SEP 14 1995 Sta. 4

ENGINEERING DATA TRANSMITTAL

1. EDT No. 613117

2. To: (Receiving Organization)	3. From: (Originating Organization)	4. Related EDT No.:
Distribution	Characterization Plans, Coordination and Reports	N/A
5. Proj./Prog./Dept./Div.:	**6. Cog. Engr.:**	**7. Purchase Order No.:**
Tank 241-BY-104/Waste Management/CPCR/Technical Basis Characterization	Cheryl J. Benar	N/A
8. Originator Remarks: This document is being released into the supporting document system for retrievability purposes.		**9. Equip./Component No.:** N/A
		10. System/Bldg./Facility: N/A
11. Receiver Remarks: For release.		**12. Major Assm. Dwg. No.:** N/A
		13. Permit/Permit Application No.: N/A
		14. Required Response Date: 09/07/95

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	WHC-SD-WM-TSAP-040	N/A	0	Tank 241-BY-104 Rotary Core Sampling and Analysis Plan	Q	2	1	1

16. KEY

Approval Designator (F)	Reason for Transmittal (G)	Disposition (H) & (I)
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION (See Approval Designator for required signatures)

(G) Reason	(H) Disp.	(J) Name (K) Signature (L) Date (M) MSIN	(J) Name (K) Signature (L) Date (M) MSIN	(G) Reason	(H) Disp.
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18.	19.	20.	21. DOE APPROVAL (if required) Ctrl. No.
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BD-7400-172-2 (04/94) GEF097

RELEASE AUTHORIZATION

Document Number:	WHC-SD-WM-TSAP-040, REV 0
Document Title:	Tank 241-BY-104 Rotary Core Sampling and Analysis Plan
Release Date:	9/14/95

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

APPROVED FOR PUBLIC RELEASE

WHC Information Release Administration Specialist:

Kara M. Broz 9/14/95

Kara M. Broz

SUPPORTING DOCUMENT

1. Total Pages 17

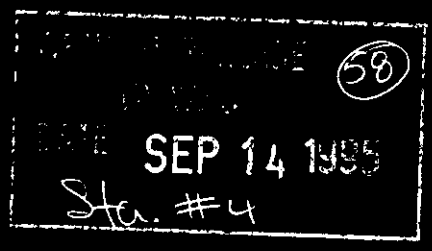
2. Title	3. Number	4. Rev No.
Tank 241-BY-104 Rotary Core Sampling and Analysis Plan	WHC-SD-WM-TSAP-040	0

5. Key Words	6. Author
Tank 241-BY-104, Tank BY-104, BY-104, BY Farm, Rotary Core, Sampling, Analysis, Plan	Name: Cheryl J. Benar Signature: Cheryl J. Benar Organization/Charge Code 75310/MDR21

7. Abstract

N/A

8. RELEASE STAMP



WHC-SD-WM-TSAP-040
Revision 0

Tank 241-BY-104 Rotary Core Sampling and Analysis Plan

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

by

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LIST OF ABBREVIATIONS

BY-104	Tank 241-BY-104
cm	centimeters
DQO	data quality objective
DSC	differential scanning calorimetry
GEA	gamma energy analysis
HPGE/MCA	high purity germanium - multi channel analysis

LIST OF ABBREVIATIONS (Cont.)

IC	ion chromatography
ICP	inductively coupled plasma - atomic emission spectroscopy
LiBr	lithium bromide
RSST	reactive system screening tool - adiabatic calorimetry
SAP	Sampling and Analysis Plan
SARP	Safety and Analysis Report for Packaging
TCP	Tank Characterization Plan
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
WHC	Westinghouse Hanford Company

1.0 SAMPLING AND ANALYSIS OBJECTIVES

This Sampling and Analysis Plan (SAP) will identify characterization objectives pertaining to sample collection, laboratory analytical evaluation, and reporting requirements for vapor samples and two rotary-mode core samples from tank 241-BY-104 (BY-104). It is written in accordance with the *Tank Safety Screening Data Quality Objective* (Babad et al. 1995), *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objective Process* (Meacham et al. 1995), *Strategy for Sampling Hanford Site Tank Wastes for Development of Disposal Technology* (Kupfer et al. 1995), *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Buckley and Baide 1995), *Test Plan for Samples From Hanford Waste Tanks 241-BY-103, BY-104, BY-105, BY-106, BY-108, BY-110, TY-103, U-105, U-107, U-108, and U-109* (Meacham 1995), and *Historical Model Evaluation Data Requirements* (Simpson, McCain 1995). These data quality objectives (DQOs) are described in the Tank Characterization Plan (TCP) for BY-104 (Schreiber 1994d). This SAP will also identify procedures and requirements for collecting and characterizing vapor and rotary-mode core samples from BY-104.

2.0 SAMPLING EVENT REQUIREMENTS

As of May 31, 1995, tank BY-104 contained 1,540 kL of noncomplexed waste corresponding to a waste depth of 394 cm. The tank contents were comprised of 1,390 kL of saltcake and 150 kL of sludge with no pumpable liquid remaining (Hanlon 1995).

Prior to core sampling, the dome space (below the riser) shall be sampled and analyzed for the presence of flammable gases. The sample shall be taken from within 3 feet of the waste surface and the data reported as a percentage of the LFL. The results shall be submitted to the project coordinator within one week of the sampling event. The necessity for recurring sampling of tanks for flammable gas concentrations and the frequency of such sampling will be determined by the Flammable Gas Program.

Tank BY-104 is currently scheduled to be core sampled using a rotary-mode core sampling truck. At least two samples are expected to be taken from risers 11 and 13. If a different riser is necessary to meet sampling and analysis requirements, this change must be recorded and approved by the sampling cognizant engineer before sampling. The risers used may be recorded on a permanent data sheet or recorded directly in the work package.

Nine segments per core are expected to be taken from tank BY-104. Segment 1 is expected to be 8 cm in depth, and segments 2 - 9 are expected to be 48 cm in depth. The sampling objective is to obtain a vertical profile of the waste; therefore, more or less segments may need to be taken depending on the accuracy of the current waste volume records. For detailed information regarding the sampling activities, refer to work package ES-95-0276. This document contains the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank BY-104 shall be obtained by filling a sampler with deionized water. This field blank is to accompany the samples to the laboratory hot cell. All collected samples and the field blank shall be shipped to the laboratory following the *Load/Transport Sample Cask(s)* procedure (TO-080-090). Core samples shall be transported to the laboratory within three calendar days from the time each segment is removed from the tank.

Occasionally, lithium bromide (LiBr) solution may be used to aid in the collection of the core samples. If LiBr solution is used, Sampling Operations must state this in the chain-of-custody form that accompanies the sample to the laboratory, and must provide a LiBr solution blank to the laboratory. The LiBr solution blank shall consist of a container filled with LiBr solution from the same batch of LiBr solution used during the sampling. This blank shall be analyzed for lithium and bromide in order to determine the concentration of the tracer at the time the core was taken. Only one LiBr solution blank per tank is required.

3.0 LABORATORY ANALYSIS REQUIREMENTS

3.1 ANALYSIS SCHEME

In order to comply with the safety screening, organic, organic test plan, ferrocyanide, and historical DQO documents, the following steps shall be performed on each sample:

- Extrude segment.
- Analyze drainable liquids and quarter-segment and/or half-segment subsamples from each segment, and solid composite samples from each core.
- Filter liquids prior to analysis.
- Remove 125 mL of solid composite for process development work.
- Archive at least 10 mL of each subsegment or drainable liquid from each segment.
- Archive 100 mL of solid and liquid composite for the Pretreatment Program.

As a precautionary measure, the Safety and Analysis Report for Packaging (SARP) in the *Load/Transport Sample Cask(s)* procedure (TO-080-090) has been reviewed for any safety issues involved with transportation of tank BY-104 core samples. **For core samples from tank BY-104, the shipping container must be vented every 47 days to release any accumulated gas.**

Any decisions, observations, or deviations made to this work plan or during the sample breakdown and analyses shall be documented in writing with justification. These decisions and observations shall also be reported in the data report. The reporting formats for analyses are contained in Table 1.

3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Table 1 to be performed on tank BY-104 core samples are based on the safety screening, organic, organic test plan, historical, and ferrocyanide DQO documents referenced

in Section 1.0. The laboratory procedure numbers, which shall be used for the analyses, are included in the table.

3.3 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from samples taken from a tank is insufficient to perform the analyses requested in the respective SAP and permit a minimum 10 mL archive per sample, the laboratory shall notify the Tank Cognizant Engineer within one working day. At that time, a prioritization of the analyses may be provided to the laboratories. Any analyses prescribed by the SAP, but not performed, shall be identified in the appropriate data report with justification for non-performance.

4.0 QUALITY ASSURANCE CONTROL

4.1 LABORATORY OPERATIONS

Laboratories performing analysis in support of this Tank Sampling and Analysis Plan shall have approved and implemented quality assurance project plans. These QA plans shall meet the *Hanford Analytical Services Quality Assurance Plan* (DOE 1995) minimum requirements as the baseline for laboratory quality systems. Quality requirements for conducting Characterization Project sampling and analysis are described in *TWRS Characterization Program Quality Assurance Program Plan* (Whelan 1994), *Fiscal Year 1995 Tank Waste Remediation System Tank Waste Analysis Plan* (Haller 1994), and in this SAP. Characterization Project sampling and analysis shall be conducted in conformance with these quality requirements.

Sample quality control (duplicates, spikes, standards) are identified in Table 1. If no criteria are provided, the performing laboratory shall perform to its quality assurance plan.

4.2 SAMPLE COLLECTION

Before sampling can be performed on a tank, available risers must be identified for use in the sampling event. The selected risers must be inspected and prepared to confirm their ability to be used in sampling. Safety hazards must be identified and special precautions must be made if needed. If deemed necessary by the sampling and tank cognizant engineers, video surveillance should be performed to identify any potential problems that may occur during the sampling event.

Samples are to be taken from a tank and shipped to the performing laboratory by Sampling Operations in accordance with the respective work package. That work package shall also initiate the chain-of-custody for the samples. Approved procedure TO-080-090 ("Load/Transport Sample Cask(s)") is 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

Table 1: Tank BY-104 Chemical, Radiological, and Physical Analytical Requirements

SOLID ANALYSES

Project Name	BY-104 Rotary Mode Core Sample	COMMENTS	REPORTING LEVELS
Plan Number	WHC-SD-WM-TSAP-040, REV. 0	Homogenization Test - Per Laboratory Discretion	FORMAT I: Early Notify
		Field Blank - Required	FORMAT II: Process Control
PROGRAM	PROGRAM CONTACTS	Hot Cell Blank - Per Laboratory Discretion	FORMAT III: Safety Screening
A. Safety Screening	Safety Screening E. J. Lipke	LiBr Solution Blank - Required	FORMAT IV: Waste Management
B. Ferrocyanide	TWRS K. E. Bell	TANK: BY-104; #CORES: 2	FORMAT V: RCRA Compliance
C. Organic			FORMAT VI: Special
D. Historical Model			
E. Organic Test Plan			

PROGRAM	PRIMARY ANALYSES: METHOD	ANAL.	WHC PROCEDURE	SAMPLE[1]: ¼ SEG SLDG	½ SEG SLDG	½ SEG SC	CORE COMP	PREP[2]	QUALITY CONTROL[3]: DUP	SPIKE	BLK	STD	PR	AC	CRITERIA: UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	REPORT
A,B,C,D,E	DSC	Energy	LA-514-113	X		X		d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g	≥ 480[4]	0 - 250	I, III, IV
A, B, C, D	TGA	% H_2O	LA-560-112	X		X		d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	20 - 30	I, III, IV
A, B	Distillation	^-CN [7]	LA-695-102	X		X		d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	40 - 90	I, III
A	α counting	Total Alpha	LA-508-101	X[11]				f or a	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	0.1 - 0.4	I, III
C	Persulfate	TOC[12]	LA-342-100		X			d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	> 30,000	10E3 - 12E3	I, III, IV
D	IC	full suite	LA-533-105		X	X	X	w	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	varies	IV
D	GEA	^{137}Cs	LA-548-121		X	X	X	f	ea smpl	N/A	ea PB	ea AB	±10	90-110	µCi/g	none	100 - 400	IV
E	RSST	Energy[17]	see [8]	X				d	N/A	N/A	N/A	ea AB	N/A	90-110	J/g	none	>480	IV
D	β counting	Sr-90	LA-220-101		X		X	f or a	ea smpl	see [5]	ea PB	ea AB	CL	CL	µCi/g	none	125 - 185	IV
A	Gravimetry	bulk density	LO-160-103		X		X	d	N/A	N/A	N/A	ea AB	N/A	90-110	g/mL	none	1.0 - 1.5	III
A, B, C, D	ICP	Al, Ca, Cr, Bi, Fe, Li[10], Mn, Na, Ni, P, Si, U	LA-505-151	X		X	X	f[16]	ea smpl	see [6]	ea PB	ea AB	±10	90-110	µg/g	Li > 100, Ni > 8,000	varies	I, III, IV

PROGRAM	SECONDARY ANALYSES: METHOD	ANAL.	WHC PROCEDURE	SAMPLE[1]: ¼ SEG SLDG	½ SEG SLDG	½ SEG SC	CORE COMP	PREP[2]	QUALITY CONTROL[3]: DUP	SPIKE	BLK	STD	PR	AC	CRITERIA: UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	REPORT
A	α counting	$^{239/40}Pu$[13]	LA-503-156	X				f	ea smpl	see [5]	ea PB	ea AB	±10	90-110	µCi/g	> 41	0.05 - 0.10	I, III
D	β counting	Total Beta[14]	LA-508-101				X	f or a	ea smpl	N/A	ea PB	ea AB	CL	CL	µCi/g	none	150 - 250	IV
D	Florimetry	Total U[14]	LA-925-007		X			f	ea smpl	1/mtrx	ea PB	ea AB	CL	CL	µg/g	none	1500 - 4000	IV
C	Gravimetry	% H_2O[15]	LA-564-101		X	X		d	ea smpl	N/A	N/A	ea AB	±20	80-120	wt%	none	20 - 30	I, IV
C	Titration	^-OH [16]	LA-661-103		X			d	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV
A, C	IC	$^-NO_2$, ^-Br [9]	LA-533-105	X				w	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	Br > 1200	varies	I, III
D	ICP	full suite[14]	LA-505-151				X	w	ea smpl	see [6]	ea PB	ea AB	CL	CL	µg/g	none	varies	IV
A, B	RSST	Energy[7]	see [8]	X		X		d	N/A	N/A	N/A	ea AB	N/A	90-110	J/g	480[4]	>480	III, IV
B	Light Scattering	Particle Size[7]	LT-519-101	X				d	N/A	N/A	ea AB	ea AB	N/A	70-130	J/g	none	unknown	IV
B	Persulfate	TC[7]	LA-342-100	X				d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	none	unknown	IV

Table 1: Tank BY-104 Chemical, Radiological, and Physical Analytical Requirements

[1]¼ SEG SLDG-¼ segment, sludge; ½ SEG SC-½ segment saltcake

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

[3]PR-precision, AC-accuracy, ea-each, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, AB-analytical batch, PB-preparation blank, mtrx-matrix, CL-control limit.

[4]Dry weight basis.

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

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

[7]Analysis required if DSC exceeds notification limits.

[8]The RSST method may be found in WHC-SD-WM-TP-104.

[9]Br performed if total alpha exceeds notification limit, NO_2 performed if TOC > 125% of the DSC value.

[10]If the chain-of-custody form indicates that LiBr solution was used to obtain the segment, Li analysis is to be performed on that segment.

[11]Analyze only the bottom quarter segment of each segment extruded.

[12]This is a primary analysis for the organic DQO, but is also a secondary analyses for the safety screening DQO. Therefore, if the DSC limit is exceeded, this analysis must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.

[13]Performed if total alpha exceeds notification limit.

[14]Performed if historical model is verified by primary analyses.

[15]This analysis required if moisture analysis by TGA exceeds notification limit.

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

[17]This analysis is to be run on the quarter segment sludge, or half segment saltcake, that exhibits the highest DSC value for each core.

[18]Non-nickel crucible required.

Table 1: Tank BY-104 Chemical, Radiological, and Physical Analytical Requirements

LIQUID ANALYSES

Project Name	BY-104 Rotary Mode Core Sample	COMMENTS	REPORTING LEVELS
Plan Number	WHC-SD WM-TSAP-040, REV. 0	Homogenization Test - Per Laboratory Discretion	FORMAT I: Early Notify
		Field Blank - Required	FORMAT II: Process Control
PROGRAM	PROGRAM CONTACTS	Hot Cell Blank - Per Laboratory Discretion	FORMAT III: Safety Screening
A. Safety Screening	Safety Screening E. J. Lipke	LiBr Solution Blank - Required	FORMAT IV: Waste Management
B. Ferrocyanide	TWRS K. E. Bell	TANK: BY-104 / #CORES: 2	FORMAT V: RCRA Compliance
C. Organic			FORMAT VI: Special
D. Test Plan			

PROGRAM	PRIMARY ANALYSES: METHOD	ANAL.	WHC PROCEDURE	SAMPLE[1]: FB & S-LEV LIQ	PREP[2]	QUALITY CONTROL[3]: DUP	SPIKE	BLK	STD	PR	AC	CRITERIA: UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	REPORT
A, B, C, D	DSC	Energy[11]	LA-514-113	X	d[5]	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g	≥ 480	unknown	I, III, IV
A,B,C	TGA	% H_2O	LA-560-112	X	d[5]	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	70 - 85	III, IV
A	ICP	Li[9]	LA-505-151	X	d[5]	ea smpl	see[6]	ea AB	ea AB	±10	90-110	µg/mL	Li > 100[8]	0 - 1200	I, III
B	Distillation	CN[10]	LA-695-102	X	d[5]	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µCi/g	>39,000[8]	20 - 50	I, III
C	Persulfate	TOC[11]	LA-344-100	X	d[5]	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/mL	> 30,000[8]	2000 - 4000	I, III, IV
A, C	Visual	Organic Layer	LA-519-151	X	d[5]	N/A	N/A	N/A	N/A	N/A	N/A		presence	n/a	I, III, IV

PROGRAM	SECONDARY ANALYSES: METHOD	ANAL.	WHC PROCEDURE	SAMPLE[1]: FB & S-LEV LIQ	PREP[2]	QUALITY CONTROL[3]: DUP	SPIKE	BLK	STD	PR	AC	CRITERIA: UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	REPORT
C	Gravimetric	% H_2O[15]	LA-564-101	X	d[5]	ea smpl	N/A	N/A	ea AB	±20	80-120	µg/mL	none	70 - 85	I, IV
C	Titration	^{-}OH [13]	LA-166-103	X	d[5]	ea smpl	N/A	ea AB	ea AB	±20	80-120	µg/mL	none	1.6E5 - 2.5E5	IV
A, C	IC	$^{-}NO_2$ [13] Br^{-}[7]	LA-533-105	X	w	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/mL	Br > 1,200[8]	0 - 2500	I, III
B, C	ICP	Al[10,14] Ca, Fe, P, Na, Bi, Cr, Mn	LA-505-151	X	d	ea smpl	see[6]	ea PB	ea AB	±10	90-110	µg/mL	none	varies	IV
B	Persulfate	TC[10]	LA-342-100	X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	none	unknown	IV
A, B, C	Furnace Oxidation	TOC[10, 12]	LA-344-105	X	d[5]	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg C/mL	> 30,000	2000 - 4000	I, III

Table 1: Tank BY-104 Chemical, Radiological, and Physical Analytical Requirements

[1]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 and matrix spike duplicate, AB-analytical batch, PB-preparation blank, N/A-not

[4]Dry weight basis.

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

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

[7]Performed only if Li exceeds notification limit.

[8]Converted from µg/g limit assuming a liquid density of 1.0 g/mL.

[9]If the chain-of-custody form indicates that LiBr solution was used to obtain the segment, Li analysis is to be performed on that segment.

[10]Perform this analysis if DSC is $\geq$ 480 J/g

[11]These analyses are primary analyses for the organic DQO, but also are secondary analyses for the ferrocyanide and safety screening DQOs. Therefore, if the DS is exceeded, these analyses must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.

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

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

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

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

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 sample collected to the performing laboratory within one working day of removing the sample from the tank, but must transport each segment or sample within three calendar days. Sampling Operations is responsible for verbally notifying the WHC 222-S Laboratory (373-2435) at least 24 hours in advance of an expected shipment.

4.3 SAMPLE CUSTODY

The chain-of-custody form is initiated by the sampling team as described in the work package. 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.

5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

5.1 EXCEPTIONS TO DQO REQUIREMENTS

If necessary, the Pretreatment Program will contact the Characterization Project and request analyses on archived composite samples. Therefore, the Pretreatment Program has requested that a 125 mL composite sample for process development and a 100 mL composite sample for archive shall be obtained from this sampling event (Slankas 1994).

Requirements in the organic DQO for analyses of 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.

The ferrocyanide DQO lists cyanide as a secondary analyte, however discussions with the Ferrocyanide Program indicate that cyanide should be a primary analyte.

5.2 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 1. Each of these issues is discussed below:

- Any exothermic reaction (in cal/g or J/g) determined by differential scanning calorimetry (DSC) must be reported on a dry weight basis as shown in equation (1) using the weight percent water determined from thermogravimetric analysis (TGA).

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

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

- The safety screening DQO (Babad et al. 1995) requires that additional analyses be performed if total alpha activity is greater than 1 g/L. Total alpha is measured in μCi/g rather than g/L. To convert the notification limit for total alpha into a number more readily usable by the laboratory, it was assumed that all alpha decay originates from Pu-239. The notification limit may then be calculated as shown in equation (2):

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

NOTE: Samples measured for total alpha shall also be measured for density. The notification limit shall be 41 μCi/g unless the measured density exceeds 1.5 g/mL when the notification limit will be adjusted according to equation 2.

- The Pretreatment Program has requested 125 g of the solid composite material for process development work. Two test plans (Lumetta 1994, and Temer 1994) will be used to guide this process development work. The Characterization Project will direct the performing laboratory, via letter of instruction, to allow shipment of the sample material to Oak Ridge National Laboratory and/or Battelle Pacific Northwest Laboratory.

- Secondary analyses required per the organic DQO are run depending on the comparison between the value of the energy equivalent for TOC and the DSC energetics value. The energy equivalent of TOC, X, is given in equation (3).

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

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.

6.0 DELIVERABLES

All analyses will be reported as Format I, III, or IV as indicated in Table 1. Additional information regarding reporting formats is given in (Schreiber 1994a).

6.1 FORMAT I REPORTING

Table 1 contains the notification limits for each analyte. Any results exceeding their notification limits shall be reported via telephone by the 222-S Laboratory Shift Manager to the East Tank Farm Operations Shift Manager as soon as the data are obtained and reviewed (Kristofzski 1995). This verbal notification must be followed within one working day by written communication to the Safety Screening Representative, Process Control, and the Characterization Project Office documenting the observations (Schreiber 1994c). Additional analyses for verification purposes may be contracted between the performing laboratory and the contacts above by a revision to this document, or by a memorandum of understanding.

6.2 FORMAT III REPORTING

A Format III report, giving the results of the primary analyses required by the safety screening DQO, shall be issued to the Safety Screening Representative, the Characterization Project, Process Control, the Tank Characterization Resource Center, Tank Characterization Database, and Historical DQO points of contact (Schreiber 1994c) within 45 days of receipt of the last sample at

the laboratory loading dock. The DSC and TGA scans have been requested due to the interpretive nature of the analysis. If analyses for the safety screening secondary analytes are required, these results shall be provided within 90 days of receipt of the last 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).

6.3 FORMAT IV REPORTING

This report shall include the results of the analyses required by the historical DQO (see Table 1 for delineation of program requests). The data package shall be provided to the Characterization Project, the Historical DQO point of contact, the Tank Characterization Resource Center, and Tank Characterization Database representatives within 105 days of the sampling event. Detailed information regarding the contents of this reporting format are given in Schreiber (1994a).

In addition to this data package, an electronic version of the analytical results shall be provided to the Tank Characterization Database representative. 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 et al. (1994).

7.0 CHANGE CONTROL

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

At the request of the Characterization Program, additional analysis of sample material from this characterization project shall be performed following a revision of this Sampling and Analysis Plan.

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