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		Design Agent									
2	1	Cog.Eng.	J.H. Baldwin	<i>J.H. Baldwin</i>	1/16/97	1	1	E.Q. Le	<i>Elvick</i>	1-29-97	
2	1	Cog. Mgr.	J.W. Hunt	<i>J.W. Hunt</i>	1/16/97	1	1	W.J. Kennedy	<i>W.J. Kennedy</i>	1/16/97	
2	1	QA	T.C. Tribble	<i>T.C. Tribble</i>	1/21/97	1	1	A.D. Rice	<i>A.D. Rice</i>	2-4-97	
		Safety									
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Tank 241-AP-108 Grab Sampling and Analysis Plan

John H. Baldwin

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Release Approval

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Tank 241-AP-108 Grab Sampling and Analysis Plan

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

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LIST OF ABBREVIATIONS

AP-108	Tank 241-AP-108
DOE	Department of Energy
DQO	data quality objective
DSC	differential scanning calorimetry
GC/MS	gas chromatography/mass spectroscopy
GEA	gamma energy analysis
HPGE/MCA	high purity germanium - multi channel analysis
IC	ion chromatography
ICP	inductively coupled plasma
MS	matrix spike
MSD	matrix spike duplicate
OSD	Operation safety documentation
QA	quality assurance
RSST	reactive system screening tool - adiabatic calorimetry
SARP	Sampling and Analysis Report for Packaging
SAP	Sampling and Analysis Plan
SpG	Specific gravity
SVOA	semivolatile
TC	tank coordinator
TGA	thermogravimetric analysis
TC	total carbon
TIC	total inorganic carbon
TOC	total organic carbon
VOA	volatile
WHC	Westinghouse Hanford Company

1.0 SAMPLING AND ANALYSIS OBJECTIVES

This Sampling and Analysis Plan (SAP) will identify characterization objectives for sample collection, laboratory analytical evaluation, and reporting requirements for tank 241-AP-108 (AP-108). It is written in accordance with the *Tank 241-AP-108 Tank Characterization Plan* (Homi 1996), the *Tank Safety Screening Data Quality Objective* (Dukelow, et al. 1995), the *242-A Evaporator/Liquid Effluent Retention Facility Data Quality Objectives* (Von Bargaen 1995), and the *Data Quality Objectives for Tank Farms Waste Compatibility Program* (Fowler 1995).

2.0 SAMPLING EVENT REQUIREMENTS

Tank AP-108 will be sampled following a supernatant waste transfer of approximately 1,363 kl (360 kgal) from tank 241-AN-106 and 2,725 kl (720 kgal) from tank 241-AY-101. This transfer will bring the total waste volume in AP-108 to approximately 4266 kl (1,127 kgal). This waste transfer will support the Evaporator Campaign 97-2. In addition to a surface sample, a total of three subsurface sample sets will be collected as outlined in Campbell (1996). Specific sample identification numbers, analysis type, sample/bottle type, sample location, and sample elevation are described in Table 1. A process memo and the sampling work package will initiate and direct the sampling event.

Prior to 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 the dome space and the data reported as a percentage of the Lower Flammability Limit (LFL). The results shall be submitted to the Characterization tank coordinator (TC) within one week of the sampling event. If the limits are above 25% of the LFL when analyzing by gas chromatography/mass spectrometer (GC/MS) or gas-specific monitoring gauges, or above 10% 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 Program. It is however specified in Dukelow (1995) that, if combustible gas meter results are over 10% of the LFL, then a type 3 or type 4 vapor sample must be taken and analyzed to address the flammability issue (Osborne 1995). Operation, however, shall adhere to stricter requirements if specified in the applicable operation safety documentation (OSD).

Tank AP-108 is currently scheduled to be grab sampled in order to prepare for processing through the 242-A Evaporator. One surface grab sample will be taken from tank AP-108, consisting of one 125-mL bottle. Grab samples from three subsurface locations will be taken as well. At each subsurface sampling location, four 125-mL bottles will be drawn: one for boildown and mixing study analyses, two for organic

analyses (one for semivolatiles [SVOA], and one for volatiles [VOA]), and one for inorganic and radionuclide analyses.

The sample bottles for inorganic/radiological analyses, organic (SVOA), TOC (surface) and the mixing/boildown studies must be 125-mL, precleaned, amber colored bottles with a certification of EPA level 1 procedure A, sealed with a Teflon¹ cap. Sample bottles used for organic (VOA) analyses must be 125-mL, precleaned, amber colored bottles with a certification of EPA level 1 procedure B, sealed with a septum cap and lined septum.

Grab sampling field activities will be performed per the applicable Process Memo and procedure TO-080-403, "Supernatant or Sludge Sampling of All Watchlist and Non-Watchlist Tanks Located in Tank Farm Facilities, Excluding SV-Farm".

With respect to sampling quality control, one field blank consisting of four 125-mL bottles shall be taken during the sampling event: one 125-mL bottle for SVOA organics, one 125-mL bottle for VOA organics, and two 125-mL bottles for inorganic/radiological analyses. Two trip blanks (125-mL each) shall be collected during the sampling event (one for SVOA organics, one for VOA organics) and shall only be analyzed for those constituents that are detected in the field blank. Field and trip blank bottles used for organic (VOA) analyses must be 125-mL, precleaned, amber colored bottles with a certification of EPA level 1 procedure B, sealed with a septum cap and lined septum. The field blank bottle used for organic (SVOA) and inorganic/radiological analyses must be a 125-mL, precleaned, amber colored bottle with a certification of EPA level 1 procedure A, sealed with a Teflon cap (Von Barga 1995). After completion of organic analyses, the remaining liquid in the field blank samples can be used for inorganic/radiological analyses. It is expected that one set of blanks will be collected during this sampling event; however, if sampling duration exceeds 4 days, the Evaporator program should consider collecting additional blanks if sampling conditions change (Basra 1995).

Table 1 summarizes the sample number for each bottle, analysis to be performed on the waste, bottle type, sample location, and depths for sampling operations.

¹Teflon is a registered trademark of E.I. DuPont de Nemours

Table 1. Tank AP-108 Grab Sampling Depths

SAMPLE NUMBER	ANALYSIS TO BE PERFORMED ON WASTE ¹	SAMPLE BOTTLE TYPE ²	SAMPLE LOCATION	SAMPLE ELEV. ³ (inch)	Cable ⁴ Length
8AP-96-1B1	Inorg/Rad/SS	Field Blank, T	Either Riser	N/A	180"
8AP-96-1B2	Inorg/Rad/SS	Field Blank, T	Either Riser	N/A	180"
8AP-96-0B1	Organic/VOA	Field Blank, S	Either Riser	N/A	180"
8AP-96-0B2	Organic/SVOA	Field Blank, T	Either Riser	N/A	180"
8AP-96-1	Surface TOC/SS	Surface, T	Riser 1, 270°	≈410 ⁵	239"
8AP-96-2A	Organic/VOA	Supernate, S	Riser 1, 270°	276	373"
8AP-96-2B	Organic/SVOA	Supernate, T	Riser 1, 270°	276	373"
8AP-96-2C	Inorg/Rad/SS	Supernate, T	Riser 1, 270°	276	373"
8AP-96-2D	Mixing/Boildown	Supernate, T	Riser 1, 270°	276	373"
8AP-96-3A	Organic/VOA	Supernate, S	Riser 1, 30°	308	341"
8AP-96-3B	Organic/SVOA	Supernate, T	Riser 1, 30°	308	341"
8AP-96-3C	Inorg/Rad/SS	Supernate, T	Riser 1, 30°	308	341"
8AP-96-3D	Mixing/Boildown	Supernate, T	Riser 1, 30°	308	341"
8AP-96-4A	Organic/VOA	Supernate, S	Riser 1, 30°	103	546"
8AP-96-4B	Organic/SVOA	Supernate, T	Riser 1, 30°	103	546"
8AP-96-4C	Inorg/Rad/SS	Supernate, S	Riser 1, 30°	103	546"
8AP-96-4D	Mixing/Boildown	Supernate, T	Riser 1, 30°	103	546"
8AP-96-TB1	Organic/VOA	Trip Blank, S	N/A	N/A	N/A
8AP-96-TB2	Organic/SVOA	Trip Blank, T	N/A	N/A	N/A

¹Analysis type: Inorg = inorganic, Rad = radiological, SS = safety screening

²Bottle type: T = Teflon Cap, S = Septum Cap (with lined septum)

³Sample elevation is defined as the distance from the bottom of the tank to the mouth of the sample bottle. If a sample cannot be obtained at the depth specified, the sampling assembly should be raised or lowered as necessary and the actual depth recorded in the process memo or J5 in the work package.

⁴The cable length is measured from top of riser to mouth of the sample bottle.

⁵Due to the potential elevation discrepancy for the surface of TK-108-AP, after the 106-AN to 108-AP and 101-AY to 108-AP transfers, operators are to obtain zipcord or local measuring device readings to determine the exact depth of the surface liquid level. Add one inch to this depth and record this number to the table in the process memo or the J5 in the process memo. This number is the cable length for sample.

3.0 LABORATORY ANALYSIS REQUIREMENTS

3.1 ANALYSIS SCHEME

In order to comply with the safety screening, evaporator, and compatibility DQOs, the following steps shall be performed on each sample:

- ▶ For all samples, report any visually observed immiscible (potentially organic) layer by Format 1 reporting, and retain the layer in a separate jar.
- ▶ Perform TOC and safety screening analyses (designated by the letter A on Table 2) on the surface sample.
- ▶ The samples collected at each sampling point should be analyzed as described below:
 - Boildown and mixing study sample: Archive the 125-ml bottle and wait for further instruction pending a letter of instruction from the Evaporator Program (see Section 5.2).
 - Semivolatile analysis sample: Perform SVOA analyses as listed in Table 2.
 - Volatile analysis sample: Perform VOA analyses as listed in Table 2.
 - Inorganic/radiological/safety screening analysis sample: Perform inorganic, radiological, and safety screening (designated by the letter A) analyses as listed in Table 2.
- ▶ Retain 40 mL from each bottle for liquid archive.

As a precautionary measure, the Safety and Analysis Report for Packaging (SARP) has been reviewed for any safety issues involved with transportation of grab samples. **For grab samples, the shipping containers must be vented every four days to release retained gas.** However, Sampling Operations should transport the shipping containers within three days. Since the shipping containers are opened at the time the samples are received at the laboratory, no safety issues should exist for grab samples with respect to transportation.

Any decisions, observations, or deviations made to this work plan, or during the sample breakdown and analyses shall be documented in writing. These decisions and observations shall also be reported in the data report. The reporting formats for analyses are contained in Table 2.

3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Table 2, to be performed on the tank AP-108 grab samples, are based on the safety screening, evaporator and compatibility DQOs referenced in Section 1.0. The laboratory procedure numbers, which shall be used for the analyses, are included in Table 2. For these analyses, the laboratory and sampling organization should strive to meet SW-846 (EPA 1986) holding times. However, adherence to SW-846 holding times is not strictly required if documented cases show that additional time was required to ship, process, and analyze the samples (Morant 1994).

3.3 INSUFFICIENT LIQUID GRAB 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 Characterization TC and Evaporator program contact (given in Table 2) within one working day. A prioritization of the analyses required in this Sampling and Analysis Plan is given in Section 3.4. Any analyses prescribed by this document, but not performed, shall be identified in the appropriate data report.

3.4 PRIORITIES OF REQUESTED ANALYSES

In order to complete the entire compatibility, safety screening, and compliance assessments for tank AP-108, results from all of the analyses in Table 2 must be received. Therefore, if the amount of sample received is insufficient to perform all the analyses, the tank shall need to be resampled at a later date. However, analyses are still requested on any sample obtained, and should be performed in an order based on holding times. Those analyses with the shortest holding times shall be analyzed first.

Table 2: AP-108 Chemical, Radiological and Physical Analytical Requirements

Project Name		AP-108 Liquid Grab Sample					COMMENTS							REPORTING LEVELS			
Plan Number		WHC-SD-WM-TSAP-125, REV. 0					Homogenization Test - Not Required							FORMAT I		Early Notify	
							Field Blank - Required							FORMAT II		Process Control	
PROGRAM		PROGRAM CONTACTS					Trip Blank - Required							FORMAT III		Safety Screen	
A. Safety Screening		TWRS Characterization J. H. Baldwin					Hot Cell Blank - Performed as Necessary							FORMAT IV		Waste Management	
B. Evaporator		Safety Screening R. J. Cash												FORMAT V		RCRA Compliance	
C. Compatibility		Evaporator B.H. Von Bargaen					TANK		#SAMPLES		#RISERS			FORMAT VI		Special	
		Compatibility T.M. Blaak					AP-108		19		2						
		222-S Laboratory G. L. Miller															
PROGRAM	PRIMARY ANALYSES					QUALITY CONTROL ^{1,17}								CRITERIA			FOR-MAT
	METHOD	ANAL.	WHC PROCEDURE	PREP ²	FB	TB ³	HCB ⁴	DUP	SPK/MSD	BLK	CALIB STD	PR ⁵	AC ⁶	UNITS	NOTIFICATION LIMIT ⁷	EXPECTED RANGE ⁷	
A, B, C	DSC	Energy	LA-514-113	d ⁸				ea smpl	N/A	N/A	ea AB	±20	80-120	J/g	see notes 9 & 10	unknown	I, II, III, IV
A, C	TGA	% H ₂ O	LA-560-112	d ⁸				ea smpl	N/A	N/A	ea AB	±20	80-120	wt%	none specified	unknown	II, III, IV
B, C	Electrode	pH	LA-212-106	d ⁸				1/SE ¹²	N/A	N/A	ea AB	±0.3 ¹¹	N/A	pH	< 8.0	unknown	II, IV
A, B	Visual (surface sample)	Organic Layer	LA-519-151	d ⁸				N/A	N/A	N/A	N/A	N/A	N/A	N/A	presence	unknown	I, III, IV
B	Visual	Mixing and Compatibility Study	LT-519-183	d ⁸				N/A	N/A	N/A	N/A	N/A	N/A	N/A	Unusual changes in color, temperature, clarity, etc.	unknown	II ¹⁸
B	Visual/ Thermometric/ Gravimetric	Boildown Study	LT-519-183	d ⁸				N/A	N/A	N/A	N/A	N/A	N/A	N/A	Unusual changes in color, temperature, clarity, etc.	unknown	II ¹⁸
A, B, C	Gravimetric	SpG	LA-510-112	d ⁸				1/SE ¹²	N/A	N/A	N/A	±20 ¹¹	N/A	none	> 1.41	unknown	II, IV
B, C	Titration	OH ⁻	LA-211-102	d ⁸	X		X	1/SE ¹²	N/A	ea PB	ea AB	±20 ¹¹	N/A	µg/mL	< 170 or > 170,000	unknown	II, IV
B, C	IC	NO ₂ NO ₃ F ⁻ , SO ₄ ²⁻ PO ₄ ³⁻	LA-533-105	d ⁸	X		X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	< 506 or > 253,000 > 341,000 none specified > 9,450	unknown	II, IV
A ²¹ , B, C	ICP	Al Na ²¹	LA-505-151 LA-505-161	a	X		X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	none specified > 184,000	unknown unknown	II, IV II, IV
B	Combustion/ Coulometry	TC	LA-344-105	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	TC - TIC > 87	unknown	IV
B, C	Coulometric Autotitration	TIC	LA-342-100	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	TC - TIC > 87	unknown	II, IV
B, C	Combustion/ Coulometric Autotitration	TOC ¹⁹	LA-344-105	d ⁸	X		X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	> 30,000 ²²	unknown	I, II, IV
B	Combustion/ Coulometric Autotitration	TOC (Surface)	LA-344-105	d ⁸			X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	> 2,600	unknown	IV
B	Kjeldahl or ISE	NH ₃	LA-631-001	d ⁸	X		X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µg/mL	> 5,000	unknown	IV

Table 2: AP-108 Chemical, Radiological and Physical Analytical Requirements

PROGRAM	PRIMARY ANALYSES				QUALITY CONTROL ¹¹⁷																	CRITERIA	FOR- MAT
	METHOD	ANAL.	WHC PROCEDURE	PREP ²	FB	TB ³	HCB ⁴	DUP	SPK/ MSD	BLK	COALIB STD	PR ⁵	AC ⁶	UNITS	NOTIFICATION LIMIT ⁷	EXPECTED RANGE ⁷							
B	GC/MS	Acetone	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-110	µg/mL	> 87 ¹⁵	unknown	IV						
B	GC/MS	1-Butanol	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	30-110	µg/mL	> 26 ¹³	unknown	IV						
B	GC/MS	2-Butoxyethanol	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	30-110	µg/mL	> 95 ¹³	unknown	IV						
B	GC/MS	2-Butanone	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-110	µg/mL	> 58 ¹³	unknown	IV						
B	GC/MS	2-Hexanone	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-125 ²⁶	µg/mL	none specified	unknown	IV						
B	GC/MS	Methyl isobutyl Ketone	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-110	µg/mL	none specified	unknown	IV						
B	GC/MS	2-Pentanone	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-125 ²⁶	µg/mL	none specified	unknown	IV						
B	GC/MS	Tetrahydrofuran	LA-523-405	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	30-110	µg/mL	none specified	unknown	IV						
B	GC/MS	Tributyl Phosphate	LA-523-406	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	40-125 ²⁶	µg/mL	> 10,150 ¹³	unknown	IV						
B, C	Ion Exchange/Solvent Extraction/AEA	^{239/240} Pu	LA-943-128	a	X	X	X	N/A	1/SE ^{12,14}	ea PB	ea AB	±25 ¹¹	70-130	µCi/mL	^{239/240} Pu + 1.077E-10 * (U-gross) > 0.005 g/L	unknown	II, IV						
B	Laser Induced Fluorimetry	U-gross	LA-925-009	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20	70-130	µg/mL	^{239/240} Pu + 1.077E-10 * (U-gross) > 0.005 g/L	unknown	IV						
A, B	Alpha	Total Alpha	LA-508-101	a	X	X	X	ea smpl	1/SE ¹²	ea PB	ea AB	±25	70-130	µCi/mL	Evaporator > 0.10*SpG Safety Screen > 41	unknown	I, III, IV						
B	Beta	Total Beta	LA-508-101	a	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	70-130	µCi/mL	none specified	unknown	IV						
B	Distillation/Liquid Scintillation	³ H	LA-218-114	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	70-130	µCi/mL	none specified	unknown	IV						
B	Persulfate Oxidation/ Liquid Scintillation	¹⁴ C	LA-348-104	d ⁸	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±25	70-130	µCi/mL	> 0.26	unknown	IV						
B	Anion-Cation Exchange/Dist./Liquid Scintillation	⁷⁵ Se	LA-365-132	a	X	X	X	1/SE ^{12,14}	N/A	ea PB	N/A	±25 ¹¹	N/A	µCi/mL	> 0.078	unknown	IV						
B, C	Sep. & β counting	⁹⁰ Sr	LA-220-101	a	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20	75-125	µCi/mL	> 220	unknown	II, IV						
B	HPGE/MCA	⁶⁰ Co ¹³⁷ Cs	LA-548-121	a	X	X	X	1/SE ¹²	N/A	ea PB	ea AB	±25 ¹¹	N/A	µCi/mL	> 1.2	unknown	IV						
B	HPGE/MCA	¹⁰⁶ Ru ¹³⁵ Cs ¹⁴⁴ Ce ¹⁵⁴ Eu ¹⁵⁹ Eu ²⁴¹ Np	LA-548-121	a	X	X	X	1/SE ¹²	N/A	ea PB	N/A	±25 ¹¹	N/A	µCi/mL	> 53 > 15 none specified > 5 > 7 > 0.098 > 0.033	unknown unknown unknown unknown unknown unknown unknown	IV IV IV IV IV IV IV						
B	Solvent/Extraction/Liquid Scintillation	⁹⁹ Tc	LA-438-101	a	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20 ¹¹	75-125	µCi/mL	> 2	unknown	IV						
B	Extraction/Filtration/GEA	¹³⁹ Np	LA-378-103	d ⁸	X	X	X	N/A	1/SE ^{12,14}	ea PB	ea AB	±20	75-125	µCi/mL	> 0.0026	unknown	IV						
B	Extraction/Alpha Count	²³⁹ Np	LA-935-141	a	X	X	X	N/A	1/SE ¹²	ea PB	ea AB	±20	70-130	µCi/mL	none specified	unknown	IV						
B	Ion Exchange/ Solvent Extraction/AEA	²³⁸ Pu	LA-943-128	a	X	X	X	1/SE ^{12,14}	N/A	ea PB	N/A	±25 ¹¹	N/A	µCi/mL	> 0.0013	unknown	IV						

Table 2: AP-108 Chemical, Radiological and Physical Analytical Requirements

PROGRAM	PRIMARY ANALYSES				QUALITY CONTROL ^{1,17}								CRITERIA			FOR-MAT	
	METHOD	ANAL.	WHC PROCEDURE	PREP ²	FB	TB ³	HCBS ⁴	DUP	SPK/MSD	BLK	CALIB STD	PR ⁵	AC ⁶	UNITS	NOTIFICATION LIMIT ⁷		EXPECTED RANGE ⁷
B, C	Ion Exchange/ Solvent Extraction/AEA	²⁴¹ Am	LA-953-103	a	X		X	1/SE ^{12,14}	1/SE ^{12,14}	ea PB	ea AB	±20 ¹¹	70-130	µCi/mL	> 1	unknown	II, IV
B	Ion Exchange/ Solvent Extraction/AEA	²⁴⁴ Cm	LA-953-103	a	X		X	1/SE ^{12,14}	N/A	ea PB	N/A	±20 ¹¹	N/A	µCi/mL	> 0.013	unknown	IV
PROGRAM	SECONDARY ANALYSES				QUALITY CONTROL								CRITERIA			FOR-MAT	
	METHOD	ANAL.	WHC PROCEDURE	PREP ²	FB	TB ³	HCBS ⁴	DUP	SPK/MSD	BLK	CALIB STD	PR ⁵	AC ⁶	UNITS	NOTIFICATION LIMIT ⁷		EXPECTED RANGE ⁷
A	RSST ¹⁵	Energy	see 15 below	d ⁸				N/A	N/A	N/A	N/A	N/A	N/A	J/g	> 480	unknown	I, IV
A	Distillation ¹⁶	CN	LA-695-102	d ⁸				ea smpl	1/SE ¹²	ea PB	ea AB	±20	80-120	µg/mL	> 39,000	unknown	I, IV
A	Hot Persulfate ¹⁶	TOC	LA-342-100	d ⁸				N/A	1/SE ¹²	ea PB	ea AB	±20	80-120	µg/mL	> 30,000 ²²	unknown	I, IV

¹FB-field blank, TB-trip blank, HCB-hot cell blank, DUP-duplicate, SPK/MSD-spike and matrix spike duplicate, PR-precision, AC-accuracy, ea-each, smpl-sample, AB-analytical batch, PB-preparation batch, N/A-not applicable, mtrx-matrix

²d-direct, a-acid digestion

³Trip blank analyses to be performed only when detected in the field blank.

⁴Analyses on the hot cell blank shall be performed only when applicable.

⁵Precision is the RPD between spike and MSD results, except as noted (note 11).

⁶The unit for accuracy is percent recovery.

⁷Units for notification limits and expected range are those listed in the "units" column unless otherwise indicated.

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

⁹Absolute value of ratio of exotherm to endotherm > 1 or exotherm < 335° F (168 °C). Action limit is applicable up to 500 °C. Notification limit is for the Evaporator DQO.

¹⁰Action limit for safety screening DQO is 480 J/g (dry weight basis). Secondary analyses required if action limit is exceeded.

¹¹For these analytes, precision is evaluated on the RPD between a sample (unspiked) and sample replicate (except for pH, where the precision requirement is < 0.3 pH units between sample/replicate).

¹²SE Sampling Event. 1/SE means that only one sample from the total number of samples must be spiked or run as duplicates.

¹³For individual organic species limits in the candidate feed tank, the sum of the fractions rule applies.

¹⁴Tracer or carrier may be used in place of a spike and results corrected for recovery.

¹⁵RSST performed only if DSC exceeds 480 J/g (dry wt basis). The RSST method, yet to be proceduralized, may be found in WHC-SD-WM-TP-104.

¹⁶This analysis required if DSC exceeds 480 J/g.

¹⁷If a QC standard does not meet the QC criteria, no rerun samples can be analyzed until the QC problem is resolved.

¹⁸The mixing and compatibility study as well as the bolldown study are to be completed only after all tanks designated for a campaign are sampled. When the studies are complete, an Internal Memo will be issued from the 222-S Laboratory to Evaporator Project Plant Engineering describing the results.

¹⁹TOC is not calculated as TC - TIC, but must be determined analytically.

²⁰For these analytes, the upper limit for accuracy has been changed from the values given in the Evaporator DQO to reflect results achieved with tank waste samples previously.

²¹Fe, Mn, U, Cr, Ni, Al, Na, and Si are all secondary analytes for safety screening and should be reported if found above detection limits.

²²Dry weight basis. The 3 wt% limit was converted to 30,000 µg/mL assuming a density of 1g/mL.

4.0 QUALITY ASSURANCE

4.1 LABORATORY OPERATIONS

Laboratories performing analyses in support of this SAP shall have approved and implemented quality assurance project plans. These QA plans shall meet the *Hanford Analytical Services Quality Assurance Requirements Document* (DOE 1996) minimum requirements as the baseline for laboratory quality systems. Quality requirements for conducting Characterization Project sampling and analysis are described in *Tank Waste Remediation System Characterization Project Quality Policies* (Tribble 1996), *Fiscal Year 1996 Tank Waste Analysis Plan* (Homi 1995), and in this SAP. Characterization Project sampling and analysis shall be conducted in conformance with these quality assurance requirements.

Method specific quality control such as calibrations and blanks are also found in the analytical procedures. Sample quality control (duplicates, spikes, standards) are identified in Table 2. If no criteria are provided in Table 2, the performing laboratory shall perform to its quality assurance plan.

4.2 SAMPLE COLLECTION

Grab samples from tank AP-108, including field and trip blanks, are to be taken and shipped to the performing laboratory by Sampling Operations.

The following documents will be used as guidance in the handling and shipment of compatibility grab samples:

- TO-100-052, "Segregate, Package, and Inventory Radioactive Waste."
- WHC-CM-2-14, "Responsibilities and Procedures for all Hazardous Material Shipments."
- WHC-SD-TP-SARP-001, "Sample Pig Transport System Safety Analysis Report for Packaging (on-site)."
- WHC-SD-WM-HSP-002, "Tank Farm Health and Safety Plan."

Samples shall be identified by a unique number before being shipped to the laboratory (Table 1). 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 send the samples to the laboratory within 3 days of removing the samples from the tank. The field/trip blank shall count as a sample. Sampling Operations is responsible for verbally notifying the shift manager at 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. Grab samples are shipped in a cask and sealed with a Waste Tank Sample Seal.

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

The sealed and labeled samples are shipped to the laboratory along with the chain-of-custody form. The receipt and control of samples in the WHC 222-S Laboratory are described in laboratory procedure LO-090-101.

5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

5.1 EXCEPTIONS TO DQO REQUIREMENTS

- ▶ In the Waste Compatibility DQO, several necessary specifications concerning sampling and analysis were omitted. These clarifications are addressed in (Sutey 1994).

Since historical information exists to adequately address the potential for line plugging and precipitation of solids during the transfer of waste, no viscosity or cooling curve analysis shall be required during this analysis activity.

- ▶ The accuracy range for 2-hexanone, 2-pentanone, and tributyl phosphate have been changed to 40-125%. The earlier values, given in the DQO (Von Bargaen 1995), were estimates of what might be achievable (they were not derived from

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contract laboratory program nor SW-846 standards). Recent testing of tank waste samples has shown that a range of 40-125% is more appropriate for these analytes.

5.2 CLARIFICATIONS AND ASSUMPTIONS

A number of clarifications and assumptions relating to the sampling strategy, and notification limits or decision thresholds identified in the applicable DQOs need to be made. Each of these issues are discussed below.

- ▶ The safety screening DQO (Dukelow, et al., 1995) does not explicitly state whether grab samples should be analyzed. Therefore, the safety screening analyses will be applied as appropriate when samples are taken for other programs (e.g., Evaporator, compatibility). For tank AP-108, the samples taken for the Evaporator Project have been chosen to be representative of the waste in the tank; analysis of these samples for safety screening analytes will provide data to help categorize the tank for safety screening purposes.
- ▶ The precision and accuracy of the analytes given in Table 2 are those given by the DQO with the more stringent requirements.
- ▶ 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.

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

NOTE: A large error in the DSC value may result when converting samples containing greater than 90% water to a dry weight basis. However, this conversion is still required.

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

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

- ▶ A laboratory verified data package including blank analyses, holding time checks, MSD analyses, surrogate recoveries, and any other requested information is required for the compliance samples taken for candidate feed tanks and liquid effluent retention facilities. This type of data verification was chosen because it is appropriate for an intermediate treatment facility such as the evaporator (Geier 1995).

6.0 DELIVERABLES

All analyses of tank AP-108 waste material shall be reported as Formats I, II, III, or IV as indicated in Table 2. Additional information regarding reporting formats is given in (Schreiber 1994).

6.1 FORMAT I REPORTING

Table 2 contains the notification limits for each analyte. Any results that exceed the notification limits and that are identified as having a Format I requirement shall be reported via telephone by the 222-S Laboratory Shift Manager to the East Tank Farm Operations Shift Manager at 373-2689 as soon as the data are obtained and reviewed (Kristofzski 1995). Further, any results which exceed the Operating Specification Document limits as identified in (Kristofzski 1996) shall warrant notification. This verbal notification must be followed within 1 working day by written communication to Safety Screening Representative, Process Control, Evaporator Project, the Characterization Project Office, and the TC documenting the observations (Schreiber 1995). Additional analyses for other purposes may be requested by the TC, or by a memorandum of understanding.

Some evaporator program analyses are identified in Table 2 as having notification limits without the Format I requirement. If the results of these analyses exceed their respective notification limits, notification should be made within one working day to the Evaporator Project contact and TC only.

6.2 FORMAT II REPORTING

The data found from these analyses will help determine whether waste from tank AP-108 may be used in the Evaporator operations without creating safety, compliance, or operational problems. The results of the analyses performed for the Compatibility DQO (Program C on Table 2) shall be provided as needed to make compatibility decisions. The data shall also be provided in the final Format IV) report. The reporting contacts for the Evaporator and Compatibility programs are listed on Table 2.

When the mixing/buildown and compatibility studies are complete, an Internal Memo(s) will be issued to the Evaporator Project describing the results.

6.3 FORMAT III REPORTING

A Format III report, providing the results of the primary safety screen analyses required by (Dukelow, et al. 1995) shall be issued to the Safety Screening Representative, the Characterization Project, Process Control, and the Tank Characterization Resource Center 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 given in Schreiber (1994).

6.4 FORMAT IV REPORTING

This final report shall include the results of the analyses required by the compatibility, safety screening, and Evaporator DQOs. The data package will be provided to the Evaporator Project, Characterization Project, the Safety Screening Representative, the Tank Characterization Resource Center, and Tank Characterization Database representatives. The laboratory should strive to complete the data package within 136 days of the sampling event (136 day annual average reporting cycle required for Evaporator candidate feed sampling events), but must complete the report within 176 days of the sampling event (Von Barga 1995). Detailed information regarding the contents of this reporting format are given in (Schreiber 1994).

For the Evaporator Project feed candidate compliance samples and analyses, the data shall be verified by the laboratory, including blank analyses, holding time checks, MSD analyses, surrogate recoveries, and any other requested information.

In addition to this data package, an electronic version of the analytical results shall be in the Tank Characterization Database within 7 days of the release of the final report. The data must be available to the Washington State Department of Ecology within 7 days of the release of the final data package. The electronic version shall be in the standard electronic format specified in (Bobrowski et al. 1994).

7.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Program. These changes shall be documented through the use of internal characterization change notices or analytical deviation reports for minor low impact changes and documented in applicable laboratory notebooks. All significant changes (such as changes in scope) shall be documented by Characterization Plans, Coordination, and Reports via an Engineering Change Notice to this plan. All changes shall also be clearly documented in the final data report. Significance is determined solely by the TC.

At the request of the Characterization Program, additional analysis of sample material from this characterization project shall be performed according to a revision of this plan.

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