 1/4 segment has a DSC exotherm which exceeds the notification limit, these analyses are to be run on sam¹⁶Ni analysis performed on every other 1/4 segment, beginning with bottommost 1/4 segment.¹⁷Analyses performed if DSC exotherm exceeds notification limit (for saltcake sample).¹⁸If the chain of custody form indicates that HHF fluid with LiBr tracer was used to obtain the segment, Li analysis is to

Chemical and Physical Analytical Requirements

QUALITY CONTROL ³				CRITERIA			FOR-MAT	PRO-GRAM
BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
ea PB	ea AB	±10	90-110	µg/g ⁵	none	unknown	IV	B
ea AB	ea AB	±20	80-120	µg/g ⁵	none	unknown	IV	B
ea AB	ea AB	±20	80-120	µg/g ⁵	none	unknown	IV	B
ea PB	ea AB	±20	80-120	µCi/g ⁵	none	unknown	IV	B
ea PB	ea AB	±20	80-120	µCi/g ⁵	none	unknown	IV	B
ea PB	ea AB	±15	85-115	µCi/g ⁵	none > 41	unknown	I, IV	B
ea PB	ea AB	±20	80-120	µCi/g ⁵	none	unknown	IV	B
ea PB	ea AB	±10	90-110	µg/g ⁵	none	unknown	IV	B
N/A	ea AB	±20	80-120	g/mL ⁵	none	unknown	IV	B

al batch, PB-preparation blank, N/A-not applicable, mtrx-matrix

be run.

from that 1/4 segment of sludge ONLY AFTER CONSULTATION WITH THE FERROCYANIDE PROGRAM.

erformed on that segment.

Table A-3: BY-106 Chemical, Radiolog

Project Name		BY-106 Rotary Mode Core Sample							
Plan Number		WHC-SD-WM-TP-217, REV. 1							
PROGRAM CONTACTS									
A. Compatibility			J. M. Jones			Homogenization T Field Blank - Requ Hot Cell Blank - Pe			
B. Ferrocyanide			J. E. Meacham						
C. Safety Screening			H. Babad						
TWRS			R. D. Schreiber						
222-S Laboratory			J. G. Kristofzski						
325 Laboratory			S. G. McKinley						
PRIMARY ANALYSES					SAMPLE ¹	PREP ²	QUA		
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	FB & S-LEV LIQ		DUP	SPK/MSD		
DSC	Energy	LA-514-113	PNL-ALO-508	X	d ⁶	ea smpl	N/A		
TGA	% H ₂ O	LA-560-112	PNL-ALO-508	X	d ⁶	ea smpl	N/A		
Furnace Oxidation	TOC	LA-344-105	PNL-ALO-380	X ¹³	d ⁶	ea smpl	1/mtrx		
Visual	Organic Layer	LA-519-151	PNL-ALO-501	X	d	N/A	N/A		
HPGE/MCA	GEA	Cs-137	LA-548-121	PNL-ALO-450	X	d ⁶	ea smpl	N/A	
Sep. & β counting	Sr-90		LA-220-101	PNL-ALO-431 PNL-ALO-433	X	d ⁶	ea smpl	1/mtrx ⁸	
ICP	Al Fe Na		LA-505-151	PNL-ALO-211	X	d ⁶	ea smpl	see ⁹	
Titration ¹⁰	OH ⁻	LA-361-103	PNL-ALO-228	X	d ⁶	ea smpl	N/A		
IC	Cl ⁻ F ⁻ NO ₃ ⁻ NO ₂ ⁻ PO ₄ ³⁻ SO ₄ ²⁻	LA-533-105	PNL-ALO-212	X	d ⁶	ea smpl	1/mtrx		
pH	[H ⁺]	LA-212-103	PNL-ALO-225	X	d ⁶	ea smpl	N/A		
TIC	CO ₃ ²⁻	LA-622-102	PNL-ALO-381	X	d ⁶	ea smpl	N/A		
Sep. & α counting	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422	X	d ⁶	ea smpl	1/mtrx ⁸		
Sep. & α counting	Am-241	LA-503-103	PNL-ALO-489 PNL-ALO-422	X	d ⁶	ea smpl	1/mtrx ⁸		
SpG	Density	LA-510-112	PNL-ALO-501	X	d ⁶	ea smpl	N/A		
Centrifugation	% Sol	LA-519-132	PNL-ALO-504	X	d ⁶	ea smpl	N/A		
ICP ¹⁴	Li	LA-505-151	PNL-ALO-211	X	d ⁶	ea smpl	see ⁹		

Chemical and Physical Analytical Requirements

LIQUID ANALYSES					REPORTING LEVELS			
COMMENTS					FORMAT I	Early Notify		
- Per Laboratory Discretion					FORMAT III	Safety Screen		
d Laboratory Discretion					FORMAT IV	Waste Management		
					FORMAT V	RCRA Compliance		
					FORMAT VI	Special		
TANK		#CORES						
BY-106		2						
CRITERIA ³			CRITERIA			FOR-MAT	PRO-GRAM	
BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
N/A	ea AB	±20	80-120	J/g ¹⁵	net > 0'	unknown	I, VI	B, C
N/A	ea AB	±20	80-120	wt%	<((0.0932*DSC)-10.7)	43.2 to 64.8	I, VI	B, C
a AB	ea AB	±20	80-120	µg/mL	> 10,000	2,380 to 3,560	I, II, VI	A, C
N/A	N/A	N/A	N/A	none	presence	unknown	I, II, VI	A, C
a AB	ea AB	±20	80-120	µCi/mL	none	48,100 to 72,100	II	A
a AB	ea AB	±20	80-120	µCi/mL	none	11.2 to 16.8	II	A
a AB	ea AB	±10	90-110	µg/mL	none	4,060 to 6,080 unknown 91,200 to 137,000	II	A
a AB	ea AB	±20	80-120	µg/mL	≤ 170 or ≥ 136,000	166,000 to 248,000	I, II	A
a AB	ea AB	±10	90-110	µg/mL	none none ≥ 62,000 ≤ 500 or ≥ 253,000 none none	unknown unknown 12,600 to 18,800 211,000 to 317,000 unknown 16,000 to 24,000	II II I, II I, II II II	A A A A A A
N/A	ea AB	±20	80-120	none	none	unknown	II	A
a AB	ea AB	±20	80-120	µg/mL	none	1,660,000 to 2,480,000	II	A
a AB	ea AB	±20	80-120	µCi/mL	> 0.8	unknown	I, II	A
a AB	ea AB	±20	80-120	µCi/mL	> 0.1	unknown	I, II	A
N/A	ea AB	±20	80-120	g/mL	> 1.3	1.12 to 1.68	I, II	A
N/A	N/A	±20	80-120	%	none	unknown	II	A
a PB	ea AB	±10	90-110	µg/mL	> 100 ⁵	unknown	I, VI	B, C

Table A-3: BY-106 Chemical, Radiolog

SECONDARY ANALYSES				SAMPLE ¹	PREP ²	QU	
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	FB & S-LEV LIQ		DUP	SPK/MSD
RSST ¹⁰	Energy	see 10 below	N/A	X	d ⁶	ea smpl	N/A
IC ¹²	Br	LA-533-105	PNL-ALO-212	X	d ⁶	ea smpl	1/mtr
Distillation ¹¹	CN	LA-695-102	PNL-ALO-285	X	d ⁶	ea smpl	1/mtr

¹S-LEV LIQ-liquid taken from the segment level; FB - field blank²d-direct, f-fusion, a-acid, w-water³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike or matrix spike duplicate⁴Units for notification limits and expected range are those listed in the "units" column.⁵Action limit converted from weight basis to volume basis assuming liquid density of 1.0 g/mL.⁶Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.⁷Action limit is applicable up to 500 °C. If the energetics action limit is exceeded, laboratory personnel and⁸Tracer or carrier may be used in place of a spike and results corrected for recovery.⁹Either serial dilutions or matrix spikes will be performed.¹⁰RSST performed only if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may¹¹These analyses required if DSC exceeds notification limits.¹²This analysis required if Li exceeds notification limit.¹³TOC is a primary analysis for the compatibility DQO, but also a secondary analysis for the safety screening and reported within 90 days of receipt of the last sample at the laboratory loading dock.¹⁴If the chain of custody form indicates that HHF fluid with LiBr tracer was used to obtain the segment, Li an¹⁵Dry weight basis and original wet weight basis.

Chemical and Physical Analytical Requirements

TEST CONTROL ³				CRITERIA			FORMAT	PROGRAM
BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
N/A	ea AB	±20	80-120	J/g	net > 0 ⁷	unknown	I, VI	C
ea PB	ea AB	±10	90-110	µg/mL	> 1200 ¹⁰	unknown	I, VI	A, B, C
ea AB	ea AB	±10	90-110	µg/mL	> 39,000 ⁵	unknown	I, VI	C

, AB-analytical batch, PB-preparation blank, N/A-not applicable, mtrx-matrix

test Systems Engineering will decide if adiabatic calorimetry shall be performed.

are found in WHC-SD-WM-TP-104.

DOQO. Therefore, if the DSC limit is exceeded, TOC analysis must be performed

ysis is to be performed on that segment.

A5.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank BY-106 characterization project are listed in Table A-4. Procedures for both the WHC 222-S Laboratory and the PNL 325 Analytical Chemistry Laboratory are given in Table A-3 since it is as yet undecided which laboratory shall receive the samples from tank BY-106. 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-4: Tank BY-106 Project Key Personnel List

Individual	Organization	Responsibility
J. L. Deichman	WHC Analytical Services, Program Management & Integration	Manager, Analytical Services, Program Management & Integration
J. G. Kristofzski	222-S Analytical Operations	Program Support Manager of Analytical Operations
S. G. McKinley	325 Analytical Chemistry Laboratory	Project Manager for Tank Waste Characterization
J. E. Meacham	WHC Ferrocyanide Safety Program	Ferrocyanide Waste Tanks Cognizant Engineer
M. J. Kupfer	TWRS Process Engineering	Pretreatment Point of Contact
R. D. Schreiber	TWRS Characterization Support	TWRS Tank BY-106 Cognizant Engineer
Tank Farms Operations Shift Manager	Tank Farms Operations	200 East Tank Farm Operations point of contact if notification limits are exceeded (373-2689)

A6.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

A6.1 EXCEPTIONS TO DQO REQUIREMENTS

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 sample for process development and a 100 mL composite sample for archive shall be obtained from this sampling event (Slankas 1994).

In the safety screening DQO, it is specified that cyanide analyses are to be performed 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 performed 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 performed on a half-segment basis and that notification shall be made if the DSC value exceeds 481 J/g (115 cal/g dry weight basis).

In the ferrocyanide DQO, it is specified that analyses are to be performed and reported "within 216 days of receipt of the last sample". However, in accordance with the *Hanford Federal Facilities Agreement and Consent Order* (Ecology 1989), this Tank Characterization Plan specifies that the analyses be performed and reported within 216 days of the sampling event.

The Ferrocyanide Safety Program is accelerating resolution of the Ferrocyanide Safety Issue to mid fiscal year 1996 (approximately 1 1/2 years earlier than previously planned). Consequently, the need to sample all the ferrocyanide tanks and to perform extensive analyses has been reexamined. As a result, analyses will be limited to the following for sludge: Energetics on every quarter segment, water on every quarter segment, nickel on every other quarter segment (starting with the bottom most quarter segment), and total cyanide/total organic carbon on one quarter segment per core (performed on the quarter segment exhibiting the highest exotherm). Saltcake will still be limited to only energetics and moisture analyses on half segments. Liquids will be analyzed for energetics and moisture on a segment basis. These analyses are necessary to determine the potential for exothermic chemical reactions and to confirm that waste aging has occurred (Cash 1995b; see Section A10.0).

The remaining secondary and tertiary analyses specified in the ferrocyanide DQO are not required if the energy content of the waste is below 481 J/g (115 cal/g) on a dry weight basis. However, if the limit is exceeded, the secondary and tertiary analyses specified in the ferrocyanide DQO shall be performed on a quarter segment level, but ONLY after consultation with the Ferrocyanide Safety Program (Cash 1995b; see Section A10.0).

Rather than specifying accuracy and precision, the Ferrocyanide DQO (Meacham et al. 1994) provides "required analytical uncertainty". The performing laboratory shall achieve accuracy and precision as shown in Table A-3. However, if the analyte is not detected at or above the level listed in Table A-5, except for pH and TGA, the precision and accuracy in Table A-3 for that analysis need not be met. The sensitivities (analyte concentration) given are action limits for the Ferrocyanide Safety Program. If the results of these analyses are below their sensitivities (analyte concentration), with the exception of pH and TGA (see Table A-5), no decisions will be made based on these results. These results must still be reported in the appropriate data report for information only.

Table A-5: Required Sensitivities (Analyte Concentration)

Analyte	Required Sensitivity (Analyte Concentration)
DSC (energetics)	28 cal/g
TGA (% water)	35 wt%
Nickel	500 μ g/g
Total Cyanide	0.5 wt%
Total Organic Carbon	1.0 wt%
Cesium-137	50 μ Ci/g
Strontium-90	50 μ Ci/g
Al, Ca, Fe, P, Na	500 μ g/g
Chloride, Fluoride, Nitrate, Nitrite; Phosphate	500 μ g/g
pH	<4 or >12
Total Carbon	1,200 μ g/g
Total Inorganic Carbon	1,200 μ g/g
Total Alpha	2 μ Ci/g
Total Beta	50 μ Ci/g
Total Gamma	50 μ Ci/g
Pu-238	0.1 μ Ci/g
Pu-239/240	2 μ Ci/g
Am-241	2 μ Ci/g
Co-60	0.1 μ Ci/g
Eu-154/155	5 μ Ci/g
Uranium	1,000 μ g/g
Bulk Density	N/A

The ferrocyanide DQO (Meacham et al. 1994) requires consolidation and particle size analyses on ferrocyanide tanks. A new strategy for resolving the ferrocyanide safety issue early is being written, and these analyses are no longer required (Meacham 1994).

A6.2 CLARIFICATIONS AND ASSUMPTIONS

One clarification must be made with respect to the type of crucible used in the fusion dissolution step of the ICP analysis. Nickel is an analyte of interest to the Ferrocyanide Safety Program. Since the use of a nickel crucible would yield an inaccurate value for the concentration of nickel in the waste, a non-nickel crucible must instead be used.

A number of clarifications and assumptions relating to the notification limits or decision thresholds identified in the applicable DQO efforts needs to be made with respect to the analyses in Table A-3. Each of these issues are discussed below.

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

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

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

- It is assumed that the notification limit for moisture content as given in *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objectives Process* (Meacham et al. 1994) requires that all exotherms in the DSC result be attributed to oxidation of disodium nickelferrocyanide. To convert the notification limit into a number more readily usable by the laboratory, the exotherm must be converted into a percent disodium nickelferrocyanide using the DSC exotherm. This was done as follows:

- The equivalent fuel percentage was calculated according to equation (2). The DSC value is divided by 1430, which is the experimentally-determined heat of reaction of disodium nickelferrocyanide with nitrate in waste simulant (Postma et al. 1994):

$$\text{wt\% Na}_2\text{NiFe(CN)}_6 = \frac{[(\text{DSC exotherm (cal/g dry weight)}]}{1430 \text{ cal/g}} \times 100 \quad (2)$$

- The notification limit then becomes: $\text{wt\%} < [(0.0932 * \text{DSC exotherm}) - 10.7]$.
- 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 $\mu\text{Ci/mL}$ rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, two assumptions and calculations are necessary. All alpha decay will be assumed to originate from Pu-239 and a sample density of 1.50 g/mL is assumed. The notification limit may then be calculated as in equation (3):

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

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

- Neither of the DQO efforts upon which the analyses in Table A-3 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 analyses done on the segment-level liquid.
- The safety screening DQO does not sufficiently address the analyses of any drainable liquid present. To adequately characterize the tank, all analyses performed on solids for the safety screening DQO, with the exception of total alpha analyses, shall also be performed on any drainable liquids and the field blank.
- The Pretreatment Program has requested 125 mL of the solid composite material for process development work. Test plans (Lumetta 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 sample is in a position to adequately 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 letter of instruction, to allow shipment of the sample material to the Process Chemistry section of PNL.

A7.0 DELIVERABLES

All analyses of tank BY-106 waste material will be reported as Format I, II, IV, or VI as indicated in Table A-3. Additional information regarding reporting formats is given in Schreiber (1994a).

A7.1 PROGRESS REPORTS

Each laboratory performing analyses on tank BY-106 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 BY-106, 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. Variances greater than 10% and \$10,000, and 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-3 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 to the Ferrocyanide Safety Program, the Safety Screening Representative, Analytical Services, Characterization Support, Waste Tanks Process Engineering, and the Characterization Program Office documenting the observations (Schreiber 1994c). Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above either by a revision to this document or a memorandum of understanding.

A7.3 FORMAT II REPORTING

The results on the segment level liquid (waste compatibility analysis results) will be analyzed and reported using a Laboratory Information Systems (LIMS) report or electronically to J. M. Jones within 60 days of receipt of the last sample from the first core at the laboratory loading dock. In addition to the LIMS/electronic report, a letter report shall be sent to Characterization Support, the Characterization Program Office, the Tank Characterization Resource Center, and East Systems Engineering summarizing the results (Schreiber 1994c). This report shall be sent within one week of the electronic transmittal to J. M. Jones. Any observations made during sample receipt, and any specific problems or variations to sample analyses, should be included in this letter report.

A7.4 FORMAT VI REPORTING

A Format VI report, reporting the results of the primary analyses, shall be issued to the Ferrocyanide Safety Program, the Safety Screening Representative, Characterization Support, the Characterization Program, Waste Tanks Process Engineering, the Tank Characterization Resource Center Tank Characterization Database, and Los Alamos Technical Associates (LATA) representatives (Schreiber 1994c) within 60 days of receipt of the last sample at the laboratory loading dock. This report shall have the same format and information as a Format III report. The DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analyses for the safety screening secondary analytes are required, these results shall be provided within 90 days of receipt of the last sample at the laboratory loading dock. No calibration data are requested for these reports. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

A7.5 FORMAT IV REPORTING

If secondary analyses are performed for the Ferrocyanide Safety Program (see Table A-3 for delineation of program requests), these results shall be compiled into a Format IV type data package. The data package shall be provided to Analytical Services, the Characterization Program, the Ferrocyanide Safety Program, and the Tank Characterization Resource Center representative within 216 days of the sampling event. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

In addition to this data package, an electronic version of the analytical results shall be provided to the Tank Characterization Resource Center and Tank Characterization Database for entry in the Tank Characterization Database. The data must be available to the Washington State Department of Ecology within 216 days of the sampling event, so this electronic copy must be sent at the time of data package delivery or within 209 days of the sampling event, whichever is

earlier, to allow time for data entry. The electronic version shall be in the standard electronic format specified in (Bobrowski 1994).

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 Support via an Engineering Change Notice to the Tank Characterization Plan. All changes shall also be clearly documented in the final data package.

Additional analysis of core 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 GUIDANCE FOR ADDITIONAL CORE SAMPLE OBTAINED FROM RISER 10B

The first core acquired from Riser 10B resulted in inadequate sample recovery for the laboratories to perform the required analyses. Thirteen samples were taken with recoveries varying from 0 to 95%. Therefore, a test plan for resampling Riser 10B has been written (Bogen 1995) and the analyses requested from the Ferrocyanide Program were condensed and are reflected in this section. The following instructions are only to be used for the second sample obtained from Riser 10B.

A9.1 ADDITIONAL PROGRAM REQUESTS

For this additional core sample, both the ferrocyanide and pretreatment programs have requested either material or analyses. These requests are documented in the following letters (see Section A10.0):

- 1) Letter, R. J. Cash to R. J. Blanchard, 74260-95-009.
- 2) Letter, J. T. Slankas to R. D. Schreiber, TWRS-TDPO-95-004.

A9.2 SAMPLING GUIDANCE

For the additional samples from Riser 10B of tank BY-106, a test plan shall be used (Bogen 1995).

A9.3 LABORATORY GUIDANCE

A9.3.1 Sample Extrusion and Breakdown.

The breakdown required for the program requests stated in (Cash 1995; Slankas 1995) are similar to the breakdown instruction in Section A3.1, and therefore will not be discussed in this section.

A9.3.2 Requested Analyses and Material

(1) FOR ALL BUT THE BOTTOM THREE SEGMENTS IN THE TANK:

(a) If $\geq 60\%$ recovery for a particular segment is obtained, the following analyses will be run:

SLUDGE (none expected): If noticeable sludge **IS** present, run DSC/TGA and Ni on 1/4 segment basis and total alpha on 1/2 segment basis.

SALTCAKE: If there is **NO** noticeable sludge, run DSC/TGA on 1/2 segment basis. (If sample size is slightly less than 19 inches in length, divide the sample into two equal parts.)

LIQUID: DSC only on segment basis.

- Archive 20 mL of material for possible future analysis.
- Retain remaining material for Pretreatment.

(b) If $< 60\%$ recovery for a particular segment is obtained, homogenize total amount, archive 20 mL of material for possible future analysis, and send the remaining material to Pretreatment for process development.

(2) FOR THE BOTTOM THREE SEGMENTS IN THE TANK:

(a) If $\geq 60\%$ recovery for a particular segment is obtained, the following analyses will be run:

SLUDGE/SALTCAKE: DSC/TGA and Ni on 1/4 segment basis. Total alpha on a 1/2 segment basis.

LIQUID: DSC only on segment basis.

- Archive 20 mL of material for possible future analysis.
- Retain remaining material for Pretreatment.

(b) If < 60% recovery for a particular segment is obtained:

SLUDGE:

Recovery: 0% to \approx 30% Treat solids as one subsample and perform DSC/TGA, Ni, and total alpha analyses.

Recovery: 31% to 60% Divide total solids in sample into two equal subsegments and perform DSC/TGA, Ni, and total alpha analyses.

LIQUID: DSC on segment basis; i.e., treat any drainable liquid from an extruded segment as one sample.

- Archive up to 20 mL of each sample regardless of recovery (except zero) after any analyses are complete.
- Retain possible remaining material for Pretreatment.

(3) IN ALL CASES:

If a subsample shows a DSC exotherm $>$ 481 J/g (115 cal/g dry weight basis for solids, original liquid weight basis for liquids), run TOC on the subsample. In addition, if it is a solid subsample from the bottom three segments in which this DSC limit is exceeded, CN analysis shall be performed.

The analyses requested above are a subset of those identified in Table A-3. Therefore, all preparations, methods, quality control, notification limits, and reporting requirements from Table A-3 shall apply.

A9.3.3 Quality Assurance/Quality Control

The quality assurance/quality control documents referenced in Section A4.2.1 shall still be used.

A10.0 ATTACHMENTS

The following memoranda are intended to be used as: (1) interim guidance pertaining to the Ferrocyanide analysis requests until the Ferrocyanide DQO is revised (scheduled release date of April 1995); and (2) as additional guidance regarding Section A9.0.

Westinghouse
Hanford CompanyInternal
Memo

From: Ferrocyanide Safety Program 74260-95-012
 Phone: 373-3132
 Date: January 25, 1995
 Subject: REVISION: ANALYSES ON FERROCYANIDE TANK WASTE SAMPLES

To: T. J. Kelley/S. J. Eberlein S7-30/R2-12
 ccmail: D. C. Board S1-71 D. R. Bratzel S7-31
 J. E. Meacham S7-15 R. D. Schreiber R2-12
 RJC File/LB

Reference: Meacham et al., 1994, *Data Requirements for the Ferrocyanide Safety Issue Developed Through the Data Quality Objectives Process*, WHC-SD-WM-DQO-007, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

The Ferrocyanide Safety Program Data Quality Objectives (DQO) document (Reference) covers a suite of primary, secondary, and tertiary analyses. Under most expected conditions, only a fraction of these analyses are required to provide sufficient data for resolution of the Ferrocyanide Safety Issue. The following reduced set of analyses shall be performed, as specified in the DQO, when the energetics for any solids subsegment (as determined by DSC) are <481 J/g on dry weight basis:

Sludge Solids: Energetics on every quarter segment, moisture on every quarter segment, nickel on every other quarter segment (starting with the bottom-most subsegment of each segment), and total cyanide/total organic carbon on one quarter segment per core (performed on the quarter segment exhibiting the highest exotherm). Archive approximately 20 mL of sample from each quarter segment pending further analyses at a later date.

Saltcake Solids: Energetics and moisture on every half segment. Archive approximately 20 mL of sample from each half segment pending further analyses at a later date.

Free Liquid: Perform DSC/TGA on any free liquid on a segment basis; report DSC values in Joules/g on a dry weight basis. Archive approximately 20 mL of sample from each segment pending further analyses at a later date.

The remaining secondary and tertiary analyses listed in the DQO (Reference) shall not be performed on samples exhibiting <481 J/g on a dry weight basis.

To maintain the schedule for resolution of the Safety Issue, the above analyses and a corresponding report are required within 60 days of receipt of the last segment of the last core from the tank being sampled.

If a sample exhibits an exotherm >481 J/g on a dry weight basis, the remaining secondary and tertiary analyses, listed in the DQO (Reference), shall ONLY be run after consultation with the Ferrocyanide Safety Program Office.

If there are any questions, please call me at 373-3132 or J. E. Meacham at 373-1961.



R. J. Cash

Westinghouse
Hanford Company

Internal
Memo

From: Ferrocyanide Safety Program 74260-95-009
Phone: 373-3132
Date: January 17, 1995
Subject: REVISION 3A: ANALYSES REQUESTED ON SECOND SET OF CORE SEGMENT SAMPLES FROM TANK BY-106, RISER 10B

To: R. J. Blanchard

cc:mail - DR Bratzel FAX - G. Westleigh, LATA (943-6740)
GT Dukelow
JE Meacham
RD Schreiber
RJC-LB/File

This memorandum has been revised (Revision 3A) to remove the requirement for Total Alpha on saltcake material in the top segments. The Safety Screening DQO does not require Total Alpha on saltcake material. We are still requesting it on the bottom three segments, even if it appears to be saltcake, as pointed out below.

Analyses on the second set of segments should only be run on those segments with good recovery (> 60%), except the bottom three segments, as follows:

(1) FOR ALL SEGMENTS EXCEPT THE BOTTOM THREE (3) SEGMENTS FROM RISER 10B:

For \geq 60% recovery for a particular segment:

SLUDGE (None Expected) - If noticeable sludge IS present, divide segment into ~ 4.75 inch subsegments and run DSC/TGA and Ni on each.¹ Total Alpha should be run on a half-segment basis.

SALTCAKE - If there is NO noticeable sludge, divide segment into approximately equal halves; run DSC/TGA on each of the two halves.

DRAINABLE LIQUID - DSC on segment basis; i.e., treat any drainable liquid from an extruded segment as one sample.

Archive approximately 20 mL of each sample after analyses are complete.

For < 60% recovery for a particular segment: Homogenize total amount of whatever sample is obtained and archive 20 mL.

¹Try to divide sample into at least 3 or 4 subsegments depending on amount of material and position in the sampling tube. Each subsegment except the last should be approximately 4.75 inches long.

R. J. Blanchard
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January-17, 1995

74260-95-009

(2) FOR BOTTOM THREE (3) SEGMENTS FROM RISER 10B:

For \geq 60% recovery for a particular segment:

SLUDGE or SALTCAKE - Run DSC/TGA and Ni on 1/4 segment basis¹ and Total alpha on 1/2 segment basis - even if it appears to be saltcake.

DRAINABLE LIQUID - DSC on segment basis; i.e., treat any drainable liquid from an extruded segment as one sample.

For < 60% recovery for a particular segment:

Recovery: 0% to ~30% - Treat solids as one sample and do DSC/TGA, Ni, and Total Alpha analyses on the one sample.

Recovery: 31% to 60% - Break total solids in segment into two approximately equal subsegments and do DSC/TGA, Ni, and Total Alpha analyses on each of the two samples.

DRAINABLE LIQUID - DSC on segment basis; i.e., treat any drainable liquid from an extruded segment as one sample.

Archive approximately 20 mL of each sample regardless of recovery (except zero) after analyses are complete.

(3) FOR ALL SAMPLES:

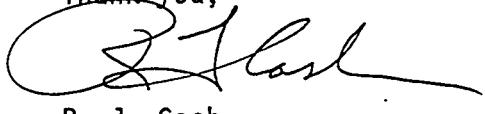
If any sample DSC shows exothermic activity \geq 115 cal/g (dry weight basis), run TOC on the sample. If any sample from the bottom three (3) segments show energies \geq 115 cal/g (dry weight basis), also run a total cyanide analysis on that sample.

Archive approximately 20 mL of each sample regardless of recovery (except zero) after any analyses are complete; remainder of sample may be given to Pretreatment Program.

Duplicates of each analysis are to be performed by the Analytical Laboratory as per previous instructions. Duplicates for each sample are still needed from a statistic point of view and the meeting today did not change that conclusion.

Please call if there are questions.

Thank you,



R. J. Cash



Pacific Northwest Laboratories
Battelle Boulevard
P.O. Box 999
Richland, Washington 99352
Telephone (509) 372-6373

January 12, 1995

Ms. Ruth D. Schreiber
Westinghouse Hanford Company
P.O. Box 1970, MS R2-12
Richland, WA 99352

Dear Ruth,

This is a confirmation of previous discussions and cc:mail messages on the need for the Pretreatment Program to use additional sample from BY-106. Pretreatment fully supports taking more material from the same riser and finds this to be an excellent target-of-opportunity to obtain needed sample material for process investigations.

With regard to the sludge, this tank has been identified by Pretreatment in a letter to you dated December 19, 1994 as satisfactory for sample to support enhanced sludge washing investigations while simultaneously satisfying Safety Program sample needs. The tank was identified as a primary alternate/phase II sample in the recently revised Disposal Sampling Strategy. Sample from this tank can readily fill the need for a phase I sample (initial set of samples to be further evaluated). Pretreatment would still require 125 grams.

Pretreatment could definitely use the sludge from the second proposed core for the Pretreatment enhanced sludge washing scaling task plus any left from the first core. Pretreatment has identified the need for seven different one-liter sludge samples to support a programmed 3.3 million dollar program which is now in its first year of funding at 1.3 million dollars. This sample should fill one of those needs or at least allow a more exact determination of actual sample quantity to provide accurate scaling data. Pretreatment does not care whether the material has been disturbed--all we need is sample material for process investigation and actual test sample size optimization. We will gain needed information on sample size to optimize the experiment and will acquire scaling data on settling, compaction rates, and supernatant content (particulate and radionuclide amount/form).

With regard to saltcake, this is an excellent tank to sample. Brett Simpson states that this tank is representative of the BY tank farm saltcakes, which comprise over one-third the total tank saltcake inventory. Additionally, Brett points out that this saltcake is high in organic content based on previous process

Ms. Ruth D. Schreiber
January 12, 1995
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history and recent vapor sample results.

Thanks again for all of your efforts, as well as that of others in the Characterization Program, to identify and use opportunities such as this that increase efficiency and cut schedule. This letter has been coordinated with the Pretreatment Program Office and Disposal Engineering.

Sincerely,

John T. Slankas

John T. Slankas
Program Element Manager, Pretreatment
Technology Development Program Office

JTS/mfm

cc: JN Appel G3-21
RJ Blanchard R1-17
KA Gasper G3-21
DJ Washenfelder H5-27
File/LB

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