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TANK 241BY106 ROTARY CORE SAMPLING & ANALYSIS PLAN

OCT 27 1995

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Page 1 of 1

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2	1	Cog.Eng. K.E. Bell	<i>Kevin Bell</i>	10/19/95	
2	1	Cog. Mgr. J.G. Kristofzski	<i>J.G. Kristofzski</i>	10/27/95	
2	1	QA T.C. Triple	<i>T.C. Triple</i>	10/24/95	
		Safety			
		Env.			
		Ops J.S. Lee	<i>J.S. Lee</i>	10/20/95	
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A.E. Young <i>A.E. Young</i> 10/18/95 Signature of EDT Date Originator		N/A Authorized Representative Date for Receiving Organization		J.G. Kristofzski <i>J.G. Kristofzski</i> 10/27/95 Cognizant Manager Date	
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Tank 241-BY-106 Rotary Core Sampling and Analysis Plan

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

TABLE OF CONTENTS

1.0 SAMPLING AND ANALYSIS OBJECTIVES	1
2.0 SAMPLING EVENT REQUIREMENTS	1
3.0 LABORATORY ANALYSIS REQUIREMENTS	2
3.1 ANALYSIS SCHEME	2
3.2 SPECIFIC METHODS AND ANALYSES	3
3.3 INSUFFICIENT SEGMENT RECOVERY	3
4.0 QUALITY ASSURANCE CONTROL	10
4.1 LABORATORY OPERATIONS	10
4.2 SAMPLE COLLECTION	10
4.3 SAMPLE CUSTODY	10
5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS	11
6.0 DELIVERABLES	12
6.1 FORMAT I REPORTING	12
6.2 FORMAT III REPORTING	13
6.3 FORMAT IV REPORTING	13
7.0 CHANGE CONTROL	13
8.0 REFERENCES	14

LIST OF TABLES

1: Tank BY-106 Chemical, Radiological, and Physical Analytical Requirements	4
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LIST OF ABBREVIATIONS

BY-106	Tank 241-BY-106
cm	centimeters
DQO	data quality objective
DSC	differential scanning calorimetry
GEA	gamma energy analysis
HPGE/MCA	high purity germanium - multi channel analysis
IC	ion chromatography
ICP	inductively coupled plasma - atomic emission spectroscopy
LiBr	lithium bromide
RSST	reactive system screening tool - adiabatic calorimetry

LIST OF ABBREVIATIONS (Continued)

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 one rotary-mode core sample from tank 241-BY-106 (BY-106). It is written in accordance with the *Tank Safety Screening Data Quality Objective* (Hunt 1995), *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objective Process* (Meacham et al. 1995), *Data Quality Objective to Support Resolution of the Complexant Safety Issue* (Turner 1995), *Historical Model Evaluation Data Requirements* (Simpson, McCain 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 *Flammable Gas Tank Safety Program: Data Requirements for Core Sample Analysis Developed through the Data Quality Objectives (DQO) Process* (McDuffie and Johnson 1995). These data quality objectives (DQOs) are described in the Tank Characterization Plan (TCP) for BY-106 (Homi 1995). The flammable gas DQO is not identified in the TCP for BY-106; however, the Flammable Gas Safety Program has requested that tanks such as BY-106 which appear on the potential flammable gas Watch List (OSD 1995) are sampled and analyzed as if they were on the Flammable Gas Watch List. This SAP will also identify procedures and requirements for collecting and characterizing vapor and rotary-mode core samples from BY-106.

2.0 SAMPLING EVENT REQUIREMENTS

As of July 1995, tank BY-106 contained 2,430 kL of noncomplexed waste corresponding to a waste depth of 612 cm. The tank contents were comprised of 2,070 kL of saltcake and 360 kL of sludge with 806 kL of 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 lower flammability limit (LFL). The results shall be submitted to the project coordinator within one week of the sampling event. If the limits are above 25% of the LFL when analyzing by gas chromatography/mass spectrometer (GC/MS) or gas-specific monitoring gauges, or above 10% of the LFL when analyzing with a combustible gas meter, the necessity for recurring sampling for flammable gas concentration and the frequency of such sampling will be determined by the Flammable Gas Program.

Tank BY-106 is currently scheduled to be core sampled using a rotary-mode core sampling truck. At least 13 segments are expected to be taken from riser 11B. 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. Segment 1 is expected to be 33 cm in depth, and segments 2-13 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-106 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, ferrocyanide, historical, and flammable gas DQO documents, as well as the organic test plan, the steps below shall be performed on each sample. Before sample removal and breakdown begins, the Flammable Gas Safety Program shall be contacted by the BY-106 Project Coordinator. However, extrusion should not be delayed for more than one half of a work day (4 hours) to accommodate program observers. Several analyses listed in Table 1 require a 45-day reporting time.

- Carefully extrude segments without shaking or mixing the sample.
- Collect aliquots from four quarters of the segment without disturbing the stratification of the segment. These subsamples will be used for the quarter-segment archives and the safety-screening, organic, historical, and ferrocyanide DQO analyses.
- Collect at least 15 mL of sample from any drainable liquid present for the safety-screening, organic, and ferrocyanide DQO analyses. Collect a minimum of 10 mL as an archive sample.
- Remove 80 mL from each segment for the shear strength measurements requested by the flammable gas DQO. Samples should be prepared in duplicate, without disturbing the stratification of the core segment.

- Prepare and homogenize the stratum composite samples for the flammable gas DQO analyses identified in Table 1.
- Collect aliquots of each segment-level liquid to generate a liquid core composite for the flammable gas DQO liquid analyses identified in Table 1.

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-106 core samples. **For core samples from tank BY-106, 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-106 core samples are based on the safety screening, organic, flammability, ferrocyanide, and historical DQO documents, and the organic test plan, 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, a prioritization of the analyses may be provided to the laboratories by the BY-106 Project Coordinator. 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 *Tank Waste Remediation System Characterization Project Quality Policies* (Trible 1995), *Fiscal Year 1995 Tank Waste Remediation System Tank Waste Analysis Plan* (Haller 1994), and in this SAP.

Table 1: Tank BY-106 Chemical, Radiological, and Physical Analytical Requirements.

SOLID ANALYSES																		
Project Name		BY-106 Rotary Mode Core Sample						COMMENTS							REPORTING LEVELS			
Plan Number		WHC-SD-WM-TSAP-050, 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		#CORES					FORMAT V		RCRA Compliance	
C. Organic		Organic				D. A. Turner		BY-106		1					FORMAT VI		Special	
D. Organic Test Plan		Ferrocyanide				R. J. Cash												
E. Historical		Historical				B. C. Simpson												
F. Flammable Gas		Flammable Gas				G. D. Johnson												
PROGRAM	PRIMARY ANALYSES				SAMPLE ¹			PREP ²	QUALITY CONTROL ³						CRITERIA			REPORT
	METHOD	ANALYSIS	WHC PROCEDURE	SEG	¼ SEG SLDG	¼ SEG SC	STRAT COMP		DUP	SPIKE	BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	
A	CGM	% LFL	work package					d	N/A	N/A	N/A	ea AB	N/A	90-110	% LFL	> 25	< 25	I, III
A,B,C,D,E,F	DSC	Energy	LA-514-113		X	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g	≥ 480 ⁴	unknown	I, III, IV
A,B,C,E,F	TGA	% H ₂ O	LA-560-112		X	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	30-40	I, III, IV
B	Distillation	CN ¹⁷	LA-695-102		X	X		d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III, IV
A,E,F	α counting	Total Alpha	LA-508-101		X ⁸	X ⁹	X	f	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µCi/g	> 41	<41	I, III, IV
A,C,D,F	Persulfate	TOC ¹⁰ , TIC	LA-342-100		X	X	X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	> 30,000	1E3-2E3	I, III, IV
A,D,E,F	IC	full suite ¹⁸	LA-533-105		X ⁸	X ⁸	X	w	ea smpl	1/mtrx	ea PB	ea AB	±15	85-115	µg/g	none	varies	IV
A,E,F	Gravimetry	bulk density	LO-160-103		X	X	X	d	N/A	N/A	N/A	ea AB	N/A	90-110	g/mL	none	1.1 - 1.6	III, IV
A,B,F	ICP	full suite ²⁰	LA-505-151		X ⁸	X ⁹	X	f ¹⁹ & a	ea smpl	see ⁶	ea PB	ea AB	±10	90-110	µg/g	Ni > 8,000	Ni: 1E3-2E3	I, III, IV
F	Vane Test	Shear Strength ¹³	see ¹⁴	X				d	ea smpl	N/A	N/A	ea AB	±25	80-120	d/cm	none	unknown	IV
F	Visual	Settling Rate ¹⁸	see ¹⁴				X	d	ea smpl	N/A	N/A	ea AB	±25	80-120	cm/hr	none	unknown	IV
F	Viscometer	Viscosity ¹³	see ¹⁴				X	d	ea smpl	N/A	N/A	ea AB	±25	90-110	cP	none	unknown	IV
F	Spectrophotometry	Cr (VI)	LA-265-101				X	w	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µg/g	none	unknown	IV
F	Sep. & β counting	Sr-90	LA-220-101				X	f	ea smpl	see ⁵	ea PB	ea AB	±10	90-110	µCi/g	none	5.0-10	IV
F	β counting	Total Beta	LA-508-101				X	f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV
F	Sep. & α counting	Pu-239/240, Pu-238	LA-503-156				X	f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	3E-3 - 5E-3	IV
F	Sep. & α counting	Am-241, Cm-242, Cm-243/244	LA-548-121				X	f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV
F	Liquid Scintillation	Tc-99	LA-438-101				X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV
F	Liquid Scintillation	H-3	LA-114-101				X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV
F	GEA	Co-60, Cs-137, Eu-154, Np-237, total gamma	LA-548-121				X	f	ea smpl	N/A	ea PB	ea AB	±20	80-120	µCi/g	none	Cs: 100-200	IV
F	GEA	I-129	LA-378-103				X	f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV
F	Titration	OH	LA-661-103				X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	none	1E4 - 2E4	IV
E,F	Laser Florimetry	Total Uranium	LA-925-009				X	f	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µg/g	none	3E3 - 4E3	IV

Table 1: Tank BY-106 Chemical, Radiological, and Physical Analytical Requirements.

PROGRAM	SECONDARY ANALYSES			SAMPLE ¹		PREP ²	QUALITY CONTROL ³						CRITERIA			REPORT	
	METHOD	ANAL.	WHC PROCEDURE	% SEG SLDG	% SEG SC		STRAT COMP	DUP	SPIKE	BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT		EXPECTED RANGE
A	α counting		LA-503-156	X	X		f	ea smpl	see ⁵	ea PB	ea AB	±10	90-110	µCi/g	> 41	3E-3 - 5E-3	I, III
C	Gravimetry	% H ₂ O ¹²	LA-564-101	X	X		d	ea smpl	N/A	N/A	ea AB	±20	80-120	wt%	< 25	30-40	I, III, IV
A, B, D	RSST	Energy ⁷	see ⁷	X	X		d	N/A	N/A	N/A	ea AB	N/A	90-110	J/g	≥ 480 ⁴	> 481	I, 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	µm	none	unknown	IV
B	Persulfate	TC ⁷	LA-342-100	X	X		d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	none	> 1000	IV
E	ICP	full suite ²⁰	LA-505-151		X		w	ea smpl	see ⁶	ea PB	ea AB	±10	90-110	µg/g	none	varies	
D	Fluorimetry	Total Uranium ²²	LA-925-007	X	X		f	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µg/g	none	3E3 - 4E3	IV
C	CG/MS	Organic Speciation ²¹	LA-523-437	X			d	ea smpl	N/A	ea AB	ea AB	±20	80-120	µg/g	none	varies	IV

¹1/2 SEG SLDG-1/2 segment, sludge; 1/2 SEG SC-1/2 segment saltcake; STRAT COMP-stratum composite.²d-direct, f-fusion, a-acid, w-water.³PR-precision, AC-accuracy, ea-each, DUP-duplicate, AB-analytical batch, PB-preparation blank, mtrx-matrix.⁴Dry weight basis.⁵Tracer or carrier may be used in place of a spike and results corrected for recovery.⁶Either serial dilutions or matrix spikes will be performed.⁷Analysis required if DSC exceeds notification limits. The RSST method may be found in WHC-SD-WM-TP-104.⁸Only Li and Br are required on a 1/2 segment basis.⁹Analyze only the bottom two quarter-segments of each segment extruded if sludge (i.e., quarter-segments C and D), or the bottom half-segment (if saltcake).¹⁰This is a primary analysis for the organic DQO, but is also a secondary analyses for the safety screening, and ferrocyanide DQOs. Therefore, if the DSC limit is exceeded, this analysis¹¹must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.¹²Performed if total alpha exceeds notification limit.¹³This analysis required if moisture analysis by TGA is less than 25 wt%.¹⁴Viscosity and shear strength measurements shall be done at 40°, 65°, and 90° C.¹⁵Not performed on a routine basis.¹⁶This analysis is to be run on the quarter segment sludge, or half segment saltcake, that exhibits the highest DSC value for each core.¹⁷Solid settling rate shall be performed at 40° and 60° C.¹⁸This is a primary analysis for the ferrocyanide DQO, but is also a secondary analysis for the safety screening DQO. Therefore, if the DSC limit is exceeded, this analysis¹⁹must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.²⁰Non-nickel crucible required.²¹C full suite: Cl, F, NO₂, NO₃, PO₄³⁻, SO₄²⁻, formate, acetate, oxalate, Br⁻.²²CP full suite: Al, Cr, Ca, Fe, K, Mn, Na, Ni, P, U, Zn, Zr, Ba, Si, B, Bi, Li.²³Perform this analysis if waste exhibits propagation using the RSST.²⁴Perform if the results of the fingerprint analyses are consistent with the Historical DQO.

Table 1: Tank BY-106 Chemical, Radiological, and Physical Analytical Requirements

LIQUID ANALYSES

Project Name		BY-106 Rotary Mode Core Sample		WHC-SD-WM-TSAP-050, REV. 0		Homogenization Test - Per Laboratory Discretion		FORMATT I		FORMATT II		FORMATT III		FORMATT IV		FORMATT V		FORMATT VI		Special	
PROGRAM		PROGRAM CONTACTS		Safety Screening		TVRS		K. E. Bell		E. J. Lipke		Hot Cell Blank - Per Laboratory Discretion		Libr Solution Blank - Required		TANK		BY-106			
PROGRAM ANALYSES		SAMPLE ¹		PREP ²		QUALITY CONTROL ³		CRITERIA		REPORT											
PROGRAM		METHOD		ANALYSIS		WHC PROCEDURE		FB & S-LEV LIQ COMP		DUP		SPIKE		BLK		STD		PR		AC	
		UNITS		NOTIFICATION LIMIT		EXPECTED RANGE															
A, B, C, D, E		DSC		Energy		LA-514-113		X		X		d		ea smpl		N/A		ea AB		±10	
A, B, C, E		TGA		% H ₂ O		LA-560-112		X		X		d		ea smpl		N/A		ea AB		±10	
B		Distillation		CN		LA-695-102		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±10	
A, E		ICP		full suite ¹²		LA-505-151		X ⁶		X		d ⁵		ea smpl		see ⁶		ea AB		±10	
A, E		α counting		Total Alpha		LA-508-101		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±10	
A, C, D		Paraffate		TOC ¹⁰ , TIC		LA-344-100		X				d ⁵		ea smpl		1/mtrix		ea AB		±20	
A, D, E		Visual		Organic Layer		LA-519-151		X				d		ea smpl		N/A		ea AB		N/A	
A, D, E		IC		full suite ¹³		LA-533-105		X ⁶		X		d ⁵		ea smpl		1/mtrix		ea AB		±10	
A, E		Gravimetry		Specific Gravity		LA-510-112		X		X		d ⁵		ea smpl		N/A		ea AB		±20	
E		Titration		OH		LA-661-103		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±10	
E		Centrifugation		Vol. % Solids ¹⁵		LA-519-132		X		X		d		ea smpl		N/A		ea AB		±10	
E		pH		[H ⁺]		LA-121-103		X		X		d		ea smpl		N/A		ea AB		±10	
E		Liquid Scintillation		H-3		LA-218-114		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±20	
E		Spectrophotometry		Cr (VI)		LA-265-101		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±20	
B		β counting		Total Beta		LA-508-101				X		d ⁵		ea smpl		N/A		ea AB		±20	
B		GEA		Cs-137, Np-237, Co-60, Eu-154		LA-548-121		X		X		d ⁵		ea smpl		see ⁶		ea AB		±20	
B		GEA		I-129		LA-378-103		X		X		d ⁵		ea smpl		see ⁶		ea AB		±20	
B		α counting		Am-241, Cm-242, Cm-243/244		LA-548-121		X		X		d ⁵		ea smpl		see ⁶		ea AB		±20	
B		α counting		Pu-239/240, Pu-238		LA-503-156		X		X		d ⁵		ea smpl		see ⁶		ea AB		±20	
B		Liq. Scintillation		Tc-99		LA-438-101		X		X		d ⁵		ea smpl		1/mtrix		ea AB		±20	
B		β counting		Sr-90		LA-220-101		X		X		d ⁵		ea smpl		see ⁶		ea AB		±20	
E		Laser Fluorimetry		Total Uranium		LA-925-009				X		d ⁵		ea smpl		1/mtrix		ea AB		±20	

Table 1: Tank BY-106 Chemical, Radiological, and Physical Analytical Requirements

PROGRAM	SECONDARY ANALYSES			SAMPLE ¹	PREP ²	QUALITY CONTROL ³						CRITERIA		REPORT	
	METHOD	ANAL.	WHC PROCEDURE			FB & S-LEV LIQ	DUP	SPIKE	BLK	STD	PR	AC	UNITS		NOTIFICATION LIMIT
C	Gravimetric	% H ₂ O ¹¹	LA-564-101	X		ea smpl	N/A	ea AB	ea AB	±20	80-120	%	none	75-90	III
B	Persulfate	TC ⁷	LA-342-100	X		ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/mL	none	0.01-0.1	III
A, B, C	Furnace Oxidation	TOC ⁷	LA-344-105	X		ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg C/mL	> 30,000	0.01-0.1	I, III
D	GC/MS	organic speciation ¹⁴	LA-523-437	X		ea smpl	N/A	ea AB	ea AB	±20	80-120	µg/mL	none	varies	III

¹S-LEV LIQ-liquid taken from the segment level, FB-field blank, COMP-composite.²d-direct.³PR-precision, AC-accuracy, ea-each, smpl-sample, DUP-duplicate, AB-analytical batch, N/A-not applicable, mtrx-matrix.⁴Dry weight basis.⁵Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.⁶Either serial dilutions or matrix spikes will be performed.⁷Performed only if DSC exceeds notification limit.⁸Converted from µg/g limit assuming a liquid density of 1.0 g/mL.⁹Only Br and Li are required on a segment basis.¹⁰These analyses are primary analyses for the organic DQO, but also are secondary analyses for the ferrocyanide and safety screening DQOs.

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.

¹¹This analysis required if moisture analysis by TGA is less than 25 wt%.¹²CP full suite: Al, Cr, Ca, Fe, K, Mn, Na, Ni, P, U, Zn, Zr, Ba, Si, B, Bi, Li¹³C full suite: Cl⁻, F⁻, NO₂⁻, NO₃⁻, PO₄³⁻, SO₄²⁻, formate, acetate, oxalate, Br⁻¹⁴Perform on liquid from waste samples exhibiting propagation using the RSST.¹⁵Measured at 40 and 60 degrees C.

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 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

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.

In the flammable gas DQO (McDuffie and Johnson 1995), solubility measurements in 2.5 M NaOH and in water are requested; however, no procedure exists. Therefore, solubility tests shall be done under one of the test plans currently being written.

In the Flammable gas DQO (McDuffie and Johnson 1995), formate and oxalate measurements by the chromatographic method are requested; however, this is to be analyzed using ion chromatography.

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).

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

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

- ▶ 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 (2).

$$X = (\text{wt\% TOC dry weight basis}) * \frac{151 \text{ cal/g}}{5} \quad (2)$$

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.
- ▶ Tables 8-3 and 8-4 of (Jewett 1992) which address stratum composites were used as quality control requirements.
 - ▶ Although the results for these core sample analyses are required to be generated and reported within 216 days of the sampling event, this reporting requirement may not be met for the results of the shear strength analysis. The sample aliquot for this analysis, which cannot be performed at this time, shall be stored at WHC 222-S Laboratory until the designated laboratory can accept and perform analyses.

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

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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