DISTRIBUTION SHEET

To	From	Page 1 of 2
Distribution	Characterization Support	Date 1-24-95
Project Title/Work Order		EDT No. N/A
Tank 241-C-103 Tank Characterization Plan (WHC-SD-WM-TP-207), Revision 1		ECN No. 617829

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ENGINEERING CHANGE NOTICE

Page 1 of 34 RDB

1. ECN No 617829

Proj. ECN

2. ECN Category (mark one)	3. Originator's Name, Organization, MSIN, and Telephone No.	4. Date
Supplemental [] Direct Revision [X] Change ECN [] Temporary [] Standby [] Supersedure [] Cancel/Void []	R. D. SCHREIBER, 71520, R2-12, 373-5589	1-20-95
	5. Project Title/No./Work Order No.: TANK 241-C-103 TANK CHARACTERIZATION PLAN	6. Bldg./Sys./Fac. No.: 241-C
		7. Approval Designator: Q
	8. Document Numbers Changed by this ECN (includes sheet no. and rev.): WHC-SD-WM-TP-207, REV. 0	9. Related ECN No(s.): N/A
		10. Related PO No.: N/A

11a. Modification Work	11b. Work Package No.	11c. Modification Work Complete	11d. Restored to Original Condition (Temp. or Standby ECN only)
[] Yes (fill out Blk. 11b) [X] No (NA Blks. 11b, 11c, 11d)	N/A	N/A Cog. Engineer Signature & Date	N/A Cog. Engineer Signature & Date

12. Description of Change

Removed Pretreatment analyses from Rev. 0.

13a. Justification (mark one)							
Criteria Change	[X]	Design Improvement	[]	Environmental	[]	As-Found	[]
Facilitate Const.	[]	Const. Error/Omission	[]	Design Error/Omission	[]		

13b. Justification Details

The Pretreatment Program requested that the analyses be deleted from the Tank Characterization Plan.

14. Distribution (include name, MSIN, and no. of copies)

See attached Distribution Sheet

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DATE JAN 24 1995

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A-7900-013-2 (06/94) GEF095

ENGINEERING CHANGE NOTICE

1. ECN (use no. from pg. 1): 617829

15. Design Verification Required	16. Cost Impact				17. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[] Yes	Additional	[] $	Additional	[] $	Improvement	[]
[X] No	Savings	[] $	Savings	[] $	Delay	[]

18. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 12. Enter the affected document number in Block 19.

SDD/DD	[]	Seismic/Stress Analysis	[]	Tank Calibration Manual	[]
Functional Design Criteria	[]	Stress/Design Report	[]	Health Physics Procedure	[]
Operating Specification	[]	Interface Control Drawing	[]	Spares Multiple Unit Listing	[]
Criticality Specification	[]	Calibration Procedure	[]	Test Procedures/Specification	[]
Conceptual Design Report	[]	Installation Procedure	[]	Component Index	[]
Equipment Spec.	[]	Maintenance Procedure	[]	ASME Coded Item	[]
Const. Spec.	[]	Engineering Procedure	[]	Human Factor Consideration	[]
Procurement Spec.	[]	Operating Instruction	[]	Computer Software	[]
Vendor Information	[]	Operating Procedure	[]	Electric Circuit Schedule	[]
OM Manual	[]	Operational Safety Requirement	[]	ICRS Procedure	[]
FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
Safety Equipment List	[]	Cell Arrangement Drawing	[]	Process Flow Chart	[]
Radiation Work Permit	[]	Essential Material Specification	[]	Purchase Requisition	[]
Environmental Impact Statement	[]	Fac. Proc. Samp. Schedule	[]	Tickler File	[]
Environmental Report	[]	Inspection Plan	[]		[]
Environmental Permit	[]	Inventory Adjustment Request	[]		[]

19. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision | Document Number/Revision | Document Number Revision

20. Approvals

Signature	Date	Signature	Date
OPERATIONS AND ENGINEERING		ARCHITECT-ENGINEER	
Cog. Eng. R.D. SCHREIBER R.D. Schreiber	1/20/95	PE	
Cog. Mgr. S. J. EBERLEIN S.J. Eberlein	1/23/95	QA	
QA K.K. CUTSFORTH T.C. Thomas	1/23/95	Safety	
Safety		Design	
Environ.		Environ.	
Other J. T. SLANKAS R.D. Schreiber per telecon	1-20-95	Other	
J.G. KRISTOFZSKI A.G. Dick FOR JGK	1-20-95		
S.G. MCKINLEY see next page	1-23-95		
V. W. HALL R.D. Schreiber for C.L. Thomas per telecon	1-20-95	DEPARTMENT OF ENERGY	
		Signature or a Control Number that tracks the Approval Signature	
		ADDITIONAL	

ENGINEERING CHANGE NOTICE

1. ECN (use no. from pg. 1): 617829

15. Design Verification Required	16. Cost Impact				17. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[] Yes	Additional	[] $	Additional	[] $	Improvement	[]
[X] No	Savings	[] $	Savings	[] $	Delay	[]

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FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
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OPERATIONS AND ENGINEERING		ARCHITECT ENGINEER	
Cog. Eng. R.D. SCHREIBER R.D. Schreiber	1/20/95	PE	
Cog. Mgr. S. J. EBERLEIN S.J. Eberlein	1/23/95	QA	
QA K.K. CUTSFORTH T.C. Tonkin	1/23/95	Safety	
Safety		Design	
Environ.		Environ.	
Other J. T. SLANKAS R.D. Schreiber per telecon	1-20-95	Other	
J.G. KRISTOFZSKI A.G. Dick for JGK	1-20-95		
S.G. MCKINLEY S.G. McKinley	1-23-95		
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ECN 617829
Page 4 of 4

REVIEW COMMENT RECORD (RCR)

1. Date	2. Review No.
3. Project No.	4. Page 1 of

5. Document Number(s)/Title(s)	6. Program/Project/ Building Number	7. Reviewer	8. Organization/Group	9. Location/Phone
WHC-SD-WM-TP-207, REV. 1/Tank 241-C-103 Tank Characterization Plan	Tank Farms	A. D. Rice	222-S Lab Program Support	200W/M0028

17. Comment Submittal Approval:

Organization Manager (Optional) — Date

10. Agreement with indicated comment disposition(s)

Reviewer/Point of Contact — Date

R D Schmel
Author/Originator

11. CLOSED

Reviewer/Point of Contact — Date

Author/Originator

12. Item	13. Comment(s)/Discrepancy(s) (Provide technical justification for the comment and detailed recommendation of the action required to correct/ resolve the discrepancy/problem indicated.)	14. Hold Point	15. Disposition (Provide justification if NOT accepted.)	16. Status
1	The Organic Safety Program has requested TOC analysis by furnace oxidation as a secondary analysis. The WHC 222-S Laboratory does not have the capability to run this analysis on solid samples (the sample is injected into the instrument).			

RELEASE AUTHORIZATION

Document Number:	WHC-SD-WM-TP-207, REV.1
Document Title:	TANK 241-C-103 TANK CHARACTERIZATION PLAN
Release Date:	January 24, 1995

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

APPROVED FOR PUBLIC RELEASE

WHC Information Release Administration Specialist:

Kara M. Broz — January 24, 1995

SUPPORTING DOCUMENT

1. Total Pages 33

2. Title	3. Number	4. Rev No.
TANK 241-C-103 TANK CHARACTERIZATION PLAN	WHC-SD-WM-TP-207	1

5. Key Words

CHARACTERIZATION, SAFETY SCREENING, QUALITY CONTROL, SINGLE-SHELL TANK, SAMPLING, ANALYSIS, TANK CHARACTERIZATION PLAN, ORGANIC WATCH LIST, PRETREATMENT

6. Author

Name: R. D. SCHREIBER

R.D. Schreiber
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, WHC 222-S Laboratory, and PNL 325 Analytical Chemistry Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of samples from tank 241-C-103.

8. RELEASE STAMP

OFFICIAL RELEASE BY WHC (5)
DATE JAN 24 1995
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RECORD OF REVISION

RECORD OF REVISION	(1) Document Number WHC-SD-WM-TP-207	Page 1

(2) Title
TANK 241-C-103 TANK CHARACTERIZATION PLAN

CHANGE CONTROL RECORD

(3) Revision	(4) Description of Change - Replace, Add, and Delete Pages	Authorized for Release	
		(5) Cog. Engr.	(6) Cog. Mgr. Date
0	(7) WHC-SD-WM-TP-207, REV 0, EDT 159074, October 3, 1994		
1 RS	Revised in its entirety, ECN 617829	R.D. Schreiber	[signature] 1/23/95

A-7320-005 (08/91) WEF168

WHC-SD-WM-TP-207
Revision 1

Tank 241-C-103
Tank Characterization Plan

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

by

Los Alamos Technical Associates
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CONTENTS

SAMPLING EVENT A: PUSH MODE CORE SAMPLING IN FISCAL YEAR 1995

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ACL	Analytical Chemistry Laboratory
B	B-Plant high level waste
BL	B-Plant low level waste
BNW	Battelle Northwest
C-103	Tank 241-C-103
CW	Cladding Waste
DQO	Data Quality Objective
DSC	Differential Scanning Calorimetry
DSSF	Double Shell Slurry Feed
DST	Double Shell Tank
DW	Decontamination Waste
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)
LW	Lab Waste
MW	Metal Waste (from bismuth phosphate operations)
N	N-Reactor Waste
NCPLX	Noncomplexed Waste
OWW	Organic Wash Waste
P	Purex High Level Waste
PL	Purex Low Level Waste
PSS	Purex Sludge Supernatant
R	Redox Waste
RCRA	*Resource Conservation and Recovery Act of 1976*
SST	Single-Shell Tank
TBP	Tri-butyl Phosphate
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

The Defense Nuclear Facilities Safety Board has advised the DOE to concentrate the near-term sampling and analysis activities on identification and resolution of safety issues (Conway 1993). The data quality objective (DQO) process was chosen as a tool to be used to identify the sampling analytical needs for the resolution of safety issues. As a result, a revision in the Federal Facility Agreement and Consent Order (Tri-Party Agreement) milestone M-44-00 has been made, which states that "A Tank Characterization Plan (TCP) will also be developed for each double shell tank (DST) and single-shell tank (SST) using the DQO process . . . Development of TCPs by the DQO process is intended to allow users (e.g., Hanford Facility user groups, regulators) to ensure their needs will be met and that resources are devoted to gaining only necessary information." This document satisfies that requirement for the tank 241-C-103 (C-103) fiscal year 1995 sampling activity.

1.1 RELEVANT SAFETY ISSUES

There are four Watch List tank classifications (ferrocyanide, organic salts, hydrogen/flammable gas, and high heat load). These classifications cover the six safety issues related to public and worker health that have been associated with the Hanford Site underground storage tanks. These safety issues are as follows: ferrocyanide, flammable gas, organic, criticality, high heat, and vapor safety issues (Babad 1992). Tank C-103 is one of the twenty tanks currently on the Organic Salts Watch List. This TCP will identify characterization objectives pertaining to sample collection, hot cell sample isolation, and laboratory analytical evaluation and reporting requirements in accordance with the appropriate DQO documents. In addition, the current contents and status of the tank are projected from historical information.

The organic safety issue arises due to wastes added to SSTs containing quantities of complexants used in waste management operations, as well as degradation products of these complexants and solvents used in fuel reprocessing and metal recovery operations. These waste tanks also contain a presumed stoichiometric excess of sodium nitrite and sodium nitrate that are sufficient to exothermally oxidize the organic compounds in the tank.

The relevant safety issues that are of concern for tanks on the Organic Salts Watch List are as follows:

1) The potential for an exothermic reaction occurring from the flammable mixture of organic materials and nitrate/nitrite salts that could result in a release of radioactive material.
2) The possibility that other safety issues may exist for the tank.

The Pretreatment DQO addresses the characterization needs of the Pretreatment, High-Level Waste Disposal, and Low-Level Waste Disposal Programs. These programs are responsible for developing long-term treatment and storage processes for the Hanford Site waste. This technology development effort will require comprehensive physical and chemical information from waste tank samples. The pretreatment process must be able to separate the waste into feed streams that satisfy the safety issues associated with the operating requirements for the low-level and high-level vitrification facilities.

1.1.1 Tank C-103 Characterization Objectives

The characterization effort applicable to this TCP is focused on the relevant safety issues listed above. The two key safety questions that should be answered from analytical data on tank C-103 waste are as follows:

(1) Is the tank SAFE and/or does it belong on the Organic Salts Watch List?
(2) Is the tank CONDITIONALLY SAFE OR UNSAFE?

Based on the answers to these two questions, actions will be identified and implemented to mitigate or remediate the conditions that resulted in classifying the tank as UNSAFE (Babad et al. 1994).

1.1.2 Organic Salts, Pretreatment, and Safety Screening Data Quality Objectives

The sampling and analytical needs associated with organic salts tanks, as well as the safety screening of all tanks, have been identified through the Data Quality Objective (DQO) process. In addition, data needs associated with tank C-103 are identified in the pretreatment DQO. Additional data needs associated with tank C-103 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 Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994), which describes the sampling and analytical requirements for tanks on the Organic Salts Watch List, including tank C-103.

(2) *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), which describes the sampling and analytical requirements for screening all waste tanks for unidentified safety issues.

(3) *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 6.1.

1.1.3 Data Quality Objectives Integration

The organic, safety screening, and pretreatment DQO efforts all require a minimum of two core samples to be taken from risers separated radially to the maximum extent possible by the existing installed risers. The safety screening DQO requires tank samples to be analyzed in half-segments. The organic DQO also requires half-segments. However, the Pretreatment Program requires samples from solid and liquid core composites for their specific requests.

It should be noted that for this Tank Characterization Plan, the Pretreatment Program has requested that only composite samples for archive and a solid composite sample for process development be obtained. For further information, refer to Section 6.1.

The analytes identified in the safety screening DQO for the various safety issues are a subset of the suite of analyses identified in the organic DQO with the exception of analytes measured for the criticality safety issue. If notification limits for immediate reporting of analytes identified in the DQO efforts were found to be conflicting, the most stringent limits were used in this TCP.

2.0 TANK, WASTE, AND SAMPLING INFORMATION

This section summarizes some of the available information for tank C-103. Discussions of the process history, recent sampling events for the tank, and general information about the tank are included.

2.1 AGE AND PROCESS HISTORY

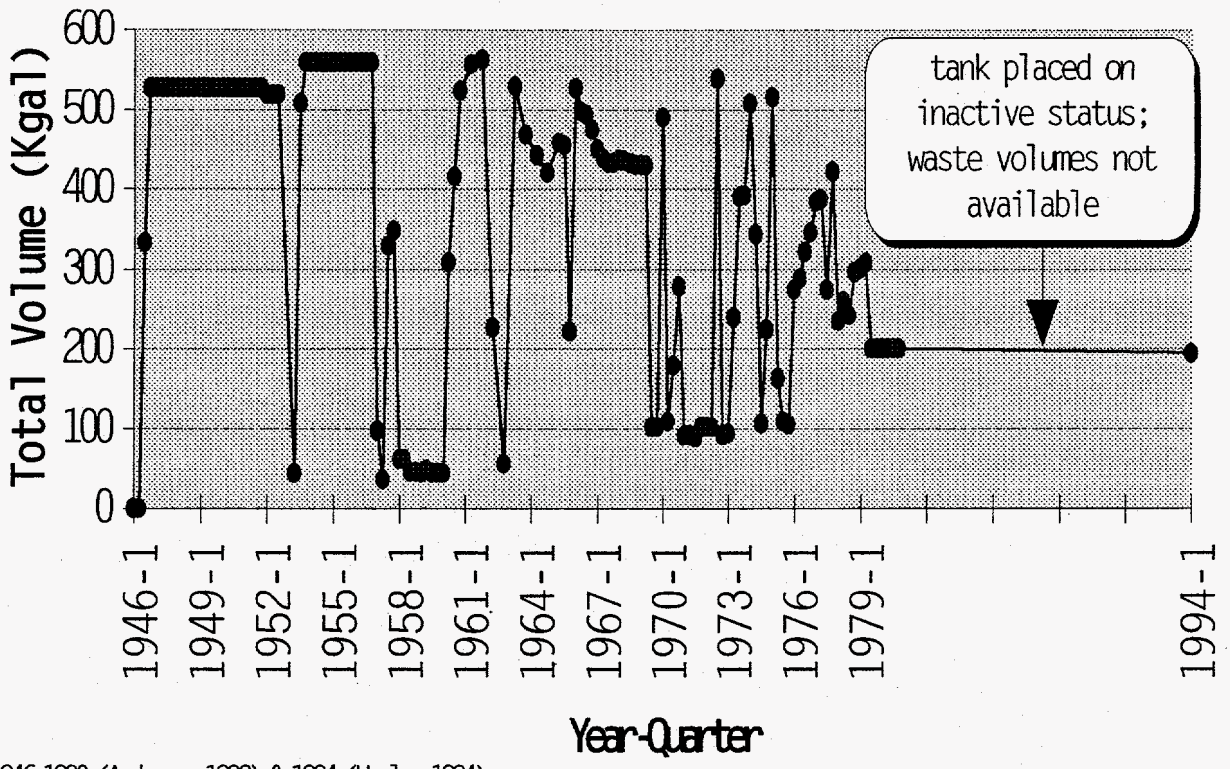
Tanks C-101, C-102, and C-103 comprise a cascade. A cascade is a system where tanks were connected in series by pipes. Waste added to the first, or primary, tank overflowed to the secondary tanks after the primary tank became full. Note that tank C-103 was the last tank in its particular cascade.

Tank C-103 is a 2,014,000 liter (530,000 gallon) SST in the 241-C Tank Farm. It has been declared sound and has been partially interim isolated (December 1982). This tank is classified as nonstabilized. Tank C-103 currently contains non-complexed waste (NCPLX) with a total waste volume of 741,000 liters (195,000 gallons) this tank volume is equivalent to 167.64 cm (66 inches) of waste as measured from the bottom of the tank. The waste in tank C-103 is comprised of 235,600 liters (62,000 gallons) of sludge and 505,400 liters (133,000 gallons) of supernatant liquid. The source of the waste for tank C-103 is from the PUREX plant and insoluble strontium-rich sluicing solids from the operation of 244-CR Vault.

The last solids update was obtained October 22, 1990, and the last photo was taken July 28, 1987 (Hanlon 1994). The photograph shows a dark brown liquid surface, but no visible sludge. In addition, there appear to be pieces of equipment that were cut off and allowed to fall back into the tank. Figure 1 and Table 1 depict the fill history of tank C-103 since it began accepting waste in August 1946.

An Unreviewed Safety Question (USQ) concerning tank C-103 was declared in September 1992 due to the presence of an organic layer on the surface of the waste (Hanlon 1994). This tank has, therefore, been identified as an Organic Salts Watch List tank. Organic chemicals are potentially flammable, and a combination of organic materials mixed with nitrite and nitrate salts can deflagrate. Tank C-103 is listed on the Organic Salts Watch List due to the "potential for release of high level waste because of uncontrolled increases in the temperature or pressure" (Hanlon 1994). This safety concern is associated with tanks that primarily contain solids since their temperature may increase as liquid levels decrease, and "high organic concentrations in the tanks could support an exothermic reaction at elevated temperatures (350 °F/180 °C)" (Hanlon 1994). Tank C-103, however, is largely liquid and has displayed temperature readings below 350°F for the first three months of 1994 (Hanlon 1994).

Figure 1: Fill History for Tank C-103



1946-1980 (Anderson 1992) & 1994 (Hanlon 1994)

Table 1: Waste Status Summary for Tank C-103

Year-Qtr.	Total Vol. (Kgal)	Type Waste	Waste rec'd from:	Kgal rec'd:	Waste moved to:	Kgal re-moved:	Remarks
1946-4	528	MW					began filling August 1946; filled in October 1946
1952-3	519	MW			C-109		supernate pumped out
1952-4	—	MW					984 Kgal in Cascade; processing-feed to TBP plant
1953-3	508	TBP	C-101		C-106		removed MW waste; received TBP waste
1953-4	560	TBP					received TBP waste
1957-3	329	TBP, P	A-102	292			scavenged then waste received
1957-4	348	TBP, P	A-102	19			
1958-1	62	TBP, P			BY-103	286	waste removed
1960-4	524	TBP, P, CW					SS 265; SS 107; 108 CW rec'd
1963-2	530	CW, P	A-105	473			waste received
1963-4	469	P	A-105	—			supernatant received
1965-4	222	P	A-101	202	A-103	435	Cs recovery in process
1966-1	527	P	A-101	446	A-103	141	Cs recovery in process
1968-2	435	P					Cs recovery operation ended
1968-3	433	P					Cs feed
1968-4	431	P					Cs feed
1969-3	103	P			C-105	326	326 Kgal removed
1970-1	491	P, BL	BX-101	385			waste received and removed
1970-2	109	BL, EB	B-102	415	C-105	798	plus 3 Kgal added from water flush
1970-3	180	BL, PSS	C-106	69			
1970-4	279	BL, PSS	C-106	99			
1971-1	92	—	C-106	257	C-106	444	waste received and removed
1972-3	539	CW, OWW	C-104	437			waste received and removed
1972-4	92	CW			C-104	443	
1973-2	239	BNW, N, LW, CW, PL	C-104	145			waste received
1973-3	390	BNW, N, LW, CW, PL	C-104	151			
1974-1	508	BNW, N, LW, P, B, CW, DW, EB ,IX, PL	C-104	114			waste received and removed; five dry wells drilled
1974-2	343	BNW, LW, R, B, CW, DW, IX, PL			C-104	165	
1974-3	107	BNW, N, LW, PL, B, CW, EB	C-104	59	S-107	297	
1974-4	224	BL	C-106	409	S-107	7	
					TX-101	281	
1975-1	516	BNW, N, LW, CW, DW, IX, EB, B, PL, BL	C-104	108	SX-106	349	waste received and removed
			C-106	404			
			C-107	65			
			C-112	66			
1975-2	164	BNW, N, PL, EB, P, B	C-104	399	SX-106	426	
			C-106	258	TX-101	584	
1975-3	109	OWW, CW, IX	C-107	195	SX-106	1014	
			C-109	364			
			C-112	400			
1975-4	106	CW, OWW	C-102	111	SX-106	711	
			C-108	426			
			C-109	85			
			C-112	85			
1976-1	274	BNW, N, LW, CW, DW, IX, TBP, R, OWW	C-107	1			waste received
			C-108	27			
			C-109	9			
			C-110	62			
			C-111	63			
			C-112	3			
1976-2	288	NW, N, LW, CW, DW, IX, TBP, R OWW, RIX, EB	C-108	1			plus 5 Kgal water added
			C-110	4			
			C-111	2			
			C-112	1			
1976-3	321	Feed Dil.					Purex Waste Storage
1977-4	422	Feed Dil., SR Sludge					Evap. B Plant Waste Recovery
1978-2	260	NCPLX					Active-SW RCR-Evap. Feed
1979-4	200	NPCLX					Inactive
1994-1	195	NPCLX					195 Kgal total waste (133-supernatant; 62-sludge)

2.2 EXPECTED TANK CONTENTS

On December 15, 1993, Pacific Northwest Laboratory (PNL) received five samples of organic liquid and one sample of underlying aqueous supernatant liquid taken from tank C-103. The purpose of the analytical work was to provide data for a Safety Analysis Report that addresses an Unreviewed Safety Question concerning the potential for a pool fire in the organic layer of tank C-103, with subsequent loss of containment and possible radiation release to the surrounding environment.

Data obtained from execution of the analytical plan are summarized in Table 2. Column 2 gives results for the organic layer, and Column 3 gives results for the aqueous layer (Pool 1994).

Table 2: Most Recent Analysis of Liquid Samples[1]

Determination	Results of Organic Supernatant Phase	Results of Aqueous Supernatant Phase
Flash point	approx. 246 °F, with tester in "Setaflash" mode	NA
Organics by GC/MS	TBP:NPH = 67:33(w:w), 62:38(v:v)	NA
Organic volatiles	1.4 mg/L organic material equilibrated above surface at 40 °C (mostly NPH and TBP)	NA
Peroxides	Less than 2.5 μEquivalent/g	NA
Nitroalkanes	No indication of nitroalkanes by RSST experiment; less than 2 μmol/g by infrared analysis	NA
Density	0.876 g/mL at 25 °C, 0.868 g/mL at 44 °C	1.078 g/mL at 25 °C, 1.076 g/mL at 44 °C
Viscosity	4 cP at 25 °C, 2.5 cP at 40 °C	1.5-4.5 cP at 25 °C (depending on shear rate), 2 cP at 40 °C
Gross alpha, beta	Alpha = 479 pCi/mL Beta = 9.2×10^5 pCi/mL	Alpha = 4.35×10^4 pCi/mL Beta = 7.06×10^7 pCi/mL
^{90}Sr	4.8×10^5 pCi/mL	NA
Alpha emitters	^{238}Pu = 79 pCi/mL $^{239+240}Pu$ = 170 pCi/mL ^{241}Am = 157 pCi/mL	NA
Gamma emitters	^{60}Co = 6.53×10^{-4} μCi/mL ^{137}Cs = 3.62×10^{-2} μCi/mL ^{154}Eu = 2.78×10^{-4} μCi/mL ^{155}Eu = 2.76×10^{-4} μCi/mL ^{241}Am = 1.85×10^{-4} μCi/mL	NA
Water content	1.31 wt%	NA

Ammonia	24 μg NH_3/g	215 μg NH_3/g
IC	F, Cl, NO_2, NO_3, SO_4, all <50 μg/mL	F^- = 1,185, Cl^- = 431, NO_2^- = 24,794, NO_3^- = 2,587, SO_4^{2-} = 3,234, PO_4^{3-} = 2,16 (μg/mL)
ICP/AES (2% HNO_3 leach)	Ag = 0.8, Al = 1.6, B = 9.6, Ca = 1.8, Cd = 1.8, Cu = 1.9, Fe = 0.29, Na = 61.3, Ni = 8.7, P = 530 (μg/mL)	Ag = 12, Ca = 5, Cd = 0.9, Cr = 57, Fe = 3, K = 323, Na = 32,771, Ni = 72, P = 2,533, U = 2102, Zr = 302 (μg/mL)
Extractable organics by GC/MS	NA	TBP = 86 μg/mL DBBP = 7 μg/mL NPH = 1-3 μg/mL
Volatiles by GC/MS	NA	Only NPH and TBP identified in headspace
Gamma analysis	NA	^{60}Co = 0.0511 μCi/mL ^{137}Cs = 57.9 μCi/mL
pH	NA	10.00, 9.99
Carbon	NA	TOC = 7200 μg/mL, TIC = 5200 μg/mL
DSC/STG TOC of dried solids	NA	Exotherm of 180 calories/g between 240 and 430 °C. TOC = 4.4 wt%

[1] (Pool 1994)
Note: 10^{12} pCi = 1 Ci

2.3 SAMPLING INFORMATION

Tank C-103 is currently scheduled to be sampled by the push mode core sampling method. Samples shall be taken from risers 2 and 7. For detailed information regarding the sampling activities, refer to work plan TFPE-YP-0229 for Riser 2 and work plan TFPE-YP-0230 for Riser 7. In addition, refer to work package 2E-93-00451. These documents contain operating procedures and the chain of custody records for this sampling event.

Current records indicate that there are 741,000 liters (195 Kgals) of waste in tank C-103. The waste consists of 235,600 liters (62 kgals) of sludge and 505,400 liters (133 Kgals) of supernatant liquid (Hanlon 1994). The most current liquid level is approximately 111.76 cm (44 inches) of supernate in a total of 167.64 cm (66 inches) of waste. Tank C-103 is considered sound with respect to tank integrity.

One push mode core sample from each riser shall be collected from risers 2 and 7 of tank C-103. Based on the information above, each core is expected to consist of four segments each. The first segment from each core is expected to contain 22.86 cm (9 inches) of waste material, while the three final segments should contain 48.26 cm (19 inches) of waste each. It should be noted that the sampling objective is to obtain a vertical profile of the waste; therefore, more or less segments may need to be taken depending on the accuracy of the volume estimates above.

Hydrostatic head fluid (HHF) with lithium bromide (LiBr) as a tracer shall be used to aid in the collection of all core samples. An HHF blank shall be prepared as part of the sampling procedure. The blank shall consist of a container filled with HHF (with LiBr tracer) from the same batch of HHF used during the push mode core sampling. It shall be analyzed for Li (and Br, if necessary) in order to determine the concentration of the tracer at the time the core was taken. Only one HHF blank per tank is required. This blank is required in addition to the field/trip blank (sampler filled with water). For specific information concerning sample handling custody, and transport, refer to the quality assurance/quality control requirements in Section 4.2.

3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

3.1 TANK-SPECIFIC ANALYTICAL PROCEDURES

A flowchart depicting the general organic, pretreatment, and safety screening sample breakdown and analysis scheme is presented in Figures 2, 3, and 4. 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 3 require a 45 day reporting time, as noted. The 45 day reporting format, Format III, is explained in Section 7.3.

Any decisions, observations, or deviations and justifications made to this work plan or during the sample breakdown shall be documented in writing. These decisions and observations shall also be reported in the data report. The reporting formats for analyses are contained in Table 3.

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

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

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

Step 3 Separate any drainable liquid from the solids by allowing the liquid to drain into a bottle. Measure and record the volume. Retain drainable liquids for further processing.

Step 4 Is the segment 100% drainable liquid?

Yes: Proceed to Step 14
No: Proceed to Step 5

SOLIDS PATH

Step 5 Divide each extruded core segment into two equal subsamples.

Step 6 Homogenize each subsample using the appropriate approved procedure.

Step 7 Will a homogenization test be performed?

Yes: Proceed to Step 8
No: Proceed to Step 9

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

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

Step 9 Collect sufficient aliquots from each homogenized subsample to perform the appropriate preparations and analyses listed in Table 3 in duplicate.

NOTE: If there is an insufficient amount of sample available in any subsample to perform all required analyses on the half segment, notify the Characterization Program within one business day and follow the prioritization of analyses given in Section 3.3.

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

Step 11 Combine 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 include 125 mL of material for process development and 100 mL for archive.

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

Step 13 Remove 125 mL of the solid composite for process development work (see Section 6.2).

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

LIQUIDS PATH

Step 14 Closely inspect the liquid sample for the presence and approximate volume of any potential organic layers. Does the sample contain any immiscible (potentially organic) layers?

Yes: Proceed to Step 15A
No: Proceed to Step 16

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

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

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

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

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

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

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

Step 19 Remove sufficient aliquots from the segment-level liquid sample to perform the appropriate analyses listed in Table 3 in duplicate.

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

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

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

PRIMARY ANALYSIS PATH

Step 23 Perform primary analyses as listed in Table 3.

Step 24 Compare the primary analysis data with notification limits.

Step 25A Do the results exceed the notification limits (Table 3)?

Yes: If the results exceed the notification limits. Proceed to Step 25B.
No: If results do not exceed the notification limits, proceed to Step 28.

Step 25B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section 7.2.

SECONDARY ANALYSIS PATH

Step 26 Perform secondary analyses according to Table 3.

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

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

Step 27B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section 7.2.

REPORTING PATH

Step 28 Report and deliver data obtained using reporting requirements (Section 7.0)..

Figure 2: Laboratory Flow Chart A

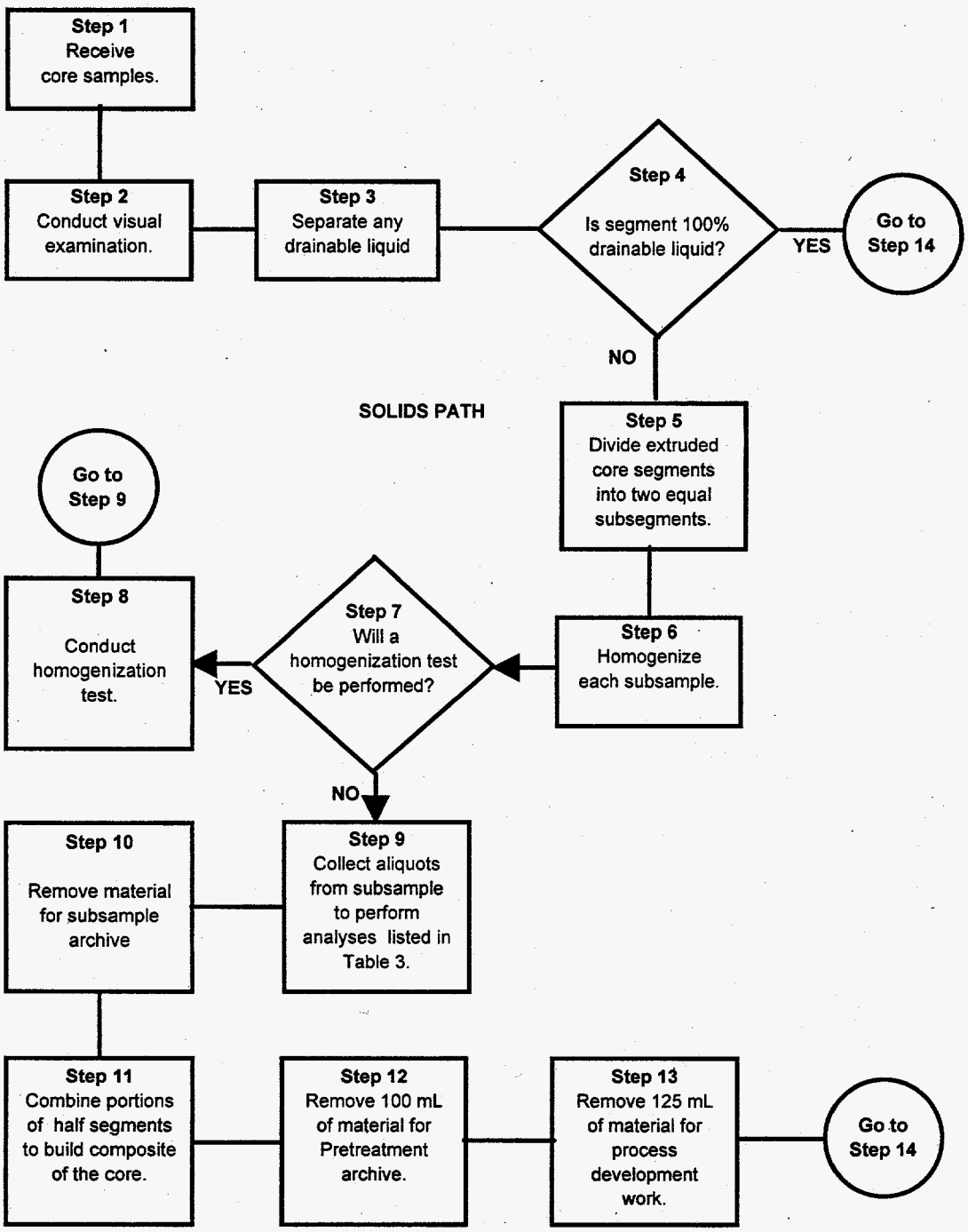


Figure 3: Laboratory Flow Chart B

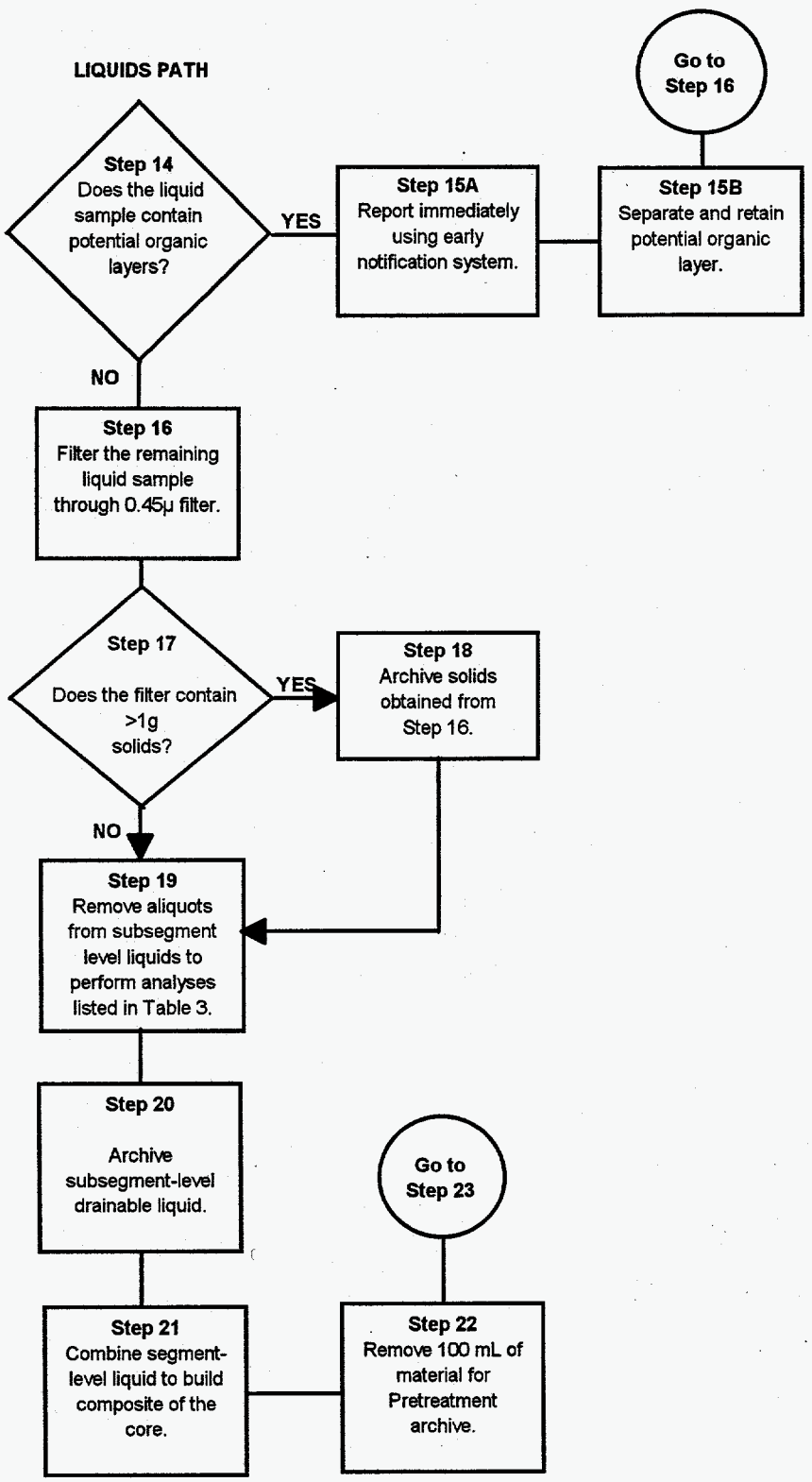
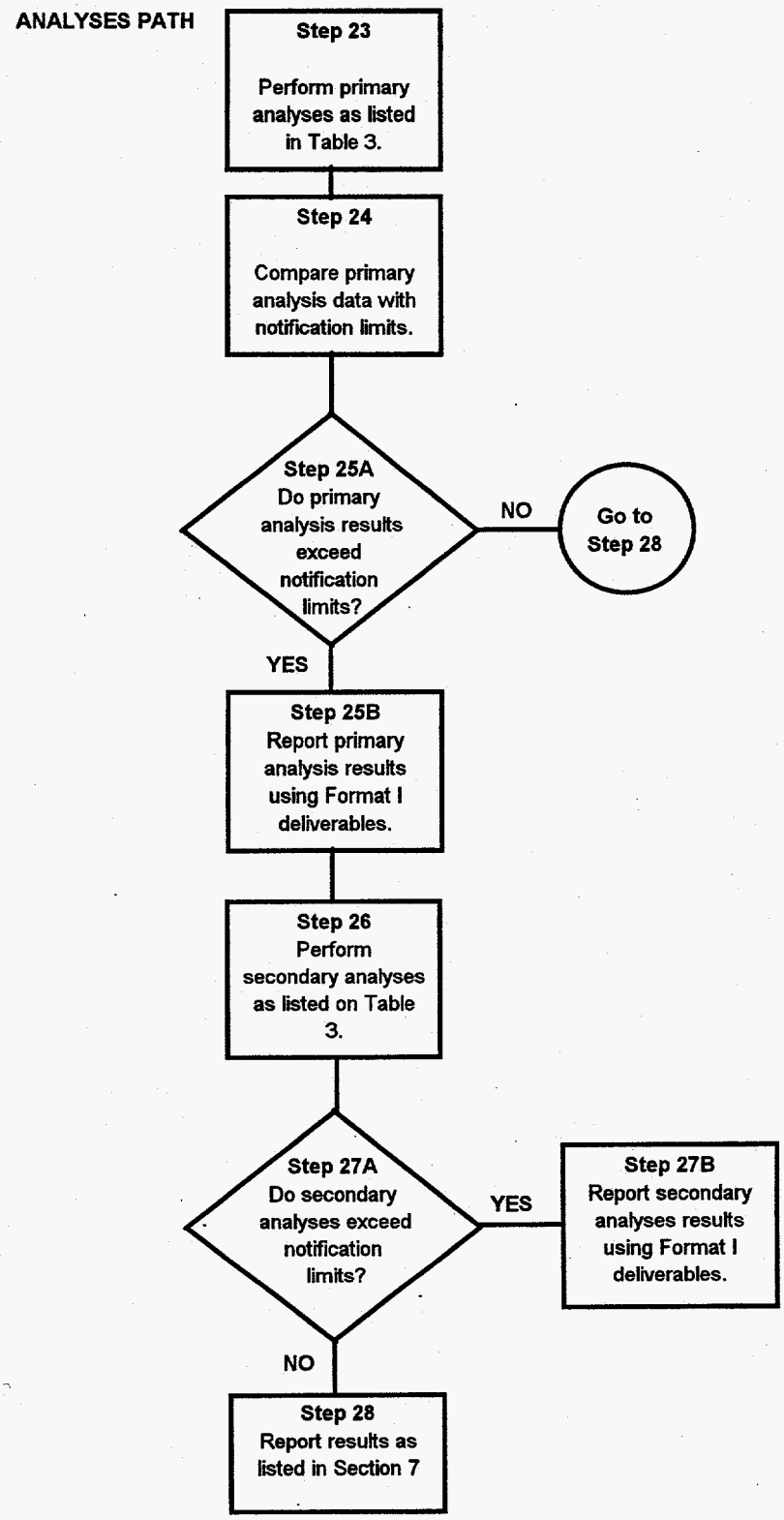


Figure 4: Laboratory Flow Chart C



3.2 INSUFFICIENT SEGMENT RECOVERY

Historically, segments have been found to be only partially filled such that there is insufficient material to perform all requested analyses. If the amount of material recovered from core samples taken from tank C-103 is insufficient to perform the analyses requested and permit a minimum 10 mL archive per segment, the laboratory shall notify the Tank Cognizant Engineer and the manager of Analytical Services, Program Management and Integration within one working day. (See Table 4). A prioritization of the analyses requested in this document is given in Section 3.3. Any analyses prescribed by this document, but not performed, shall be identified in the appropriate data report, with justification for non-performance.

3.3 PRIORITIES OF REQUESTED ANALYSES

The analyses to be performed for the tank safety screening and organic programs have been prioritized below. Confirmation of prioritization levels or revision of sample breakdown procedures may be provided based upon the sample recovery, readily observable physical property distinctions within the sample, and the requested sample breakdown procedures provided in section 3.1.

PRIORITY LEVEL 1

The total organic carbon (TOC), DSC, TGA, Total Alpha, and Li analyses shall be performed.

PRIORITY LEVEL 2

Cyanide, RSST, bromide, and gravimetric analyses shall be performed.

PRIORITY LEVEL 3

Hydroxide, GC/MS, nitrate and nitrite analyses shall be performed.

4.0 SPECIFIC ANALYTE, QUALITY CONTROL, AND DATA CRITERIA

4.1 SPECIFIC METHODS AND ANALYSES

The analyses in Table 3 to be performed on the tank C-103 core samples are based on the Organic, and Safety Screening DQOs referenced in Section 1.1.2. The laboratory procedure numbers, which shall be used for the analyses, are included in the table.

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 of analyzing the waste tank core samples at the 222-S Laboratory. If the analyses are performed at the 325 Analytical Chemistry Laboratory (ACL), the analysis shall be guided by the 325 Quality Assurance Plan (Kuhl-Klinger 1994). Additionally, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994), when implemented, 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 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

Two core samples with an expected four segments each are to be taken from tank C-103 and shipped to the performing laboratory by Sampling Operations in accordance with work package 2E-93-00451. That work package shall also initiate the chain-of-custody for the samples. Approved work plans TFPE-YP-0229 and TFPE-YP-0230, and procedure TO-080-090 ("Load/Transport Sample Cask(s)") are to be used during the sampling event. Samples shall be identified by a unique number before being shipped to the performing laboratory. The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook. Sampling Operations shall enter this information in the comment section of the chain-of-custody form for addition to the data reports.

Sampling Operations should transport each segment collected to the performing laboratory within 1 working day of removing the segment from the tank, but must transport each segment within 3 calendar days. The field blank 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.

4.2.3 Sample Custody

The chain-of-custody form is initiated by the sampling team as described in work package 2E-93-00451. Core samples are shipped in a cask and sealed with a Waste Tank Sample Seal.

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

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

Table 3: C-103 Chemical, Radiolo

			SO
Project Name	C-103 Push Mode Core Sample		
Plan Number	WHC-SD-WM-TP-207, REV. 1		Hom
			Field
PROGRAM	**PROGRAM CONTACTS**		Hot C
A. Safety Screening	Safety Screening	H. Babad	
C. Organic	Organic	D. A. Turner	
	TWRS	R. D. Schreiber	
	222-S Laboratory	J. G. Kristofzski	
	325 Laboratory	S. G. McKinley	

PROGRAM	PRIMARY ANALYSES				SAMPLE[1]		PRI
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	½ SEG SLDG	½ SEG SC	
A,C	DSC	Energy	LA-514-113	PNL-ALO-508	X	X	c
A,C	TGA	% H_2O	LA-560-112	PNL-ALO-508	X	X	c
A	Alpha	Total Alpha	LA-508-101	PNL-ALO-421 PNL-ALO-420	X		f o
A,C	ICP	Li	LA-505-151	PNL-ALO-211	X	X	f o
A,C	Hot Persulfate	TOC	LA-342-100	PNL-ALO-381	X[12]	X[12]	c

PROGRAM	SECONDARY ANALYSES				SAMPLE[1]		PRE
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	½ SEG SLDG	½ SEG SC	
A,C	RSST[10]	Energy	see 10 below	N/A	X	X	c
A	Distillation[10]	CN	LA-695-102	PNL-ALO-285	X	X	c
A	Sep. & α counting[11]	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422	X		f
A	ICP[11]	Fe U	LA-505-151	PNL-ALO-211	X		f o
A,C	ICP[11,15]	Mn	LA-505-151	PNL-ALO-211	X		f o
C	ICP[15]	Cr	LA-505-151	PNL-ALO-211	X		f o
C	IC[13]	NO_2^- NO_3^-	LA-533-105	PNL-ALO-212	X		w
A,C	IC[6]	Br	LA-533-105	PNL-ALO-212	X	X	w
C	Titration[13]	OH^-	LA-661-103	PNL-ALO-225	X		d
A,C	Furnace Oxidation[14]	TOC	LA-344-105	PNL-ALO-380	X	X	d
C	Gravimetric[7]	% H_2O	LA-564-101	PNL-ALO-504	X		d

[1]½ SEG SLDG-½ segment, sludge; ½ SEG SC-½ segment, saltcake

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

[3]PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicat

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

[5]Dry weight basis.

[6] This analysis required if Li exceeds notification limit.

cal and Physical Analytical Requirements

) ANALYSES

COMMENTS	REPORTING LEVELS	
nization Test - Per Laboratory Discretion	FORMAT I	Early Notify
nk - Not Required	FORMAT II	Process Control
Blank - Per Laboratory Discretion	FORMAT III	Safety Screen
	FORMAT IV	Waste Management
	FORMAT V	RCRA Compliance
	FORMAT VI	Special

TANK	#CORES	RISER#
C-103	2	2 and 7

QUALITY CONTROL[3]						CRITERIA			FOR-MAT
DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
ea smpl	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III, IV
ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III, IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III, IV
ea smpl	see[9]	ea PB	ea AB	±10	90-110	µg/g	100	unknown	I, III, IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg C/g	> 30,000	unknown	I, III[12], IV

QUALITY CONTROL[3]						CRITERIA			FOR-MAT
DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
ea smpl	N/A	N/A	ea AB	±20	80-120	J/g	> 481	unknown	I, III, IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III, IV
ea smpl	1/mtrx[8]	ea PB	ea AB	±15	85-115	µCi/g	> 41	unknown	I, III, IV
ea smpl	see[9]	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV
							none	unknown	III, IV
ea smpl	see[9]	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV
ea smpl	see[9]	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV
							none	unknown	IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	1200	unknown	I,III,IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV
ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg C/g	> 30,000	unknown	I, III, IV
ea smpl	N/A	N/A	ea AB	±20	80-120	wt%	< 17	unknown	I, IV

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

Table 3: C-103 Chemical, Radiolog

[7]This analysis is required if moisture analysis by TGA exceeds the notification limit.

[8]Tracer or carrier may be used in place of a spike and results corrected for recovery.

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

[10]This analysis required if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be

[11]Performed only if total alpha exceeds notification limit.

[12]These analyses are primary analyses for the organic DQO, but also are secondary analyses for the safety scr
and reported within 90 days of receipt of the last sample at the laboratory dock.

[13]This analysis is required if the energy equivalent of the TOC assay by hot persulfate is > 125% of the DSC val

[14]This analysis required if the energy equivalent of the TOC by hot persulfate is < 75% of the DSC value.

[15]This analysis is required if the energy equivalent of the TOC assay by furnace combustion is < 75% of the DS

cal and Physical Analytical Requirements

und in WHC-SD-WM-TP-104.

ning DQO. Therefore, if the DSC limit is exceeded, these analyses must be performed

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/alue.

Table 3: C-103 Chemical, Radiolog

					LIQU
Project Name	C-103 Push Mode Core Sample				
Plan Number	WHC-SD-WM-TP-207, REV. 1				Ho
					Fie
PROGRAM	**PROGRAM CONTACTS**				Ho
A. Safety Screening	Safety Screening	H. Babad			
C. Organic	Organic	D. A. Turner			
	TWRS	R. D. Schreiber			
	222-S Laboratory	J. G. Kristofzski			
	325 Laboratory	S. G. McKinley			

PROGRAM	PRIMARY ANALYSES				SAMPLE[1]	PREP[2]	
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID		
A,C	DSC	Energy	LA-514-113	PNL-ALO-508	X	d[6]	e
A,C	TGA	% H_2O	LA-560-112	PNL-ALO-508	X	d[6]	e
A,C	Hot Persulfate	TOC	LA-342-100	PNL-ALO-381	X[12]	d[6]	e
A,C	ICP	Li	LA-505-151	PNL-ALO-211	X	d[6]	e
A,C	Visual	Organic Layer	LA-519-151	PNL-ALO-501	X	d	
PROGRAM	**SECONDARY ANALYSES**				**SAMPLE[1]**	**PREP[2]**	
	METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID		
A,C	RSST[10]	Energy	see 10 below	N/A	X	d[6]	e
A	Distillation[10]	CN	LA-695-102	PNL-ALO-285	X	d[6]	e
C	IC[13]	NO_2^- NO_3^-	LA-533-105	PNL-ALO-212	X	d[6]	e
A,C	IC[16]	Br	LA-533-105	PNL-ALO-212	X	d[6]	e
C	Titration[13]	OH^-	LA-661-103	PNL-ALO-228	X	d[6]	e
A,C	Furnace Oxidation[14]	TOC	LA-344-105	PNL-ALO-380	X	d[6]	e
C	ICP[15]	Cr Mn	LA-505-151	PNL-ALO-211	X	d[6]	e
C	Gravimetric[11]	% H_2O	LA-564-101	PNL-ALO-504	X	d[6]	e

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

[3]PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate,

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

[5]Dry weight basis.

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

[7]Action limit converted from weight basis assuming liquid density of 1.0 g/mL.

[8]Tracer or carrier may be used in place of a spike and results corrected for recovery.

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

[10]RSST performed only if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be fo

:al and Physical Analytical Requirements

ANALYSES

COMMENTS	REPORTING LEVELS	
genization Test - Per Laboratory Discretion	FORMAT I	Early Notify
Blank - Not Required	FORMAT II	Process Control
ell Blank - Per Laboratory Discretion	FORMAT III	Safety Screen
	FORMAT IV	Waste Management
	FORMAT V	RCRA Compliance
	FORMAT VI	Special

TANK	#CORES	RISER#
C-103	2	2 and 7

QUALITY CONTROL[3]						CRITERIA			FOR-MAT
JP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
mpl	N/A	N/A	ea AB	±10	90-110	J/g[5]	> 481	unknown	I, III, IV
mpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III, IV
mpl	1/mtrx	ea PB	ea AB	±10	90-110	µg C/mL	> 30,000[7]	5,780 to 8,670	I, III[12], IV
mpl	see[9]	ea PB	ea AB	±10	90-110	µg/mL	100	unknown	I,III,IV
/A	N/A	N/A	N/A	N/A	N/A	none	presence	unknown	I, III, IV

QUALITY CONTROL[3]						CRITERIA			FOR-MAT
JP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	
mpl	N/A	N/A	ea AB	±20	80-120	J/g[5]	> 481	unknown	I,III,IV
mpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	> 39,000[7]	unknown	I,III,IV
mpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	19,800 to 29,800	IV
							none	2,070 to 3,100	IV
mpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	1200	unknown	I,III,IV
mpl	N/A	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV
mpl	1/mtrx	ea PB	ea AB	±10	90-110	µg C/mL	> 30,000[7]	5,780 to 8,670	I, III, IV
mpl	see[9]	ea PB	ea AB	±10	90-110	µg/mL	none	45.7 to 68.6	IV
							none	unknown	IV
mpl	N/A	N/A	ea AB	±20	80-120	wt%	< 17	unknown	I, IV

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

d in WHC-SD-WM-TP-104.

Table 3: C-103 Chemical, Radiolo

[11]This analysis is required if moisture analysis by TGA exceeds the notification limit.
[12]These analyses are primary analyses for the organic DQO, but also are secondary analyses for the safety scr
and reported within 90 days of receipt of the last sample at the laboratory dock.
[13]This analysis is required if the energy equivalent of the TOC assay by hot persulfate is > 125% of the DSC val
[14]This analysis required if the energy equivalent of the TOC by hot persulfate is < 75% of the DSC value.
[15]This analysis is required if the energy equivalent of the TOC assay by furnace oxidation is < 75% of the DSC
[16] This analysis required if Li exceeds notification limit.

ning DQO. Therefore, if the DSC limit is exceeded, these analyses must be performed

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5.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank C-103 characterization project are listed in Table 4.

Table 4: Tank C-103 Project Key Personnel List

Individual	Organization	Responsibility
J. G. Kristofzski	222-S Analytical Operations	Program Support Manager of Analytical Operations
S. G. McKinley	325 Analytical Chemistry Laboratory	Project Manager for Single Shell Tank (Core Sampling) Projects
R. D. Schreiber	TWRS Characterization Support	Tank C-103 Tank Characterization Plan Cognizant Engineer
D. A. Turner	Organic Tanks Safety Program	Organic Safety Program Manager
H. Babad	Characterization Program	Safety Screening Point of Contact
J. L. Deichman	Analytical Services	Manager of Analytical Services Program Management and Integration
M. J. Kupfer	Process Systems Engineering	Pretreatment Point of Contact
East Tank Farm Operations Shift Manager	Tank Farm Operations	200 East Tank Farm Point of Contact if Action Limit is Exceeded (373-2689)

6.0 EXCEPTIONS, CLARIFICATIONS AND ASSUMPTIONS

6.1 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 the samples taken. If necessary, the Pretreatment Program will personally contact the laboratory via Letter of Instruction or Memorandum of Understanding 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 run on a quarter-segment level and that the notification limit for the DSC analysis is 125 cal/g. However, the soon to be released revision of the safety screening DQO has changed the requirements such that the cyanide analysis is now to be run on a half-segment level and the DSC notification limit is 115 cal/g (dry weight basis). Therefore, although this Tank Characterization Plan uses the current Safety Screening

DQO, it specifies that cyanide is to be run on a half-segment basis and that notification shall be made if the DSC value exceeds 115 cal/g (dry weight basis).

In the organic DQO, Cs^{137} and Sr^{90} on half-segments are requested as secondary analyses. However, these have not been requested as primary analyses, nor are there any other primary analyses that would require Cs^{137} or Sr^{90} to be conducted should it exceed a notification limit. Therefore, it has been decided by both the Organic Safety Program and the Characterization Program that Cs^{137} and Sr^{90} on a half-segment level should not be analyzed.

In the organic DQO the method of analyses for principal organic species, equilibrium moisture content, and Cr and Mn oxidation states has not been developed at this point. Therefore, if it is necessary to analyze these secondary constituents, archived samples will be used for analyses at a later date.

6.2 CLARIFICATIONS AND ASSUMPTIONS

A number of clarifications and assumptions relating to the notification limits or decision thresholds identified in the applicable DQO efforts need to be made with respect to the analyses in Table 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 thermal gravimetric analysis.

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

NOTE: If there is greater than 90 percent water in a sample, converting to a dry weight bases may lead to a large error in the DSC value.

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

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

NOTE: If a density of 1.5 is assumed, the notification limit becomes 41 $\mu Ci/g$.

- Neither the safety screening nor the organic DQO, upon which some of the analyses in Table 3 are based, sufficiently addresses the analyses of any drainable liquid present. To adequately characterize the tank, all analyses performed on the solids for the safety screening and organic DQOs shall also be performed on any drainable liquids and the field blank, with the exception of total alpha analysis.

- The Pretreatment Program has requested 125 mL of the solid composite material for process development work. Two test plans (Lumetta 1994, and 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 plans. This approval will not only ensure that the DQO process has been used in the generation of the test plans 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 plans are approved by the Characterization Program, the Characterization Program will direct the performing laboratory, via letter of instruction, to allow shipment of the sample material.

- In the organic DQO it is unclear as to when secondary analyses are to be run. Whether or not a secondary analysis is run depends on the comparison between the value of the energy equivalent for TOC, X, and the DSC energetics value. The energy equivalent of TOC is given in equation (3).

$$X = (wt\%\ TOC\ dry\ weight\ basis) * \frac{151\ cal/g}{5} \qquad (3)$$

NOTE: 151 cal/g represents the energy equivalent of 5 wt% TOC (based on sodium acetate average energetics standard).

Secondary analyses for the Organic Safety Program are run on half-segments based on this equation. Therefore the following rules apply:

- If X by hot persulfate is ≤ 75% of the DSC value, run TOC by furnace combustion on the half-segment.
- If X by hot persulfate is ≥ 125% of the DSC value, run nitrite, nitrate, and hydroxide analyses on the half-segment.
- If X by furnace combustion is ≤ 75% of the DSC, analyze for the presence of Mn and Cr on the half-segment.

7.0 DELIVERABLES

All analyses of tank C-103 waste material shall be reported as Formats I, III, or IV as indicated in Table 3. Additional information regarding reporting formats is given in Schreiber (1994a).

7.1 PROGRESS REPORTS

Each laboratory performing analyses on tank C-103 waste material from this core sampling project shall provide monthly status reports to the Characterization Program. This report shall contain 1) a summary of the activities on the analysis of tank C-103, 2) preliminary results to the program, and 3) schedule and cost information on a DQO basis.

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

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

Schedule reporting shall consist of the following:

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

7.2 FORMAT I REPORTING

Table 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 Organic 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.

7.3 FORMAT III REPORTING

A Format III report, reporting the results of the primary safety screen analyses, shall be issued to the Organic Safety Program, the Safety Screening Representative, Characterization Support, Waste Tanks Process Engineering, the Characterization Program Office, and the Los Alamos Technical Associates, Tank Characterization Resource Center and Tank Characterization Database representatives within 45 days of receipt of the last segment of the last core sample at the laboratory loading dock. The DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analyses for the safety screening secondary analytes are required, these results shall be provided within 90 days of receipt of the last segment of the last core sample at the

laboratory loading dock. No calibration data are requested for these reports. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

7.4 FORMAT IV REPORTING

Analytical results requested for the characterization project of tank C-103 shall be compiled into a Format IV type data package. The data package shall be provided to Analytical Services, Los Alamos Technical Associates, and the Tank Characterization Resource Center representatives 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 Database representative 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).

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 documented. Changes may be documented through the use of internal characterization changes notices or analytical deviation reports for minor low-impact changes and documented in applicable laboratory notebooks. All significant changes (such as changes in scope) shall be documented by Characterization Support via an Engineering Change Notice to this Tank Characterization Plan. All changes shall also be clearly documented in the final data report.

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

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