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ENGINEERING DATA TRANSMITTAL

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Tank 241-AP-104 Grab Sampling and Analysis Plan

Andrew M. Templeton

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Tank 241-AP-104 Grab Sampling and Analysis Plan

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CH2M HILL Hanford Group, Inc.

Date Published
October 2000

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Hanford Group, Inc.

Prepared for the U.S. Department of Energy
Office of River Protection

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LIST OF TERMS

AEA	alpha energy analysis
AMU	atomic mass unit
CHG	CH2M HILL Hanford Group, Inc.
CPO	Characterization Project Operations
CVAA	cold vapor atomic absorption
DQO	Data Quality Objective
DOE-RL	U.S. Department of Energy, Richland Operations Office
EDTA	ethylenediaminetriacetic acid
ft	feet
g/mL	grams per milliliter
GEA	gamma energy analysis
HEDTA	N-(hydroxyethyl)-ethylenediaminetiracetic acid
HLW	high-level waste
IC	ion chromatography
ICD	Interface Control Document
ICP	inductively coupled plasma
ICP/AES	inductively coupled plasma/atomic emission spectroscopy
ICP/MS	inductively coupled plasma/mass spectrometry
in.	inches
ISE	ion-specific electrode
kgal	kilogallon
kL	kiloliter
L	liter
L&H	low activity waste and high-level waste feed
LAW	low activity waste
LCS	laboratory control standard
LFL	lower flammability limit
m	meters
M	molar
Mgal	million gallons
mL	milliliter
MRQ	minimum reportable quantity
N/A	not applicable
QA	quality assurance
QC	quality control
RPD	relative percent difference
RPP	River Protection Project
RSD	relative standard deviation
SAP	sampling and analysis plan
SpG	specific gravity

LIST OF TERMS (Continued)

TBD	to be determined
TIC	total inorganic carbon
TOC	total organic carbon
TWRS	Tank Waste Remediation System
wt%	weight percent
%	percent
$\mu\text{Ci/g}$	microcuries per gram
$\mu\text{Ci/mL}$	microcuries per milliliter
$\mu\text{g C/g}$	micrograms carbon per gram
$\mu\text{g C/mL}$	micrograms carbon per milliliter
$\mu\text{g/g}$	microgram per gram
$\mu\text{g/mL}$	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 samples obtained from tank 241-AP-104. The purpose of this sampling event is to obtain information about the characteristics of the contents of 241-AP-104 required to provide sample material to the Waste Treatment Contractor in accordance with *Interface Control Document for Waste Treatability Samples* (Interface Control Document-23 [ICD-23]) (BNFL 2000) and to satisfy requirements of *Low Activity Waste and High-Level Waste Feed Data Quality Objectives* (L&H DQO) (Patello et al. 1999), and *Data Quality Objectives For RPP Privatization Phase I: Confirm Tank T Is An Appropriate Feed Source For Low Activity Waste Feed Batch X* (LAW DQO) (Nguyen 1999a). Adams et al. (2000a) lists these DQOs/issues as the only ones pertaining to this sample event.

Grab samples will be obtained from riser 001 to provide sufficient material for the chemical analyses and tests required to satisfy these data quality objectives and ICD-23. The 222-S Laboratory will receive samples; composite the samples; perform chemical analyses on composite samples; and provide samples to the Waste Treatment Contractor and the Process Chemistry Laboratory. The Process Chemistry Laboratory at the 222-S Laboratory Complex will perform process tests to evaluate the behavior of the 241-AP-104 waste undergoing the retrieval and treatment scenarios defined in the applicable DQOs. The Waste Treatment Contractor will perform process verification and waste form qualification tests. Requirements for analyses of samples originating in the L & H DQO process tests will be documented in the corresponding test plan (Person 2000) and are not within the scope of this SAP.

The following sections provide the general methodology and procedures to be used in the preparation, retrieval, transport, analysis, and reporting of results from grab samples retrieved from tank 241-AP-104.

2.0 SAMPLING EVENT REQUIREMENT

Tank 241-AP-104 is located in the AP Tank Farm in the 200 East Area. The tank is a 3,785,400-L (1 Mgal), 23-m (75-ft) diameter double-shell underground storage tank. As of October 25, 2000, the surface level ENRAF¹ indicated that tank 241-AP-104 contained a total waste volume of approximately 4,202 kL (1,110 kgal). This waste volume is equivalent to 403.75 inches of waste as measured from the inside bottom of the tank). Currently, it is unknown how much solids are contained in the tank. The tank contains waste transferred from tank 241-SY-102.

Table 1 identifies the number of samples, the riser to be used for sampling, and the elevations and depths at which the samples are to be obtained.

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 Interpretation within ten working days of the sampling event. 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 Interpretation 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.

2.1 SAMPLE COLLECTION

Four samples will be collected from each of five different waste depths from riser 001 using 500-mL bottles (total of twenty samples). If a different riser is necessary to meet sampling and analysis requirements, this change must be recorded and approved by the cognizant engineer and the tank coordinator (TC) before sampling. Table 1 provides the information for this sampling event. The exact time that each sample bottle is collected and the appearance of each sample (solids or no solids) shall be recorded on the chain-of-custody form.

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 in the work packages.

¹ ENRAF is a trademark of the ENRAF Corporation, Houston, Texas.

These work packages contain the operating procedures and the chain-of-custody records for the sampling events.

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.*

A measurement of the solids in tank is required prior to sampling. This may or may not be possible depending on the properties of the solids.

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 packages. The chain-of-custody forms for these work packages shall identify samples by a unique number and state the size of the sample for each sample before being shipped to the 222-S Laboratory. Pertinent sampling information (e.g., unusual waste characteristics, solids content, or detecting debris) *should be noted in the comment section of the chain-of-custody form.*

Characterization Project Operations should transport each sample on the same day it is collected or store the samples in a heated building (70 degrees F). A 500-mL sample bottle field blank is required during the sampling event. The laboratory shall provide the de-ionized water. The samples should be shipped to the performing laboratory within three 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 1. Tank Grab Sampling Information

Level	Sample Number	Expected Sample Type ²	Sample Location	Sample Elevation ¹	Sample Depth ¹ ft
#1	4AP-00-1B	Field Blank	Riser 001	400 in.	21' 1"(253 in)
	4AP-00-1	Liquid	Riser 001	379 in.	22' 6"(270 in.)
	4AP-00-2	Liquid		379 in	22' 6"(270 in.)
	4AP-00-3	Liquid		379 in	22' 6"(270 in.)
	4AP-00-4	Liquid		379 in	22' 6"(270 in.)
#2	4AP-00-5	Liquid	Riser 001	290 in.	29' 11"(359 in)
	4AP-00-6	Liquid		290 in	29' 11"(359 in)
	4AP-00-7	Liquid		290 in	29' 11"(359 in)
	4AP-00-8	Liquid		290 in.	29' 11"(359 in)
#3	4AP-00-9	Liquid	Riser 001	201 in.	37' 4"(448 in)
	4AP-00-10	Liquid		201 in	37' 4"(448 in)
	4AP-00-11	Liquid		201 in	37' 4"(448 in)
	4AP-00-12	Liquid		201 in .	37' 4"(448 in)
#4	4AP-00-13	Liquid	Riser 001	113 in.	44' 8"(536 in)
	4AP-00-14	Liquid		113 in.	44' 8"(536 in)

Table 1. Tank Grab Sampling Information

Level	Sample Number	Expected Sample Type ²	Sample Location	Sample Elevation ¹	Sample Depth ¹ ft
	4AP-00-15	Liquid		113 in .	44' 8"(536 in)
	4AP-00-16	Liquid		113 in..	44' 8"(536 in)
#5	4AP-00-17	Solids/Liquid	Riser 001	24 in.	52' 1"(625 in)
	4AP-00-18	Solids/Liquid		24 in .	52' 1"(625 in)
	4AP-00-19	Solids/Liquid		24 in .	52' 1"(625 in)
	4AP-00-20	Solids/Liquid		24 in .	52' 1"(625 in)
#6	4AP-00-21	Solids/Liquid	Riser 001	6 in ³	53' 7"(643 in)

Notes:

N/A = not applicable.

¹Sample elevation is defined as distance from tank bottom (Reference drawing H-14-010503, Tank Bottom Elevation 624 (ft) msl) to mouth of sample bottle; sample depth is defined as the distance from the top of the cover block to the mouth of the sample bottle.

²Shipping information:

¹³⁷Cs-⁹⁰Sr 3.2E-3 TBq/500ml bottle (Type A) for the supernatant samples.

¹³⁷Cs-⁹⁰Sr 3.2E-3 TBq/500ml bottle (Type A) for the solid/liquid samples

Sample activity is estimated based on the best basis inventory.

³Sample should be taken as close to the bottom of the tank as possible

1. A sludge measurement in the tank is required prior to sampling
2. CPO should transport each sample on the same day it is collected or store the samples in a heated building (70 degrees F). The samples are to be shipped to 222S Laboratory within three 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.

3.0 LABORATORY ANALYSIS REQUIREMENTS

The extrusion, sub-sampling, forming a composite, and analysis requirements are described below.

3.1 ANALYSIS SCHEME

Data quality objectives requirements drive the analysis of the samples. This section provides direction to the laboratory in regards to the sample handling and analysis scheme required to comply with the L&H and LAW DQOs and the sample handling required to comply with ICD-23.

Four sets of grab samples will be collected representing 5 elevations in the tank. One set (Samples 4AP-00-02, 06, 10, 14, 18) of 500-mL bottles will be used to support the requirements of the L&H DQO, and part of the LAW DQO. The second set 4AP-00-03, 07, 11, 15, 19 of 500-mL bottles will be archived for future support of the LAW DQO. The third and fourth sets (Samples 4AP-00-04, 05, 08, 09, 12, 13, 16, 17, 20, 21) of 500-mL bottles will be provided to the Waste Treatment Plant (WTP) Contractor to support the requirements of ICD-23. Record date and times of all steps.

- (1) 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 upon receipt and after 16 hours, if present. A consistent approach to this measurement shall be established. Video tape the samples at time of receipt.
- (2) Closely inspect the sample for the presence and approximate volume of any potential organic layers. Record the volume of separable organic phase, if present.
- (3) Package the third and fourth set of samples for shipment to the WTP contractor. Copies of the laboratory load in reports, sample chains-of-custody, and sample photographs associated with these samples must be provided to the Waste Treatment Contractor point of contact.
- (4) Archive the second set of samples for the LAW DQO.
- (5) Allow the remaining set of samples to settle for at least 16 hours. Record volume percent settled solids, and note any evidence of gas releases or separated liquid phases.
- (6) Prepare six separate tank composites as directed by the tank coordinator. These six composites will consist of liquid or slurry composites representative of the waste present in the grab samples.
- (7) Mix the composites and allow to settle for at least 16 hours. Record the total composite weight and volume % settled solids for each composite, and note evidence of any floating layer (an organic layer or solids) or gas generation.
- (8) Submit four of the composites to the Technology Project Management Group for

testing per L&H DQO. These tests will be performed per Person (2000).

- (9) Archive one of the remaining composites.
- (10) Separate the remaining composite by centrifugation. The responsible chemist shall ensure that the centrifuged solids contain no separate liquid phase. Each of these composites must contain sufficient material to perform the analyses in Tables 2 and 3. If insufficient solids are present to perform all the solids analyses in Table 3, refer to Section 5.0 for a priority of the analyses.
- (11) Analyze 3 sub-samples of the centrifuged solid fraction of each composite separated in step 10 per Table 3.
- (12) Analyze 3 sub-samples of the liquid fraction of each composite separated in step 10 per Table 2.
- (13) Analyze the field blank per Table 2.
- (14) Archive all remaining sample material.

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 2 and 3 and are described in Section 7.0.

A material balance shall be performed to account for all the material sampled and composited.

3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Tables 2 and 3 to be performed on the tank 241-AP-104 grab samples are based on the L&H and LAW DQOs. 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-549-142 for fusion digestion followed by water leaching; LA-505-158, or LA-505-163 for acid digestion of samples; LA-504-101 for water leach of solids; and LA-505-100 for acid digestion of solids and liquids for selenium analysis by graphite furnace atomic absorption (GFAA). The laboratory is to notify the tank coordinator once all analyses are complete.

Duplication of effort shall be avoided where practical. For example, many of the analyses required for the L&H DQO are also required by the LAW DQO. If process testing per the LAW DQO determines that dilution is not required for waste retrieval, analyses per the L&H DQO can meet the corresponding data needs of the LAW DQO. However, if process testing determines that dilution of the waste is required, then separate analyses of the diluted samples will be as specified in a test plan.

The L&H and LAW DQOs specify minimum reportable quantities (MRQs) for analytes listed in Tables 2 and 3. The laboratory shall notify the tank coordinator of any circumstances which prevent achieving detection limits at or below the L&H DQO's MRQ for any analyte, and recommend a corrective course of action. The MRQs for the requested analytes are provided in Tables 3 and 4; the HLW DQO's MRQs are provided in these tables for information only.

The L&H DQO requires reparation and/or reanalysis of a subsample under certain conditions. Repreparation and/or reanalysis of a subsample applies to Group 1 analytes whose relative standard deviation (RSD) exceeds the criteria in Section 4 and whose concentration is greater than the MRQ. In such circumstances, only one additional analysis is required, and the results of all analyses shall be reported. The decision to reanalyze a subsample or reprepare and reanalyze a new subsample shall be made by the project coordinator based on the previous results. If after reanalysis, the RSD of all subsamples exceeds the criteria, a decision will be made on the need for further analysis by the project coordinator, the tank coordinator and the Waste Integration Team (WIT) technical point of contact. Group 1 analytes, as defined by the L&H DQO for LAW and HLW are listed in Table 5.

3.3 INSUFFICIENT SAMPLE RECOVERY

If the amount of material recovered from samples taken from 241-AP-104 is insufficient to form the composites and perform the analyses requested in the SAP, the laboratory shall notify the tank coordinator within one working day. It is unknown how much solids are contained in the tank. Based upon the results of the sampling (amount of solids), a priority of the analyses and/or compositing scheme will 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*.

If the amount of centrifuged solids obtained from the composite solids is not sufficient to perform the analyses required by Table 3, analyses shall be performed in order of priority until all sample is consumed. Analytical priorities for the centrifuged solids, from highest priority to lowest, are listed below. For multi-analyte methods, all analytes required by Table 3 are requested. However, if material constraints require prioritization of the analytes, the highest priority analytes are also provided. The analyses should be performed on three subsamples as described in Section 3.1 unless there is insufficient sample to complete the first ten analytical methods listed below. If there is insufficient material, only two subsamples shall be analyzed.

If the same acid digest preparation is used for both inductively coupled plasma/atomic emission spectroscopy (ICP/AES) and inductively coupled plasma/mass spectrometry (ICP/MS), the analytical priorities are:

- (1) ICP/AES (Al, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, and U are highest priority analytes)
- (2) ICP/MS (Ba, K, La, and ^{99}Tc are highest priority analytes)
- (3) Gamma Energy Analysis (GEA) (^{154}Eu , ^{60}Co , and ^{137}Cs are highest priority analytes)
- (4) Total alpha
- (5) ^{99}Tc by separation and liquid scintillation and ^{90}Sr by separation and beta counting
- (6) ^{129}I by separation and GEA
- (7) Alpha Energy Analysis (AEA) (^{238}Pu , $^{239/240}\text{Pu}$, and ^{241}Am are highest priority AEA analytes)
- (8) Wt. % oxides
- (9) Ion Chromatography (IC)
- (10) Total inorganic carbon/total organic carbon (TIC/TOC) by furnace oxidation and persulfate oxidation
- (11) Hg
- (12) Weight percent solids
- (13) CN

If separate acid digest preparations are required for ICP/AES and ICP/MS, the priority of the ICP/MS analysis will be lowered to between TIC/TOC and Hg.

RPP-7266, REV. 0

Table 2: Tank 241-AP-104 Chemical, Radiological, and Physical Analytical Requirements: Liquids

LIQUID ANALYSES													
Project Name		241-AP-104		RPP-7266, Rev. 0		REPORTING LEVELS							
PROGRAM		PROGRAM CONTACTS		COMMENTS		FORMAT I							
A. Waste Disposal Division		PNNL CP Devel.		Homogenization Test - Per Laboratory Discretion		FORMAT II							
B. Waste Feed Delivery		J. H. Balkwin		Field Blank - Required		FORMAT III							
C. Process Control		RPP DD & I		Hot Cell Blank - Per Laboratory Discretion		FORMAT IV							
						FORMAT V							
						# of samples							
						2 Cores @ 4 segments ea.							
PRIMARY ANALYSES				QUALITY CONTROL ³			CRITERIA						
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SAMP ¹ LIQUID/ COMP.	PREP ² a/d/fw/e	SING/DUP/ TRIP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁵	FORMAT
A, B	Visual	Organic	LA-519-151	LIQUID	d	N/A	N/A	N/A	N/A	none	presence	unknown	I, IV
A, B	Gravimetry	SpG	LA-510-112	COMP	d	TRIP	N/A	N/A	ea AB	none	none	1.08	IV
A, B	Gravimetry	Wt % solids	LA-564-101	COMP	d	TRIP	N/A	N/A	ea AB	wt%	none	10	IV
A, B	ICP/AES	Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Eu, K, Fe, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, S, Sb, Se, Sr, Si, Th, Ti, Tl, U, V, Zn, Zr ⁵	LA-505-151 LA-505-161	COMP/FB	a	TRIP ⁶	1/mtr ⁷	ea PB	ea AB	µg/mL	As>11 Ba>33 Be> 3.8 Cd>70 Ca>1300 Ce>1800 Co>8.8 La>1000 Nd>5.6 Sb>6.4 Se>280 Tl>37 U>11000 V>2.1 Na>3.3E5	unknown	I, IV
A, B	ICP/MS	As, B, Ba, Be, Ce, Co, K, La, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ⁹⁰ AMU ⁸	LA-506-101	COMP	a	TRIP	1/mtr ⁷	ea PB	ea AB	µg/mL	As>11 Ba>33 Be> 3.8 Ce>1800 Co>8.8 La>1000 Sb>6.4 Se>280 Te>2.7 Tl>37 V>6.4	unknown	I, IV
A, B	ICP/MS	Cs, Eu ²³⁵ Np, ²⁴³ AMU, ²³⁵ U, ²³⁴ U, ²³⁶ U, ²³⁸ U, ²³¹ Pa, ²⁴² AMU, ¹²⁶ Sn, ⁹⁹ Tc, ²⁴¹ AMU ⁸	LA-506-101	COMP	a	TRIP	N/A	ea PB	N/A	µg/mL	none	unknown	IV
A, B	GFAA	Sb, Se	LA-505-102	COMP	d or a	TRIP	1/mtr ⁷	ea PB	ea AB	µg/mL	Sb>6.4 Se>280	unknown	I, IV
A, B	IC	NO ₂ , NO ₃ , PO ₄ , SO ₄ , Cl, F, formate, oxalate, acetate, citrate, NTA ⁹	LA-533-105 LA-533-115	COMP/FB	d	TRIP ⁶	1/mtr ⁷	ea PB	ea AB	µg/mL	[NO ₂], <0.01M see also note 10	unknown	I, IV
A, B	CVAA	Hg	LA-325-106	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	ISE	NH ₃ /NH ₄	LA-631-001	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	Pot. titration	OH ⁻	LA-211-102	COMP	d	TRIP	none	ea PB	ea AB	µg/mL	<0.01M see also note 10	unknown	I, IV
A, B	pH direct	pH	LA-212-106	COMP/FB	d	TRIP ⁶	none	none	ea AB	pH	<12	unknown	I, IV
A, B	Furnace oxidation	TOC, TC	LA-344-105	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µgC/mL	TOC >45,000 ^{11, 12}	unknown	I, IV
A, B	Ag catalyzed persulfate	TIC, TOC	LA-342-100	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µgC/mL	TOC >45,000 ^{11, 12}	unknown	I, IV
A, B	Distillation/ colorimetric	CN	LA-695-102	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µg/mL	>91	unknown	I, IV
A, B	Alpha counting	Total alpha	LA-508-101	COMP/FB	d or a	TRIP ⁶	1/mtr ⁷	ea PB	ea AB	µCi/mL	>61.5 ¹³	unknown	I, IV
A, B	Beta counting	Total beta	LA-508-101	COMP/FB	d or a	TRIP ⁶	1/mtr ⁷	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	Sep & beta count	⁹⁰ Sr	LA-220-101	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	Separation/AEA	²³⁸ , ^{239/240} Pu, ²⁴¹ Am, ^{243/244} Cm, ²⁴² Cm	LA-953-104	COMP	d	TRIP	1/mtr ⁷	ea PB	ea AB	µCi/mL	^{239/240} Pu>61.5 ¹³ ²³⁸ Pu>0.0481 ^{239/240} Pu>0.207 ²⁴¹ Am>0.919 ^{243/244} Cm > 0.033	unknown	I, IV

Table 2: Tank 241-AP-104 Chemical, Radiological, and Physical Analytical Requirements: Liquids

PRIMARY ANALYSES				QUALITY CONTROL ³				CRITERIA					
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SAMP ¹ LIQUID/ COMP.	PREP ² a/d/f/w/e	SING/DUP/ TRIP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	FORMAT
A, B	Sep/liquid scintillation	³ H	LA-218-114	COMP/FB	d	TRIP ⁵	1/mtrix	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	Sep/liquid scintillation	¹⁴ C	LA-348-104	COMP	d	TRIP	1/mtrix	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	Sep/liquid scintillation	⁷⁵ Se	LA-365-132	COMP	d or a	TRIP	N/A	ea PB	N/A	µCi/mL	none	unknown	IV
A, B	Sep/liquid scintillation	⁸⁶ Tc	LA-438-101	COMP	d or a	TRIP	see ¹⁵	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	Sep/liquid scintillation	⁹⁹ Tc (pertechnetate) ¹⁶	LA-438-101	COMP	d	TRIP	see ¹⁵	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	GEA	^{152,154,156} Eu, ¹³⁷ Cs, ⁶⁰ Co, ¹²⁵ Sb ¹⁷	LA-548-121	COMP/FB	d	TRIP ⁶	N/A ¹⁸	ea PB	ea AB	µCi/mL	¹³⁷ Cs>1580 ⁶⁰ Co>0.188 ¹⁵⁴ Eu>1.13	unknown	I, IV
A, B	Separation/GEA	¹²⁹ I	LA-378-103	COMP	d	TRIP	1/mtrix ¹⁴	ea PB	ea AB	µCi/mL	none	unknown	IV
SECONDARY ANALYSES				QUALITY CONTROL ³				CRITERIA					
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SAMP ¹ COMP/FB	PREP ² a/d/f/w/e	SING/DUP/ TRIP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	FORMAT
A	Capillary zone electrophoresis	EDTA, HEDTA ¹⁹	LA-533-113	COMP	d	TRIP	1/mtrix	ea AB	ea AB	µg /ml	none	unknown	IV
A	TBD	Organic speciation ¹⁹	TBD	COMP	d	TRIP	1/mtrix	ea AB	ea AB	µg/g	none	unknown	IV

¹Liquid = every bottle, COMP = centrifuged supernate from tank composite, FB = field blank²d = direct, f = fusion, a = acid, w = water, e = extraction³SING = single analysis, DUP = duplicate analyses, TRIP = triplicate subsamples analyzed singly, BLK = blank, LCS = laboratory control sample, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtrix = matrix, N/A = not applicable⁴Units for notification limits and expected range are those listed in the "units" column. Unless otherwise indicated, immediate notifications to be made to On-Call Process Engineer only.⁵CP/AES is not expected to meet MRQs for the following analytes: As, B, Ba, Ce, Co, K, La, Li, Mo, Sb, Se, Th, Ti, and V and may not meet the MRQs for Be.⁶Analyze field blank in duplicate.⁷Either serial dilutions or matrix spikes will be performed.⁸Analytical results for the following ICP/MS analytes will be semi-quantitative: Ba, Mo, Pd, Rb, Ru, Sb, Se, Te, and ²³¹Pa⁹Oxalate, formate, acetate, citrate, and NTA are secondary analyses; required only if the TOC is greater than 5,000 mg/L (5,000 µg/mL).¹⁰Immediate notification to be made to both the On-Call Process Engineer and Tank Farm Shift Manager if tank corrosion specifications are not met. The specifications are:a. For NO₃ ≤ 1.0 M: 0.010 M ≤ OH ≤ 8.0 M, 0.011 M < NO₂ < 5.5 M, and NO₃/(OH+NO₂) < 2.5 M.b. For 1.0 M < NO₃ ≤ 3.0 M: 0.1x NO₂ ≤ OH ≤ 10 M and OH + NO₂ ≥ 0.4 x NO₃c. For NO₃ > 3.0 M: 0.3 M ≤ OH < 10 M, OH + NO₂ ≥ 1.2 M, and NO₃ ≤ 5.5 M¹¹Dry weight basis¹²These 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 Data Development and Interpretation).¹³Immediate notification to be made to both the On-Call Process Engineer and Tank Farm Shift Manager.¹⁴Tracer or carrier may be used in place of a spike and corrected for recovery.¹⁵Every aliquot is spiked in order to calculate results. Additional spike analysis is not typically performed.¹⁶The radiochemical analysis for pertechnetate is to be performed on an unoxidized sample. That is, do NOT use a sample preparation that converts all Tc to the pertechnetate form.¹⁷An extended counting time in the presence of relatively high gamma activity may be required to achieve the reportable quantities for analytes other than ¹³⁷Cs¹⁸The measurement is a direct reading of the energy and the analysis is not affected by the sample matrix; therefore a matrix spike is not required¹⁹If TOC or organic from IC > 40,000 mg/L, analyze for chelator fragments by capillary zone electrophoresis, MeCl extraction/derivatization

GC/mass spectroscopy and ion-pair chromatography.

Table 3: Tank 241-AP-101 Chemical, Radiological, and Physical Analytical Requirements: Solids

SOLID ANALYSES													
Project Name	241-AP-104	Plan Number	RPP-7266, Rev. 0	COMMENTS		REPORTING LEVELS							
PROGRAM		PROGRAM CONTACTS		COMMENTS		FORMAT I	Immediate Notification						
A. Waste Disposal Division		PNNL CP Devel.		Homogenization Test - Per Laboratory Discretion		FORMAT II	Process Control						
B. Waste Feed Delivery		J. H. Baldwin		Field Blank - Required		FORMAT III	Safety Screening						
C. Process Control		RPP DD & I		Hot Cell Blank - Per Laboratory Discretion		FORMAT IV	Waste Management						
						FORMAT V	RCRA Compliance						
						# of samples							
						2 Cores @ 4 segments ea.							
PRIMARY ANALYSES				QUALITY CONTROL ³			CRITERIA						
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SAMP ¹	PREP ²	SING/DUP/ TRIP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	FORMAT
A	Centrifugation	Bulk density	LA-519-132	COMP	d	TRIP	N/A	N/A	N/A	g/ml	none	1.3 - 1.8	IV
A, B	Gravimetry	Wt% solids	LA-564-101	COMP	d	TRIP	N/A	N/A	ea AB	wt%	none	50	IV
A	Gravimetry	Wt % oxides @ 1050°C	see ⁸	COMP ⁷	d	TRIP	N/A	N/A	N/A	wt%	none	unknown	IV
A, B	ICP/AES	Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, S, Sb, Se, Si, Sr, Th, Ti, Tl, U, V, Y, Zn, Zr ⁹	LA-505-151 LA-505-161	COMP	f or a	TRIP	1/mtrix ⁹	ea PB	ea AB	µg/g	As>2800 Ba>2900 Be>120 Cd>12000 Ca>12000 Ce>1200 Co>2100 La>15000 Nd>3500 Sb>450 Se>10000 Tl>7400 U>21000 V>60 Na>2.37E5 Cr>74000	unknown	I, IV
A, B	ICP/MS	Ag, As, B, Be, Ce, Co, K, La, Li, Mo, Na, Nd, Pd, Pr, Rb, Rh, Ru, S, Sb, Se, Ta, Te, Th, Tl, V, W, Y, Zn, ⁹⁰ AMU ¹⁰	LA-506-101	COMP	f or a	TRIP	1/mtrix ⁹	ea PB	ea AB	µg/g	As>2800 Ba>2900 Be>120 Ce>1200 Co>2100 La>15000 Sb>450 Se>1000 Te>460 Tl>7400 V>60	unknown	I, IV
A, B	AA	Cs	TBD	COMP	f or a	TRIP	N/A	ea PB	N/A	µg/g	none	unknown	IV
A, B	ICP/MS	²³⁷ Np, ²⁴³ AMU, ²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁸ U, ²⁴¹ AMU, ²⁴² AMU, ¹²⁶ Sn, ⁹⁹ Tc, ¹⁵¹ AMU, ¹³⁵ Cs	LA-506-101	COMP	f or a	TRIP	1/mtrix	ea PB	ea AB	µg/g	²³⁷ Np>27.7 ²⁴¹ AMU>93.3	unknown	I, IV
A, B	IC	NO ₂ , NO ₃ , Cl, F, PO ₄ , SO ₄	LA-533-105 LA-533-115	COMP	w	TRIP	1/mtrix	ea PB	ea AB	µg/g	none	unknown	IV
A, B	CVAA	Hg	LA-325-106	COMP	d	TRIP	1/mtrix	ea PB	ea AB	µg/g	>6.0	unknown	I, IV
A, B,	GFAA	Sb, Se	LA-505-102	COMP	a	TRIP	1/mtrix	ea PB	ea AB	µg/g	Sb>450 Se>1000	unknown	I, IV
A, B	ISE/IC	NH ₃ /NH ₄	LA-631-001 LA-533-101	COMP	w	TRIP	1/mtrix	ea PB	ea AB	µg/g	>3300	unknown	I, IV
A, B	Furnace oxidation	TOC, TC	LA-344-105	COMP	w	TRIP	1/mtrix	ea PB	ea AB	µgC/g	TOC >45,000 ^{11, 12}	unknown	I, IV
A, B	Ag catalyzed persulfate	TOC, TIC	LA-342-100	COMP	d	TRIP	1/mtrix	ea PB	ea AB	µgC/g	TOC >45,000 ^{11, 12}	unknown	I, IV
A, B	Distillation/ colorimetric	CN	LA-695-102	COMP	w	TRIP	1/mtrix	ea PB	ea AB	µg/g	none	unknown	IV
A	Alpha counting	Total alpha	LA-508-101	COMP	f or a	TRIP	1/mtrix	ea PB	ea AB	µCi/g	>41 ¹³	unknown	I, IV
A	Beta counting	Total beta	LA-508-101	COMP	f or a	TRIP	1/mtrix	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	Sep & beta count	⁹⁰ Sr, ⁹⁰ Y	LA-220-101	COMP	f	TRIP	1/mtrix ¹⁴	ea PB	ea AB	µCi/g	>1250	unknown	I, IV

Table 3: Tank 241-AP-101 Chemical, Radiological, and Physical Analytical Requirements: Solids

PRIMARY ANALYSES				QUALITY CONTROL ³				CRITERIA					
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SAMP ¹ COMP	PREP ² a/d/f/w/e	SING/DUP/ TRIP	SPIKE	BLK	LCS	UNITS	NOTIFICATION LIMIT ⁴	EXPECTED RANGE ⁴	FORMAT
A, B	Separation/AEA	²³⁸ , ^{239/240} Pu, ²⁴¹ Am, ^{243/244} Cm, ²⁴² Cm	LA-953-104	COMP	f	TRIP	1/mtr ¹⁴	ea PB	ea AB	µCi/g	^{239/240} Pu>41 ¹³ ²³⁸ Pu>1.73 ^{239/240} Pu>38.4 ²⁴¹ Am>93.3 ²⁴⁴ Cm>0.384	unknown	I, IV
A, B	Sep/liquid scintillation	³ H	LA-218-114	COMP	w	TRIP	1/mtr ¹⁴	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	Sep/liquid scintillation	¹⁴ C	LA-348-104	COMP	w	TRIP	1/mtr ¹⁴	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	Sep/liquid scintillation	⁹⁹ Tc	LA-438-101	COMP	f	TRIP	see ¹⁵	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	GEA	^{152,154,155} Eu, ¹³⁷ Cs, ⁶⁰ Co, ¹²⁵ Sb ¹⁶	LA-548-121	COMP	f	TRIP	N/A ¹⁷	ea PB	ea AB	µCi/g	¹³⁷ Cs>1410 ⁶⁰ Co>0.531 ¹⁵⁴ Eu>7.21	unknown	I, IV
A, B	Separation/GEA	¹²⁹ I	LA-378-103	COMP	w	TRIP	1/mtr ¹⁴	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	Separation/GEA	¹²⁹ I	LA-378-103	COMP	f/w	TRIP	1/mtr ¹⁴	ea PB	ea AB	µCi/g	none	unknown	IV

¹COMP = centrifuged solids from tank composite.²d = direct, f = fusion, a = acid, w = water, f/w = fusion with water leach, e = extraction³SING = single analysis, DUP = duplicate analyses, TRIP = triplicate subsamples analyzed singly, BLK = blank, LCS = laboratory control sample, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtr = matrix, N/A = not applicable, NP = not performed⁴Units for notification limits and expected range are those listed in the "units" column. Unless otherwise indicated, immediate notifications to be made to On-Call Process Engineer only.⁵No procedure is available for w% oxides. Work will be performed to an approved test plan, HNF-5479.⁶Gravimetric wt%oxide is to be performed by heating at 1050°C. See L&H DQO section 7.3.4 for additional details⁷Weight percent oxides determination to be performed on both the solids composite prior to centrifuging and centrifuged solids.⁸CP/AES is not expected to meet MRQs for the following analytes: As, B, Ce, Co, Li, Mo, Sb, Se, Th, Ti, V, and Y and may not meet the MRQs for Ag, La, Na, Nd, S, U, and Zn.⁹Either serial dilutions or matrix spikes will be performed.¹⁰Analytical results for the following ICP/MS analytes will be semi-quantitative: Ag, Mo, Nd, Pd, Rb, Ru, Sb, Se, Te, and ²³¹Pa¹¹Dry weight basis¹²These 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 Data Development and Interpretation).¹³Immediate notification to be made to both the On-Call Process Engineer and Tank Farm Shift Manager.¹⁴Tracer or carrier may be used in place of a spike and results corrected for recovery.¹⁵Every aliquot is spiked in order to calculate results. Additional spike analysis is not typically performed.¹⁶An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ⁸⁰Co and ¹⁵⁴Eu, ¹⁵⁵Eu.¹⁷The measurement is a direct reading of the energy and the analysis is not affected by the sample matrix; therefore a matrix spike is not required

Table 4. Detection Limits and Minimum Reportable Quantities for LAW Liquids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ)	Units
Ag	ICP/AES	5.5E+00	1.7E+01	µg/mL
Al	ICP/AES	2.5E+01	7.5E+01	µg/mL
As	ICP/MS	7.5E-01	2.3E+00	µg/mL
B	ICP/MS	7.5E-01	2.3E+00	µg/mL
Ba	ICP/MS	7.5E-01	2.3E+00	µg/mL
Be	ICP/MS	7.5E-01	2.3E+00	µg/mL
Bi	ICP/AES	5.5E+01	1.7E+02	µg/mL
Ca	ICP/AES	5.0E+01	1.5E+02	µg/mL
Cd	ICP/AES	2.5E+00	7.5E+00	µg/mL
Ce	ICP/MS	7.5E-01	2.3E+00	µg/mL
Co	ICP/MS	7.5E-01	2.3E+00	µg/mL
Cr	ICP/AES	5.0E+00	1.5E+01	µg/mL
Cu	ICP/AES	5.5E+00	1.7E+01	µg/mL
Cs, total	ICP/MS	5.0E-01	1.5E00	µg/mL
Eu, total	ICP/MS	2.0E+01	6.0E+01	µg/mL
Fe	ICP/AES	2.5E+01	7.5E+01	µg/mL
Hg	CVAA	5.0E-01	1.5E+00	µg/mL
K	ICP/AES	2.5E+02	7.5E+02	µg/mL
La	ICP/MS	7.5E-01	2.3E+00	µg/mL
Li	ICP/MS	7.5E-01	2.3E+00	µg/mL
Mg	ICP/AES	5.5E+01	1.7E+02	µg/mL
Mn	ICP/AES	5.5E+00	1.7E+01	µg/mL
Mo	ICP/MS	7.5E-01	2.3E+00	µg/mL
Na	ICP/AES	5.5E+01	1.7E+02	µg/mL
Nd	ICP/AES	5.5E+01	1.7E+02	µg/mL
Ni	ICP/AES	1.0E+01	3.0E+01	µg/mL
P	ICP/AES	1.1E+02	3.3E+02	µg/mL
Pb	ICP/AES	9.9E+01	3.0E+02	µg/mL
Pd	ICP/AES	1.3E+02	3.9E+02	µg/mL
Pr	ICP/MS	7.5E-01	2.3E+00	µg/mL
Rb	ICP/MS	7.5E-01	2.3E+00	µg/mL
Rh	ICP/AES	6.0E+00	1.8E+01	µg/mL

Table 4. Detection Limits and Minimum Reportable Quantities for LAW Liquids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ)	Units
Ru	ICP/AES	1.2E+01	3.6E+01	µg/mL
S	ICP/AES	5.5E+01	1.7E+02	µg/mL
Sb	ICP/MS	7.5E-01	2.3E+00	µg/mL
Se	ICP/MS	7.5E-01	2.3E+00	µg/mL
Si	ICP/AES	3.0E+01	9.0E+01	µg/mL
Sr	ICP/AES	5.5E+00	1.7E+01	µg/mL
Ta	ICP/MS	7.5E-01	2.3E+00	µg/mL
Te	ICP/MS	7.5E-01	2.3E+00	µg/mL
Th	ICP/MS	7.5E-01	2.3E+00	µg/mL
Ti	ICP/AES	5.5E+00	1.7E+01	µg/mL
Tl	ICP/MS	7.5E-01	2.3E+00	µg/mL
U	ICP/AES	2.60E+02	7.8E+02	µg/mL
V	ICP/MS	7.5E-01	2.3E+00	µg/mL
W	ICP/MS	7.5E-01	2.3E+00	µg/mL
Y (⁹⁰ AMU)	ICP/MS	7.5E-01	2.3E+00	µg/mL
Zn	ICP/AES	5.5E+00	1.7E+01	µg/mL
Zr	ICP/AES	5.5E+00	1.7E+01	µg/mL
³ H	Liquid scint.	7.0E-03	2.1E-02	µCi/mL
¹⁴ C	Liquid scint.	2.4E-04	7.2E-04	µCi/mL
⁶⁰ Co	GEA	7.0E-04	2.1E-03	µCi/mL
⁷⁹ Se	Liquid scint.	3.0E-05	9.0E-05	µCi/mL
⁸⁹ Sr, ⁹⁰ Sr	Beta counting	1.0E-02	3.0E-02	µCi/mL
⁹⁹ Tc (total)	ICP/MS	5.0E-04	1.5E-03	µCi/mL
⁹⁹ Tc (pertechnetate)	NS	TBD	TBD	µCi/mL
¹²⁵ Sb	GEA	5.6E-01	1.7E+00	µCi/mL
¹²⁶ Sn	ICP/MS	2.0E-03	6.0E-03	µCi/mL
¹²⁹ I	GEA	5.8E-06	1.8E-05	µCi/mL
¹³⁷ Cs	GEA	1.3E-01	3.9E-01	µCi/mL
¹⁵² Eu	GEA	TBD	TBD	µCi/mL
¹⁵⁴ Eu	GEA	6.5E-03	2.0E-02	µCi/mL
¹⁵⁵ Eu	GEA	3.0E-02	9.0E-02	µCi/mL
²³¹ Pa	ICP/MS	TBD	TBD	µCi/mL
²³³ U	ICP/MS	1.4E-04	4.2E-04	µCi/mL

Table 4. Detection Limits and Minimum Reportable Quantities for LAW Liquids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ)	Units
²³⁴ U	ICP/MS	4.4E-05	1.2E-04	μCi/mL
²³⁵ U	ICP/MS	1.5E-08	4.5E-08	μCi/mL
²³⁶ U	ICP/MS	4.5E-07	1.4E-06	μCi/mL
²³⁷ Np	ICP/MS	1.3E-05	3.9E-05	μCi/mL
²³⁸ Pu	AEA	3.4E-03	1.0E-02	μCi/mL
²³⁸ U	ICP/MS	2.4E-09	7.2E-09	μCi/mL
²³⁹ Pu ²	AEA	3.4E-03	1.0E-02	μCi/mL
²⁴⁰ Pu ²	AEA	1.7E-02	5.1E-02	μCi/mL
²⁴¹ Pu	NS	NS	NS	μCi/mL
²⁴¹ Pu/Am (²⁴¹ AMU)	ICP/MS	1.6E+00	4.8E+01	μCi/mL
²⁴² Pu (²⁴² AMU)	ICP/MS	1.0E-02	3.0E-02	μCi/mL
²⁴¹ Am	AEA	1.0E-02	3.0E-02	μCi/mL
²⁴² Cm	NS	NS	NS	μCi/mL
²⁴³ Am (²⁴³ AMU)	ICP/MS	3.2E-03	9.6E-03	μCi/mL
²⁴³ + ²⁴⁴ Cm	AEA	5.0E-02	1.5E-01	μCi/mL
NH ₄ /NH ₃	ISE	4.5E+01	1.4E+02	μg/mL
Cl	IC	1.0E+02	3.0E+02	μg/mL
CN	Distillation/ colorimetric	1.5E+00	4.5E+00	μg/mL
F	IC	5.0E+01	1.5E+02	μg/mL
NO ₂	IC	7.5E+02	2.3E+03	μg/mL
NO ₃	IC	1.0E+03	3.0E+03	μg/mL
OH	Titration	2.5E+04	7.5E+04	μg/mL
oxalate	IC	6.0E+02	1.8E+03	μg/mL
PO ₄	IC	7.5E+02	2.3E+03	μg/mL
SO ₄	IC	7.7E+02	2.3E+03	μg/mL
total alpha	Prop. counting	7.5E-02	2.3E-01	μCi/mL
total beta	Beta counting	TBD	TBD	μCi/mL

Table 4. Detection Limits and Minimum Reportable Quantities for LAW Liquids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ)	Units
total inorganic carbon	Persulfate/ furnace oxidation	5.0E+01	1.5E+02	µg/mL
total organic carbon	Persulfate/ furnace oxidation	5.0E+02	1.5E+03	µg/mL

Notes:

AEA = alpha energy analysis

CVAA = cold vapor atomic absorption

GEA = gamma energy analysis

IC = ion chromatography

ICP/AES = inductively coupled plasma/atomic emission spectroscopy

ICP/MS = inductively coupled plasma /mass spectrometry

ISE = ion-specific electrode

liquid scint. = liquid scintillation

NS = not specified

prop. counting = proportional counting

TBD = to be determined

¹ The method is that which is listed in the DQO and may differ from the method used by the Laboratory for analysis.

² Combined analysis of ²³⁹Pu and ²⁴⁰Pu

Table 5. Detection Limits and Minimum Reportable Quantities for HLW Solids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity ²	Minimum Reportable Quantity (MRQ) ²	Units (per gram dried solids)
Ag	ICP/AES	3.0E+02 (5.0E+01)	9.0E+02	µg/g
Al	ICP/AES	1.2E+03 (1.1E+02)	3.6E+03 (3.3E+02)	µg/g
As	ICP/MS	2.0E+01 (1.0E+01)	6.0E+01 (3.0E+01)	µg/g
B	ICP/MS	1.0E+01 (1.0E+00)	3.0E+01 (3.0E+00)	µg/g
Ba	ICP/AES	2.0E+02	6.0E+02	µg/g
Be	ICP/MS	1.0E+01	3.0E+01	µg/g
Bi	ICP/AES	2.0E+03	6.0E+03	µg/g
Ca	ICP/AES	2.0E+03 (6.2E+01)	6.0E+03 (1.8E+02)	µg/g
Cd	ICP/AES	3.0E+02 (4.0E+00)	9.0E+02 (1.1E+01)	µg/g
Ce	ICP/MS	2.0E+00	6.0E+00	µg/g
Co	ICP/MS	2.0E+00 (1.0E+00)	6.0E+00 (3.0E+00)	µg/g
Cr	ICP/AES	4.0E+02 (4.0E+01)	1.2E+03 (1.2E+02)	µg/g
Cs	NS	NS ³ (2.0E+00)	NS ³ (6.0E+00)	µg/g
Cu	ICP/AES	2.0E+02 (6.5E+00)	6.0E+02 (1.8E+01)	µg/g
F	IC	2.5E+03	7.5E+03	µg/g
Fe	ICP/AES	4.0E+02 (5.0E+01)	1.2E+03 (1.4E+02)	µg/g
Hg	CVAA	5.0E-01	1.5E+00	µg/g
K	ICP/MS	2.0E+03 (5.0E+02)	6.0E+03 (1.5E+03)	µg/g
La	ICP/AES	1.0E+03 (2.0E+01)	3.0E+03 (6.0E+01)	µg/g
Li	ICP/MS	1.0E+01	3.0E+01	µg/g
Mg	ICP/AES	1.8E+03 (1.8E+02)	5.4E+03 (5.4E+02)	µg/g
Mn	ICP/AES	1.00E+02	3.0E+02	µg/g
Mo	ICP/MS	2.0E+00	6.0E+00	µg/g
Na	ICP/AES	1.8E+03 (5.0E+01)	5.4E+03 (1.5E+02)	µg/g
Nd	ICP/AES	1.0E+03 (2.6E+01)	3.0E+03 (7.7E+01)	µg/g
Ni	ICP/AES	6.0E+02 (5.5E+01)	1.8E+03 (1.6E+02)	µg/g
P	ICP/AES	2.0E+03 (2.0E+02)	6.0E+03 (6.0E+02)	µg/g
Pb	ICP/AES	1.2E+03 (2.0E+02)	3.6E+03 (6.0E+02)	µg/g
Pd	ICP/MS	1.0E+01 (1.0E+00)	3.0E+01 (3.0E+00)	µg/g
Pr	ICP/MS	2.0E+00	6.0E+00	µg/g
Pu	ICP/MS	2.0E+00 (8.0E+00)	6.0E+00 (2.4E+01)	µg/g
Rb	ICP/MS	2.0E+00	6.0E+00	µg/g
Rh	ICP/MS	2.0E+00	6.0E+00	µg/g
Ru	ICP/MS	4.0E+00 (1.0E+00)	1.2E+01 (3.0E+00)	µg/g
S	NS	NS ³ (4.0E+02)	NS ³ (1.2E+03)	µg/g
Sb	ICP/MS	4.0E+00	1.2E+01	µg/g
Se	ICP/MS	1.0E+02	3.0E+02	µg/g

Table 5. Detection Limits and Minimum Reportable Quantities for HLW Solids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity ²	Minimum Reportable Quantity (MRQ) ²	Units (per gram dried solids)
Si	ICP/AES	1.0E+04 (1.0E+03)	3.0E+04 (3.0E+03)	µg/g
Sr	ICP/AES	1.0E+02	3.0E+02	µg/g
Ta	ICP/MS	2.0E+00	6.0E+00	µg/g
Tc	NS	NS ³ (2.0E+00)	NS ³ (6.0E+00)	µg/g
Te	ICP/MS	6.0E+00 (2.0E+00)	1.8E+01 (6.0E+00)	µg/g
Th	ICP/MS	2.0E+00 (2.0E+02)	6.0E+00 (6.0E+02)	µg/g
Ti	ICP/AES	2.0E+02 (5.0E+01)	6.0E+02 (1.5E+02)	µg/g
Tl	ICP/MS	2.0E+00 (2.0E+02)	6.0E+00 (6.0E+02)	µg/g
U	NS	NS ³ (2.0E+02)	NS ³ (6.0E+02)	µg/g
V	ICP/MS	2.0E+00 (2.0E-02)	6.0E+00 (6.0E-02)	µg/g
W	ICP/MS	2.0E+00	6.0E+00	µg/g
Y	ICP/MS	2.0E+00 (9.0E+01)	6.0E+00 (2.7E+02)	µg/g
Zn	ICP/AES	4.0E+02 (2.0E+00)	1.2E+03 (6.0E+00)	µg/g
Zr	ICP/AES	2.0E+02	6.0E+02	µg/g
Cl	IC	7.5E+01	2.25E+02	µg/g
CN ⁻	CN ⁻ analysis	1.0E+00	3.0E+00	µg/g
CO ₃ ⁻²	NS	NS ³ (1.0E+01)	NS ³ (3.0E+01)	µg/g
NH ₃	ISE	2.0E+01	6.0E+01	µg/g
NO ₂ ⁻	IC	1.5E+02	4.5E+02	µg/g
NO ₃ ⁻	IC	1.5E+02	4.5E+02	µg/g
TOC	Persulfate/furnace oxidation	2.0E+01	6.0E+01	µg C/g
³ H	NS	NS (5.0E-03)	NS (1.5E-02)	µCi/g
¹⁴ C	Liquid scint.	2.0E-04 (6.0E-04)	6.0E-04 (1.8E-03)	µCi/g
⁶⁰ Co	GEA	4.0E-02 (4.0E-03)	1.2E-01 (1.2E-02)	µCi/g
⁹⁰ Sr ⁴	Beta counting	7.0E+00 (7.0E+01)	2.1E+01 (7.0E+01)	µCi/g
⁹⁰ Y ⁴	Beta counting	7.0E+00	2.1E+01	µCi/g
⁹³ Zr ⁵	Liquid scint.	2.0E-03	6.0E-03	µCi/g
^{93m} Nb (⁹³ AMU) ⁵	ICP/MS	4.0E+00	1.2E+01	µg/g
⁹⁹ Tc	ICP/MS	2.0E+00	6.0E+00	µg/g
¹²⁵ Sb ⁶	GEA	2.0E+00	6.0E+00	µCi/g
^{125m} Te ⁶	GEA	2.0E+00	6.0E+00	µCi/g
¹²⁶ Sn	Sep/GEA	6.0E-03 (2.0E-02)	1.8E-02 (6.0E-02)	µCi/g
¹²⁹ I	ICP/MS	1.0E+01	3.0E+01	µg/g
¹³⁵ Cs	ICP/MS	2.0E+00	6.0E+00	µg/g

Table 5. Detection Limits and Minimum Reportable Quantities for HLW Solids (4 Sheets)

Analyte	Method ¹	Estimated Quantitation Limit/Minimum Detectable Activity ²	Minimum Reportable Quantity (MRQ) ²	Units (per gram dried solids)
^{137m} Ba ⁷	GEA	3.0E-02	9.0E-02	μCi/g
¹³⁷ Cs ⁷	GEA	3.0E-02 (2.0E-02)	9.0E-02 (6.0E-02)	μCi/g
¹⁵¹ Sm (¹⁵¹ AMU)	Sep/β-LCS	NS ³	NS ³	μCi/g
¹⁵² Eu	GEA	2.0E+00	6.0E+00 (2.0E+00)	μCi/g
¹⁵⁴ Eu	GEA	1.0E-01 (2.0E-02)	3.0E-01 (6.0E-02)	μCi/g
¹⁵⁵ Eu	GEA	2.0E+00 (2.0E-02)	6.0E+00 (6.0E-02)	μCi/g
²³³ U	ICP/MS	2.0E-01 (2.0E+00)	6.0E-01 (6.0E+00)	μg/g
²³⁴ U	ICP/MS	2.0E+00	6.0E+00	μg/g
²³⁵ U	ICP/MS	2.0E+00	6.0E+00	μg/g
²³⁶ U	ICP/MS	2.0E+00	6.0E+00	μg/g
²³⁷ Np	ICP/MS	2.0E+00 (6.0E-01)	6.0E+00 (1.8E+00)	μg/g
²³⁸ Pu	Sep./AEA	2.0E-02 (2.0E-05)	6.0E-02 (6.0E-05)	μCi/g
²³⁸ U	ICP/MS	2.0E+00	6.0E+00	μg/g
²³⁹ Pu ⁸	Sep./AEA	2.0E-02 (2.0E+00 μg/g)	6.0E-02 (6.0E+00 μg/g)	μCi/g
²⁴⁰ Pu ⁸	Sep./AEA	2.0E-02	6.0E-02	μCi/g
²⁴¹ Am	Sep./AEA	6.0E-03 (4.0E-04)	1.8E-02 (1.2E-03)	μCi/g
²⁴¹ Am	GEA	2.0E+00	6.0E+00	μCi/g
²⁴² Cm ⁹	Sep./AEA	4.0E-03	1.2E-02	μCi/g
^{242m} Am ⁹	Sep./AEA	4.0E-03	1.2E-02	μCi/g
²⁴² Pu	ICP/MS	2.0E-01	6.0E-01	μg/g
²⁴² Pu (²⁴² AMU)	Sep./AEA	2.0E-02	6.0E-02	μCi/g
²⁴³ Am	Sep./AEA	2.0E-02	6.0E-02	μCi/g
²⁴³⁺²⁴⁴ Cm	Sep./AEA	4.0E-03 (2.0E-05)	1.2E-02 (6.0E-05)	μCi/g
Total alpha	Alpha counting	2.0E-01	6.0E-01	μCi/g
Total beta	Beta counting	7.0E+00	2.1E+01	μCi/g

Notes:

AEA = alpha energy analysis

CVAA = cold vapor atomic absorption

GEA = gamma energy analysis

IC = ion chromatography

ICP/AES = inductively coupled plasma/atomic emission spectroscopy

ICP/MS = inductively coupled plasma/mass spectrometry
ISE = ion-specific electrode
sep. = separation

¹The method is that which is listed in the DQOs and may differ from the method used by the Laboratory for analysis.

²Detection limits and MRQs for the L&H DQO. Where HLW DQO requirements differ from those listed, the corresponding HLW requirement is shown in parentheses. The HLW requirements are listed for information only.

³NS, not stated for the L&H DQO. In the event that testing shows that dilution is not required to meet waste transfer requirements, then the HLW MRQs apply. If process testing determines that dilution is required for transfer, requirements for analysis of the diluted waste will be as per the test plan.

⁴Combined analysis of ⁹⁰Sr and ⁹⁰Y

⁵Combined analysis of ^{93m}Nb and ⁹³Zr

⁶Combined analysis of ¹²⁵Sb and ^{125m}Te

⁷Combined analysis of ¹³⁷Cs and ^{137m}Ba

⁸Combined analysis of ²³⁹Pu and ²⁴⁰Pu

⁹Combined analysis of ²⁴²Am, ^{242m}Am, and ²⁴²Cm

Table 6. Group I Analytes (Patello et al. 1999) (2 sheets)

Analyte	LAW (liquids)	HLW (solids)
Al	X	
As		X
B		X
Ba	X	
Be		X
Ca	X	
Cd	X	
Ce		X
Cl	X	X
CN ⁻		X
Co		X
Cr	X	
Cs		X
Cu		X
F ⁻	X	
Fe	X	
Hg	X	X
K	X	
La	X	X
Li		X
Mn		X
Mo		X
Na	X	
Nd		X
NH ₃		X
Ni	X	
NO ₂ ⁻	X	X
NO ₃ ⁻	X	X
Pb	X	
PO ₄ ⁻³	X	
Pr		X
Pu		X
Rb		X
Sb		X
Se		X
SO ₄ ⁻²	X	
Sr		X
Ta		X
Tc		X
Te		X

Table 6. Group I Analytes (Patello et al. 1999) (2 sheets)

Analyte	LAW (liquids)	HLW (solids)
Th		X
TiC	X	X
Tl		X
TOC	X	X
U	X	
V		X
W		X
Y		X
Zn		X
³ H		X
¹⁴ C		X
⁶⁰ Co	X	X
⁹⁰ Sr	X	X
⁹⁹ Tc	X	X
¹²⁵ Sb		X
¹²⁶ Sn		X
¹²⁹ I		X
¹³⁷ Cs	X	X
¹⁵² Eu		X
¹⁵⁴ Eu	X	X
¹⁵⁵ Eu	X	X
²³³ U		X
²³⁵ U		X
²³⁷ Np	X	X
²³⁸ Pu	X	X
²³⁹ Pu	X	X
²⁴⁰ Pu	X	
²⁴¹ Pu	X	X
²⁴² Pu	X	
²⁴¹ Am	X	X
²⁴² Cm	X	
²⁴³ + ²⁴⁴ Cm	X	X
²⁴³ Am	X	

4.0 QUALITY ASSURANCE AND QUALITY CONTROL

Processes, services, activities, and conditions adverse to the 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 the disposition taken as required by the *Nonconformance Item Reporting and Control* (CHG 1999).

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-RL 1998) minimum requirements as the baseline for laboratory quality systems. The *222-S Laboratory Quality Assurance Plan* (Markel 2000) 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 1998) 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, 2, 6, and 7. The laboratory shall also use calibration and calibration check standards appropriate for the analytical instrumentation being used (see DOE-RL [1998] for definitions of QC samples and standards) or procedure/method specific QC criteria as applicable. 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 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.

Archive all unused sample portions. Table 7. Quality Control Parameters for Liquid Analysis (2 Sheets)

Analyte	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery	Spike %Recovery	Triplicate RSD ¹
Separable organic	Visual	N/A	N/A	N/A
Specific gravity	Gravimetry	NS	N/A	N/A
Wt% dissolved solids	Gravimetry	80 - 120%	N/A	<21%
Wt% oxide	Gravimetry	TBD	TBD	TBD
Ag, Al, As, B, Be, Ba, Bi, Ca, Cd, Ce, Cr, Cu, Eu, Fe, K, La, Li, Mg, Mn, Mo, Nd, Ni, P, Pb, S, Sb, Se, Sr, Si, Th, Ti, Tl, U, V, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ⁹⁰ AMU, ¹²⁶ Sn, ²³⁸ U, ²⁴¹ AMU, ²⁴² AMU, ⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cs, Eu	ICP/MS	NS	NS	NS
²³¹ Pa	ICP/MS	TBD	TBD	TBD
²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁷ Np, ²⁴³ AMU	ICP/MS	90 - 110%	75 - 125%	<15%
Br ⁻ , Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ⁻³ , SO ₄ ⁻² , formate, oxalate, acetate, citrate, NTA	IC	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
Sb, Se	GFAA	80 - 120%	75 - 125%	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH ⁻	Potentiometric titration	80 - 120%	N/A	<15%
TIC, TOC	Persulfate and furnace oxidation	80 - 120%	75 - 125%	<15%
CN ⁻	Distillation/colorimetric	80 - 120%	75 - 125%	<15%

Archive all unused sample portions. Table 7. Quality Control Parameters for Liquid Analysis (2 Sheets)

Analyte	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery	Spike %Recovery	Triplicate RSD ¹
Total alpha	Proportional counting	70 - 130%	70-130%	<15%
Total beta	Beta counting	70 - 130%	70 - 130%	<15%
⁹⁰ Sr	Separation/beta counting	75 - 125%	N/A	<15%
²³⁸ Pu, ^{239/240} Pu, ²⁴¹ Am, ²⁴² Cm, ^{243/244} Cm	Separation/AEA	NS	N/A	<15%
³ H	Separation/liquid scintillation	80 - 120%	NS	<15%
¹⁴ C	Separation/liquid scintillation	80 - 120%	75 - 125%	<15%
⁷⁹ Se	Liquid scintillation	NP	N/A	<15%
⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
⁹⁹ Tc, ⁹⁹ Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
⁶⁰ Co, ¹³⁷ Cs, ¹⁵² Eu, ¹⁵⁴ Eu, ¹⁵⁵ Eu	GEA	NS	N/A	<15%
¹²⁵ Sb	GEA	TBD	TBD	TBD
¹²⁹ I	Separation/GEA	NS	N/A	<15%

Notes:

AEA = alpha energy analysis
 AMU = atomic mass unit
 CVAA = cold vapor atomic absorption
 GEA = gamma energy analysis
 GFAA = graphite furnace atomic absorption
 IC = ion chromatography
 ICP/MS = inductively coupled plasma/mass spectrometry
 ICP/AES = inductively coupled plasma/atomic emission spectroscopy
 ISE = ion specific electrode
 LCS = laboratory control standard
 N/A = not applicable
 NS = not specified by DQO (Patello et al. 1999)
 TBD = to be determined
 TIC = total inorganic carbon
 TOC = total organic carbon
 RPD = relative percent difference
 RSD = relative standard deviation
 Wt% = weight percent

¹RSD requirement if the sample result is at least 10 times the instrument detection limit. $RSD = (\text{standard deviation of the mean} / \text{mean}) \times 100\%$

Table 8. Quality Control Parameters for Solids Analysis (2 Sheets)

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery	Spike % Recovery	Triplicate RSD ¹
Bulk density	Centrifugation	N/A	N/A	N/A
Wt% solids	Gravimetry	80 - 120%	N/A	<21%
Wt% oxide	Gravimetry	TBD	TBD	TBD
Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Cr, Cu, Fe, K, La, Mg, Mn, Li, Nd, Ni, P, Pb, S, Sb, Se, Si, Sr, Th, Ti, Tl, U, V, Y, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
Ag, As, B, Be, Ce, Co, K, La, Li, Mo, Nd, Pd, Pr, Rb, Rh, Ru, S, Sb, Se, Ta, Te, Th, Tl, V, W, Y, Zn, ⁹⁰ AMU	ICP/MS	80 - 120%	70 - 130%	<15%
Cs	ICP/MS	NS	NS	NS
²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁷ Np, ²⁴³ AMU	ICP/MS	90 - 110%	75 - 125%	<15%
⁹⁹ Tc, ¹²⁶ Sn, ¹³⁵ Cs, ¹⁵¹ AMU, ²³⁸ U, ²⁴¹ AMU, ²⁴² AMU	ICP/MS	80 - 120%	70 - 130%	<15%
⁹³ AMU	TBD	TBD	N/A	<15%
Br, Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ⁻³ , SO ₄ ⁻² , formate, oxalate	IC	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
Sb, Se	GFAA	80 - 120%	75 - 125%	<15%
OH	Potentiometric titration	80 - 120%	N/A	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC, TOC	Persulfate and furnace oxidation	80 - 120%	75 - 125%	<15%
CN ⁻	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
⁹⁰ Sr	Isotopic specific separation/beta counting	75 - 125%	N/A	<15%

Table 8. Quality Control Parameters for Solids Analysis (2 Sheets)

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery	Spike % Recovery	Triplicate RSD ¹
²³⁸ Pu, ^{239/240} Pu, ²⁴¹ Am, ²⁴² Cm, ^{243/244} Cm	Separation/AEA	NS	N/A	<15%
³ H	Separation/liq. Scintillation	80 - 120%	NS	<15%
¹⁴ C	Separation/liquid scintillation	80 - 120%	75 – 125%	<15%
⁹⁹ Tc	Separation/liquid scintillation	80 - 120%	70 – 130%	<15%
⁶⁰ Co	GEA	80 - 120%	N/A	<15%
¹³⁷ Cs, ¹⁵² Eu, ¹⁵⁴ Eu, ¹⁵⁵ Eu, ¹²⁵ Sb	GEA	NS	N/A	<15%
¹²⁹ I	Separation/GEA	NS	N/A	<15%

Notes:

AEA = alpha energy analysis
 CVAA = cold vapor atomic absorption
 GEA = gamma energy analysis
 GFAA = graphite furnace atomic absorption
 IC = ion chromatography
 ICP/AES = inductively coupled plasma/atomic emission spectroscopy
 ICP/MS = inductively coupled plasma/mass spectrometry
 ISE = ion specific electrode
 LCS = laboratory control standard
 N/A = not applicable
 NS = not specified
 RPD = relative percent difference
 RSD = relative Standard Deviation
 TBD = to be determined
 Wt% = weight percent

¹RSD requirement if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation /mean) x 100%

4.2 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

The L&H DQO requests analysis of both solid and liquid fractions from static Phase 1 candidate feed source tanks. Tank 241-AP-104 is assigned as a LAW feed candidate but the solids remaining after dissolution may be considered HLW; therefore the solids will be analyzed against the HLW section of the L & H DQO.

As understood by the customer the analyses of the liquids for volatile organic compounds as stated in the *Data Quality Objectives for Regulatory Requirements for Hazardous and Radioactive Air Emissions Sampling and Analysis* (Air DQO) (Mulkey 1999) will not be performed.

The L&H DQO requires full dissolution of solids samples for analysis. However, some of the analyses in Table 2 can only be performed on water digests of solids; full dissolution of the sample for analysis is not currently possible for these analytes. These analytes are: TOC (and total carbon) by furnace oxidation, anions by IC, ammonia, tritium, carbon-14, and iodine-129. A fusion digestion/water leach preparation has recently been developed at the 222-S Laboratory and, if implemented, may be used for the iodine-129 analysis.

Many of the analyses performed in triplicate as directed by the L&H DQO are also required in duplicate by the HLW DQO. Per customer request, duplication of effort is to be avoided by performing these analyses in triplicate as directed by the L&H DQO to satisfy the requirements of the HLW DQO in the event that dilution is not required. Tables 2 and 7 reflect compromises between these DQO requirements to satisfy the affected programs. Several analytes being measured by ICP/MS required by the L&H DQO will take the place of ICP/AES analyses for solids (As, B, Be, Ce, Co, Cs, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V) required by the HLW DQO.

The tables identify many analytes to be determined by ICP/MS because this technique has the sensitivity to meet the desired MRQs. However if the concentration of the analyte is high enough, the quality of the ICP/AES results will be as good as or better than the ICP/MS data. Because ICP/MS is based on the measurement of different element isotopes, the total amount of an element must be determined by summing the isotopes or by calculations assuming the natural abundance of the isotopes. However, in the production of nuclear materials these natural abundances are changed, particularly around the mass peaks of the fission product yield curves (AMU-90 and -137). The presence of the fission products in the samples can lead to unnatural isobaric interference that can lead to inaccuracies in the measurements. Another potential interference to the ICP/MS method is in the low (<80 AMU) atomic mass range where polyatomic species and ionized species from the argon plasma gas can cause interference problems. Elements in this region may be determined easier and more accurately using ICP/AES if the concentrations in the sample are high enough. Because it is not possible to precisely predict what trace analytes will be present in high enough concentrations for ICP/AES analysis, ICP/AES and ICP/MS are being requested for all possible analytes. For liquid samples, the following elements cannot be analyzed by ICP/AES at the required MRQs: As, B, Ba, Be

(borderline), Ce, Co, K, La, Li, Mo, Sb, Se, Th, Tl, and V. For solid samples the following elements cannot be analyzed by ICP/AES at the required MRQs: Ag (borderline), As, B, Ce, Co, La (borderline), Li, Mo, Na (borderline), Nd (borderline), S (borderline), Sb, Se, Th, Tl, U (borderline), V, Y, and Zn (borderline). The following analytes are expected to be semi-quantitative for the ICP/MS method: Ag, Ba, Mo, Nd, Pd, Rb, Ru, Sb, Se, Te, and ^{231}Pa . Sb and Se can be performed by graphite furnace atomic absorption (GFAA) and are, therefore, requested by that method.

Spikes and standards are not available for all the ICP/MS analytes. Pre- and post-digestion standards and spikes are analyzed by ICP/MS for the following elements and isotopes:

- Solids: As, Be, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Tl, V, W, ^{238}U , and ^{232}Th
- Liquids: As, Ba, Be, Ce, Co, Eu, La, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Tl, V, W, ^{238}U , and ^{232}Th .
- Only post-digestion standards and spikes are used for the following:
 - Solids: ^{239}Pu , ^{237}Np , ^{241}Am , ^{117}Sn , ^{99}Tc , ^{133}Cs , ^{93}AMU , and ^{151}AMU
 - Liquids: ^{239}Pu , ^{237}Np , ^{241}Am , ^{117}Sn , ^{99}Tc , ^{133}Cs , and ^{90}AMU .

The 222-S laboratory does not have analytical methods for ^{59}Ni , ^{63}Ni , ^{126}Sb , $^{126\text{m}}\text{Sb}$, $^{121\text{m}}\text{Sn}$, ^{93}Nb , or ^{93}Zr . The 222-S laboratory has indicated that it is unlikely it can meet the <3.5% RSD for Na.

5.1 CLARIFICATIONS AND ASSUMPTIONS

The laboratory is requested to report all analytical results recovered from multi-analyte methods, including 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 associated QC results (blanks, standards, and spikes, as appropriate) are reported. No reruns or additional analyses should be performed to improve recovery for analytes not specifically requested in Tables 1 or 2.

6.0 ORGANIZATION

The organization and responsibility of points of contact and key personnel involved with this tank 241-AP-104 characterization project are listed in Table 8.

Table 9. Tank 241-AP-104 Project Points of Contact and Key Personnel List

Responsibility	Organization	Individual
RPP 241-AP-104 Tank Coordinator	RPP Data Development and Interpretation (CHG)	A. M. Templeton, 373-5589
222-S Project Coordinator for 241-AP-104	Analytical Production (FH)	R. A. Esch, 373-4314
222-S Laboratory Point of Contact (day shift)	Analytical Services (FH)	R. K. Fuller, 373-1883
222-S Laboratory Point of Contact	Analytical Services (FH)	222-S Laboratory Shift Manager, 373-2435
200 East Tank Farm Point of Contact	Tank Farm Operations (CHG)	East Tank Farm Operations Shift Manager, 373-2689
Grab Sampling Manager	Characterization Project Operations (CPO)	J. F. Sickels, 373-0259
Grab Sampling Cognizant Engineer	Characterization Project Operations (CPO)	R G. Brown, 373-5694
Data Management	Manager, Data Development and Interpretation (CHG)	J. G. Field, 376-3753
Process Control Point of Contact for Immediate Notifications	RPP Process Control (CHG)	On-Call Process Engineer, 85-9654 (pager)
Technology Project Management Point of Contact	Acting Manager, Technology Project Management (NHC)	W. I. Winters, 373-1951
WFD Point of Contact	Retrieval Engineering (CHG)	J. H. Baldwin, 373-4533
Waste Treatment Contractor Point of Contact	Tank Waste Treatment Operations Project (CHG)	M. E. Johnson, 371-3621
WIT Technical Point of Contact	PNNL	I. E. Burgeson, 372-3650
Field Operations Interface	RPP CPO Requirements Planning and Support (CHG)	J. G. Douglas, 372-0989

Notes:

CHG = CH2M HILL Hanford Group, Inc.

FH = Fluor Hanford, Inc.

NHC = Numatec Hanford Corporation

PNNL = Pacific Northwest National Laboratory

WFD =Waste Feed Delivery

WIT = Waste Integration Team

7.0 DELIVERABLES

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

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 Analytical Production Team to the On-Call Process Engineer and/or the East 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 River Protection Project (RPP) Data Development and Interpretation manager, the On-Call Process Engineer, 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 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) formation of composites, 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 Section A5.0 of Adams et al. (2000b). The data package shall be issued within 150 days from the date 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. The data package shall be released via an engineering data transmittal and a copy shall be delivered to the Tank Characterization and Safety Resource Center in compact disk read-only memory format if available.

In addition to this data package, an electronic version of the analytical results shall be provided to the Tank Characterization Database following issuance of the final data package. The data must be available to the Washington State Department of Ecology via the Tank Characterization Database within seven (7) calendar days of 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 or the Characterization Project. All significant changes shall be documented by RPP Data Development and Interpretation via an Engineering Change Notice to this SAP, by a letter, or on the Characterization Change Notice form (see Figure 1). Characterization Change Notices may also be initiated by the 222-S Laboratory. 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 the extrusion logbook or memorandum to file). Significance is determined by the tank coordinator.

Additional analysis of sample material from this characterization project performed at the request of the Characterization Program shall be performed following a revision of this SAP or issuance of a letter.

Figure 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

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