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Compatibility Grab Sampling and Analysis Plan for Fiscal Year 2001.

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Office of River Protection Contract DE-AC06-99RL14047

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LIST OF ABBREVIATIONS

AEA	alpha energy analysis
CHG	CH2M HILL Hanford Group, Inc.
CPO	Characterization Project Operations
DQO	data quality objective
DSC	differential scanning calorimetry
EQL	estimated quantitation limit
FH	Fluor Hanford, Inc.
GEA	gamma energy analysis
g/mL	grams per milliliter
IC	ion chromatography
ICP/AES	inductively coupled plasma □ atomic emission spectroscopy
ICP/MS	inductively coupled plasma □ mass spectrometry
J/g	joules per gram
LCS	laboratory control standard
LFL	lower flammability limit
MDA	minimum detectable activity
N/A	not applicable
NHC	Numatec Hanford Corporation
NP	not performed
PCB	polychlorinated biphenyls
QA	quality assurance
QC	quality control
RPD	relative percent difference
RPP	River Protection Project
SAP	sampling and analysis plan
TBD	to be determined
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
wt%	weight percent
ΦCi/g	microcuries per gram
ΦCi/mL	microcuries per milliliter
Φg/g	micrograms per gram
Φg/kg	micrograms per kilogram
Φg/L	micrograms per liter
Φg/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 grab samples obtained to address waste compatibility. It is written in accordance with requirements identified in *Data Quality Objectives for Tank Farms Waste Compatibility Program* (Mulkey et al. 1999) and *Tank Farm Waste Transfer Compatibility Program* (Fowler 2000).

2.0 SAMPLING EVENT REQUIREMENTS

Table 2-1 identifies tanks scheduled to be grab sampled to address waste compatibility. The number of samples, the riser to be used for sampling, and the elevations and depths at which the samples are to be obtained are identified. This SAP will be revised and updated as necessary to include additional grab sample events.

Prior to 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 River Protection Project (RPP) Data Development and Interputation within ten working days of the sampling event (Adams et al. 2000). 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 RPP Data Development and Interputation shall notify the Flammable Gas Safety Project. 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.

Grab samples shall be obtained using plant operating procedure TO-080-403, *Supernatant or Sludge Sampling of Waste Storage Tanks Using a Glovebag* or a suitable substitute approved plant operating procedure. The procedure title and number used shall be documented in the work package.

If quality-affecting changes to the sampling requirements must be made (including the risers or samples to be obtained), 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 the work packages. These work packages contain the operating procedures and the chain-of-custody records for the sampling events.

No field/trip blanks are required during these sampling events. Samples should be shipped to the laboratory within three calendar days from the time of sampling.

Table 2-1. Tank Grab Sampling Information

Tank	Sample Number	Sample Type	Sample Location	Sample Elevation[1]	Sample Depth[2, 3]	Shipping Information[4]
241-SY-102[2, 5]	2SY-01-1 2SY-01-2 2SY-01-3	Supernate (surface) Supernate Supernate	R03	1 in. below liquid level 140 in. 80 in.	zipcord reading + 1 in. 522 in. 582 in.	^{137}Cs, ^{90}Sr. 1.44E-3 TBq/120-mL bottle (Type A)
TX-244[2]	244TX-00-1 244TX-00-2 244TX-00-3	Supernate (surface) Supernate Supernate	Riser 8	1 in. below liquid level 42 in. 10 in.	zipcord reading + 1 in. 386 in. 418 in.	^{137}Cs, ^{90}Sr. <2.5E-7 TBq/120-mL bottle (Type A)
241-U-111[3]	11U-01-1 11U-01-2 11U-01-3	Supernate (surface) Supernate Supernate	R13	128 55 10	334 407 452	^{137}Cs, ^{90}Sr. <7.2E-3 TBq/120-mL bottle (Type A)

Notes:
1 Sample elevation is defined as distance from tank bottom to mouth of sample bottle.
[2]For tanks 241-SY-102 and 244TX the sample depth is defined as the distance from the top of the riser to the mouth of the sample bottle.
[3]For tanks 241-U-111 sample depth is defined as the distance from the top of the 2 inch steel cover plate to the mouth of the sample bottle.
[4]Sample activity estimate based on previous sample results.
[5]For this sample event, obtain zipcord (surface level) and sludge level measurements from the designated riser and document the levels in the work package.

3.0 LABORATORY ANALYSIS REQUIREMENTS

3.1 ANALYSIS SCHEME

In order to comply with Mulkey et al. (1999) and Fowler (2000) the following steps shall be performed on each sample. All samples shall be analyzed to meet the requirements of the Compatibility program.

X Record visual observations such as color and clarity of the liquid and the presence of any solid particles in the liquid. Record the volume of settled solids, if present.

X Closely inspect the liquid sample for the presence and approximate volume of any potential organic layers. Record the volume of separable organic phase, if present.

X For samples expected sample type to be supernatant samples (Table 2-1):

- X Remove sufficient aliquots of liquid and perform the analyses listed in Table 3-1.
- X If the sample has greater than 25 percent settled solids, contact the tank coordinator for further instructions.

X For samples expected to contain solids:

- X Allow the solids to settle record the volume percent settled solids, and then decant the liquid (supernatant) from the solids. Unless specified in Section 5, no analyses are required for the decanted supernatant.
- X Record the bulk density of the □wet□ sludge, centrifuge the solids, and the volume percent centrifuged solids, and then decant the liquid (interstitial liquid) from the centrifuged solids.
- X Remove sufficient aliquots of interstitial liquid and perform the analyses listed in Table 3-1.
- X X Remove sufficient aliquots of centrifuged solids and perform the analyses as shown in Table 3-2. Subsample solids for polychlorinated biphenyls (PCBs) as specified in Section 5.1.

X Archive remaining sample material for possible future analyses.

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 from this work plan during the sample breakdown and analyses shall be documented in writing with justification. These decisions, observations, and deviations shall be reported in the data report. The reporting formats for analyses are specified in Tables 3-1 and 3-2 and are described in Section 7.0

3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Tables 3-1 and 3-2 to be performed on the (liquid and solid, respectively) grab samples are based on the compatibility data quality objective (DQO). 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 of solids, LA-505-159 or LA-505-163 for acid digestion of samples, LA-504-101 for water leach of solids or

appropriate approved laboratory procedure. The laboratory project coordinator and/or chemist will make appropriate procedure determination.

3.3 INSUFFICIENT SAMPLE RECOVERY

If the amount of material recovered from samples taken from the tank is insufficient to perform the analyses requested in the SAP, the laboratory shall notify the tank coordinator within one working day. A prioritization of the analyses is provided in Section 5.3. Any analyses prescribed by this SAP, but not performed, shall be identified in the appropriate data report with justification for non-performance.

Table 3-1. Chemical, Radiological, and Physical Analyses: Liquids

LIQUID ANALYSES			
Project Name	FY 2001 Compatibility Grab Samples	COMMENTS	REPORTING LEVELS
Plan Number	RPP-7023, Rev. 0	Homogenization Test - Not Required	FORMAT I: Early Notify
PROGRAM	PROGRAM CONTACTS	*Field Blank - Not Required*	*FORMAT II: Process Control*
A. Compatibility	DST/SST Compatibility: T. M. Blaak/R. E. Larson	Hot Cell Blank - Performed as Necessary	FORMAT III: Safety Screen
B. Process Control	RPP Technical Point of Contact: T. L. Lauricella		FORMAT IV: Waste Management
			FORMAT V: RCRA Compliance
			FORMAT VI: Special

PROGRAM	PRIMARY ANALYSES			SAMPLE	PREP[1]	QUALITY CONTROL[2]				CRITERIA			REPORT FORMAT
	METHOD	ANALYSIS	PROCEDURE	LIQUID		DUP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT[3]	EXPECTED RANGE[3]	
A	Titration	OH^-	LA-211-102	X	d	ea smpl	N/A	ea AB	ea AB	µg/mL	see[4]	unknown	I, II, IV
A	pH	$[H^+]$	LA-212-106	X	d	ea smpl	N/A	N/A	ea AB	pH	see[5]	unknown	I, II, IV
A	IC	Cl^-, F^-, PO_4^{-3}, SO_4^{-2}, NO_3^-, NO_2^-, oxalate[6]	LA-533-105 LA-533-115	X	d[7]	ea smpl	1/riser	ea AB	ea AB	µg/mL	see[4]	unknown	I, II, IV
A	DSC	Energy[8]	LA-514-114, LA-514-115	X	d	ea smpl	N/A	N/A	ea AB	J/g	ratio exo:endo > 1 or exo > 480[9]	unknown	I, II, IV
A	TGA	wt % H_2O	LA-514-114, LA-514-115	X	d	ea smpl	N/A	N/A	ea AB	wt%	none	unknown	II, IV
A	Visual	Organic	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	presence	unknown	I, II, IV
A	Gravimetry	Specific Gravity	LA-510-112	X	d	ea smpl	N/A	N/A	ea AB	SpG	none	unknown	II, IV
A	Gravimetry	Bulk Density[10]	LA-519-132	X	d	N/A	N/A	N/A	N/A	g/mL	none	unknown	II, IV
A	ICP/AES	Al, Cr, Fe, Mn, Na, Ni[11]	LA-505-151 LA-505-161	X	d[7]	ea smpl	1/riser[12]	ea PB	ea AB	µg/mL	see[4]	unknown	I, II, IV
A	ICP/MS	U isotopes (^{235}U, ^{238}U)	LA-506-101	X	d[7]	ea smpl	1/riser[12]	ea PB	ea AB	µg/mL	none	unknown	II, IV
A	AEA	^{238}Pu, $^{239/240}Pu$, ^{241}Am	LA-953-104	X	d[7]	ea smpl	1/riser[13]	ea PB	ea AB	µCi/mL	> 61.5[14]	unknown	I, II, IV
A	GEA	^{137}Cs	LA-548-121	X	d[7]	ea smpl	N/A	ea AB	ea AB	µCi/mL	see[15, 16]	unknown	I, II, IV
A	Sep. & ß Counting	^{90}Sr	LA-220-101	X	d[7]	ea smpl	1/riser[13]	ea AB	ea AB	µCi/mL	see[15, 17]	unknown	I, II, IV
A	Persulfate Oxidation	TIC/TOC	LA-342-100	X	d	ea smpl	1/riser	ea AB	ea AB	µg C/mL	> 45,000[9, 15, 18, 19]	unknown	I, II, IV
A	Visual	% Settled Solids[20]	LA-519-151	X	d	ea smpl	N/A	N/A	N/A	%	none	unknown	II, IV
A	Ion Selective Electrode	Ammonia	LA-631-001	X	d[7]	ea smpl	1/riser	ea PB	ea AB	µg/mL	none	unknown	II, IV
A	Phosphorescence	U Total	LA-925-009	X	d[7]	ea smpl	1/riser	ea AB	ea AB	µg/mL	none	unknown	II, IV
B	Polychlorinated Biphenyls	Aroclors (1016, 1221, 1232, 1242, 1248, 1254 1260)[21]	LA-523-115, LA-523-140	X	d	ea smpl	1/riser	ea PB	ea AB	µg/L	none	unknown	II, IV

PROGRAM	SECONDARY ANALYSES			SAMPLE	PREP[1]	QUALITY CONTROL[2]				CRITERIA			REPORT FORMAT
	METHOD	ANALYSIS	PROCEDURE	LIQUID		DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[3]	EXPECTED RANGE[3]	
A	Potentiometric Titration	Caustic Demand[22]	LA-211-104	X	d	ea smpl	N/A	N/A	N/A	mole OH/L	none	unknown	II, IV

Table 3-1. Chemical, Radiological, and Physical Analyses: Liquids

Notes:

[1] d-direct, f-fusion, a-acid, w-water

[2] DUP = duplicate, BLK = blank, LCS = laboratory control sample, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, N/A = not applicable, SE = sample event

[3] Units for notification limits and expected range are those listed in the "units" column. Except as noted, limits apply to all DSTs, SSTs, and saltwell receivers (DCRTs 244-BX, 244-S, 244-TX and 244-U and TK-003 of 244-CR Vault). Unless otherwise indicated, immediate notifications to be made to both On-Call Process Engineer and Tank Farm Operations Shift Manager

[4] OH, NO_3, NO_2 and Na Limits: For SSTs, there are no active limits for samples. For DSTs and saltwell receivers, there are variable limits dependent upon nitrate concentration:

For Saltwell receivers:

a. For $NO_3 \leq 1.0$ M: $0.010 \text{ M} \leq OH \leq 5.0$ M (upper OH limit is 8.0 M for solutions below 167°F, lower limit corresponds to pH 12 in dilute solutions.) and $0.011 \text{ M} \leq NO_2 \leq 5.5$ M.

b. For $1.0 \text{ M} < NO_3 \leq 3.0$ M: $0.1 [NO_2] \leq OH \leq 10$ M and $OH + NO_2 \geq 0.4 [NO_3]$

c. For $NO_3 > 3.0$ M: $0.3 \text{ M} \leq OH < 10$ M, $OH + NO_2 \geq 1.2$ M, and $NO_3 \leq 5.5$ M

For DSTs with tank temperatures below 212°F:

a. For $NO_3 \leq 1.0$ M: $0.010 \text{ M} \leq OH \leq 5.0$ M (upper OH limit is 8.0 M for solutions below 167°F, lower limit corresponds to pH 12 in dilute solutions), $0.011 \text{ M} \leq NO_2 \leq 5.5$ M, and $NO_3/(OH+NO_2) < 2.5$ M.

b. For $1.0 \text{ M} < NO_3 \leq 3.0$ M: $0.1x\ NO_3 \leq OH \leq 10$ M and $OH + NO_2 \geq 0.4 \times NO_3$

c. For $NO_3 > 3.0$ M: $0.3 \text{ M} \leq OH < 10$ M, $OH + NO_2 \geq 1.2$ M, and $NO_3 \leq 5.5$ M

[5] Per CPS-T-149-00010, for DSTs, DCRTs, 244-AR, and 244-CR: if tank Pu inventory is > 10 kg and supernate depth is > 30 cm (12 in.), the investigation level for pH is ≤ 8.0. If Pu< 10 kg or supernate depth is < 30 cm (12 in.), then there is no pH restriction. Notify On-Call Process Engineer if pH ≤ 8.0.

[6] Results should be reported for all IC analytes, however, only the analytes indicated here are required to meet the QC criteria in Tables 3-1 and 4-1.

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

[8] If exotherm is observed, both the endotherm and exotherm values must be reported.

[9] Dry weight basis. Limit applies to DSTs and SSTs.

[10] In order to preserve sample volume, bulk density measurement may be performed instead of specific gravity.

[11] Results should be reported for all ICP analytes, however, only the required analytes are required to meet the QC criteria in Tables 3-1 and 4-1.

[12] Either serial dilutions or matrix spikes will be performed.

[13] Tracer or carrier may be used in place of spike and results corrected for recovery.

[14] This notification limit applies to ^{239}Pu only.

[15] Immediate notification applicable to On-Call Process Engineer only.

[16] Notification required if Cs > 3.28E+05 µCi/L for SSTs, 4.10E+05 µCi/L for SY Farm, or 5.74E+05 µCi/L for AN, AP, and AW farms.

[17] Notification required if Sr > 2.30E+05 µCi/L for SSTs, 2.88E+05 µCi/L for SY Farm, or 4.04E+05 µCi/L for AN, AP, and AW farms.

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

[19] This notification limit applies to TOC only.

[20] Analysis may not be necessary if the amount ot solids is small. Under these circumstances, the project and tank coordinator shall determine if the analysis is to be performed.

[21] Analysis not required on all samples. See Sectrion 5 to determine if analysis is required for a given sample.

[22] To be performed if analysis indicates waste is hydroxide deficient.

Table 3-2. Chemical, Radiological, and Physical Analyses: Solids

SOLID ANALYSES			
Project Name	FY 2001 Compatibility Grab Samples	**COMMENTS**	**REPORTING LEVELS**
Plan Number	RPP-7023, Rev. 0	Homogenization Test - Not Required	FORMAT I: Early Notify
PROGRAM	**PROGRAM CONTACTS**	Field Blank - Not Required	FORMAT II: Process Control
A. Compatibility	DST/SST Compatibility: T. M. Blaak/R. E. Larson	Hot Cell Blank - Performed as Necessary	FORMAT III: Safety Screening
B. Process Control	RPP Technical Point of Contact: T. L. Lauricella		FORMAT IV: Waste Management
			FORMAT V: RCRA Compliance
			FORMAT VI: Special

PROGRAM	PRIMARY ANALYSES			SAMPLE	PREP[1]	QUALITY CONTROL[2]				CRITERIA			REPORT FORMAT
	METHOD	ANAL.	PROCEDURE	SOLIDS		DUP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT[3]	EXPECTED RANGE[3]	
A	Titration	OH^-	LA-211-102	X	w/s	ea smpl	N/A	ea AB	ea AB	µg/g	none	unknown	II, IV
A	pH	$[H^+]$	LA-212-105	X	d	ea smpl	N/A	N/A	ea AB	pH	none	unknown	II, IV
A	IC	Cl^-, F^-, PO_4^{-3}, SO_4^{-2}, NO_3^-, NO_2^-, oxalate[4]	LA-533-105 LA-533-115	X	w	ea smpl	1/riser	ea AB	ea AB	µg/g	none	unknown	II, IV
A	DSC	Energy[5]	LA-514-114, LA-514-115	X	d	ea smpl	N/A	N/A	ea AB	J/g	ratio exo:endo > 1 or exo ≥ 480[6]	unknown	I, II, IV
A	TGA	wt % H_2O	LA-514-114, LA-514-115	X	d	ea smpl	N/A	N/A	ea AB	wt%	none	unknown	II, IV
A	Visual	Organic	LA-519-151	X	d	N/A	N/A	N/A	N/A	N/A	presence	unknown	I, II, IV
A	Gravimetry	Bulk Density	LA-519-132	X	d	N/A	N/A	N/A	N/A	g/mL	none	unknown	II, IV
A	ICP/AES	Al, Cr, Fe, Mn, Na, Ni[7]	LA-505-151 LA-505-161	X	f or a	ea smpl	1/riser[8]	ea PB	ea AB	µg/g	none	unknown	II, IV
A	ICP/MS	U isotopes (^{235}U, ^{238}U)	LA-506-101	X	f or a	ea smpl	1/riser[8]	ea PB	ea AB	µg/g	none	unknown	II, IV
A	AEA	^{238}Pu, $^{239/240}Pu$, ^{241}Am	LA-953-104	X	f	ea smpl	1/riser[9]	ea PB	ea AB	µCi/g	> 41[10]	unknown	I, II, IV
A	GEA	^{137}Cs	LA-548-121	X	f	ea smpl	N/A	ea PB	ea AB	µCi/g	see[11, 12]	varied	II, IV
A	Sep. & ß Counting	^{90}Sr	LA-220-101	X	f	ea smpl	1/riser[9]	ea AB	ea AB	µCi/g	see[11, 13]	unknown	II, IV
A	Persulfate Oxidation	TOC/TIC	LA-342-100	X	d	ea smpl	1/riser	ea AB	ea AB	µg C/g	> 45,000[6, 11, 14]	varied	I, II, IV
A	Visual	% Settled Solids[15]	LA-519-151	X	d	ea smpl	N/A	N/A	N/A	%	none	unknown	II, IV
A	Ion Selective Electrode	Ammonia	LA-631-001	X	a	ea smpl	1/riser	ea PB	ea AB	µg/g	none	unknown	II, IV
A	Phosphorescence	U Total	LA-925-009	X	f	ea smpl	1/riser	ea AB	ea AB	µg/g	none	unknown	II, IV
B	Polychlorinated Biphenyls	Aroclors (1016, 1221, 1232, 1242, 1248, 1254 1260)[16]	LA-523-115, LA-523-140	X	d	ea smpl	1/riser	ea PB	ea AB	µg/g	none	unknown	II, IV

PROGRAM	SECONDARY ANALYSES			SAMPLE	PREP[1]	QUALITY CONTROL[2]				CRITERIA			REPORT FORMAT
	METHOD	ANAL.	PROCEDURE	SOLIDS		DUP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[3]	EXPECTED RANGE[3]	
A	Potentiometric Titration	Caustic Demand[17]	LA-211-104	X	d	ea smpl	N/A	N/A	N/A	mole OH/L	none	unknown	II, IV

Notes:

[1] d-direct, f-fusion, a-acid, w-water, w/s-half and half water and solid sample

[2] DUP = duplicate, BLK = blank, LCS = laboratory control standards, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, N/A = not applicable

[3] Units for notification limits and expected range are those listed in the "units" column. Except as noted, limits apply to all DSTs, SSTs, and saltwell receivers (DCRTs 244-BX, 244-S, 244-TX, and 244-U and TK-003 of 244-CR Vault). Unless otherwise indicated, immediate notifications to be made to both On-Call Process Engineer and Tank Farms Operations Shift Manager.

Table 3-2. Chemical, Radiological, and Physical Analyses: Solids

[4]Results should be reported for all IC analytes, however, only the analytes indicated here are required to meet the QC criteria in Tables 3-2 and 4-1.
[5]If exotherm is observed, both the endotherm and exotherm values must be reported.
[6]Dry weight basis. Limit applies to DSTs and SSTs.
[7]Results should be reported for all ICP analytes, however, only the required analytes are required to meet the QC requirements of Tables 3-2 and 4-1.
[8]Either serial dilutions or matrix spikes will be performed.
[9]Tracer or carrier may be used in place of a spike and results corrected for recovery.
[10]This notification limit applies to ^{239}Pu only.
[11]Immediate notification applicable to On-Call Process Engineer only.
[12]Notification required if Cs > 3.28E+05 µCi/L for SSTs, 4.10E+05 µCi/L for SY Farm, or 5.74E+05 µCi/L for AN, AP, and AW farms.
[13]Notification required if Sr > 2.30E+05 µCi/L for SSTs, 2.88E+05 µCi/L for SY Farm, or 4.04E+05 µCi/L for AN, AP, and AW farms.
[14]This notification limit applies to TOC only.
[15]Analysis may not be necessary if the amount of solids is small. Under these circumstances, the project and tank coordinators shall determine if the analysis is to be performed.
[16]Analysis may not be necessary if the amount ot solids is small. Under these circumstances, the project and tank coordinator shall determine if the analysis is to be performed.

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 *Nonconforming Item Reporting and Control* (CHG 2000).

Quality requirements for conducting Characterization Project sampling and analysis are described in *Tank Waste Remediation System Characterization Project, Quality Policies* (Board 1998) and this SAP. Characterization Project sampling and analysis shall be conducted in conformance with these QA requirements.

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 minimum requirements of *Hanford Analytical Services Quality Assurance Requirements Document* (DOE 1998) as a baseline for laboratory quality systems. *222-S Laboratory Quality Assurance Plan* (Markel 2000) specifies the requirements for assuring the quality of sample analysis conducted at the 222-S Laboratory.

Analytical quality control (QC) requirements (duplicates, spikes, blanks, laboratory control samples) are identified in Tables 3-1, 3-2, and 4-1. The laboratory shall also use calibration and calibration check standards appropriate for the analytical instrumentation being used (see DOE [1998] 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 any 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. 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 Characterization Project Operations (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 for

each sample. Pertinent sampling information (e.g., unusual waste characteristics or sampling problems) should be noted in the comments 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 4-1. 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]
Liquids				
DSC	# 30	N/A	N/A	80 - 120
TGA	# 20	N/A	N/A	80 - 120
Bulk density	N/A	N/A	N/A	N/A
Specific gravity	# 20	N/A	N/A	N/A
ICP/AES	# 20	75 - 125	< EQL	80 - 120
ICP/MS	# 20	75 - 125	< EQL	80 - 120
IC	# 20	75 - 125	< EQL	80 - 120
TIC/TOC	# 20	75 - 125	< EQL	80 - 120
OH	# 20	N/A	< EQL	80 - 120
pH	# 20	N/A	N/A	∀ 0.1 pH unit
AEA (Pu/Am)	# 20	N/A[5]	< MDA	70 - 130
GEA	# 20	N/A	< MDA	80 - 120
^{90}Sr	# 20	N/A[5]	< MDA	75 - 125
Ammonia	# 20	75 - 125	< EQL	80 - 120
U Total	# 20	75 - 125	< EQL	80 - 120
PCB	# 20	70 - 130	< EQL[6]	70 - 130
Caustic demand	N/A	N/A	N/A	N/A
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
ICP/AES	# 20	75 - 125	< EQL	80 - 120
ICP/MS	# 20	75 - 125	< EQL	80 - 120
IC	# 20	75 - 125	< EQL	80 - 120
TIC/TOC	# 20	75 – 125	< EQL	80 - 120
OH	# 20	75 – 125	< EQL	80 - 120
pH	# 20	N/A	N/A	∀ 0.1 pH unit
AEA (Pu/Am)	# 20	N/A[5]	< MDA	70 - 130
GEA	# 20	N/A	< MDA	80 - 120
^{90}Sr	# 20	N/A[5]	< MDA	75 - 125

Table 4-1. 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]
Ammonia	# 20	75 - 125	< EQL	80 - 120
U Total	# 20	75 - 125	< EQL	80 - 120
PCB	# 20	70 - 130	< EQL[6]	70 - 130
Caustic demand	N/A	N/A	N/A	N/A

Notes:

N/A = not applicable
NP = not performed
MDA = minimum detectable activity
EQL = estimated quantitation limit
LCS = laboratory control standard

[1]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.
[2]The criteria are recommended. Failures are permissible if the requirements in the QA section are followed.
[3]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.
[4]For some analyses, this could be a method spike or a blank spike. Ranges are percent recovery of theoretical.
[5]A tracer or carrier may be substituted for the spike.
[6]EQLs for PCB are; liquid =25 µg/L and solids = 7 µg/g, for each constituent requested.

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 per WHC-SD-TP-SARP-001, *Safety Analysis Report for Packaging (Onsite) Sample Pig Transport System,* 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.:

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

Directions for the analysis of specific tank samples for envelope analytes and other special instructions are provided in Table 5-1.

Table 5-1. Tank-Specific Analytical Exceptions

Tank	Exceptions
244-TX	Samples 244TX-00-1 through 244TX-00-3 shall be analyzed for PCBs for Process Control. Both the settled solids and decanted liquid shall be analyzed. Solid sample results are to be reported in dry weight.

5.2 CLARIFICATIONS AND ASSUMPTIONS

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).

$$Exotherm\ (dry\ weight) = \frac{[exotherm\ (wet\ weight) \times 100]}{(100 - \%\ water)}$$

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 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) are to be reported only if no additional preparatory work is required (e.g., running additional standards) and if the error associated with the results are documented. No reruns nor additional analyses should be performed to improve recovery for analytes not specifically requested in Tables 3-1 or 3-2.

5.3 PRIORITIES FOR COMPATIBILITY GRAB SAMPLES

In the event that the sample material recovered is insufficient to perform all the analyses prescribed by this SAP, the analyses have been prioritized (from highest to lowest priority) as follows:

1. Ammonia (for single-shell tank and double-contained receiver tank samples), hydroxide, ion chromatography, and pH.
2. Separable organics, DSC, and TGA
3. Specific gravity/bulk density
4. Volume percent solids, $^{235/238}U$, $^{239/240}Pu$, ^{241}Am, ICP/AES (Al, Fe, Cr, Mn, Ni)
5. TOC, ICP/AES (Na)
6. ^{90}Sr, ^{137}Cs
7. Other analytes listed in Tables 3-1 and 3-2.

6.0 ORGANIZATION

The organization and responsibility of key personnel involved with these tank characterization projects are listed in Table 6-1.

Table 6-1. Tank Project Key Personnel

Responsibility	Organization	Individual
Manager, Data Development and Interpretation	RPP Data Development and Interputation (CHG)	J. G. Field, 376-3753
Data Development and Interputation point of contact for Compatibility Samples	RPP Data Development and Interputation (CHG)	T. L. Lauricella, 373-6343
Process Control point of contact for Stabilization	RPP Process Control (CHG)	V. C. Boyles, 373-1321
Double-Shell Tank Farm Compatibility Program point of contact	Double-Shell Tank Farms: Engineering (CHG)	T. M. Blaak, 373-3880
Single-Shell Tank Farm Compatibility Program point of contact	SST Engineering (CHG)	R. E. Larson, 373-9100
Manager, Field Sampling	Characterization Project Operations (CHG)	J. F. Sickels, 373-0259
222-S Laboratory point of contact (day shift)	Hanford Analytical Laboratory Operations (FH)	R. K. Fuller (NHC), 373-1883
Analytical Programs & Client Integration	Hanford Analytical Laboratory Operations (FH)	K. L. Powell (FDH) 372-0939
222-S Laboratory point of contact (off hours)	Analytical Services (FH)	222-S Laboratory Shift Manager, 373-2435
Process Control point of contact for immediate notifications	RPP On-Call Process Engineer (CHG)	On-Call Process Engineer, Site Pager # 85-9654
Tank Farm Operations Shift Manager	Tank Farm Operations (CHG)	On-Duty Shift Manager, 373-2689

7.0 DELIVERABLES

All analyses will be reported as Format I, II, or IV as indicated in Tables 3-1 and 3-2. Additional information regarding reporting formats is given in Adams et al. (2000).

7.1 FORMAT I REPORTING

Tables 3-1 and 3-2 contain the notification limits for selected analytes. Any results exceeding their notification limits shall be reported via telephone to the Process Control On-Call Process Engineer and/or the appropriate 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 Process Control On-Call Process Engineer (and the Tank Farm Operations shift manager, if required), the RPP technical point of contact Data Development and Interpretation manager, and the On-Call Process Engineer for Compatibility Samples. Additional analyses for verification purposes may be contracted between the performing laboratory and Data Development and Interputation by either a revision to this SAP or by a letter.

7.2 FORMAT II REPORTING

A letter report documenting the results of the analyses shall be issued to the RPP Data Development and Interpretation manager, the tank coordinator responsible for the tank, the Process Control point of contact for Stabilization, and the point of contact for Tank Farm Compatibility Samples within 90 days of the receipt of the sample at the laboratory loading dock. If a turnaround time of less than 90 days is required for specific samples to meet tank farm needs, the turnaround time will be negotiated with Analytical Services and included in Section 5.0. The format II report is not required to contain supporting raw data, QC results, or associated analytical procedure numbers. However, the results require review and approval by the cognizant scientist or manager of the laboratory operation.

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 sample, including (as applicable) composites, solids, liquids, and associated blanks taken and analyzed for the sampling event. The recommended reporting format and the raw data that shall be included are given in detail in Section A5.0 of Adams et al. (2000). This data package shall be issued as a document approved for public release through the document control system within 120 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 the release of the data package. The electronic version shall be in the standard electronic format (Lang et al.1999).

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 of the Characterization Project. All significant changes (such as analysis of new samples) shall be documented by RPP Data Development and Interputation via an engineering change notice to this SAP or by a letter. All changes shall also be clearly documented in the final data report. The tank coordinator using a Characterization Change Notice (Figure 8-1) may make insignificant changes. 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.

Figure 8-1
CHARACTERIZATION CHANGE NOTICE

Document: _____

_____

Change Number: _____

ECN to TSAP Required? _____

Requestor: _____ **Date:** _____

Samples Impacted:

Proposed Change:

Reason for Change:

Date Change Effective:

Schedule Impact:

Authorization:

Tank Coordinator: _____ **Date:**_____

Project Coordinator: _____ **Date:**_____

222-S Client Services: _____ **Date:**_____

Other: _____ **Date:**_____

9.0 REFERENCES

Adams, M. R., J. W. Hunt, R. R. Thompson, 2000, *Letter of Intent for River Protection Project (RPP) Characterization Program: Process Engineering, Hanford Analytical Services, Characterization Project Operations and Quality Assurance,* RPP-5539, Rev. 0, CH2M HILL Hanford Group, Inc., Richland Washington.

Board, D. C., 1998, *Tank Waste Remediation System, Characterization Project, Quality Policies*, HNF-SD-WM-QAPP-025, Rev. 4, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

DOE, 1998, *Hanford Analytical Services Quality Assurance Requirements Document*, DOE/RL-96-68, Rev. 2, U.S. Department of Energy, Richland Field Office, Richland, Washington.

Fowler, K. D., 2000, *Tank Farm Waste Transfer Compatibility Program*, HNF-SD-WM-OCD-015, Rev. 3 CH2M HILL Hanford Group, Inc., Richland, Washington.

Kristofzski, J. G., 1996, *Directions for □ Opportunistic Analyses,□* (memorandum 75310-96-168 to J. H. Baldwin, et al., September 11), Westinghouse Hanford Company, Richland, Washington.

Lang, L. L., Bobrowski, S. F., and S. J. Harris, 1999, *Standard Electronic Format Specification for Tank Characterization Data Loader: Version 3.0*, HNF-3638, Rev. 1, prepared by Pacific Northwest National Laboratory for Lockheed Martin Hanford Corp., Richland, Washington.

CHG, 2000, *Nonconforming Item Reporting and Control*, RPP-PRO-298, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, Washington.

Markel, L. P., 2000, *222-S Laboratory Quality Assurance Plan*, HNF-SD-CP-QAPP-016, Rev. 4, Fluor Hanford, Inc., Richland, Washington.

Mulkey, C. H., M. Miller, and L. Jackson, 1999, *Data Quality Objectives for Tank Farms Waste Compatibility Program*, HNF-SD-WM-DQO-001, Rev. 3, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.