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7. Abstract

This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, WHC 222-S Laboratory, and PNL 325 Analytical Chemistry Laboratory. The scope of this plan is to provide guidance for the sampling and analysis of samples from tank 241-C-103.

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MASTER

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LIST OF ABBREVIATIONS

ACL	Analytical Chemistry Laboratory	LW	Lab Waste
B	B-Plant high level waste	MW	Metal Waste (from bismuth phosphate operations)
BL	B-Plant low level waste	N	N-Reactor Waste
BNW	Battelle Northwest	NCPLX	Noncomplexed Waste
C-103	Tank 241-C-103	OWW	Organic Wash Waste
CW	Cladding Waste	P	Purex High Level Waste
DQO	Data Quality Objective	PL	Purex Low Level Waste
DSC	Differential Scanning Calorimetry	PSS	Purex Sludge Supernatant
DSSF	Double Shell Slurry Feed	R	Redox Waste
DST	Double Shell Tank	RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
DW	Decontamination Waste	SST	Single-Shell Tank
EB	Evaporator Bottoms	TBP	Tri-butyl Phosphate
EVAP	Post-1976 (designation for evaporator feed)	TGA	Thermogravimetric Analysis
IC	Ion Chromatography	TOC	Total Organic Carbon
ICP	Inductively Coupled Plasma (atomic emission spectroscopy)	TWRS	Tank Waste Remediation System
IX	Ion Exchange Waste (from Cesium recovery)	USQ	Unreviewed Safety Question
		WHC	Westinghouse Hanford Company

1.0 SPECIFIC TANK OBJECTIVES

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

1.1 RELEVANT SAFETY ISSUES

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

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

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

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

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

1.1.1 TANK C-103 CHARACTERIZATION OBJECTIVES

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

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

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

1.1.2 ORGANIC SALTS, PRETREATMENT, AND SAFETY SCREENING DATA QUALITY OBJECTIVES

The sampling and analytical needs associated with organic salts tanks, as well as the safety screening of all tanks, have been identified through the Data Quality Objective (DQO) process. In addition, data needs associated with tank C-103 are identified in the pretreatment DQO. Additional data needs associated with tank C-103 may be identified in subsequent DQO efforts.

Pertinent documents to this effort include the following:

- (1) *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al. 1994), which describes the sampling and analytical requirements for tanks on the Organic Salts Watch List, including tank C-103.
- (2) *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994), which describes the sampling and analytical requirements for screening all waste tanks for unidentified safety issues.
- (3) *Interim Data Quality Objectives for Waste Pretreatment and Vitrification* (Kupfer et al. 1994) which describes the sampling and analytical requirements to support the TWRS technical strategy by identifying the chemical and physical composition of the waste in the tank. In addition, the DQO works to guide development efforts to define waste pretreatment processes, which will in turn define high-level and low-level waste feed to vitrification processes.

1.1.3 DATA QUALITY OBJECTIVES INTEGRATION

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

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

Many of the analytes identified in the pretreatment DQO are the same as those required by the organic and safety screening DQOs, although there are many additional analyses required that are not specified in the organic and safety screening DQOs. However, since the Pretreatment DQO analysis suite is to be run on core composite samples, little integration of the required analyses could be made. To simplify the explanation of the Pretreatment DQO analytical requirements, Figures 5 and 6 have been included in this Tank Characterization Plan which clarify their requests.

2.0 TANK, WASTE, AND SAMPLING INFORMATION

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

2.1 AGE AND PROCESS HISTORY

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

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

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

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

Figure 1: Fill History for Tank C-103

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

Table 1: Waste Status Summary for Tank C-103

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

2.2 EXPECTED TANK CONTENTS

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

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

Table 2: Most Recent Analysis of Liquid Samples¹

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

Ammonia	24 $\mu\text{g NH}_3/\text{g}$	215 $\mu\text{g NH}_3/\text{g}$
IC	F, Cl, NO_2 , NO_3 , SO_4 , all <50 $\mu\text{g/mL}$	$\text{F}^- = 1,185$, $\text{Cl}^- = 431$, $\text{NO}_2^- = 24,794$, $\text{NO}_3^- = 2,587$, $\text{SO}_4^{2-} = 3,234$, $\text{PO}_4^{3-} = 2,16$ ($\mu\text{g/mL}$)
ICP/AES (2% HNO_3 leach)	Ag = 0.8, Al = 1.6, B = 9.6, Ca = 1.8, Cd = 1.8, Cu = 1.9, Fe = 0.29, Na = 61.3, Ni = 8.7, P = 530 ($\mu\text{g/mL}$)	Ag = 12, Ca = 5, Cd = 0.9, Cr = 57, Fe = 3, K = 323, Na = 32,771, Ni = 72, P = 2,533, U = 2102, Zr = 302 ($\mu\text{g/mL}$)
Extractable organics by GC/MS	NA	TBP = 86 $\mu\text{g/mL}$ DBBP = 7 $\mu\text{g/mL}$ NPH = 1-3 $\mu\text{g/mL}$
Volatiles by GC/MS	NA	Only NPH and TBP identified in headspace
Gamma analysis	NA	$^{60}\text{Co} = 0.0511 \mu\text{Ci/mL}$ $^{137}\text{Cs} = 57.9 \mu\text{Ci/mL}$
pH	NA	10.00, 9.99
Carbon	NA	TOC = 7200 $\mu\text{g/mL}$, TIC = 5200 $\mu\text{g/mL}$
DSC/STG TOC of dried solids	NA	Exotherm of 180 calories/g between 240 and 430 $^{\circ}\text{C}$. TOC = 4.4 wt%

(Pool 1994)

Note: 10^{12} pCi = 1 Ci

2.3 SAMPLING INFORMATION

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

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

One push mode core sample from each riser, consisting of four segments each, shall be collected from risers 2 and 7 of tank C-103. The first segment from each core is expected to contain 22.86 cm (9 inches) of waste material, while the three final segments should contain 48.26 cm (19 inches) of waste each.

Hydrostatic head fluid (HHF) with lithium bromide (LiBr) as a tracer shall be used to aid in the collection of all core samples. An HHF blank shall be prepared as part

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

3.0 LABORATORY SAMPLE RECEIPT AND ANALYSIS INSTRUCTIONS

3.1 TANK-SPECIFIC ANALYTICAL PROCEDURES

A flowchart depicting the general safety screening sample breakdown and analysis scheme is presented in Figures 2, 3, and 4. These steps are described in detail to provide the hot cell and laboratory chemists with guidance for the breakdown of the segments and may be altered as appropriate by the performing laboratory. Several analyses listed in Table 3 require a 45 day reporting time, as noted. The 45 day reporting format, Format III, is explained in Section 7.3.

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

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

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

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

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

Step 4 Is the segment 100% drainable liquid?

Yes: Proceed to Step 15

No: Proceed to Step 5

SOLIDS PATH

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

Step 6 Homogenize each subsample using the appropriate approved procedure.

Step 7 Will the subsample be used for the homogenization test?

Yes: Proceed to Step 8

No: Proceed to Step 9

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

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

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

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

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

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

Step 12 Collect sufficient aliquots from the solid composite to perform the appropriate preparations and analyses listed in Figure 5 in duplicate.

Step 13 Remove 100 mL of the solid composite as the Pretreatment solid composite archive (Bratzel 1994).

Step 14 Remove 200 mL of the solid composite for process development work (see Section 6.2).

NOTE: If insufficient sample material is available to provide an archive and a sample for process development of the sizes described, divide the material remaining after Step 12 into equal portions (i.e., equal-sized portions for archive and process development work).

LIQUIDS PATH

Step 15 Closely inspect the liquid sample for the presence and approximate volume of any potential organic layers. Does the sample contain any immiscible (potentially organic) layers?

Yes: Proceed to Step 16A

No: Proceed to Step 17

Step 16A Report any visually observed immiscible (potential organic) layer immediately by the early notification system.

Step 16B Separate and retain the potential organic layer for possible future analysis.

NOTE: Steps 17 through 24 shall be performed on the remaining (probable aqueous) liquid layer only.

Step 17 Filter the remaining liquid sample through a 0.45 micron filter.

Step 18 Is there greater than 1 gram of solid on the filter?

Yes: Proceed to Step 19

No: Proceed to Step 20

Step 19 Archive the solids for possible future analysis (Bratzel 1994).

Step 20 Remove sufficient aliquots from the segment-level liquid sample to perform the appropriate analyses listed in Table 3 in duplicate.

Step 21 Archive at least 20 mL and up to 40 mL of the segment-level drainable liquid as the segment level liquid archive (Bratzel 1994).

Step 22 Combine the segment-level liquid proportional to the liquid recovery of the segment to build a liquid composite of the core.

Step 23 Collect sufficient aliquots from the liquid composite to perform the appropriate preparations and analyses listed in Figure 6 in duplicate.

Step 24 Remove 100 mL of the liquid composite as the Pretreatment liquid composite archive (Bratzel 1994).

PRIMARY ANALYSIS PATH

Step 25 Perform primary analyses as listed in Table 3.

Step 26 Compare the primary analysis data with notification limits.

Step 27A Do the results exceed the notification limits (Table 3)?

Yes: If the results exceed the notification limits. Proceed to Step 27B.

No: If results do not exceed the notification limits, proceed to Step 30.

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

SECONDARY ANALYSIS PATH

Step 28 Perform secondary analyses according to Table 3.

Step 29A Do the secondary analyses exceed the notification limits?

Yes: Proceed to Step 29B

No: Proceed to Step 30

Step 29B Report results exceeding the notification limits using Format I reporting deliverable requirements as listed in Section 7.2.

Step 30 Report results as listed in Section 7.

Figure 2: Laboratory Flow Chart A

Figure 3: Laboratory Flow Chart B

Figure 4: Laboratory Flow Chart C

ANALYSES PATH

3.2 INSUFFICIENT SEGMENT RECOVERY

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

3.3 PRIORITIES OF REQUESTED ANALYSES

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

PRIORITY LEVEL 1

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

PRIORITY LEVEL 2

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

PRIORITY LEVEL 3

Equilibrium moisture content, GC/MS, hydroxide, nitrate and nitrite, ^{137}Cs , ^{90}Sr , and Cr analyses shall be performed.

PRIORITY LEVEL 4

The Pretreatment analytical needs as listed in Figures 5 and 6.

4.0 SPECIFIC ANALYTE, QUALITY CONTROL, AND DATA CRITERIA

4.1 SPECIFIC METHODS AND ANALYSES

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

4.2 QUALITY ASSURANCE/QUALITY CONTROL

4.2.1 LABORATORY OPERATIONS

The 222-S Laboratory has a quality assurance program plan (Meznarich 1994) and a quality assurance project plan (Taylor 1993) that shall provide the primary direction for the quality assurance/quality control of analyzing the waste tank core samples at the 222-S Laboratory. If the analyses are performed at the 325 Analytical Chemistry Laboratory (ACL), the analysis shall be guided by the 325 Quality Assurance Plan (Kuhl-Klinger 1994). Additionally, the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994), when implemented, shall be used as quality assurance/quality control guidance.

Method specific quality control such as calibrations and blanks are also found in the analytical procedures. Sample quality control (duplicates, spikes, standards) are identified in Table 3. If no criteria are provided in Table 3, the performing laboratory shall perform to its quality assurance plan(s).

4.2.2 SAMPLE COLLECTION

Two core samples with four segments each are to be taken from tank C-103 and shipped to the performing laboratory by Sampling Operations in accordance with work package 2E-93-00451. That work package shall also initiate the chain-of-custody for the samples. Approved work plans TFPE-YP-0229 and TFPE-YP-0230, and procedure T0-080-090 ("Load/Transport Sample Cask(s)") are to be used during the sampling event. Samples shall be identified by a unique number before being shipped to the performing laboratory. The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook. Sampling Operations shall enter this information in the comment section of the chain-of-custody form for addition to the data reports.

Sampling Operations should transport each segment collected to the performing laboratory within 1 working day of removing the segment from the tank, but must transport each segment within 3 days. The field and hot cell blanks shall each count as a segment. Sampling Operations is responsible for verbally notifying the laboratory (373-2435 for 222-S Laboratory; 373-6704 for 325 ACL) at least 24 hours in advance of an expected shipment. If samples are going to be delivered after 3:00 pm, the laboratory shall be notified at least four hours in advance of actual sample shipment so that proper shift operations can be planned.

4.2.3 SAMPLE CUSTODY

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

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

The sealed and labeled samples are shipped to the laboratory along with the chain-of-custody form. The receipt and control of samples in the WHC 222-S Laboratory are described in laboratory procedure LO-090-101. Receipt and control of samples in the 325 ACL are described in procedure PNL-AL0-051.

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Table 3: C-103 Chemical, Radiological and Physical Requirements

Project Name		C-103 Push Mode Core Sample					SOLID ANALYSES							CHARGE CODES		ORG	REPORTING LEVELS			
Plan Number		WHC-SD-WM-TP-207, REV. 0					COMMENTS							TBD		8E200	FORMAT I	Early Notify		
PROGRAM CONTACTS							Homogenization Test - Required <td colspan="2">TBD</td> <td>8E300</td> <td>FORMAT II</td> <td>Process Control</td>							TBD		8E300	FORMAT II	Process Control		
A. Organic							D. A. Turner							TBD		8E400	FORMAT III	Safety Screen		
B. Safety Screening							H. Babad							TBD		8E700	FORMAT IV	Waste Management		
C. Pretreatment							M. J. Kupfer							TBD		PNL	FORMAT V	RCRA Compliance		
TWRS							R. D. Schreiber										FORMAT VI	Special		
222-S Laboratory							J. G. Kristofzski													
325 Laboratory							S. G. McKinley													
							TANK		#CORES		CORE#									
							C-103		2		1 and 2									
PRIMARY ANALYSES							SAMPLE ¹		PREP ²		QUALITY CONTROL ³						CRITERIA		FOR- MAT	PRO- GRAM
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	SOLID COMP	% SEG SLDG		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴					
DSC	Energy	LA-514-113	PNL-ALO-508		X	d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	cal/g ⁵	> 115	unknown	I, III, IV	A,B			
TGA	% H2O	LA-560-112	PNL-ALO-508	X	X	see ¹⁵ , d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	17	unknown	I, III, IV	A,B,C			
Alpha	Total Alpha	LA-508-101	PNL-ALO-421 PNL-ALO-423		X	f	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	unknown	I, III, IV	B			
Hot Persulfate	TOC	LA-342-100	PNL-ALO-381	X	X ¹²	see ¹⁵ , d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 30,000	unknown	I, III, IV	A, B, C			
Sep. & b counting	Sr/Y-90	LA-220-101	PNL-ALO-431 PNL-ALO-433	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5		µCi/g	none	unknown	IV	C			
ICP	Al	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Ba	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Bi	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Ca	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Ce	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Cr	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Fe	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	K	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	La	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Li	LA-505-151	PNL-ALO-211		X	f, w	ea smpl	N/A ⁹	ea PB	ea AB	±10	90-110	µg/g	100	unknown	I, III, IV	A,B,C			
ICP	Mg	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Mn	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Na	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Ni	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	P	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	S	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Si	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP	Zr	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/g	none	unknown	IV	C			
ICP ¹⁶	Ag	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	As	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	B	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	Be	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	Cd	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	Cu	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	Mo	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			
ICP ¹⁶	Pb	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/g	none	unknown	IV	C			

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Table 3: C-103 Chemical, Radiological and Physical Requirements

ICP ¹⁶	Sb	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µg/g	none	unknown	IV	C	
ICP ¹⁶	Se	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µg/g	none	unknown	IV	C	
ICP ¹⁶	Sr	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µg/g	none	unknown	IV	C	
ICP ¹⁶	Zn	LA-505-151	PNL-ALO-211	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µg/g	none	unknown	IV	C	
IC	Cl ⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
IC	F ⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
IC	NO ₂ ⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
IC	NO ₃ ⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
IC	PO ₄ ³⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
IC	SO ₄ ²⁻	LA-533-105	PNL-ALO-212	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
TIC	CO ₃ ²⁻	LA-622-102	PNL-ALO-381	X		see ¹⁵	ea smpl	N/A	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
Sep. & a counting	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5	µCi/g	> 62/density	unknown	I, IV	C	
GEA	Am-241	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
GEA	Eu-154	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl	N/A	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
GEA	Cs/Ba-137	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl	N/A	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
GEA ¹⁶	Co-60	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/g	none	unknown	IV	C	
GEA ¹⁶	Eu-155	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/g	none	unknown	IV	C	
GEA ¹⁶	Sb-125	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/g	none	unknown	IV	C	
GEA ¹⁶	Sn-113	LA-548-121	PNL-ALO-450	X		see ¹⁵	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/g	none	unknown	IV	C	
Electrode	pH	LA-212-103	PNL-ALO-290	X		see ¹⁵	ea smpl	N/A	N/A	ea AB	see Table 5	units	none	unknown	IV	C	
Titration	OH ⁻	LA-661-103	PNL-ALO-225	X		see ¹⁵	ea smpl	1/mtrx	ea PB	N/A	see Table 5	ug/g	none	unknown	IV	C	
Gravimetry	Wt% sol.	LA-564-101	PNL-ALO-504	X		see ¹⁵	ea smpl	N/A	ea AB	ea AB	see Table 5	wt%	none	unknown	IV	C	
Wt% Oxides	Wt% oxides	N/A	PNL-ALO-501	X		see ¹⁵	ea smpl	N/A	ea AB	ea AB	see Table 5	wt%	none	unknown	IV	C	
Sep. & a counting	Np-237	LA-933-141	PNL-ALO-421 PNL-ALO-425	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
Liquid Scintillation	C-14	LA-348-104	PNL-ALO-474 PNL-ALO-482	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea PB	see Table 5	µCi/g	none	unknown	IV	C	
Liquid Scintillation	Tc-99	LA-438-101	PNL-ALO-432	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
GEA	I-129	LA-378-103	PNL-ALO-454	X		see ¹⁵	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5	µCi/g	none	unknown	IV	C	
X-Ray Diff.	Crystalline Compounds	LA-507-151	PNL-ALO-268	X		see ¹⁵	ea smpl	N/A	ea PB	ea AB	see Table 5		none	unknown	IV	C	
Wt% Solids	Wt% Residual solids	LA-504-101	PNL-ALO-501	X		see ¹⁵	ea smpl	N/A	ea PB	ea AB	see Table 5	wt%	none	unknown	IV	C	
Phosphorescence ¹³	U, total	LA-925-107	PNL-ALO-445	X		see ¹⁵	ea smpl	1/mtrx	ea PB	ea AB	see Table 5	µg/g	none	unknown	IV	C	
Density	Density	LA-560-101	PNL-ALO-501	X		see ¹⁵	ea smpl	N/A	N/A	ea AB	see Table 5	g/mL	none	unknown	IV	C	
SECONDARY ANALYSES				SAMPLE ¹		PREP ²	QUALITY CONTROL ³					CRITERIA			FOR-MAT	PRO-GRAM	
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	SOLID COMP ¹⁵	½ SEG SLDG		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴			EXPECTED RANGE ⁴
Distillation ¹⁰	CN	LA-695-102	PNL-ALO-285		X	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III, IV	B
Sep. & a counting ¹¹	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422		X	f	ea smpl	1/mtrx ⁸	ea PB	ea AB	±10	90-110	µCi/g	> 62/density	unknown	I, III, IV	B
ICP ¹¹	Fe	LA-505-151	PNL-ALO-211		X	f,a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	B
ICP ²²	Cr	LA-505-151	PNL-ALO-211		X	f,a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	A

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Table 3: C-103 Chemical, Radiological and Physical Requirements

ICP ^{11,22}	Mn	LA-505-151	PNL-ALO-211		X	f,a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	A,B
ICP ¹¹	U	LA-505-151	PNL-ALO-211		X	f,a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	B
IC ¹⁷	Br	LA-533-105	PNL-ALO-212		X	w	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/g	1200	unknown	I,III,IV	A,B,C
RSST ¹⁰	Energy	see 10 below	N/A		X	d ⁶	ea smpl	N/A	N/A	ea AB	±20	80-120	cal/g ⁵	> 115	unknown	I, III, IV	A,B
Gravimetric ¹⁸	% H2O	LA-564-101	PNL-ALO-504		X	d ⁶	ea smpl	N/A	N/A	ea AB	±20	90-110	wt%	<17	unknown	I, IV	A
IC ²¹	NO2	LA-533-105	PNL-ALO-212		X	w	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV	A
IC ²¹	NO3	LA-533-105	PNL-ALO-212		X	w	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV	A
Titration ²¹	OH	LA-661-103	PNL-ALO-228		X	d	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
Furnace Combustion ¹⁴	TOC	LA-344-105	PNL-ALO-380		X	d	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	> 30,000	unknown	I,III, IV	A

1 ¼ SEG SLDG-¼ segment, sludge; SOLID COMP-composite of the sludge

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

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

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

5 Dry weight basis.

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

7 Action limit is applicable up to 500 °C.

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

9 Serial dilutions or matrix spikes will be performed.

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

11 Performed only if total alpha exceeds notification limit.

12 These analyses are secondary analyses for the safety screening DQO. Therefore, if the DSC limit is exceeded, these analyses must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.

13 If analyses performed at PNL, U-total shall be analyzed by laser fluorimetry.

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

15 For composite analyses, refer to Figure 5

16 This analysis is not required if instrument recalibrations or running the instrument using different parameters are needed to obtain the analyte result.

17 This analysis required if Li exceeds notification limit.

18 This analysis is required if moisture analysis by TGA exceeds notification limit.

19 Duplicate analysis should be performed; however, preparation of special dilution sizes to analyze these components will not be required.

20 No additional standards, spikes, or blanks should be prepared solely for the purpose of obtaining information on this analyte.

21 This analysis is required if the energy equivalent for TOC by hot persulfate is > 125% of the DSC value.

22 This analysis is required if the energy equivalent of the TOC assay by furnace combustion is < 75% of the DSC value.

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Table 3: C-103 Chemical, Radiological and Physical Requirements

Project Name		C-103 Push Mode Core Sample		LIQUID ANALYSES										CHARGE CODES		ORG	REPORTING LEVELS	
Plan Number		WHC-SD-WM-TP-207, REV. 0												TBD	8E200	FORMAT I	Early Notify	
				COMMENTS										TBD	8E300	FORMAT II	Process Control	
PROGRAM CONTACTS				Homogenization Test - Required										TBD	8E400	FORMAT III	Safety Screen	
A. Organic		D. A. Turner		Field Blank - Required										TBD	8E700	FORMAT IV	Waste Management	
B. Safety Screening		H. Babad		Hot Cell Blank - Not Required										TBD	PNL	FORMAT V	RCRA Compliance	
C. Pretreatment		M. J. Kupfer														FORMAT VI	Special	
TWRS		R. D. Schreiber		TANK		#CORES		CORE#										
222-S Laboratory		J. G. Kristofzski		C-103		2		1 and 2										
325 Laboratory		S. G. McKinley																
PRIMARY ANALYSES				SAMPLE ¹		PREP ²	QUALITY CONTROL ³							CRITERIA		FOR-MAT	PRO-GRAM	
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID COMP ¹⁵	FB & S-LEV LIQ		DUP	SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴			
DSC	Energy	LA-514-113	PNL-ALO-508		X	d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	cal/g ⁵	> 115 ⁵	unknown	I, III, IV	A,B	
TGA	% H2O	LA-560-112	PNL-ALO-508		X	d ⁶	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	< 17	unknown	I, III, IV	A,B	
Hot Persulfate	TOC	LA-342-100	PNL-ALO-381	X	X ¹²	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/mL	> 30,000	5,780 to 8,670	I, III, IV	A, B, C	
Sep. & b counting	Sr/Y-90	LA-220-101	PNL-ALO-431 PNL-ALO-433	X		d ⁶	ea smpl	1/mtrx ⁸	ea PB	ea AB	see Table 5		µCi/mL	none	unknown	IV	C	
ICP	Al	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Ba	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Ca	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	4.31 to 6.47	IV	C	
ICP	Cr	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	45.7 to 68.6	IV	C	
ICP	Fe	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	2.59 to 3.88	IV	C	
ICP	K	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	259 to 388	IV	C	
ICP	Li	LA-505-151	PNL-ALO-211		X	d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	±10	90-110	µg/mL	100	unknown	I,III,IV	A,B,C	
ICP	Mg	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Mn	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Na	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	26,200 to 39,300	IV	B	
ICP	P	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	2,030 to 3,040	IV	C	
ICP	S	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Si	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	unknown	IV	C	
ICP	Zr	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl	N/A ⁹	ea PB	ea AB	see Table 5		µg/mL	none	241 to 362	IV	C	
ICP ¹⁶	Ag	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	9.49 to 14.2	IV	C	
ICP ¹⁶	As	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Cd	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	0.69 to 1.03	IV	C	
ICP ¹⁶	Cu	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Mo	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Pb	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Se	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Sr	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
ICP ¹⁶	Zn	LA-505-151	PNL-ALO-211	X		d ⁶	ea smpl ¹⁶	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A		µg/mL	none	unknown	IV	C	
IC	Cl ⁻	LA-533-105	PNL-ALO-212	X		d ⁶	ea smpl	ea smpl	ea PB	ea AB	see Table 5		µg/mL	none	345 to 517	IV	C	
IC	F ⁻	LA-533-105	PNL-ALO-212	X		d ⁶	ea smpl	ea smpl	ea PB	ea AB	see Table 5		µg/mL	none	949 to 1,420	IV	C	
IC	NO ₂ ⁻	LA-533-105	PNL-ALO-212	X		d ⁶	ea smpl	ea smpl	ea PB	ea AB	see Table 5		µg/mL	none	19,800 to 29,800	IV	C	
IC	NO ₃ ⁻	LA-533-105	PNL-ALO-212	X		d ⁶	ea smpl	ea smpl	ea PB	ea AB	see Table 5		µg/mL	none	2,070 to 3,100	IV	C	

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Table 3: C-103 Chemical, Radiological and Physical Requirements

IC	PO ₄ ³⁻	LA-533-105	PNL-ALO-212	X		d ^a	ea smpl	ea smpl	ea PB	ea AB	see Table 5	µg/mL	none	172 to 259	IV	C	
IC	SO ₄ ²⁻	LA-533-105	PNL-ALO-212	X		d ^a	ea smpl	ea smpl	ea PB	ea AB	see Table 5	µg/mL	none	unknown	IV	C	
TIC	CO ₃ ²⁻	LA-622-102	PNL-ALO-381	X		d ^a	ea smpl	N/A	ea AB	ea AB	see Table 5	µg/mL	none	4,140 to 6,210	IV	C	
Sep. & a counting	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	> 62/density	unknown	I, IV	C	
GEA	Am-241	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
GEA	Eu-154	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl	N/A	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
GEA	Cs/Ba-137	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl	N/A	ea PB	ea AB	see Table 5	µCi/mL	none	46.3 to 70	IV	C	
GEA ¹⁸	Co-60	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/mL	none	0.041 to 0.061	IV	C	
GEA ¹⁸	Eu-155	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/mL	none	unknown	IV	C	
GEA ¹⁸	Sb-125	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/mL	none	unknown	IV	C	
GEA ¹⁸	Sn-113	LA-548-121	PNL-ALO-450	X		d ^a	ea smpl ¹⁹	N/A ²⁰	N/A ²⁰	N/A ²⁰	N/A	µCi/mL	none	unknown	IV	C	
Electrode	pH	LA-212-103	PNL-ALO-290	X		d ^a	ea smpl	N/A	N/A	ea AB	see Table 5	pH	none	unknown	IV	C	
Titration	OH-	LA-661-103	PNL-ALO-225	X		d ^a	ea smpl	1/mtrx	ea PB	N/A	see Table 5	µg/mL	none	unknown	IV	C	
Sep. & a counting	Np-237	LA-933-141	PNL-ALO-421 PNL-ALO-425	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
Liquid Scintillation	C-14	LA-348-104	PNL-ALO-474 PNL-ALO-482	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
Liquid Scintillation	Tc-99	LA-438-101	PNL-ALO-432	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
GEA	I-129	LA-378-103	PNL-ALO-454	X		d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	see Table 5	µCi/mL	none	unknown	IV	C	
Visual	Organic Layer	LA-519-151	PNL-ALO-501		X	d ^a	N/A	N/A	N/A	N/A	N/A	none	presence	unknown	I,IV	A	
Density	Density	LA-560-101	PNL-ALO-501	X		d ^a	ea smpl	N/A	N/A	ea AB	±20 80-120	g/mL	none	unknown	IV	C	
SECONDARY ANALYSES				SAMPLE ¹		PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT	PRO-GRAM
METHOD	ANAL.	WHC PROCEDURE	PNL PROCEDURE	LIQUID COMP ¹⁵	FB & S-LEV LIQ		DUP	SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
Distillation ¹⁰	CN	LA-695-102	PNL-ALO-285		X	d ^a	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/mL	> 39,000	unknown	I, III, IV	B
Sep. & a counting ¹¹	Pu-239/240	LA-503-156	PNL-ALO-423 PNL-ALO-422		X	d ^a	ea smpl	1/mtrx ^a	ea PB	ea AB	±15	85-115	µCi/mL	> 62/density	unknown	I, III, IV	B
ICP ¹¹	Fe	LA-505-151	PNL-ALO-211		X	d ^a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	III, IV	B
ICP ²²	Cr	LA-505-151	PNL-ALO-211		X	d ^a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
ICP ^{11,22}	Mn	LA-505-151	PNL-ALO-211		X	d ^a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
ICP ¹¹	U	LA-505-151	PNL-ALO-211		X	d ^a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	III, IV	B
IC ¹⁷	Br	LA-533-105	PNL-ALO-212		X	d ^a	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/mL	1200	unknown	I,III,IV	B
RSST ¹⁰	Energy	see 10 below	N/A			d ^a	ea smpl	N/A	N/A	ea AB	±20	80-120	cal/g ⁵	> 115	unknown	I, III, IV	B
Gravimetric ¹⁸	% H2O	LA-564-101	PNL-ALO-504		X	d ^a	ea smpl	N/A	N/A	ea AB	±20	90-110	wt%	< 17	unknown	I, IV	A
IC ²¹	NO2	LA-533-105	PNL-ALO-212		X	d ^a	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
IC ²¹	NO3	LA-533-105	PNL-ALO-212		X	d ^a	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
Titration ²¹	OH	LA-661-103	PNL-ALO-228		X	d ^a	ea smpl	ea smpl	ea PB	ea AB	±10	90-110	µg/mL	none	unknown	IV	A
Furnace Combustion ¹⁴	TOC	LA-344-105	PNL-ALO-380		X	d ^a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/mL	> 30,000	unknown	III, IV	A

1 LIQ COMP-composite of the liquid, S-LEV LIQ-liquid taken from the segment level, FB-field blank

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

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

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Table 3: C-103 Chemical, Radiological and Physical Requirements

- 4 Units for notification limits and expected range are those listed in the "units" column.
- 5 Dry weight basis.
- 6 Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.
- 7 Action limit is applicable up to 500 °C.
- 8 Tracer or carrier may be used in place of a spike and results corrected for recovery.
- 9 Serial dilutions or matrix spikes will be performed.
- 10 These analyses required if DSC exceeds notification limits. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.
- 11 Performed only if total alpha exceeds notification limit.
- 12 These analyses are secondary analyses for the safety screening DQO. Therefore, if the DSC limit is exceeded, these analyses must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.
- 13 If analyses performed at PNL, U-total shall be analyzed by laser fluorimetry.
- 14 This analysis required if the energy equivalent of the TOC by hot persulfate is < 75% of the DSC value.
- 15 For composite analyses, refer to Figure 6
- 16 This analysis is not required if instrument recalibrations or running the instrument using different parameters are needed to obtain the analyte result.
- 17 This analysis required if Li exceeds notification limit.
- 18 This analysis is required if moisture analysis by TGA exceeds notification limit.
- 19 Duplicate analysis should be performed; however, preparation of special dilution sizes to analyze these components will not be required.
- 20 No additional standards, spikes, or blanks should be prepared solely for the purpose of obtaining information on this analyte.
- 21 This analysis is required if the energy equivalent for TOC by hot persulfate is > 125% of the DSC value.
- 22 This analysis is required if the energy equivalent of the TOC assay by furnace combustion is < 75% of the DSC value.

Figure 5: Analytical Scheme for Solid Composite

(a) Required metals from ICP - Al, Ba, Bi, Ca, Ce, Cr, Fe, K, Mg, Mn, Na, Ni, La, P, S, Si, Zr

(b) Required Anions from IC - PO₄, SO₄, NO₃, NO₂, Cl, F

(c) Required Analytes by GEA - Cs/Ba-137, Eu-154, Am-241

NOTE: * Perform fusion of residual solids unless judged that insufficient solids remain after water digestion. If little or no solids remain, the fusion dissolution of entire sample will be used for material balance calculations

Figure 6: Analytical Scheme for Liquid Composite

(a) Required metals from ICP - Al, Ba, Ca, Cr, Fe, K, Mg, Mn, Na, P, S, Si, Zr

(b) Required Anions from IC - PO₄, SO₄, NO₃, NO₂, Cl, F

(c) Required Analytes by GEA - Cs/Ba-137, Eu-154, Am-241

5.0 ORGANIZATION

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

Table 4: Tank C-103 Project Key Personnel List

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

6.0 EXCEPTIONS, CLARIFICATIONS AND ASSUMPTIONS

6.1 EXCEPTIONS TO DQO REQUIREMENTS

In the Safety Screening DQO, it is specified that cyanide analyses are to be run on a quarter-segment level and that the notification limit for the DSC analysis is 125 cal/g. However, both the soon to be released revision of the Safety Screening DQO and the Ferrocyanide DQO (Meacham et al. 1994) have changed the requirements such that the cyanide analysis is now to be run on a half-segment level and the DSC notification limit is 115 cal/g (dry weight basis). Therefore, although this Tank Characterization Plan uses the current Safety Screening DQO, it specifies that cyanide is to be run on a half-segment basis and that notification shall be made if the DSC value exceeds 115 cal/g (dry weight basis).

In the organic DQO, Cs¹³⁷ and Sr⁹⁰ on half-segments are requested as secondary analyses. However, these have not been requested as primary analyses, nor are there any other primary analyses that would require Cs¹³⁷ or Sr⁹⁰ to be conducted should it exceed a notification limit. Therefore, it has been decided by both the Organic Safety Program and the Characterization Program that Cs¹³⁷ and Sr⁹⁰ on a half-segment level should not be analyzed.

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

6.2 CLARIFICATIONS AND ASSUMPTIONS

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

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

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

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

- The safety screening DQO (Babad and Redus 1994) requires that additional analyses be performed if total alpha activity measures greater than 1 g/L. Total alpha is measured in $\mu\text{Ci/g}$ rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it was assumed that all alpha decay originates from Pu-239. The notification limit may then be calculated as shown in equation (2):

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

- Neither the safety screening nor the organic DQO, upon which some of the analyses in Table 3 are based, sufficiently addresses the analyses of any drainable liquid present. To adequately characterize the tank, all analyses performed on the solids for the safety screening and organic DQOs shall also be performed on any drainable liquids and the field blank.
- The Pretreatment Program has requested 200 mL of the solid composite material for process development work. Two test plans (Beahm 1994; Lumetta 1994) will be used to guide this process development work. Since the Characterization Program is responsible for the taking of tank samples, the Characterization Program will need to approve the test plans. This approval will not only ensure that the DQO process has been used in the generation of the test plans and that there is justification for the samples, but also that the facility receiving the sample is in a position to adequately handle radioactive material. At such time that the test plans are approved by the Characterization Program, the Characterization Program will direct the performing laboratory, via letter of instruction, to allow shipment of the sample material to Oak Ridge National Laboratory and Pacific Northwest Laboratory.

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

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

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

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

- If the energy equivalent for TOC by hot persulfate is $\leq 75\%$ of the DSC value, run TOC by furnace combustion on the half-segment.
- If the energy equivalent for TOC by hot persulfate is $\geq 125\%$ of the DSC value, run nitrite, nitrate, and hydroxide analyses on the half-segment.
- If the energy equivalent of the TOC assay by furnace combustion is $\leq 75\%$ of the DSC, analyze for the presence of Mn and Cr on the half-segment.
- In the pretreatment DQO the achieved precision and accuracy are listed at two concentrations: greater than the minimum detectable quantity detection limits and near (< 10 times) the minimum detectable quantity. Table 5 is a list of the Pretreatment precision and accuracy requirements.

Table 5: Required Accuracy and Precision for Pretreatment Sample Analytes

Analyte	Sample Accuracy in Percent	Sample Precision in Percent*
Al	20 ⁽¹⁾	20
	50 ⁽²⁾	40
Ba	40	40
	80	80
Bi	50	20
	120	50
Ca	20	20
	50	50
Ce	10	30
	20	40
Cr	20	20
	40	50
Fe	50	30

Analyte	Sample Accuracy in Percent	Sample Precision in Percent*
	50	60
K	20	20
	40	40
La	10	20
	50	70
Mg	20	30
	30	100
Mn	50	30
	70	100
Na	20	20
	20	30
P	Presence/absence only	Presence/absence only
	Presence/absence only	Presence/absence only
S	Presence/absence only	Presence/absence only
	Presence/absence only	Presence/absence only
Ni	20	10
	30	50
Si	20	30
	30	80
Zr	20	30
	50	60
Cl	20	20
	50	30
F	30	30
	70	60
SO4	20	20
	60	30
NO3	20	20
	50	30
NO2	20	20
	50	30
PO4	20	20
	50	50
CO3	20	20

Analyte	Sample Accuracy in Percent	Sample Precision in Percent*
	50	50
TOC	50	50
	100	100
acid H+ (pH)	10	20
	50	100
Hydroxide OH-	10	20
	50	100
U (total)	20	20
	60	60
¹⁴ C	40	50
	50	200
⁹⁹ Tc	60	20
	100	50
¹²⁹ I	60	100
	100	100
¹³⁷ Cs	10	20
	40	60
⁹⁰ Sr	20	30
	30	60
¹⁵⁴ Eu	10	20
	40	60
^{239/240} Pu	20	40
	40	70
²⁴¹ Am	20	30
	50	80
²³⁷ Np	60	40
	140	80

- (1) The first row for an analyte lists the accuracy and precision at high concentrations (>10 times the detection limit).
- (2) The second row gives the values at low concentrations (<10 times the detection limit).

* Precision requirements are based on relative percent difference between duplicates rather than standard deviation of main value.

Several ICP and GEA analyses are listed in the Pretreatment DQO and Table 3 for which the Pretreatment Program has requested information without additional quality

assurance/quality control requirements (see footnotes, Table 3). These results will not be used in any of the Pretreatment decision rules; rather, this information will assist in detecting unexpected analyte concentrations, and could indicate the need for obtaining more extensive analyses from a core at a later date. The Pretreatment Program has requested a reporting of the instrument results for these analyses, with the following caveats:

- If instrument recalibration or running the instrument using different parameters is required to obtain the analyte result, the additional analysis is not required.
- No additional standards, spikes, or blanks should be prepared solely for the purpose of obtaining information on the these additional analytes.
- Duplicate analyses should be performed; however, preparation of special dilution sizes to analyze these components will not be required.

In the Pretreatment DQO, Sn-123 by GEA is listed as one of the additional analytes for which no quality assurance/quality control is required. However, Sn-123 is not an isotope obtained by GEA, although Sn-113 is such an analyte. Therefore, it is assumed in this Tank Characterization Plan that Sn-113 rather than Sn-123 is the analyte of interest.

7.0 DELIVERABLES

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

7.1 PROGRESS REPORTS

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

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

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

Schedule reporting shall consist of the following:

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

7.2 FORMAT I REPORTING

Table 3 contains the notification limits for each analyte. Any results exceeding their notification limits shall be reported by calling the East Tank Farm Operations Shift Manager at 373-2689 and the Characterization Program (Schreiber 1994b). This verbal notification must be followed within 1 working day by written communication to D. A. Turner of the Organic Tank Safety Program, J. L. Deichman of Analytical Services, R. D. Schreiber of Characterization Support, S. J. Eberlein, H. Babad and D. R. Bratzel of the Characterization Program, and N. W. Kirch of Data Trending, Interpretation, and Reporting, documenting the observations. Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above either by a revision to this document, by a letter of instruction, or by a memorandum of understanding.

7.3 FORMAT III REPORTING

A Format III report, reporting the results of the primary safety screen analyses, shall be issued to D. A. Turner, H. Babad, R. D. Schreiber, S. J. Eberlein, D. R. Bratzel, P. Sathyanarayana, and N. W. Kirch within 45 days of receipt of the last sample at the laboratory loading dock. Although normally raw data would not be attached to this type of report, the DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analyses for the safety screening secondary analytes are required, these results shall be provided within 90 days of receipt of the last sample at the laboratory loading dock. No calibration data are requested for

these reports. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

7.4 FORMAT IV REPORTING

Analytical results requested for the characterization project of tank C-103 shall be compiled into a Format IV type data package. The data package shall be provided to Analytical Services and the Characterization Program within 216 days of the sampling event. Detailed information regarding the contents of this reporting format are given in (Schreiber 1994a).

In addition to this data package, an electronic version of the analytical results shall be provided to Characterization Support for entry in the Tank Characterization Database. The data must be available to the Washington State Department of Ecology within 216 days of the sampling event, so this electronic copy must be sent at the time of data package delivery or within 209 days of the sampling event, whichever is earlier, to allow time for data entry. The electronic version shall be in the standard electronic format specified in (Bobrowski 1994).

8.0 CHANGE CONTROL

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

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

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