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TANK 241U105 PUSH MODE CORE SAMPLING & ANALYSIS
PLAN

ENGINEERING CHANGE NOTICE

Page 1 of 2

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13a. Justification (mark one) Criteria Change ☒ Design Improvement ☐ Environmental ☐ Facility Deactivation ☐ As-Found ☐ Facilitate Const ☐ Const. Error/Omission ☐ Design Error/Omission ☐							
13b. Justification Details This ECN will complete the documentation process necessary to sample and analyze Tank 241-U-105 in Fiscal Year 1996.							
14. Distribution (include name, MSIN, and no. of copies) See attached distribution sheet.						RELEASE STAMP OFFICIAL RELEASE BY WHC DATE OCT 23 1995 Sta 4	

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ECN-625753

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

Document Number/Revision	Document Number/Revision	Document Number Revision

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RELEASE AUTHORIZATION

Document Number: WHC-SD-WM-TSAP-048, REV 1

Document Title: Tank 241-U-105 Push Mode Core Sampling and Analysis Plan

Release Date: 10/23/95

**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 Broz

10/23/95

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Tank 241-U-105 Push Mode 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

U-105	Tank 241-U-105
cm	centimeters
DQO	data quality objective
DSC	differential scanning calorimetry
GEA	gamma energy analysis
GC/MS	gas chromatography/mass spectroscopy

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 three push mode core samples from tank 241-U-105 (U-105). It is written in accordance with the *Tank Safety Screening Data Quality Objective* (Hunt 1995), *Flammable Gas Tank Safety Program: Data Requirements for Core Sample Analysis Developed through the Data Quality Objectives (DQO) Process* (McDuffie and Johnson 1995), *Data Quality Objective to Support Resolution of the Organic Complexant Safety Issue* (Turner 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 tank U-105 (Homi 1995a). This SAP will also identify procedures and requirements for collecting and characterizing push-mode samples from U-105.

2.0 SAMPLING EVENT REQUIREMENTS

Currently there are 1,581 kL of waste in U-105. The waste is comprised of 140 kL of supernatant, 1,320 kL of saltcake, and 121 kL of sludge with 594 kL of pumpable liquid remaining. (The tank is identified on the Hydrogen and Organic Watch Lists, is passively ventilated, and is categorized as sound with partial interim isolation completed. Tank U-105 is equipped to cascade to tank 241-U-106 and is second in the three-tank cascade series. Tank U-105 has 12 risers, two of which (risers 2 and 7) are available for use. (Brevick 1994)

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. 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 U-105 is currently scheduled to be core sampled using a push mode core sampling truck. At least 8 segments are expected to be taken from risers 2, 7, and 20. 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 41 cm in depth, and segments 2 - 8 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 WS-95-0206. That document contains the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank U-105 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 flammable gas, safety screening, organic, and historical 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 U-105 Project Coordinator. However, extrusion should not be delayed for more than one half of a work day (4 hours) to accommodate program observers.

For the Core with the Highest Recovery Rate:

- Carefully extrude segments without shaking or mixing the sample.
- Collect adequate sample from both halves of the segment without disturbing the stratification of the segment. These subsamples will be used for the half-segment archives and the safety-screening, organic, and historical DQO analyses.
- Collect at least 15 mL of sample from any drainable liquid present for archiving and the safety-screening, organic, and historical DQO analyses.
- 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.

For the Second and Third Cores:

- Carefully extrude segments without shaking or mixing the sample.

- Divide the segment into two equal sub-segments and remove 7-10 g for the half-segment safety-screening, organic, and historical DQO analyses, and at least 15 g for the half-segment archive sample.
- Collect at least 15 mL of sample from any drainable liquid present for archiving and the safety-screening, organic, and historical DQO analyses.
- Collect aliquots of each segment-level liquid to generate a liquid core composite for flammable gas liquid analyses (see 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 U-105 core samples. **For core samples from tank U-105, 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 U-105 core samples are based on the safety-screening, organic, historical, and flammable gas 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 this SAP and permit a minimum 10 mL archive per sample, a prioritization of the analyses shall be provided to the laboratories by the U-105 Project Coordinator. Any analyses prescribed by this 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 SAP 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

Table 1: Tank 241-U-105 Chemical, Radiological, and Physical Analytical Requirements

SOLID ANALYSES									
Project Name		U-105 Push-Mode Core Sample			COMMENTS				
Plan Number		WHC-SD-WM-TSAP-048, REV. 1			Homogenization Test - Per Laboratory Discretion				
PROGRAM		PROGRAM CONTACTS			Field Blank - Required				
A. Safety Screening		Safety Screening			Hot Cell Blank - Per Laboratory Discretion				
B. Flammable Gas		TWRS			Libr Solution Blank - Required				
C. Organic		Flammable Gas			G. D. Johnson				
D. Organic Test Plan		Organic			D. A. Turner				
E. Historical		Historical			B. C. Simpson				
U-105					3				
FORMAT I		Early Notify			FORMAT VI				
FORMAT II		Process Control			FORMAT V				
FORMAT III		Safety Screening			RCRA Compliance				
FORMAT IV		Waste Management			Special				

PROGRAM	METHOD	ANALYSIS	PRIMARY ANALYSES				SAMPLE ¹		PREP ²		QUALITY CONTROL ³					CRITERIA			REPORT
			WHC PROCEDURE	SEG	% SEG	STRATUM COMP			DUP	SPIKE	BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT	EXPECTED RANGE		
A	CGM	% LFL	work package				d	N/A	N/A	N/A	N/A	ea AB	N/A	90-110	% LFL	> 25	< 25	I, III	
A, B, C, E	DSC	Energy	LA-514-113	X	X		d	ea smpl	N/A	N/A	N/A	ea AB	±10	90-110	J/g	> 480 ⁴	0 - 250	I, III, IV	
B, C, E	TGA	% H ₂ O	LA-560-112	X	X		d	ea smpl	N/A	N/A	N/A	ea AB	±10	90-110	wt%	none	20 - 30	III, IV	
B, C, D	Persulfate	TOC	LA-342-100	X	X		d	ea smpl	1/mtrix	ea AB	ea AB	±20	80-120	µg C/g	> 30,000	10E3 - 12E3	I, III, IV		
B	Persulfate	TIC	LA-342-100	X			d	ea smpl	1/mtrix	ea AB	ea AB	±15	85-115	µg C/g	none	unknown	IV		
A, B, E	ICP	Li, Na, Al, Cr, Ca, Fe, K, Ni, Zn, Zr, Ba, Si, B, Bi, Mn, U	LA-505-151	X ⁶	X		f and a	ea smpl	see ⁸	ea PB	ea AB	±15	85-115	µg/g	LI > 100	varies	I, III, IV		
A, B, D, E	IC	Br, NO ₂ , NO ₃ , PO ₄ , SO ₄ , Cl ⁻ , F ⁻ , oxalate, formate, acetate	LA-533-105	X ¹⁰	X		w	ea smpl	1/mtrix	ea PB	ea AB	±15	85-115	µg/g	Br > 1200	varies	I, III, IV		
B	Titration	OH	LA-661-103		X		d	ea smpl	1/mtrix	ea AB	ea AB	±10	90-110	µg/g	none	1.6E5 - 2.5E5	IV		
D	RSST	Energy ¹⁶	see ⁸	X			d	N/A	N/A	N/A	ea AB	N/A	90-110	J/g	none	>480	IV		
A, B, E	Gravimetry	Bulk Density	LO-160-103		X		d	N/A	N/A	N/A	ea AB	±10	90-110	g/mL	none	1.0 - 1.5	III, IV		
B	Viscometer	Viscosity ¹²	see ¹³		X		d	ea smpl	N/A	N/A	ea AB	±25	80-120	d/cm	none	varies	IV		
B	Vane Test	Shear Strength ¹²	see ¹³	X			d	ea smpl	N/A	N/A	ea AB	±25	80-120	cm/hr	none	varies	IV		
B	Visual	Settling Rate ¹⁵	see ¹³	X			d	ea smpl	N/A	N/A	ea AB	±25	80-120	µg/g	none	varies	IV		
B, E	Fluorimetry	Total Uranium	LA-925-009	X			f	ea smpl	1/mtrix	ea PB	ea AB	±20	80-120	µg/g	none	unknown	IV		
B	Spectroscopy	Cr (VI)	LA-265-101	X			w	ea smpl	1/mtrix	ea PB	ea AB	±20	80-120	µg/g	none	unknown	IV		
A, B, E	α counting	Total Alpha	LA-508-101	X	X ¹¹		f	ea smpl	1/mtrix	ea PB	ea AB	±10	90-110	µCi/g	> 41	0.1 - 0.4	I, III, IV		
B, E	β counting	Total Beta	LA-508-101	X			f	ea smpl	1/mtrix	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV		
B, E	GEA	CS-137, Np-237, Co-60, Eu-154	LA-548-121	X			f	ea smpl	N/A	ea PB	ea AB	±20	80-120	µCi/g	none	varies	IV		
B	GEA	H-129	LA-378-103	X			f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV		
B	Liq. Scintillation	H-3	LA-114-101	X			d	ea smpl	1/mtrix	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV		
B	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		
B	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	0.005 - 0.02	IV		
B	Liq. Scintillation	Tc-99	LA-438-101	X			d	ea smpl	1/mtrix	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV		
B, E	β counting	St-90	LA-220-101				f	ea smpl	see ⁵	ea PB	ea AB	±20	80-120	µCi/g	none	15-40	IV		

Table 1: Tank 241-U-105 Chemical, Radiological, and Physical Analytical Requirements

PROGRAM	METHOD	ANAL.	SECONDARY ANALYSES					SAMPLE ¹	PREP ²	QUALITY CONTROL ³					CRITERIA		REPORT
			WHC PROCEDURE	% SEG	STRATUM COMP	DUP	SPIKE			BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	
C	Gravimetry	% H ₂ O ¹⁴	LA-564-101	X		d	ea smpl	N/A	ea AB	±20	80-120	w%	none	20 - 30	I, IV		
A	α counting	Pu-239/40 ²⁰	LA-503-156	X		f	ea smpl	see ⁵	ea PB	±10	90-110	µCi/g	> 41	0.005 - 0.02	I, III		
A, B	RSST	Energy ⁷	see ⁸	X		d	N/A	N/A	ea AB	N/A	90-110	J/g	>480 ⁹	>480	III, IV		
C, A	Distillation	CN ¹¹	LA-695-102	X		d	ea smpl	1/mtx	ea AB	±20	80-120	µg C/g	none	unknown	IV		
E	ICP	full suite ¹⁸	LA-505-151		X	w	ea smpl	see ⁶	ea PB	±10	90-110	µg C/g	none	values	IV		
C	CG/MS	organic speciation ¹⁹	LA-523-437	X		d	ea smpl	N/A	ea AB	±20	80-120	µg C/g	none	values	IV		

¹ ½ SEG - ½ segment, COMP - Composite

² d-direct, f-fusion, a-acid, w-water

³ PR-precision, AC-accuracy, ea-each, DUP-duplicate, AB-analytical batch, PB-preparation blank, mtx-matrix

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 Only Li is required on a 1/2 segment basis.

¹⁰ Only Br is required on a 1/2 segment basis.

¹¹ Analyze only the bottom half segment of each segment extruded.

¹² Measured at 40, 65, and 90 degrees C.

¹³ Not performed on a routine basis.

¹⁴ This analysis required if moisture analysis by TGA exceeds 17% H₂O.

¹⁵ Measured at 40 and 60 degrees C.

¹⁶ This analysis is to be run on the or half segment that exhibits the highest DSC value for each core.

¹⁷ Performed if TOC < 3%.

¹⁸ Perform if results of the fingerprint analyses for this waste type are consistent with the historical DQO.

¹⁹ Perform this analysis if waste exhibits propagation using the RSST.

²⁰ Performed if total alpha exceeds notification limit.

Table 1: Tank U-105 Chemical, Radiological, and Physical Analytical Requirements

LIQUID ANALYSES																	
Project Name		U-105 Push-Mode Core Sample				COMMENTS								REPORTING LEVELS			
Plan Number		WHC-SD-WM-TSAP-048, REV. 1				Homogenization Test - Per Laboratory Discretion Field Blank - Required Hot Cell Blank - Per Laboratory Discretion LiBr Solution Blank - Required								FORMAT I		Early Notify	
														FORMAT II		Process Control	
														FORMAT III		Safety Screening	
														FORMAT IV		Waste Management	
PROGRAM		PROGRAM CONTACTS				TANK U-105		#CORES 3						FORMAT V		RCRA Compliance	
A. Safety Screening		Safety Screening		E. J. Lipke										FORMAT VI		Special	
B. Flammable Gas		TWRS		K. E. Bell													
C. Organic		Flammable Gas		G. D. Johnson													
D. Organic Test Plan		Organic		D. A. Turner													
PROGRAM	PRIMARY ANALYSES			SAMPLE ¹		PREP ²	QUALITY CONTROL ³							CRITERIA			REPORT
	METHOD	ANALYSIS	WHC PROCEDURE	FB & SEG LIQUID	LIQUID COMP		DUP	SPIKE	BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT	EXPECTED RANGE		
A	Visual	Organic Layer	LA-519-151	X		d	N/A	N/A	N/A	N/A	N/A	N/A	none	presence	0	I, III	
A, B, C	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	
B, C	TGA	% H ₂ O	LA-560-112	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	70 - 80	I, III, IV	
B, C, D	Persulfate	TOC	LA-342-100	X	X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	> 30,000	10E3 - 12E3	I, III, IV	
B	Persulfate	TIC	LA-342-100		X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±15	85-115	µg C/g	none	unknown	IV	
A, B	ICP	full suite	LA-505-151	X ⁸	X	d ⁷	ea smpl	see ⁵	ea AB	ea AB	±15	85-115	µg/g	Li > 100	varies	I, III, IV	
A, B, D	IC	Br, NO ₂ , NO ₃ , PO ₄ , SO ₄ , Cl, F, oxalate, formate, acetate	LA-533-105	X ⁹	X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±15	85-115	µg/g	Br > 1200	varies	I, III, IV	
B	Titration	OH	LA-661-103		X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	none	1.6E5 - 2.5E5	IV	
A, B	Gravimetry	Specific Gravity	LA-510-112	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	g/mL	none	1.0 - 1.5	III, IV	
B	Centrifugation	Vol. % Solids ¹¹	LA-519-132		X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	%	none	varies	IV	
B	Fluorimetry	Total Uranium	LA-925-009		X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/g	none	unknown	IV	
B	Spectroscopy	Cr (VI)	LA-265-101		X	d ⁷	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/g	none	unknown	IV	
A, B	α counting	Total Alpha	LA-508-101	X ¹⁰	X	d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µCi/g	> 41	0.1 - 0.4	I, III, IV	
B	β counting	Total Beta	LA-508-101		X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV	
B	GEA	Cs-137, Np-237, Co-60, Eu-154	LA-548-121		X	d	ea smpl	N/A	ea AB	ea AB	±20	80-120	µCi/g	none	varies	IV	
B	GEA	I-129	LA-378-103		X	d ⁷	ea smpl	see ⁵	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV	
B	Liq. Scintillation	H-3	LA-114-101		X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV	
B	α counting	Am-241, Cm-242, Cm-243/244	LA-548-121		X	d	ea smpl	see ⁵	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV	
B	α counting	Pu-239/240	LA-503-156		X	d	ea smpl	see ⁵	ea AB	ea AB	±20	80-120	µCi/g	none	0.005 - 0.02	IV	
B	Liq. Scintillation	Tc-99	LA-438-101		X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µCi/g	none	unknown	IV	
B	β counting	Sr-90	LA-220-101		X	d	ea smpl	see ⁵	ea AB	ea AB	±20	80-120	µCi/g	none	15-40	IV	

Table 1: Tank U-105 Chemical, Radiological, and Physical Analytical Requirements

PROGRAM	SECONDARY ANALYSES			SAMPLE ¹		PREP ²	QUALITY CONTROL ³						CRITERIA			REPORT
	METHOD	ANAL.	WHC PROCEDURE	FB & SEG LIQUID	LIQUID COMP		DUP	SPIKE	BLK	STD	PR	AC	UNITS	NOTIFICATION LIMIT	EXPECTED RANGE	
D	GC/MS	organic speciation ¹³	LA-523-437	X		d	ea smpl	N/A	ea AB	ea AB	±20	80-120	µg C/g	none	unknown	IV
A, C	Distillation	CN ¹²	LA-695-102	X		d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg C/g	none	unknown	IV

¹FB - Field Blank, SEG - segment, COMP - Composite.

²d-direct.

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.

⁷Direct liquid samples may be diluted in acid or water to adjust to the proper analyte concentration or pH.

⁸Only Li is required on a segment basis.

⁹Only Br is required on a segment basis.

¹⁰Analyze only the bottom half segment of each segment extruded.

¹¹Measured at ambient and 60 degrees C.

¹²Performed if TOC < 3%.

¹³Perform on liquid from waste samples exhibiting propagation using the RSST.

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

The organic DQO document (Turner 1995) requires determination of total organic carbon (TOC) by furnace oxidation if the energy equivalent by TOC (See Section 5.2.) is less than 75% of any differential scanning calorimetry (DSC) exotherm. Determination of TOC by furnace oxidation is only possible on liquid or water-digested samples, therefore analysis of TOC by furnace oxidation will not be performed until funding is provided for method development and/or new instrumentation. Analyses may then be performed on archived samples. The organic DQO also requires analysis for organic acids by liquid chromatography (HPLC) if the waste exhibits propagation during adiabatic calorimetry testing. Selected organic acids will be determined by ion chromatography, however the 222-S Laboratory currently has no procedure for determination by HPLC. The program may request analysis of archive samples at a later date if funding is provided for procedure development.

Analysis for complexants is required in the organic test plan (Meacham 1995), however a procedure at the 222-S Laboratory is not available at this time. Analyses may be performed at a later date on archived samples if funding is provided by the program for procedure development. Likewise, investigation of any propagating reaction using a tube propagation test is not possible at the 222-S Laboratory at this time. Analysis may be performed at a later date after funding is provided for on-site analytical development, or samples may be sent off-site for analysis.

The flammable gas DQO document (McDuffie and Johnson 1995) requires solubility measurements in 2.5 M NaOH and water, and determination of nickel-63 activity. No procedure exists for the solubility tests as requested; they may be carried out at a later date using archived samples. No procedure exists at the 222-S Laboratory for nickel-63 analysis. Analysis shall be performed on archived material at a later date if funding is made available for procedure development or transfer.

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 determined by 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 the weight percent water value is greater than 90%, however the conversion is still required.

The safety screening DQO (Hunt 1995) requires that additional analyses be performed if total alpha activity is greater than 1 g/L. Total alpha activity is measured in $\mu\text{Ci/g}$ rather than g/L. To convert the notification limit for total alpha activity 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):

$$\left(\frac{1 \text{ g}}{\text{L}} \right) \left(\frac{1 \text{ L}}{10^3 \text{ mL}} \right) \left(\frac{1}{\text{density}} \frac{\text{mL}}{\text{g}} \right) \left(\frac{0.062 \text{ Ci}}{1 \text{ g Pu-239}} \right) \left(\frac{10^6 \mu\text{Ci}}{1 \text{ Ci}} \right) = \frac{61.5}{\text{density}} \frac{\mu\text{Ci}}{\text{g}} \quad (2)$$

NOTE: Samples measured for total alpha activity shall also be measured for density. The notification limit shall be 41 $\mu\text{Ci/g}$ unless the measured density exceeds 1.5 g/mL when the notification limit will be adjusted according to equation 2.

Secondary analyses are required per the organic DQO 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).

$$X = [\text{wt\% TOC (dry weight basis)}] * \frac{632 \text{ J/g}}{5} \quad (3)$$

NOTE: 632 J/g represents the energy equivalent of 5 wt% TOC based on a sodium acetate average energetics standard (Turner 1995).

6.0 DELIVERABLES

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

WHC-SD-WM-TSAP-048, REV. 1

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Trible, T.C., 1995, *Tank Waste Remediation System Characterization Project Quality Policies*, WHC-SD-WM-QAPP-025, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

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