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TANK 241SX108 AUGER SAMPLING & ANALYSIS PLAN

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Assistant Secretary for Environmental Management



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LIST OF TERMS

DQO	data quality objective
DSC	Differential Scanning Calorimetry
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma (atomic emission spectroscopy)
RSST	Reactive System Screening Tool
SAP	Sampling and Analysis Plan
SARP	Safety and Analysis Report for Packaging
TCP	Tank Characterization Plan
TGA	thermogravimetric analysis
TOC	total organic carbon
TWRS	Tank Waste Remediation System
WHC	Westinghouse Hanford Company

TANK 241-SX-108 AUGER SAMPLING AND ANALYSIS PLAN

1.0 SAMPLING AND ANALYSIS OBJECTIVES

This Sampling and Analysis Plan (SAP) identifies characterization objectives pertaining to sample collection, laboratory analytical evaluation, and reporting requirements for the 241-SX-108 auger sampling event in accordance with the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). The analytical requirements in *Historical Model Evaluation Data Requirements* (Simpson and McCain 1995) will be applied as well. This is a secondary priority in this document.

If sufficient sample is recovered, composite material will be retained for pretreatment studies as requested in Kupfer et al. (1995). These data quality objectives (DQOs) are described in the Tank Characterization Plan (TCP) for tank 241-SX-108 (Homi 1995). This SAP also identifies procedures and requirements for collecting and characterizing samples from tank 241-SX-108 by the auger sampling method.

Quality requirements for conducting Tank Waste Remediation System (TWRS) Characterization Project sampling and analysis are described in the *TWRS Characterization Program Quality Assurance Program Plan* (Whelan 1994), in the *Fiscal Year 1995 Tank Waste Remediation System Tank Waste Analysis Plan* (Haller 1994), and in this SAP. Characterization Project sampling and analysis will be conducted in conformance to these requirements.

2.0 SAMPLING EVENT REQUIREMENTS

Tank 241-SX-108 contains about 115 kgal of waste, which corresponds to a waste height of about 48 in. as measured from the baseline of the tank. The waste consists of sludge with some drainable liquid (Brevick et al. 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 ft of the waste surface and the data reported as a percentage of the lower flammability limit. 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 241-SX-108 is currently scheduled to be auger sampled. Samples shall be collected from risers R7, R16, and R17 of the tank. If a different riser meets the intent of other requirements in the DQO, it may be used if the riser number is recorded and approved, in

writing, in advance by the sampling cognizant engineer. The risers used may be recorded on a permanent data sheet, or recorded directly in the work package.

Based on current waste volume information, one 20-in. auger bit will be used for sampling each riser. However, only 15 in. of waste will be taken to avoid exceeding the Safety and Analysis Report for Packaging (SARP) limits of the work procedure TO-080-090. The objective of this sampling event is to obtain a vertical profile of the top layer of the waste. Further sampling is scheduled for the future. For detailed information about sampling activities, refer to work package WS-95-00129. This document contains the operating procedures and the chain-of-custody records for this sampling event. All collected samples shall be shipped to the laboratory according to procedure TO-080-090 ("Load/Transport Sample Cask(s)"). Samples shall be transported to the laboratory within three calendar days after a sample is removed from the tank.

3.0 LABORATORY ANALYSIS REQUIREMENTS

3.1 ANALYSIS SCHEME

To comply with the data measurements of the safety screening DQO, the historical model data requirements DQO, and Pretreatment Program DQO, the following must be accomplished for each sample.

- Extrude solids according to laboratory procedure LO-160-103.
- Analyze samples on a homogenized half-segment basis according to Table 1.
- Archive samples on a homogenized half-segment basis.
- Analyze core composite samples according to Table 1.
- Filter liquids according to procedure LA-505-101 (or centrifuge and decant according to procedure LO-160-103) liquids and analyze according to Table 1.
- Provide core composites to the Pretreatment Program, if sufficient sample material is available after safety screening analyses are complete.

As a precautionary measure, the Safety and Analysis Report for Packaging (SARP) in the work procedure TO-080-090 ("Load/Transport Sample Cask(s)") has been reviewed for any safety issues involved with the transporting samples from tank 241-SX-108. For samples from tank 241-SX-108, the shipping containers must be vented every 15 days from the time of the cask sealing to release any retained gas.

Table 1. Tank 241-SX-108 Chemical, Radiological, and Physical Analytical Requirements.
(sheet 1 of 3)

SOLID ANALYSES															
Project Name		SX-108 Auger Sample			COMMENTS				REPORTING LEVELS						
Plan Number		WHC-SD-WM-TSAP-007, REV. 0			Homogenization Test - Per Laboratory Discretion				FORMAT I						
PROGRAM		PROGRAM CONTACTS			Field Blank -Not Required				FORMAT II						
A. Safety Screening		Safety Screening E. J. Lipke			Hot Cell Blank - Per Laboratory Discretion				FORMAT III						
B. Historical Model Evaluation		Historical B. C. Simpson			Lithium Bromide Solution Blank - Not Required				FORMAT IV						
		TWRS R. F. Eggers			TANK				FORMAT V						
					SX-108				FORMAT VI						
					3				Special						

Table 1. Tank 241-SX-108 Chemical, Radiological, and Physical Analytical Requirements.
(sheet 2 of 3)

PROGRAM	PRIMARY ANALYSES			SAMPLE [1]		QUALITY CONTROL [3]					CRITERIA		FOR-MAT		
	METHOD	ANAL.	WHC PROCEDURE	% SEG	SLD COMP	DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS		NOTIFICATION LIMIT [4]	EXPECTED RANGE [4]
1 1/2 SEG - 1/2 segment, SLD COMP- solid core composite															
24-direct, fusion dissolution, a-acid dissolution, w-water dissolution															
3PR-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 applicable, mtr-matrix															
4Units for notification limits and expected range are those listed in the "units" column.															
5Dry weight basis.															
6Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.															
8Tracer or carrier may be used in place of a spike and results corrected for recovery.															
9Either serial dilutions or matrix spikes will be performed.															
10This analysis required if DSC exceeds 480 J/g. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.															
11Fe, Mn, and U are secondaries for safety screening and are reported within 90 days if total alpha exceeds notification limit.															
12These analyses are secondary analyses for the safety screening DQO. Therefore, if the DSC limit is exceeded, these analyses must be performed and reported within 90 days of receipt of the last sample at the laboratory dock.															
13 The historical fingerprint analyses for SX-108 are Na, Al, Cr, % water, Cs-137, and Sr-90.															
14 Performed only after consultation with TWRS point of contact.															
15 Total alpha analyses required on lower half-segment only.															
16 Gravimetric analysis performed if moisture content is less than 25% by TGA and sufficient sample remains.															

Table 1. Tank 241-SX-108 Chemical, Radiological, and Physical Analytical Requirements.
(sheet 3 of 3)

LIQUID ANALYSES																	
Project Name		SX-108 Auger Sample				COMMENTS				REPORTING LEVELS							
Plan Number		WHC-SD-WM-TSAP-007, REV. 0				Homogenization Test - Per Laboratory Discretion				FORMAT I		Early Notify					
PROGRAM		PROGRAM CONTACTS				Field Blank -Not Required				FORMAT II		Process Control					
A. Safety Screening		Safety Screening				E. J. Lipke				FORMAT III		Safety Screen					
B. Historical Model Eval.		Historical				B. C. Simpson				FORMAT IV		Waste Management					
TWRS		R. F. Eggers				#AUGERS				FORMAT V		RCRA Compliance					
						SX-108				3		FORMAT VI		Special			
PROGRAM	METHOD	PRIMARY ANALYSES			PREP [2]	QUALITY CONTROL 3						CRITERIA		FOR- MAT			
		ANAL.	WHC PROCEDURE	S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT [4]		EXPECTED RANGE [4]		
A	DSC	Energy	LA-514-113 or LA-514-114	X	d [6]	ea smpl	N/A	N/A	ea AB	±10	90-110	J/g [5]	> 480	unknown	I, III		
A	TGA	% H2O	LA-560-112 or LA-514-114	X	d [6]	ea smpl	N/A	N/A	ea AB	±10	90-110	wt%	none	unknown	I, III		
A	Visual	Organic Layer	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	N/A	N/A	presence	unknown	I, III		
PROGRAM	METHOD	SECONDARY ANALYSES			PREP [2]	QUALITY CONTROL 3						CRITERIA		FOR- MAT			
		ANAL.	WHC PROCEDURE	S-LEV LIQ		DUP	SPK/ MSD	BLK	CALIB STD	PR	AC	UNITS	NOTIFICATION LIMIT [4]		EXPECTED RANGE [4]		
A	RSST [12]	Energy	see 12 below	X	d [6]	N/A	N/A	N/A	ea AB	±20	80-120	J/g [5]	> 480	unknown	I, III		
A	Hot Persulfate [13]	TOC	LA-344-100	X	d [6]	ea smpl	1/mtrix	ea AB	ea AB	±20	80-120	µg C/g [5]	> 30,000 [11]	unknown	I, III		
A	Distillation [13]	CN	LA-695-102	X	d [6]	ea smpl	1/mtrix	ea AB	ea AB	±10	90-110	µg/mL	> 39000 [11]	unknown	I, III		

1 S-LEV LIQ-liquid taken from the segment level.

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

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 applicable, mtrix-matrix

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

5 Dry weight basis.

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

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

9 Either serial dilutions or matrix spikes will be performed.

10 This analyses is required if DSC exceeds 480 J/g. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

11 Corrected from weight basis to volumetric basis assuming a liquid density of 1.0 g/mL.

12 RSST performed only if DSC exceeds notification limit. The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

13 This analysis required if DSC exceeds notification limits.

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. Table 1 shows the reporting formats for analyses.

3.2 SPECIFIC METHODS AND ANALYSES

Table 1 shows the analyses to be performed on the tank 241-SX-108 auger samples, based on the safety screening DQO and the historical model data requirements DQO referenced in Section 1.0. Also, the applicable analytical procedures are listed in Table 1.

Procedures used in sample preparation may include procedures for fusion (LA-549-141); acid digestion of aqueous samples (LA-505-158); acid digestion of solids (LA-505-159); and water leach of solids (LA-504-101).

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 applicable SAP and permit a minimum 10 mL archive sample, the laboratory shall notify the TWRS sample coordinator 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

Work conducted under this Sampling and Analysis Plan will be conducted in conformance with *TWRS Characterization Program Quality Assurance Plan* (Whelan 1994). The Westinghouse Hanford Company 222-S Laboratory has a quality assurance plan (Meznarich 1995) that describes the primary quality assurance measures to be applied, when analyzing waste tank samples. Laboratories performing analyses in support of this SAP shall have approved and implemented quality assurance project plans. These plans shall meet the minimum requirements given in the *Hanford Analytical Services Quality Assurance Plan* (DOE 1994) as the baseline for laboratory quality systems.

Sample quality control measures (duplicates, spikes, standards) are identified in Table 1. If no criteria are provided, the performing laboratory shall perform according 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 for which special precautions must be made. If deemed necessary by the sampling and tank cognizant engineers, video surveillance should be performed to help identify 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 applicable 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 1 working day of removing the sample from the tank, but must transport each segment or sample within 3 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 ORGANIZATION

Table 2 lists the organization and responsibility of key personnel involved with this tank 241-SX-108 characterization project.

Table 2. Tank 241-SX-108 Project Key Personnel List.

Individual	Organization	Responsibility
R. F. Eggers	TWRS Characterization Plans, Coordination, and Reports	Tank Sample Coordinator
E.J. Lipke	WHC Safety Program	Safety Screening Point of Contact
B.C. Simpson	TWRS Characterization Technical Basis Integration	Historical DQO Point of Contact
M.J. Kupfer	TWRS Disposal Engineering	Pretreatment Point of Contact
West Tank Farm Operations Shift Manager	Tank Farm Operations	200 West Tank Farm Point of Contact if Action Limit is Exceeded (373-3475)

6.0 CLARIFICATIONS AND ASSUMPTIONS

6.1 CLARIFICATIONS AND ASSUMPTIONS

Many clarifications and assumptions relating to the notification limits, decision thresholds, or analytical strategies identified in the applicable DQOs need to be made with respect to the analyses in Table 1. Discussion of these follow:

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

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

Note: If there is greater than 90 percent water in a sample, converting to a dry weight basis may lead to a large error in the DSC value. However, the conversion is still required.

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

$$\left(\frac{1 \text{ g}}{\text{L}}\right) \left(\frac{1 \text{ L}}{10^3 \text{ mL}}\right) \left(\frac{1 \text{ mL}}{\text{density g}}\right) \left(\frac{0.0615 \text{ Ci}}{1 \text{ g}}\right) \left(\frac{10^6 \mu\text{Ci}}{1 \text{ Ci}}\right) = \frac{61.5 \mu\text{Ci}}{\text{density g}} \quad (2)$$

Note: If a density of 1.5 g/mL is assumed for solid material, the notification limit becomes 41 $\mu\text{Ci/g}$.

- If the analytical results on a sample fall below the minimum criterion for moisture content stated in Table 1, but the segment from which the sample was taken contained drainable liquid, the moisture content of the sample should be adjusted to reflect the drainable liquid present. That is, the drainable liquid should be distributed to each partial segment (on a relative mass basis) and the projected moisture content of the sample recalculated. If the adjusted moisture content still does not meet the criterion, then a notification to appropriate personnel may be made (see Section 7.1).
- The laboratory is expected to report all analytical results recovered from inductively coupled plasma spectroscopy (ICP), ion chromatography (IC), and gamma energy analysis (GEA), even though only specific analytes are requested. These results should be reported only if no additional preparatory work is required (e.g. running additional standards) or if the results meet internal quality control criteria. No reruns nor additional analyses should be performed to improve recovery for analytes not specifically requested in Table 1.
- Based on the data collected for each analyte a mean value of the concentration will be determined. If an upper notification limit is established for the analyte in Table 1, the 95 percent confidence upper limit of the true concentration of the analyte will be determined and compared to this notification threshold. If the 95 percent confidence upper limit of the true value exceeds the notification limit notification is made. The equation for determining the upper 95 percent confidence limit of the true value of the concentration is

$$\hat{\mu} + t_{(n-1)} * \frac{\sqrt{\hat{\sigma}^2}}{\sqrt{n}} \quad (3)$$

The equation for determining the lower 95 percent confidence limit of the true value is

$$\hat{\mu} - t_{(n-1)} * \frac{\sqrt{\hat{\sigma}^2}}{\sqrt{n}} \quad (4)$$

$\hat{\mu}$ = sample mean
 t_{n-1} = Student t statistic with n-1 degrees of freedom
 $\hat{\sigma}^2$ = sample variance

These equations are appropriate for confidence limit estimates of the mean when the sample size is small. This equation, and a table of values for the Student t statistic, is available in an introductory statistics textbook (e.g. Lapin 1983). For a sample run in duplicate, n equals 2.

7.0 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).

7.1 FORMAT I REPORTING

Table 1 contains the notification limits for each analyte. Any results exceeding their notification limits shall be immediately reported (upon review by the laboratory shift manager) via the 222-S shift office by calling the West Tank Farm Operations Shift Manager (Kristofzski 1995). 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.

7.2 FORMAT III REPORTING

A Format III report of 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, and the Tank Characterization Resource Center, Tank Characterization Database, and Historical DQO point-of-contact (Schreiber 1994b) 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 provided in Schreiber (1994a).

7.3 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 Analytical Services, the Characterization Program, the historical DQO point of contact, and the Tank Characterization Resource Center and Tank Characterization Database representatives within 216 days of the sampling event. Detailed information regarding the contents of this reporting format are provided 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 (1994).

8.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Program. These changes shall be discussed with the TWRS sample coordinator for minor low-impact changes and documented in applicable laboratory reports. All significant changes (such as changes in scope) shall be documented by via an Engineering Change Notice to the appropriate Tank Characterization Plan (Homi 1995) or this Sampling and Analysis Plan. All changes shall also be clearly documented in the final data package.

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

9.0 REFERENCES

- Dukelow, G. T., J. W. Hunt, H. Babad, J. E. Meacham, 1995, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 2, Westinghouse Hanford Company, Richland, Washington.
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