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Tank 241-TX-113 Rotary Mode Core Sampling and Analysis Plan

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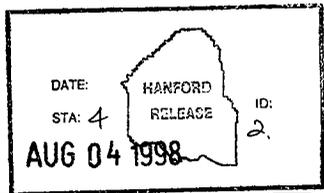
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HNF-3039
Revision 0

Tank 241-TX-113

Rotary Mode Core

Sampling and Analysis Plan

D. J. McCain
Lockheed Martin Hanford Corporation

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
GEA	gamma energy analysis
g	gram
g/L	gram per liter
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
MSL	mean sea level
PIC	person in charge
PNNL	Pacific Northwest National Laboratory
PRSSST	Propagating Reactive System Screening Tool
QA	quality assurance
QC	quality control
RSST	Reactive System Screening Tool
SAP	sampling and analysis plan
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
TWRS	Tank Waste Remediation System
TX-113	241-TX-113
WMH	Waste Management Hanford
μ Ci	microcurie
μ Ci/g	microcurie per gram
μ g C/g	micrograms of carbon per gram
μ g/g	micrograms per gram
μ g/mL	micrograms per milliliter

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 push mode core samples from tank 241-TX-113 (TX-113). It is written in accordance with *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), *Data Quality Objective to Support Resolution of the Flammable Gas Safety Issue* (Bauer 1998) and the *Historical Model Evaluation Data Requirements* (Simpson and McCain 1997). The *Tank Characterization Technical Sampling Basis* document (Brown et al. 1998) also identifies Retrieval, Pretreatment and Immobilization as an issue that applies to tank TX-113. As a result, a 150 gram composite of solids shall be made and archived for that program.

2.0 SAMPLING EVENT REQUIREMENTS

As of April 30, 1998, tank TX-113 contained a total waste volume of approximately 2,300 kL (607 kgal), consisting entirely of saltcake with 60.6 kL (16 kgal) of drainable liquid (Hanlon 1998). This waste volume is equivalent to 5.80 meters (228 inches) of waste as measured from the centerline of the tank. A physical profile prediction based on waste fill history and previous sampling information is provided in Appendix A. Historical data suggests Al-clad sludge is part of the tank waste (Place 1997).

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 1998). If the dome space results are above 10 percent of the LFL when analyzing with a combustible gas meter or above 25 percent of the LFL when analyzing by gas chromatography/mass spectrometry or gas-specific monitoring, the necessity for recurring sampling for the flammable gas concentration and the frequency of such sampling will be determined by the Flammable Gas Safety Project.

During core sampling, the drill string will be monitored for flammable gas. If the monitoring instrument in the drill string indicates a level greater than 10% of the LFL during intrusive activities, then a vapor grab sample shall be taken and sent to Pacific Northwest National Laboratory (PNNL) for analysis (Bauer 1998). Any additional vapor sampling is not within the scope of this SAP.

Tank TX-113 will be rotary mode core sampled using a rotary mode core sampling system. Two core samples, each consisting of 13 segments, are expected to be taken from risers 3 and 5. 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 in comparison to presampling zip cord readings.

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 sampling work packages WS-98-00072 (riser 3) and WS-98-00073 (riser 5). These work packages contain the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank TX-113 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 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), Bauer (1998), and Simpson and McCain (1997), 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.
- Form stratum composite samples and core composite samples from each core per tank coordinator direction.
- Analyze drainable liquids, homogenized half-segment subsamples, stratum composite, and core composite samples from each core as shown in Tables 1 and 2.
- Form a 150 gram solid composite sample from one (or both) cores and archive this material for the Retrieval, Pretreatment, and Immobilization Program.
- Archive all remaining material.

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.

For core samples from tank TX-113, the shipping container must be vented every 27 days to release any accumulated gas.

Opportunistic analyses as defined in Kristofzski (1996) are to be included 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 TX-113 core samples are based on the safety screening data quality objective (DQO), the flammable gas DQO, and the historical model DQO referenced in Section 1.0. The laboratory procedure numbers which shall 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.

The historical model DQO requires phased analysis periods, and selection of optimal segments for full suite analysis. The laboratory is to notify the tank coordinator once primary analyses are complete for each segment.

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 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 the SAP, but not performed, shall be identified in the appropriate data report with justification provided for nonperformance.

Table 1: Tank TX-113 Chemical, Radiological, and Physical Analytical Requirements: Solids (2 Sheets)

Project Name		SOLID ANALYSES		COMMENTS		REPORTING LEVELS									
		TX-113 Rotary Mode Core Sample	PROCEDURE	Homogenization Test - Per Laboratory Discretion	Field Blank - Required	FORMAT I	Immediate Notification								
Plan Number	PROGRAM	HNF-3039, Rev. 0	LA-514-113	Energy	LA-514-113	LA-514-113	FORMAT II								
A. Safety Screening	PROGRAM CONTACTS	R. J. Cash	LA-514-114	% H ₂ O	LA-514-114	LA-514-114	FORMAT III								
B. Historical Data	TWRS Safety	B. C. Simpson	LA-514-115	Blank density	LA-514-115	LA-514-115	FORMAT IV								
C. Process Control	TWRS Proc. Eng.	D. J. MacCam	LA-508-101	Total Alpha	LA-508-101	LA-508-101	FORMAT V								
D. Flammable Gas	TWRS Safety	R. E. Bauer	LA-220-101	%Sr	LA-220-101	LA-220-101	FORMAT V								
			LA-548-121	%Cs ¹³⁷	LA-548-121	LA-548-121	FORMAT VI								
			LA-342-100	TIC	LA-342-100	LA-342-100	Special								
		PRIMARY ANALYSES		SAMPLE ¹		QUALITY CONTROL ³		CRITERIA		FORMAT					
PROGRAM	METHOD	ANALYSIS	PROCEDURE	1/2 SBG SLDG/SC	STRAT /COMP	COMP	PREP	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁵	FORMAT
A, B	DSC		LA-514-113	x	x	x	d	ca smpl	N/A	N/A	ca AB	J/g	> 400 ^{5,6}	unknown	I, III, IV
A, B, D	TGA		LA-514-114	x	x	x	d	ca smpl	N/A	N/A	ca AB	wt%	none	63.2	III, IV
A, B, D	Gravimetry		LA-514-115	x ¹⁵	x	x	d	N/A	N/A	N/A	N/A	g/mL	none	1.41	III, IV
A, B	Alpha counting		LA-508-101	x ¹⁶	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µCi/g	> 41 ⁶	unknown	I, III, IV
B, D	Beta counting		LA-220-101	x ¹⁷	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µCi/g	none	81	IV
B, D	GEA		LA-548-121	x	x	x	f	ca smpl	N/A	ca AB	ca AB	µCi/g	none	189	IV
D	Acidification/ Peroxalate oxidation		LA-342-100	x	x	x	d	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg Ci/g	> 45,000 ^{5,6}	unknown	I, IV
A, C	ICP/AES	Li, Al, Cr, Fe, Mn, Na, Ni, Si, U, ¹¹	LA-505-151 LA-505-161	x	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	varied	III, IV
B	ICP/AES	Na, Al, Cr ¹⁶	LA-505-151 LA-505-161	x	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	varied	IV
D	ICP/AES	Ca, Fe, K, Ni, Al, Ba, Si, B, Bi, Na, Zn, Zn ¹⁷	LA-505-151 LA-505-161	x	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	varied	IV
B	ICP/AES	Na, Al, Cr ¹⁹	LA-505-151 LA-505-161	x ²	x	x	w	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	varied	IV
C	IC	B ¹⁰	LA-533-105	x	x	x	w	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/mL	none	varied	IV
B	IC	NO ₃ , SO ₄ ¹⁸	LA-533-105	x	x	x	w	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/mL	none	varied	IV
D	IC	formate, oxalate, NO ₃ , PO ₄ , Cl ¹⁹	LA-533-105	x	x	x	w	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/mL	none	varied	IV
B, D	Percalate	TOC	LA-542-100	x ²	x	x	d	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg Ci/g	> 45,000 ^{5,6}	15,900	I, IV
B	Beta counting	Total Beta	LA-508-101	x ²	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µCi/g	none	unknown	IV
B	Kinetic phosphorescence	Uranium	LA-925-009	x ²	x	x	f or a	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	1,180	IV
D	Titration	OH	LA-211-102	x	x	x	w	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	125,000	IV
D	Spectrophotometry	Cr (VI)	LA-265-101	x	x	x	d	ca smpl	1/mtr ²⁰	ca AB	ca AB	µg/g	none	2,570	IV
D	Gravimetry	Settling behavior	see ²	x	x	x	w	ca smpl	N/A	N/A	N/A	cm/hr	none	unknown	IV
D	Centrifuge	Vol% solids	LA-510-152	x	x	x	d	N/A	N/A	N/A	N/A	vol%	none	unknown	IV

Table 1: Tank TX-113 Chemical, Radiological, and Physical Analytical Requirements: Solids (2 Sheets)

PROGRAM	SECONDARY ANALYSES			SAMPLE ¹			QUALITY CONTROL ²				CRITERIA		FORMAT	
	METHOD	ANALYSIS	PROCEDURE	1/2 SEG SLD/SC	STRAT /COMP	COMP	PREP ³	DUP	SPIKE	BLK	STD	UNITS		NOTIFICATION LIMIT ⁴
A	Alpha counting	²³⁸ Pu ¹	LA-935-104	X			f	ca. smpl	1/mtr ²	ca. PB	ca. AB	µCi/g	none	0.064
A	FRSST	propagating behavior ⁶	L7-510-105	X			d	N/A	N/A	N/A	N/A	J/g	none	unknown
A	Elemental Oxidation	TOC ¹⁴	LA-344-105	X ¹⁵			w	ca. smpl	1/mtr ²	ca. AB	ca. AB	µg C/g	> 45,000 ¹⁶	15,900

¹1/2 SEG SLD/SC = 1/2 segment, sludge or slitsaker; STRAT/COMP = stratum subsample or composite COMP = Composite for Historical DQO

²d = direct, f = fusion, a = acid, w = water

³DUP = duplicate, BLK = blank, STD = calibration standard, ca = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtr = matrix, N/A = not applicable

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

⁵Dry weight basis

⁶These 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).

⁷Tracer or carrier may be used in place of a spike and results corrected for recovery.

⁸Results should be reported for all GEA analytes, however the QC requirements of Table 3 apply to ¹³⁷Cs.

⁹Br is required to correct ZHGO measurement (to be performed by Process Engineering).

¹⁰Li is required to correct ZHGO measurement (to be performed by Process Engineering). Al, Cl, Fe, Mn, Na, Ni, Si, and U are required as secondary safety screening analytes,

to be performed if total alpha activity exceeds notification limit.

¹¹No procedure is available for the settling rate test. Work will be performed to an approved test plan, which will be referenced in the data package.

¹²Analysis to be performed if total alpha activity limit is exceeded.

¹³Analysis to be performed only on the subsegment with the highest total (other than historical DQO optimum segment) if the sample's DSC result exceeds 480 J/g.

¹⁴TOC/FRSST is required only on the subsegment with the highest total (other than historical DQO optimum segment) if the half-segment selected for additional historical DQO analyses is an upper half-segment. Dry should be done on this sample as well.

¹⁵Na, Al, Cr are fingerprint analytes for historical. Results should be reported for all ICP/AES analytes; however, the QC requirements of Table 3 apply only to those identified here.

¹⁶Ca, Fe, K, Ni, Zn, Al, Ba, Si, Bi, Na, Zn, and Zr are from the flammable gas DQO, and are required only on the stratum samples. Results should be reported for all ICP/AES analytes; however, the QC requirements of Table 3 apply only to those identified here.

¹⁷Nitrate and Sulfate are fingerprint analytes for historical DQO. Results should be reported for all IC analytes. However, the QC requirements of Table 3 only apply to those identified in this table.

¹⁸Formate, oxalate, nitrite, phosphate, chloride, and fluoride are required only on the flammable gas stratum samples. Results should be reported for all IC analytes.

¹⁹However, the QC requirements of Table 3 only apply to those identified in this table.

²⁰Either serial dilutions or matrix spikes will be performed.

²¹Calibration of heater resistance, time, temperature, pressure, containment volume, and sample weight will be performed to measure accuracy as described in procedure L7-510-103.

Table 2: Tank TX-113 Chemical, Radiological, and Physical Analytical Requirements: Liquids (2 Sheets)

Project Name Plan Number		LIQUID ANALYSES				COMMENTS				REPORTING LEVELS				
PROGRAM		PROGRAM CONTACTS				Homogenization Test - Per Laboratory Discretion				FORMAT I				
A. Safety Screening		R. J. Cash				Field Blank - Required				Immediate Notification				
B. Process Control		D. J. McCain				Hot Cell Blank - Per Laboratory Discretion				FORMAT II				
C. Flammable Gas		R. E. Bauer				LBr Solution Blank - Required if HHF is used				Safety Screening				
		TANK				#CORES				FORMAT III				
		TX-113				2				FORMAT IV				
										FORMAT V				
										RCRA Compliance				
										FORMAT VI				
										Special				
PROGRAM	METHOD	PRIMARY ANALYSES				QUALITY CONTROL ²				CRITERIA				
		ANALYSIS	PROCEDURE	SAMP ¹ FB & SEG LJO	STRAT	PREP ³	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁵	FORMAT
A	DSC	Energy	LA-514-113 LA-514-114	x		d	ea snpl	N/A	N/A	ca AB	1/g	≥ 480 ^{5,6}	unknown	I, III, IV
A,C	TGA	% H ₂ O	LA-514-114	x	x	d	ea snpl	N/A	N/A	ca AB	wt%	none	45.9	III, IV
A	Alpha counting	Total Alpha	LA-508-101	x		d	ea snpl	1/mtrx	ca PB	ca AB	µCi/mL	> 61.5 ⁶	unknown	I, III, IV
A, C	Gravimetry	Specific Gravity	LA-510-112	x	x	d	ea snpl	N/A	N/A	ca AB	none	none	1.43	IV
A, B, C	ICP/AES	Li, Al, Ca, Fe, Mn, Na, Ni, Si, U, Cs, K, Zn, Zr, Ba, B, Bi ⁷	LA-505-151 LA-505-161	x	x	d ⁸	ea snpl	1/mtrx ⁹	ca PB	ca AB	µg/mL	none	varied	III, IV
C	Ion selective electrode	Ammonia ¹⁷	LA-631-001	x		d ⁸	ea snpl	1/tank	ca AB	ca AB	µg/mL ¹²	none	968	IV
B, C	IC	Br, formate, oxalate, NO ₃ , NO ₂ , PO ₄ , SO ₄ , Cl ₂ and F ¹¹	LA-533-105	x	x	d ⁸	ea snpl	1/mtrx	ca AB	ca AB	µg/mL	none	varied	IV
C	Acidification/ Persulfate	TIC/TOC	LA-342-100	x		d	ea snpl	1/mtrx	ca AB	ca AB	µg Ci/mL	TOC > 45,000 ^{5,6,16}	unknown	I, IV
A	Visual	Organic	LA-519-151	x		d	N/A	N/A	N/A	N/A	N/A	presence	none	I, III, IV
C	Thiation	OH	LA-211-102	x	x	d	ea snpl	1/mtrx	ca AB	ca AB	µg/mL	none	75,500	IV
C	Spectrophotometry	Cr (VI)	LA-265-101	x	d	d	ea snpl	1/mtrx	ca AB	ca AB	µg/mL	none	5,050	IV
C	GEA	¹³⁷ Cs, ¹³⁹ Cs, ⁸⁸ Sr	LA-548-121	x	d	d	ea snpl	1/mtrx ¹⁴	ca AB	ca AB	µCi/mL	none	152	IV
C	Beta counting gravimetric	Setting behavior isotols fraction	LA-220-101 seg ¹⁵	x	x	d	ea snpl	1/mtrx ¹⁴	ca AB	ca AB	µCi/mL	none	53.8	IV
C	centrifuge		LA-519-132	x		d	N/A	N/A	N/A	N/A	cm/hr	none	unknown	IV
PROGRAM	METHOD	SECONDARY ANALYSES				QUALITY CONTROL ²				CRITERIA				
		ANALYSIS	PROCEDURE	SEG LIQ	STRAT	PREP ³	DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁵	FORMAT
A	Alpha counting	²³² Th/ ²³² Pu ¹³	LA-933-104	x		d	ea snpl	1/mtrx ¹⁴	ca AB	ca AB	µCi/mL	none	0.02	III, IV
A	PRSSST	propagating behavior ¹⁸	LT-510-103	x		d	N/A	N/A	N/A	seg ²	1/g	none	unknown	IV
A	Purnee Oxidation	TOC ¹⁶	LA-344-103	x		d	ea snpl	1/mtrx	ca AB	ca AB	µg Ci/mL	> 45,000 ^{5,6,16}	unknown	I, III, IV

Table 2: Tank TX-113 Chemical, Radiological, and Physical Analytical Requirements: Liquids (2 Sheets)

¹PB = field blank, SEGLIQ = segment-level drainable liquid, STRAT = stratum or composite liquid

²d = direct

³DUJP = duplicate, BLK = blank, STD = calibration standard, ea = each, empl = sample, AB = analytical batch, PB = preparation blank, mtrx = matrix, N/A = not applicable

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

⁵Dry weight basis

⁶These 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).

⁷Li is required to correct %H₂O measurement (to be performed by Process Engineering). Al, Cs, Fe, Mn, Na, Ni, Si, and U are required as secondary safety screening analytes; required if total alpha exceeds notification limits. The remainder are for the flammable gas DQO. Results should be reported for all ICP/AES analytes; however, the QC requirements of Table 3 apply only to those identified here.

⁸Direct liquid samples may be diluted in acid or water to adjust to proper sample size and/or pH.

⁹Other serial dilutions or matrix spikes will be performed.

¹⁰TOC by the furnace oxidation method is performed on a sample if the sample's DSC result exceeds 480 J/g.

¹¹Br is required to correct %H₂O measurement (to be performed by Process Engineering). The remainder are required for the flammable gas DQO.

¹²Results should be reported for all IC analytes; however, the QC requirements of Table 3 apply only to those listed.

¹³Micrograms ammonia per milliliter of as-received sample.

¹⁴Performed if total alpha exceeds notification limit.

¹⁵Tracer or carrier may be used in place of a spike and results corrected for recovery.

¹⁶No procedure is available for the settling rate test. Work will be performed to an approved test plan, which will be referenced in the data package.

¹⁷Corrected from weight basis to volumetric basis assuming a liquid density of 1.0 g/mL.

¹⁸Ammonia analysis should be performed as rapidly as possible following subsampling to minimize volatilization.

¹⁹Calibration of heater resistance, time, temperature, pressure, containment volume, and sample weight will be performed to measure accuracy as described in procedure LT-510-103.

²⁰Results should be reported for all GEA analytes; however, the QC requirements of Table 3 apply only to ¹⁷Cs.

²¹No procedure is available for the settling rate test. Work will be performed to an approved test plan, which will be referenced in the data package.

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, controlled, reported, and dispositioned as required by the *Nonconformance Item Reporting and Control* (PHMC 1997).

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 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 (retained gas sampler or universal 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, LiBr solution used, or detecting debris) should be noted 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) ¹	Spike Criteria (% recovery) ²	Preparation Blank Criteria ³	LCS Criteria (% recovery) ⁴
Solids				
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 Uranium	≤ 20	75 - 125	< EQL	80 - 120
Total alpha	≤ 20	75 - 125	< MDA	70 - 130
Total beta	≤ 20	75 - 125	< MDA	80 - 120
ICP/AES	≤ 20	75 - 125	< EQL	80 - 120
PRSSST ⁶	N/A	N/A	N/A	N/A
IC	≤ 20	75 - 125	< EQL	80 - 120
TOC/TIC	≤ 20	75 - 125	< EQL	80 - 120
⁹⁰ Sr	≤ 20	75 - 125 ⁵	< MDA	80 - 120
GEA	≤ 20	N/A	< MDA	80 - 120
^{239/240} Pu	≤ 20	75 - 125 ⁵	< MDA	70 - 130
Cr (VI)	≤ 20	75 - 125	< EQL	80 - 120
OH	≤ 10	75 - 125	< EQL	90 - 110
Settling Rate	≤ 10	N/A	N/A	N/A
Solids Fraction	N/A	N/A	N/A	N/A

Analysis/Method	Duplicate Criteria (RPD) ¹	Spike Criteria (% recovery) ²	Preparation Blank Criteria ³	LCS Criteria (% recovery) ⁴
Liquids				
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
PRSS ⁶	N/A	N/A	N/A	N/A
IC	≤ 20	75 - 125	< EQL	80 - 120
Ammonia	≤ 10	75 - 125	< EQL	90 - 110
TIC/TOC	≤ 20	75 - 125	< EQL	80 - 120
OH	≤ 10	75 - 125	< EQL	90 - 110
Cr (VI)	≤ 20	75 - 125	< EQL	80 - 120
GEA	≤ 20	N/A	< MDA	80 - 120
^{239/240} Pu	≤ 20	75 - 125 ⁵	< MDA	70 - 130
⁹⁰ Sr	≤ 20	75 - 125 ⁵	< MDA	80 - 120
Settling Rate	≤ 10	N/A	N/A	N/A
Solids Fraction	N/A	N/A	N/A	N/A

Notes:

N/A = not applicable

MDA = minimum detectable activity

EQL = estimated quantitation limit

LCS = laboratory control standard

¹For 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.

²The criteria are recommended. Failures are permissible if the requirements in the QA section are followed.

³When 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.

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

⁵A tracer or carrier may be substituted for the spike.

⁶See Section 5.1.

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 Propagating Reactive System Screening Tool (PRSST) analysis is now preferred over the RSST testing. As such, the RSST requirement in Tables 1 and 2 has been removed replaced by a PRSST requirement.

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 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 dry weight basis value may result when the wt% water value is greater than 90 percent water, 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 Pu-239. The notification limit may then be calculated as shown in equation (2):

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

NOTE: 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 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.

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

Where $\hat{\mu}$ is the sample mean, σ^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), gamma energy analysis (GEA), 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 or additional analyses should be performed to improve recovery for analytes not specifically requested in Tables 1 or 2.

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 TX-113 characterization project are listed in Table 4.

Table 4: Tank TX-113 Project Key Personnel List

Responsibility	Organization	Individual
TX-113 Tank Coordinator	TWRS Process Engineering (LMHC)	D. J. McCain, 373-1023
222-S Laboratory Point of Contact (day shift)	Analytical Services (WMH)	D. B. Hardy, 376-4878
222-S Laboratory Point of Contact (off-hours)	Analytical Services (WMH)	222-S Laboratory Shift Manager, 373-2435
Single-Shell Tank Farm Point of Contact	Tank Farm Operations	Single-Shell 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 (1998).

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 Faculty Planning Team to the Single-Shell 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 Tank Waste Remediation System (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). The data package shall be issued 180 days after the last sample is received at the laboratory. 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).

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 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.

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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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APPENDIX A:

EXPECTED PHYSICAL PROFILE OF TANK TX-113 CORE SAMPLES

Table A-1: Tank 241-TX-113 Physical Profile Estimate
Riser 3

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	17	644.20 - 645.60		metal tapes under riser
	2	644.02 - 644.20	dry, soft saltcake	limited to good recovery with push mode, limited recovery with rotary mode due to N2 purge
2	19	642.44 - 644.02	dry, soft saltcake	limited to good recovery with push mode, limited recovery with rotary mode due to N2 purge
3	19	640.85 - 642.44	dry, hard saltcake	limited recovery
4	19	639.27 - 640.85	dry, hard saltcake	limited recovery
5	19	637.69 - 639.27	dry, hard saltcake	limited recovery
6	19	636.10 - 637.69	dry, hard saltcake	limited recovery
7	19	634.52 - 636.10	dry, hard saltcake	limited recovery
8	19	632.94 - 634.52	dry, hard saltcake	limited recovery
9	19	631.35 - 632.94	dry, hard saltcake	limited recovery
10	19	629.77 - 631.35	damp, soft sludge/saltcake mix	limited to good recovery, saltcake will look like sludge/saltcake
11	0.5	629.73 - 629.77	damp, soft sludge/saltcake mix	limited to good recovery, saltcake will look like sludge/saltcake
	18.5	628.19 - 629.73	damp, soft sludge	good recovery expected
12	19	626.60 - 628.19	damp, soft sludge	good recovery expected
13	19	625.02 - 626.60	damp, soft sludge	good recovery expected

Note: elevations based on inside tank center bottom for Tank 241-TX-113 of 624.73 ft. MSL

Figure A-1: Tank 241-TX-113 Physical Profile Estimate
(Riser 3)

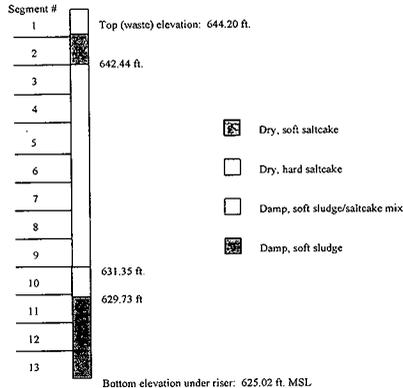
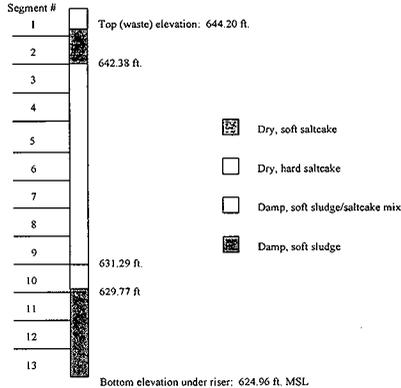


Table A-2: Tank 241-TX-113 Physical Profile Estimate
Riser 5

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	16	644.20 - 645.54		metal tapes under riser
	3	643.96 - 644.20	dry, soft saltcake	limited to good recovery with push mode, limited recovery with rotary mode due to N2 purge
2	19	642.38 - 643.96	dry, soft saltcake	limited to good recovery with push mode, limited recovery with rotary mode due to N2 purge
3	19	640.79 - 642.38	dry, hard saltcake	limited recovery
4	19	639.21 - 640.79	dry, hard saltcake	limited recovery
5	19	637.63 - 639.21	dry, hard saltcake	limited recovery
6	19	636.04 - 637.63	dry, hard saltcake	limited recovery
7	19	634.46 - 636.04	dry, hard saltcake	limited recovery
8	19	632.88 - 634.46	dry, hard saltcake	limited recovery
9	19	631.29 - 632.88	dry, hard saltcake	limited recovery
10	18	629.77 - 631.29	damp, soft sludge/saltcake mix	limited to good recovery, saltcake will look like sludge/saltcake
	1	629.71 - 629.77	damp, soft sludge/saltcake mix	limited to good recovery, saltcake will look like sludge/saltcake
11	19	628.13 - 629.71	damp, soft sludge	good recovery expected
12	19	626.54 - 628.13	damp, soft sludge	good recovery expected
13	19	624.96 - 626.54	damp, soft sludge	good recovery expected

Note: elevations based on inside tank center bottom for Tank 241-TX-113 of 624.73 ft. MSL

Figure A-2: Tank 241-TX-113 Physical Profile Estimate
(Riser 5)



DISTRIBUTION SHEET

To	From	Page 1 of 1
Distribution	Technical Basis and Planning	Date 07/30/98
Project Title/Work Order		EDT No. EDT-622460
HNF-3039, Rev. 0, "Tank 241-TX-113 Rotary Mode Core Sampling and Analysis Plan"		ECN No. N/A

Name	MSIN	Text With All Attach.	Text Only	Attach./Appendix Only	EDT/ECN Only
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Central Files	B1-07	X			
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Los Alamos Technical Associates

R. W. Lambie	S7-04	X			
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Numatec Hanford Corporation

W. I. Winters	T6-50	X			
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Waste Management Federal Services of Hanford, Inc.

R. Akita	T6-20	X			
R. A. Esch	T6-12	X			
R. K. Fuller	T6-50	X			
D. B. Hardy	T6-12	X			
K. L. Powell	T6-12	X			
F. H. Steen	T6-12	X			