

Waste Characterization Plan for the Hanford Site Single-Shell Tanks

Appendix I - Test Plan for Sampling and Analysis of Ten Single-Shell Tanks

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CONTENTS

11.0	INTRODUCTION	I1-1
11.1	SORT ON RADIOACTIVE WASTE TYPE MODEL	I1-2
11.1.1	Sort on Radioactive Waste Type Model Assumptions	I1-3
11.1.2	Sort on Radioactive Waste Type Model Results	I1-4
11.2	PRELIMINARY RECOMMENDATIONS REPORT	I1-6
11.2.1	Analyte Priorities, Concentration Thresholds, and Detection Limit Goals	I1-6
11.2.2	Decision Quality	I1-16
11.2.3	Evaluation of Impacts	I1-20
12.0	SUMMARY OF LESSONS LEARNED FROM PHASE IA/IB	I2-1
12.1	SAMPLING OPERATIONS	I2-1
12.2	HOT CELL OPERATIONS	I2-2
12.3	ANALYTICAL OPERATIONS	I2-2
12.3.1	Metal Ions	I2-2
12.3.2	Anions and Wet Chemical Analyses	I2-2
12.3.3	Radiochemical Analysis	I2-3
12.3.4	Organic Analyses	I2-4
12.3.5	Characteristic Testing	I2-4
12.4	ADMINISTRATIVE/ORGANIZATIONAL OPERATIONS	I2-4
13.0	SELECTION OF THE NEXT TEN SINGLE-SHELL TANKS FOR CHARACTERIZATION	I3-1
13.1	SINGLE-SHELL TANK SELECTION CRITERIA	I3-1
13.1.1	Primary Single-Shell Tank Selection Criteria	I3-1
13.1.2	Secondary SST Selection Criteria	I3-2
13.1.3	Next Ten SSTs Selected	I3-3
13.2	PRELIMINARY INTEGRATED CORE SAMPLE SCHEDULE	I3-7
14.0	DESCRIPTION OF THE SINGLE-SHELL TANKS SELECTED FOR SAMPLING	I4-1
14.1	TANK 241-B-111	I4-1
14.2	TANK 241-C-112	I4-1
14.3	TANK 241-C-109	I4-1
14.4	TANK 241-C-106	I4-2
14.5	TANK 241-C-110	I4-2
14.6	TANK 241-T-111	I4-2
14.7	TANK 241-T-110	I4-3
14.8	TANK 241-BX-107	I4-3
14.9	TANK 241-BX-103	I4-3
14.10	TANK 241-S-104	I4-3
14.11	DESCRIPTION OF THE SAMPLING	I4-4
15.0	OBJECTIVES FOR SAMPLING AND ANALYSIS	I5-1
15.1	BASELINE CASE SINGLE-SHELL TANKS	I5-1
15.1.1	Single-Shell Tank Waste Constituent Inventory	I5-1
15.1.2	Physical Properties	I5-6
15.1.3	Waste Designation	I5-9
15.1.4	Evaluation of Transport Constants	I5-10
15.1.5	Analysis of Errors	I5-10

CONTENTS (continued)

15.1.6	Vertical Spatial Variation	I5-11
15.1.7	Horizontal Spatial Variation	I5-11
15.1.8	Holding Time	I5-12
15.1.9	Single-Shell Tank Waste Standards Program	I5-12
15.1.10	Tank Stability	I5-12
5.2	SINGLE-SHELL TANKS IDENTIFIED IN PUBLIC LAW 101-510	I5-13
15.2.1	Single-Shell Tank C-112	I5-13
15.2.2	Single-Shell Tank C-109	I5-14
15.2.3	Single-Shell Tank C-106	I5-14
15.2.4	Single-Shell Tank T-110	I5-16
I6.0	SAMPLING AND ANALYSIS SCHEME AND TEST PROCEDURES	I6-1
I6.1	BASELINE-CASE SINGLE-SHELL TANKS	I6-2
16.1.1	Baseline-Case Sample and Analysis Scheme	I6-2
16.1.2	Core Compositing	I6-8
16.1.3	Baseline-Case Single-Shell Tank Core Sample Utilization	I6-10
I6.2	SINGLE-SHELL TANKS IDENTIFIED IN PUBLIC LAW 101-510	I6-17
16.2.1	Ferrocyanide Tanks (C-112 and C-109)	I6-17
16.2.2	High Heat Single-Shell Tanks (C-106)	I6-32
16.2.3	Gas Generating Tanks (T-110)	I6-43
I6.3	DATA REPORTING	I6-49
I7.0	REFERENCES	I7-1

LIST OF FIGURES

I1-1	Recommended Core Sample Locations for a Typical SST	I1-19
I1-2	Dose Impacts (Total) of Different Scenarios	I1-26
I1-3	Dose Impacts of Alternate Configurations for Each Scenario . . .	I1-27
I3-1	Preliminary Integrated Core Sample Schedule	I3-9
I6-1	Core Sample Analysis Scheme	I6-1
I6-2	Baseline Case Single-Shell Tank Sample and Analysis Flow Diagram	I6-3
I6-3	Single-Shell Tank Extrusion Logsheet	I6-4
I6-4	Baseline Case Single-Shell Tank Sample Allocation	I6-9
I6-5	Sample and Analysis Flow Diagram for Single-Shell Tanks C-112 and C-109	I6-19
I6-6	Sample Allocation for Single-Shell Tanks C-112 and C-109	I6-25
I6-7	Sample and Analysis Flowsheet for Single-Shell Tank C-106 . . .	I6-33
I6-8	Sample Allocation for Single-Shell Tank C-106	I6-37
I6-9	Sample and Analysis Flow Diagram for Single-Shell Tank T-110 . .	I6-44
I6-10	Sample Allocation for Single-Shell Tank T-110	I6-48

LIST OF TABLES

I1-1	Summary of Waste Type by SORWT	I1-5
I1-2	Analyte Priority List Based on Combination of All Three Methods	I1-8
I1-3	List of Analytes Without Tank Inventories	I1-9
I1-4	Detection Limit Goals and Analytical Detection Limits for Type I Carcinogens	I1-10
I1-5	Detection Limit Goals and Analytical Detection Limits for Type I Noncarcinogens.	I1-11
I1-6	Detection Limit Goals and Analytical Detection Limits for Type II Carcinogens	I1-12
I1-7	Detection Limit Goals and Analytical Detection Limits for Type II Noncarcinogens.	I1-12
I1-8	Detection Limit Goals and Analytical Detection Limits for Type III Carcinogens	I1-13
I1-9	Detection Limit Goals and Analytical Detection Limits for Type III Noncarcinogens	I1-14
I1-10	Detection Limit Goals and Analytical Detection Limits for Unranked Carcinogens	I1-14
I1-11	Detection Limit Goals and Analytical Detection Limits for Unranked Noncarcinogens	I1-15
I1-12	Process Category 1 - Sampling Operations.	I1-22
I1-13	A&B Process Category 2 Sample Receipt and Handling	I1-22
I1-14	Process Category 3 Sample Transfer (from hotcell to hood).	I1-23
I1-15	Process Category 4 Sample Analysis (Empirical)	I1-23
I1-16	Process Category 4 Sample Analysis (Inferred)	I1-24
I1-17	Annual Dose per Characterization Worker	I1-25
I3-1	Preliminary Optimized SST Sampling Order	I3-4
I4-1	Sample Breakdown and Subsampling Procedures.	I4-5
I5-1	Analytes and Methods for Analysis of Core Composites From Next Ten Single-Shell Tanks	I5-2

LIST OF TABLES (continued)

I5-2	Baseline Case SST Physical Analysis	I5-7
I5-3	Additional Analyses Required for SST C-106	I5-15
I6-1	Prehomogenized Sample Utilization	I6-11
I6-2	Estimate of Segment Archive Sample Utilization	I6-12
I6-3	Estimate of Core Composite Sample Utilization	I6-13
I6-4	Water Digestion Sample Utilization	I6-14
I6-5A	Baseline Case % Recovery Study Rheology Core	I6-15
I6-5B	Baseline Case % Recovery Study Non-Rheology Core	I6-16
I6-6	Prehomogenized Sample Utilization (C-112 and C-109)	I6-27
I6-7	Estimate of Subsegment Sample Utilization (C-112 and C-109)	I6-28
I6-8	Estimate of Subsegment Archive Sample Utilization (C-112 and C-109)	I6-28
I6-9	Estimate of Core Composite Sample Utilization (C-112 and C-109)	I6-29
I6-10	Water Digestion Sample Utilization	I6-30
I6-11	% Recovery Sensitivity Study (C-112 and C-109)	I6-31
I6-12	Prehomogenized Sample Utilization (C-106)	I6-38
I6-13	Estimate of Segment Sample Utilization (C-106)	I6-39
I6-14	Estimate of Core Composite Sample Utilization (C-106)	I6-40
I6-15	Water Digestion Sample Utilization (C-106)	I6-41
I6-16	% Recovery Sensitivity Study for SST C-106	I6-42
I6-17	Prehomogenized Sample Utilization (T-110)	I6-50
I6-18	Estimate of Segment Sample Utilization (T-110)	I6-51
I6-19	Estimate of Segment Archive Sample Utilization	I6-51
I6-20	Estimate of Core Composite Sample Utilization (T-110)	I6-52

LIST OF TABLES (continued)

I6-21	Water Digestion Sample Utilization (T-110)	I6-53
I6-22	% Recovery Sensitivity Study for SST T-110	I6-54

METRIC CONVERSION CHART		
1 inch	=	2.54 centimeters
1 foot	=	30. centimeters
1 gallon	=	3.8 liters
1 ton	=	0.9 metric tons
$1 \text{ } ^\circ\text{F} = \left(\frac{9}{5} \text{ } ^\circ\text{C}\right) + 32$		
1 Btu/h = 2.980711 E-01 watts (International Table)		

LIST OF TERMS

1C	first-cycle decontamination
224	lanthanum fluoride
2C	second-cycle decontamination
5-6	Cell 5-6 at B Plant
AA	atomic absorption
ADL	analytical detection limit
ANOVA	analysis of variance
BL	B Plant low-level waste
CCPLX	Concentrated Complexant
CLASS	Waste Classification Model
CN	Cyanide
COD	chemical oxygen demand
CT	concentration threshold
CW	coating waste
DLG	detection limit goal
DQO	Data Quality Objectives
DSC	differential scanning calorimetry
DSSF	Double-Shell Slurry Feed
DW	dangerous waste
EB	evaporator bottoms
Ecology	Washington State Department of Ecology
EDTA	ethylenediaminetetraacetic acid
EHW	extremely hazardous waste
EPA	U.S. Environmental Protection Agency
EVAP	evaporator feed
FeCN	ferrocyanide waste
FP	fission product
FY	fiscal year
Gas	gas-generator tank
GC	gas chromatography
GEA	gamma energy analysis
GFAA	Graphite Furnace Atomic Absorption
HCN	Hydrogen Cyanide
HEDTA	hydroxyethylethylenediaminetriacetic acid
HH	high-heat waste
HP_C	high-performance liquid chromatography
HYAA	hydride atomic absorption
IC	ion chromatography
ICP	inductively coupled plasma
IX	ion-exchange
KOH	Potassium Hydroxide
LTRR	Long-Term Release Risk
MS	mass spectrometry
MSD	matrix spike duplicate
NCPLX	None-Complexed Waste
NPH	normal paraffin hydrocarbon
NRC	U.S. Nuclear Regulatory Commission
OSM	Office of Sample Management
OWW	organic wash waste
P	PUREX high-level waste

LIST OF TERMS

PA	Performance Assessment
pH	
PLM	Polarized Light Microscopy
PNL	Pacific Northwest Laboratory
PRR	Preliminary Recommendations Report
PSS	PUREX supernatant sludge
Public Law 101-510	U.S. Congress, House, 1990, "Safety Measures for Waste Tanks at Hanford Nuclear Reservation," Public Law 101-510, Section 3137, H.R. 4739, Government Printing Office, Washington D.C.
	plutonium-uranium extraction
PUREX	Quality Assurance
QA	high-level REDOX
R	reduction oxidation
REDOX	Supplemental Environmental Impact Statement
SEIS	Sort On Radioactive Waste Type
SORWT	strontium sludge
SRS	single-shell tank
SST	strontium semiworks/hot semiworks
SSW/HS	Short-Term Intruder Risk
STIR	tributyl phosphate
TBP	ferrocyanide-scavenged tributyl phosphate
TBP-F	toxic characteristic leach procedure
TCLP	toxic equivalent concentration
TEC	Thermal Gravimetry
TGA	total organic carbon
TOC	Track Radioactive Components
TRAC	Hanford Federal Facility Agreement
Tri-Party Agreement	Tank Waste Characterization Technology
TWCT	volatile organic analysis
VOA	Waste Classification
WC	Waste Characterization Plan
WCP	Westinghouse Hanford Company
Westinghouse Hanford	X-Ray Diffraction
XRD	

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11.0 INTRODUCTION

This appendix describes the sampling and analysis of the next ten single-shell tanks (SST) following the successful sampling of SSTs B-201 and B-202. SST T-203 shall not be core sampled sequentially after B-201 and B-202, as originally planned in Appendix F, because this tank would not have provided information on tank safety issues and it contains an identical waste type as the previous two SSTs. Therefore, sampling and analysis of T-203 at the present time was considered repetitious and not an efficient utilization of the limited available resources. This test plan will outline methodology for characterization of the next ten SSTs, summarize lessons learned in the laboratory during Phase IA/IB, identify criteria for tank selection, and detail the analysis to be performed during the characterization of each tank.

The sampling, analysis, and data collection, detailed by this test plan, are being performed to support the final SST closure date of 2,018 identified in the Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement). The criteria governing SST closure decisions must be formulated in the Supplemental Environmental Impact Statement (SEIS). In order to meet the 2018 closure date, the SST Systems Engineering Study has identified a need to accelerate the SEIS and the Record of Decision to 1996. A proposal to pursue this option is presently being reviewed. The data to support the SST closure criteria development must be collected well in advance of the 1996 date. The data collection deadline for criteria development has been estimated as December 1993. Therefore, to efficiently characterize the waste in the SSTs, given the limited amount of time and resources, an integrated and optimized sampling schedule must be developed from a representative sample group of SSTs. It is imperative to acquire waste characterization data on as many different waste types as possible and to have every program take maximum advantage of each sampling event before closure of the SEIS data gathering window. The only feasible method to accomplish this task before the SEIS closure deadline is to attempt to categorize the tanks into groups that have similar chemical compositions and physical characteristics.

A model has been developed to categorize SSTs into groups expected to exhibit similar chemical and physical characteristics based on major waste types and processing histories identified from historical records. This method has identified 29 different groups of tanks. These 29 groups encompass 131 tanks and 90% of the total waste volume contained in SSTs. The 18 remaining SSTs were not predicted to fall into any group and were encompassed in a 30th ungrouped category. The validity of the predicted groups was then statistically tested using quantitative information from a limited number of tanks. The groups predicted by this method were shown to be statistically significant based on the available data from core samples obtained in 1985 and 1986. The analytical variability was shown to be reduced by grouping the tanks according to this model. The model has been a valuable tool aiding in the selection of the next 10 SSTs.

The Phase IC analytical plan was supposed to be based upon a recommendations report prepared by Pacific Northwest Laboratory (PNL) using Phase IA and IB information. Phases IA, IB, and IC are described in detail in Chapter 1.1. The verification and preparation of data packages for Phase IA

and IB analysis has taken longer than anticipated. Pacific Northwest Laboratory has prepared sections of the preliminary recommendations report focusing on analyte priorities, concentration threshold limits, decision quality, and impact analysis. The scopes of these efforts have been summarized in Sections 1.2 through 1.2.3. The original purpose of the program was to issue a generic Phase IC waste analysis plan for the remaining SSTs. Although completion of a generic Phase IC Waste Characterization Plan will not be possible until all the Phase IA and IB information has been analyzed, development and initiation of limited Phase IC sampling and analysis can proceed. The characterization goals and strategies will be iterated based upon new analytical results from each SST sampled. In addition, the sampling and analysis needs for those tanks identified in U.S. Congress, House, *Safety Measures for Waste Tanks at Hanford Nuclear Reservation*, Public Law 101-510 Section 3137, will be different than the generic plan. Therefore, the sampling and analysis of these early Phase IC tanks will be described in test plans for each set of tanks.

The objectives for sampling and analysis of the next 10 SSTs are to characterize the physical and chemical properties of the waste contained in the selected tanks. This characterization information will directly support most of the programs involved in the effort to close the SST operable units. The acquired data can also be used to check the laboratory's analytical performance and to statistically verify the grouping results of the SORWT model.

11.1 SORT ON RADIOACTIVE WASTE TYPE MODEL

The Sort On Radioactive Waste Type (SORWT) model has been developed to categorize tanks into groups expected to have similar physical characteristics and chemical compositions. In light of the complex physical and chemical histories of the SSTs, especially when several different waste types have been mixed or processed together, the SORWT model does not attempt to predict the composition of a waste tank nor does it use existing predictions of tank compositions (TRAC). Instead, the sorting method concentrates on the different types of waste introduced into each SST and the process history of each SST. Although the actual chemical reactions and phase equilibria may be unknown when two waste types are combined in an SST, it can be assumed that similar reactions and similar equilibria occur in other SSTs when the same two waste types are mixed.

The fundamental thesis of the SORWT model is that SSTs that predominantly received the same mixture of waste types will be more similar to one another than to SSTs that received different mixtures of waste types. In addition, largely supernatant waste types do not have as significant an effect on the character of the wastes remaining in the tank as solids-forming waste types. Therefore, if the primary and secondary solids-forming waste types can be identified for each SST, then the tanks can be grouped based on this criteria. Thus, information about the character of the waste in the remaining members of a group can be deduced from the information obtained by the analysis of samples from a tank representative of that group.

The waste type judged to be the most significant contributor to the solids volume in any specific SST was identified as the primary waste type. This evaluation was made on the basis of waste volume introduced into each tank and the solids accumulation during the regime of that particular waste. The second most significant solids-forming waste type was identified as the secondary waste type. When appropriate, a tertiary and other waste type also were identified.

The principal source of SST waste type information used by this model was *A History of the 200 Area Tank Farms* (Anderson 1990). This document contains much of the available processing history for each of the 149 SSTs from 1944 until 1980. Although this source contains extensive information pertaining to waste types, volumes, and tank transfers, the source information contained many inconsistencies. The historical records used to generate Anderson (1990) were often inaccurate and/or incomplete. The methods utilized to measure accumulated solid and liquid volumes during the early history of the Hanford Site produced inconsistent inventories. In fact, solids inventories were not routinely taken until the mid-1950s. Often, tank transfer information was missing. Despite these deficiencies, the Anderson document is the best source of SST historical information and a qualitative assessment about the main solids-forming waste types contained in each SST can be accurately determined.

The volumes of waste contained in each SST were obtained from the *Tank Farm Surveillance and Waste Status Summary Report* (Hanlon 1990). These values include, on a per tank basis, Total Waste Volume, Volume of Salt Cake, Volume of Sludge, and Volume of Supernate. It is assumed that these values are more accurate than those final values found in Anderson (1990) because they were obtained more recently.

11.1.1 Sort on Radioactive Waste Type Model Assumptions

The underlying assumptions utilized by the SORWT model are as follows:

- The information contained within Anderson (1990) was sufficient to qualitatively identify and rank, relative to one another, the waste types that contributed to the accumulated solids in each individual SST.
- The primary and secondary solids-forming waste types were responsible for the majority of the physical characteristics and chemical compositions of the waste remaining in each SST.
- Supernatant wastes that were not allowed to remain in a tank for a great period of time (and later pumped out of the SST) had less influence on the physical and chemical character of the waste relative to the solid waste types that remained in the tanks.
- Single-shell tanks were often sluiced sometime during their processing history. Waste types present in the tank before the most recent sluicing were not considered relevant by this model.

- Use of broad-ranging, less descriptive waste types, such as non-complexed (NCPLX), concentrated complexed (CCPLX), evaporator feed (EVAP), and/or double-shell slurry feed (DSSF), were avoided whenever possible. The previous nomenclature for those waste types was preferred, if available. However, a broad category identifying the tank waste as either Non-Complexed, Complexed, or Ferrocyanide-Scavenged Waste has been included in the SORWT model to aid in evaluating the results of the model.

11.1.2 Sort on Radioactive Waste Type Model Results

The SORWT model has predicted the existence of 29 waste type groups ranging from a high of 21 tanks per group to a low of 2 tanks per group. These 29 waste type groups encompass 131 tanks and 90% of the total waste volume. A thirtieth group contains the 18 solitary SSTs, which did not fall into any waste type group. Table 11-1 presents a summary of the SST waste type groups predicted by the SORWT model.

The first column of Table 11-1 identifies the group number. The second column contains the primary and secondary waste types that were used as the grouping criteria. Column 3 reports the number of tanks in each individual group. An asterisk in column 3 indicates that this group has already been core sampled at least one time. Most of these previous core sample analyses were not as complete as core sample analyses conducted under the current characterization program and these tanks must be resampled in the future. These core samples do provide some preliminary chemical characterization for these groups. The fourth, fifth, and sixth columns respectively contain the volume of salt cake, sludge, and total waste represented by each waste type group. Columns 7, 8, and 9, respectively, report the percentage volume of salt cake, sludge, and total waste compared to all 149 SSTs. A total has been accumulated for columns 3 through 9, encompassing the 29 waste type groups predicted by the SORWT model. The ungrouped tanks were not included in this total. A review of Table 11-1 will quickly reveal that Group I is by far the most significant group. This group includes 21 tanks, 36% of the total salt cake volume, and over 1/4 of the total waste in all 149 SSTs. The first 3 groups represent nearly 1/2 of the total waste volume in all 149 SSTs which demonstrates the potential usefulness of the SORWT model. Table 11-1 also identifies groups which have relatively no significance, such as Groups XII and XIX, that contain almost no waste. This information can be used in allocating time and resources for core sampling.

To support the accelerated SEIS, it has been determined that some kind of SST grouping methodology must be developed and implemented. The SORWT SST grouping model presents a methodology that is both simple to understand and logical in its assumptions and construction. The SST groups predicted by the SORWT model are statistically significant and reduce the variability in the concentrations for a selected set of analytes.

Table II-1. Summary of Waste Type by SORW.

GROUP NUMBER	PRIMARY & SECONDARY WASTE GROUP TYPE		NUMBER OF TANKS IN GROUP	VOLUME SALT CAKE IN GROUP (KGAL)	VOLUME SLUDGE IN GROUP (KGAL)	TOTAL VOLUME IN GROUP (KGAL)	% VOLUME SALT CAKE ALL TANKS	% VOLUME SLUDGE ALL TANKS	% TOTAL VOLUME ALL TANKS
I.	R	EB	21	8361	1328	9798	36%	11%	27%
II.	TBP-F	EB-ITS	10	3344	636	3980	14%	5%	11%
III.	EB	1C	9 *	3945	40	3985	17%	0%	11%
IV.	224		8	0	277	280	0%	2%	1%
V.	R		7	0	888	892	0%	7%	2%
VI.	TBP	CW	7 *	3	458	489	0%	4%	1%
VII.	EB	R	5	1864	127	2037	8%	1%	6%
VIII.	1C	TBP	5	0	709	715	0%	6%	2%
IX.	TBP-F	1C	5	0	465	478	0%	4%	1%
X.	EB	CW	4	1520	124	1755	6%	1%	5%
XI.	1C	EB	4	0	552	553	0%	4%	2%
XII.	HS		4	0	11	11	0%	0%	0%
XIII.	DSSF	NCPLX	4 *	1717	387	2113	7%	3%	6%
XIV.	2C	224	3	0	892	904	0%	7%	2%
XV.	2C	5-6	3 *	0	511	516	0%	4%	1%
XVI.	R	RIX	3	0	368	368	0%	3%	1%
XVII.	CW	EB	3	10	190	204	0%	2%	1%
XVIII.	CW	MIX	3	0	145	192	0%	1%	1%
XIX.	CW		3	0	10	13	0%	0%	0%
XX.	TBP	EB-ITS	2	771	87	907	3%	1%	2%
XXI.	CW	TBP	2 *	0	574	577	0%	5%	2%
XXII.	EB	TBP	2	481	0	481	2%	0%	1%
XXIII.	SRS	TBP	2 *	0	372	429	0%	3%	1%
XXIV.	1C	EB-ITS	2	152	257	429	1%	2%	1%
XXV.	TBP		2	0	248	248	0%	2%	1%
XXVI.	TBP	1C-F	2 *	0	205	208	0%	2%	1%
XXVII.	CCPLX	DSSF	2	40	9	151	0%	0%	0%
XXVIII.	R	DIA	2	0	148	148	0%	1%	0%
XXIX.	1C	CW	2	0	117	119	0%	1%	0%
	TOTAL		131	22208	10135	32980	95%	80%	90%
XXX.	UNGROUPED TANK		18	1241	2509	3794	5%	20%	10%

* - Waste Groups Already Sampled

11.2 PRELIMINARY RECOMMENDATIONS REPORT

The proposed objectives of the Preliminary Recommendations Report (PRR) is to support continued sampling and analysis of SSTs by providing recommendations as to the number of cores required, the number of duplicates needed, and the identification of whether laboratory analyses should be performed on core composites or individual segments. The report will address three major areas:

- Analyte priorities and concentration thresholds
- Decision quality and Data Quality Objectives
- Evaluation of impacts to worker exposure, schedule, and costs.

Each of these topics is discussed in the Sections 1.2.1 through 1.2.3.

11.2.1 Analyte Priorities, Concentration Thresholds, and Detection Limit Goals

The U.S. Environmental Protection Agency (EPA) has defined a process named Data Quality Objectives (DQO), which assists in defining the type, quality, and quantity of the data needed to evaluate waste sites, or in this case, SSTs. These DQOs help focus characterization and streamline the remediation and closure process. Analyte priorities and proposed detection limit goals (based on concentration threshold concept) are preliminary DQOs that have been developed for the SST waste characterization effort based on health risk and regulation criteria. Volume 2 of the PRR (Buck et al. 1991), provides a detailed description and analysis of these DQOs.

The vast number of analytes that are known or suspected to be in SSTs require that priorities for chemical and radiological analysis be established. The criteria that has been developed for determining the importance of analytes is based on public health risk concerns, and state and federal regulations.

Three different methods were used to prioritize the SST analytes: Long-Term Release Risk (LTRR), Short-Term Intruder Risk (STIR), and Waste Classification (WC). The LTRR method used an integrated source term, transport, and exposure code to develop a health risk-based analyte priority list based on site-specific information. The STIR method used generic intruder scenarios developed by the U.S. Nuclear Regulatory Commission (NRC) to prioritize SST analytes based on source term and toxicity/dose parameters. The WC method was based on guidance from NRC's 10 CFR 61 (classification of waste for near-surface disposal) regulation for radioactive waste and Washington State Department of Ecology (Ecology) WAC 173-303-084 regulation for dangerous waste. All three methods used Track Radioactive Components (TRAC) inventory estimates as input in lieu of better source term data.

Each of these three methods produced a list of prioritized SST analytes that could be used, independently or combined, to improve the design of the SST waste characterization plan. A combined analyte priority list, based on the highest relative risk or waste class type for each analyte (Type I

analytes are more significant than Type II analytes) from the three methods, was used to define Type I, II, and III analytes.

Type I analytes constitute 99% of the cumulative risk or waste class index for all analytes and tank groups; Type II analytes constitute 0.9% of the cumulative risk or waste class index for all analytes and tank groups; and Type III analytes constitute less than 0.1% of the cumulative risk or waste class index for all analytes and tank groups. The Type I and II analyte groups were further divided into two subgroups each (Type I-A, I-B, II-A, and II-B) to provide more detail. A list of the carcinogen and noncarcinogen Type I-A and -B, Type II-A and -B, and Type III analytes, based on the combined analyte priority list, is provided in Table II-2.

Twenty-four analytes could not be prioritized because tank inventories were not available. These analytes, listed on Table II-3, do not appear to be present in the tanks in large quantities based on the absence of these analytes in TRAC and other historical sources of information. A preliminary assumption is that these analytes do not present a significant health risk to the public and were not used for this analyte priorities study. Future efforts will be conducted to confirm this assumption.

In characterizing SST waste, it is important to know at what quantity an analyte is considered a significant health risk or waste class contributor. A concept called the concentration threshold (CT) was developed to assist in determining when an analyte is in sufficient quantities in the tank to be considered a significant risk or waste class contributor. The CT value is defined as the tank concentration of the analyte that represents 1% of the cumulative health risk or waste class for a tank group. A CT value was computed for each analyte, each tank group, and each of the three prioritizing methods (LTRR, STIR, and WC). The CT values provide information to

- (1) conduct qualitative analysis in planning waste characterization and
- (2) evaluate remediation technologies.

The CT concept provides information on when an analyte is in sufficient quantities in a tank to be considered a health risk. This concept can be carried further to define detection limit goals (DLGs) that represent the "lowest concentration of interest" in a tank for each analyte. The lowest concentration of interest for an analyte is defined as a detection limit goal (DLG), and can be used to identify current analytical detection limits (ADLs) that may not be adequate, based on health risk and waste class criteria. DLGs provide information on the quality of data needed to characterize SST waste.

These DLGs are computed by taking the most restrictive CT values for an analyte and dividing by 10 for CT_{STIR} and CT_{WC} values or dividing by 100 for CT_{LTRR} values. A safety factor of 10 is assigned to all DLGs because of the general variability in the ADL methods. An additional safety factor is assigned to the CT_{LTRR} because of the uncertainty in the risk-based code used. Future efforts will be conducted to reduce the uncertainty in the LTRR method and to determine statistically significant DLG values based on the CT concept.

Table II-2. Analyte Priority List Based on Combination of All Three Methods.

Carcinogen					Noncarcinogen				
Type I-A	Type I-B	Type II-A	Type II-B	Type III	Type I-A	Type I-B	Type II-A	Type II-B	Type III
241Am 14C 137Cs 129I 239Pu 240Pu 90Sr 99Tc 238U 90Y	242mAm 63Ni 238Pu 241Pu 235U	244Cm 93mNb 237Np	243Am 242Cm 234U	225Ac 227Ac 242Am 245Cm 135Cs 231Pa 233Pa 210Pb 210Po 223Ra 225Ra 226Ra 228Ra 106Ru 79Se 151Sm 229Th 230Th 234Th 233U	Al Bi Cr+6 EDTA F Fe Mn Na Ni NO2 NO3 OH PO4	Ba C6H5O7 Cd CM CO3 HEDTA Pb SiO3 SO4 Zr	Ag Fe(CN)6	C2H3O3 Ca K	C2O4 Ce Cl La SeO4 Sn WO4

C2O4 - Oxalate
 C2H3O3 - Hydroxyacetic acid
 C6H5O7 - Citric acid
 Fe(CN)6 - Ferrocyanide

Type IA analytes include 0.00% to 90.00% of cumulative ranking index
 Type IB analytes include 90.00% to 99.00% of cumulative ranking index
 Type IIA analytes include 99.00% to 99.90% of cumulative ranking index
 Type IIB analytes include 99.90% to 99.99% of cumulative ranking index
 Type III analytes less than 99.99% of cumulative ranking index
 (Note 'm' as part of the isotope number indicates metastable state)

Tables I1-4 through I1-11 provide the ADL and DLG values for each SST analyte group by Type (I, II, III, or unranked) and health effect (carcinogen or noncarcinogen). If the ADL is greater than the computed DLG, the ADL is identified as potentially inadequate and additional analytical methods development is required. To easily identify suspect ADLs, a ratio ADL-to-DLG was computed and included in Tables I1-4 through I1-11. If this ratio is significantly greater than 1.0, the ADL is considered inadequate with respect to the DLG. However, if the analyte concentration in the tank is orders-of-magnitude greater than either the ADL or the DLG, then additional efforts to revise the ADL will be unnecessary. It is important to understand that the ADL is suspect only if (1) it is greater than the DLG and (2) the quantities of that analyte in the tank is at or below the ADL.

In conclusion, the analyte priority list and the DLGs were developed using a health-risk code and regulatory criteria to determine preliminary DQOs for the SST waste characterization plan. It is important to note that these results are preliminary and will change as more information is gained from future sampling and analysis efforts. The analyte priority list and DLGs will be updated and refined for the continuing SST waste characterization effort.

Table I1-3. List of Analytes Without Tank Inventories.

<u>Radionuclides</u>	<u>Chemicals</u>
⁶⁰ Co	S ⁻²
³ H	Co
⁹⁴ Nb	NH ₄
⁵⁹ Ni	Tl
²⁴² Pu	Th
¹²⁶ Sn	Ti
²³² Th	U *
²³⁶ U	Zn
⁹³ Zr	As
	V
	Be
	Sb
	Hg
	Cu
	Sr *

*The inventory of these two analytes could have been calculated based on curie content.

Table II-4. Detection Limit Goals and Analytical Detection Limits for Type I Carcinogens.

Carcinogen Analyte	Detection Limit Goal ($\mu\text{Ci/g}$)	Analytical Detection Limit ($\mu\text{Ci/g}$)	Ratio of Limit ADL to DLG (None)
^{241}Am	3.0 E-05	4.0 E-04	1.3 E+01
^{242m}Am	3.0 E-05 ^a	1.0 E-04	3.3 E+00
^{14}C	6.4 E-06	5.0 E-05	7.8 E+00
^{137}Cs	3.8 E-02	1.0 E-03	2.6 E-02
^{129}I	2.1 E-08	7.0 E-06	3.3 E+01 ^b
^{63}Ni	5.8 E-03	TBD	NC
^{238}Pu	3.0 E-05	7.0 E-05	2.3 E+00
^{239}Pu	3.0 E-05	7.0 E-05	2.3 E+00
^{240}Pu	3.0 E-05	7.0 E-05	2.3 E+00
^{241}Pu	1.0 E-03 ^a	1.0 E-05	1.0 E-02
^{90}Sr	2.3 E-02	4.0 E-03	1.7 E-01
^{99}Tc	1.4 E-06	9.0 E-04	6.4 E+02 ^b
^{235}U	4.4 E-08 ^a	3.0 E-08	6.8 E-01
^{238}U	6.8 E-08 ^a	2.0 E-07	2.9 E+00
^{90}Y	5.7 E-02	TBD	NC

^aDetermined based upon the ADL of a different isotope.

^bSuspect Analytical Detection Limit.

NA = data or method is Not Available

TBD = detection limit To Be Determined

NC = value could not be computed.

Table 11-5. Detection Limit Goals and Analytical Detection Limits for Type I Noncarcinogens.

Noncarcinogen Analyte	Detection Limit Goal ($\mu\text{g/g}$)	Analytical Detection Limit ($\mu\text{g/g}$)	Ratio of ADL to DLG (None)
Al	6.3 E+00	1.3 E+01	2.1 E+00
Ba	8.7 E-01	9.2 E-01	1.1 E+00
Bi	3.9 E+00	1.3 E+00	3.3 E-01
Cd	5.3 E-01	1.6 E+00	3.0 E+00
C ₆ H ₅ O ₇	4.4 E+02	TBD	NC
CN	3.8 E-03	TBD	NC
CO ₃	3.3 E+02	TBD	NC
Cr ³⁶	7.9 E-02	5.7 E+00	7.2 E+01
EDTA	8.8 E-04	5.0 E+01	5.7 E+04*
F	1.1 E+00	2.0 E+01	1.8 E+01
Fe	4.4 E+01	2.0 E+00	4.5 E-02
HEDTA	4.4 E+01	5.0 E+01	1.1 E+00
Mn	1.9 E+00	3.7 E-01	1.9 E-01
Na	4.8 E+02	3.3 E+01	6.9 E-02
Ni	3.9 E+00	5.0 E+00	1.3 E+00
NO ₂	4.3 E-02	4.0 E+01	9.3 E+02
NO ₃	9.4 E-01	4.0 E+01	4.3 E+01
OH	4.4 E+01	NA	NC
Pb	2.7 E+00	5.0 E-01	1.9 E-01
PO ₄	4.3 E+01	4.0 E+01	9.3 E-01
SiO ₃	1.3 E+01	1.5 E+01	1.2 E+00
SO ₄	4.3 E+02	4.0 E+01	9.3 E-02
Zr	4.1 E+02	2.5 E+00	6.1 E-03

NA = data or method is Not Available

TBD = detection limit To Be Determined

NC = value could not be computed

*Suspect Analytical Detection Limit.

Table II-6. Detection Limit Goals and Analytical Detection Limits for Type II Carcinogens.

Carcinogen Analyte	Detection Limit Goal ($\mu\text{Ci/g}$)	Analytical Detection Limit ($\mu\text{Ci/g}$)	Ratio of ADL to DLG (None)
²⁴³ Am	3.0 E-05 ^a	7.0 E-03	2.3 E+02 ^b
²⁴² Cm	2.2 E-04 ^a	5.0 E-05	2.3 E-01
²⁴⁴ Cm	3.0 E-05	4.0 E-04	1.3 E+01
^{93m} Nb	NC	TBD	NC
²³⁷ Np	3.0 E-05	1.6 E+00	5.3 E+04 ^b
²³⁴ U	3.5 E-04 ^a	1.0 E-11	2.9 E-08

^aDetermined based upon the ADL of a different isotope.

^bSuspect Analytical Detection Limit.

NA = data or method is Not Available

TBD = detection limit To Be Determined

NC = value could not be computed.

Table II-7. Detection Limit Goals and Analytical Detection Limits for Type II Noncarcinogens.

Noncarcinogen Analyte	Detection Limit Goal ($\mu\text{g/g}$)	Analytical Detection Limit ($\mu\text{g/g}$)	Ratio of ADL to DLG (None)
Ag	1.9 E+01	3.9 E+00	2.1 E-01
Ca	1.8 E+02	3.6 E-01	2.0 E-03
C ₂ H ₃ O ₃	4.4 E+02	TBD	NC
Fe(CN) ₆	8.9 E+01	TBD	NC
K	4.4 E+02	1.3 E+02	3.0 E-01

NA = data or method is Not Available

TBD = detection limit To Be Determined

NC = value could not be computed.

Table II-8. Detection Limit Goals and Analytical Detection Limits for Type III Carcinogens.

Carcinogen Analyte	Detection Limit Goal ($\mu\text{Ci/g}$)	Analytical Detection Limit ($\mu\text{Ci/g}$)	Ratio of ADL to DLG (None)
²²⁵ Ac	4.1 E-03	TBD	NC
²²⁷ Ac	NC	TBD	NC
²⁴² Am*	4.1 E-05	7.0 E-07	1.7 E-02
²⁴⁵ Cm*	3.0 E-05	2.0 E-01	6.7 E+03
¹³⁵ Cs	NC	TBD	NC
²³¹ Pa	NC	TBD	NC
²³³ Pa	2.5 E-02	TBD	NC
²¹⁰ Pb	NC	TBD	NC
²¹⁰ Po	1.7 E-03	TBD	NC
²²³ Ra	5.0 E-03	TBD	NC
²²⁵ Ra	5.8 E-03	TBD	NC
²²⁶ Ra	NC	TBD	NC
²²⁸ Ra	NC	TBD	NC
¹⁰⁶ Ru	1.6 E-02	TBD	NC
⁷⁹ Se	NC	TBD	NC
¹⁵¹ Sm	NC	TBD	NC
²²⁹ Th	NC	TBD	NC
²³⁰ Th	1.4 E-04	TBD	NC
²³⁴ Th	2.3 E-01	TBD	NC
²³³ U*	3.5 E-04	7.0 E-12	2.0 E-08

*Determined based on the ADL of a different isotope.
 NA = data or method is Not Available
 TBD = detection limit To Be Determined
 NC = value could not be computed.

Table II-9. Detection Limit Goals and Analytical Detection Limits for Type III Noncarcinogens.

Noncarcinogen Analyte	Detection Limit Goal ($\mu\text{g/g}$)	Analytical Detection Limit ($\mu\text{g/g}$)	Ratio of ADL to DLG (None)
Ce	3.8 E+02	5.4 E+01	1.4 E-01
Cl	1.1 E+00	2.0 E+01	1.8 E+01
C ₂ O ₄	3.2 E+01	TBD	NC
La	4.4 E+02	4.8 E+00	1.1 E-02
SeO ₄	2.9 E-01	5.0 E-01	1.7 E+00
Sn	4.5 E+01	TBD	NC
WO ₄	2.1 E+02	TBD	NC

NA = data or method is Not Available
 TBD = detection limit To Be Determined
 NC = value could not be computed.

Table II-10. Detection Limit Goals and Analytical Detection Limits for Unranked Carcinogens.

Carcinogen Analyte	Detection Limit Goal ($\mu\text{Ci/g}$)	Analytical Detection Limit ($\mu\text{Ci/g}$)	Ratio of ADL to DLG (None)
As	1.7 E-03**	7.0 E-03**	4.1 E+00
⁶⁰ Co	2.6 E-03	TBD	NC
³ H	3.3 E-04	8.0 E-05	2.4 E-01
⁹⁴ Nb	3.3 E-05	TBD	NC
⁵⁹ Ni	3.6 E-02	TBD	NC
²⁴² Pu*	1.1 E-04	3.0 E-01	2.7 E+03+
¹²⁶ Sn	9.6 E-02	TBD	NC
²³² Th	2.8 E-05	TBD	NC
²³⁶ U*	2.6 E-08	1.0 E-09	3.8 E-02
⁹³ Zr	1.4 E-01	TBD	NC

*Determined based on the ADL of a different isotope.

**As values are in $\mu\text{g/g}$.

NA = data or method is Not Available
 TBD = detection limit To Be Determined
 NC = value could not be computed.
 +Suspect Analytical Detection Limit.

Table II-11. Detection Limit Goals and Analytical Detection Limits for Unranked Noncarcinogens.

Noncarcinogen Analyte	Detection Limit Goal ($\mu\text{g/g}$)	Analytical Detection Limit ($\mu\text{g/g}$)	Ratio of ADL to DLG (None)
Be	3.3 E-03	4.0 E-02	1.2 E+01
Co	2.2 E+00	1.0 E+02	4.5 E+01
Cu	TBD	4.1 E+00	NC
Hg	7.8 E-03	2.0 E-01	2.9 E+01
NH ₃	4.4 E+00	3.0 E+00	6.8 E-01
Sb	9.0 E-03	2.7 E+01	3.0 E+03+
Sr	4.4 E+02	3.4 E-01	7.7 E-04
S ⁻²	TBD	TBD	NC
Th	TBD	4.1 E+01	NC
Tl	4.4 E-01	7.8 E+02	NC
Ti	TBD	3.0 E+00	NC
U	6.3 E+00	3.3 E+02	5.2 E+01+
V	4.9 E-01	2.7 E+00	5.5 E+00
Zn	4.4 E+01	1.2 E+00	2.7 E-02

NA = data or method is Not Available
 TBD = detection limit To Be Determined
 NC = value could not be computed.
 +Suspect Analytical Detection Limit.

11.2.2 Decision Quality

The fundamental requirement for the SST characterization data is that tank closure decisions are adequately supported. Under the Hanford Defense Waste Environmental Impact Statement (DOE 1987) decisions on the remediation (closure) strategy for SST's will be made on a tank-by-tank basis. The options available include retrieval and treatment of SST waste in the same facilities that will be used to separate and isolate DST waste, and a range of in place disposal options. The reliability with which these decisions are made is a direct consequence with the data available on tank inventories. Thus a statistical simulation of decision making (Decision Simulation) is being employed to determine the effects of the various features of the characterization activity on decision quality. These features include the number of cores per tank, the degree to which cores are analyzed as composites or as segments, and the degree of analytical error which will determine the number and distribution of sample duplicates. Based on these studies Westinghouse Hanford will develop a core sampling and chemical analysis plan commensurate with preliminary results from the Decision Simulation and other characterization objectives and requirements. The Decision Simulation and its implementation uses information proceeding from various other statistical activities.

11.2.2.1 Statistical Activities to Date. The following are brief descriptions of the statistical analyses of data from the sampling of SST B-110 in Phases IA and IB. Results used to formulate recommendations for Phase IC sampling that relate to the sampling plan for each tank (number of cores to be taken, etc.) and the analytical protocol (specification of segment or composite analysis and the number of homogenized replicate aliquots, etc.) are emphasized.

- **Analysis of Sources of Variability and Comparison of Core Composite and Segment Analysis Results**

An analysis of variance (ANOVA) was performed on concentrations of metals, anions, and radionuclides to obtain estimates of components of variability. These components are analytical variance, homogenization variance, and spatial (horizontal and vertical) variance. Analytical variability attributable to the error associated with any particular analysis can be measured by performing duplicate analyses on the same sample and comparing the results. Homogenization uncertainty is the error due to the inability to sufficiently mix a heterogeneous sample to obtain an aliquot that is representative of the entire mixture. It can be determined by obtaining two separate aliquots from different locations within the homogenized sample and comparing the analytical results from each aliquot. This procedure assumes that the analytical uncertainty is well understood and smaller in magnitude than the homogenization error. The homogenization error can be measured for both homogenized segments and homogenized core composites. The horizontal and vertical variabilities are respectively associated with the distribution of constituents across the tank in the horizontal and vertical planes. For most of the constituents examined, analytical variance was largest in magnitude, followed by segment and composite homogenization variance, and

horizontal variance. However, for many Type I and Type II constituents (see Table 11-2), the ordering was generally reversed, for analysis results from fusion and some acid leach preparations. For cases in which the spatial variance components are largest, more core samples may be needed. Alternatively, if the dominant components of variability are homogenization and analytical uncertainty, relatively fewer cores are needed, but more replicated analyses of homogenized aliquots and duplicates from aliquots are required.

- Statistical Adequacy of Core Composite Procedure

Even though much of the SST waste is thought to exhibit distinct vertical layering, the determination of the average tank inventories can be based on analysis of core composites under certain conditions. This would greatly simplify and reduce the cost of the SST characterization effort. Comparisons between core composite and segment level analysis were made to help resolve the need for further segment level analysis during processing of 241-B-110 samples. The comparisons involved testing the difference between the average core composite concentration and the average of the average segment concentrations for significance (from zero). For most constituents, this comparison resulted in significant differences between individual core estimates for at most one core out of seven analyzed. (These calculations are not yet publicly available.) Thus the preliminary conclusion is that core composite level analyses may be sufficient to determine average SST constituent inventories.

- Analysis of Holding Time Data

The primary purpose of the holding time study was to evaluate if any of the regulatory constituents for which holding times are important were significantly affected by the slower processing requirements of highly radioactive samples. The analytical objective was to identify any decrease in constituent concentrations over time that would result in false negative or low results. No holding time effects were observed for the analytes examined in this study. (These calculations are not yet publicly available.)

In addition, the presence of a long-term analytical or batch effect was investigated. In more than half of the analytes examined (6 of 11), long-term analytical or "batch" effects were observed. This suggests that standard duplicate analyses underestimate the total analytical variability. There were insufficient data to distinguish between either of these effects (if present) in Cr⁶ measurements.

- Numbers of Cores and Sampling Geometry

The variability in constituent concentrations within tanks is a central issue in planning the waste characterization program. Extreme spatial variability in constituent concentrations essentially reduces the information content of data from samples and requires a larger number of samples to provide a given level of

confidence in decision-making. The decision simulation model is used to simulate the sampling process for alternative numbers of cores-per-tank and under alternative degrees of decision stringency. The results are tabulated (relative frequency) probabilities of correct, incorrect, and inconclusive decisions (for individual tanks and in aggregate). The constituents considered are the majority of those Type I and II analytes in Table I1-2 and the surrogate decision criteria are limits for sums-of-fractions (summed ratios of individual concentrations to their respective limits) for long-lived and short-lived radionuclides (based on 10 CFR 61) and toxic chemicals (WAC). The final decision criteria will be established at a later date (SEIS). The spatial and analytical variabilities relative to tank average concentrations estimated from the B-110 tank were assumed to apply to all tanks. The TRAC concentrations were assumed to be the true tank means.

A generality that was drawn from the decision simulation results is that 3 cores is sufficient for classifying an SST as either "leave" or "retrieve," provided that the spatial variation in each tank is like that of SST B-110 and that the TRAC estimated concentrations for Type I and II analytes are accurate. It was found that tanks which had high or low concentrations relative to the decision thresholds were in most cases correctly classified with 2 cores per tank. A few tanks in which concentrations were close to the decision thresholds required as many as 5 to 6 cores for reliable decisions. While this analysis is preliminary in the sense that it depends on the assumption that the spatial variability in B-110 can also be found in all of the SSTs, it does illustrate the importance of this feature in structuring a reliable characterization scheme. For this reason it may be desirable to obtain a greater number of cores from the early SSTs sampled.

The issue of adequately determining spatial variability also effects sampling geometry. Although geometry for core samples is often constrained by riser locations and availability, the spatial pattern of samples should be considered when selecting specific risers for samples. The assessment of spatial covariability involves taking core samples in a configuration which results in an even distribution of pairwise sampling distances over short, medium, and long distances. Lastly, if concentration estimates at arbitrary locations in a tank are needed, then core samples should be configured so as to provide reasonable lateral "coverage" of the tank. Thus, in addition to configuring the design to support estimation of the covariogram, the sampling layout must also exhibit sufficient coverage to achieve other stated objectives.

I1.2.2.2 Sampling Strategies. Recommended strategies for the sampling of the next 10 SSTs were based upon the results and conclusions from the foregoing statistical efforts. These recommendations are as follows:

- Core Sample Analysis

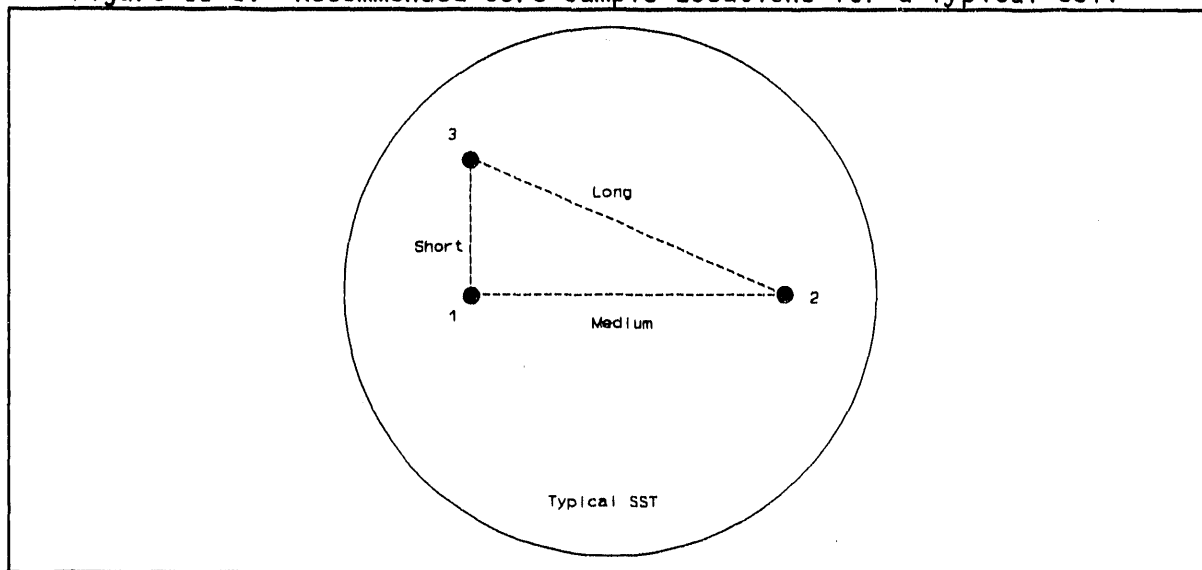
For each core composite the minimal set of constituents to be analyzed are the Type I and II analytes listed in Table I1-2. These

analytes are considered to be significant contributors to the overall risk associated with the SST waste. Most of them are also significant with respect to waste classifications (see 1.2.1). Concentrations should be determined in duplicate for both replicate composites and replicate aliquots from core composites to ensure adequate information from which to estimate various components of variability.

- Spatial Variability

At the present time, the only source of information about the spatial variability of various SST waste constituents is data from Tank B-110. It is not known whether constituents in other tanks exhibit similar patterns of spatial variability. In general it is desirable to resolve this spatial variability issue early, and therefore to take more than 2 cores per tank during the early stages of the characterization effort. In order to estimate spatial correlation, the 3 pairwise distances between risers should be as evenly spaced between short, medium, and long distances as possible. A diagram depicting recommended sampling locations for a typical single-shell tank has been presented as Figure I1-1. The recommended sampling configuration will provide improved estimates of the covariogram (spatial correlation). Additional cores will provide additional spatial resolution, provided that they can be taken at locations which preserve the uniform spacing among intercore distances.

Figure I1-1. Recommended Core Sample Locations for a Typical SST.



- Validation of the SORWT Model

The SORWT grouping model, used as a tool for characterization planning has not been validated and is currently under technical review. The selection of the next ten SSTs should take into account the need to validate the model results.

- Holding Times

Examination of the anion data led to the conclusion that no kinetic holding time effect exists (for the constituents examined). For other analytes, such as Cr(VI), there were insufficient data to distinguish between a batch effect and a holding time effect. Since Cr(VI) is a Type I analyte, its analysis plan should facilitate this distinction. In particular, six replicate segment analyses for Cr(VI)--each with homogenization replicates and sample duplicates--should be done for one segment in one core. These analysis groups should be done at regularly spaced times with the last set being analyzed at the maximum time that the laboratory expects to hold sample material.

11.2.3 EVALUATION OF IMPACTS

The Impact Analysis Study is concerned with evaluating the impacts of the waste characterization plan on radiological exposure to workers, costs, and schedules. These impact analyses will assist in evaluating alternative sampling and analytical testing programs for SSTs during subsequent characterization phases. Recommendations are provided for choosing among sampling alternatives that provide decision-making capability using minimal resources and identification of process areas where improvements can yield reductions in resource needs and schedule compression. Dose impact analysis provides for postulating the occupational dose acquired by the radiological worker as a result of his involvement with SST waste characterization.

The scenarios being evaluated vary according to the number of cores sampled and analyzed per tank, the number of segments and core composite samples analyzed per core, and the number of duplicate and spiked samples analyzed per segment (or core composite). The current set of cases are:

- Case 2A: Two cores per tank. Duplicate and spiked samples are analyzed for one segment of five¹ and the core composite.
- Case 2C: Two cores per tank. Segments are analyzed for physical properties and volatile constituents. All other tests are run on core composite samples only, including the duplicate and spiked samples.
- Case 3A: Three cores per tank. Duplicate and spiked samples are analyzed for one segment of five and the core composite.
- Case 3B: Three cores per tank. Duplicate and spiked samples are analyzed for all five segments and the core composite.
- Case 3C: Three cores per tank. Segments are analyzed for physical properties and volatile constituents. All other tests are run on core composite samples only, including the duplicate and spiked samples.

¹Five segments are assumed to be in each core sample for this study.

Case 4A: Four cores per tank. Duplicate and spiked samples are analyzed for one segment of five and the core composite.

Waste characterization has been divided into four process categories of work that must be performed on a core sample from a tank for the purposes of the impact analyses. The process categories are:

- (1) Tank sampling
- (2) Segment receipt and handling (at the laboratory)
- (3) Sample transfer (from hotcell to hood, where appropriate)
- (4) Sample analysis

Westinghouse Hanford performs the tasks associated with process categories 1 and 3 (if required). Both Westinghouse Hanford and PNL personnel are assumed to participate in process categories 2 and 4. The laboratory work is alternated between labs on a tank-by-tank basis.

II.2.3.1. Radiological Dose Impacts. The radiological characteristics of the SST waste are determined by the radionuclides present. The primary radioactive species of concern with regard to external exposure are those emitting beta particles, gamma rays, or both. Only those beta particles with sufficient energy to penetrate the walls of the sample container and reach a worker present an exposure potential. Preliminary analytical data indicate that only Cesium-137 and Yttrium-90 are of concern in the context of extremity exposure.

Empirical data obtained during Phase IA and IB in combination with process background data and the TRAC database were used to calculate extremity dose received during sampling and analysis of tanks during Phase IA and IB. Empirical data consisted of personnel dosimetry, radiochemical analysis results, and radiological surveys obtained during Phase IA and IB. Process background data consisted of information obtained from procedures and analysis scenario. Process background data determined constraints such as the sample weight required for a sample analysis, the number of segments retrieved from each tank, and the number of duplicate and/or spiked samples for each analysis. Process background data is used directly in the derivation process or indirectly as the basis for simplifying assumptions.

Tables II-12 through II-16 present a summary of the actual personnel dose data from Phase IA and IB used in the impact analyses. The data shown from process categories 1, 2, and 3 are empirical data taken from dosimetry records during Phase IA and IB characterization work. The data shown from process category 4 (Tables II-15 and II-16) show the analyses that are assumed to be performed during the remainder of SST waste characterization. Empirical data was used for the first eight analyses (Table II-15). Empirical data was not available for the remaining nine analyses (Table II-16), however, the average dose per analysis inferred from similar analyses where data was available. Also note that during the period of time for which the SST characterization dose was recorded the workers did not work exclusively on SST samples. The dose received from working on any other samples was embedded in the personnel dose reports, therefore the reported extremity exposure are conservatively high.

Table I1-12. Process Category 1 - Sampling Operations.

PERSONNEL	AMOUNT (segment)	TOTAL DOSE (mrem)	DOSE PER SEGMENT
A	5	9.4	1.9 mrem/segment
B	5	10	2 mrem/segment
C	5	10	2 mrem/segment
D	5	20	4 mrem/segment
E	5	10	2 mrem/segment
F	5	10	2 mrem/segment

Process Category 1 Total = 13.9 mrem/segment

Table I1-13. A&B Process Category 2
Sample Receipt and Handling.

A. Sample Receipt

PERSONNEL	AMOUNT (segment)	TOTAL DOSE (mrem)	DOSE PER SEGMENT
1	5	9.4	1.9 mrem/segment
2	5	23	4.6 mrem/segment
3	5	13	2.6 mrem/segment
4	5	3	0.6 mrem/segment

Process Category 2A Total = 9.7 mrem/segment

B. Sample Handling

PERSONNEL	AMOUNT (segment)	TOTAL DOSE (mrem)	DOSE PER SEGMENT
1	5	13	2.6 mrem/segment
2	5	3	0.6 mrem/segment
3	5	13	2.6 mrem/segment
4	5	3	0.6 mrem/segment

Process Category 2B Total = 6.4 mrem/segment

Process Category 2 Total = 16.1 mrem/segment

Table II-14. Process Category 3 Sample Transfer
(from hotcell to hood).

PERSONNEL	AMOUNT (gram)	TOTAL DOSE (mrem)	DOSE PER SEGMENT
A	136.1	20	0.15 mrem/segment

Table II-15. Process Category 4 Sample Analysis (Empirical).

ANALYSIS	MONTH	CHEMIST	SAMPLE WEIGHT (gram)	NUMBER OF SAMPLES	TOTAL DOSE (mrem)	AVERAGE DOSE (mrem)
Acid Digestion	1	A	1	6	10	0.9 mrem
	2	A	1	40	30	
Water Leach	1	B	1	9	40	1.9 mrem
	2	B	1	19	45.2	
	3	B	1	22	50	
		C	1	28	13.3	
pH	1	C	2.5	53	110	1.4 mrem
	2	C	2.5	45	50	
	3	C	2.5	72	85.7	
Fusion	1	D	0.25	34	8	0.3 mrem
	2	D	0.25	16	5	
	3	D	0.25	19	6.2	
Percent Water	1	D	2	54	102	2.2 mrem
	2	B	2	1	4.8	
		D	2	22	55	
	3	C	2	1	1	
		D	2	32	83.8	
Total Gamma Analysis	3	A	0.25	8	25	3.1 mrem
DSC	3	A	0.25	8	25	3.1 mrem
Volatile Organic Analysis	N/A	F	1	58	9.4	0.2 mrem

Table II-16. Process Category 4 Sample Analysis (Inferred).

ANALYSIS	ANALYSIS BASIS	SAMPLE WEIGHT (gram)	AVERAGE DOSE (mrem)
Semi-Volatile Organic Analysis	Volatile Organic Analysis	1	0.2 mrem
Extractable Organic Halides	Volatile Organic Analysis	1	0.2 mrem
Carbon-14	pH	1	0.6 mrem
Sulfide	pH	1	0.6 mrem
Mercury	pH	1	0.6 mrem
Cyanide	pH	1	0.6 mrem
Arsenic	pH	1	0.6 mrem
Selenium	pH	1	0.6 mrem
Particle Size	Fusion	0.25	0.3 mrem

The total dose (in rem) acquired by all workers exposed during the entire course of SST waste characterization sampling and analysis is shown in Figure I1-2 for the scenarios of interest. The dose is separated into amounts received for each process category. Comparison of the same number of cores, but alternative numbers of replicate samples (by observing Cases 3A, 3B, and 3C in Figure I1-2) show more extremity dose is received by laboratory personnel from process category 3 (sample transfer) and process category 4 (analysis and testing) following the "B" alternative. The "B" alternative performs analyses on duplicate and spiked samples for all segments of a core sample. While this alternative may provide additional data quality, it is at the expense of increased radiological dose to the laboratory workers.

The dose impact of SST waste characterization work on an individual worker performing tasks in each process category is shown in Table I1-17. The annual effects for process categories 1 and 2 are shown for an assumed maximum of one crew supporting one sampling rig handling 24 cores per year. Process categories 3 and 4 are shown for an assumed maximum of one laboratory worker supporting two crews and sampling rigs handling a total of 48 cores per year. The more restrictive "B" alternative is used for this analysis.

Table I1-17. Annual Dose per Characterization Worker.

Process Category	Number of Cores Handled Annually	Annual Dose per Person
1	24	2.0 rem
2	24	3.6 rem
3	48	2.9 rem
4	48	3.1 rem

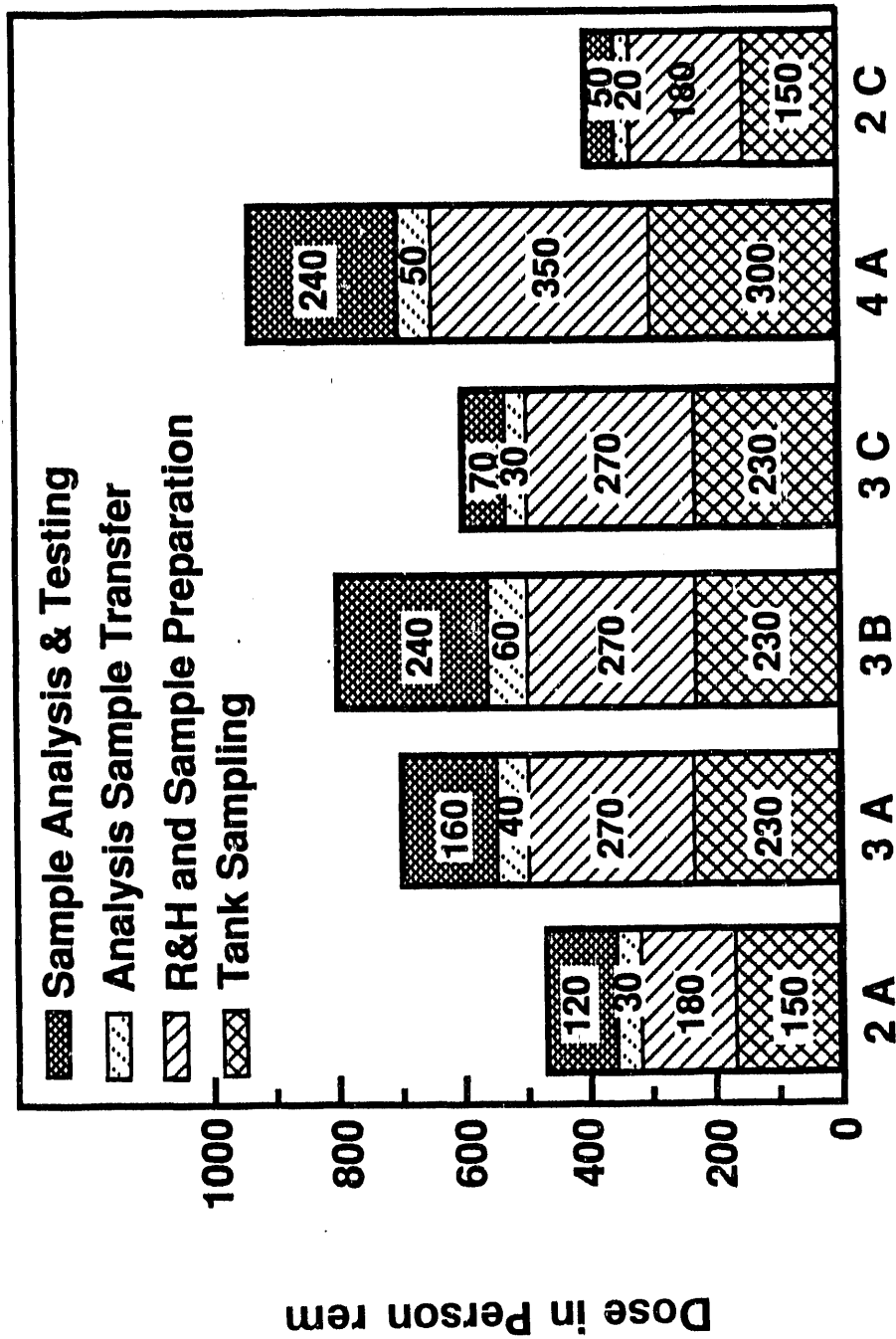
The dose impact was calculated based upon three alternative laboratory analysis scenario configurations. These three configurations were: (1) all testing and analyses were performed in fume hoods; (2) all testing and analyses were performed in hotcells; and (3) testing and analyses were performed alternatively in fume hoods and hotcells (Basecase). These comparisons are illustrated in Figure I1-3.

I1.2.3.2. Schedule and Cost Impacts. The process logic and associated schedule and cost impacts were extrapolated from information obtained through interviews with laboratory management personnel. The schedule estimates are based on multiples of cores for the different scenarios of interest. The duration for processing a core sample is calculated using a standard scheduling tool. Cost figures are estimated based on the personnel and time that is required to process the core sample. Overhead charges are applied to account for management, use of equipment and supplies, and waste handling.

Several simplifying assumptions were made in order to arrive at the preliminary estimates. The key assumptions were that no rework occurs, tank sampling equipment was always available, and laboratory resources were

Figure 11-2. Dose Impacts (Total) of Different Scenarios.

Dose Impacts (Total) of Different Scenarios

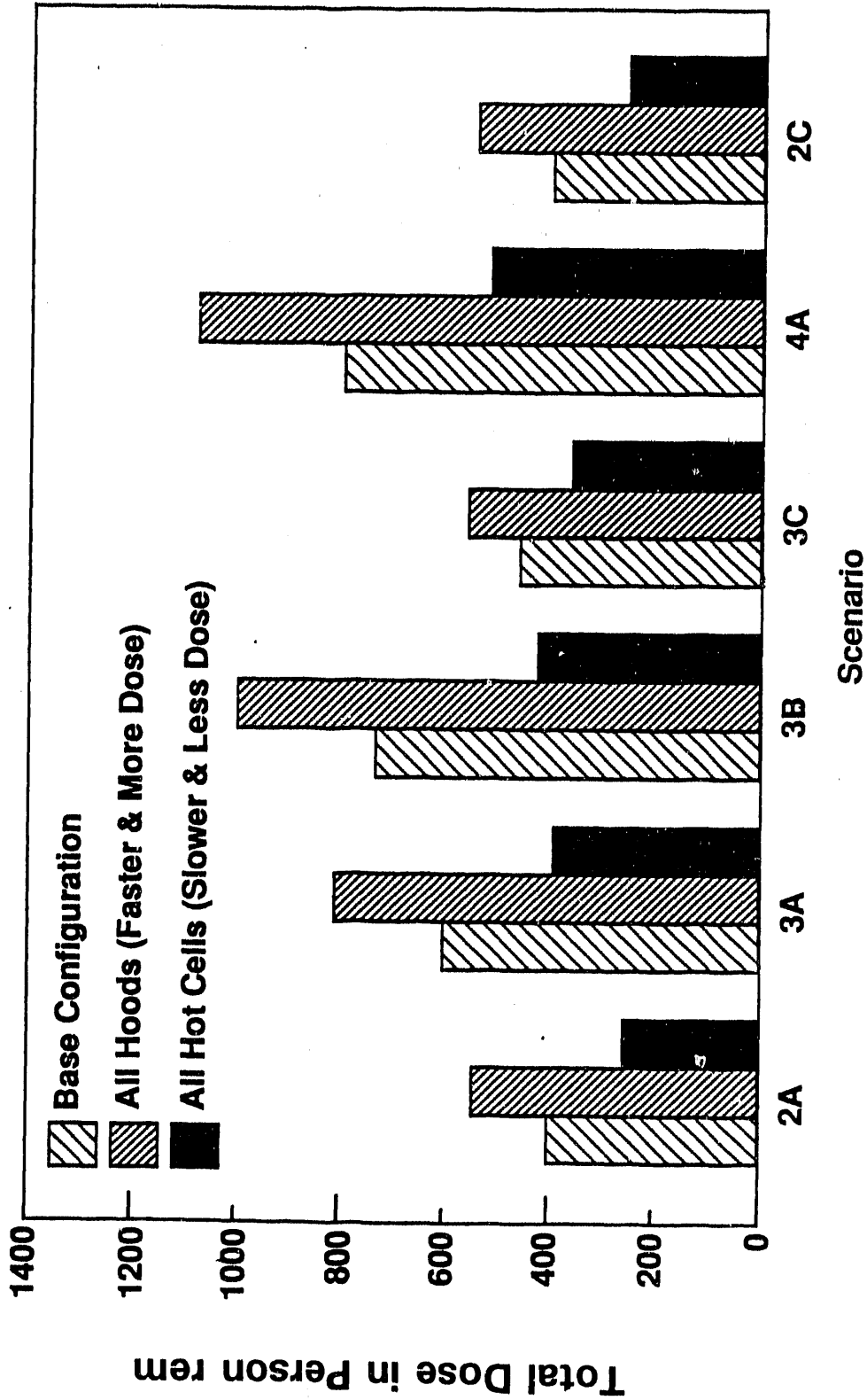


Scenario

S9105031.6

Figure 11-3. Dose Impacts Alternative Configurations for Each Scenario.

Dose Impacts of Alternate Configurations For Each Scenario



S9105031.5

available when needed. Since sample control and sample analysis procedures are being evaluated for performance during Phase IA and IB, and continuous process improvement and learning is expected during the lifetime of the SST waste characterization program, the assumptions implicit in these initial estimates will be adequate for a first iteration.

Additional data and closer correlation with work processes are needed to confirm the dose estimating model and to provide a more detailed estimate of resource requirements. Dose impact analysis during Phase IC will focus on gathering empirical data for (1) more precise correlation between occupational dose and SST samples actually handled for (2) substantiation of the preliminary dose estimate calculations.

The focus of the schedule and cost impact analyses during Phase IC, and subsequent the phases of SST waste characterization, will be on gathering and analyzing empirical data for calculation of schedule and cost impacts, including (1) data package preparation, data analysis, (2) identification of resource constraints, and (3) how to prevail over the limitations that the resources suggest.

12.0 SUMMARY OF LESSONS LEARNED FROM PHASE IA/IB

One of the primary objectives of Phase IA/IB was to evaluate technical and administrative procedures used to sample, analyze, and report data. Lessons learned from this evaluation are described for the following operations: sampling, hot cells, analytical, and administrative/organizational.

12.1 SAMPLING OPERATIONS

Chain-of-custody procedures were implemented during Phase IA/IB. Some improvements have been made in the chain-of-custody form based on experience from Phase IA/IB. A new, disposable sampler will be implemented starting with SSTs B-201 and B-202 which will reduce the potential for cross contamination between samples, and reduce the time and cost involved in transporting and cleaning the sampler.

The sampling equipment did not perform well for the drier wastes found in Tank U-110. (The average recovery for sampling this waste was about 50%.) The selection of tanks with softer waste will be important until a system capable of sampling drier and harder waste is available. Incomplete sample recovery impacts the interpretation of the data and the representativeness of the core composite.

Additional needed improvements in the sampling operations were noted as a result of phase IA/IB. These included increasing shipping cask inventories to enable continued core sampling while allowing for decontamination of the shipping casks and liners. Shipping procedures have also been modified to incorporate road closure when core samples are shipped to the 300 Area. Core Sample Truck operating delays for riser set-up and break-down can be reduced by the addition of a three man support crew.

Normal paraffin hydrocarbons (NPH), used in the sampling process to provide a hydrostatic head, seriously affect the analytical procedures for determining organics of regulatory interest and total organic carbon (TOC) analyses. The NPH contamination of samples requires large sample dilutions before analyses, making it impossible to meet reasonable detection limits. In addition, the NPH affects the long-term performance of the gas chromatography (GC) columns and mass spectrometer, and can cause more frequent down times and instrument repairs.

In order to alleviate the adverse effects of NPH sample contamination two projects are currently underway. For near-term core sampling events, an NPH clean-up technique is being developed to remove the NPH contaminatin and allow volatile analysis using a GC/MS system. The long-term solution to this probelm will be replacement of NPH as a hydrostatic head with a pressurized inert gas.

12.2 HOT CELL OPERATIONS

Both laboratories extruded, homogenized, and composited waste samples in the hot cell. In addition, PNL performed dissolution and some separation operations in the hot cell.

The Omni Mixer used by PNL worked well on the soft/wet tank B-110 waste. After mixing the tank B-110 segments, a small volume of separable aqueous phase sometimes resulted. The Stomacher Mixer used by the 222-S Laboratory did not work well on the drier tank U-110 wastes. This waste sometimes contained lumps of harder material that would puncture the plastic bags used with the mixer. The Stomacher may still be adequate for softer waste; however, improved mixing systems will be needed for drier/harder forms of wastes.

Complex operations such as distillation of cyanide performed very inefficiently in the hot cell because of the limitations in setting up multiple systems and because of the large number of analyses required to meet quality control requirements. More efficient hot cell distillation systems will be needed to meet the stratified testing requirements for ferrocyanide wastes.

12.3 ANALYTICAL OPERATIONS

12.3.1 Metal Ions

Regulatory-based acid digestion procedures were implemented for the analysis of metals by inductively coupled plasma (ICP) and Graphite Furnace Atomic Absorption (GFAA). The 222-S Laboratory did not utilize interelement corrections for ICP analysis of tank U-110 wastes. This resulted in false positive results for some environmentally sensitive metals. Interelement corrections will be required for the complex matrices found in SST wastes. Improved data handling and reporting systems for the ICP are needed for both labs because of the large volume of data generated in the analysis of the samples for 20 to 30 elements and associated quality control requirements.

Pacific Northwest Laboratory used GFAA to measure Arsenic, Selenium, and Lead. Initial results for these analyses had relatively high less than values (20 $\mu\text{g/g}$) probably due to high dilution factors and small sample sizes; however, these later improved to 2 $\mu\text{g/g}$. GFAA equipment at PNL needs to be upgraded to improve the performance. Arsenic and selenium were determined by hydride atomic absorption (HYAA) methods at the 222-S Laboratory. The GFAA capabilities need to be added at this laboratory to confirm ICP Pb analyses and to improve detection limits for metals such as Sb and Ti if required.

12.3.2 Anions and Wet Chemical Analyses

A water digestion method was implemented for leaching the anions and water-soluble organics from the SST waste. Even though no obvious problems were noted with this procedure, further evaluation of its performance should

be documented. The effect of time, temperature, and mixing method on leaching completeness should be studied and documented to optimize the method.

The 222-S ion chromatography (IC) results contained numerous high less than values, particularly for sulfate. The cause of these high less than results needs to be evaluated to determine if the high values are the result of large dilutions or from matrix effects such as high aluminum or high phosphate. Data specifications and Detection Limit Goals detailed in Section 11.2.1 are needed to provide the laboratory with guidance about required detection levels.

The effect of water-soluble organics on the IC chromatograms also should be evaluated to ensure false positive results are not reported. The potential of analyzing these organic compounds on the IC also should be evaluated since they will be important in the characterization of complexant waste tanks.

Faster cyanide methodology for hot cell applications will be needed if cyanide analysis on segments or layers is required. Analysis of cyanide at PNL was one of the most manpower-intensive methods. Alternate methods are being evaluated. These methods also must be applicable to the highly insoluble cesium nickel ferrocyanide compounds and be effective for the high cyanide concentrations expected in the ferrocyanide tanks.

A method with better detection limits needs to be implemented for ammonia analysis at the 222-S Laboratory. High less than values were reported during Phase IA/IB.

12.3.3 Radiochemical Analysis

A fusion/acid digestion method was used for the preparation of samples for all radiochemical analyses except ^{14}C and tritium, which were analyzed on the water digestion. Additional data are needed to support the fusion/digestion procedure to determine the effect of the high temperature and acid treatment on the recovery of potentially volatile radionuclides such as ^{129}I and ^{99}Tc from the SST waste matrices.

The total alpha procedures at both laboratories did not perform well with the high salt and chloride matrix of fused samples. Method improvements need to be evaluated that can determine total alpha in the waste at 10 to 25 nCi/g levels. Determination of total alpha in the acid digestions would eliminate the high salts from the KOH fusion but would still have a chloride problem. Comparisons of total alpha from fusion and acid digestions would be needed to verify that the acid result recoveries are comparable to the fusion.

The PNL method for radiochemical spike evaluation did not reflect the effect of the sample matrix. Pacific Northwest Laboratory spiking procedures need to be changed so that the effect of the sample matrix on the recovery of the spike can be evaluated. The 222-S Laboratory needs to report results for ^{238}Pu and ^{244}Cm . These results are available from alpha energy analysis but may be very low (^{244}Cm) or may have interferences from spike materials that require additional corrections (^{238}Pu). The 222-S Laboratory also needs to lower its detection limit for ^{237}Np analyses.

Improved data-handling and reporting methods are needed for radiochemical data.

12.3.4 Organic Analyses

The detection limits for TOC need to be improved at the 222-S Laboratory. This will probably require new equipment with larger sample-handling capabilities. This new equipment should include the ability to determine TOC directly on the solids.

Several problems were identified in the determination of organics that are of regulatory interest. The major problem is the interference caused by contamination of the sample with normal paraffin hydrocarbon (NPH) used in the sampling process. This material required the samples to be diluted to the point that the trace organics were not detectable. In addition, NPH seriously impacted the performance and reliability of the GC/MS instrumentation. New sampling procedures or methods to selectively remove the NPH from the sample are needed before organic analyses are continued.

Organic analyses in Phase IA/IB also indicated that there was an unknown polar substance affecting the volatile organic method. Analyses also indicated that the high nitrate in the sample may be reacting with the surrogate organics used to evaluate the method. These areas need further evaluation.

The method used by 222-S Laboratory to determine complexants ethylenediametetraacetic acid (EDTA) and hydroxyethylenediametetraacetic acid (HEDTA) is no longer functional because of changes in High-Performance Liquid Chromatography (HPLC) column material. Improved methods for identification of complexants (i.e., water soluble organics) will be valuable in safety assessments for the waste in the tanks and for establishing distribution coefficients in performance assessment evaluations.

12.3.5 Characteristic Testing

Experience gained in EP-toxicity testing in Phase IA/IB will be applied to implementing the TCLP tests for wastes from new tanks. The PNL pH procedure needs to be modified so that the results are in compliance with corrosivity testing requirements.

12.4 ADMINISTRATIVE/ORGANIZATIONAL OPERATIONS

Batching of samples is important in improving laboratory efficiency and for ensuring that proper quality control of measurements is maintained. Experience gained in Phase IA/IB will improve batching procedures for the different operations.

Data compilation and report generation was more manpower-intensive for Phase IA/IB than expected. Until more efficient data management systems are available, a significant staff will be required to compile the data and prepare the reports.

Quantitative DQOs were not clearly established for Phase IA/IB. This resulted in the laboratory reporting some data that may not be useful and can not be evaluated. The laboratory needs more specific guidance on required measurement limits and variability goals.

Data evaluation procedures need to be better defined, and implemented on a more real-time basis to permit quick response to problems and to prevent reporting erroneous results. Either improved data management systems or increased staff will be needed to perform more extensive data reviews.

Solid standards are needed to evaluate the entire analytical measurement system. A system of analyzing these standards and tracking the laboratory performance is needed to evaluate the laboratory procedures and personnel, and to provide an indication of long-term analytical variances. An interlaboratory sample exchange program needs to be implemented to substantiate the results and identify potential problems in methodology.

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13.0 SELECTION OF THE NEXT TEN SINGLE-SHELL TANKS FOR CHARACTERIZATION

The selection of the next 10 SSTs to be core sampled as part of Phase IC of the Waste Characterization Plan was based on a number of criteria and assumptions. The ultimate goals of the selection process were to obtain the greatest amount of information on as many different waste type groups as possible and to analyze the variance of the chemical and physical characteristics of SST groups predicted by the SORWT model.

There are many uncertainties pertaining to programmatic priorities, safety assessments, and sampling capabilities. During the course of sampling the next ten SSTs, the need to alter the list of selected tanks may become apparent due to shifting priorities or the inability to safely sample a selected tank. If a new tank is selected in addition to or to replace the selected tanks then the justification and schedule for the change shall be appropriately documented.

13.1 SINGLE-SHELL TANK SELECTION CRITERIA

The SST selection criteria have been separated into primary and secondary criteria. Groups of tanks that satisfy the primary criteria are SSTs that are considered a high priority to sample. One SST from each high priority group is then selected based upon the secondary criteria. The primary selection criteria are listed in Section 13.1.1. The secondary selection criteria are listed in Section 13.1.2.

13.1.1 Primary Single-Shell Tank Selection Criteria

The primary selection criteria are as follows:

- Single-shell tanks should belong to a large SORWT Group. In order to obtain the most characterization information in the shortest possible time, larger SORWT groups have a higher selection priority than smaller SORWT groups.
- Single-shell tanks should contain relatively soft waste. The current sampling technology is only capable of obtaining sufficiently complete core samples from soft waste. It has been shown that incomplete core samples significantly impacts the confidence bounds of the tank inventory (Jensen 1988). A sampler capable of sampling harder material will not be available before the end of fiscal year (FY) 1992. Therefore, only SSTs containing softer material will be core sampled until the new sampler is available. An SST was considered to hold soft waste if it was on the "Push-Mode" list (Kelly 1991) or if recent surveillance photographs indicated a soft, moist surface.
- Single-shell tanks should satisfy multi-programmatic needs. There are a number of open safety concerns pertaining to SST waste such as FeCN₆-scavenged waste and high-heat SSTs. The selected

tanks should satisfy the sampling needs of the safety program to enable closure of these issues. In addition, other SST programs, such as retrieval, have legitimate sampling and analysis needs that must be taken into account.

- Selected SORWT groups must contain a large waste volume. Some of the SST groups predicted by the SORWT model do not contain significant quantities of waste even though they represent a large number of tanks. These low-waste-volume groups should not be sampled as a high priority. Some SORWT groups represent only a limited number of tanks but possess large volumes of waste. These high-waste-volume SORWT groups should be given a higher sampling priority.

13.1.2 Secondary SST Selection Criteria

The secondary selection criteria are as follows:

- Single-shell tanks with the highest volume within a group should be sampled. The SST containing the largest waste volume within each group that satisfies the primary SST selection criteria should be sampled. The largest waste volume SST should be the most representative, on a volumetric basis, of the entire SORWT group.
- Variance of SORWT groups.
In order to measure the variance of the physical and chemical properties of groups of SSTs predicted by the SORWT model, two tanks from each of five SORWT groups should be collected. Single-shell Tanks B-201 and B-202 are already scheduled to be sampled and constitute one of the five SORWT groups to be measured for variability. In order to most efficiently sample SSTs before closure of the SEIS database, the same SORWT group should not be sampled more than twice during the next 10 sampling events. This will provide a larger and more comprehensive database on which to write the SEIS.
- Single-shell tanks should possess at least two risers.
In order to collect two representative core samples, samples from two different risers from opposite ends of the tank would be preferred. The configuration of recommended sample locations can be found on Figure 11-1. The riser configurations can be checked from references. However, the ability to collect core samples from a particular riser can not be assured until they are opened and inspected.
- Tri-Party Agreement milestones.
Westinghouse Hanford and the U.S. Department of Energy (DOE) has committed to interim milestone M-10-06, which requires 20 core samples be obtained from SSTs prior to September 1992. This revision of the Waste Compliance Plan (WCP) supports this milestone's requirements.

13.1.3 Next Ten SSTs Selected

The SST selection criteria are reasonably efficient even if tanks do not fall into groups as predicted by the SORWT model. Single-shell tanks are selected and prioritized not only on the basis of group representation, but also for programmatic needs, technological feasibility, total waste represented, and variety of waste represented. Thus, the tank selection criteria should aid in bounding the design and safety criteria and enable informed decisions to be made pertaining to the final disposition of the SST operable units, regardless of the existence of SORWT groups. The distinct possibility that SSTs belong to groups of tanks with similar physical and chemical characteristics, however, should make these selection criteria a very effective method of choosing which tanks to sample.

The next 10 SSTs selected to be core sampled are presented in Table I3-1. In addition, a short description of the technical justification for each selection and its placement on the sampling order has been provided. In order to estimate the spatial distribution of waste constituents in a SORWT group, three cores per tank will be collected and analyzed for the first tank sampled from an individual SORWT group. A minimum of two cores per tank will be collected for the rest of the non-Public Law 101-510 list tanks sampled in a SORWT group.

No SSTs have been selected from the largest three SORWT groups (see Table I1-1) because these tanks contain saltcake and can not presently be sampled. The second Core Sample Truck should be available for rotary-mode sampling by the end of FY 1992.

The first column in Table I3-1 lists the chronological sampling order for the next ten SSTs. An asterisk (*) next to the sample order indicates that this tank is on the Push-Mode List as defined by Internal Memo WHC-86431-91-002 (Kelly 1991). These tanks contain waste that is soft and should not pose any difficulties for sampling. The remaining SSTs on Table I3-1 are considered candidates for push-mode sampling based upon the waste types contained in the tank and interpretation of recent surveillance photos. The next column identifies which of the tanks are identified by Public Law 101-510 and the safety issue associated with the particular tank. Columns 3, 4, and 5 identify the specific SST proposed to be sampled, the number of cores per tank, and the number of segments per core, respectively. The next column contains the waste type group number in which this tank was predicted by the SORWT model. The seventh column categorizes the primary and secondary solids-forming waste types expected to be present in the tank. This information was used by the SORWT model to organize the SSTs into groups. The next column contains the number of tanks that belong to the same group as the sampled tank. The next two columns respectively report the salt cake and sludge volume contained in the proposed SST. The eleventh column presents the total waste volume contained in the entire group to which the proposed SST belongs. The final three columns respectively report the percentage of salt cake, sludge, and total waste found in the group as compared to all 149 SSTs.

As can be seen in Table I3-1, sampling the 10 SSTs proposed by this plan will gather information on 29 tanks and approximately 30% of the total sludge volume. These totals do not include information gathered as a result of sampling and analysis of SSTs B-110, U-110, B-201, and B-202 nor the 18 SSTs

Table I3-1. Preliminary Optimized SST Sampling Order.

PRELIMINARY OPTIMIZED SST SAMPLING ORDER

Sample Order	Wyden Bill List	Tank I.D. No.	Number Cores Per Tank	Number Segments Per Core	SORWT Waste Type Group	Primary & Secondary Waste Type	Number of Tanks In Group	Volume SaltCake In Tank (KGAL)	Volume Sludge In Tank (KGAL)	Total Volume In Group (KGAL)	% of Total SaltCake In Group	% of Total Sludge In Group	% of Total Waste In Group
1		B-111	2	5	XV	2C 5-6	3	0	236	516	0%	4%	2%
2	F	C-112	3	3	IX	TBP-F 1C	5	0	109	478	0%	4%	1%
3	F	C-109	3	2	IX	TBP-F 1C	(5)	0	62	#	#	#	#
4	H	C-106	2	4	XXIII	SRS TBP	2	0	197	429	0%	3%	1%
5		C-110	3	4	VIII	1C TBP	5	0	196	#	#	#	#
6		T-111	2	9	XIV	2C 224	3	0	456	904	0%	7%	2%
7	G	T-110	3	8	XIV	2C 224	(3)	0	376	#	#	#	#
8		BX-107	2	4	VIII	1C TBP	(5)	0	348	715	0%	6%	2%
9		BX-103	3	2	VI	TBP CW	7	0	62	489	0%	4%	1%
10		S-104	3	6	V	R	7	0	293	892	0%	7%	2%
Total			26				32	0	2335	4423	0%	34%	13%

* Tank is on the Push Mode List (WHC 86421-91-002)
 F Tank is on the Wyden Bill List as a Ferrocyanide Tank.
 G Tank is on the Wyden Bill List as a Gas Generating Tank.
 H Tank is on the Wyden Bill List for High Heat
 (#) Tank is member of a group previously sampled and not included in the totals.

core sampled in 1985 and 1986. Table I3-1 demonstrates the power of tank grouping by obtaining large amounts of characterization information with relatively few core sampling events. These 10 tanks also include four tanks identified in Public Law 101-510 and 1 tank requested by the SST retrieval program, therefore, satisfying most programmatic needs. Single-shell tank selection based upon SORWT groups will go a long way towards characterizing a significant portion of all SST waste before the closure of the SEIS database and still satisfy other SST analytical data needs.

The following are technical justifications for SST selection and placement in the optimized sampling order.

<u>Sample Order</u>	<u>Tank No.</u>	<u>Justification</u>
1.	B-111	This tank contains 237,000 gal of waste and belongs to a three tank group representing 516,000 gal of waste. The waste types held by these tanks are 2C and 5-6. Single-shell tank B-110 also is a member of this SORWT group and has been previously sampled. Core sampling of B-111 will provide a pair of tanks from this SORWT group from which to measure the group variability.
2.	C-112	This tank is a member of a five-tank group representing 478,000 gal of waste. This tank also is one of the primary in-farm scavenged-ferrocyanide tanks. Sampling and analysis of this tank will provide a great deal of knowledge pertaining to the FeCN safety issue. Although this tank is not on the Push-Mode List, examination of recent tank surveillance photographs (9/90) indicate that the waste surface is moist and relatively soft. There should be no technical difficulties in obtaining a core sample from this waste. However, significant safety issues must be addressed before sampling this tank because of its presence on the Public Law 101-510 List.
3.	C-109	This tank also is a member of the same SORWT group as C-112 and will provide additional data concerning the FeCN safety issue. Core sampling this tank will also furnish a pair of tanks from this group as outlined in the selection criteria.

<u>Sample Order</u>	<u>Tank No.</u>	<u>Justification</u>
4.	C-106	This tank is part of a SORWT group with only two SSTs containing 429,000 gal of strontium rich sludge. The retrieval program has requested a sample from this tank to characterize the physical properties of this waste to support retrieval equipment design to achieve interim stabilization, a TPA milestone. This tank also is identified in Public Law 101-510 as a high-heat tank. This tank will be sampled immediately after C-109 to alleviate the need to move the core sample truck to a different tank farm between core samples. This will minimize the time delay between SSTs.
5.	C-110	This tank is a member of a five-tank group, which represents 715,000 gal of waste. Although this tank is not on the Push-Mode List, surveillance photographs indicate that the waste is relatively soft and should not pose technical difficulties in obtaining a core sample. This tank also is in the C Tank Farm and can be obtained without inter-farm transport of the core sample truck.
6.	T-111	Although this tank is a member of a group that contains only three tanks, this group represents 904,000 gal of waste. This tank is on the Push-Mode List and presents no expected technical or safety issues.
7.	T-110	Single-shell tank T-110 is a member of the same group as T-111 and will satisfy the criteria requirement of two tanks per SORWT group. T-111 also is on the Push-Mode List and should be sampled without technical difficulty. This tank also is identified in Public Law 101-510 as a gas-generating tank and can satisfy safety programmatic sampling needs. This tank should be sampled after T-111 to remove the necessity to move the truck between tank farms between sampling events.
8.	BX-107	This tank is a member of the same SORWT group as SST C-110 and will satisfy the

Sample Order	Tank No.	Justification
9.	BX-103	<p>criteria requirement for pairs of tanks from the same group. Successful sampling and analysis of this tank will provide the five pairs of tanks from different SORWT groups to perform the variability study. This tank is not on the Push-Mode List; however, examination of recent tank surveillance photos (9/90) indicates that the crust is moist and relatively soft. This waste should not pose any technical or safety issues in sampling.</p>
10.	S-104	<p>This tank is a member of a seven-tank group representing 489,000 gal of waste. This tank is on the Push-Mode List and should not present any technical or safety-oriented difficulties. In light of the previously obtained sampling data (mid 1980s) on two other tanks in this group, additional sampling and analysis will provide further verification of the validity of the grouping methodology.</p> <p>This tank is a member of a seven-tank group containing exclusively REDOX (R) type waste representing 892,000 gal of waste. This tank is on the Push-Mode List and can be sampled with no technical or safety restrictions.</p>

13.2 PRELIMINARY INTEGRATED CORE SAMPLE SCHEDULE

The Preliminary Integrated Core Sample Schedule, presented in Figure I3-1, has been compiled as a result of the SORWT tank grouping model, resource availability, knowledge of programmatic needs, technological feasibility, and tank waste characterization technology (TWCT) best engineering judgement.

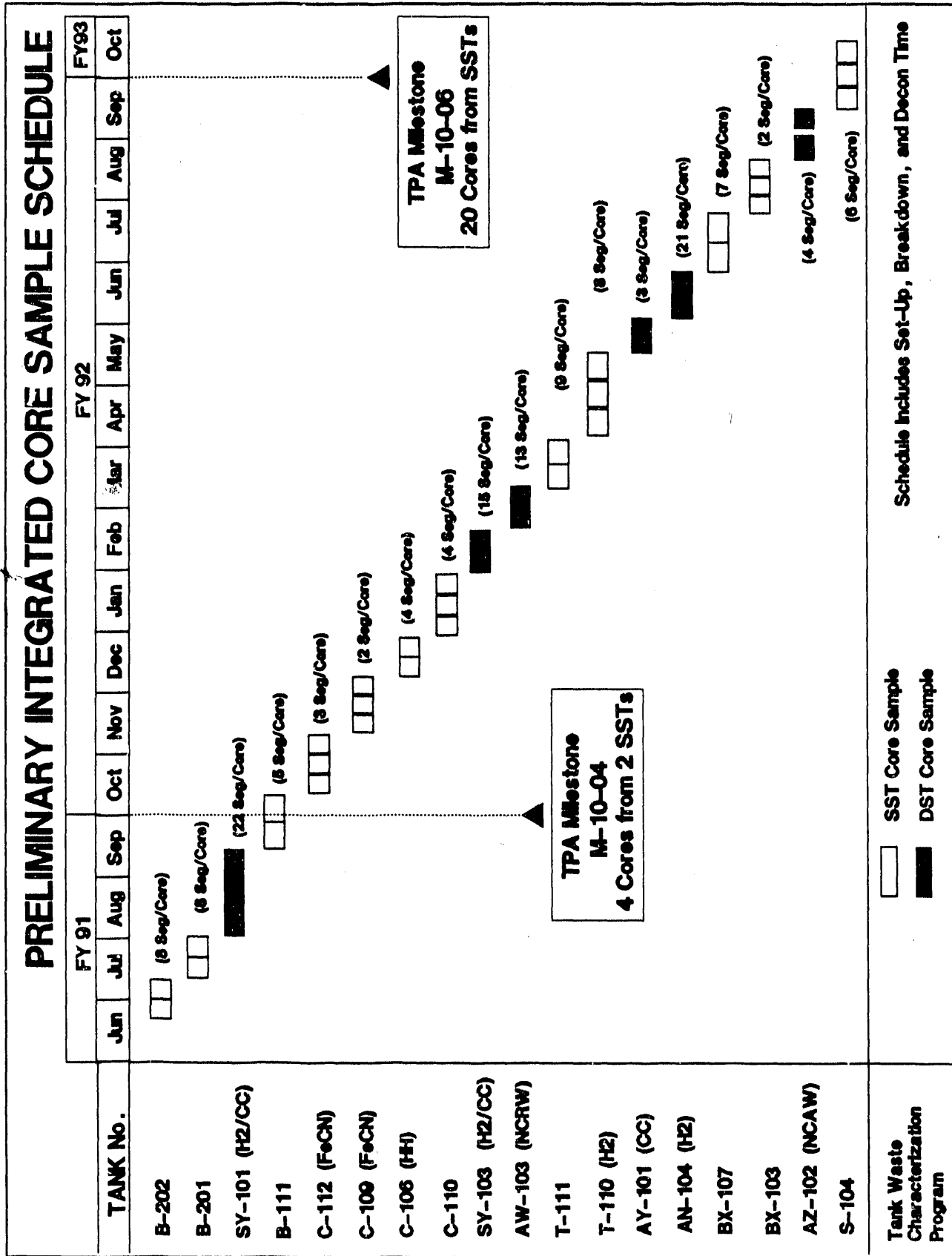
A number of assumptions have been made pertaining to the availability of the core sample trucks. These assumptions are as follows:

- The core sample truck is capable of obtaining three segments per day.
- The integrated core sample schedule includes down time for: set-up, breakdown, transportation, and equipment decontamination.
- Seven core samples from six different DSTs must be obtained between August 1991 and the end of FY 1992.

- The second core sample truck will be available for rotary mode sampling of hard cake waste at the end of FY 1992.
- The first core sample truck will begin sampling by push mode in June 1991.

The unshaded rectangles in Figure I3-1 indicate core samples from SSTs. The shaded rectangles indicate core samples from DSTs. The number of segments per core sample have been identified on the schedule. Three core samples are expected to be collected for FeCN Tanks C-112 and C-109 as well as SSTs T-110, C-110, BX-103, and S-104. This will support horizontal spatial variation studies detailed in Section I1.2.2. Applicable Tri-Party Agreement milestones M-10-04 and M-10-06 have been placed on the schedule. The proposed schedule indicates 24 core samples will be obtained in FY 1992.

Figure I3-1. Preliminary Integrated Core Sample Schedule.



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I4.0 DESCRIPTION OF THE SINGLE-SHELL TANKS SELECTED FOR SAMPLING

I4.1 TANK 241-B-111

Tank 241-B-111 (B-111) was constructed in 1943-1944 and was removed from service in 1976. Tank B-111 has a diameter of 75 ft and a nominal capacity of 500,000 gal. A sketch of this type of tank is provided in Figure A-12 in Appendix A. Tank B-111 contains 2C waste, 5-6 waste, and fission product (FP) waste as its predominant waste types. The waste is expected to be classified as extremely hazardous waste (EHW), class C low-level waste, and nontransuranic, based on TRAC evaluations. The tank has about 236,000 gal of sludge-type waste and 22,000 gal of drainable liquid remaining. Tank B-111 has an approximate solid waste height of 86 in. Eighty-six in. of waste should produce four full segments and one partial segment of sample material.

I4.2 TANK 241-C-112

Tank 241-C-112 (C-112) was constructed in 1943-1944 and was removed from service in 1976. The design of Tank C-112 is similar to that of Tank B-111. Tank C-112 contains ferrocyanide-scavenged tributyl phosphate (TBP-F) waste and first-cycle decontamination (1C) waste as its predominant waste types, with varying amounts of several miscellaneous wastes, such as coating waste (CW), FP waste, strontium semiworks/hot semiworks (SSW/HS) waste, and ion-exchange (IX) waste making up most of the remainder of the tank contents. The waste is expected to be classified as EHW, greater than class C low-level waste, and is believed to have a transuranic concentration between 100 nCi/g and 500 nCi/g, based on TRAC evaluations. Tank C-112 has been declared an Unresolved Safety Question because of the amount of ferrocyanide believed to be in the tank and the potential for a release from a ferrocyanide/nitrate reaction. Evaluation of this potential hazard is still ongoing and a full Readiness Review is expected to be performed before Tank C-112 is sampled. The tank has about 109,000 gal of sludge-type waste and 5,000 gal of drainable liquid remaining. Tank C-112 has an approximate solid-waste height of 40 in. Forty in. of waste should produce two full segments and one partial segment of sample material.

I4.3 TANK 241-C-109

Tank 241-C-109 (C-109) was constructed in 1943-1944 and was removed from service in 1976. The design of Tank C-109 is similar to that of Tank C-112 and B-111. Tank C-109 contains TBP-F waste and 1C waste as its predominant waste types, with varying amounts of several miscellaneous wastes, such as CW, evaporator bottoms (EB), SSW/HS waste, and IX waste making up most of the remainder of the tank contents. The waste is expected to be classified as EHW, class A low-level waste, and nontransuranic, based on TRAC evaluations. Tank C-109 has been declared an Unresolved Safety Question and has a status similar to that of C-112. The contains about 62,000 gal of sludge-type waste, and 4,000 gal of drainable liquid remaining. Tank C-109 has an approximate solid-waste height of 24 in. Twenty-four in. of waste should produce one full segment and one partial segment of sample material.

I4.4 TANK 241-C-106

Tank 241-C-106 (C-106) was constructed in 1943-1944 and was removed from service in 1979. The design of Tank C-106 is similar to that of Tank C-112. Tank C-106 contains strontium sludge (SRS) and tributyl phosphate (TBP) as its predominant waste types, with varying amounts of several miscellaneous wastes, such as Plutonium-Uranium Extraction (PUREX) high-level waste (P), PUREX supernatant sludge (PSS), and B Plant low-level waste (BL), making up most of the remainder of the tank contents. The waste is expected to be classified as dangerous waste (DW), greater than class C low-level waste, and is believed to have a transuranic concentration greater than 500 nCi/g, based on TRAC evaluations. Tank C-106 has been declared a high-heat tank and is under operating restrictions. The tank has about 197,000 gal of sludge-type waste and 48,000 gal of drainable liquid remaining. Tank C-106 has an approximate solid-waste height of 60 in. Eighty-three in. of waste should produce four full segments and one partial segment of sample material.

I4.5 TANK 241-C-110

Tank 241-C-110 (C-110) was constructed in 1943-1944 and was removed from service in 1976. The design of Tank C-110 is similar to that of Tank C-112. Tank C-110 contains TBP waste and 1C waste as its predominant waste types, with varying amounts of several miscellaneous wastes, such as PUREX organic wash waste (OWW), EB, and IX waste, making up most of the remainder of the tank contents. The waste is expected to be classified as EHW, greater than class C low-level waste, and is believed to have a transuranic concentration between 100 nCi/g and 500 nCi/g, based on TRAC evaluations. Tank C-110 is under no operating restrictions. The tank has about 196,000 gal of sludge-type waste and 21,000 gal of drainable liquid remaining. Tank C-110 has an approximate solid-waste height of 73 in. Seventy-three inches of waste should produce three full segments and one partial segment of sample material.

I4.6 TANK 241-T-111

Tank 241-T-111 (T-111) was constructed in 1943-1944 and was removed from service in 1974. The design of Tank T-111 is similar to that of Tank C-112 and B-111. Tank T-111 contains lanthanum fluoride (224) waste and 2C waste as its predominant waste types with no significant amounts of any other waste types. The waste is expected to be classified as EHW, class C low-level waste, and is believed to be nontransuranic, based on TRAC evaluations. Tank T-111 is under no operating restrictions. The tank has about 456,000 gal of sludge-type waste and 51,000 gal of drainable liquid remaining. Tank T-111 has an approximate solid-waste height of 167 in. One hundred sixty-seven inches of waste should produce eight full segments and one partial segment of sample material.

14.7 TANK 241-T-110

Tank 241-T-110 (T-110) was constructed in 1943-1944 and was removed from service in 1976. The design of Tank T-110 is similar to that of Tank C-112 and B-111. Tank T-110 contains 224 waste and 2C waste as its predominant waste types with no significant amounts of any other waste types. The waste is expected to be classified as EHW, class C low-level waste, and is believed to be nontransuranic, based on TRAC evaluations. Tank T-110 is classified as an Unresolved Safety Question because of the observed hydrogen gas generation behavior. Evaluation of this potential hazard is still on-going and a full Readiness Review is expected to be performed before Tank T-110 is sampled. The tank has about 376,000 gal of sludge-type waste and 42,000 gal of drainable liquid remaining. Tank T-110 has an approximate solid-waste height of 137 in. One hundred sixty-seven inches of waste should produce seven full segments and one partial segment of sample material.

14.8 TANK 241-BX-107

Tank 241-BX-107 (BX-107) was constructed in 1943-1944 and was removed from service in 1977. The design of Tank BX-107 is similar to that of Tank C-112 and B-111. Tank BX-107 contains TBP waste and 1C waste as its predominant waste types with varying amounts of miscellaneous wastes, such as EVAP, and IX waste. The waste is expected to be classified as EHW, class C low-level waste, and is believed to be nontransuranic, based on TRAC evaluations. Tank BX-107 is not under any operating restrictions. The tank has about 348,000 gal of sludge-type waste and 24,000 gal of drainable liquid remaining. Tank BX-107 has an approximate solid-waste height of 127 in. One hundred twenty-seven inches of waste should produce six full segments and one partial segment of sample material.

14.9 TANK 241-BX-103

Tank 241-BX-103 (BX-103) was constructed in 1943-1944 and was removed from service in 1977. The design of Tank BX-103 is similar to that of Tanks C-112 and B-111. Tank BX-103 contains TBP waste and CW as its predominant waste types with varying amounts of miscellaneous wastes, such as EVAP, OWW, and IX waste. The waste is expected to be classified as EHW, class C low-level waste, and is believed to be nontransuranic, based on TRAC evaluations. Tank BX-103 is not under any operating restrictions. The tank has about 66,000 gal of sludge-type waste and 4,000 gal of drainable liquid remaining. Tank BX-103 has an approximate solid-waste height of 24 in. Twenty-four inches of waste should produce one full segment and one partial segment of sample material.

14.10 TANK 241-S-104

Tank 241-S-104 (S-104) was constructed in 1950-1951 and was removed from service in 1968. Tank S-104 has a diameter of 75 ft and a nominal capacity of 750,000 gal. A sketch of this type of tank is provided in Figure A-12. Tank S-104 contains R waste as its sole waste type. The waste is expected to be classified as EHW, greater than class C low-level waste, and is believed to

have a transuranic concentration of 100 nCi/g to 500 nCi/g, based on TRAC evaluations. Tank S-104 is not under any operating restrictions. The tank has about 293,000 gal of sludge-type waste and 29,000 gal of drainable liquid remaining. Tank S-104 has an approximate solid-waste height of 107 in. One hundred seven inches of waste should produce five full segments and one partial segment of sample material.

14.11 DESCRIPTION OF THE SAMPLING

Sampling will be performed in the same manner as described in Section I3.2 and Appendix B of the WCP. All core sampling in the next 10 SSTs will be accomplished in push-mode. Further sampling requirements have been identified in the safety analysis that was developed for the hydrogen-generation tank, SY-101, and will be identified in the forthcoming safety analysis for the ferrocyanide tanks. Sampling Procedures for tanks identified in Public Law 101-510 will be specific, but there will be only one Sampling Procedure for non-Public Law 101-510 list tanks. These further safety requirements will be incorporated into the sampling procedure for each tank identified as needing additional precautions. Samples are taken and shipped in accordance with Tank Farms Operations procedures T0-020-450, "Perform Core Sampling," and T0-080-090, "Ship Core Samples." The design of the sampler has been changed to eliminate decontamination of the sampler and to permit sampling to within a range of one and a half to 3 in. of the bottom of the tank. This sampler is made of stainless steel and is slightly smaller in diameter than the old sampler. Because of the smaller diameter, the total volume of sample is reduced from 250 mL to 187 mL. The sampling of these tanks will be done using NPH as the hydrostatic fluid until a replacement system can be developed.

Sample breakdown and subsampling will be performed as described in Chapter I6.0 of this appendix in accordance with the procedures in Table I4-1. Subsampling for composites has been modified for these tanks so that samples to be analyzed for physical properties (rheology) are not homogenized and core composite subsamples are obtained by either taking random aliquots from different locations along the length of the segment or by splitting the sample along its length. New extrusion equipment compatible with the new sampler will be used.

Table I4-1. Sample Breakdown and Subsampling Procedures.

WESTINGHOUSE HANFORD COMPANY PROCESS CHEMISTRY
LABORATORIES DESK INSTRUCTIONS

No.	Title
LT-151-101	Core Segment Receipt and Preparation
LT-549-101	Core Segment Extrusion
LT-549-102	Homogenization and Homogenized Segment Sampling
LT-549-103	Core Compositing and Sampling

BATTELLE-PACIFIC NORTHWEST LABORATORY PROCEDURES

No.	Title
PNL-AL0-010 Rev. 0	325 Laboratory Single-shell tank Sample Receiving and Subsample Analysis System
325-A-29 Rev. 0	Receiving of Waste Tank Samples in Onsite Transfer Cask
PNL-AL0-130 Rev. 0	Receipt and Inspection of SST Samples
325-EXT-1 Rev. 0	Receipt and Extrusion of Core Samples at 325A Shielded Facility
PNL-AL0-135 Rev. 0	Homogenization of Solutions, Slurries, and Sludges

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15.0 OBJECTIVES FOR SAMPLING AND ANALYSIS

The objectives for sampling and analysis of the next 10 SSTs are to characterize the physical and chemical properties of the waste contained in these selected tanks. This characterization information will directly support most of the programs involved in the effort to close the SST operable units. The acquired data can also be used to check the laboratory's analytical performance and to statistically verify the grouping results of the SORWT model. The various measurements performed in order to accomplish the sampling and analysis objectives have been outlined below. These sampling and analysis objectives are for (1) the baseline case SST and (2) tanks identified in Public Law 101-510.

15.1 BASELINE CASE SINGLE-SHELL TANKS

15.1.1 Single-Shell Tank Waste Constituent Inventory

The primary objective of the sampling and analysis plan for the next ten SSTs is to obtain estimates of the total quantity of Type I and Type II analytes in each SST sampled. These inventory estimates are essential for making risk assessment-based disposal decisions and for the design of pretreatment and final waste-disposal systems. Estimated inventories are direct inputs into Long-Term Release Risk (LTRR), Short-Term Intruder Risk (STIR), and waste classification model (CLASS) models for determining the risk to the public health and the environment associated with the tank waste.

The constituent inventories can be calculated by either treating the core samples as random samples and averaging the results or by using a spatial model. The calculated inventories will include an estimated total quantity of each selected analyte and its corresponding confidence interval based upon the analytical and sampling variability. The use of a spatially dependant model will require at least three cores to produce better results than the simple random sample model.

The analytical data necessary to estimate the constituent inventories will be collected by obtaining at least two cores from two different risers in each SST and compositing representative portions of each homogenized 19 in. segment. Aliquots will be taken from each homogenized core composite and will be analyzed in the laboratory for Type I and II analytes and for other compounds of regulatory concern.

A list of the analytes to be measured and the associated laboratory procedures is presented in Table I5-1. The first column of Table I5-1 identifies the preparation used to obtain analytical results. The preparations can be either acid digestion, water digestion, or fusion/acid digestion. The acid digestion is performed to satisfy regulatory metals (ICP) analyses. The water digestions are conducted primarily to obtain water soluble anions, but are also analyzed for water soluble cations such as Cr^{+6} . The fusion/acid digestion are done primarily to obtain a total dissolution of

Table I5-1. Analytes and Methods for Analysis of Core Composites From Next Ten Single-Shell Tanks. (4 sheets)

Preparation	Method	Analyte	PNL preparation procedure	PNL measurement procedure	MHC preparation procedure	MHC measurement procedure	Priority type	Basis	Other	
Acid digestion No. 1 (PNL-ALO-101) (LA-505-159)	ICP--metals	Ag	PNL-ALO-101	PNL-SP-7	LA-505-159	LA-505-151	II	LTRR	TCLP	
		Al	"	"	"	"	I	CLASS	REG	
		As	"	"	"	"	II	LTRR	TCLP	
		Ba	"	"	"	"	I	STIR	TCLP	
		Be	"	"	"	"	II	LTRR	REG	
		Bi	"	"	"	"	I	STIR	--	
		Ca	"	"	"	"	II	STIR	REG	
		Cd	"	"	"	"	I	STIR	TCLP	
		Co	"	"	"	"	NE	--	REG	
		Cr	"	"	"	"	I	STIR	TCLP	
		Cu	"	"	"	"	NE	--	REG	
		Fe	"	"	"	"	I	STIR	REG	
		K	"	"	"	"	II	CLASS	REG	
		Mg	"	"	"	"	NE	--	REG	
		Mn	PNL-ALO-101	"	PNL-SP-7	LA-505-159	LA-505-151	I	STIR	REG
		Na	"	"	"	"	"	I	LTRR	REG
		Ni	"	"	"	"	"	I	STIR	REG
		Pb	"	"	"	"	"	I	STIR	TCLP
		Sb	"	"	"	"	"	II	LTRR	REG
		Se	"	"	"	"	"	III	--	TCLP
		Sn	"	"	"	"	"	III	--	REG
		Tl	"	"	"	"	"	NE	--	REG
		V	"	"	"	"	"	II	LTRR	REG
		Zn	"	"	"	"	"	NE	--	REG
		Zn (a)	"	"	"	"	"	I	CLASS	TD
		Si	"	"	"	"	"	III	--	S
		Th	"	"	"	"	"	I	LTRR	--
		U	"	"	"	"	"	I	LTRR	--
Zr	"	"	"	"	"	I	STIR	--		

Table I5-1. Analytes and Methods for Analysis of Core Composites From Next Ten Single-Shell Tanks. (4 sheets)

Preparation	Method	Analyte	PNL preparation procedure	PNL measurement procedure	WHC preparation procedure	WHC measurement procedure	Priority type	Basis	Other	
Acid digestion No. 2	GFAA--metals	As	PNL-ALO-101	PNL-ALO-214	NC	NC	Same	Same	Same	
		Se	"	PNL-ALO-215	"	"	"	"	"	
		Sb	"	PNL-ALO-219	"	"	"	"	"	
Direct acid digestions	HYAA metals	As	NC	NC	NA	LA-355-131	"	"	"	
		Se	"	"	"	LA-365-131	"	"	"	
Fusion/acid digestion	CVAA metals	Hg	NA	PNL-ALO-213	NA	LA-325-102	II	LTRR	TCLP	
		Same as acid digestion No. 1	PNL-ALO-102	PNL-SP-7	LA-504-141	LA-505-151	Same	Same	Same	
Water digestion No. 1	ICP--metal anions (b)	Cr(VI)	PNL-ALO-103	PNL-SP-7	LA-504-101	LA-505-151	Same	Same	Same	
		Mn(VII) Si(V)	" "	" "	" "	" "	" "	" "	" "	
Water digestion No. 1	IC--anions UV-VIS (d) anion SIE anion (d) TIC--anion TBD-- complexants (e)	NO ₃ ⁻	PNL-ALO-103	PNL-ALO-212	LA-504-101	LA-533-105	I	LTRR	--	
		NO ₂ ⁻	"	"	"	"	"	I	STIR	--
		F ⁻	"	"	"	"	"	I	LTRR	--
		Cl ⁻	"	"	"	"	"	III	S	--
		SO ₄ ⁻²	"	"	"	"	"	I	STIR	--
		PO ₄ ⁻³	"	"	"	"	"	I	STIR	--
		NO ₂ ⁻	NA	NA	NA	LA-504-101	LA-645-101	Same	Same	--
		F ⁻	PNL-ALO-103	HMVP-2	NA	NA	NA	Same	Same	--
		CO ₃ ⁻²	PNL-ALO-103	7-40.7	LA-504-101	LA-622-102	LA-622-102	I	STIR	TD
		EDTA, HEDTA, citrate, acetate, oxalate	TBD	TBD	TBD	TBD	TBD	I	LTRR STIR CLASS --	TD
		NH ₃	NA	PNL-ALO-226	"	LA-634-102	LA-634-102	III	--	TD, REG
		C ^f	PNL-ALO-103	7-40.7	LA-504-101	LA-344-105	LA-344-105	NA	--	S

Table I5-1. Analytes and Methods for Analysis of Core Composites From Next Ten Single-Shell Tanks. (4 sheets)

Preparation	Method	Analyte	PNL preparation procedure	PNL measurement procedure	WHC preparation procedure	WHC measurement procedure	Priority type	Basis	Other
Water digestion No. 2	pH anion	OH ⁻	NA	PNL-ALO-225	NA	LA-212-103	I	STIR	REG
Direct distillation	UV-VIS spec anion SIE--anion	CN ⁻ S ^{2-(e)}	NA NA	PNL-ALO-270 TBD	NA NA	LA-695-101 TBD	I NE	LTRR --	REG REG
Fusion/acid digestion (PNL-ALO-102) (LA-549-141)	Fluorimeter radiochem Alpha--radiochem	U Total α ⁹ 239, 240, Pu 238 Pu 241, Am 244 Cm 237 Np	PNL-ALO-102 PNL-ALO-427 PNL-ALO-423 PNL-ALO-424 PNL-ALO-425	PNL-ALO-445 PNL-ALO-421 PNL-ALO-421 PNL-ALO-422 PNL-ALO-421 PNL-ALO-422 PNL-ALO-421 PNL-ALO-422	LA-549-141 LA-548-101 LA-503-156 LA-503-156 LA-933-141	LA-925-106 LA-508-104 LA-508-104 LA-508-051 LA-508-104 LA-508-051 LA-508-104 LA-508-051	I I, II I, I II I II II	LTRR LTRR STIR LTRR STIR STIR LTRR	-- MRC S MRC MRC MRC MRC
	Beta--radiochem	242m Am 243 Am (e) Total B	Calculated PNL-ALO-430	PNL-ALO-431	LA-548-103	LA-503-105	-- -- --	-- -- --	S
	LEPS--radiochem	90 SrY 99 Tc 129 I 93 Nb (e) 137 Cs others	PNL-ALO-433 PNL-ALO-432 PNL-ALO-454 PNL-ALO-456 TBD	PNL-ALO-431 PNL-ALO-431 PNL-ALO-451 TBD	LA-220-102 LA-438-101 LA-378-103 TBD	LA-508-105 LA-508-121 (h) LA-508-052	I I I	STIR LTRR LTRR	MRC MRC MRC
Fusion/acid digestion (PNL-ALO-102) (LA-549-141)	GEA--radiochem	79 Se	PNL-ALO-451	PNL-ALO-451	LA-548-121	LA-508-052	II I	LTRR LTRR	-- MRC
	Liquid scintillation radiochem Mass spec radiochem	238 U, 234 U Pu-- isotopic 240 Pu 241 Pu U-- isotopic 238 U, 234 U 235 U, 234 U	PNL-ALO-440 PNL-ALO-102 PNL-ALO-102	PNL-ALO-442 PNL-ALO-455 PNL-ALO-455	LA-365-132 NC NC	LA-508-121 NC NC	III I I	LTRR LTRR STIR	-- MRC --

Table I5-1. Analytes and Methods for Analysis of Core Composites From Next Ten Single-Shell Tanks. (4 sheets)

Preparation	Method	Analyte	PNL preparation procedure	PNL measurement procedure	WMC preparation procedure	WMC measurement procedure	Priority type	Basis	Other
Water digestion (PNL-ALO-103) (LA-504-101)	Liquid scintillation radiochem	¹⁴ C	PNL-ALO-444	PNL-ALO-442	LA-348-104	LA-508-121	I	LTRR	MRC
Acid digestion (PNL-ALO-101) (LA-505-159)	Beta radionuclide	³ H ⁶³ Ni	PNL-ALO-441 PNL-ALO-101	PNL-ALO-443 TBD	LA-218-113 NM	LA-508-121 NM	ME I	-- CLASS	MRC MRC
Purge/trap	Organic	Volatile organics	PNL-ALO-335	PNL-ALO-335	NC	NC	ME	--	REG
Extraction	Organic	Semi-volatile organic	PNL-ALO-120	PNL-ALO-345	NC	NC	ME	--	S
Extraction	Organic (EOX)	Halogen organics	NA	PLN-ALO-320	NC	NC	ME	--	REG
Direct	Gravimetric wt% H ₂ O	H ₂ O	NA	PNL-ALO-504	NA	LA-564-101	MA	NA	REG
Acetic Acid digestion	Characteristic TCLP	Ag, Ba, Cd, Cr, Pb, As, Se, Hg	TBD	Same as ICP	TBD	Same as ICP	Same	Same	REG
Water digestion No. 2	Characteristic corrosivity	pH	Same	Same	Same	Same	Same	Same	REG
Direct	Characteristic ignitibility reactivity	DSC TGA Flash point (e)	NA	RDS-TA-1	MA	T043 A-01712F T044 A-01712F LA-560-112 LA-514-113	MA	NA	REG
	Reactivity	CN ⁻ , S ²⁻ , e	TBD	TBD	TBD	TBD	MA	MA	REG

a The following metals may be determined more accurately on fused sample.
 b Performed to evaluate speciation of metals.
 c Performed to check for acid insoluble metals (e.g., Si, Zr, Th, Al, U, Bi, Fe).
 d Methods used by laboratories in place of IC analysis.
 e Analyte will not be analyzed for these ten tanks.
 f ICP will analyze for water soluble organic carbon to estimate complexant levels.
 g Will not be performed on next ten tanks unless improved method is identified.
 Westinghouse Hanford uses liquid scintillation counting.
 Class = waste classification.
 CVAA = cold vapor atomic absorption.
 GEA = gamma energy analysis.
 GFAA = graphite furnace atomic absorption.
 HYAA = hydride atomic absorption.
 IC = ion chromatography.
 ICP = inductively coupled plasma.
 LEPS = low energy photon spectroscopy.

LTRR = long-term release risk.
 MA = not applicable.
 NC = no capability.
 NE = not evaluated.
 NM = no method.
 NRC = U.S. Nuclear Regulatory Commission Low-Level waste classification.
 PNL = Pacific Northwest Laboratory.
 REG = regulatory interest (CLP or Appendix IX).
 S = screen.
 STIR = short-term intruder risk.
 TBD = to be determined.
 TCLP = metal required for toxic characterization leach procedure.
 TIC = total inorganic carbon.
 TD = technology development.
 WHC = Westinghouse Hanford Company.

samples for radionuclide analysis but are also analyzed for acid insoluble cations such as silica. The second column in Table I5-1 indicates the analytical method that will be employed to measure the concentration of each analyte. Column 3 identifies the individual analytes that are required to be analyzed. Columns 4 and 5 respectively list the analytical procedures employed by the PNL 325 Laboratory to perform the specified preparation and measurement. The next two columns presents the procedures for the same preparation and measurement used at the Westinghouse Hanford 222-S Laboratory. The eighth column identifies the analyte priority as determined by the risk-based assessment models described in Section I1.2.1. Column 9 indicates which assessment model was used to classify the analyte priority. The final column lists other justifications for measurement of the particular analyte.

I5.1.2 Physical Properties

The second major objective is to measure the physical properties of the waste to support waste-retrieval technology development. The physical characteristics of SST waste are required to (1) develop design criteria for waste-retrieval equipment, (2) provide a basis for simulated waste development, and (3) provide a basis for validation of equipment testing using design criteria and simulated waste. The analytical methods to determine the physical properties of the waste as it actually exists in the tank require a substantial amount of unhomogenized sample. Rheological properties are of particular interest in the design of waste-retrieval equipment and require 50 to 100 g of unhomogenized sample.

The large quantities of sample needed for rheology tests mean that the chemical and radiological analysis on that segment must be limited. Several alternatives were evaluated for obtaining both physical and chemical analysis from a single core. The alternative of taking a second core for physical characterization was eliminated because of the limited number (and size) of risers in some tanks and because taking two samples from the same riser could impact the chemical or physical characteristics of the second core. In addition, taking additional cores for physical measurements will significantly increase the hot cell workload. Another alternative was to select certain segments for physical measurements, removing only enough of the selected segment by either random sampling or splitting along the length of the segment to prepare two core composites and a small segment archive sample. This is the alternative chosen for these next tanks. Selection of every other segment for physical measurements would give data for the entire tank depth. However, if the waste is soft and uniform like Tank 110-B, only one segment may be required to obtain the rheological properties of the tank. If samples are too dry and viscous for rheology measurements, they must be diluted. Since the most accurate and random subsampling can be done when the segment is in the extrusion tray, the decision to choose the segment for rheology must be made before or immediately after the sample is extruded. Comparison and selection of segments is limited. Therefore, for these next ten tanks, segments near the top, middle, and bottom of the first core sampled will be used for rheology. If, during the sampling of core 1, a unique phase is found that is not analyzed for rheology, an effort will be made to find a similar segment in core 2 for rheological analysis.

The physical properties that have been identified as important for all SSTs are presented in Table I5-2. The first column in Table I5-2 lists the physical characteristic to be measured. The next column identifies the

Table I5-2. Baseline Case SST Physical Analysis.
(2 sheets)

Physical Characteristic	Level of Analysis	Collection of Aliquot	PML 325 Procedure	WHC 222-S Procedure
Bulk Density	Every Segment Every Core	At Extrusion	WHC-053-1	LT-549-101
Volume of Solids	Every Segment Every Core	At Extrusion	325-EXT-1	LT-549-101
Volume of Liquid	Every Segment Every Core	At Extrusion	325-EXT-1	LT-549-101
Particle Size	Every Segment Every Core	Pre-Homogenized	2-50.3	T044 A-01712F
Penetrometer	Every Segment Every Core	Pre-Homogenized	PML-ALO-506	LT-549-101
Rheologic Properties - Shear Strength - Shear Rate ^a - Absolute Viscosity vs. Shear Rate - Yield Stress - Absolute Viscosity ^a	Every Other Segment One Core Per Tank	Pre-Homogenized	WHC-053-1	TBD
Settling Velocity ^b	Every Other Segment One Core Per Tank	Pre-Homogenized	WHC-053-1	TBD
Weight Percent Solids	Every Segment Every Core	Homogenized Segment	PML-ALO-504	LA-564-101
Differential Scanning Calorimetry	Every Visible Phase for Every Segment	Pre-Homogenized Segment	RDF-TA-1	T042 A-01712F
Thermogravimetry	Every Visible Phase for Every Segment	Pre-Homogenized Segment	RDF-TA-1	T045 A-00712F
Bulk Slurry Density ^c	Every Core	Homogenized Core Composite	WHC-053-1	LA-560-101
Centrifuged Solids Density ^c	Every Core	Homogenized Core Composite	WHC-053-1	TBD

Table I5-2. Baseline Case SST Physical Analysis.
(2 sheets)

Physical Characteristic	Level of Analysis	Collection of Aliquot	PNL 325 Procedure	WHC 222-S Procedure
Centrifuged Supernate Density ^c	Every Core	Homogenized Core Composite	WHC-053-1	LA-510-112
Weight Percent Centrifuged Solids ^c	Every Core	Homogenized Core Composite	WHC-053-1	TBD
Volume Percent Centrifuged Solids ^c	Every Core	Homogenized Core Composite	WHC-053-1	LA-519-132
Weight Percent Dissolved Solids ^c	Every Core	Homogenized Core Composite	WHC-053-1	LA-504-101

^aSelected rheologic properties should be measured on the undisturbed sample and on dilutions of 1:1 and 3:1 at two different temperatures, ambient and 95 °C.

^bThe settling velocity for solids particles and bulk density should be determined for all rheologic dilutions.

^cThese physical properties shall be measured only for slurry sample.

TBD - To Be Determined

frequency in which this parameter will be measured. Column 3 indicates from which subsample the aliquot was obtained. The remaining two columns respectively report the procedures utilized by the 325-A and 222-S Laboratories.

The bulk density, penetrometer, volume of solids, and volume of liquids will be determined for every segment from every core at the time of extrusion into the hot cell. The particle size shall be measured for every segment. In order to evaluate tank reactivity safety concerns, differential scanning calorimetry (DSC) and thermal gravimetry (TGA) analyses will be performed on every visible phase in every segment prior to homogenization. Rheologic properties and settling velocity will be measured for every other segment from the first core collected out of an SST. Porosity and compressive strength are applicable only to hard saltcake and sludges, and will not be needed for wastes in these tanks. Rheological measurements will be made on direct samples when possible and on 1:1 (water:sample) and 3:1 dilutions at ambient hot cell temperatures and an elevated temperature (95 °C). Solid settling velocities will be determined for the diluted samples. The weight percent solids will be determined for every segment from every core. The remainder of the physical measurements will be conducted on one core composite from each core.

15.1.3 Waste Designation

The inorganic core composite analyses for chemicals are used to designate waste, using the toxic equivalent concentration (TEC) calculation. A reference compound is identified for each inorganic analyte and an appropriate toxicity class is determined. As reported in Washington State Dangerous Waste Regulations (WAC 173-303-101), the TEC calculations are a sum of the fractions based on the weight percent of the constituent and its toxicity class. Designation of a waste as either EHW or DW can be determined from the Toxic Dangerous Waste Mixtures Graph (WAC 173-303-9906). The regulatory threshold TEC value for EHW for SSTs is 0.01%.

Volatile and semi-volatile organic analyses for designation, based on toxicity and carcinogenicity, will be performed for every segment from every core. Samples for volatile organic analysis will be taken as soon as possible from unhomogenized segments not chosen for physical analysis in the same manner as Phase IA and IB. Semi-volatile analysis aliquots will be taken from the homogenized core composite. If problems are encountered (with GC/MS equipment or NPH contamination during the initial testing) that cannot be resolved within a reasonable time (1 wk), these analyses will be discontinued until problems are resolved. However, sample shall be archived in a sealed container to be analyzed at a later date. Organic analyses will include all the analytes described in the WCP.

The TCLP will be performed on an aliquot from one core composite from each riser and analyzed for the eight toxic metals. Matrix spikes will be used to evaluate ICP and atomic absorption (AA) performance for each metal. Results will be used to designate waste and to evaluate new TCLPs that have been modified for hot cell applications.

15.1.4 Evaluation of Transport Constants

Work on the recommendations report has identified the verification of the uncertainty in the transport model as a key factor affecting decisions concerning the waste. The two major transport factors that depend on the properties of the waste are (1) the solubility of the analytes and (2) the adsorption coefficient (K_d) of the analyte in the soil. Even though the experimental design for tests (to determine these factors on core composites) is not currently available, all remaining sample from each core composite after all analyses have been completed should be archived for evaluation at a later time.

15.1.5 Analysis of Errors

Estimation of the sources of error is essential to accurately characterize SSTs. The components of the total error can be broken down into its component parts. These component parts are:

- Analytical error
- Sampling error
- Segment homogenization error
- Composite homogenization error.

The analytical error will be estimated by performing duplicate analyses for all parameters. This also will allow the responsible chemist to identify anomalous results requiring reruns at the time of analysis.

Evaluation of tank-sampling errors by taking two cores from one riser, as done in Phase IA and IB, will not be done for the next 10 tanks because information from different risers provides more useful information.

The error due to segment homogenization will be determined by performing a homogenization test on every other segment for the second core (nonphysical analysis core) and at least once per core for all remaining cores from each selected SST. The test will be carried out by homogenizing the segment and then taking two subsamples from two opposite (left/right or top/bottom) locations for analysis. Duplicate 1 g aliquots of each subsample will be acid-digested and analyzed for metals by ICP, ^{137}Cs by gamma energy analysis (GEA) and total alpha analysis. Analytical errors should be small enough to permit detection of homogenization errors of at least 10%. If larger errors are noted that indicate problems with homogenization, it is the responsibility of the inorganic or radiochemical technical leader to bring this to the attention of the hot cell technical leader for evaluation. If segment samples differ significantly in consistency, the hot cell technical leader is responsible for initiating and requesting additional homogenization tests to support the work. Segments chosen for rheological analyses will not be homogenized and cannot be used in this test.

Errors associated with core compositing will be evaluated by preparing two composites for each core from each tank, homogenizing the composites, and analyzing the composites in duplicate. This also will provide combined homogenization and analytical error estimates for all the analytes analyzed in composites. Additionally, this provides a balanced data set that may be used in evaluating decision quality.

15.1.6 Vertical Spatial Variation

The vertical spatial variation will only be determined for those tanks identified in Public Law 101-510 during the next 10 sampling and analysis events. These are the only SSTs where chemical and radiological analyses will be performed on a segmental basis. The analytical results data will be utilized to generate a three-dimensional model of the spatial distribution for each analyte of concern. The distribution model will be prepared using a three-dimensional kriging technique and software developed by PNL. A constituent inventory can be estimated by integrating the concentration distribution over the entire tank. The three-dimensional distribution model should be able to indicate whether large concentrations of safety-related compounds, such as ferrocyanides, have congregated into distinct layers. Completion of the three-dimension spatial variation study should significantly assist in the resolution of the unclosed safety issues associated with ferrocyanides.

Vertical distribution studies are not planned for the remaining non-Public Law 101-510 list tanks. Segment samples from these three tanks will be archived so analyses can be performed at a later date, if required. Core-composite analytical results are sufficient to produce constituent inventories. A preliminary leave-retrieve sorting of SSTs can be accomplished based upon the constituent inventories. If risk-assessment and waste-designation evaluations indicate that a specific SST might be a candidate for in situ treatment and disposal, the archived samples can be analyzed on a segmental basis to provide a more complete characterization of the subject waste and to re-evaluate the candidacy for in place disposal based upon the segmental level analyses. Additional core sampling and analyses would be required for an SST in-situ disposal is still considered appropriate after the analyses on the archived samples.

15.1.7 Horizontal Spatial Variation

The horizontal spatial variation can be estimated for those SSTs where three cores from three different risers were obtained. Three distinct analytical results data points will allow for triangulation, which cannot be accomplished by two cores per tank. Three cores are planned to be obtained from six of the next ten SSTs sampled. These tanks are C-112, C-109, C-110, T-110, BX-103, and S-104. The first two C Farm tanks are ferrocyanide tanks, which will be characterized using the techniques discussed in the preceding section, thus, horizontal distribution studies will not be performed on these tanks. The horizontal distribution of the constituents will be determined for the other four specified tanks using two-dimension kriging techniques currently available with commercial software. Two-dimension concentration contour maps can be prepared depicting the horizontal distribution of analyte concentrations.

The horizontal distribution of the SSTs (with only two cores per tank) can only be determined in one dimension--between the two sample points. However, these tanks are members of SORWT groups from which the horizontal spatial variation has been determined from a previously sampled tank. If the SSTs are reasonably similar in physical and chemical characteristics, the horizontal spatial variation of the reference tank could be imposed upon the remaining members of the SORWT group.

I5.1.8 Holding Time

Phase IA/IB statistical data analyses have indicated that significant holding time effects are not present for the analytes included in the holding time study. Since an insufficient amount of analytical results data was available to determine if holding time effects were present for Cr^{+6} , a limited holding time study for this analyte will be accomplished on one tank. Six aliquots will be collected from a homogenized core composite from SST S-104 and analyzed for water leach ICP at six different dates. These dates shall be 15, 30, 45, 60, 120, and 180 days after collecting the sample. SST S-104 was chosen for the holding time study because it was expected to contain the largest concentration of Cr^{+6} out of the next ten SSTs to be sampled. Additional holding time studies are not planned for the next 10 SSTs. Volatile organic compounds were not included in the Phase IA/IB holding time study because of the NPH contamination difficulty. When this NPH analysis problem is solved, a further holding time study for volatile organic compounds should be enacted.

I5.1.9 Single-Shell Tank Waste Standards Program

A Hanford Site SST Waste Standards Program should be implemented to evaluate the performance of both the 325 and 222S laboratories. The standards program should monitor all analytical systems from solids dissolution to final measurement of all parameters. The standards program should be designed to:

- Evaluate interlaboratory calibration and instrument control using independent standards
- Evaluate short-term performance on varying sample matrices through a referee or exchange program using actual core composite samples
- Evaluate long-term performance of both laboratories by analyzing a working standard that is prepared in bulk from several SST segments or composites containing components of interest over an extended period of time.

The results of this program will be used to monitor and maintain high quality analytical systems in support of the SST waste characterization program and to produce continuity of results over the life of the program.

I5.1.10 Tank Stability

The waste reactivity will be evaluated by performing DSC tests on each distinct visible phase of waste or at least once per segment for every core in each tank. These samples are taken before homogenization so that actual tank

conditions are being evaluated. Chemical analyses for nitrates, nitrites, ammonia, TOC, and other organics in composites will support further reactivity evaluations.

15.2 SINGLE-SHELL TANKS IDENTIFIED IN PUBLIC LAW 101-510

In addition to the sampling and analysis objectives specified in the preceding sections, SSTs identified in Public Law 101-510 require further analytical measurements in order to resolve the safety concerns associated with those tanks. Four of the next 10 SSTs to be core sampled are identified in Public Law 101-510. The tanks and their unresolved safety concerns are C-112 (FeCN), C-109 (FeCN), C-106 (HH), and T-110 (gas). Each of the programmatic organizations responsible for these associated safety questions were contacted for their specific analytical requirements for resolution of the safety concern. The following sections identify the additional sampling and analysis objectives for these Public Law 101-510 list tanks.

15.2.1 Single-Shell Tank C-112

Single-shell Tank C-112 has been identified as the primary receiver tank for in-farm ferrocyanide-scavenged waste. Three cores are planned to be collected from this tank. Each core is expected to contain two full segments and a partial third segment. In order to enhance the resolution of the vertical distribution study, each segment will be divided into two 9.5-in. segments. This can aid in identifying the potential for formation of localized layers of ferrocyanide. Limited physical and chemical analyses will be performed before core compositing. The core composites will be treated as described for baseline SSTs. The following measurements will be conducted using the analytical procedures identified in Tables I5-1 and I5-2, unless otherwise specified.

CN ⁻	Total cyanide analysis will be performed on each 9.5-in. subsegment.
TOC	Total organic carbon will be performed using Pacific Northwest Laboratory's method for solids on every subsegment. (Note: NPH contamination may produce false positive results from this method.)
Fusion	The following list of analytes will be measured from a fusion digestion for every subsegment: ¹³⁷ Cs, ⁹⁰ Sr, GEA, Plutonium, Americium, Uranium, ⁹⁹ Tc, total alpha, total beta, and ICP analyses.
Acid	An ICP analysis will be performed from an acid-digestion for every subsegment.
Water	An IC analysis for anions (including NO ₂), pH, and TOC using the 222-S method will be performed from a water-digestion of each 9.5-in. subsegment.
PSA	Particle-size analysis will be conducted for each 9.5-in. subsegment.

Adiabatic subsegment	Adiabatic calorimetry will be performed for every Calorimetry (where an exotherm was observed during the DSC analysis). The procedure for this analysis is in development.
Speciation	X-Ray Diffraction (XRD) and PLM shall be conducted for all subsamples that contain greater than 5,000 $\mu\text{g/g}$ total CN. The procedure for x-ray diffraction at Westinghouse Hanford is LA-507-151 and 152. Polarized Light Microscopy is performed using document no. RHO-RE-ST-28P. Procedures for both XRD and PLM must still be developed at PNL.
Further Analyses	Two chemical analyses have been identified for further studies on homogenized subsegments. These analyses are FeCN speciation and chemical oxygen demand (COD). Neither of these tests are currently performed at the Hanford Site. An archived sample will be taken to perform these tests at a later date when they become available.

In light of the segmental and core-composite level analyses to be performed on this tank, the spatial variability study recommended by PNL (Section II.2.2) can be conducted. In addition, a further comparison of segmental versus core-composite analyses can be evaluated.

15.2.2 Single-Shell Tank C-109

This tank also is a ferrocyanide-receiver tank and will be analyzed in an identical manner as SST C-112.

15.2.3 Single-Shell Tank C-106

This tank has been identified as a high-heat tank because of a quantity of strontium-rich sludge. Currently, the tank is cooled by addition of evaporation water. In order to stabilize this SST, a number of options have been proposed. These options range from retrieving the waste to creating a freeze barrier. To support design criteria for these alternatives, a number of physical parameters have been requested to be measured. The responsible programmatic organizations have identified the physical properties to be of most interest for C-106. Therefore, rheological and physical properties will be measured for every segment for each core. Because a limited retrieval of C-106 is an alternative option, the vertical distribution of ^{90}Sr must be determined. A fusion dissolution for GEA, ^{90}Sr , and ICP metals will be performed for every segment. Two cores are expected to be obtained from this SST. The core composites will be treated the same as baseline-case SST core composites. Some of these parameters can not currently be performed onsite, and others would require funding to develop procedures and techniques to conduct the analyses. The additional requested physical and chemical parameter measurements are summarized in Table I5-3.

Table 15-3. Additional Analyses Required for SST C-106.

Measurement Parameter	Level of Analysis	Collection of Aliquot	PML 325 Procedure	MHC 222-S Procedure	Status
Fusion Dissolution GEA	Every Segment Every Core	Homogenized Segment	See Table 15-1	See Table 15-1	This analyses is typically done for SST characterization.
Fusion Dissolution 90 Sr	Every Segment Every Core	Homogenized Segment	See Table 15-1	See Table 15-1	This analyses is typically done for SST characterization.
Particle Density	Every Segment Every Core	Pre-Homogenized Segment	TBD	TBD	This measurement has been previously performed at Hanford.
Thermal Output	Every Segment Every Core	Pre-Homogenized Segment	TBD	TBD	The Hanford Site does not currently possess the ability to perform the sensitive calorimetry required to accurately measure this parameter. However, this can be calculated based upon heat generation of the radionuclides found in the sample.
Thermal Conductivity	Every Segment Every Core	Pre-Homogenized Segment	TBD	TBD	The apparatus to measure the thermal conductivity is not currently on-site. The equipment should be purchased and a method developed to measure this parameter.
Specific Heat	Every Segment Every Core	Pre-Homogenized Segment	See Table 15-2	See Table 15-1	This parameter can be estimated using the DST procedure typically performed for SST characterization. However, this procedure does not resolve the heat addition due to phase changes, decompositions, and water losses from the rise in temperature.
Freezing Point of Sludge	Every Segment One Core	Pre-Homogenized Segment	TBD	TBD	A procedure for this analyses must be developed prior to measurement of this parameter.
Expansion of Sludge on Freezing	Every Segment One Core	Pre-Homogenized Segment	TBD	TBD	A procedure to measure the small volume increase of a sludge sample upon freezing is not currently available. A procedure and apparatus must be developed prior to analysis.
Thermal Conductivity of Frozen Sludge	Every Segment One Core	Pre-Homogenized Segment	TBD	TBD	The technology to measure the thermal conductivity at a sub-ambient temperature in a hot-cell is not currently available. Research and development must be accomplished prior to initiation of a procedure.

TBD - To Be Determined

15.2.4 Single-Shell Tank T-110

This SST has been identified as a hydrogen gas generator due to a fluctuating liquid level. The safety program has requested some additional analytical measurements to better understand the mechanisms for level fluctuation. Three cores are expected to be obtained from this SST. The requested additions can be measured using the typical procedures identified in Tables I5-1 and I5-2, except where otherwise noted. The additions are summarized below.

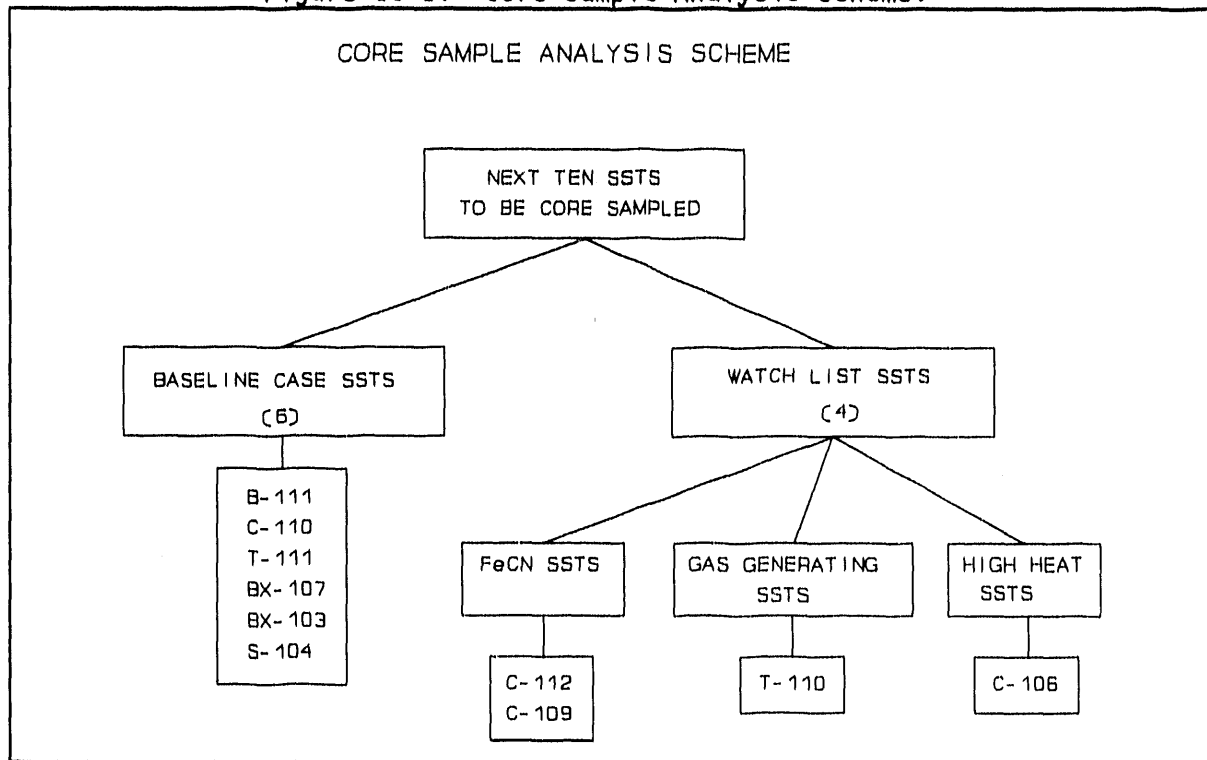
DSC/TGA	Both a DSC and a thermogravimetry should be performed at least twice per segment. An aliquot from each visually discernable facie should be collected for analysis. If no facies are visually obvious, the aliquot should be collected from a location 4.75 in. and 14.25 in. along the length of each segment. These lengths correspond to one-fourth and three-fourths of the length of a segment.
Fusion GEA	An aliquot should be collected and analyzed for GEA using the fusion-dissolution preparation for each homogenized segment sample.
Water Adsorption	The deliquescence or water adsorption properties of the SST waste in this tank should be studied. This measurement might explain the cyclic nature of the liquid levels. This property should be measured on both the liquid and solid-phase core composites. A procedure should be developed to achieve this parameter and an archive sample will be retained if the procedure cannot be in place at the time of analysis.

**16.0 SAMPLING AND ANALYSIS SCHEME
AND TEST PROCEDURES**

The scheme for sampling and analysis of the next 10 SSTs has been divided into 2 sections. Section 16.1 through 16.1.3 will describe the test procedures for baseline-case SSTs expected to be used on typical non-Public Law 101-510 list SSTs. Section 16.2 through 16.2.3.2 will detail the analytical scheme required by the individual safety programs involved in resolution of open safety issues. A flowchart outlining which SSTs shall be sampled under which scenario has been presented as Figure I6-1.

If any new tanks are selected in addition to or to replace the tanks listed in Figure I6-1, then these new tanks shall be sampled and analyzed according to the proper analytical scenario described below. SSTs not identified in Public Law 101-510 shall be analyzed according to the baseline case scenario, SSTs identified in Public Law 101-510 shall be analyzed according to the appropriate scenario outlined for the particular unresolved safety question associated with that tank. The addition or substitution of any new tanks to the selected list shall be properly documented.

Figure I6-1. Core Sample Analysis Scheme.



I6.1 BASELINE--CASE SINGLE--SHELL TANKS

Six of the next ten SSTs to be core sampled have been identified as non-Public Law 101-510 list tanks. The selected non-Public Law 101-510 list tanks are B-111, C-110, T-111, BX-107, BX-103, and S-104.

I6.1.1 Baseline--Case Sample and Analysis Scheme

A flowchart depicting the general sampling and analysis scheme for non-Public Law 101-510 List SSTs is presented in Figure I6-2. The individual steps shown in Figure I6-2 are described in detail as follows:

- Step 1--Tank Farm operations will obtain one core from two or three different risers in each SST listed in Table I3-1 using procedure TO-020-450, "Perform Core Sampling." The number of cores per tank required for characterization also is identified on Table I3-1. One field blank will be taken for each tank by preparing a sampler, as normal, using any necessary sealants but filling it in the field with deionized water from the laboratory.
- Step 2--The decision to ship core samples to laboratory 325 or 222-S, will be made by the Office of Sample Management (OSM) before initiation of the particular sampling event. Core samples will be transported to the laboratories in accordance with procedure TO-080-090, "Ship Core Samples."
- Step 3--Samples will be received, broken down, and extruded at each laboratory using the procedures shown in Table I4-1. The visual observations will be recorded on a SST Extrusion Logsheet. (A copy of this logsheet is presented in Figure I6-3.) The visual observations will include a sketch of the extruded core and such pertinent descriptive information as color, texture, homogeneity, and consistency. The physical parameters identified on the extrusion logsheet will be measured and recorded. The physical parameters listed include:

Drainable liquid

- Volume of liquid in liner (mL)
- Weight of liquid in liner (g)
- Volume of liquid in sampler (mL)
- Weight of liquid in sampler (g)

Bulk solid

- Weight of segment (g)
- Length of segment (in.)
- Length of segment (cm)
- Diameter of segment (cm)
- Volume of segment (cc)
- Bulk density (g/mL)
- Percent recovery
- Penetrometer

Figure I6-2. Baseline Case Single-Shell Tank Sample and Analysis Flow Diagram.

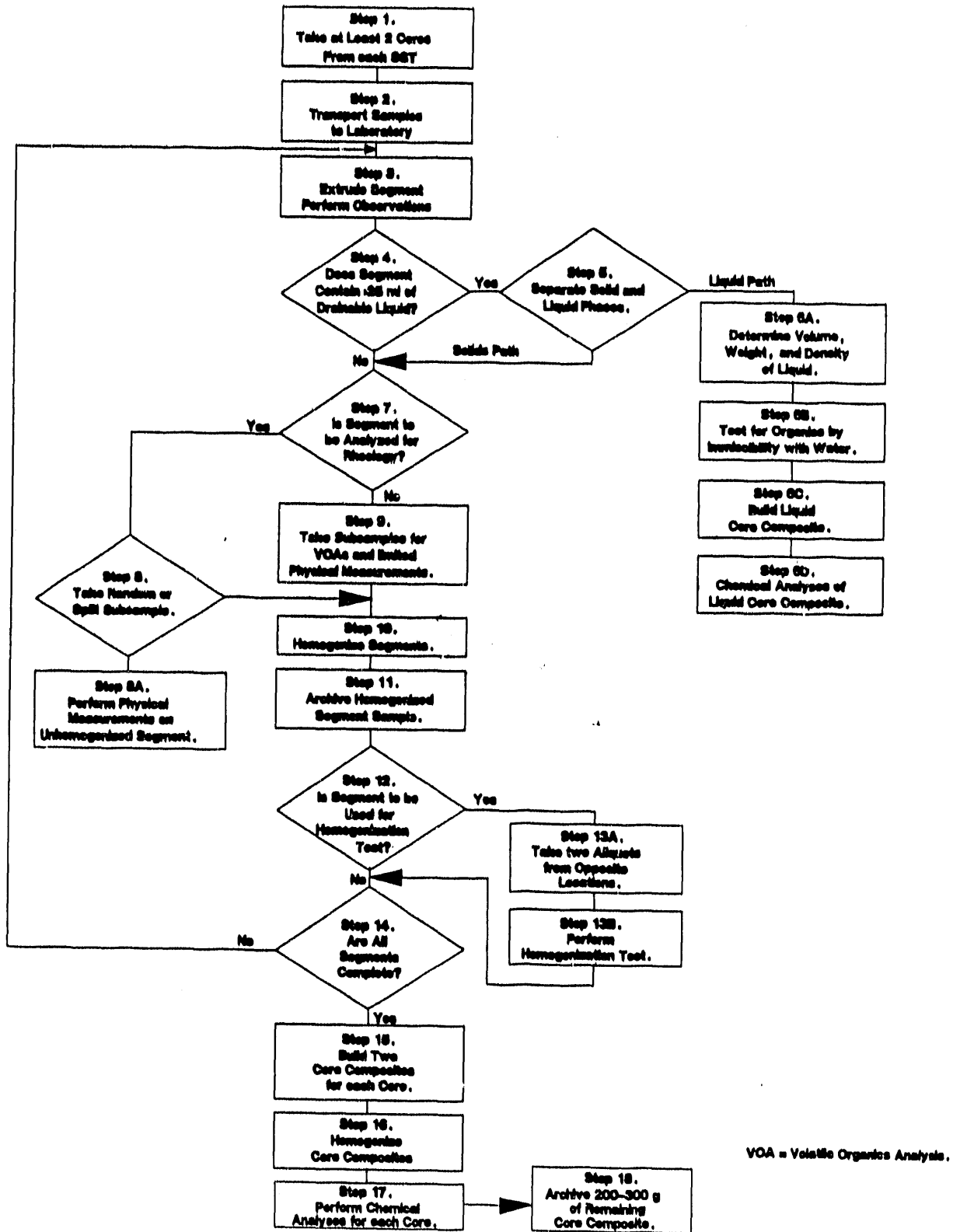


Figure I6-3. Single-Shell Tank Extrusion Logsheet.
Hanford Site
Single-Shell Tank Core Extrusion Logsheet

Tank ID	Date Sampled	Extruded by		
Core No.	Date Extruded			
Seg No.				
DRAINABLE LIQUID		Visual Observations (Color, Texture, Homogeneity, Consistency, and Other)	Segment Sketch	0
Volume of Liquid in Liner (ml)				1
Weight of Liquid in Liner (g)				2
Volume of Liquid in Sampler (ml)				3
Weight of Liquid in Sampler (g)				4
BULK SOLID				5
Weight of Segment (g)				6
Length of Segment (in)				7
Length of Segment (cm)				8
Diameter of Segment (cm)				9
Volume of Segment (cc)				10
Bulk Density (g/ml)				11
% Recovery				12
Penetrometer				13
				14
				15
				16
				17
				18
				19

The bulk density will be obtained by dividing the weight of the segment by the volume of the segment so that:

$$\text{Bulk Density} = \frac{\text{Weight of Segment}}{\text{Volume of Segment}}$$

The percent recovery can be determined by dividing the volume of material actually collected in the sampler by the volume expected from a particular segment and then multiplying by 100.

$$\text{Percent Recovery} = \frac{\text{Liquid Volume} + \text{Solid Volume}}{\text{Expected Volume}} \times 100 \%$$

A color photograph documenting the extruded segment will be taken after completely extruding the entire segment.

- Step 4--If the sample contains more than 25 mL of drainable liquid, the liquid should be analyzed separately from the solids.

If the liquid is <25 mL, then it must be determined whether the limited quantity of liquid is actually NPH will be made. If the small quantity (<25 mL) of liquid is resolved to be NPH, then it should be drained off and analyzed by GC to determine if any organic compound other than NPH is present. If the drained NPH is highly colored then an acid digestion shall be prepared and analyzed for ICP, GEA, and total alpha. The NPH should not be discarded unless until directed by TWCT personnel. If the small quantity of liquid is not NPH, it should be retained with the sample for eventual homogenization. Proceed to Step 7.

If the amount of drainable liquid is greater than 25 mL then proceed to Step 5.

- Step 5--Separate the drainable liquid from the solids by allowing the liquid to drain into a clean, plastic bottle. The liquid may be drained from the extrusion tray or through a coarse, inert (stainless steel, glass, or Teflon) filter that will permit the solids to be recovered without significant losses. The solids are to be retained in the extrusion tray for further subsampling and analysis.
- Step 6A--The weight, volume, and density are determined on the liquid.
- Step 6B--Sometimes NPH from the drilling is trapped in the sampler. GC analysis, immiscibility test, and density measurement are used to

determine if it is NPH. If the liquid is NPH, analyze it by GEA, ICP, and total alpha to evaluate if it is significantly contaminated with waste. Also record its color. If its density indicates it is some other organic save for ignitibility testing.

- Step 6C--Prepare a liquid core composite from the liquids from each segment. If the volume is small (25 to 50 mL) and found in only one or two segments, composite the liquids proportionately with the solid composite and homogenize before subsampling.
- Step 6D--If a liquid core composite is prepared, analyze for the same analytes as the solid core composite as shown in Table I5-1.
- Step 7--Every other segment from the first core obtained from each SST will be used for extensive physical rheologic measurements. If a segment is chosen for rheological examination, then proceed to Step 8; otherwise, continue with Step 9. Incomplete core recovery and other factors may require these segment selections to be changed. These segments are chosen to provide rheology information for waste at different depths in the tank. If incomplete segments are obtained, Section I6.1 should be consulted for guidance on how to use the sample and the change in plan discussed with OSM and Tank Waste Characterization Technology.
- Step 8--While the core is unhomogenized and still in the extrusion tray, either randomly remove ~30 g of sample from every 4 to 5 in. of the segment (enough [120-150 g] to make two core composites and segment archive) for the entire length of the segment or split the sample lengthwise into a portion for rheology and a portion for composites. This should be done in a manner that disturbs the physical nature of the waste as little as possible and fast enough that segments do not dry significantly. The random or one half of the split sample is transferred to a glass jar for homogenization (Step 10).
- Step 8A--The remaining unhomogenized segment material is then subsampled for particle size, rheologic properties, settling velocities, weight percent solids, DSC, and thermal gravimetry according to procedures indicated for each analysis in Table I5-2. VOA shall not be performed on the rheologic segments due to sample size constraints.
- Step 9--If the segment is not used for rheology, take subsamples for volatile organic analysis (VOA) and a limited number of physical tests. The required physical tests are weight percent solids, DSC, and thermal gravimetry. The procedures for these analyses are listed in Table I5-2. Randomly sampled aliquots are collected from the length of the core until about 10 g are obtained for VOA. These should be collected and sealed as soon as possible after extrusion. A similar procedure is used to obtain 1 to 3 g for particle size. Differential scanning calorimetry samples should not be combined. Choose a small (~0.5 g) sample from each distinctive region of the segment. Attempts should be made to run the DSC on different phases based on visual observations with the objective of locating

concentrated areas of potentially exothermic materials. Thermal gravimetric analysis should be performed on these same portions to estimate water content and to support DSC analysis evaluations. Because of problems keeping radioactive VOA samples cooled, these samples shall be analyzed as quickly as possible. If NPH interferences can not be removed, then no VOA will be performed until a sufficient NPH clean-up can be accomplished.

- Step 10--Homogenize the solids from Step 9 or the random/split sample from Step 8 using procedure LT-549-102 at 222-S Laboratory and procedure PNL-ALO-135 at the 325 Laboratory.
- Step 11--Approximately 50 g of each homogenized segment should be archived in a sealed glass jar for future analytical studies. This archival procedure will eventually generate a large quantity of archived samples, which can not be permanently stored in the hot cells. This will require establishing a permanent SST sample archive facility.
- Step 12--Determine if the segment is to be used for a homogenization test. Every other segment from the second (nonrheologic) core will be used for a homogenization test or at least two homogenization tests per tank. If problems homogenizing samples are encountered then the frequency of the homogenization tests should be increased.
- Step 13A--If a homogenization test is to be done, take one 3-to-5-g subsample from opposite locations of the homogenized segment. (i.e., two subsamples).
- Step 13B--Prepare duplicate 1-g aliquots of the subsamples (through the acid digestion) for ICP analysis and GEA using the same procedures identified for acid digestion listed in Table I5-1. Analyze acid-digested samples for ICP metals, GEA and total alpha. Use the same analysis procedures described in Table I5-1.
- Step 14--When all the segments have been either subsampled for core composites (rheology segments) or homogenized, the core composites can be built.
- Step 15--Using portions of the homogenized segments from Step 10, build two core composites for each core. Identify and report all segments and weights used to make the composites. (See Section I6-1 for a discussion of core compositing.)
- Step 16--Homogenize each of the core composites.
- Step 17--Perform duplicate analyses for all the parameters identified in Table I5-1 for each core composite.
- Step 18--Archive 200 to 300 g of remaining segments for analysis verification and performance assessment parameters.

Most of the analytical procedures are the same as were used in Phase IA and IB. The 222-S Laboratory will be implementing a new micro-distillation

system for cyanide. This system uses sulfuric acid, MgSO_4 , and heat to distill off HCN gas through a semi-permeable membrane where it is trapped in a small volume of NaOH. Cyanide is determined by the same calorimetric procedure used before. The distillation tubes are disposable and the heating system can process up to 20 samples at a time.

Pacific Northwest Laboratory will test some new cleanup technology for removing NPH in the volatile and semivolatile organic analyses. The volatile cleanup uses HPLC and removes 99% of the NPH. If the procedure development is completed, this new technique shall be implemented for the next ten SSTs. The semi-volatile cleanup removes about 85% of the NPH and does not require any special equipment. This should improve semi-volatile detection limits and will be evaluated on the next ten tanks.

The old method for EDTA and HEDTA is not usable because the same chromatographic columns are no longer available and a new chromatographic system has not been developed. A sulfide method is still in the process of being developed and will not be available for the analysis of these tanks. Radiochemical methods for ^{63}Ni and $^{93\text{m}}\text{Nb}$ are not developed for the 222-S Laboratory. The 325 Laboratory has a method for ^{63}Ni that must be run on the acid digestion (because of nickel interference from the fusion crucible), but has not been routinely implemented. This is not expected to cause a problem unless highly insoluble nickel species are present. Also, PNL has a potential $^{93\text{m}}\text{Nb}$ method that could be evaluated but is not ready for routine use.

Modified TCLP methods for metals analysis has to be developed at both laboratories and is planned to be implemented for the next ten SSTs. Flashpoint/ignitibility methods have not been developed and are not planned unless liquid organics are found in the tank. Cyanide and sulfide reactivity measurements are not planned since these tanks are not expected to contain significant quantities of cyanide or sulfide.

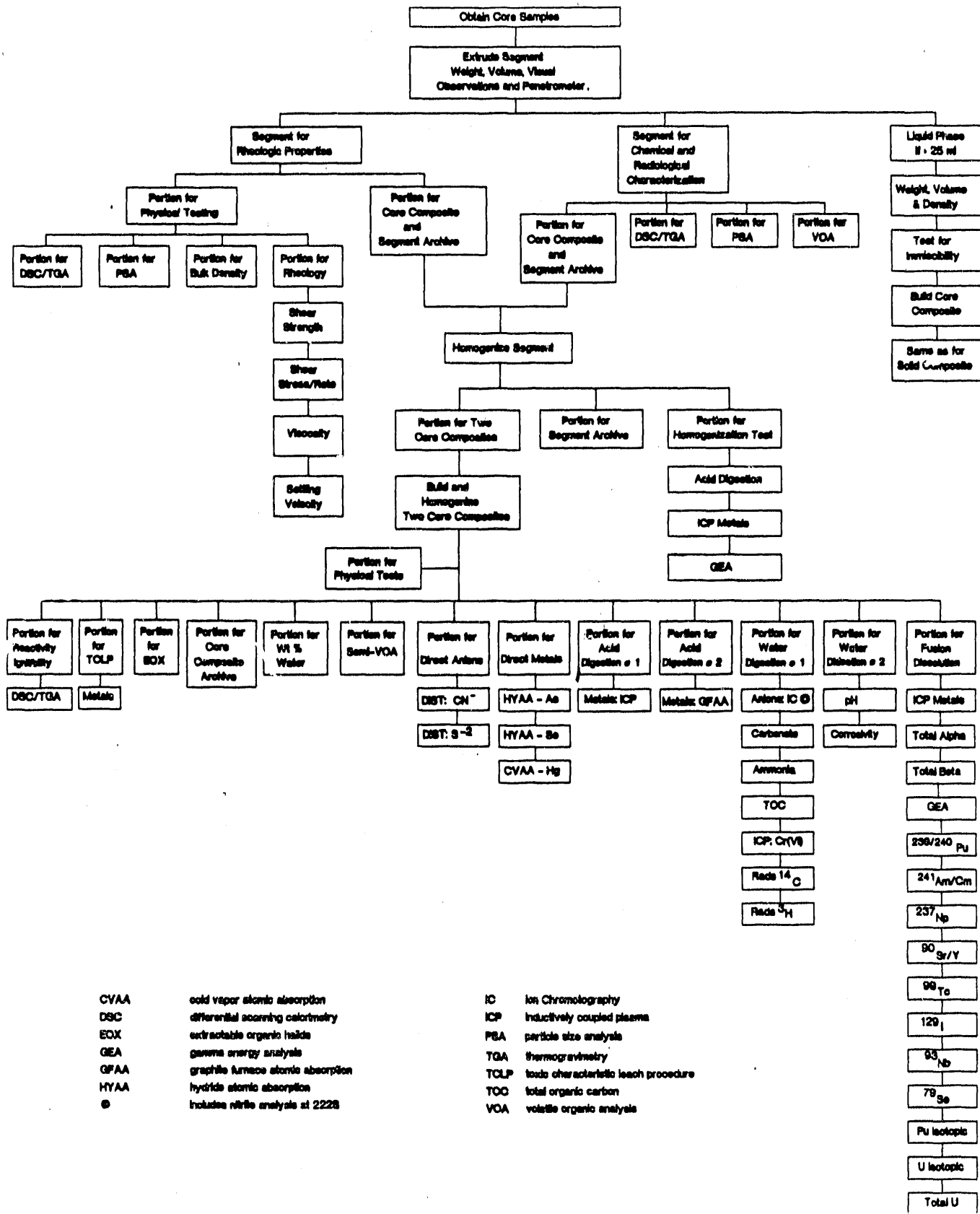
The information in Figure I6-2 and in Tables 5-1 through 5-2 have been combined into Figure I6-4, "Baseline Case SST Sample Allocation." Analytes not planned or that only may be tested for evaluation (VOAs, $^{93\text{m}}\text{Nb}$) are noted in Figure I6-3.

I6.1.2 Core Compositing

In Phase IA and IB, the core composites were built using quantities of segments based on a proportion of the total weight of sample for the core. This assumes that the sample obtained is representative of what is in the tank. However, when partially filled segments are obtained, this procedure assumes that the tank does not contain any waste in this area. Actually, the incomplete recovery for a segment may be the result of sampling problems rather than voids in the waste. If this is true, the composite results could be weighted more heavily to components and concentrations found in full segments.

Another approach is to composite equal quantities of segments and assume that whatever is obtained in a partial segment is representative of the entire segment. Some inaccuracies may be introduced because of density differences between segments but these would probably be insignificant because the density

Figure I6-4. Baseline Case Single-Shell Tank Sample Allocation.



differences are small compared to the other errors in sampling and analysis. If full segments are obtained for the entire core there, will be little difference between the two approaches. Since it seems more likely that the partial segments are the result of sampling problems rather than voids in the waste, this equal quantity-per-segment approach to compositing will be used for the next 10 tanks. The top or first segment from a core is taken so that remaining segments will be full; therefore, it is normally a partial segment and will be composited proportionately. This approach also should help simplify the compositing procedure.

I6.1.3 Baseline-Case Single-Shell Tank Core Sample Utilization

Using the constant quantity approach, the amount of material that can be used to build a core composite will be limited by the amount of segment sample remaining from the segment with the lowest partial recovery. The complexity of the SST characterization program which uses one set of samples to perform tests to satisfy multiple data users, makes it difficult to write a definitive test plan when incomplete samples are recovered. Tables I6-1 through I6-5 look at sample utilization, the impact of partial recovery, and sample quantity requirements for different options. These tables are intended to help develop analysis strategies when partial segments are received.

Table I6-1 estimates the amount of prehomogenized segment sample used and remaining for cores in which (1) rheology samples are taken and (2) no rheology is performed. A maximum and minimum quantity of sample needed is estimated. The maximum is based on large sample sizes, full quality control, and sufficient sample for reruns. The minimum is based on smaller samples, and reduced quality control and rerun requirements. The basis for the numbers is provided. For example, 5S + 5D + 10MSD + 10RR means 5 g for sample + 5 g for duplicate + 10 g for matrix spike duplicate + 10 g for reruns. Obviously, cores used for rheology require the most sample. For the cores without rheology, the VOA consumes the most samples. Roughly 56 g to 141 g of the original segment sample will remain for core compositing from a rheology core segment, providing 100% core recovery. On the other hand, 95 g to 184 g would remain from a nonrheology core segment with 100% core recovery.

Table I6-2 estimates the amount of segment sample that needs to be archived. Since the analysis of semi-volatile organics and TCLP testing will probably not be required on segments, archive samples of 15 to 30 g should be adequate for most chemical and radiochemical tests.

Table I6-3 estimates the amount of sample needed for completing the analysis on a single core composite. Since two core composites must be made for each core, the values required for compositing (listed as the first subtotal) must be doubled. The remaining analyses and archives only require one quantity per core.

Table I6-4 estimates the volume of water-digested sample needed to complete the analysis. This estimate indicates that the digestion procedure should be changed from 1 g in 100 mL of water to 2 g in 200 mL of water. This allows larger sample sizes and better detection limits.

Table I6-1. Prehomogenized Sample Utilization.

Task Performed	Rheology Core			No rheology Core			Basis
	Maximum Used (g)	Minimum Used (g)	Balance Range (g)	Maximum Used (g)	Minimum Used (g)	Balance Range (g)	
Extrude Segment	0	0	234	0	0	234	180mL x 1.3 g/mL
Portion for DSC	3	1	231-233	3	1	231-233	3 phases x 1g
Portion for Particle Size	1	1	230-232	1	1	230-232	Random 1g
Portion for Bulk Density	0	0	230-232	0	0	230-232	Calculations for weight and volume
Portion for Rheology	120	65	110-167	0	0	230-232	90mL and 50mL samples
Portion for VOA	0	0	110-167	30	12	200-220	5S + 5D + 10MSD + 10RR 2S + 2D + 4SD + 4RR
Transfer Loss	24	12	86-155	24	12	176-208	5% = 12g, 10% = 24g
Portion for Homog. Test	0	0	86-155	6	4	170-204	2S + 2D + 2RR 2S + 2D
Portion for Seg. Archive	30	14	56-141	75	20	95-184	No TCLP, Semi-VOA or EOX on Rheology
Amount Remaining for Core Composites	-	-	56-141	-	-	95-184	
Total g Used	178	93		139	50		

S = Sample
D = Duplicate
MSD = Matrix Spike Duplicate
RR = Rerun
Seg. = Segment
Homog. = Homogenization

Table I6-2. Estimate of Segment Archive Sample Utilization.

Analysis	Segment Maximum	Amount Minimum	Basis
ICP/Acid	6	2	1S + 1D + 2MSD + 2RR 1S + 1D
Anions/Water	4	2	1S + 1D + 2RR 1S + 1D
Rads/Fusion	1	1	0.25S + .25D + 0.5RR
pH/Corrosivity	5	5	2.5S + 2.5D
GFAA/Acid	6	2	Same as ICP
CVAA/Hg	2	1	0.2S + 0.2D + 0.4MSD + 0.4RR 0.2S + 0.2D
Cyanide	6	1	1.0S + 1.0D + 2.0MSD + 2.0RR (Large Dist.) 0.2S + 0.2D + 0.4MSD (Micro Dist.)
Subtotal	30	14	
Semi-VOA	30	6	5.0S + 5.0D + 10MSD + 10RR 2.0S + 2.0D + 2MSD
TCLP	10	0	10S
EOX	5	0	5S
Total	75g	20g	

Table I6-4. Water Digestion Sample Utilization.

Analysis	Water Maximum mL	Water Minimum mL	Basis (Maximum, mL) (Minimum, mL)
IC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.2RR
Carbonate	1	1	0.2S + 0.2D + 0.2PS + 0.2RR 0.2S + 0.2D
Ammonia	15	6	5S + 5D + 5RR 2S + 2D + 2RR
TOC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.25RR
ICP	80	50	20S + 20D + 20PS + 20RR 10S + 10D + 20PS + 10RR
C-14	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
H-3	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
Total	192 mL	109 mL	

PS = post adjustment spike

Table I6-5A. Baseline Case % Recovery Study Rheology Core.

% Recovery	Max/Min 234x _{f_R} -178 234x _{f_R} -93	Max 2	Min 2	Max 3	Min 3	Max 5	Min 5	Max 7	Min 7
100	56 141	112	282	168	423	280	705	392	987
90	33 118	66	236	99	354	165	509	231	826
80	9 94	18	188	27	282	45	470	63	658
70	0 71	0	142	0	213	0	354	0	497
60	0 47	0	94	0	141	0	234	0	329
50	0 24	0	48	0	72	0	234	0	329
40	0 1	0	2	0	3	0	5	0	7
30	0 0	0	0	0	0	0	0	0	0
20	0 0	0	0	0	0	0	0	0	0

Table 16-5B. Baseline Case % Recovery Study
Non-Rheology Core.

% Recovery	Max/Min 234xf _R -139 234xf _R -50	Max 2	Min 2	Max 3	Min 3	Max 5	Min 5	Max 7	Min 7
100	95 184	190	368	285	552	475	920	665	1288
90	72 161	144	322	216	483	360	305	504	1127
80	48 137	96	274	144	411	240	685	336	959
70	25 114	50	288	75	342	125	570	175	798
60	1 90	2	180	3	270	5	450	7	630
50	0 67	0	134	0	201	0	335	0	469
40	0 44	0	88	0	132	0	220	0	308
30	0 20	0	40	0	60	0	100	0	140
20	0 0	0	0	0	0	0	0	0	0

Tables 6-5A and 5B respectively estimate the amount of sample remaining in each segment for compositing samples with different percent recoveries for rheologic cores and nonrheologic cores. The total quantities that could be composited are estimated for 2-segment, 3-segment, 5-segment, and 7 segment cores with minimum and maximum posthomogenization sample sizes. As can be clearly seen, the amount of available sample is heavily dependant upon the percent recovery. Most of the SSTs selected are predicted to contain soft waste and should be able to be sampled with high recoveries. The amount of available sample also is sensitive to the number of segments per core. Some of the selected tanks do not contain vast quantities of waste and the waste heights will provide only two to three segments. For these tanks, minimum sample sizes will be required to be used to enable the full spectrum of characterization activities to be performed.

If during the process of sampling, extrusion, and analysis, it becomes apparent that there will be insufficient samples to perform all of the tests designated in this, then sample sizes must be reduced and/or some analyses and archives eliminated. These decisions must be made on a case-by-case basis by Tank Waste Characterization Technology in conjunction with the OSM.

The evaluation of sample utilization indicates that for cores that require rheology, it would be best to select a segment with >80% recovery for the rheology test, which permits the largest core composite to be constructed. If the recovery for a segment becomes too low, it may be necessary to choose a segment with higher recovery for the basis of the core composite, even though the composite may not be weighted properly for the low-recovery segments. This is always the case for a core that contains segments from which no sample is recovered.

For segments that are expected to be only partially full, such as the first segment of each core, it should be composited in proportion to the amount that was expected. If incomplete segments cause a change in the plan, the changes should be discussed and approved by OSM and Tank Waste Characterization Technology. All changes in the plan will be documented in the data packages and subsequent reports.

16.2 SINGLE-SHELL TANKS IDENTIFIED IN PUBLIC LAW 101-510

A separate flowsheet depicting the specific sampling and analysis scheme for each type of tank identified in Public Law 101-510 has been generated. They are presented in detail in the following sections.

16.2.1 Ferrocyanide Tanks (C-112 and C-109)

Two ferrocyanide tanks are expected to be core sampled and analyzed during the next ten tanks. Three cores are expected to be obtained from each of these two tanks. The 19 in. segments shall be divided into 9 1/2 in. subsegments to enhance the resolution of the vertical distribution of such key components as FeCN and radionuclides.

I6.2.1.1 Sample and Analysis Scheme for Ferrocyanide Tanks (C-112 and C-109). The flowsheet for FeCN SSTs C-112 and C-109 is presented in Figure I6-5. Each individual step on the flowsheet has been described in detail below.

- Step 1--Tank Farm operations will obtain one core from three different risers in each SST (C-112 and C-109) using procedure TO-020-450, "Perform Core Sampling." One field blank will be taken for each tank by preparing a sampler, as normal, using any necessary sealants but filling it in the field with deionized water from the laboratory.
- Step 2--The decision to ship core samples to laboratory 325 or 222-S, will be made by the OSM before initiation of the particular sampling event. Core samples will be transported to the laboratories in accordance with procedure TO-080-090, "Ship Core Samples."
- Step 3--Samples will be received, broken down, and extruded at each laboratory using the procedures shown in Table I4-1. The visual observations will be recorded on a SST Extrusion Logsheet. (A copy of this logsheet is presented in Figure I6-2.) The visual observations will include a sketch of the extruded core and such pertinent descriptive information as color, texture, homogeneity, and consistency. The physical parameters identified on the extrusion logsheet will be measured and recorded. The physical parameters listed include:

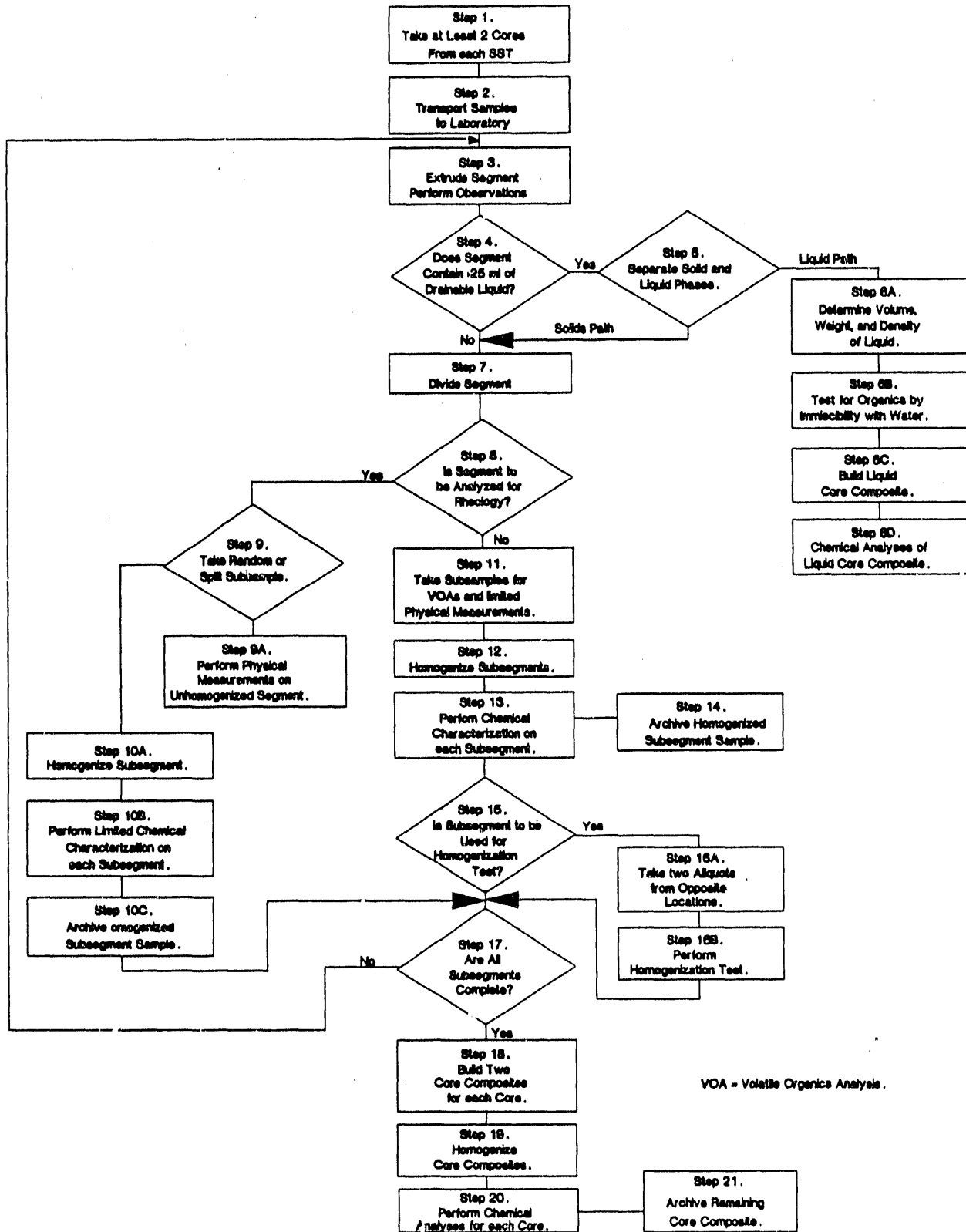
Drainable liquid

- Volume of liquid in liner (mL)
- Weight of liquid in liner (g)
- Volume of liquid in sampler (mL)
- Weight of liquid in sampler (g)

Bulk solid

- Weight of segment (g)
- Length of segment (in.)
- Length of segment (cm)
- Diameter of segment (cm)
- Volume of segment (cc)
- Bulk density (g/mL)
- Percent recovery
- Penetrometer

Figure I6-5. Sample and Analysis Flow Diagram for Single-Shell Tanks C-112 and C-109.



The bulk density will be obtained by dividing the weight of the segment by the volume of the segment so that:

$$\text{Bulk Density} = \frac{\text{Weight of Segment}}{\text{Volume of Segment}}$$

The percent recovery can be determined by dividing the volume of material actually collected in the sampler by the volume expected from a particular segment and then multiplying by 100.

$$\text{Percent Recovery} = \frac{\text{Liquid Volume} + \text{Solid Volume}}{\text{Expected Volume}} \times 100\%$$

A color photograph documenting the extruded segment shall also be taken after completely extruding the entire segment.

- Step 4--If the sample contains more than 25 mL of drainable liquid, the liquid should be analyzed separately from the solids.

If the liquid is <25 mL, then it must be determined whether the limited quantity of liquid is actually NPH will be made. If the small quantity (<25 mL) of liquid is resolved to be NPH, then it should be drained off and analyzed by GC to determine if any organic compound other than NPH is present. If the drained NPH is highly colored then an acid digestion shall be prepared and analyzed for ICP, GEA, and total alpha. The NPH should not be discarded unless until directed by TWCT personnel. If the small quantity of liquid is not NPH, it should be retained with the sample for eventual homogenization. Proceed to Step 7.

If the amount of drainable liquid is greater than 25 mL then proceed to Step 5.

- Step 5--Separate the drainable liquid from the solids by allowing the liquid to drain into a clean, plastic bottle. The liquid may be drained from the extrusion tray or through a coarse, inert (stainless steel, glass, or Teflon) filter that will permit the solids to be recovered without significant losses. The solids are to be retained in the extrusion tray for further subsampling and analysis.
- Step 6A--The weight, volume, and density are determined on the liquid.
- Step 6B--Sometimes NPH from the drilling is trapped in the sampler. GC analysis, immiscibility test, and density measurement are used to determine if it is NPH. If the liquid is NPH, analyze it by GEA, ICP, and total alpha to evaluate if it is significantly contaminated with waste. Also, record its color. If its density indicates it is some other organic save for ignitibility testing.

- Step 6C--Prepare a liquid core composite from the liquids from each segment. If the volume is small (25 to 50 mL) and found in only one or two segments, composite the liquids proportionately with the solid composite and homogenize before subsampling.
- Step 6D--If a liquid core composite is prepared, analyze for the same analytes as the solid core composite as shown in Table I5-1.
- Step 7--Each 19-in. segment will be divided into two 9.5-in. subsegments. The two subsegments should remain unhomogenized in the sample tray. The upper subsegment will be labeled with the suffix "A" and the lower with the suffix "B." The naming convention for the root segment name will remain the same.
- Step 8--Every other segment from the first core obtained from each SST will be used for extensive physical and rheologic measurements. If a segment is chosen for rheological examination, then proceed to Step 9 otherwise, continue with Step 11. Incomplete core recovery and other factors may require these segment selections to be changed. These segments are chosen to provide rheology information for waste at different depths in the tank. If incomplete segments are obtained, Section I6.1 should be consulted for guidance on how to use the sample and the change in plan discussed with OSM and Tank Waste Characterization Technology.
- Step 9--While the subsegments are unhomogenized and still in the extrusion tray, either randomly remove ~20 g of sample from every 2 to 3 in. of the segment (enough [60-80 g] to make two core composites, a subsegment archive, and for limited chemical analysis on the subsegment) for the entire length of the subsegment or split the sample lengthwise into a portion for rheology and a portion for composites. This should be done in a manner that disturbs the physical nature of the waste as little as possible and fast enough that subsegments do not dry significantly. The random or split sample is transferred to a glass jar for homogenization (Step 10).
- Step 9A--The remaining unhomogenized subsegments are subsampled for particle size, DSC, and thermal gravimetry on a subsegmental level. The remaining sample material, after all subsampling, shall be used for measurement of rheologic properties, settling velocities, and weight percent solids on a segmental basis. All physical measurements will be made according to procedures indicated for each analysis in Table I5-2.
- Step 10A--The random or split subsegment sample obtained in Step 9 will be thoroughly homogenized using procedure T046 at the 222-S laboratory and procedure PNL-ALO-135 at the 325 Laboratory.

- Step 10B--Due to the significant sample volume requirements for the physical and rheologic measurements, only a limited amount of chemical characterization can be performed on the subsegment samples from the rheologic segments. An aliquot will be taken from the homogenized subsegment to perform each of the following preparations and analyses in duplicate:

<u>Preparation</u>	<u>Analyses</u>
Fusion dissolution	GEA ⁹⁰ Sr ICP
Water leach	Anions
Direct	CN ⁻ TOC Wt % H ₂ O

The procedures for each of these preparations and analyses can be found in Table I5-1.

- Step 10C--Approximately 15 g of each subsegment should be archived in a sealed, smoked-glass jar for future analysis.
- Step 11--If the segment is not used for rheology, take subsamples for VOA and a limited number of physical tests. The required physical tests are weight percent solids, DSC, and thermal gravimetry. The procedures for these analyses are listed in Table I5-2. Randomly sampled aliquots are collected from the length of the core until about 10 g are obtained for VOA. These should be collected and sealed as soon as possible after extrusion. A similar procedure is used to obtain 1 to 3 g for particle size. Differential scanning calorimetry samples should not be combined. Choose a small (~0.5 g) sample from each distinctive region of the segment. Attempts should be made to run the DSC on different phases based on visual observations with the objective of locating concentrated areas of potentially exothermic materials. Thermal gravimetric analysis should be performed on these same portions to estimate water content and to support DSC analysis evaluations. Because of problems keeping radioactive VOA samples cooled, these samples will be analyzed as quickly as possible.
- Step 12--Homogenize the subsegment sample material remaining from Step 11 using procedure T046 at the 222-S Laboratory and procedure PNL-ALO-135 at the 325 Laboratory.

- Step 13--A sufficient aliquot will be collected from each of the homogenized subsegment samples to perform the following preparations and analyses in duplicate:

<u>Preparation</u>	<u>Analyses</u>
Fusion dissolution	GEA ⁹⁰ Sr Plutonium Americium Uranium ⁹⁹ Tc Total Alpha Total Beta ICP
Acid digestion	ICP
Water digestion	IC pH TOC CN ⁻
Water digestion Residual solids	XRD (if CN ⁻ > 5,000 µg/g)
Direct	CN ⁻ TOC PLM Wt % H ₂ O Adiabatic Calorimetry (if DSC observes exotherm)

The procedures for each of these preparations and analyses, except where otherwise noted, can be found in Table I5-1. The procedure for x-ray diffraction at Westinghouse Hanford is LA-507-151 and 152. Polarized light microscopy is performed using procedures document no. RHO-RE-ST-28P at the 222S lab. Procedures for both XRD and PLM must be developed at PNL.

- Step 14--Approximately 25 g of each homogenized subsegment should be archived for future analytical studies in a sealed glass jar.
- Step 15--Determine if the subsegment is to be used for a homogenization test. Every fourth subsegment from the second (nonrheologic) core will be used for a homogenization test. If problems homogenizing the samples is encountered then the frequency of the homogenization test should be increased.

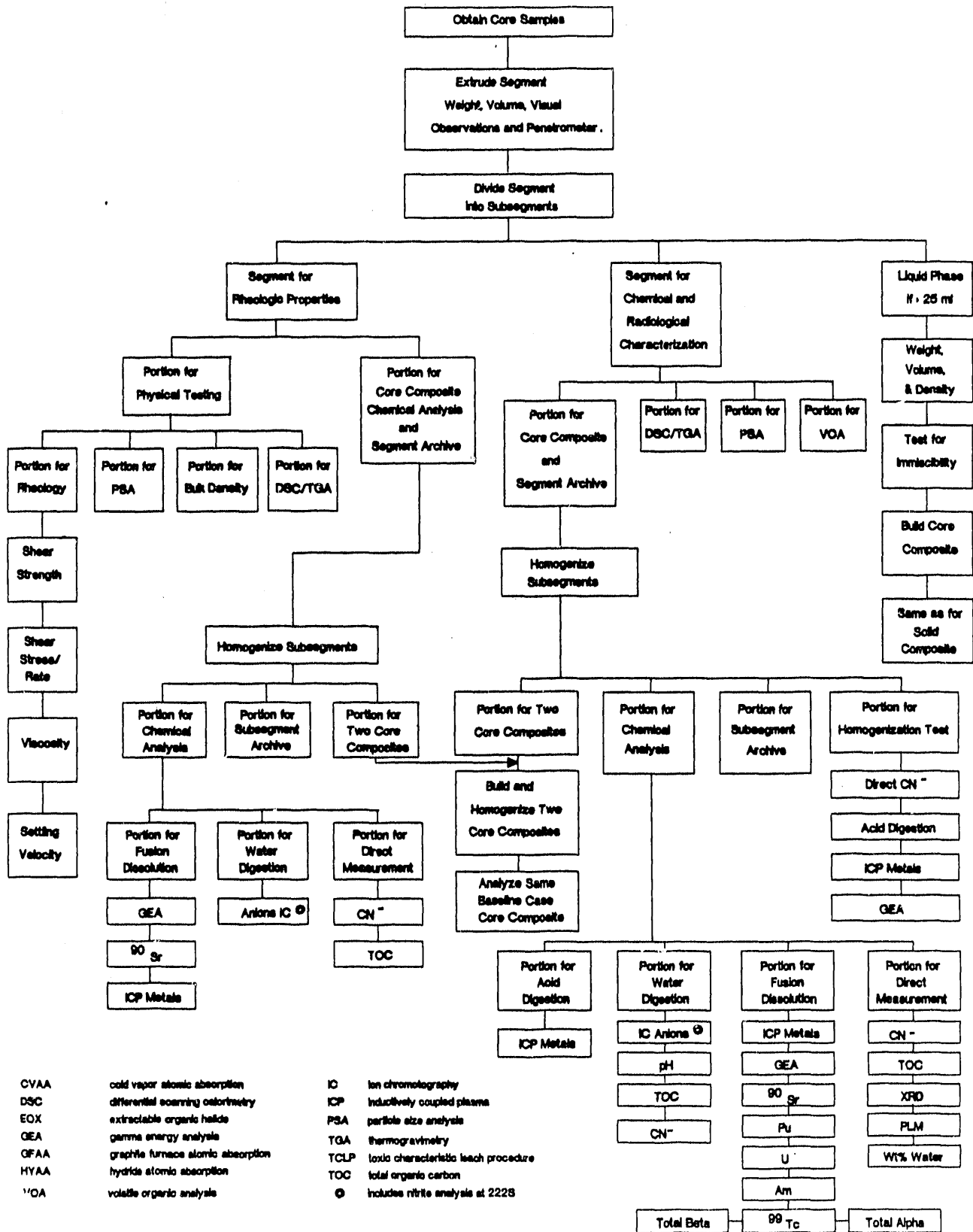
- Step 16A--If a homogenization test is to be done, take one 3-to-5-g subsample from opposite locations of the homogenized subsegment (two subsamples).
- Step 16B--Prepare duplicate 1-g aliquots of the subsamples (through acid digestion) analyses for ICP, GEA, and total alpha using the same procedures identified for acid digestion listed in Table I5-1. Since the distribution of CN is a primary objective of the sampling and analysis of these two tanks, measurement of CN using the same analysis procedures described in Table I5-1 shall be conducted for the homogenization test.
- Step 17--When all the subsegments have been either subsampled for core composites (rheology segments) or homogenized, the core composites can be built.
- Step 18--Using portions of the homogenized subsegments from either Step 10C, Step 15, or Step 16B, build two core composites for each core. Identify and report all subsegments and weights used to make the composites. (See Section I6.1.2 for a discussion of core compositing.)
- Step 19--Homogenize each of the core composites.
- Step 20--Perform duplicate analyses for all the parameters identified in Table I5-1 for each core composite. In addition, perform CN reactivity analysis on each core composite.
- Step 21--Archive 200 to 300 g of remaining segments for analysis verification and performance assessment parameters.

Figure I6-6 depicts the sample allocation for analysis of SSTs C-112 and C-109.

I6.2.1.2 FeCN Core Sample Utilization. Tables 6-6 through 6-11 look at sample utilization, the impact of partial recovery, and sample quantity requirements for FeCN SSTs C-112 and C-109. These tables are intended to help develop analysis strategies when partial segments are received.

Table I6-6 estimates the amount of prehomogenized subsegment sample used and remaining for cores in which (1) rheology samples are taken and (2) no rheology is performed. A maximum and minimum quantity of sample needed is estimated. The maximum is based on large sample sizes, full quality control, and sufficient sample for reruns. The minimum is based on smaller samples, and reduced quality control and rerun requirements. The basis for the numbers is provided. For example, 5S + 5D + 10MSD + 10RR means 5 g for sample + 5 g for duplicate + 10 g for matrix spike duplicate + 10 g for reruns. The large sample required for rheologic analyses is off-set by the VOA and the more extensive chemical analyses performed on nonrheologic cores. Due to the limited amount of samples contained in a 9.5-in. subsegment, maximum sample sizes and Quality Assurance (QA) will totally consume the subsegment. Therefore, minimum sample sizes and QA will be required for these two FeCN tanks. Approximately 40 g and 35 g of subsegment sample will remain from a

Figure I6-6. Sample Allocation for Single-Shell Tanks C-112 and C-109.



rheologic and nonrheologic core, respectively. If VOA is not analyzed on the nonrheologic cores, then 92 g of subsegment sample will remain for core compositing.

Table I6-7 estimates the amount of homogenized subsample that will be required to perform limited chemical analyses. Fewer chemical analyses are performed on rheologic cores due to the larger sample requirements of the physical testing.

Table I6-8 estimates the amount of subsegment sample that needs to be archived. Because a limited chemical characterization will be performed, no subsegment sample shall be archived for chemical analyses. Only those chemical tests that are not currently available or are dependant upon the result of a previous test will be archived. Aliquots for a chemical speciation for FeCN and a COD test will be archived only for nonrheologic cores. The adiabatic calorimetry test will be performed only if the DSC analysis indicates the presence of an exotherm.

Table I6-9 estimates the amount of sample needed for completing the analysis on a single core composite. Since two core composites must be made for each core, the values required for compositing (listed as the first subtotal) must be doubled. The remaining analyses and archives only require one quantity per core.

Table I6-10 estimates the volume of water-digested sample needed to complete the analysis. This estimate indicates that the digestion procedure should be changed from 1 g in 100 mL of water to 2 g in 200 mL of water. This allows larger sample sizes and better detection limits.

Table I6-11 estimates the amount of sample remaining in each segment for compositing samples with different percent recoveries for rheologic cores and nonrheologic cores. The total quantities that could be composited are estimated for four and five subsegments per core using only minimum posthomogenization sample sizes. Estimates of sample availability for nonrheologic cores without VOA also has been made. As can be clearly seen, the amount of available sample is heavily dependant upon the percent recovery. These two SSTs are predicted to contain soft waste and should be able to be sampled with high recoveries. The Performance Assessment (PA) archive sample will not be attained from these two tanks due to the limited amount of sample. If under a 90% core recovery is achieved, then an insufficient amount of sample will be available to perform a full core composite characterization on two composites per core. In this circumstance, only one core composite per core will be built to enable a full characterization to be performed on that single core. This decision will be made by Tank Waste Characterization Technology in conjunction with the OSM.

Table I6-6. Prehomogenized Sample Utilization (C-112 and C-109).

Task Performed	Rheology Core			No rheology Core			Basis
	Max (g)	Min (g)	Balance (g)	Max (g)	Min (g)	Balance (g)	
Extrude Segment	0	0	234	0	0	234	180mL x 1.3 g/mL
Divide Segment into Subsegments	0	0	117	0	0	117	234 + 2
Portion for DSC	3	2	114-115	3	2	114-115	3 phases x 1g (Minimum of 2 per Subsegment)
Portion for Particle Size	1	1	113-114	1	1	113-114	Random 1g
Portion for Bulk Density	0	0	113-114	0	0	113-114	Calculations for weight and volume
Portion for Rheology	60	33	53-81	0	0	113-114	½ (90mL and 50mL samples)
Portion for VOA	0	0	53-81	30	12	83-102	5S + 5D + 10MSD + 10RR 2S + 2D + 4SD + 4RR
Transfer Loss	12	6	41-75	12	6	71-96	5% = 6g, 10% = 12g
Portion for Chemical Analysis	19	12	22-63	35	25	36-71	See Table I6-7
Portion for Homog. Test	0	0	22-63	6	4	30-67	2S + 2D + 2RR 2S + 2D
Portion for Subseg Archive	15	15	7-48	27	19	3-48	See Table I6-8.
Amount Remaining for Core Composites	-	-	7-48	-	-	3-48	
Total g Used	110	69		114	69		

Table I6-7. Estimate of Subsegment Sample Utilization (C-112 and C-109).

Analysis Performed	Rheology Core		No rheology Core		Max Basis Min Basis
	Maximum Used (g)	Minimum Used (g)	Maximum Used (g)	Minimum Used (g)	
Acid Digestion	0	0	6	4	1S + 1D + 2 MSD + 2RR 1S + 1D + 1 MSD + 1RR
Water Digestion IC	8	6	8	6	2S + 2D + 4RR (IC) 2S + 2D + 2RR (IC)
pH, TOC	0	0	10	8	2.5S + 2.5D + 5.CRR (pH) 2.5S + 2.5D + 2.5RR (pH)
Fusion Dissolution (ICP, Rads)	1	1	1	1	0.25S + 0.25D + 0.5RR 0.25S + 0.25D + 0.5RR
Direct CN ⁻ Macro	6	4	6	4	1S + 1D + 2MSD + 2 RR
S ⁻² Micro	2	1	2	1	0.2S + 0.2D + 0.4MSD + 0.4RR
	2	1	2	1	Same as Micro CN ⁻
Total Subsegment	19	12	35	25	

Table I6-8. Estimate of Subsegment Archive Sample Utilization (C-112 and C-109).

Analysis	Segment Maximum	Amount Minimum	Basis
Adiabatic Calorimetry	15	15	
FeCN Speciation	6	2	1.0S + 1.0D + 2MSD + 2RR 0.5S + 0.5D + 1MSD
COD	6	2	1.0S + 1.0D + 2MSD + 2RR 0.5S + 0.5D + 1MSD
Total	51g	27g	

Table I6-9. Estimate of Core Composite Sample Utilization (C-112 and C-109).

Analysis	CC Max. (g)	CC Min. (g)	Basis (Max) Basis (Min)
Acid Digestion # 1 (ICP)	6	4	1S + 1D + 2MSD + 2RR 1S + 1D + 1MS + 1RR
Acid Digestion # 2 (GFAA)	6	4	Same as ICP
Direct As, Se (HYAA)	3	2	(0.25S + 0.25D + 0.5MSD + 0.5RR) (2)
CVAAs (Hg)	2	1	.2S + .2D + .4MSD + .4RR
Water Digestion # 1 (IC, NH ₃ , TOC, ICP, Ra's)	8	6	2S + 2D + 4RR 2S + 2D + 2RR
Water Digestion # 2 (pH, Corrosivity)	10	8	2.5S + 2.5D + 5.0RR 2.5S + 2.5D + 2.5RR
Fusion Portion (Rads, ICP)	1	1	0.25S + 0.25D + 0.5RR 0.25S + 0.25D + 0.25RR
Direct Anions CN ⁻ Portion (Macro) (Micro) S ⁻² Portion	6 2 2	4 1 1	1S + 1D + 2MSD + 2RR 0.2S + 0.2D + 0.4MSD + 0.4RR Same as Micro CN ⁻
Wt% H ₂ O	4	3	1S + 1D + 2RR 1S + 1D + 1RR
DSC/TGA	1	1	0.1S + 0.1D + 0.2RR (a)
Subtotal x 2	51 102	36 72	
Semi-VOA	50	20	5S + 5D + 10MSD + 10RR 5S + 5S + 5MS + 5RR
EOX	12	8	2S + 2D + 4MSD + 4RR 2S + 2D + 2MS + 2RR
Subtotal	164	100	
TCLP	20	10	10S + 10D or RR 10S
Subtotal	184	110	
Analysis Archive	75	20	Same as Basecase Archive
Subtotal	259	130	
PA Archive	150	100	Performance Assessment Tests (Duplicates)
Total	409	230	

CC = Core Composite
(a) = Lowest estimate is rounded to 1 g

Table I6-10. Water Digestion Sample Utilization.

Analysis	Water Maximum mL	Water Minimum mL	Basis (Maximum, mL) (Minimum, mL)
IC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.2RR
Carbonate	1	1	0.2S + 0.2D + 0.2PS + 0.2RR 0.2S + 0.2D
Ammonia	15	6	5S + 5D + 5RR 2S + 2D + 2RR
TOC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.25RR
ICP	80	50	20S + 20D + 20PS + 20RR 10S + 10D + 20PS + 10RR
C-14	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
H-3	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
Total	192 mL	109 mL	

PS = post adjustment spike

Table I6-11. % Recovery Sensitivity Study (C-112 and C-109).

% Recovery	Min		Rheology		Min		No Rheology			
	$117 \times f_R-69$		4 Min	5 Min	$117 \times f_R-69$ $117 \times f_R-57^{(a)}$		4 Min	4 Min (a)	5 Min	
100	48		192	240	48 60		192	240	240	300
90	36.3		145	182	36.3 48.3		145	193	182	242
80	24.6		98	123	24.6 36.6		98	146	123	183
70	12.9		52	65	12.9 24.5		52	98	65	123
60	1.2		5	6	1.2 13.2		5	53	6	66
50	0		0	0	0		0	0	0	0
40	0		0	0	0		0	0	0	0
30	0		0	0	0		0	0	0	0
20	0		0	0	0		0	0	0	0

(a) No VOA Analyses

I6.2.2 HIGH HEAT SINGLE-SHELL TANKS (C-106)

One high heat SST, C-106, is expected to be sampled during the next ten SSTs. Two cores are planned to be obtained from this tank.

I6.2.2.1 Sample and Analysis Scheme for Single-Shell Tank C-106. A flowchart depicting the general sampling and analysis scheme for SST C-106 is presented in Figure I6-7. The individual steps shown on Figure I6-7 are described in detail as follows:

- Step 1--Tank Farm operations will obtain one core from two different risers in SST C-106 using procedure T0-020-450, "Perform Core Sampling." One field blank will be taken by preparing a sampler, as normal, using any necessary sealants but filling it in the field with deionized water from the laboratory.
- Step 2--The decision to ship core samples to laboratory 325 or 222-S, will be made by the OSM before initiation of the particular sampling event. Core samples will be transported to the laboratories in accordance with procedure T0-080-090, "Ship Core Samples."
- Step 3--Samples will be received, broken down, and extruded at each laboratory using the procedures shown in Table I4-1. The visual observations will be recorded on a SST Extrusion Logsheet. (A copy of this logsheet is presented in Figure I6-2.) The visual observations will include a sketch of the extruded core and such pertinent descriptive information as color, texture, homogeneity, and consistency. The physical parameters identified on the extrusion logsheet will be measured and recorded. The physical parameters listed include:

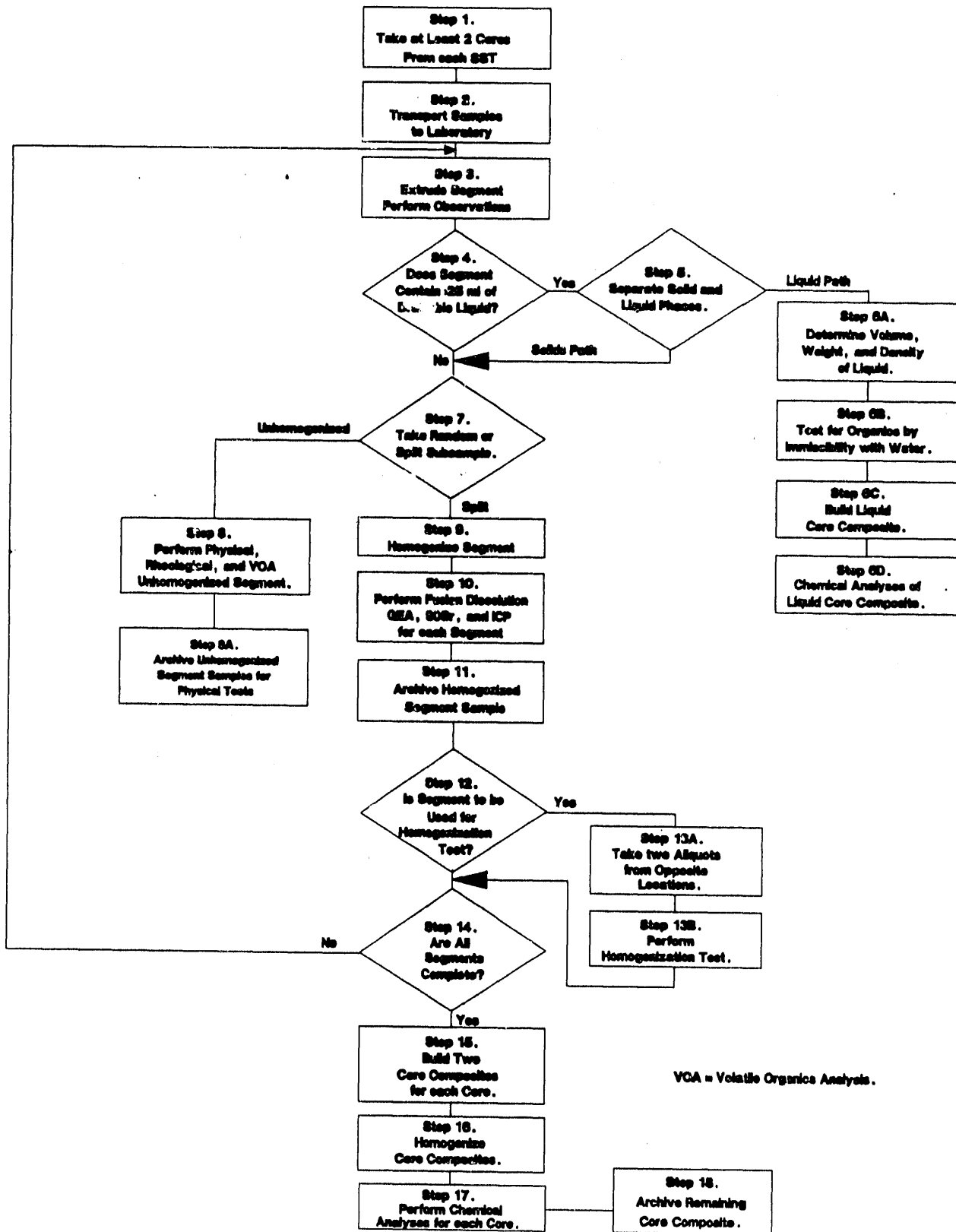
Drainable liquid

- Volume of liquid in liner (mL)
- Weight of liquid in liner (g)
- Volume of liquid in sampler (mL)
- Weight of liquid in sampler (g)

Bulk solid

- Weight of segment (g)
- Length of segment (in.)
- Length of segment (cm)
- Diameter of segment (cm)
- Volume of segment (cc)
- Bulk density (g/mL)
- Percent recovery
- Penetrometer

Figure I6-7. Sample and Analysis Flowsheet for Single-Shell Tank C-106.



The bulk density will be obtained by dividing the weight of the segment by the volume of the segment so that:

$$\text{Bulk Density} = \frac{\text{Weight of Segment}}{\text{Volume of Segment}}$$

The percent recovery can be determined by dividing the volume of material actually collected in the sampler by the volume expected from a particular segment and then multiplying by 100.

$$\text{Percent Recovery} = \frac{\text{Liquid Volume} + \text{Solid Volume}}{\text{Expected Volume}} \times 100 \%$$

A color photograph documenting the extruded segment will be taken after completely extruding the entire segment.

- Step 4--If the sample contains more than 25 mL of drainable liquid, the liquid should be analyzed separately from the solids.

If the liquid is <25 mL, then, it must be determined whether the limited quantity of liquid is actually NPH will be made. If the small quantity (<25 mL) of liquid is resolved to be NPH, then it should be drained off and analyzed by GC to determine if any organic compound other than NPH is present. If the drained NPH is highly colored then an acid digestion shall be prepared and analyzed for ICP, GEA, and total alpha. The NPH should not be discarded unless until directed by TWCT personnel. If the small quantity of liquid is not NPH, it should be retained with the sample for eventual homogenization. Proceed to Step 7.

If the amount of drainable liquid is greater than 25 mL, then proceed to Step 5.

- Step 5--Separate the drainable liquid from the solids by allowing the liquid to drain into a clean, plastic bottle. The liquid may be drained from the extrusion tray or through a coarse, inert (stainless steel, glass, or Teflon) filter that will permit the solids to be recovered without significant losses. The solids are to be retained in the extrusion tray for further subsampling and analysis.
- Step 6A--The weight, volume, and density are determined on the liquid.
- Step 6B--Sometimes NPH from the drilling is trapped in the sampler. GC analysis, immiscibility test, and density measurement are used to determine if it is NPH. If the liquid is NPH, analyze it by GEA, ICP, and total alpha to evaluate if it is significantly contaminated with waste. Also, record its color. If its density indicates it is some other organic save for ignitibility testing.

- Step 6C--Prepare a liquid core composite from the liquids from each segment. If the volume is small (25 to 50 mL) and found in only one or two segments, composite the liquids proportionately with the solid composite and homogenize before subsampling.
- Step 6D--If a liquid core composite is prepared, analyze for the same analytes as the solid core composite as shown in Table I5-1.
- Step 7--While the core is unhomogenized and still in the extrusion tray, either randomly remove ~40 g of sample from every 4 to 5 in. of the segment (enough [160-200 g] to make two core composites to perform limited chemical analyses, and segment archive) for the entire length of the segment or split the sample lengthwise into a portion for rheology and a portion for composites. This should be done in a manner that disturbs the physical nature of the waste as little as possible and fast enough that segments do not dry significantly. The random or split sample is transferred to a glass jar for homogenization (Step 9).
- Step 8--The remaining unhomogenized segment material is then subsampled for particle size, rheologic properties, settling velocities, weight percent solids, VOA, DSC, and thermal gravimetry according to procedures indicated for each analysis in Table I5-2. Subsamples also will be taken to perform all of the physical measurements listed in Table I5-3.
- Step 8A--Some of the unhomogenized subsamples will have to be archived because the procedures or technology are not currently available onsite.
- Step 9--Homogenize the solids from Step 8 using procedure T046 at 222-S Laboratory and procedure PNL-ALO-135 at the 325 Laboratory.
- Step 10--The homogenized segment shall be subsampled to perform a fusion dissolution GEA, and ⁹⁰Sr and ICP analyses as well as weight % water.
- Step 11--Approximately 50 g of each homogenized segment should be archived in a sealed glass jar for future analytical studies.
- Step 12--Determine if the segment is to be used for a homogenization test. Two segments per core from this tank will be used for a homogenization test unless problems are encountered during homogenization.
- Step 13A--If a homogenization test is to be done, take one 3-to-5-g subsample from opposite locations of the homogenized segment (two subsamples).
- Step 13B--Prepare duplicate 1-g aliquots of the subsamples (through the acid digestion) analyses for ICP, GEA, ⁹⁰Sr, and total alpha using the same procedures identified for acid digestion listed in Table I5-1.

- Step 14--When all the segments have been either subsampled for core composites (rheology segments) or homogenized, the core composites can be built.
- Step 15--Using portions of the homogenized segments from Step 10, build two core composites for each core. Identify and report all segments and weights used to make the composites. (See Section I6-1 for a discussion of core compositing.)
- Step 16--Homogenize each of the core composites.
- Step 17--Perform duplicate analyses for all the parameters identified in Table I5-1 for each core composite.
- Step 18--Archive 200 to 300 g of remaining segments for analysis verification and performance assessment parameters.

Figure I6-8 depicts the allocation of the core samples for SST C-106.

I6.2.2.2 Core Sample Utilization for Single-Shell Tank C-106. Tables 6-12 through 6-16 look at sample utilization, the impact of partial recovery, and sample quantity requirements for SST C-106. These tables are intended to help develop analysis strategies when partial segments are received.

Table I6-12 estimates the amount of prehomogenized segment sample used for SST C-106. Rheological properties will be determined for each segment. A maximum and minimum quantity of sample needed is estimated. The maximum is based on large sample sizes, full quality control, and sufficient sample for reruns. The minimum is based on smaller samples, and reduced quality control and rerun requirements. The basis for the numbers is provided. For example, 5S + 5D + 10MSD + 10RR means 5 g for sample + 5 g for duplicate + 10 g for matrix spike duplicate + 10 g for reruns. Use of the maximum sample size and QA requirements consume the entire segment. Therefore, minimum sample sizes and QA will be required for this tank. Approximately 94 g of segment sample will remain to build core composites.

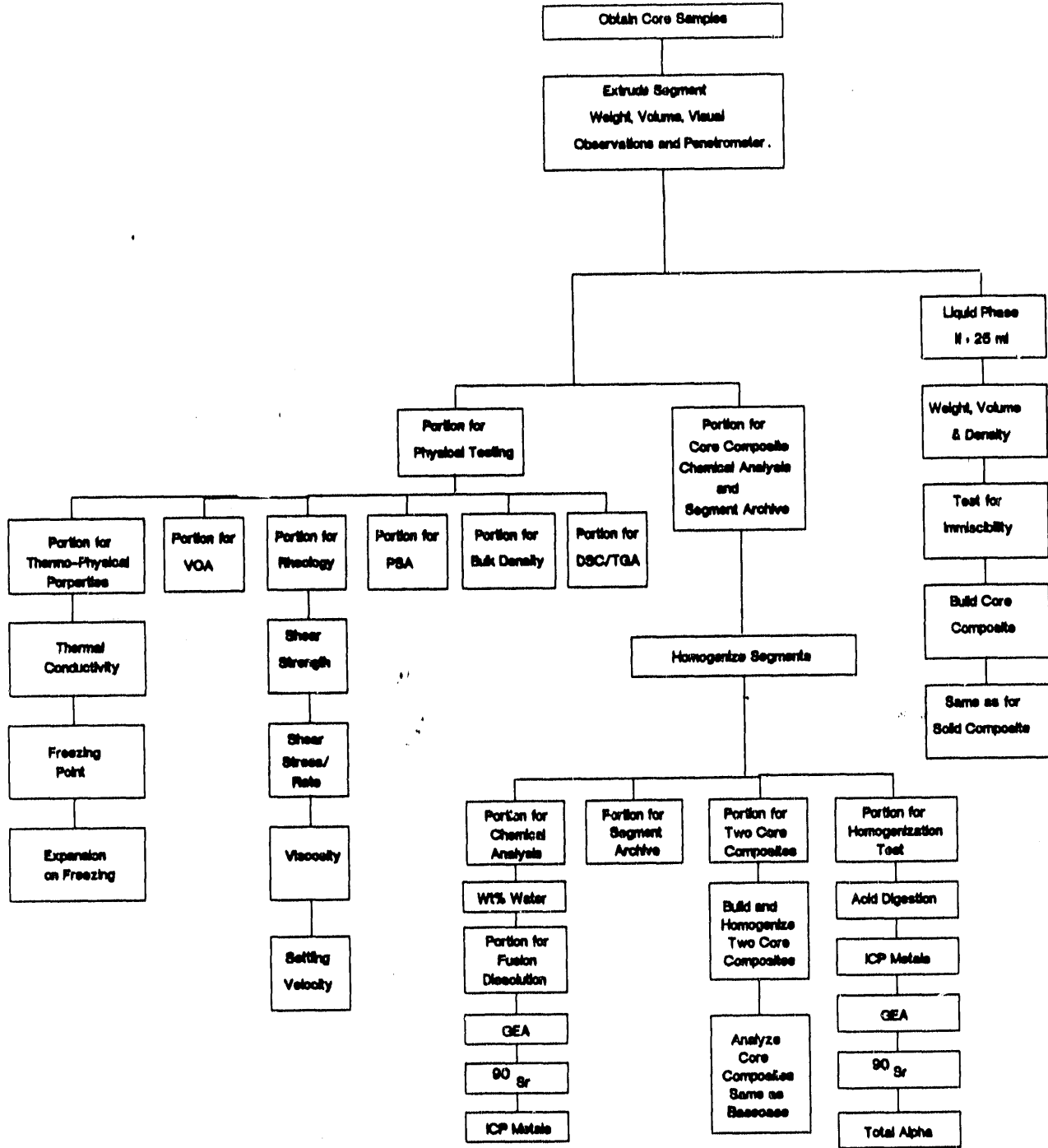
Table I6-13 estimates the amount of homogenized subsample that will be required to perform limited chemical analyses. Only a limited number of fusion dissolution analyses will be performed on a segmental basis.

Table I6-14 estimates the amount of sample needed for completing the analysis on a single core composite. Since two core composites must be made for each core, the values required for compositing (listed as the first subtotal) must be doubled. The remaining analyses and archives only require one quantity per core.

Table I6-15 estimates the volume of water-digested sample needed to complete the analysis. This estimate indicates that the digestion procedure should be changed from 1 g in 100 mL of water to 2 g in 200 mL of water. This allows larger sample sizes and better detection limits.

Table I6-16 estimates the amount of sample remaining in each segment for compositing samples with different percent recoveries. The total quantities that could be composited are estimated for four segments per core using only

Figure I6-8. Sample Allocation for Single-Shell Tank C-106.



- | | | | |
|------|------------------------------------|------|--------------------------------------|
| CVAA | cold vapor atomic absorption | IC | Ion Chromatography |
| DSC | differential scanning calorimetry | ICP | inductively coupled plasma |
| EOX | extractable organic halide | PSA | particle size analysis |
| GEA | gamma energy analysis | TGA | thermogravimetry |
| GFAA | graphite furnace atomic absorption | TCLP | toxic characteristic leach procedure |
| HYAA | hydride atomic absorption | TOC | total organic carbon |
| | | VOA | volatile organic analysis |

Table I6-12. Prehomogenized Sample Utilization (C-106).

Task Performed	Max (g)	Min (g)	Balance (g)	Basis
Extrude Segment	0	0	234	180mL x 1.3 g/mL
Portion for DSC	3	2	231-233	3 phases x 1g (Minimum of 2 per Segment)
Portion for Particle Size	1	1	230-232	Random 1g
Portion for Bulk Density	0	0	230-232	Calculations for weight and volume
Portion for Rheology	120	65	110-167	90mL and 50mL samples
Portion for VOA	30	12	80-155	5S + 5D + 10MSD + 10RR 2S + 2D + 4SD + 4RR
Transfer Loss	24	12	56-143	5% = 6g, 10% = 12g
Portion for Chemical Analysis	7	5	49-138	See Table I6-
Portion for Homog. Test	6	4	43-134	2S + 2D + 2RR 2S + 2D
Thermo-Physical Tests	30	30	13-114	
Portion for Segment Archive	75	20	0-94	Full basecase segment archive.
Amount Remaining for Core Composites	-	-	0-94	
Total g Used	296	140		

Table I6-13. Estimate of Segment Sample Utilization (C-106).

Analysis Performed	Rheology Core		Max Basis Min Basis
	Maximum Used (g)	Minimum Used (g)	
Fusion	6	4	Same as ICP Acid
Dissolution	1	1	0.25S + 0.25D + 0.5RR
ICP			0.25S + 0.25D + 0.5RR
Rads			
Total Segment	7	5	

Table I6-14. Estimate of Core Composite Sample Utilization (C-106).

Analysis	CC Max. (g)	CC Min. (g)	Basis (Max) Basis (Min)
Acid Digestion # 1 (ICP)	6	4	1S + 1D + 2MSD + 2RR 1S + 1D + 1MS + 1RR
Acid Digestion # 2 (GFAA)	6	4	Same as ICP
Direct As, Se (HYAA)	3	2	(0.25S + 0.25D + 0.5MSD + 0.5RR) (2)
CVAA (Hg)	2	1	.2S + .2D + .4MSD + .4RR
Water Digestion # 1 (IC, NH ₃ , TOC, ICP, Rads)	8	6	2S + 2D + 4RR 2S + 2D + 2RR
Water Digestion # 2 (pH, Corrosivity)	10	8	2.5S + 2.5D + 5.0RR 2.5S + 2.5D + 2.5RR
Fusion Portion (Rads, ICP)	1	1	0.25S + 0.25D + 0.5RR 0.25S + 0.25D + 0.25RR
Direct Anions CN ⁻ Portion (Macro) (Micro) S ⁻² Portion	6 2 2	4 1 2	1S + 1D + 2MSD + 2RR 0.2S + 0.2D + 0.4MSD + 0.4RR Same as Micro CN ⁻
Wt% H ₂ O	4	3	1S + 1D + 2RR 1S + 1D + 1RR
DSC/TGA	1	1	0.1S + 0.1D + 0.2RR (a)
Subtotal x 2	51 102	36 72	
Semi-VOA	50	20	5S + 5D + 10MSD + 10RR 5S + 5S + 5MS + 5RR
EOX	12	8	2S + 2D + 4MSD + 4RR 2S + 2D + 2MS + 2RR
Subtotal	164	100	
TCLP	20	10	10S + 10D or RR 10S
Subtotal	184	110	
Analysis Archive	75	20	Same as Basecase Archive
Subtotal	259	130	
PA Archive	150	100	Performance Assessment Tests (Duplicates)
Total	409	230	

CC = Core Composite
(a) = Lowest estimate is rounded to 1 g

Table I6-15. Water Digestion Sample Utilization (C-106).

Analysis	Water Maximum mL	Water Minimum mL	Basis (Maximum, mL) (Minimum, mL)
IC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.2RR
Carbonate	1	1	0.2S + 0.2D + 0.2PS + 0.2RR 0.2S + 0.2D
Ammonia	15	6	5S + 5D + 5RR 2S + 2D + 2RR
TOC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.25RR
ICP	80	50	20S + 20D + 20PS + 20RR 10S + 10D + 20PS + 10RR
C-14	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
H-3	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
Total	192 mL	109 mL	

PS = post adjustment spike

Table I6-16. % Recovery Sensitivity Study for SST C-106.

% Recovery	Min	Rheology
	234 x f _p -140	4 Min
100	94	376
90	71	284
80	47	188
70	24	96
60	0	0
50	0	0
40	0	0
30	0	0
20	0	0

(a) No VOA Analyses

minimum posthomogenization sample sizes because maximum sample sizes did not leave any sample for compositing. The amount of available sample is dependant upon the percent recovery. The PA archive sample will not be attained from this tank due to the limited amount of sample. If under an 80% core recovery is achieved, then an insufficient amount of sample will be available to perform a full core composite characterization on two composites per core. In this circumstance, only one core composite per core will be built to enable a full characterization to be performed on that single core. This decision will be made by Tank Waste Characterization Technology in conjunction with the OSM.

16.2.3 GAS GENERATING TANKS (T-110)

One gas generating SST, T-110, is planned to be sampled during the next ten SSTs. Three cores are expected to be obtained from this SST.

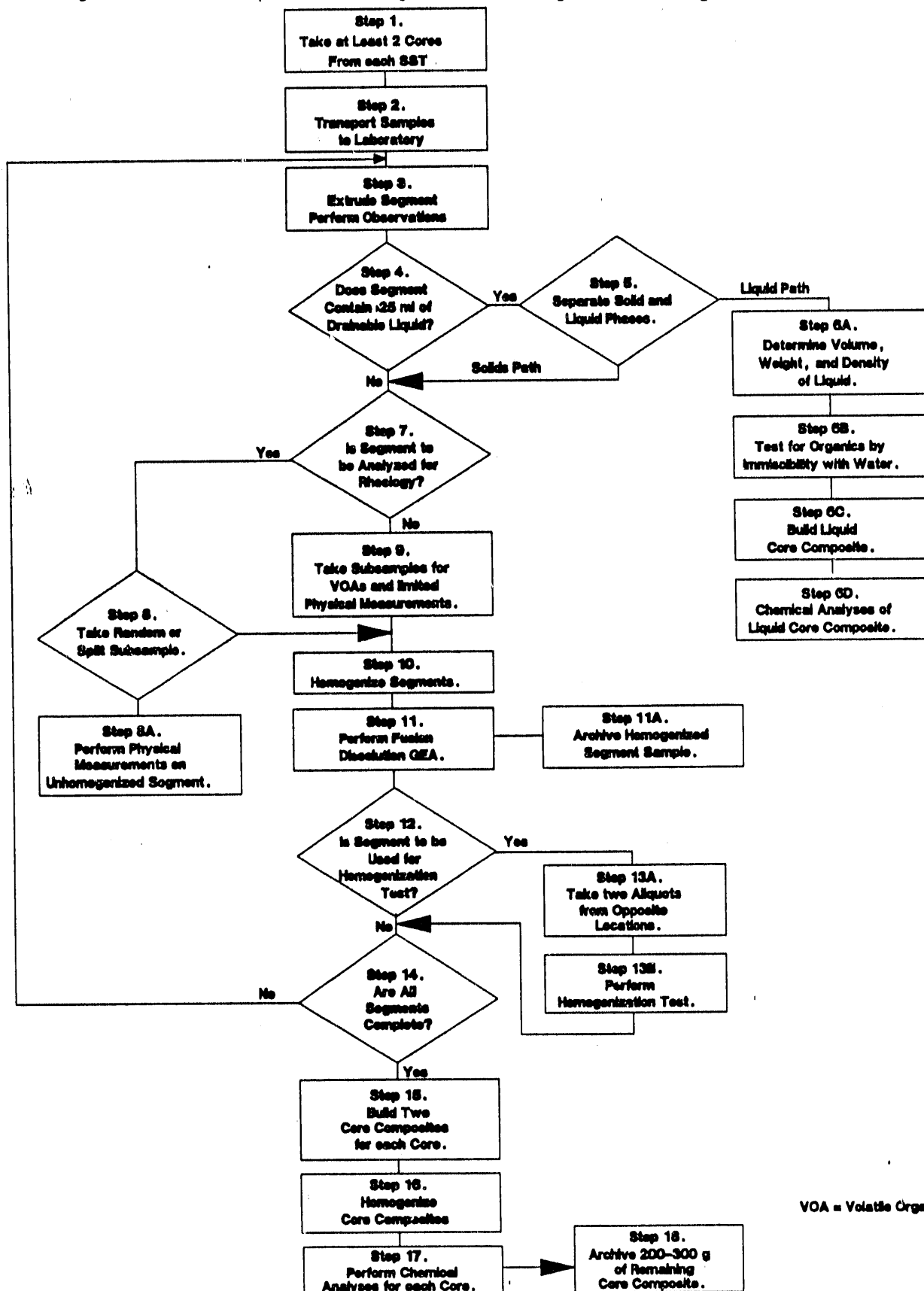
16.2.3.1 Sample and Analysis Scheme for Single-Shell Tank T-110. A flowchart depicting the sampling and analysis scheme for SST T-110 is presented in Figure I6-9. The individual steps shown in Figure I6-9 are described in detail as follows:

- Step 1--Tank Farm operations will obtain one core from three different risers in SST T-110 using procedure TO-020-450, "Perform Core Sampling." One field blank will be taken by preparing a sampler, as normal, using any necessary sealants but filling it in the field with deionized water from the laboratory.
- Step 2--The decision to ship core samples to laboratory 325 or 222-S, will be made by the OSM before initiation of the particular sampling event. Core samples will be transported to the laboratories in accordance with procedure TO-080-090, "Ship Core Samples."
- Step 3--Samples will be received, broken down, and extruded at each laboratory using the procedures shown in Table I4-1. The visual observations will be recorded on a SST Extrusion Logsheet. (A copy of this logsheet is presented in Figure I6-2.) The visual observations will include a sketch of the extruded core and such pertinent descriptive information as color, texture, homogeneity, and consistency. The physical parameters identified on the extrusion logsheet will be measured and recorded. The physical parameters listed include:

Drainable liquid

- Volume of liquid in liner (mL)
- Weight of liquid in liner (g)
- Volume of liquid in sampler (mL)
- Weight of liquid in sampler (g)

Figure I6-9. Sample and Analysis Flow Diagram for Single-Shell Tank T-110.



VOA = Volatile Organics Analysis.

Bulk solid

- Weight of segment (g)
- Length of segment (in.)
- Length of segment (cm)
- Diameter of segment (cm)
- Volume of segment (cc)
- Bulk density (g/mL)
- Percent recovery
- Penetrometer

The bulk density will be obtained by dividing the weight of the segment by the volume of the segment such that:

$$\text{Bulk Density} = \frac{\text{Weight of Segment}}{\text{Volume of Segment}}$$

The percent recovery can be determined by dividing the volume of material actually collected in the sampler by the volume expected from a particular segment and then multiplying by 100.

$$\text{Percent Recovery} = \frac{\text{Liquid Volume} + \text{Solid Volume}}{\text{Expected Volume}} \times 100$$

A color photograph documenting the extruded segment will be taken after completely extruding the entire segment.

- Step 4--If the sample contains more than 25 mL of drainable liquid, the liquid should be analyzed separately from the solids.

If the liquid is <25 mL, then it must be determined whether the limited quantity of liquid is actually NPH will be made. If the small quantity (<25 mL) of liquid is resolved to be NPH, then it should be drained off and analyzed by GC to determine if any organic compound other than NPH is present. If the drained NPH is highly colored then an acid digestion shall be prepared and analyzed for ICP, GEA, and total alpha. The NPH should not be discarded unless until directed by TWCT personnel. If the small quantity of liquid is not NPH, it should be retained with the sample for eventual homogenization. Proceed to Step 7.

If the amount of drainable liquid is greater than 25 mL, then proceed to Step 5.

- Step 5--Separate the drainable liquid from the solids by allowing the liquid to drain into a clean, plastic bottle. The liquid may be drained from the extrusion tray or through a coarse, inert (stainless steel, glass, or Teflon) filter that will permit the solids to be recovered without significant losses. The solids are to be retained in the extrusion tray for further subsampling and analysis.

- Step 6A--The weight, volume, and density are determined on the liquid.
- Step 6B--Sometimes NPH from the drilling is trapped in the sampler. GC analysis, immiscibility test, and density measurement are used to determine if it is NPH. If the liquid is NPH, analyze it by GEA, ICP, and total alpha to evaluate if it is significantly contaminated with waste. Also, record its color. If its density indicates it is some other organic save for ignitibility testing.
- Step 6C--Prepare a liquid core composite from the liquids from each segment. If the volume is small (25 to 50 mL) and found in only one or two segments, composite the liquids proportionately with the solid composite and homogenize before subsampling.
- Step 6D--If a liquid core composite is prepared, analyze for the same analytes as the solid core composite as shown in Table I5-1.
- Step 7--Every other segment from the first core obtained from each SST will be used for extensive physical rheologic measurements. If a segment is chosen for rheological examination, then proceed to Step 8; otherwise, continue with Step 9. Incomplete core recovery on other factors may require these segment selections to be changed. These segments are chosen to provide rheology information for waste at different depths in the tank. If incomplete segments are obtained, Section I6.1 should be consulted for guidance on how to use the sample and the change in plan discussed with OSM and Tank Waste Characterization Technology.
- Step 8--While the core is unhomogenized and still in the extrusion tray, either randomly remove ~30 g of sample from every 4 to 5 in. of the segment (enough [120-150 g] to make 2 core composites and segment archive) for the entire length of the segment or split the sample lengthwise into a portion for rheology and a portion for composites. This should be done in a manner that disturbs the physical nature of the waste as little as possible and fast enough that segments do not dry significantly. The random or split sample is transferred to a glass jar for homogenization (Step 10).
- Step 8A--The remaining unhomogenized segment material is then subsampled for particle size, rheologic properties, settling velocities, weight percent solids, DSC, and thermal gravimetry according to procedures indicated for each analysis in Table I5-2.
- Step 9--If the segment is not used for rheology, take subsamples for VOA and a limited number of physical tests. The required physical tests are weight percent solids, DSC, and thermal gravimetry. The procedures for these analyses are listed in Table I5-2. Randomly sampled aliquots are collected from the length of the core until about 10 g are obtained for VOA. These should be collected and sealed as soon as possible after extrusion. A similar procedure is used to obtain 1 to 3 g for particle size. Differential scanning calorimetry samples should not be combined. Choose a small (~0.5 g) sample from each distinctive region of the segment. Attempts should

be made to run the DSC on different phases based on visual observations with the objective of locating concentrated areas of potentially exothermic materials. Thermal gravimetric analysis should be performed on these same portions to estimate water content and to support DSC analysis evaluations. Because of problems keeping radioactive VOA samples cooled, these samples will be analyzed as quickly as possible.

- Step 10--Homogenize the solids from Step 9 or the random/ split sample from Step 8 using procedure T046 at the 222-S Laboratory and procedure PNL-ALO-135 at the 325 Laboratory.
- Step 11--A sufficient aliquot will be obtained to perform fusion a dissolution GEA, and ⁹⁰Sr and ICP metals analyses.
- Step 11A--Approximately 50 g of each homogenized segment should be archived in a sealed glass jar for future analytical studies.
- Step 12--Determine if the segment is to be used for a homogenization test. Every other segment from the second (nonrheologic) core will be used for a homogenization test or at least two homogenization tests for this tank. The frequency of the homogenization tests should be increased if problems homogenizing samples are encountered.
- Step 13A--If a homogenization test is to be done, take one 3-to-5-g subsample from opposite locations of the homogenized segment (2 subsamples).
- Step 13B--Prepare duplicate 1-g aliquots of the subsamples (through the acid digestion) analyses for ICP, GEA, and total alpha using the same procedures identified for acid digestion listed in Table I5-1.
- Step 14--When all the segments have been either subsampled for core composites (rheology segments) or homogenized, the core composites can be built.
- Step 15--Using portions of the homogenized segments from Step 10, build two core composites for each core. Identify and report all segments and weights used to make the composites. (See Section I6-1 for a discussion of core compositing.)
- Step 16--Homogenize each of the core composites.
- Step 17--Perform duplicate analyses for all the parameters identified in Table I5-1 for each core composite.
- Step 18--Archive 200 to 300 g of remaining segments for analysis verification and performance assessment parameters.

Figure I6-10 depicts the sample allocation scheme for analysis of SST T-110.

16.2.3.2 Core Sample Utilization for Single-Shell Tank T-110. Tables 6-17 through 6-22 look at sample utilization, the impact of partial recovery, and sample quantity requirements for SST T-110. These tables are intended to help develop analysis strategies when partial segments are received.

Table I6-17 estimates the amount of prehomogenized segment sample used and remaining for cores in which (1) rheology samples are taken and (2) no rheology is performed. A maximum and minimum quantity of sample needed is estimated. The maximum is based on large sample sizes, full quality control, and sufficient sample for reruns. The minimum is based on smaller samples, and reduced quality control and rerun requirements. The basis for the numbers is provided. For example, 5S + 5D + 10MSD + 10RR means 5 g for sample + 5 g for duplicate + 10 g for matrix spike duplicate + 10 g for reruns. Obviously, cores used for rheology require the most samples. For the cores without rheology, the VOA consumes the most sample.

Table I6-18 estimates the amount of segment sample that needs to be archived. Since the analysis of semi-volatile organics and TCLP testing will probably not be required on segments, archive samples of 15 to 30 g should be adequate for most chemical and radiochemical tests.

Table I6-19 estimates the amount of segment sample utilization for chemical analyses.

Table I6-20 estimates the amount of sample needed for completing the analysis on a single core composite. Since two core composites must be made for each core, the values required for compositing (listed as the first subtotal) must be doubled. The remaining analyses and archives only require one quantity per core.

Table I6-21 estimates the volume of water-digested sample needed to complete the analysis. This estimate indicates that the digestion procedure should be changed from 1 g in 100 mL of water to 2 g in 200 mL of water. This allows larger sample sizes and better detection limits.

Table I6-22 estimates the amount of sample remaining in each segment for compositing samples with different percent recoveries for rheologic cores and nonrheologic cores. The total quantities that could be composited are reported for maximum and minimum posthomogenization sample sizes. The amount of available sample is dependant upon the percent recovery. Table I6-23 indicates that there is sufficient sample full core composite characterization (on a minimum sample-size basis) for all percent recoveries greater than 50.

16.3 DATA REPORTING

Data reporting requirements are according to the statement of work for each laboratory. The data reporting (Section I1.7), has been modified to reflect recent revisions to the Tri-Party Agreement (Ecology et al. 1989).

Table I6-17. Prehomogenized Sample Utilization (T-110).

Task Performed	Rheology Core			No rheology Core			Basis
	Maximum Used (g)	Minimum Used (g)	Balance Range (g)	Maximum Used (g)	Minimum Used (g)	Balance Range (g)	
Extrude Segment	0	0	234	0	0	234	180mL x 1.3 g/mL
Portion for DSC	3	1	231-233	3	1	231-233	3 phases x 1g
Portion for Particle Size	1	1	230-232	1	1	230-232	Random 1g
Portion for Bulk Density	0	0	230-232	0	0	230-232	Calculations for weight and volume
Portion for Rheology	120	65	110-167	0	0	230-232	90mL and 50mL samples
Portion for VOA	0	0	110-167	30	12	200-220	5S + 5D + 10MSD + 10RR 2S + 2D + 4SD + 4RR
Transfer Loss	24	12	86-155	24	12	176-208	5% = 12g, 10% = 24g
Portion for Homog. Test	0	0	86-155	6	4	170-204	2S + 2D + 2RR 2S + 2D
Portion for Chemical Test	7	5	79-150	7	5	163-199	
Portion for Seg. Archive	30	14	49-136	75	20	88-179	No TCLP, Semi-VOA or EOX on Rheology
Amount Remaining for Core Composites	-	-	49-136	-	-	88-179	
Total g Used	185	98		146	55		

S = Sample
D = Duplicate
MSD = Matrix Spike Duplicate
RR = Rerun
Seg. = Segment
Homog. = Homogenization

Table I6-18. Estimate of Segment Sample Utilization (T-110)

Analysis Performed	Rheology Core		Max Basis Min Basis
	Maximum Used (g)	Minimum Used (g)	
Fusion	6	4	Same as ICP Acid
Dissolution	1	1	0.25S + 0.25D + 0.5RR
ICP			0.25S + 0.25D + 0.5RR
Rads			
Total Segment	7	5	

Table I6-19. Estimate of Segment Archive Sample Utilization.

Analysis	Segment Maximum	Amount Minimum	Basis
ICP/Acid	6	2	1S + 1D + 2MSD + 2RR 1S + 1D
Anions/Water	4	2	1S + 1D + 2RR 1S + 1D
Rads/Fusion	1	1	0.25S + .25D + 0.5RR
pH/Corrosivity	5	5	2.5S + 2.5D
GFAA/Acid	6	2	Same as ICP
CVAA/Hg	2	1	0.2S + 0.2D + 0.4MSD + 0.4RR 0.2S + 0.2D
Cyanide	6	1	1.0S + 1.0D + 2.0MSD + 2.0RR (Large Dist.) 0.2S + 0.2D + 0.4MSD (Micro Dist.)
Subtotal	30	14	
Semi-VOA	30	6	5.0S + 5.0D + 10MSD + 10RR 2.0S + 2.0D + 2MSD
TCLP	10	0	10S
EOX	5	0	5S
Total	75g	20g	

Table I6-20. Estimate of Core Composite Sample Utilization (T-110).

Analysis	CC Max. (g)	CC Min. (g)	Basis (Max) Basis (Min)
Acid Digestion # 1 (ICP)	6	4	1S + 1D + 2MSD + 2RR 1S + 1D + 1MS + 1RR
Acid Digestion # 2 (GFAA)	6	4	Same as ICP
Direct As, Se (HYAA)	3	2	(0.25S + 0.25D + 0.5MSD + 0.5RR) (2)
CVAAs (Hg)	2	1	.2S + .2D + .4MSD + .4RR
Water Digestion # 1 (IC, NH ₃ , TOC, ICP, Rads)	8	6	2S + 2D + 4RR 2S + 2D + 2RR
Water Digestion # 2 (pH, Corrosivity)	10	8	2.5S + 2.5D + 5.0RR 2.5S + 2.5D + 2.5RR
Fusion Portion (Rads, ICP)	1 ?6	1 ?4	0.25S + 0.25D + 0.5RR 0.25S + 0.25D + 0.25RR
Direct Anions CN ⁻ Portion (Macro) (Micro) S ⁻² Portion	6 2 ?6	4 1 ?4	1S + 1D + 2MSD + 2RR 0.2S + 0.2D + 0.4MSD + 0.4RR ?Same as Macro CN ⁻
Wt% H ₂ O	4	3	1S + 1D + 2RR 1S + 1D + 1RR
DSC/TGA	1	1	0.1S + 0.1D + 0.2RR (a)
Subtotal x 2	61 122	43 86	
Semi-VOA	50	20	5S + 5D + 10MSD + 10RR 5S + 5S + 5MS + 5RR
EOX	12	8	2S + 2D + 4MSD + 4RR 2S + 2D + 2MS + 2RR
Subtotal	184	114	
TCLP	20	10	10S + 10D or RR 10S
Subtotal	204	124	
Analysis Archive	75	20	Same as Segment Estimate
Subtotal	279	144	
PA Archive	150	100	Performance Assessment Tests (Duplicates)
Total	429	244	

CC = Core Composite
(a) = Lowest estimate is rounded to 1 g

Table I6-21. Water Digestion Sample Utilization (T-110).

Analysis	Water Maximum mL	Water Minimum mL	Basis (Maximum, mL) (Minimum, mL)
IC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.2RR
Carbonate	1	1	0.2S + 0.2D + 0.2PS + 0.2RR 0.2S + 0.2D
Ammonia	15	6	5S + 5D + 5RR 2S + 2D + 2RR
TOC	8	6	1S + 1D + 5PS + 1RR 0.25S + 0.25D + 5PS + 0.25RR
ICP	80	50	20S + 20D + 20PS + 20RR 10S + 10D + 20PS + 10RR
C-14	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
H-3	40	20	10S + 10D + 10PS + 10RR 5S + 5D + 5PS + 5RR
Total	192 mL	109 mL	

PS = post adjustment spike

Table I6-22. % Recovery Sensitivity Study for SST T-110.

% Recovery	Min	Rheology		Min	No Rheology	
		8 Max	8 Min		8 Max	8 Min
	234 x f _R -185 234 x f _R -98			234 x f _R -146 234 x f _R -55		
100	49 136	392	1088	88 179	704	1432
90	27 112	216	896	65 156	520	1248
80	2 899	16	712	41 132	328	1056
70	0 65	0	528	18 108	144	864
60	0 42	0	336	0 85	0	680
50	0 19	0	152	0 62	0	496
40	0 0	0	0	0 39	0	312
30	0 0	0	0	0 15	0	120
20	0 0	0	0	0 0	0	0

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