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TANK 241BY105 ROTARY CORE SAMPLING & ANALYSIS PLAN

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ENGINEERING DATA TRANSMITTAL

Page 1 of 1

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Tank 241-BY-105
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-105	Tank 241-BY-105
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
LFL	lower flammability limit
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 two rotary-mode core samples from tank 241-BY-105 (BY-105). It is written in accordance with the *Tank Safety Screening Data Quality Objective* (Dukelow, 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 Complexant Safety Issue* (Turner, et al. 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 241-BY-105 (BY-105). This SAP will also identify procedures and requirements for collecting and characterizing samples from tank BY-105 by the rotary core sampling method. This SAP supersedes Appendix A of the *Tank 241-BY-105 Tank Waste Characterization Plan* (Homi 1995a).

2.0 SAMPLING EVENT REQUIREMENTS

As of May 31, 1995, tank BY-105 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, using a combustible gas meter, 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 BY-105 was scheduled to be core sampled using a rotary-mode core sampling truck. At least two samples were expected to be taken from risers 11 and 12A. If a different riser is necessary to meet sampling and analysis requirements, this change must be recorded and approved by the 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-105. 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.

Rotary mode core sampling through riser 12A was initiated under Appendix A of the TCP (Homi 1995a). For detailed information regarding the sampling activities, refer to work packages

ES-95-0434 and ES-95-0556. This documents contain the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank BY-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 if the lithium notification limit is exceeded, 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 in addition to the field/trip blank.

3.0 LABORATORY ANALYSIS REQUIREMENTS

3.1 ANALYSIS SCHEME

In order to comply with the safety screening, organic, organic test plan, ferrocyanide, disposal, 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 as indicated in Table 1.
- ▶ 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-105 core samples. For core samples from tank BY-105, the shipping container must be vented every 47 days to release any accumulated gas.

Opportunistic analyses as defined in Kristofzski (1995a) will be included at the discretion of the project coordinator when the laboratory is not operating at maximum capacity.

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

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

Project Name			BY-105 Rotary Mode Core Sample				SOLID ANALYSES										REPORTING LEVELS	
Plan Number			WHC-SD-WM-TSAP-040, Rev. 0														FORMAT I	Early Notify
							COMMENTS										FORMAT II	Process Control
PROGRAM CONTACTS						Homogenization Test - Per Laboratory Discretion Field Blank - Required Hot Cell Blank - Per Laboratory Discretion LiBr Blank - Required if LiBr used during sampling										FORMAT III	Safety Screen	
A. Historical DQO			B. C. Simpson													FORMAT IV	Waste Management	
B. Ferrocyanide DQO			J. E. Meacham													FORMAT V	RCRA Compliance	
C. Safety Screening DQO			E. J. Lipke													FORMAT VI	Special	
D. Organic Complexant Issue DQO			D. A. Turner															
E. Safety Test Plan			J. E. Meacham															
TWRS			L. M. Sasaki															
PRIMARY ANALYSES			SAMPLE ¹			PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT	PRO-GRAM	
METHOD	ANAL.	WHC PROCEDURE	½ SEG SLDG	½ SEG SC	COMP		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴			
DSC	Energy	LA-514-113	X	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g ⁵	≥ 480	unknown	I, III, IV	A, B, C, D, E	
TGA	% H ₂ O	LA-580-112	X	X	X	d	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	unknown	III, IV	A, B, C, D	
Alpha	Total Alpha	LA-508-101	X ⁶	X ⁶		f	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µg/g	>41	unknown	I, III, IV	C	
Distillation	CN	LA-695-102	X			d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, IV	B	
ICP	Na, Al, Bi, Ni, Ca, Fe, P	LA-505-151	X	X	X	a or f	ea smpl	see ⁷	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV	A, B, D	
IC	NO ₃ , SO ₄	LA-533-105	X	X	X	w	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV	A	
Sep. & β counting	Sr-90	LA-220-101	X	X	X	f	ea smpl	1/mtrx ⁸	ea PB	ea AB	±10	90-110	µCi/g	none	unknown	IV	A	
HPGE/MCA	GEA (Cs ¹³⁷)	LA-548-121	X	X	X	f	ea smpl	N/A	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV	A	
Gravimetric	bulk density	LO-160-103	X ⁹	X ⁶		d	ea smpl	N/A	N/A	ea AB	±10	90-110	g/mL	none	unknown	III, IV	C	
Direct Persulfate	TOC	LA-344-100	X	X		d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/g	> 30,000 ⁵	unknown	I, III ⁹ , IV	B, C, D, E	
SECONDARY ANALYSES			SAMPLE ¹			PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT	PRO-GRAM	
METHOD	ANAL.	WHC PROCEDURE	½ SEG SLDG	½ SEG SC	COMP		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴			
Distillation	CN	LA-695-102		X ¹⁰		d	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/g	> 39,000	unknown	I, III, IV	B, C	
Direct Persulfate	TOC	LA-344-100	X ¹¹	X ¹¹	X	d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/g	> 30,000 ⁵	unknown	I, IV	A	
Sep. & α counting	Pu-239/240	LA-503-156	X ¹²	X ¹²		f	ea smpl	1/mtrx ⁸	ea PB	ea AB	±15	85-115	µCi/g	none	unknown	III, IV	C	
RSST	Energy	see ¹³	X ¹³	X ¹³		d	ea smpl	N/A	N/A	ea AB	±20	80-120	J/g ⁵	none	unknown	III, IV	B, C, D, E	
Direct Persulfate	TC	A-344+C12-10	X ¹⁴	X ¹⁴		d	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/g	none	unknown	IV	B	
Beta	Total Beta	LA-508-101	X ¹¹	X ¹¹	X	f	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µCi/g	none	unknown	IV	A	
Phosphor-escence	U, total	LA-925-009	X ^{11, 12}	X ^{11, 12}	X	f	ea smpl	1/mtrx	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	A, C	
Gravimetric	bulk density	LO-160-103	X ¹¹	X ¹¹	X	d	ea smpl	N/A	N/A	ea AB	±20	80-120	g/mL	none	unknown	IV	A	
Particle Size	particle size	LT-519-101	X ¹⁴	X ¹⁴									see ¹⁵	none	unknown	IV	B	
ICP	Na, Al, Bi, Ni	LA-505-151	X ¹¹	X ¹¹	X	w	ea smpl	see ⁹	ea PB	ea AB	±10	90-110	µg/g	none	unknown	IV	A	
ICP	Fe, Mn, U, Cr, Ni, Al, Na, Si	LA-505-151	X ¹²	X ¹²		f	ea smpl	see ⁹	ea PB	ea AB	±10	90-110	µg/g	none	unknown	III, IV	C	
Alpha	Total Alpha	LA-508-101	X ¹¹	X ¹¹	X	f	ea smpl	1/mtrx	ea PB	ea AB	±20	80-120	µCi/g	>41	unknown	I, IV	A	

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

SECONDARY ANALYSES			SAMPLE ¹			PREP ²	QUALITY CONTROL ³						CRITERIA			FOR-MAT	PRO-GRAM
METHOD	ANAL.	WHC PROCEDURE	½ SEG SLDG	½ SEG SC	COMP		DUP	SPK/MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
Gravimetric	% water	LA-564-101	X ¹⁶	X ¹⁶		d	ea smpl	N/A	N/A	ea AB	±10	90-110	%	none	unknown	IV	D
GC/MS	organic speciation	LA-564-101	X ¹⁷	X ¹⁷		d	ea smpl	N/A	ea AB	ea AB	±20	80-120	µg/g	none	unknown	IV	D

¹½ SEG SLDG-½ segment, sludge; ½ SEG SC-½ segment, saltcake; COMP-saltcake and sludge core composites

²d-direct, f-fusion, a-acid, w-water

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

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

⁵Dry weight basis.

⁶Perform on lower half of each segment only.

⁷Either serial dilutions or matrix spikes will be performed.

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

⁹TOC by persulfate is a secondary analysis for the Safety Screening DQO; results not required for 45-day report.

¹⁰Performed if TOC does not account for the exotherm or if DSC>480 J/g (dry weight basis)

¹¹Performed on one sludge and one saltcake subsegment after consultation with the Historical DQO point of contact.

¹²Analysis performed if total alpha exceeds notification limit.

¹³If Analysis performed on sample with highest exotherm above 480 J/g (dry weight basis). RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

If DSC<1200 J/g (dry weight basis), perform a second RSST, spiking the sample with sodium acetate.

¹⁴Analysis performed if TGA<0.022(DSC-1200).

¹⁵Estimate of total number and mass of particles under 2µm in diameter is required.

¹⁶Analysis to be performed if TGA<25wt%.

¹⁷Analysis to be performed if TOC>30,000 or if AC/RSST indicates propagation.

¹⁸Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.

¹⁹Analysis to be performed if TOC does not account for the exotherm.

²⁰Analysis to be performed if TOC by persulfate does not account for the exotherm.

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

Project Name			BY-105 Rotary Mode Core Sample		LIQUID ANALYSES									REPORTING LEVELS	
Plan Number			WHC-SD-WM-TSAP-040, Rev. 0											FORMAT I	Early Notify
					COMMENTS									FORMAT II	Process Control
PROGRAM CONTACTS					Homogenization Test - Per Laboratory Discretion Field Blank - Required Hot Cell Blank - Per Laboratory Discretion LiBr Blank - Required if LiBr used during sampling									FORMAT III	Safety Screen
B. Ferrocyanide DQO			J. E. Meacham											FORMAT IV	Waste Management
C. Safety Screening DQO			E. J. Lipke											FORMAT V	RCRA Compliance
D. Organic Complexant Issue DQO			D. A. Turner											FORMAT VI	Special
E. Safety Test Plan			J. E. Meacham												
TWRS			L. M. Sasaki												
PRIMARY ANALYSES			SAMPLE ¹	PREP ²	QUALITY CONTROL ³						CRITERIA			FOR- MAT	PRO- GRAM
METHOD	ANAL.	WHC PROCEDURE	FB & S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
DSC	Energy	LA-514-113	X	d	ea smpl	N/A	N/A	ea AB	±20	80-120	J/g ⁵	≥ 480	unknown	I, III, IV	B, C, D, E
TGA	% H ₂ O	LA-560-112	X	d	ea smpl	N/A	N/A	ea AB	±20	80-120	wt%	none	unknown	III, IV	C, D
Direct Persulfate	TOC	LA-344-100	X	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/mL	> 30,000 ⁵	unknown	I, III ⁹ , I	B, C, D, E
Alpha	Total alpha	LA-508-101	X	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µCi/g	>41	unknown	I, III, IV	C
Specific Gravity	Density	LA-510-112	X	d	ea smpl	N/A	N/A	ea AB	±20	80-120	g/mL	none	unknown	III, IV	C
Visual	Organic Layer	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	N/A	none	presence	unknown	III, IV	C
SECONDARY ANALYSES			SAMPLE ¹	PREP ²	QUALITY CONTROL ³						CRITERIA			FOR- MAT	PRO- GRAM
METHOD	ANAL.	WHC PROCEDURE	S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴		
RSST	Energy	see ¹³	X	d	ea smpl	N/A	N/A	ea AB	±20	80-120	J/g	none	unknown	III, IV	C, D, E
Distillation	CN	LA-695-102	X ¹⁹	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±10	90-110	µg/mL	> 39,000	unknown	I, III, IV	C
GC/MS	organic speciation	LA-564-101	X ¹⁷	d	ea smpl	N/A	ea AB	ea AB	±20	80-120	µg/g	none	unknown	IV	D
Furnace Oxidation	TOC	LA-344-105	X ²⁰	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/mL	> 30,000	unknown	I, III, IV	C, D
Furnace Oxidation	TC	LA-344-105	X ¹⁴	d ⁶	ea smpl	1/mtrx	ea AB	ea AB	±20	80-120	µg/mL	none	unknown	IV	B

¹S-LEV LIQ-liquid taken from the segment level; FB - field blank²d-direct, f-fusion, a-acid, w-water³PR-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-matrix⁴Units for notification limits and expected range are those listed in the "units" column.⁵Dry weight basis.⁶Perform on lower half of each segment only.⁷Either serial dilutions or matrix spikes will be performed.⁸Tracer or carrier may be used in place of a spike and results corrected for recovery.⁹TOC by persulfate is a secondary analysis for the Safety Screening DQO; results not required for 45-day report.¹⁰Performed if TOC does not account for the exotherm or if DSC>480 J/g (dry weight basis)¹¹Performed on one sludge and one saltcake subsegment after consultation with the Historical DQO point of contact.

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

¹²Analysis performed if total alpha exceeds notification limit.

¹³If Analysis performed on sample with highest exotherm above 480 J/g (dry weight basis). RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

If DSC<1200 J/g (dry weight basis), perform a second RSST, spiking the sample with sodium acetate.

¹⁴Analysis performed if TGA<0.022(DSC-1200).

¹⁵Estimate of total number and mass of particles under 2µm in diameter is required.

¹⁶Analysis to be performed if TGA<25wt%.

¹⁷Analysis to be performed if TOC>30,000 or if AC/RSST indicates propagation.

¹⁸Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.

¹⁹Analysis to be performed if TOC does not account for the exotherm.

²⁰Analysis to be performed if TOC by persulfate does not account for the exotherm.

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-105 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 SAP shall have approved and implemented quality assurance project plans. These QA plans shall meet the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994) 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* (Sparks 1995), *Fiscal Year 1996 Tank Waste Analysis Plan* (Homi 1995b), 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 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 personally contact the laboratory to run analyses on the archived composite samples. Therefore, the Pretreatment Program has directed 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).

Several requested analytical methods are currently not available. The organic and ferrocyanide DQO documents (Turner, et al. 1995) (Meacham, et al. 1995) require determination of total organic carbon (TOC) by furnace oxidation if the energy equivalent by TOC does not match 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 on solid samples until new methods are developed and/or new instrumentation is available. Analyses may then be performed on archived samples. The organic DQO also requires analysis for organic acids by high performance liquid chromatography (HPLC) and organic complexants by gas chromatography/mass spectroscopy (GC/MS) if the waste exhibits propagation

during adiabatic calorimetry testing. The organic test plan (Meacham 1995) also requires HPLC and organic complexants by derivitization followed by GC/MS when the TOC is greater than or equal to 3 wt%. Selected organic acids will be determined by ion chromatography, however, the 222-S laboratory currently has no procedures for determination by HPLC. A GC/MS method exists at the 222-S Laboratory for the analysis of some organic species, but there is no method for organic complexants by derivitization and GC/MS. Should a method be developed, analyses may be performed on archived samples.

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).

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

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.

- ▶ The safety screening DQO (Dukelow, et al. 1995) requires that additional analyses be performed if total alpha activity is 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):

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

NOTE: Samples measured for total alpha 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.

- ▶ 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 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 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 required on half-segments based on this equation. Therefore, 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.

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 the Tank Cognizant Engineer by calling the West Tank Farm Operations Shift Manager within two working days (Kristofzski 1995b). This verbal notification must be followed within one working day by written communication to the Safety Screening Representative, Analytical Services, Process Control, the Historical DQO point of contact, and the Characterization Program Office documenting the observations (Schreiber 1994b). 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 Program, Process Control, the Tank Characterization Resource Center, Tank Characterization Database, and Historical DQO point of contact (Schreiber 1994b) within 45 days of the issuance of this sampling and analysis plan. 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 ferrocyanide, organic, organic test plan, and historical DQOs (see Table 1 for delineation of program requests). The data package shall be provided to Analytical Services, the Characterization Program, 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 on the same day that the final data package is issued. 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 to make decisions concerning a sample without review of the data by the customer or the Characterization Program. 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 and Reports via an Engineering Change Notice to this plan. All such changes shall also be clearly documented in the final data report. Any non-significant changes may be made by the project coordinator in the permanent record (e.g., a notation in the extrusion logbook or a memo to file); the significance of any change is determined solely by the project coordinator.

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 or issuance of a letter of instruction.

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