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## Tank 241-SX-105 Rotary Mode Core Sampling and Analysis Plan

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
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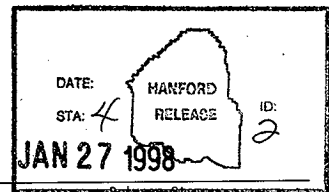
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# **Tank 241-SX-105 Rotary Mode Core Sampling and Analysis Plan**

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

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## LIST OF ABBREVIATIONS

Ci	curie
CPO	Characterization Project Operations
DQO	data quality objective
DSC	differential scanning calorimetry
g	gram
g/L	gram per liter
GEA	gamma energy analysis
IC	ion chromatography
ICP/AES	inductively coupled plasma - atomic emission spectroscopy
kgal	kilogallon
kL	kiloliter
L	liter
LFL	lower flammability limit
LiBr	lithium bromide
LMHC	Lockheed Martin Hanford Corporation
mL	milliliter
MOU	memorandum of understanding
PHMC	Project Hanford Management Contractor
PIC	person in charge
PRSST	propagating reactive system screening tool
QA	quality assurance
QC	quality control
RSST	reactive system screening tool - adiabatic calorimetry
SX-105	single-shell tank 241-SX-105
SAP	sampling and analysis plan
TGA	thermogravimetric analysis
TOC	total organic carbon
TWRS	Tank Waste Remediation System
WMH	Waste Management Hanford
$\mu$ Ci	microcurie
$\mu$ Ci/g	microcuries per gram

## 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 rotary mode core samples from tank 241-SX-105 (SX-105). It is written in accordance with *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995) and *Memorandum of Understanding for the Organic Complexant Safety Issue Data Requirements* (Schreiber 1997a). Vapor screening issues apply as well, but are outside the scope of this SAP.

## 2.0 SAMPLING EVENT REQUIREMENTS

As of October 31, 1997, tank SX-105 contained 2,585 kL (683 kgal) of waste (Hanlon 1997). This waste volume is equivalent to 645 centimeters (254 inches) of waste as measured from the centerline of the tank. The tank contents are comprised of 2,309 kL (610 kgal) of saltcake and 276 kL (73 kgal) of sludge, with 1,170 kL (309 kgal) of drainable interstitial liquid (Hanlon 1997). A physical profile prediction based on waste fill history and previous sampling information is provided in Appendix A.

Prior to core sampling, the dome space (below the riser) shall be measured for the presence of flammable gases. The measurement shall be taken from within the dome space and the data reported as a percentage of the lower flammability limit (LFL). The results shall be transmitted to the tank coordinator within ten working days of the sampling event (Schreiber 1997b). If the results are above 25 percent of the LFL when analyzing by gas chromatography/mass spectrometry or gas-specific monitoring gauges or above 10 percent 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 Safety Project. Any additional vapor sampling is not within the scope of this SAP.

Tank SX-105 will be rotary mode core sampled using a rotary mode core sampling system. Two core samples, each consisting of approximately 14 segments (13 complete and 1 partial), are expected to be taken from risers 3 and 14. The sampling objective is to obtain two vertical profiles of the waste; therefore, more or fewer segments may need to be taken depending on the accuracy of the current waste volume records.

Universal samplers will be used for these samples. If quality-affecting changes to the sampling requirements must be made (including the risers, sampling truck, or segments to be sampled), the change must be recorded and approved by the cognizant engineer and tank coordinator before sampling. This information may be recorded on a permanent data sheet or recorded directly in work packages WS-97-00207 (riser 3) and WS-97-00208 (riser 14). These work packages contain the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank SX-105 shall be obtained in accordance with procedure TO-060-003. The Characterization Project Operations (CPO) person in charge (PIC) or the PIC designate will verify that the field blank is properly created and shipped. For sampling events having multiple PICs, CPO shall determine which PIC will be responsible for the field blank. This field blank is to accompany the samples to the laboratory. All collected samples shall be shipped to the laboratory following the Load/Transport Sample Cask(s) procedure (TO-080-090). Core samples should be transported to the laboratory within three calendar days from the time each segment is removed from the tank.

If lithium bromide (LiBr) solution is used in the collection of the core samples, it should be a  $0.3 \pm 0.01$  molar solution with a pH greater than 8. Characterization Project Operations must state the batch number and amount of fluid added at each segment. This information should be indicated on the chain-of-custody form that accompanies the sample to the laboratory. A sample of the LiBr solution must be provided to the laboratory. This sample shall consist of a container filled with LiBr solution from the same batch of LiBr solution used during the sampling. This solution shall be analyzed for lithium and bromide in order to determine the concentration of the tracer at the time the sample was taken. If analysis of the waste samples indicates contamination by the LiBr solution, these data will be used to determine the amount of contamination. If more than one batch of LiBr solution is used during the sampling event, one solution sample must be provided for each batch in addition to the field blank.

### 3.0 LABORATORY ANALYSIS REQUIREMENTS

#### 3.1 ANALYSIS SCHEME

In order to comply with Dukelow et al. (1995) and Schreiber (1997a), the following steps shall be performed on each sample:

- Extrude segments, videotaping the extrusion and photographing the extruded segments. The extrusion procedure is LO-160-103 at the 222-S Laboratory.
- Filter or centrifuge drainable liquids as needed prior to analysis.
- Separate solids into half-segments and homogenize.
- Analyze drainable liquids and homogenized half-segment subsamples from each core as shown in Tables 1 and 2.
- Archive at least 10 mL of each subsegment and drainable liquid samples, if possible (Bratzel 1994).

If liner liquid is observed during extrusion and the liquid is in sufficient quantity to collect, the liner liquid may be retained and analyzed at the discretion of the tank coordinator. In this event, this addition of analyses may not require a revision to this SAP.

As a precautionary measure, the Safety and Analysis Report for Packaging shall be reviewed for any safety issues involved with transportation of tank SX-105 core samples.

Table 1: Tank SX-105 Chemical, Radiological, and Physical Analytical Requirements: Solids (2 Sheets)

SOLID ANALYSES												
Project Name			COMMENTS				REPORTING LEVELS					
Plan Number			Homogenization Test - Per Laboratory Discretion				FORMAT I		Immediate Notification			
PROGRAM			Field Blank - Required				FORMAT II		Process Control			
A. Safety Screening			Hot Cell Blank - Per Laboratory Discretion				FORMAT III		Safety Screening			
B. Organic			LiBr Solution Blank - Required if LiBr solution is used				FORMAT IV		Waste Management			
C. Process Control			TANK				FORMAT V		RCRA Compliance			
			SX-105				FORMAT VI		Special			
			2									
PRIMARY ANALYSES			SAMPLE <sup>1</sup>			QUALITY CONTROL <sup>3</sup>			CRITERIA			
			PREP <sup>2</sup>									
			CORE COMP									
PROGRAM	METHOD	ANALYSIS	PROCEDURE	1/2 SEG SLDG/ SC	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT <sup>4</sup>	EXPECTED RANGE <sup>4</sup>	FORMAT
A, B	DSC	Energy	LA-514-113 LA-514-114	x	d	ca smpl	N/A	ca AB	J/g	≥ 480 <sup>5,6</sup>	0	I, III, IV
A, B	TGA	% H <sub>2</sub> O	LA-560-112 LA-514-114	x	d	ca smpl	N/A	ca AB	wt%	none	38.3	III, IV
A	Gravimetry	Bulk density	LO-160-103	x <sup>7</sup>	d	N/A	N/A	N/A	g/mL	none	1.59	III, IV
A	Alpha counting	Total Alpha	LA-508-101	x <sup>7</sup>	f or a	ca smpl	1/mtrx	ca PB	µCi/g	> 41 <sup>6</sup>	0.26	I, III, IV
C	IC	Br <sup>10</sup>	LA-533-105	x	w	ca smpl	1/mtrx	ca AB	µg/g	none	sec <sup>13</sup>	IV
A, C	ICP/AES	Li, Al, Cr, Fe, Mn, Na, Ni, Si, U <sup>11</sup>	LA-505-151 LA-505-161	x	f or a	ca smpl	1/mtrx <sup>12</sup>	ca PB	µg/g	none	sec <sup>13</sup>	III, IV
SECONDARY ANALYSES			SAMP <sup>1</sup>			QUALITY CONTROL <sup>3</sup>			CRITERIA			
			PREP <sup>2</sup>									
			CORE COMP									
PROGRAM	METHOD	ANALYSIS	PROCEDURE	1/2 SEG SLDG/ SC	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT <sup>4</sup>	EXPECTED RANGE <sup>4</sup>	FORMAT
A	Alpha counting	<sup>239,240</sup> Pu <sup>14</sup>	LA-953-104	x	f	ca smpl	1/mtrx <sup>8</sup>	ca PB	µCi/g	none	unknown	III, IV
A, B	Persulfate	TOC	LA-342-100	x <sup>9</sup>	d	ca smpl	1/mtrx	ca AB	µg C/g	> 30,000 <sup>5,6</sup>	1,150	I, III, IV
TERTIARY ANALYSES			SAMP <sup>1</sup>			QUALITY CONTROL <sup>3</sup>			CRITERIA			
			PREP <sup>2</sup>									
			CORE COMP									
PROGRAM	METHOD	ANALYSIS	PROCEDURE	1/2 SEG SLDG/ SC	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT <sup>4</sup>	EXPECTED RANGE <sup>4</sup>	FORMAT
B	Furnace Oxidation	TOC <sup>15</sup>	LA-344-105	x	w	ca smpl	1/mtrx	ca PB	µg C/g	none	1,150	IV
B	CZE	EDTA, HEDTA <sup>16,17</sup>	LA-533-113	x	w <sup>18</sup>	ca smpl	ca AB	ca PB	µg/g	none	unknown	IV



Table 1: Tank SX-105 Chemical, Radiological, and Physical Analytical Requirements: Solids (2 Sheets)

B	IC	acetate, glycolate, formate, oxalate, citrate, NTA, IDA <sup>16,19</sup>	LA-533-105 LA-533-115	x	w <sup>18</sup>	ea smpl	ea AB	ea PB	ea AB	µg/g	none	unknown	IV
B	Ion pair chromatography	EDTA, ED3A, HEDTA, EDDA <sup>16,17</sup>	sec <sup>20</sup>	x	w <sup>18</sup>	ea smpl	ea AB	ea PB	ea AB	µg/g	none	unknown	VI
B	Derivatization GC/MS and GC with FID	chelator and chelator fragments: EDTA, NTA IDA, ED3A, HEDTA, succinate, citrate <sup>6,17</sup>	sec <sup>20</sup>	x	w <sup>18</sup>	ea smpl	ea AB	ea PB	ea AB	µg/g	none	unknown	VI
B	PRST	propagating behavior <sup>16</sup>	LT-510-103	X	d	N/A	N/A	N/A	sec <sup>21</sup>	J/g	none	unknown	IV

<sup>1</sup>1/2 SEG SLD/GSC = 1/2 segment, sludge or saltcake; CORE COMP = core composite<sup>2</sup>d = direct, f = fusion, a = acid, w = water<sup>3</sup>DU = duplicate, BLK = blank, STD = calibration standard, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtr = matrix, N/A = not applicable<sup>4</sup>Units for notification limits and expected range are those listed in the "units" column.<sup>5</sup>Dry weight basis<sup>6</sup>These analytes are to be compared to the limit by calculating the one-sided, upper 95% confidence limit for the sample result (to be performed by Process Engineering).<sup>7</sup>These analyses required on the lower half-segment of each segment only.<sup>8</sup>Tracer or carrier may be used in place of a spike and results corrected for recovery.<sup>9</sup>TOC by the persulfate oxidation method is performed on a sample if the sample's DSC result exceeds 480 J/g, or on all tank samples if greater than 25% of the samples exhibit exothermic activity.  
<sup>10</sup>Br is required to correct %H<sub>2</sub>O measurement (to be performed by Process Engineering).<sup>11</sup>Results should be reported for all IC analytes. However, the QC requirements of Table 3 only apply to those identified in this table.<sup>12</sup>Li is required to correct %H<sub>2</sub>O measurement (to be performed by Process Engineering). Al, Cr, Fe, Mn, Ni, Si, and U are required as secondary safety screening analytes,<sup>13</sup>to be performed if total alpha activity exceeds notification limit. Results should be reported for all ICP/AES analytes. However, the QC requirements of Table 3 apply only to those identified.<sup>14</sup>Either serial dilutions or matrix spikes will be performed.<sup>15</sup>Al = 64100, Cr = 106000, Fe = 123000, Mn = 20.4, Na = 1330000, Ni = 858, Si = 1040, U = 1010,<sup>16</sup>Analysis to be performed if total alpha activity limit is exceeded.<sup>17</sup>Furnace oxidation TOC is to be run on those samples which exhibit exotherms, and have persulfate TOC results that account for <75% of the exotherm energy (see Equation 4)<sup>18</sup>These analytes are to be performed on the solid subsample with the highest TOC result if the ANOVA model classifies the tank as "conditionally safe" or "unsafe"<sup>19</sup>(to be performed by Process Engineering). Contact tank coordinator before proceeding with these analyses.<sup>20</sup>Analyses of interest are listed here. However, quantitative results or qualitative, if quantitative is not possible, should be reported for all analytes detected.<sup>21</sup>The solid sample with the highest TOC result is to be centrifuged prior to further analysis, and then both the solid and liquid fractions are to be analyzed.<sup>22</sup>If the subsample is too dry or crumbly to centrifuge, it need not be centrifuged.<sup>23</sup>Results should be reported for all organic acids IC analytes found; however, the requirements of Table 3 apply only to those analytes identified here.<sup>24</sup>These analyses will be performed at the PNNL 329 Laboratory. Methods are being formalized at this time. Analyses shall not be performed until the Laboratory has accepted procedures in compliance with HASQARD.<sup>25</sup>Calibrations of heater resistance, time, temperature, pressure, containment volume, and sample weight will be performed to measure accuracy as described in procedure LT-510-103.

Table 2: Tank SX-105 Chemical, Radiological, and Physical Analytical Requirements: Liquids (2 Sheets)

LIQUID ANALYSES												
Project Name		SX-105 Rotary Mode Core Sample			COMMENTS				REPORTING LEVELS			
Plan Number		HNF-2107 Rev. 0			Homogenization Test - Per Laboratory Discretion				FORMAT I			
					Field Blank - Required				FORMAT II			
PROGRAM		PROGRAM CONTACTS			Hot Cell Blank - Per Laboratory Discretion				FORMAT III			
A. Safety Screening		TWRS Safety R. J. Cash			LJB: Solution Blank - Required if HHE is used				FORMAT IV			
B. Organic		TWRS Safety J. E. Meacham			TANK				FORMAT V			
C. Process Control		TWRS Proc. Eng. B. C. Simpson			SX-105				FORMAT VI			
					#CORES				RCRA Compliance			
					2				Special			
					</							

<sup>1</sup>FB = field blank, LIQ = drainable liquid<sup>2</sup>d = direct<sup>3</sup>DUP = duplicate, BLK = blank, STD = calibration standard, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtrx = matrix, N/A = not applicable<sup>4</sup>Units for notification limits and expected range are those listed in the "units" column.<sup>5</sup>Dry weight basis<sup>6</sup>These analytes are to be compared to the limit by calculating the one-sided, upper 95% confidence limit for the sample result (to be performed by Process Engineering).

Table 2: Tank SX-105 Chemical, Radiological, and Physical Analytical Requirements: Liquids (2 Sheets)

- <sup>7</sup>Li is required to correct %H<sub>2</sub>O measurement (to be performed by Process Engineering). Al, Cr, Fe, Mn, Na, Ni, Si, and U are required as secondary safety screening analytes; required if total alpha exceeds notification limits. Results should be reported for all ICP/AES analytes, however, the QC requirements of Table 3 apply only to those identified here.
- <sup>8</sup>Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.
- <sup>9</sup>Either serial dilutions or matrix spikes will be performed.
- <sup>10</sup>Ammonia should be analyzed on both acidified and non-acidified samples.
- <sup>11</sup>Br is required to correct %H<sub>2</sub>O measurement (to be performed by Process Engineering). Results should be reported for all IC analytes, however, the QC requirements of Table 3 apply only to Br.
- <sup>12</sup>Micrograms ammonia per milliliter of as-received sample.
- <sup>13</sup>Performed if total alpha exceeds notification limit.
- <sup>14</sup>Tracer or carrier may be used in place of a spike and results corrected for recovery.
- <sup>15</sup>Corrected from weight basis to volumetric basis assuming a liquid density of 1.0 g/mL.
- <sup>16</sup>Furnace oxidation TOC is to be run on those samples which exhibit exotherms and have persulfate TOC values that account for <75% of the exothermic energy (see Equation 4)
- <sup>18</sup>TOC by the persulfate oxidation method is performed if the sample's DSC result exceeds 480 J/g, or on all tank samples if greater than 25% of the samples exhibit any exothermic activity.

For core samples from tank SX-105, the shipping container **must** be vented every 45 days to release any accumulated gas.

Opportunistic analyses as defined in Kristofzski (1996) may be included at the discretion of the tank 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 observations shall be reported in the data report. The reporting formats for analyses are contained in Tables 1 and 2 and are described in Section 7.0.

### 3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Tables 1 and 2 to be performed on tank SX-105 core samples are based on the safety screening data quality objective (DQO) and the organic complexant Memorandum of Understanding (MOU), referenced in Section 1.0. The laboratory procedure numbers to be used for the analyses are included in the tables. Sample preparation procedures that may be used at the 222-S Laboratory are LA-549-141 for fusion digestion, LA-505-159 or LA-505-163 for acid digestion of samples, and LA-504-101 for water leach of solids.

### 3.3 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from samples taken from the tank is insufficient to perform the analyses requested in the SAP and permit a minimum 10 mL archive per sample, the laboratory shall notify the tank coordinator within one working day. At that time, a prioritization of the analyses may be provided to the laboratory. 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 AND QUALITY CONTROL

Processes, services, activities, and conditions adverse to quality which do not conform to requirements specified in this SAP or references herein shall be controlled to prevent inadvertent use. Nonconforming sampling and analysis processes shall be identified, processed, reported, and dispositioned as required by the *Project Hanford Policy and Procedure System* (PHMC 1997), "*Nonconformance Item Reporting and Control*."

### 4.1 LABORATORY OPERATIONS

Laboratories performing analyses in support of this SAP shall have approved and implemented Quality Assurance (QA) Plans. These QA plans shall meet the *Hanford Analytical Services Quality Assurance Requirements Document* (DOE 1997) minimum requirements as the baseline for laboratory quality systems. The *222-S Laboratory Quality Assurance Plan* (Markel 1997) specifies the requirements for assuring the quality of sample analysis conducted at the 222-S Laboratory. Quality requirements for conducting Characterization Project sampling and

analysis are described in *Tank Waste Remediation System Characterization Project, Quality Policies* (Board 1997) and this SAP. Characterization Project sampling and analysis shall be conducted in conformance with these QA requirements.

Analytical quality control (QC) requirements (duplicates, spikes, blanks, laboratory control samples) are identified in Tables 1 through 3. The laboratory shall also use calibration and calibration check standards appropriate for the analytical instrumentation being used (see DOE [1997] for definitions of QC samples and standards). The criteria presented are goals for demonstrating reliable method performance. It is understood that the laboratory will follow its internal QC system for required actions whenever QC failures occur. If sample QC failures occur or if all analyses cannot be performed (e.g., insufficient sample), analysts shall consult with supervisors/customers to determine the proper action. The laboratory should provide a suggested course of action at that time. All sample QC failures and limitations on the associated data shall be discussed in the narrative of the data report. Proper notification of all data not meeting QC requirements shall be included with the data.

#### 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 taken if needed. If deemed necessary by the sampling cognizant engineers and tank coordinator, 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 CPO in accordance with the respective work package(s). The chain-of-custody form for this work package shall identify samples by a unique number and state the type of sampler used (universal sampler or RGS sampler) for each sample before being shipped to the 222-S Laboratory. Approved procedure TO-080-090 [Load/Transport Sample Cask(s)] is to be used during the sampling event. Pertinent sampling information (e.g., unusual waste characteristics, x-ray scan results, or detecting possible debris) should also be entered in the comment section of the chain-of-custody form.

Characterization Project Operations should transport each sample collected to the performing laboratory within 3 calendar days of removing the sample from the tank. A verbal notification by CPO is to be made to the 222-S Laboratory at 373-2435 at least 24 hours in advance of an expected shipment.

Table 3. QC Precision and Accuracy Requirements for the Analyses

Analysis/Method	Duplicate Criteria (RPD) <sup>1</sup>	Spike Criteria (% recovery) <sup>2</sup>	Preparation Blank Criteria <sup>3</sup>	LCS Criteria (% recovery) <sup>4</sup>
<b>Solids</b>				
DSC	≤ 30	N/A	N/A	80 - 120
TGA	≤ 30	N/A	N/A	80 - 120
Bulk density	N/A	N/A	N/A	N/A
Total alpha	≤ 20	75 - 125	< MDA	70 - 130
ICP/AES	≤ 20	75 - 125	< EQL	80 - 120
IC	≤ 20	75 - 125	< EQL	80 - 120
TOC	≤ 20	75 - 125	< EQL	80 - 120
<sup>239/240</sup> Pu	≤ 20	75 - 125 <sup>5</sup>	< MDA	70 - 130
PRSST <sup>6</sup>	N/A	N/A	N/A	N/A
Organic speciation <sup>7</sup>	≤ 30	70 - 130	< EQL	70 - 130
<b>Liquids</b>				
DSC	≤ 20	N/A	N/A	80 - 120
TGA	≤ 20	N/A	N/A	80 - 120
Specific gravity	≤ 20	N/A	N/A	N/A
Total alpha	≤ 20	75 - 125	< MDA	70 - 130
ICP/AES	≤ 20	75 - 125	< EQL	80 - 120
IC	≤ 20	75 - 125	< EQL	80 - 120
TOC	≤ 20	75 - 125	< EQL	80 - 120
<sup>239/240</sup> Pu	≤ 20	75 - 125 <sup>5</sup>	< MDA	70 - 130

Table 3. QC Precision and Accuracy Requirements for the Analyses

Analysis/Method	Duplicate Criteria (RPD) <sup>1</sup>	Spike Criteria (% recovery) <sup>2</sup>	Preparation Blank Criteria <sup>3</sup>	LCS Criteria (% recovery) <sup>4</sup>
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Notes:

N/A = not applicable

MDA = minimum detectable activity

EQL = estimated quantitation limit

LCS = laboratory control standard

<sup>1</sup>For the calculation of the relative percent difference (RPD), both the sample and duplicate results must exceed the EQL or MDA. Failures are permissible if the requirements in the QA section are followed.

<sup>2</sup>The criteria are recommended. Failures are permissible if the requirements in the QA section are followed.

<sup>3</sup>When a blank exceeds the EQL or MDA, sample results that exceed the contribution from the blank twenty-fold or more are reportable. See also the QA section of this SAP.

<sup>4</sup>For some analyses, this could be a method spike or a blank spike. Ranges are percent recovery of theoretical.

<sup>5</sup>A tracer or carrier may be substituted for the spike.

<sup>6</sup>See Section 5.0.

<sup>7</sup>These requirements apply to the organic acids IC, GC/MS, CZE, and ion pair chromatography analyses (see Table 1).

### 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 (see below).

WASTE TANK SAMPLE SEAL	
Supervisor	Sample No.:
Date of Sampling:	Time of Sampling:
Shipment No.:	Serial No.:

Each sample number shall be created using the sample's core and segment number. For instance, segment 1 of core 197 would be sample number 197-01. 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 222-S Laboratory are described in laboratory procedure LO-090-101.

## 5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

### 5.1 EXCEPTIONS TO DQO REQUIREMENTS

The safety screening DQO (Dukelow et al. 1995) states that cyanide analysis is required when the energetics notification limit is exceeded and the total organic carbon (TOC) is less than its notification limit. Because the ferrocyanide safety issue has been closed, cyanide analysis is no longer required as a secondary analyte under the safety screening DQO (Cash 1997).

Analysis by the Reactive System Screening Tool (RSST) method is currently required as a secondary analysis by the Safety Screening DQO. However, the energetics requirements of the Safety Screening DQO are superseded by those in the organic complexant MOU, which requires Propagating Reactive System Screening Tool (PRSST) analysis instead of RSST testing. As such, the RSST requirement in Tables 1 and 2 has been removed.

Analysis for organic solvents such as tributyl phosphate and normal paraffin hydrocarbons is required by the organic complexant MOU (Schreiber 1997a). However, this MOU is undergoing revision to delete the analytical requirement. Because this analysis is not necessary, the requirement has been deleted from this SAP.

### 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 Tables 1 and 2. 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 percent 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. For solids, 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  $^{239}\text{Pu}$ . The notification limit may then be calculated as shown in equation 2:



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

**NOTE:** Solid samples measured for total alpha activity shall also be measured for density. The notification limit for solid subsamples shall be 41  $\mu\text{Ci/g}$  until the density is measured. At such time, the notification limit will be adjusted according to equation 2 using the highest recorded density, and the total alpha activity results will be reevaluated against the new limit.

- The safety screening DQO (Dukelow et al. 1995) states that the analytical results should be compared to their notification limits at a 95% confidence level (one-tailed test). The equation for determining the upper confidence value is shown in equation 3.

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

Where  $\hat{\mu}$  is the sample mean,  $\hat{\sigma}^2$  is the sample variance, n is the number of observations (for a sample run in duplicate, n equals 2), and  $t_{(n-1)}$  is the quantile from Student's t distribution with (n-1) degrees of freedom (for a one-sided 95% confidence interval and when n is 2, t is 6.314). This equation is appropriate for confidence limit estimates of the mean when the sample size is small. This equation, as well as a table of values for the Student t statistic, should be found in any introductory statistics textbook (e.g., Lapin 1983).

- The laboratory is requested to report all analytical results recovered from the inductively coupled plasma - atomic emission spectroscopy (ICP/AES) and ion chromatography (IC) analyses, even though only specific analytes are requested. These opportunistic analyses (Kristofzski 1996) should be reported only if no additional preparatory work is required (e.g., running additional standards) and if the error associated with the results is documented. No reruns nor additional analyses need to be performed to improve recovery for analytes not specifically requested in Tables 1 or 2.

The organic complexant MOU requires TOC analysis by furnace oxidation to be performed on samples if the results from TOC analysis by persulfate oxidation do not account for 75% of the exotherms observed in the DSC analysis. The energy equivalent of TOC is given in equation 4.

$$X = (\text{wt\% TOC dry weight basis}) * \frac{1,200 \text{ J/g}}{4.5} \quad (4)$$

**NOTE:** 1,200 J/g represents the energy equivalent of 4.5 wt% TOC (based on sodium acetate average energetics standard).

If tank SX-105 is found to be "conditionally safe" or "unsafe", PRSST and organic speciation analyses are performed on the solid subsample with the highest TOC result (see Table 1). Prior to organic speciation analysis, the subsample is to be centrifuged, and both the solid and liquid fractions are to be analyzed. If the original solid subsample is too dry to centrifuge effectively, the centrifugation step may be omitted.

Tank SX-105 is not a bounding tank for the organic complexant safety issue. If some of the tank samples have exothermic activity, but less than 25% of the total tank samples show exothermic activity, the tank coordinator is to be notified. Additional instruction to the laboratory may be provided at that time, if necessary.

No specific requirements for analytical accuracy and precision are presented with respect to the PRSST method. It is understood that the final results of this measurement are strongly subject to interpretation and that accuracy is dependent upon absolute calibration. To that end, accuracy must be maintained through the calibration of heater resistance, time, temperature, pressure, containment volume, and sample weight as described in procedure LT-510-103.

## 6.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank SX-105 characterization project are listed in Table 4.

Table 4. Tank S-110 Project Key Personnel

Responsibility	Organization	Individual
TWRS SX-105 tank coordinator	TWRS Process Engineering (LMHC)	B. C. Simpson, 373-5915
222-S Laboratory point of contact	Analytical Services (WMH)	Dayshift: D. B. Hardy, 376-4878 After hours: 222-S Laboratory shift manager, 373-2435
200 West Tank Farm point of contact	Tank Farm Operations	West Tank Farm Operations shift manager, 373-3475

## 7.0 DELIVERABLES

All analyses will be reported as Format I, III, or IV as indicated in Tables 1 and 2. Additional information regarding reporting formats is given in Schreiber (1997b).

### 7.1 FORMAT I REPORTING

Tables 1 and 2 contain 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 West Tank Farm Operations shift manager as soon as the data are obtained and reviewed by the responsible scientist. This verbal notification must be followed within one hour by electronic notification to the tank farm operations shift manager, the TWRS Process Engineering Data Assessment and Interpretation manager, and the tank coordinator responsible for the tank. Additional analyses for verification purposes may be contracted between the performing laboratory and the tank coordinator by either a revision to this SAP or by a letter.

## 7.2 FORMAT III REPORTING

Analyses identified as primary safety screening analyses in Tables 1 and 2 must be completed within 45 calendar days of the receipt of the last sample at the laboratory sample receiving/loading dock. If no safety screening criteria were exceeded, the laboratory shall electronically notify the tank coordinator and shall follow the notification with a letter to the TWRS Process Engineering Data Assessment and Interpretation manager and the tank coordinator confirming work completion. If any analysis results exceeded the safety screening criteria, a letter identifying the results which exceeded the criteria will be issued.

Any secondary safety screening analyses must be completed within 90 calendar days of the receipt of the last sample at the laboratory sample receiving/loading dock. When the secondary analyses are complete, the laboratory shall issue a letter to the TWRS Process Engineering Data Assessment and Interpretation manager and tank coordinator confirming work completion. If any secondary analysis notification limits were exceeded, the results which exceeded the limits shall be identified.

## 7.3. FORMAT IV REPORTING

The format IV report shall be a data package reporting the results of analyses performed and will resemble a regulatory data package without third party validation. The data package should be prepared by tank and include the data for all samples, including (as applicable) composites, segments, subsegments, drainable liquids, and associated blanks taken and analyzed for this sampling event. The recommended reporting format and the raw data that shall be included are given in detail in Section A5.0 of Schreiber (1997b). This data package shall be issued as a document approved for public release through the document control system within 140 calendar days of the receipt of the last sample at the laboratory sample receiving/loading dock. The raw data shall be accessible to the program in accordance with the laboratory's Records Inventory and Disposition Schedule and until the respective waste tank is closed or the waste is treated.

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 7 days of release of the data package. The electronic version shall be in the standard electronic format (Bobrowski et al. 1994).

## 7.4 FORMAT VI REPORTING

The analyses performed at Pacific Northwest National Laboratory shall be administered and reported as specified in the Letter of Instruction (Funderburke 1997).

## 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 Project. All significant changes (such as DQO additions or analysis of new, additional samples) shall be documented by TWRS Process Engineering via an engineering change notice to this SAP or by a letter. All changes shall also be clearly documented in the final data report. Insignificant changes (such as changes in procedure numbers) may be made by the tank or project coordinator by placing a notation in the permanent record (i.e., note change in extrusion log book or memo to file). Significance is determined by the tank coordinator.

At the request of the Characterization Project, additional analysis of sample material from this characterization project shall be performed following a revision of this SAP or issuance of a letter.

## 9.0 REFERENCES

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Table A-1: Tank 241-SX-105 Physical Estimate Profile  
Riser 3

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	11	630.21 - 631.11	Air	limited recovery of segments 1 - 12 with rotary mode due to N <sub>2</sub> purge
	8	629.53 - 630.21	thin dry, granular crust over wet, soft saltcake	anticipate good recovery with push mode
2	19	627.95 - 629.53	wet, soft saltcake	anticipate good recovery with push mode
3	19	626.36 - 627.95	wet, soft saltcake	anticipate good recovery with push mode
4	19	624.78 - 626.36	damp, soft saltcake	anticipate good recovery with push mode; could contain hard interface
5	19	623.20 - 624.78	damp, soft saltcake	anticipate good recovery with push mode
6	19	621.61 - 623.20	damp, soft saltcake	anticipate good recovery with push mode
7	19	620.03 - 621.61	damp, soft saltcake	anticipate good recovery with push mode
8	19	618.45 - 620.03	damp, soft saltcake	anticipate good recovery with push mode
9	19	616.86 - 618.45	damp, soft saltcake	anticipate good recovery with push mode
10	19	615.28 - 616.86	damp, soft saltcake	anticipate good recovery with push mode
11	19	613.70 - 615.28	damp, soft saltcake	anticipate good recovery with push mode
12	13	612.61 - 613.70	damp, soft saltcake	anticipate good recovery with push mode
	6	612.11 - 612.61	damp, soft R1 sludge	anticipate good recovery with push mode
13	4	611.78 - 612.11	damp, soft R1 sludge	anticipate good recovery with push mode
	15	610.53 - 611.78	damp, hard sludge w/ lg chunks	limited recovery

Note: elevations based on inside tank center bottom for Tank SX-105 of 610.11 ft. MSL

Figure A-1. Tank 241-SX-105 Physical Profile Estimate  
(Riser 3)

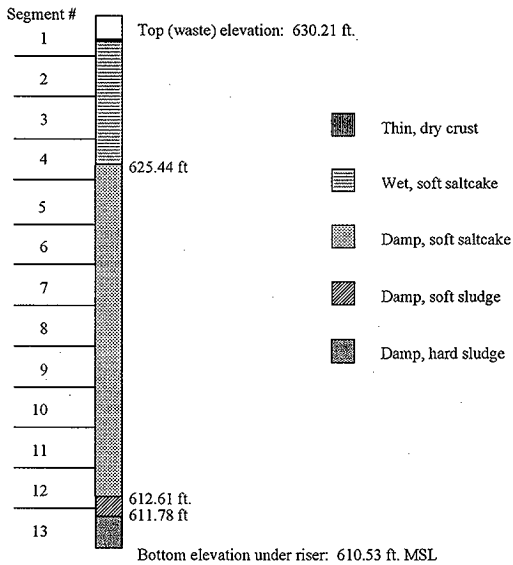


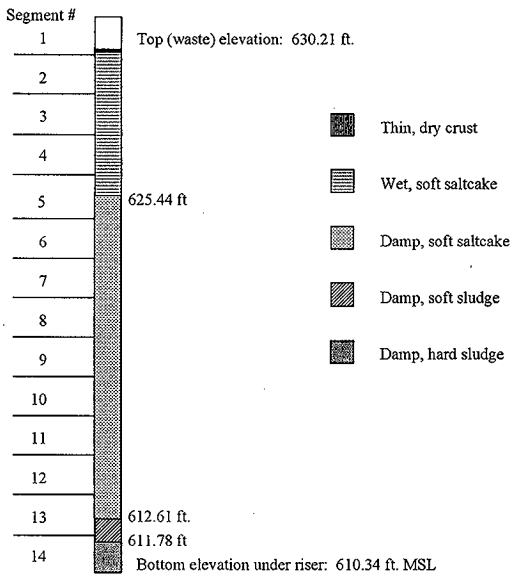


Table A-2: Tank 241-SX-105 Physical Estimate Profile  
Riser 6

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	17	631.11 - 632.51	Air	limited recovery of segments 1 -13 with rotary mode due to N <sub>2</sub> purge
	2	630.92 - 631.11	thin dry, granular crust over wet, soft saltcake	anticipate good recovery with push mode
2	19	629.34 - 630.92	wet, soft saltcake	anticipate good recovery with push mode
3	19	627.76 - 629.34	wet, soft saltcake	anticipate good recovery with push mode
4	19	626.17 - 627.76	wet, soft saltcake	anticipate good recovery with push mode
5	19	624.59 - 626.17	damp, soft saltcake	anticipate good recovery with push mode; could contain hard interface
6	19	623.01 - 624.59	damp, soft saltcake	anticipate good recovery with push mode
7	19	621.42 - 623.01	damp, soft saltcake	anticipate good recovery with push mode
8	19	619.84 - 621.42	damp, soft saltcake	anticipate good recovery with push mode
9	19	618.26 - 619.84	damp, soft saltcake	anticipate good recovery with push mode
10	19	616.67 - 618.26	damp, soft saltcake	anticipate good recovery with push mode
11	19	615.09 - 616.67	damp, soft saltcake	anticipate good recovery with push mode
12	19	613.51 - 615.09	damp, soft saltcake	anticipate good recovery with push mode
13	11	612.61 - 613.51	damp, soft saltcake	anticipate good recovery with push mode
	8	611.92 - 612.61	damp, soft R1 sludge	anticipate good recovery with push mode
14	2	611.78 - 611.92	damp, soft R1 sludge	anticipate good recovery with push mode
	17	610.34 - 611.78	damp, hard sludge w/ lg chunks	limited recovery

Note: elevations based on inside tank center bottom for Tank SX-105 of 610.11 ft. MSL

Figure A-2. Tank 241-SX-105 Physical Profile Estimate  
(Riser 6)



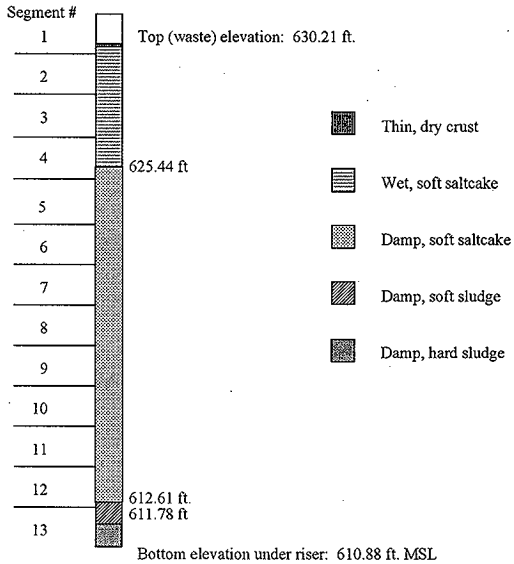
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Table A-3: Tank 241-SX-105 Physical Estimate Profile  
Riser 14

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	15	630.21 - 631.46	Air	limited recovery of segments 1 - 12 with rotary mode due to N <sub>2</sub> purge
	4	629.88 - 630.21	thin dry, granular crust over wet, soft saltcake	anticipate good recovery with push mode
2	19	628.30 - 629.88	wet, soft saltcake	anticipate good recovery with push mode
3	19	626.71 - 628.30	wet, soft saltcake	anticipate good recovery with push mode
4	19	625.13 - 626.71	damp, soft saltcake	anticipate good recovery with push mode; could contain hard interface
5	19	623.55 - 625.13	damp, soft saltcake	anticipate good recovery with push mode
6	19	621.96 - 623.55	damp, soft saltcake	anticipate good recovery with push mode
7	19	620.38 - 621.96	damp, soft saltcake	anticipate good recovery with push mode
8	19	618.80 - 620.38	damp, soft saltcake	anticipate good recovery with push mode
9	19	617.21 - 618.80	damp, soft saltcake	anticipate good recovery with push mode
10	19	615.63 - 617.21	damp, soft saltcake	anticipate good recovery with push mode
11	19	614.05 - 615.63	damp, soft saltcake	anticipate good recovery with push mode
12	17	612.61 - 614.05	damp, soft saltcake	anticipate good recovery with push mode
	2	612.46 - 612.61	damp, soft R1 sludge	anticipate good recovery with push mode
13	9	611.78 - 612.46	damp, soft R1 sludge	anticipate good recovery with push mode
	10	610.88 - 611.78	damp, hard sludge w/ lg chunks	limited recovery

Note: elevations based on inside tank center bottom for Tank SX-105 of 610.11 ft. MSL

Figure A-3. Tank 241-SX-105 Physical Profile Estimate  
(Riser 14)



# DISTRIBUTION SHEET

To	From	Page 1 of 1
Distribution	Technical Basis and Planning	Date 01/19/98
Project Title/Work Order		EDT No. EDT-622420
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