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ENGINEERING CHANGE NOTICE

Page 1 of 2

1. ECN 655005

Proj. ECN

2. ECN Category (mark one)	3. Originator's Name, Organization, MSIN, and Telephone No.	4. USQ Required?	5. Date
Supplemental [] Direct Revision [X] Change ECN [] Temporary [] Standby [] Supersedure [] Cancel/Void []	Juergen H. Rasmussen, Data Development and Interpretation, R2-12, 373-1128	[] Yes [X] No	07/30/99
	6. Project Title/No./Work Order No.	7. Bldg./Sys./Fac. No.	8. Approval Designator
	Tank 241-AZ-102	N/A	Q
	9. Document Numbers Changed by this ECN (includes sheet no. and rev.)	10. Related ECN No(s).	11. Related PO No.
	HNF-4577, Rev. 2	ECNs: 654977, 654981, 654986	N/A

12a. Modification Work	12b. Work Package No.	12c. Modification Work Complete	12d. Restored to Original Condition (Temp. or Standby ECN only)
[] Yes (fill out Blk. 12b) [X] No (NA Blks. 12b, 12c, 12d)	N/A	N/A Design Authority/Cog. Engineer Signature & Date	N/A Design Authority/Cog. Engineer Signature & Date

13a. Description of Change

13b. Design Baseline Document? [] Yes [X] No

This revision descopes safety screening and flammable gas DQO requirements from this sampling event. These changes affect the sample analysis scheme and ripple throughout the document.

14a. Justification (mark one)

Criteria Change	[X]	Design Improvement	[]	Environmental	[]	Facility Deactivation	[]
As-Found	[]	Facilitate Const	[]	Const. Error/Omission	[]	Design Error/Omission	[]

14b. Justification Details

This tank sampling and analysis plan is descoped to reflect an evaluation which determined that safety screening requirements have already been met for this tank and that the Flammable Gas DQO will no longer be applied to the condensed phases of this tank.

15. Distribution (include name, MSIN, and no. of copies)

See attached distribution.

RELEASE STAMP
AUG 02 1999
DATE
STA 4
HANFORD RELEASE
ID: 4

A-7900-013-2 (05/96) GEF095

A-7900-013-1

ENGINEERING CHANGE NOTICE

1. ECN (use no. from pg. 1) ECN-655005

16. Design Verification Required	17. Cost Impact				18. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[ ] Yes	Additional	[ ] $	Additional	[ ] $	Improvement	[ ]
[X] No	Savings	[ ] $	Savings	[ ] $	Delay	[ ]

19. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 13. Enter the affected document number in Block 20.

SDD/DD	[ ]	Seismic/Stress Analysis	[ ]	Tank Calibration Manual	[ ]
Functional Design Criteria	[ ]	Stress/Design Report	[ ]	Health Physics Procedure	[ ]
Operating Specification	[ ]	Interface Control Drawing	[ ]	Spares Multiple Unit Listing	[ ]
Criticality Specification	[ ]	Calibration Procedure	[ ]	Test Procedures/Specification	[ ]
Conceptual Design Report	[ ]	Installation Procedure	[ ]	Component Index	[ ]
Equipment Spec.	[ ]	Maintenance Procedure	[ ]	ASME Coded Item	[ ]
Const. Spec.	[ ]	Engineering Procedure	[ ]	Human Factor Consideration	[ ]
Procurement Spec.	[ ]	Operating Instruction	[ ]	Computer Software	[ ]
Vendor Information	[ ]	Operating Procedure	[ ]	Electric Circuit Schedule	[ ]
OM Manual	[ ]	Operational Safety Requirement	[ ]	ICRS Procedure	[ ]
FSAR/SAR	[ ]	IEFD Drawing	[ ]	Process Control Manual/Plan	[ ]
Safety Equipment List	[ ]	Cell Arrangement Drawing	[ ]	Process Flow Chart	[ ]
Radiation Work Permit	[ ]	Essential Material Specification	[ ]	Purchase Requisition	[ ]
Environmental Impact Statement	[ ]	Fac. Proc. Samp. Schedule	[ ]	Tickler File	[ ]
Environmental Report	[ ]	Inspection Plan	[ ]		[ ]
Environmental Permit	[ ]	Inventory Adjustment Request	[ ]		[ ]

20. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision	Document Number/Revision	Document Number Revision
N/A		

21. Approvals

Signature	Date	Signature	Date
Design Authority		Design Agent	
Cog. Eng. J.H. Rasmussen	7/30/99	PE	
Cog. Mgr. K.M. Hall	7/30/99	QA	
QA J.S. Sparks	7/30/99	Safety	
Safety		Design	
Environ.		Environ.	
Other D.B. Hardy	7/30/99	Other	
		DEPARTMENT OF ENERGY	
J.S. Lee	7-30-99	Signature or a Control Number that tracks the Approval Signature	
		ADDITIONAL	

Tank 241-AZ-102 Privatization Push Mode Core Sampling and Analysis Plan

Juergen H. Rasmussen
Lockheed Martin Hanford, Corp., Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-96RL13200

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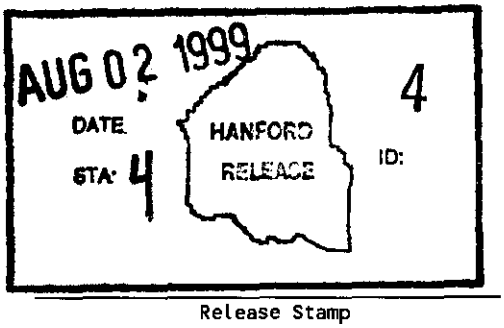
Abstract: N/A



8/2/99
Release Approval Date

Release Stamp

Approved for Public Release

RECORD OF REVISION	(1) Document Number HNF-4577	Page 1

(2) Title
Tank 241-AZ-102, Privatization Push Mode Core Sampling And Analysis Plan.

CHANGE CONTROL RECORD

(3) Revision	(4) Description of Change - Replace, Add, and Delete Pages	Authorized for Release	
		(5) Cog. Engr.	(6) Cog. Mgr. Date
0	(7) Initially released 06/04/99 on EDT-611470.	A. M. Templeton	J.W. Hunt
RS 0-A	Incorporate per ECN-654977.	A. M. Templeton	K. M. Hall 6/14/99
1 RS	Incorporate per ECN-654981.	A.M. Templeton	K.M. Hall Kathleen McDowell 6/24/99
2 RS	Incorporate per ECN-654986.	A.M. Templeton	K.M. Hall for K.M. Hall 7/7/99
3 RS	Incorporate per ECN-655008.	J.H. Rasmussen	K.M. Hall for K.M. Hall 7/30/99

HNF-4577
Revision 3

Tank 241-AZ-102 Privatization Push Mode Core Sampling and Analysis Plan

J. H. E. Rasmussen
Lockheed Martin Hanford Corporation

Prepared for the Office of River Protection

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LIST OF ABBREVIATIONS

AEA	alpha energy analysis
ALC	air lift circulator
AMU	Atomic Mass Unit
Ci	curie
CPO	Characterization Project Operations
CVAA	cold vapor atomic absorption
DQO	data quality objective
DSC	differential scanning calorimetry
g	gram
g/L	gram per liter
GEA	gamma energy analysis
GPC	gas flow proportional counter
HLW	high level waste
IC	ion chromatography
ICP/AES	inductively coupled plasma - atomic emission spectroscopy
ICP/MS	inductively coupled plasma mass spectroscopy
ISE	ion-specific electrode
kgal	kilogallon
kL	kiloliter
L	liter
LAW	low activity waste
LCS	laboratory control standard
LFL	lower flammability limit
LiBr	lithium bromide
LMHC	Lockheed Martin Hanford Corporation
LSC	liquid scintillation
MDA	minimum detectable activity
mL	milliliter
MRQ	minimum reportable quantity
MSL	mean sea level
N/A	not applicable
NA	not available
NCAW	neutralized current acid waste
NHC	Numatec Hanford Corporation
ORP	Office of River Protection
PIC	person in charge
PNNL	Pacific Northwest National Laboratory
PRSST	Propagating Reactive System Screening Tool
QA	quality assurance
QC	quality control
RPP	River Protection Project
RSD	relative standard deviation
RSST	Reactive System Screening Tool

SAP	sampling and analysis plan
Sep	separation required
TBD	to be determined
TGA	thermogravimetric analysis
TIMS	thermal ionization mass spectroscopy
TOC	total organic carbon
TRU	transuranic
TWRS	Tank Waste Remediation System
WAPS	Waste Acceptance Product Specifications
WMH	Waste Management Hanford
wt%	weight percent
µCi	microcurie
µCi/g	microcurie per gram
µCi/mL	microcuries per milliliter
µg/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-AZ-102. The purpose of this sampling event is to obtain information about the characteristics of the contents of 241-AZ-102 required to satisfy the *Data Quality Objectives For TWRS Privatization Phase 1: Confirm Tank T Is An Appropriate Feed Source For High-Level Waste Feed Batch X* (HLW DQO) (Nguyen 1999a), *Data Quality Objectives For TWRS Privatization Phase 1: Confirm Tank T Is An Appropriate Feed Source For Low-Activity Waste Feed Batch X* (LAW DQO) (Nguyen 1999b), *Low Activity Waste and High Level Waste Feed Data Quality Objectives* (L&H DQO) (Patello et al. 1999) and *Characterization Data Needs for Development, Design, and Operation of Retrieval Equipment Developed through the Data Quality Objective Process* (Equipment DQO) (Bloom 1996). The *Tank Characterization Technical Sampling Basis* document (Brown et al. 1998) indicates that these issues, except the Equipment DQO apply to tank 241-AZ-102 for this sampling event. The Equipment DQO is applied for shear strength measurements of the solids segments only. Poppiti (1999) requires additional americium-241 analyses of the sludge segments. Brown et al. (1998) also identify safety screening, regulatory issues and provision of samples to the Privatization Contractor(s) as applicable issues for this tank. However, these issues will not be addressed via this sampling event. Reynolds et al. (1999) concluded that information from previous sampling events was sufficient to satisfy the safety screening requirements for tank 241-AZ-102.

Push mode core samples will be obtained from risers 15C and 24A to provide sufficient material for the chemical analyses and tests required to satisfy these data quality objectives. The 222-S Laboratory will extrude core samples, composite the liquids and solids, perform chemical analyses, and provide subsamples to the Process Chemistry Laboratory. The Process Chemistry Laboratory will prepare test plans and perform process tests to evaluate the behavior of the 241-AZ-102 waste undergoing the retrieval and treatment scenarios defined in the applicable DQOs. Requirements for analyses of samples originating in the process tests will be documented in the corresponding test plan.

2.0 SAMPLING EVENT REQUIREMENTS

As of June 21, 1999, surveillance readings indicated that tank 241-AZ-102 contained a total waste volume of approximately 3,487 kL (921 kgal), consisting of 3,091 kL (817 kgal) of supernate and 396 kL (104 kgal) of sludge (sludge volume per Hanlon 1999). This waste volume is equivalent to 8.51 meters (335 inches) of waste as measured from the inside bottom of the tank. A physical profile prediction based on waste fill history and previous sampling information is provided in Appendix A. Previous characterization data indicate that the waste consists primarily of aging waste (Schreiber 1995). Air Lift Circulator (ALC) operation prior to this sampling event may result in suspension of solids in the liquid layer. Because the settling behavior of these solids is not well known, the entire liquid layer must be represented by the full core samples.

Prior to core sampling, the dome space (below the riser) shall be measured for the presence of flammable gases. The measurement shall be taken from within the dome space and the data

reported as a percentage of the lower flammability limit (LFL). The results shall be transmitted to the tank coordinator within ten working days of the sampling event (Schreiber 1998). If the dome space results are above 10 percent of the LFL when analyzing with a combustible gas meter or above 25 percent of the LFL when analyzing by gas chromatography/mass spectrometry or gas-specific monitoring, the necessity for recurring sampling for the flammable gas concentrations and the frequency of such sampling will be determined by the Flammable Gas Safety Program.

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

Tank 241-AZ-102 will be push mode core sampled using a push or rotary mode core sampling system. Two core samples, consisting of 18 segments each, are expected to be taken from two 6-inch diameter risers 15C, and 24A. The sampling objective is to obtain two full vertical profiles of the waste; therefore, additional segments may need to be taken depending on the accuracy of the current waste volume records in comparison to pre-sampling zip cord readings.

Universal zero-flow samplers will be used for these samples. The lower 2 segments of each core shall be x-rayed to ensure adequate solids recovery. If quality-affecting changes to the sampling requirements must be made (including the risers, sampling truck, or segments to be sampled), the change must be recorded and approved by the cognizant engineer and tank coordinator before sampling. This information may be recorded on a permanent data sheet or recorded directly in sampling work packages ES-99-00070 for riser 15C and ES-99-00071 for riser 24A. These work packages contain the operating procedures and the chain-of-custody records for this sampling event.

One field blank for tank 241-AZ-102 shall be obtained in accordance with procedure TO-060-003. The Characterization Project Operations (CPO) person in charge (PIC) or the PIC designate will verify that the field blank is properly created and shipped. For sampling events having multiple PICs, CPO shall determine which PIC will be responsible for the field blank. This field blank is to accompany the samples to the laboratory. All collected samples shall be shipped to the laboratory following the Load/Transport Sample Cask(s) procedure (TO-080-090). Core samples should be transported to the laboratory within three calendar days from the time each segment is removed from the tank.

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

by the LiBr solution, these data will be used to determine the amount of contamination. If more than one batch of LiBr solution is used during sampling event, one solution sample must be provided for each batch in addition to the field blank.

3.0 LABORATORY ANALYSIS REQUIREMENTS

The extrusion, subsampling, compositing, and analysis requirements are described below. **For core samples from tank 241-AZ-102, the shipping container must be vented every 27 days to release any accumulated gas.**

3.1 ANALYSIS SCHEME

Data quality objectives requirements drive the analysis of the samples. In order to comply with the LAW, HLW, L&H, and Equipment DQO, the following steps shall be performed on each sample:

(1) Extrude segments, videotaping the extrusion and photographing the extruded segments. The extrusion procedure is LO-160-103 at the 222-S Laboratory. If the segment contains solids, the solids shall be examined to confirm that layering is not present. If observations indicate that layers are present the cognizant scientist directs subsampling; the samples will be taken from the top, middle, and bottom of the segment, leaving the remainder of the segment undisturbed. Combine and homogenize the three samples. Three subsamples are to be taken from this homogenized sample and analyzed for ^{241}Am per Poppiti (1999). The subsamples will also be analyzed for total Alpha, solids density, wt% solids by gravimetry, weight fraction of centrifuged solids and centrifuged solids density.

(2) Analyze each solids segment for shear strength, and subsample for particle size analysis before waste is handled further. If observations indicate that layers are present, the cognizant scientist directs subsampling for particle size.

(3) Homogenize solids segments. If subsamples for Am-241 analysis were not taken in step (1), then take three subsamples of each solids segment and analyze for ^{241}Am per Poppiti (1999). Analyze for total Alpha, solids density and % wt solids by gravimetry, weight fraction of centrifuged solids and centrifuged solids density.

(4) Allow drainable liquid samples to settle for at least 16 hours. Record volume percent solids, and note any evidence of gas releases or separated liquid phases.

(5) Prepare 10 separate composites for each riser as directed by tank coordinator per the guidelines in Appendix B.

(6) Archive one of the composites.

(7) Mix solids and liquids and allow to settle for at least 16 hours. Record weight, volume, and volume % settled solids for each composite, and note any evidence of floating layers (organics or solids) or gas generation.

(8) Submit 4 of the composites from each riser to the Process Chemistry Laboratory for rheological measurements, dilution, water washing, and caustic washing tests per HLW and LAW DQOs. These tests will be performed per an approved Test Plan to be issued at a later date.
(9) Submit 4 of the composites from each riser to the Process Chemistry Laboratory for solids dissolution screening tests per L&H DQO. These tests will be performed per an approved Test Plan to be issued at a later date.
(10) Homogenize the remaining composite from each riser and analyze a representative subsample of the slurry for wt% oxides (@305°) and wt% oxides (@1050°C).
(11) Separate the remainder of each composite subsampled in step 10 by centrifugation. The responsible chemist shall ensure that the centrifuged solids contain no separate liquid phase.
(12) Analyze 3 subsamples of solid fraction of each riser composite separated in step 11 per Table 2.
(13) Analyze 3 subsamples of liquid fraction of each riser composite separated in step 11 per Table 1.

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

Opportunistic analyses as defined in Kristofzski (1996) are to be included when the laboratory is not operating at maximum capacity. Any decisions, observations, or deviations made to this work plan, or during the sample breakdown and analyses shall be documented in writing with justification. These decisions and observations shall be reported in the data report. The reporting formats for analyses are contained in Tables 1 and 2 and are described in Section 7.0.

3.2 SPECIFIC METHODS AND ANALYSES

The analyses in Tables 1 and 2 to be performed on the tank 241-AZ-102 core samples are based on the HLW, LAW, L&H, and Equipment 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-505-159 or LA-505-163 for acid digestion of samples, and LA-504-101 for water leach of solids. The laboratory is to notify the tank coordinator once 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 HLW and LAW DQOs. If process testing per the HLW and LAW DQOs determines that dilution is not required for waste retrieval, analyses per the L&H DQO can meet the corresponding data needs of the HLW and LAW DQOs. However, if process testing determines that dilution of the waste is required, then separate analyses of the diluted samples will be as specified in the test plan.

The HLW, LAW, and L&H DQOs specify Minimum Reportable Quantities (MRQs) of those analytes listed in Tables 1 and 2. The laboratory is to notify the tank coordinator of any

circumstances which prevent achieving detection limits at or below the MRQ for any analyte, and recommend a corrective course of action.

The L&H DQO requires that a material balance be performed for all material sampled and composited as part of this sampling event. Weights of all liquids and solids subsampled shall be reported in the Format IV data package.

3.3 INSUFFICIENT SEGMENT RECOVERY

If the amount of material recovered from samples taken from 241-AZ-102 is insufficient to form the composites and perform the analyses requested in the SAP and permit a minimum 100 mL archive per sample, the laboratory shall notify the tank coordinator within one working day. At that time, a prioritization of the analyses and/or compositing scheme may be provided to the laboratory. Any analyses prescribed by the SAP, but not performed, shall be identified in the appropriate data report with justification provided for nonperformance.

Table 1: Tank AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Liquids (3 Sheets)

LIQUID ANALYSES													
Project Name	241-AZ-102 Samples for Retrieval and Disposal			Plan Number	HNF-4577, REV. 3				REPORTING LEVELS			
PROGRAM	PROGRAM CONTACTS			COMMENTS					FORMAT I	Immediate Notification		
A. Waste Disposal Division	PNNL CP Devel.	K. D. Wiemers		Homogenization Test - Per Laboratory Discretion					FORMAT II	Process Control		
B. Waste Feed Delivery	Waste Feed Delivery	J. H. Baldwin		Field Blank - Required					FORMAT IV	Waste Management		
C. Process Control	RPP Process Engr	J. H. E. Rasmussen		Hot Cell Blank - Per Laboratory Discretion					FORMAT V	RCRA Compliance		
				TANK	# of samples				FORMAT VI	Special		
				241-AZ-101	2 Cores @ 18 segments ea.							

PRIMARY ANALYSES				SAMP[1]	PREP[2]	QUALITY CONTROL[3]					CRITERIA			
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SEG/ COMP	a/d/f/w/e	DUP/ TRIP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	FORMAT
A, B	Visual	Organic	LA-519-151	SEG	d	N/A	N/A	N/A	N/A	none	none	unknown	IV
C	IC	Br	LA-533-105	SEG	d	DUP	1/mtrx	ea PB	ea AB	µg/mL	none	unknown	IV
C	ICP/AES	Li	LA-505-151 LA-505-161	SEG	d or a	DUP	see[10]	ea PB	ea AB	µg/mL	none	unknown	IV
C	pH direct	pH	LA-212-106	SEG	d	DUP	N/A	N/A	N/A	pH	< 12	unknown	IV, I
A, B	Gravimetry	Sp-G	LA-510-112	COMP	d	N/A	N/A	N/A	ea AB	none	none	unknown	IV
A, B	Gravimetry	Wt % solids[15]	LA-560-101	COMP	d	TRIP	N/A	N/A	ea AB	Wt %	none	unknown	IV
A, B	ICP/AES	Ag, Al, B, Ba, Bi, Ca, Cd, Cr, Cu, K, Fe, La, Mg, Mn, Na, Nd, Ni, P, Pb, S, Sr, Si, Sn, Ti, U, Zn, Zr	LA-505-151 LA-505-161	COMP	d or a	TRIP	see[10]	ea PB	ea AB	µg/mL	none	unknown	IV
A. B	ICP/MS	As, B, Ba, Be, Ce, Co, La, Li, Mo, Pd, Pr, Pt, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ^{90}AMU	LA-506-101	COMP	d or a	TRIP	see[10]	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	ICP/MS	Cs, Eu[7]	LA-506-101	COMP	d or a	TRIP	N/A	ea PB	N/A	µg/mL	none	unknown	IV
A, B	ICP/MS	^{237}Np, ^{243}AMU, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{231}Pa,	LA-506-101	COMP	d or a	TRIP	1/mtrx[8]	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	ICP/MS	^{242}AMU, ^{126}Sn, ^{99}Tc, ^{241}AMU, ^{129}I	LA-506-101	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	IC	NO_2, NO_3, PO_4, SO_4, Cl, F, formate, oxalate, acetate, citrate, NTA	LA-533-105 LA-533-115	COMP	d	TRIP	see[10]	ea PB	ea AB	µg/mL	$[NO_2^-]$, $<0.01\underline{M}$	unknown	IV, I
A, B	CVAA	Hg	LA-325-106	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	ISE	NH3/NH4	LA-631-001	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	Pot. Titration	$[OH^-]$	LA-211-102	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µg/mL	$<0.01\underline{M}$	unknown	IV, I
A, B	pH	$[H^+]$	LA-212-106	COMP	d	TRIP	1/mtrx	ea PB	ea AB	pH	< 12	unknown	IV
A, B	furnace	TIC (CO3), TOC	LA-344-105	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µgC/mL	>45,000[5,6] (TOC only)	unknown	IV, I

Table 1: Tank AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Liquids (3 Sheets)

PRIMARY ANALYSES				SAMP[1]	PREP[2]	QUALITY CONTROL[3]				CRITERIA			
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SEG/ COMP	a/d/f/w/e	DUP/ TRIP	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	FORMAT
A, B	Ag catalyzed persulfate	TOC, TIC (CO3)	LA-342-100	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µgC/mL	>45,000[5,6] (TOC only)	unknown	IV, I
A	distillation/ colorimetric	CN	LA-695-102	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µg/mL	none	unknown	IV
A, B	Sep & beta count	^{90}Sr	LA-220-101	COMP	d	TRIP	1/mtrx	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/mtrx	ea PB	ea AB	µCi/mL	>61.5 (for $^{239/240}Pu$)	unknown	IV, I
A, B	sep/liquid scintillation	^{3}H	LA-218-114	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	sep/liquid scintillation	^{14}C	LA-348-104	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	none	unknown	IV
A	sep/liquid scintillation	^{79}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	^{99}Tc	LA-438-101	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	none	unknown	IV
A	sep/beta count	^{99}Tc (pertechnetate)[14]	LA-438-101	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	alpha counting	Total Alpha	LA-508-101	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	>61.5	unknown	IV, I
A	beta counting	Total Beta	LA-508-101	COMP	d or a	TRIP	1/mtrx	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	GEA[13]	$^{152,154,155}Eu$, ^{137}Cs, ^{60}Co, ^{125}Sb	LA-548-121	COMP	d	TRIP	N/A[12]	ea PB	ea AB	µCi/mL	none	unknown	IV
A, B	separation/GEA	^{129}I	LA-378-103	COMP	d	TRIP	N/A[12]	ea PB	ea AB	µCi/mL	none	unknown	IV
A	GEA	Total Gamma	LA-548-121	COMP	d or a	TRIP	N/A[12+H29]	ea PB	ea AB	µCi/mL	none	unknown	IV
SECONDARY ANALYSES				**SAMP[1]**	**PREP[2]**	**QUALITY CONTROL[3]**				**CRITERIA**			
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SEG/ COMP	a/d/f/w/e	DUP/ TRIP[3]	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	FORMAT
A	capillary zone electrophoresis	EDTA, HEDTA[11]	LA-533-113	COMP		TRIP	1/mtrx	ea AB	ea AB	µg /ml	none	unknown	IV
A	TBD[11+B25]	Organic Speciation	TBD	COMP	d	TRIP	1/mtrx	ea AB	ea AB	µg/g	none	unknown	IV

[1]COMP = Centrifuged supernate from Riser composite, SEG=every segment

[2]d = direct, f = fusion, a = acid, w = water, e = extraction

[3]DUP = duplicate, TRIP= triplicate subsamples analyzed singly, BLK = blank, STD = calibration standard, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtrx = matrix, N/A = not applicable

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

[5]Dry weight basis

[6]These analytes are to be compared to the limit by calculating the one-sided, upper 95% confidence limit for the sample result (to be performed by Process Engineering).

[7]Total Cs and Eu are sums of all isotopes, therefore spikes and LCS do not apply

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

Table 1: Tank AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Liquids (3 Sheets)

[9]Analysis to be performed if total alpha activity limit is exceeded.

[10] Either serial dilutions or matrix spikes wil be performed.

[11]If TOC or organic from IC>40,000 mg/L, analyze for chelator fragments by capillary zone electrophoresis, MeCl extraction/derivitization GC/mass spectroscopy and ion-pair chromatography.

[12]The 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. A tracer is used to correct for analyte loss during sample preparation and analysis

[13]An extended counting time in the presence of relatively high gamma activity may be required to achieve the reportable quantities for analytes other than ^{137}Cs

[14]The 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.

[15]Gravimetric Wt. % solids is to be performed @105^{0}C. See L&H DQO Section 7.3.4 for additional details.

Table 2: Tank 241-AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Solids (3 Sheets)

SOLID ANALYSES													
Project Name		AZ-102 Samples for Retrieval and Disposal			Plan Number	HNF-4577, REV. 3				REPORTING LEVELS			
PROGRAM		PROGRAM CONTACTS			COMMENTS					FORMAT I	Immediate Notification		
A. Waste Disposal Division		PNNL CP Devel.	K. D. Wiemers		Homogenization Test - Per Laboratory Discretion					FORMAT II	Process Control		
B. Waste Feed Delivery		Waste Feed Delivery	J. H. Baldwin		Field Blank - Required					FORMAT IV	Waste Management		
D. Process Control		RPP Process Engr	J. H. E. Rasmussen		Hot Cell Blank - Per Laboratory Discretion					FORMAT V	RCRA Compliance		
					TANK	# of samples				FORMAT VI	Special		
					AZ-101	2 Cores @ 18 segments ea.							
PRIMARY ANALYSES				SAMP[1]	PREP[2]	QUALITY CONTROL[3]				CRITERIA			
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SEG/ COMP	a/d/f/w/e	DUP/ TRIP[3]	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	FORMAT
B	Rot. Viscometer	Shear Strength[12]	LT-519-115	SEG	d	N/A	N/A	N/A	ea AB	kPa	none	unknown	IV
B	Particle Size Analysis	Particle Size Distribution[12]	LT-519-101	SEG	d	N/A	N/A	N/A	ea AB	kPa	none	unknown	IV
A	Gravimetry	Wt% solids[14]	LA-564-101	SEG	d	TRIP	N/A	N/A	ea AB	Wt%	none	45.9	IV, VI
A	Alpha counting	Total Alpha	LA-508-101	SEG	f or a	TRIP[16]	1/mtrx	ea PB	ea AB	μCi/g	> 41	unknown	I, IV, VI
A	Centrifugation	Weight fraction solids[17]	LA-519-132	SEG	d	TRIP	N/A	N/A	N/A	grams	None	unknown	IV, VI
A, B	Gravimetry	Wt% solids[14]	LA-564-101	COMP	d	TRIP	N/A	N/A	ea AB	Wt%	none	unknown	IV, VI
A, B	Centrifugation	Bulk Density	LO-160-103 LA-519-132	SEG, COMP	d	TRIP	N/A	N/A	N/A	g/ml	none	unknown	IV, VI
A	Gravimetry	Wt % oxides @ 1050^{0}C	see[13]	COMP	d	TRIP	N/A	N/A	N/A	Wt%	none	unknown	IV
A, B	ICP/AES	Ag, Al, B, Ba, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Mg, Mn, Li, Na, Nd, Ni, P, Pb, S, Sr, Si, Tc, Ti, U, Y, Zn, Zr	LA-505-151 LA-505-161	COMP	f or a	TRIP	see[8]	ea PB	ea AB	µg/g	none	unknown	IV
D	ICP/AES	Li	LA-505-151 LA-505-161	SEG	f or a	DUP	see[8]	ea PB	ea AB	µg/g	none	unknown	IV
D	IC	Br	LA-533-105	SEG	w	DUP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV
A, B	ICP/MS	As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ^{90}AMU	LA-506-101	COMP	f or a	TRIP	see[8]	ea PB	ea AB	µg/g	none	unknown	IV
A, B	ICP/MS	Cs[6]	LA-506-101	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	LA-506-101	COMP	f or a	TRIP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV
A, B	ICP/MS	^{241}AMU, ^{242}AMU, ^{126}Sn, ^{99}Tc, ^{151}AMU, ^{135}Cs, ^{129}I	LA-506-101	COMP	f or a	TRIP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV

Table 2: Tank 241-AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Solids (3 Sheets)

PRIMARY ANALYSES				SAMP[1]	PREP[2]	QUALITY CONTROL[3]				CRITERIA			
PROGRAM	METHOD	ANALYSIS	PROCEDURE	SEG/ COMP	a/d/f/w/e	DUP/ TRIP[3]	SPIKE	BLK	STD	UNITS	NOTIFICATION LIMIT[4]	EXPECTED RANGE[4]	FORMAT
A, B	IC	NO_2, NO_3, Cl, F, PO_4, SO_4, formate, oxalate	LA-533-105 LA-533-115	COMP	w	TRIP	1/mtrx	ea PB	ea AB	µg/g	$[NO_2^-]$, $<0.01\underline{M}$	unknown	I, IV
A, B	CVAA	Hg	LA-325-106	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV
A, B	ISE	NH3/NH4	LA-631-001 LA-533-101	COMP	d or w	TRIP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV
A, B	furnace	TIC (CO3), TOC	LA-344-105	COMP	w	TRIP	1/mtrx	ea PB	ea AB	µgC/g	>45,000[5] (TOC only)	unknown	IV, I
A, B	Ag catalyzed persulfate	TOC, TIC	LA-342-100	COMP	d	TRIP	1/mtrx	ea PB	ea AB	µgC/g	>45,000[5] (TOC only)	unknown	IV, I
A, B	distillation/ colorimetric	CN	LA-695-102	COMP	d or w	TRIP	1/mtrx	ea PB	ea AB	µg/g	none	unknown	IV
A, B	Sep & beta count	^{90}Sr, ^{90}Y	LA-220-101	COMP	f	TRIP	1/mtrx[7]	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	Separation/AEA	$^{238, 239/240}Pu$, ^{241}Am, $^{243/244}Cm$, ^{242}Cm	LA-953-104	COMP	f	TRIP	1/mtrx[7]	ea PB	ea AB	µCi/g	>41 (for $^{239/240}Pu$)	unknown	IV, I
B	sep/liquid scintillation	^{99}Tc	LA-438-101	COMP	w or a	TRIP	1/mtrx	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	sep/liquid scintillation	^{3}H	LA-218-114	COMP	w	TRIP	1/mtrx	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	sep/liquid scintillation	^{14}C	LA-348-104	COMP	w	TRIP	1/mtrx	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	alpha counting	Total Alpha	LA-508-101	COMP	f or a	TRIP	1/mtrx[8]	ea PB	ea AB	µCi/g	>41	unknown	IV, I
A, B	beta counting	Total Beta	LA-508-101	COMP	f or a	TRIP	1/mtrx[8]	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	GEA[15]	$^{152,154,155}Eu$, ^{137}Cs, ^{60}Co, ^{125}Sb[15]	LA-548-121	COMP	f	TRIP	N/A[10]	ea PB	ea AB	µCi/g	none	unknown	IV
A, B	separation/GEA	^{129}I	LA-378-103	COMP	f	TRIP	1/mtrx[7]	ea PB	ea AB	µCi/g	none	unknown	IV
A	Separation/AEA	^{241}Am	LA-953-104	SEG	f	TRIP[16]	1/mtrx[7]	ea PB	ea AB	µCi/g	none	unknown	IV, VI
A	Separation/GEA[11]	^{59}Ni, ^{63}Ni	TBD	COMP	f	TRIP	N/A[10]	ea PB	NP	µCi/g	none	unknown	IV
A	ICP/MS[11]	^{59}Ni, ^{63}Ni	LA-506-101	COMP	d	TRIP	N/A	ea PB	NP	µg/g	none	unknown	IV
A	ICP/MS[11]	^{93}AMU	LA-506-101	COMP	d	TRIP	N/A	ea PB	N/A	µg/g	none	unknown	IV
A	ICP/MS[11]	^{121m}Sn	LA-506-101	COMP	d	TRIP	N/A	ea PB	NP	µg/g	none	unknown	IV
A	separation/GEA[11]	^{126m}Sb, ^{126}Sb	TBD	COMP	d	TRIP	N/A[10]	ea PB	NP	µCi/mL	none	unknown	IV
A	GEA	Total Gamma	LA-548-121	COMP	f	TRIP	1/mtrx	ea PB	ea AB	µCi/g	none	unknown	IV

[1]SEG=every segment, COMP = Centrifuged solids from riser composite

[2]d = direct, f = fusion, a = acid, w = water, e = extraction

[3]DUP = duplicate, TRIP= triplicate subsamples analyzed singly, BLK = blank, STD = calibration standard, ea = each, smpl = sample, AB = analytical batch, PB = preparation blank, mtrx = matrix, N/A = not applicable, NP= not performed

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

[5]Dry weight basis

[6]Total Cs is the sums of all isotopes, therefore spikes and LCS do not apply.

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

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

Table 2: Tank 241-AZ-102 Chemical, Radiological, and Physical Analytical Requirements: Solids (3 Sheets)

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

[10]The 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

[11]Radionuclide only required for WAPS justification. Analysis is of low priority if unique separation or analysis is required

[12]Unhomogenized samples are to be used for particle size and shear strength

[13]No procedure is available for Wt% Oxides. Work will be performed to an approved test plan, which will then be referenced in the data package. Gravimetric wt%oxide is to be performed at 1050^{0}C. See L&H DQO section 7.3.4 for additional details

[14]Gravimetric Wt. % solids is to be performed @105^{0}C. See L&H DQO Section 7.3.4 for additional details.

[15]An extended counting time in the presence of relativley high gamma-activity may be required to achieve the minimum reportable quantitiy for ^{60}Co and ^{154}Eu, ^{155}Eu.

[16]For ^{241}Am and total alpha analysis perform in duplicate on three subsamples for a total of six results for each segment.

[17]Centrifuge, separate solids and liquids, and weigh.

Table 3. Detection Limits and Minimum Reportable Quantities for LAW Liquids (3 Sheets)

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	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 (1.0E+00)	2.3E+00 (3.0E+00)	µg/mL
B	ICP/MS	7.5E-01	2.3E+00	µg/mL
Ba	ICP/MS	7.5E-01 (2.6E+01)	2.3E+00 (7.8E+01)	µg/mL
Be	ICP/MS	7.5E-01 (3.0E+00)	2.3E+00 (9.9E+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
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 (3.0E+01)	7.5E+01 (9.9E+01)	µg/mL
Hg	CVAA	5.0E-01	1.5E+00 (2.0E+00)	µg/mL
K	ICP/AES	2.5E+02	7.5E+02 (7.5E+01)	µg/mL
La	ICP/MS	7.5E-01 (2.5E+01)	2.3E+00 (3.5E+01)	µg/mL
Li	ICP/MS	7.5E-01	2.3E+00	µg/mL
Mg	ICP/AES	5.5E+01 (5.0E+01)	1.7E+02 (1.5E+02)	µg/mL
Mn	ICP/AES	5.5E+00	1.7E+01	µg/mL
Mo	ICP/MS	7.5E-01 (3.0E+01)	2.3E+00 (9.0E+01)	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
Pt	ICP/AES	NS[2] (6.0E-01)	NS[2] (1.8E+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
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 (3.5E+01)	2.3E+00 (1.0E+02)	µg/mL
Se	ICP/MS	7.5E-01 (5.5E+01)	2.3E+00 (1.7E+02)	µg/mL
Si	ICP/AES	3.0E+01	9.0E+01	µg/mL

Table 3. Detection Limits and Minimum Reportable Quantities for LAW Liquids (3 Sheets)

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	Units
Sn	ICP/AES	NS[2] (5.0E+02)	NS[2] (1.5E+03)	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 (2.0E-01)	7.8E+02 (6.0E+02)	µg/mL
V	ICP/MS	7.5E-01 (3.0E+01)	2.3E+00 (9.0E+01)	µg/mL
W	ICP/MS	7.5E-01	2.3E+00	µg/mL
Y (^{90}AMU)	ICP/MS	7.5E-01 (1.0E+02)	2.3E+00 (2.0E+02)	µg/mL
Y	ICP/AES	NS[2] (1.0E+02)	NS[2] (2.0E+02)	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	LSC	7.0E-03	2.1E-02	µCi/mL
^{14}C	LSC	2.4E-04	7.2E-04	µCi/mL
^{60}Co[3]	GEA	7.0E-04	2.1E-03	µCi/mL
^{79}Se	LSC	3.0E-05	9.0E-05	µCi/mL
^{89}Sr, ^{90}Sr	beta count	1.0E-02 (5.0E-02)	3.0E-02 (1.5E-01)	µCi/mL
^{99}Tc (total)	ICP/MS	5.0E-04	1.5E-03	µCi/mL
^{99}Tc (pertechnetate)	TBD	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 (3.5E-04)	1.8E-05 (1.1E-03)	µCi/mL
^{137}Cs	GEA	1.3E-01 (3.0E+00)	3.9E-01 (9.0E+00)	µCi/mL
^{152}Eu[3]	GEA	TBD	TBD	µCi/mL
^{154}Eu[3]	GEA	6.5E-03	2.0E-02 (2.0E-03)	µCi/mL
^{155}Eu[3]	GEA	3.0E-02	9.0E-02	µCi/mL
^{231}Pa	ICP/MS	TBD	TBD	µCi/mL
^{233}U	ICP/MS	1.4E-04 (6.0E-04)	4.2E-04 (1.8E-03)	µCi/mL
^{234}U	ICP/MS	4.4E-05 (4.0E-08)	1.2E-04 (1.2E-07)	µCi/mL
^{235}U	ICP/MS	1.5E-08 (1.1E-06)	4.5E-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 (9.1E-03)	3.9E-05 (2.7E-02)	µCi/mL
^{238}Pu	AEA	3.4E-03 (3.2E-03)	1.0E-02 (9.6E-03)	µCi/mL
^{238}U	ICP/MS	2.4E-09 (1.7E-07)	7.2E-09 (5.0E-07)	µCi/mL
^{239}Pu	AEA	3.4E-03 (3.20E-03)	1.0E-02 (9.6E-03)	µCi/mL
^{240}Pu	AEA	1.7E-02 (3.2E-03)	5.1E-02 (9.6E-03)	µCi/mL

Table 3. Detection Limits and Minimum Reportable Quantities for LAW Liquids (3 Sheets)

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	Units
^{241}Pu	AEA	(3.2E-03)	(9.6E-03)	µCi/mL
$^{241}Pu/Am$	ICP/MS	1.6E+00	4.8E+01	µCi/mL
^{242}Pu	ICP/MS	1.0E-02 (3.2E-03)	3.0E-02 (9.6E-03)	µCi/mL
^{241}Am	AEA	1.0E-02 (2.4E-04)	3.0E-02 (7.2E-04)	µCi/mL
^{243}Am	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	distil./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 (2.5E+03)	µg/mL
SO_4	IC	7.7E+02	2.3E+03	µg/mL
Total Alpha	prop. counter	7.5E-02	2.3E-01	µCi/mL
Total Beta	beta count	TBD	TBD	µCi/mL
Total Inorganic Carbon	Persulfate/ combustion furnace	5.0E+01	1.5E+02	µg/mL
Total Organic Carbon	Persulfate/ combustion furnace	5.0E+02	1.5E+03	µg/mL

Acronyms:
AEA – Alpha Energy Analysis
AMU - Atomic Mass Unit
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
ISE – Ion Selective Electrode
LSC – Liquid Scintillation Counter
N/A – Not applicable
NS - Not stated
RSD – Relative Standard Deviation
Wt% - Weight percent

Footnotes:
[1]Detection limits and MRQ's for the L&H DQO. Where HLW or LAW DQO requirements differ from those listed, the corresponding HLW or LAW requirement is shown in parentheses. The HLW and LAW requirements apply only if testing shows that dilution is not needed to meet waste transfer requirements. If process testing determines that dilution is required for transfer, requirements for analysis of the diluted waste will be as per the test plan.
[2]NS 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 and LAW MRQ's 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.
[3]An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

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

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	Units (per gram dried solids)
Ag	ICP/AES	300 (50)	900 (900 14)	µg
Al	ICP/AES	1200 (110)	3600 (330)	µg
As	ICP/MS	20 (10)	60 (30)	µg
B	ICP/MS	10 (1)	30 (3)	µg
Ba	ICP/AES	200	600	µg
Be	ICP/MS	10	30	µg
Bi	ICP/AES	2000	6000	µg
Ca	ICP/AES	2000 (62)	6000 (180)	µg
Cd	ICP/AES	300 (4)	900 (11)	µg
Ce	ICP/MS	2	6	µg
Co	ICP/MS	2 (1)	6 (3)	µg
Cr	ICP/AES	400 (40)	1200 (120)	µg
Cu	ICP/AES	200 (6.5)	600 (18)	µg
F	IC	2500	7500	µg
Fe	ICP/AES	400 (50)	1200 (140)	µg
Hg	CVAA	0.5	1.5	µg
K	ICP/MS	2000 (500)	6000 (1500)	µg
La	ICP/AES	1000 (20)	3000 (60)	µg
Li	ICP/MS	10	30	µg
Mg	ICP/AES	1800 (180)	5400 (540)	µg
Mn	ICP/AES	100	300	µg
Mo	ICP/MS	2	6	µg
Na	ICP/AES	1800 (50)	5400 (150)	µg
Nd	ICP/AES	1000 (26)	3000 (77)	µg
Ni	ICP/AES	600 (55)	1800 (160)	µg
P	ICP/AES	2000 (200)	6000 (600)	µg
Pb	ICP/AES	1200 (200)	3600 (600)	µg
Pd	ICP/MS	10 (1)	30 (3)	µg
Pr	ICP/MS	2	6	µg
Pu	ICP/MS	2 (8)	6 (24)	µg
Rb	ICP/MS	2	6	µg
Rh	ICP/MS	2	6	µg
Ru	ICP/MS	4 (1)	12 (3)	µg
S	ICP/MS	NS[2] (40)	NS[2] (120)	µg
Sb	ICP/MS	4	12	µg
Se	ICP/MS	100	300	µg
Si	ICP/AES	10000 (1000)	30000 (3000)	µg
Sr	ICP/AES	100	300	µg
Ta	ICP/MS	2	6	µg
Tc	ICP/MS	(2)	(6)	µg

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

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	Units (per gram dried solids)
Te	ICP/MS	6 (2)	18 (6)	µg
Th	ICP/MS	2 (200)	6 (600)	µg
Ti	ICP/AES	200 (50)	600 (150)	µg
Tl	ICP/MS	2 (200)	6 (600)	µg
V	ICP/MS	2 (0.02)	6 (0.06)	µg
W	ICP/MS	2	6	µg
Y	ICP/MS	2 (90)	6 (270)	µg
Zn	ICP/AES	400 (2)	1200 (6)	µg
Zr	ICP/AES	NS[2] (200)	NS[2] (600)	µg
Cl	IC	75	225	µg
CN^-	CN^- analysis	1	3	µg
CO_3^{-2}	Persulfate. Combustion furnace	NS[2] (10)	NS[2] (30)	µg
NH_3	ISE	20	60	µg
NO_2^-	IC	150	450	µg
NO_3^-	IC	150	450	µg
TOC	Persulfate. Combustion furnace	20	60	µg C
^{3}H	NS	NS (5.0E-03)	NS (1.5E-02)	µCi
^{14}C	ß-LSC	2.0E-04 (6.0E-04)	6.0E-04 (1.8E-03)	µCi
^{59}Ni	Sep/GEA	1.0E-02	3.0E-02	µCi
^{60}Co	GEA	4.0E-02 (4.0E-03)	1.2E-01 (1.2E-02)	µCi
^{63}Ni	Sep/ß-LSC	2.0E-03	6.0E-03	µCi
^{90}Sr[3]	Sep/ß-GPC	7.0E+00	2.1E+01	µCi
^{90}Y[3]	Sep/ß-GPC	7.0E+00	2.1E+01	µCi
^{93}Zr[8]	ß-LSC	2.0E-03	6.0E-03	µCi
^{93}AMU	ICP/MS	4	12	µCi
^{93m}Nb[8]	ICP/MS	4	12	µCi
^{99}Tc	ICP/MS	NA (2.0E+00)	NA (6.0E+00)	µCi
^{121m}Sn[9]	Sep/GEA	9.0E-02	2.7E-01	µCi
^{125}Sb[4]	GEA	2.0E+00	6.0E+00	µCi
^{125m}Te[4]	GEA	2.0E+00	6.0E+00	µCi
^{126m}Sb[5]	Sep/GEA	6.0E-03	1.8E-02	µCi
^{126}Sb[5]	Sep/GEA	6.0E-03	1.8E-02	µCi
^{126}Sn[5]	ICP/MS	6.0E-03 (2.0E-02)	1.8E-02 (6.0E-02)	µCi
^{129}I	ICP/MS	10	30	µCi
^{135}Cs	ICP/MS	2	6	µCi

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

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity[1]	Minimum Reportable Quantity (MRQ)[1]	Units (per gram dried solids)
^{137m}Ba[6]	GEA	3.0E-02	9.0E-02	µCi
^{137}Cs[6]	GEA	3.0E-02 (2.0E-02)	9.0E-02 (6.0E-02)	µCi
^{151}AMU	ICP/MS	2	6	µCi
^{152}Eu	GEA	2.0E+00	6.0E+00 (2.-E+00)	µCi
^{154}Eu	GEA	1.0E-01 (2.0E-02)	3.0E-01 (6.0E-02)	µCi
^{155}Eu	GEA	2.0E+00 (2.0E-02)	6.0E+00 (6.0E-02)	µCi
^{233}U	ICP/MS	0.2 (2.0E+00)	0.6 (6.0E+00)	µCi
^{234}U	ICP/MS	2	6	µCi
^{235}U	ICP/MS	2	6	µCi
^{236}U	ICP/MS	2	6	µCi
^{237}Np	ICP/MS	2 (6.0E-01)	6 (1.8E+00)	µCi
^{238}Pu	Sep/AEA	2.0E-02 (2.0E-05)	6.0E-02 (6.0E-05)	µCi
^{238}U	ICP/MS	2	6	µCi
^{239}Pu[7]	Sep/AEA	2.0E-02 (2.0E+00)	6.0E-02 (6.0E+00)	µCi
^{240}Pu[7]	Sep/AEA	2.0E-02	6.0E-02	µCi
^{241}Am	Sep/AEA	6.0E-03 (4.0E-04)	1.8E-02 (1.2E-03)	µCi
^{241}AMU	ICP/MS	2	6	µCi
^{242}Cm[10]	Sep/AEA	4.0E-03	1.2E-02	µCi
^{242m}Am[10]	Sep/AEA	4.0E-03	1.2E-02	µCi
^{242}Pu	ICP/MS	0.2	0.6	µCi
^{242}Pu	Sep/AEA	2.0E-02	6.0E-02	µCi
^{243}Am	ICP/MS	2	6	µCi
$^{243+244}Cm$	Sep/AEA	4.0E-03 (2.0E-05)	1.2E-02 (6.0E-05)	µCi

Acronyms:
AEA – Alpha Energy Analysis
GEA – Gamma Energy Analysis
GPC – Gas flow proportional counter
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
LSC – Liquid Scintillation Counter
NA – Not applicable
NM – Not measured
Sep – separation required
TIMS – Thermal Ionization Mass Spectrometry

Notes:
[1]Detection limits and MRQ's for the L&H DQO. Where HLW or LAW DQO requirements differ from those listed, the corresponding HLW or HAW requirement is shown in parentheses. The HLW and LAW requirements apply only if testing shows that dilution is not needed to meet waste transfer requirements. If process testing determines that dilution is required for transfer, requirements for analysis of the diluted waste will be as per the test plan.
[2]NS 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 and LAW MRQ's 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.
[3]Combined analysis of ^{90}Sr and ^{90}Y
[4]Combined analysis of ^{125}Sb and ^{125m}Te
[5]Combined analysis of ^{126}Sn, ^{126m}Sb, and ^{126}Sb
[6]Combined analysis of ^{137}Cs and ^{137m}Ba
[7]Combined analysis of ^{239}Pu and ^{240}Pu.
[8]Combined analysis of ^{93m}Nb and ^{93}Zr
[9]No method currently available
[10]Combined analysis of ^{242}Am, ^{242m}Am, and ^{242}Cm

4.0 QUALITY ASSURANCE AND QUALITY CONTROL

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

4.1 LABORATORY OPERATIONS

Laboratories performing analyses in support of this SAP shall have approved and implemented Quality Assurance (QA) Plans. These QA plans shall meet the *Hanford Analytical Services Quality Assurance Requirements Document* (DOE-RL 1998) minimum requirements as the baseline for laboratory quality systems. The *222-S Laboratory Quality Assurance Plan* (Markel 1999) 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, 5, and 6. 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). The criteria presented are goals for demonstrating reliable method performance. It is understood that the laboratory will follow its internal QC system for required actions whenever QC failures occur. If sample QC failures occur, or if all analyses cannot be performed (e.g., insufficient sample), analysts shall consult with supervisors/customers to determine the proper action. The laboratory should provide a suggested course of action at that time. All sample QC failures and limitations on the associated data shall be discussed in the narrative of the data report. Proper notification of all data not meeting QC requirements shall be included with the data. It should be noted that the L&H DQO requires specific quality control/quality assurance requirements. These requirements are outlined in section 7.7 of the L&H DQO.

4.2 SAMPLE COLLECTION

Before sampling can be performed on a tank, available risers must be identified for use in the sampling event. The selected risers must be inspected and prepared to confirm their ability to be used in sampling. Safety hazards must be identified and special precautions must be taken if needed. If deemed necessary by the sampling cognizant engineers and tank coordinator, video surveillance should be performed to identify any potential problems that may occur during the sampling event.

Samples are to be taken from a tank and shipped to the performing laboratory by CPO in accordance with the respective work packages. The chain-of-custody forms for these work packages shall identify samples by a unique number and state the type of sampler used (retained gas sampler or universal sampler) for each sample before being shipped to the 222-S Laboratory.

Approved procedure TO-080-090 [Load/Transport Sample Cask(s)] is to be used during the sampling event. Pertinent sampling information (e.g., unusual waste characteristics, X-ray scan results, LiBr solution used, or detecting debris) should be noted in the comment section of the chain-of-custody form.

Characterization Project Operations should transport each sample collected to the performing laboratory within 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 5. Quality Control Parameters for Liquid Analysis (3 Sheets)

Liquid Fraction[a]	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[b]	Spike %Recovery[c]	Triplicate RSD[d]
Ag, Al, , B, Ba, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Li, Mg, Mn, Nd, Ni, P, Pb, Pd, Pt, Rh, Ru, S, Sn, Sr, Si, Ti, U, 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, Pt, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ^{90}AMU[e]	ICP/MS	80 - 120%	70 - 130%	<15%
Br^-, Cl^-, F^-, NO_2^-, NO_3^- PO_4^{-3}, SO_4^{-2},formate[h], oxalate[h], acetate[h], citrate[h], NTA[h]	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/ colorimetric	80 - 120%	75 - 125%	<15%
Cs[f], Eu[f]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	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/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A[i]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[j]	GEA	NP	N/A[k]	<15%
^{79}Se	Liq. scintillation	NP	N/A[i]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[i]	<15%
^{99}Tc	ICP/MS, Sep/Liq scintillation	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)[g]	Separation/beta count	80 - 120%	70 - 130%	<15%

Table 5. Quality Control Parameters for Liquid Analysis (3 Sheets)

Liquid Fraction[a]	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[b]	Spike %Recovery[c]	Triplicate RSD[d]
^{125}Sb	GEA	TBD	TBD	TBD
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	Separation/GEA	NP	N/A[k]	<15%
^{137}Cs	GEA	NP	N/A[k]	<15%
^{152}Eu[i]	GEA	NP	N/A[k]	<15%
^{154}Eu[i,j]	GEA	NP	N/A[k]	<15%
^{155}Eu[i,j]	GEA	NP	N/A[k]	<15%
^{231}Pa	ICP/MS	TBD	TBD	TBD
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np[l]	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu[l]	Separation/AEA	NP	N/A[i]	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[i]	<15%
^{242}Cm	Separation/AEA	NP	N/A[i]	<15%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 – 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[i]	<15%
Total Alpha[l]	Proportional counter	70 - 130%	75-125% (80-120% RPD for Safety Screening	<15%[o]
Total Beta	Beta counting	70 - 130%	70 – 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Specific Gravity	Density	N/A	N/A	N/A
Wt% dissolved solids[m]	Gravimetric	80 - 120%	N/A	<21%

Acronyms:

AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
LCS – Laboratory Control Standard

N/A – Not applicable
NP - not performed
RPD - relative percent difference
TBD – To be determined
RSD – Relative Standard Deviation
Wt% - Weight Percent

Footnotes:

(a) analytes for the Solubility Screening Test are a subset of this analyte list.
(b) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.
(c) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured, less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices.
For other analytes, the accuracy is determined based on use of serial dilutions.
(d) RSD = Relative Standard Deviation between the samples, defined as: (standard deviation of the mean/mean) x 100. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit.
(e) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.
(f) Total Cs and Eu are sums of all isotopes; therefore, spiking and LCS do not apply.
(g) The 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.
(h) Secondary analytes for the L&H DQO
(i) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.
(j) An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{154}Eu, ^{155}Eu.
(k) The 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.
(l) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting TRU when total alpha measurement in the liquid fraction is equal to or exceeds 6.0E-05 Ci/L for Envelopes A and B and 4.0E-04 Ci/L for Envelope C. The selected trigger values correspond to 70% of the LAW envelope limits for TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes which are defined as alpha-emitting TRU (e.g., ^{237}Np, ^{242}Pu, ^{242}Cm, ^{243}Am, and $^{243+244}Cm$) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the minimal detectable activities (MDA). Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination.
(m) Weight percent dissolved solids method is described in L&H DQO Section 7.3.4.

Table 6. Quality Control Parameters for Solids Analysis (3 Sheets)

Solids Fraction[a]	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[b]	Spike % Recovery[c]	Triplicate RSD[d]
Ag, Al, B, Ba, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Li, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Tc, Ti, U, Zn, Zr, Y	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, ^{90}AMU[e]	ICP/MS	80 - 120%	70 - 130%	<15%
Acetate, Br, Cl^-, citrate, F^-, formate, NO_2^-, NO_3^-, oxalate, NTA, PO_4, SO_4	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/ colorimetric	80 - 120%	75 - 125%	<15%
Cs[f]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^{3}H	Sep/LSC	80 - 120%	N/A[g]	<15%
^{14}C	Sep/LSC	80 - 120%	75 – 125%	<15%
^{63}Ni[h]	ICP/MS	80 - 120%	70 – 130%	<15%
^{59}Ni[h]	ICP/MS	80 - 120%	70 – 130%	<15%
^{59}Ni[h]	Sep/GEA	NP	N/A[f]	<15%
^{63}Ni[h]	Isotopic specific separation/beta-LSC	NP	N/A[g]	<15%
^{60}Co[j]	GEA	80 - 120%	N/A[f]	<15%
^{90}Sr[k]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{90}Y[k], [h]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{93}Zr[h], [l]	Beta-LSC	NP	N/A[g]	<15%

Table 6. Quality Control Parameters for Solids Analysis (3 Sheets)

Solids Fraction[a]	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[b]	Spike % Recovery[c]	Triplicate RSD[d]
^{93}AMU	ICP/MS	80 - 120%	70 – 130%	<15%
^{99}Tc	ICP/MS, Sep/LSC	80 - 120%	70 – 130%	<15%
^{121m}Sn[h]	Sep/GEA	NP	N/A[f]	<15%
^{125}Sb[m]	GEA	NP	N/A[f]	<15%
^{126}Sb[n], [h]	Sep/GEA	NP	N/A[f]	<15%
^{126m}Sb[h], [n]	Sep/GEA	NP	N/A[f]	<15%
^{126}Sn[n]	ICP/MS	80 - 120%	70 – 130%	<15%
^{129}I	Sep/GEA	NP	N/A[f]	<15%
^{135}Cs[h]	ICP/MS	80 - 120%	70 – 130%	<15%
^{137}Cs	GEA	NP	N/A[f]	<15%
^{151}Sm	Isotopic specific separation/beta-LSC	NP	N/A[g]	<15%
^{152}Eu[j]	GEA	NP	N/A[f]	<15%
^{154}Eu[j]	GEA	NP	N/A[f]	<15%
^{155}Eu[j]	GEA	NP	N/A[f]	<15%
^{233}U	ICP/MS	90 - 110%	75 – 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np[o]	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu[o]	Sep/AEA	NP	70 – 130%	<15%
^{241}Pu/Am, ^{242}Pu[o]	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am[o]	Sep/AEA	80 – 120%	N/A[g]	<15%
^{242}Cm[o]	Sep/AEA	NP	N/A[g]	<15%
^{243}Am/Cm[o]	ICP/MS	90 - 110%	75 – 125%	<15%
$^{243+244}$Cm[o]	Sep/AEA	NP	N/A[g]	<15%
Total Alpha[o]	Proportional counter	70 - 130%	75 – 125%	<15%
Total Beta	Beta counting	70 - 130%	70 – 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density	Gravimetric	N/A	N/A	N/A
Wt% solids[p]	gravimetric	80 - 120%	N/A	<21%
Wt % oxide[p]	gravimetric	TBD	TBD	TBD
Shear Strength	Rot Viscometer	N/A	N/A	N/A
Particle size	Particle Size Analysis	N/A	N/A	N/A

Acronyms:

AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis

IC – Ion Chromatography
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
ICP/AES - Inductively Coupled Plasma Atomic Emission Spectroscopy
LCS – Laboratory Control Standard
N/A – Not applicable
TBD – To be determined
RSD – Relative Standard Deviation
Wt% - Weight Percent
NP - Not performed
N/A - Not available

Footnotes:

(a) Analytes for the Solubility Screening Test are a subset of this analyte list
(b) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.
(c) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured, less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions as described in Section 7.6.2.2.
(d) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit.
RSD = (standard deviation of the mean/mean) x 100
(e) ICP/MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.
(f) Total Cs and Eu are sums of all isotopes; therefore, spiking and LCS do not apply.
(g) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result is generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.
(h) Radionuclide only required for Waste Acceptance Product Specifications (WAPS) justification. Analysis is lower priority if unique separation or analysis is required.
(i) The 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.
(j) An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{154}Eu, ^{155}Eu.
(k) Combined analysis of ^{90}Sr and ^{90}Y.
(l) Combined analysis with ^{93m}Nb.
(m) Combined analysis with ^{125m}Te.
(n) Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.
(o) Trigger level based on total alpha and specific isotopes to be measured . . . TBD.
(p) Weight percent solids and weight percent oxide methods are described in L&H DQO Section 7.3.4.

4.3 SAMPLE CUSTODY

The chain-of-custody form is initiated by the sampling team as described in the work packages. Samples are shipped in a cask and sealed with a Waste Tank Sample Seal (see below).

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

Each sample number shall be created using the sample's core and segment number. For instance, segment 1 of core 197 would be sample number 197-01. The sealed and labeled samples are shipped to the laboratory along with the chain-of-custody form. The receipt and control of samples in the 222-S Laboratory are described in laboratory procedure LO-090-101.

5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

5.1 EXCEPTIONS TO DATA QUALITY OBJECTIVES REQUIREMENTS

The solids dissolution rate determination required by the HLW and LAW DQOs is waived per customer request for tank 241-AZ-102 only. Consequently, no special testing steps are required when the solids and liquids are mixed during preparation of the composite.

The requirement to dry the solids fraction of the tank composite at 105 °C ("solids B") prior to analysis (Patello et al. 1999) is waived per customer request for tank 241-AZ-102 only.

Many of the analyses performed in triplicate as directed by the L&H DQO are also required in duplicate by the HLW and LAW DQOs. 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 and LAW DQOs in the event that dilution is not required. Tables 1 through 6 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 liquids (Ba and La) and solids (As, B, Be, Ce, Co, Cs, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V) required by the HLW and LAW DQOs.

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 interferences which 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, it is recommended that the ICP/AES be performed for all possible analytes and that ICP/MS be applied to those analytes that are below the ICP/AES quantitation limit and above the desired MRQ.

For the Equipment DQO (Bloom 1996) only shear strength is required.

No analytical method is currently available to perform the sludge particle density measurements required by the HLW DQO. (Nguyen 1999a). These analyses may be requested at a later date if and when a method becomes available.

5.2 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 error associated with the results is documented. No reruns or additional analyses should be performed to improve recovery for analytes not specifically requested in Tables 1 or 2.

6.0 ORGANIZATION

The organization and responsibility of key personnel involved with this tank 241-AZ-102 characterization project are listed in Table 7.

Table 7: Tank 241-AZ-102 Project Key Personnel List

Responsibility	Organization	Individual
RPP 241-AZ-102 Tank Coordinator	RPP Process Engineering (LMHC)	J. H. E. Rasmussen, 373-1128
222-S Laboratory Point of Contact (day shift)	Analytical Services (WMH)	D. B. Hardy, 376-4878
222-S Laboratory Point of Contact (off-hours)	Analytical Services (WMH)	222-S Laboratory Shift Manager, 373-2435
200 East Tank Farm Point of Contact	Tank Farm Operations	East Tank Farm Operations Shift Manager, 373-3475 or 373-2689
Data Management	Manager, Data Development and Interpretation	K. M. Hall, 376-5029
Process Engineering Point of Contact for Immediate Notifications	RPP Process Engineering (LMHC)	On-Call Process Engineer 539-2074 or 85-9654 (pager)
Process Chemistry Point of Contact	Manager, Process Chemistry (NHC)	L. L. Lockrem, 373-4471
RPP Privatization Point of Contact	Manager, Privatization Interface	K. A. Gasper, 373-1948

7.0 DELIVERABLES

All analyses will be reported as Formats I, IV, or VI as indicated in Tables 1 and 2. Additional information regarding reporting formats is given in Schreiber (1998).

7.1 FORMAT I REPORTING

Tables 1 and 2 contain the notification limits for each analyte. Any results exceeding their notification limits shall be reported via telephone by the 222-S Laboratory Facility Planning Team to 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) Process Engineering 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, segments, subsegments, drainable liquids, and associated blanks taken and analyzed for this sampling event and for core sample 254 which was obtained from tank 241-AZ-102 in 1998. The recommended reporting format and the raw data that shall be included are given in Section A5.0 of Schreiber (1998). The data package shall be issued 180 days after the last sample is received at the laboratory. The raw data shall be accessible to the program in accordance with the laboratory's Records Inventory and Disposition Schedule and until the respective waste tank is closed or the waste is treated.

In addition to this data package, an electronic version of the analytical results shall be provided to the Tank Characterization Database representative on the same day that the final data package is issued. The data must be available to the Washington State Department of Ecology within 7 days of release of the data package. The electronic version shall be in the standard electronic format (Lang et al., 1999).

7.3 FORMAT VI REPORTING

The Americium-241 analyses subsampled in triplicate and performed in duplicate for each solids segment per Poppiti (1999) must be completed within 45 calendar days after the completion of the sampling event. The laboratory shall issue a letter report specific to this Americium-241 analysis within this 45-day period. The report shall include identification of the segment locations, the weight fraction of solids and liquids for each segment, and a LABCORE report including the Americium -241, total alpha, solids density, centrifuged solids density, and weight fraction of centrifuged solids measurements. The report shall be provided to Dr. Neil R. Brown, Waste Processing and Disposal Program, Mr. James F. Thompson, Jr. Program Development Division, Ms Karyn Wiemers, Chemical Process Development Group, and Ms Kathleen Hall, Data Development and Interpretation.

8.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Project. All significant changes (such as analyte additions or analysis of new, additional samples) shall be documented by RPP Process Engineering via an Engineering Change Notice to this SAP or by a letter. All changes shall also be clearly documented in the final data report. Insignificant changes may be made by the tank or project coordinator by placing a notation in the permanent record (i.e., note change in extrusion log book or memorandum to file). Significance is determined by the tank coordinator.

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

9.0 REFERENCES

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APPENDIX A:

EXPECTED PHYSICAL PROFILE OF TANK 241-AZ-102 CORE SAMPLES

APPENDIX A:

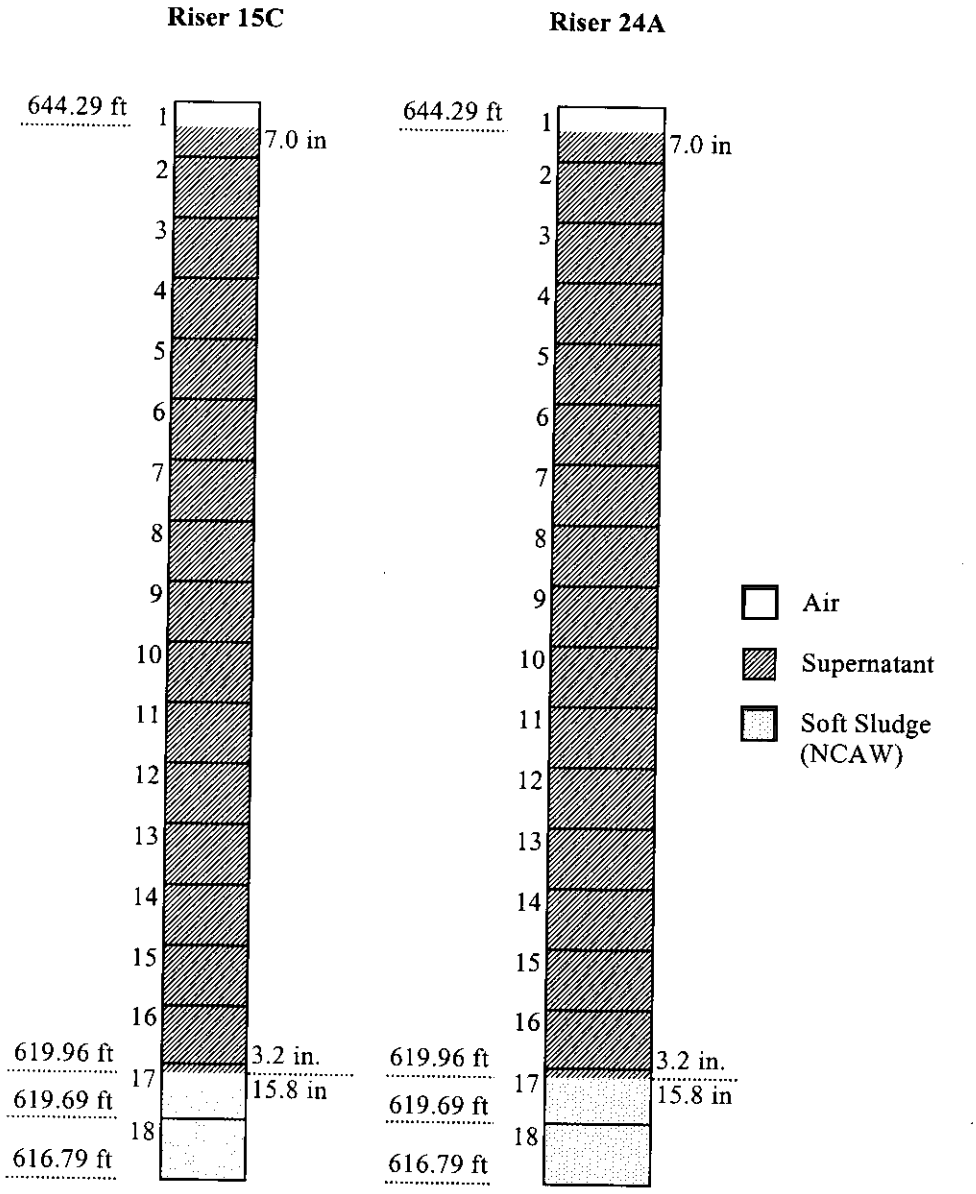
EXPECTED PHYSICAL PROFILE OF TANK 241AZ-102 CORE SAMPLES

Table A-1: Tank 241-AZ-102 Physical Profile Estimate Risers 15C and 24A

Segment #	Inches	Elevation Range (ft. MSL)	Waste Type	Comments
1	12.0	645.29 - 644.29	Air	
	7.0	644.29 - 643.71	Liquid	Good Recovery
2	19.0	643.71 - 642.12	Liquid	Good Recovery
3	19.0	642.12 - 640.54	Liquid	Good Recovery
4	19.0	640.54 - 638.96	Liquid	Good Recovery
5	19.0	638.96 - 637.37	Liquid	Good Recovery
6	19.0	637.37 - 635.79	Liquid	Good Recovery
7	19.0	635.79 - 634.21	Liquid	Good Recovery
8	19.0	634.21 - 632.62	Liquid	Good Recovery
9	19.0	632.62 - 631.04	Liquid	Good Recovery
10	19.0	631.04 - 629.46	Liquid	Good Recovery
11	19.0	629.46 - 627.87	Liquid	Good Recovery
12	19.0	627.87 - 626.29	Liquid	Good Recovery
13	19.0	626.29 - 624.71	Liquid	Good Recovery
14	19.0	624.71 - 623.12	Liquid	Good Recovery
15	19.0	623.12 - 621.54	Liquid	Good Recovery
16	19.0	621.54 - 619.96	Liquid	Good Recovery
17	3.2	619.96 619.69	Liquid	Good Recovery
	15.8	619.69 - 618.37	Soft sludge, NCAW waste.	Good Recovery
18	19.0	618.37 - 616.79	Soft sludge, NCAW waste.	Good Recovery

Note: elevations based on inside tank bottom elevation for Tank 241-AZ-102 of 616.54 ft. MSL
MSL = Mean Sea Level
NCAW = Neutralized Current Acid Waste

Figure A-2: Physical/Chemical Profile Estimate Chart of Tank 241-AZ-102



Note:
1. The waste level is based on a tank inside bottom elevation of 616.54 ft MSL
2. Each segment is 19 inch long and partial segments were specified
3. Waste depth based on the manual tape measurement of 5/15/99

APPENDIX B:

241-AZ-102 WASTE COMPOSITE PREPARATION GUIDELINES

These preparation steps for the 241-AZ-102 waste composite satisfies the HLW, LAW and the L&H DQO objective that the composite be representative of the waste to be retrieved and allows the composite to be prepared in multiple jars.

Core Sample Extrusion:

Cores shall be extruded onto the extrusion tray. Solids and drainable liquids shall be placed in separate jars. A chemist shall note the degree of separation achieved both for the solids and the drainable liquids. If the drainable liquid contains a high concentration of solids, the chemist needs to determine if centrifuging is needed to separate the solids. The composite preparation assumes minimal solids in the drainable liquids and minimal liquid in the extruded solids. Error is introduced in the composite preparation when there is a high concentration of solids in the drainable liquid because of the difficulty in keeping the solids suspended during the liquid transfer into the composite jars. When excess liquid is in the solids, again maintaining homogeneity of the transferred solids is difficult. When making a decision to centrifuge, the composite preparation errors need to be balanced with errors associated with increased sample handling and transfer associated with the centrifuging operation.

During the entire sample extrusion process and possible subsequent sample preparation, a thorough material balance needs to be maintained and unaccounted for mass loss shall be less than 10%. Observations of sample jars are very important to understanding and interpretation of later analyses.

Composite Preparation:

The composite preparation steps assume that each core segment has a jar of drainable liquids and a jar of solids. If a segment has only one jar, the steps below still apply. The number of composite jars prepared is dependent on the quantity of composite needed to meet the requirements in the Sampling and Analysis Plan (SAP). This compositing procedure requires that the composite be prepared using two aliquots of solids and two aliquots of liquid from each segment that is used as part of the composite.

Use the following steps to prepare a set of whole tank composites in individual composite jars.

1. Based on the quantity of composite required, the number of segments, and distribution of segments sampling locations in the tank, determine the weight of solids required from each segment to prepare each individual composite jar (reference calculations in the L&H DQO).

2. Determine the weight of the solids aliquot for each segment where the solids aliquot weight equals the solids weight determined above divided by two.
3. Based on the quantity of composite required, the number of segments, and distribution of segments sampling locations in the tank, determine the weight of liquids required from each segment to prepare each of the composite jars (reference calculations in the DQO).
4. Determine the weight of the liquid aliquot for each segment where the liquid aliquot weight equals the liquid weight determined above divided by two.
5. Homogenize the contents of the first segment solids jar by mixing.
6. Distribute a solids aliquot from the homogenized segment solids jar (weight calculated above) into each individual composite jar.
7. Homogenize the contents of the first segment liquids jar by mixing.
8. Distribute a liquid aliquot from the homogenized segment liquid jar (weight calculated above) into each individual composite jar.
9. Repeat steps 5 through 8 for each segment to be used in preparing the composites. Fill the composite jars in a random order so that the fill order is changed every time that steps 5 through 8 are repeated Note that homogenization of the sample jar before transferring an aliquot is important.

The first solids aliquot and liquid aliquot from each segment should now have been transferred to each individual composite jar. The following steps will complete the composite preparation by distributing the second solids aliquot and second liquid aliquot from each segment into the individual composite jars.

10. Rehomogenize the contents of the first segment solids jar by mixing.
11. Distribute a solids aliquot from the homogenized segment solids jar (weight calculated above) into each individual composite jar.
12. Rehomogenize the contents of the first segment liquids jar by mixing.
13. Distribute a liquid aliquot from the homogenized segment liquid jar (weight calculated above) into each individual composite jar.
14. Repeat steps 10 through 13 for each segment to be used in preparing the composites. Fill the composite jars in a random order so that the fill order is changed every time that steps 10 through 13 are repeated Note that homogenization of the sample jar before transferring an aliquot is important.

The composite preparation is now complete. The following measurements are required for the composites.

15. Allow the composite jars to settle undisturbed for 12 to 24 hours.
16. Measure and document the volume % settled solids for each composite jar.

Compositing steps shall be conducted in the presence of a chemist. The attending chemist is to compare jar-to-jar variability based on observed volume % settled solids. Any adjustments to the composite are to be documented and are to accommodate preservation of the sample pedigree to the extent possible. The attending chemist shall direct adjustments to the composite.

DISTRIBUTION SHEET

To	From	Page 1 of 2
Distribution	Data Development and Interpretation	Date 07/30/99
Project Title/Work Order		**EDT No.** N/A
HNF-4577, Rev. 3, "Tank 241-AZ-102 Privatization Push Mode Core Sampling and Analysis Plan"		**ECN No.** ECN-655005

Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
Office of River Protection					
W. Liou	S7-54	X			
J. A. Poppiti	S7-54	X			
DOE Reading Room	H2-53	X			
Lockheed Martin Hanford Corp.					
D. G. Baide	S5-05	X			
J. H. Baldwin	R3-73	X			
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Waste Management Federal Services of Hanford, Inc.					
R. Akita	T6-50	X			
K. E. Bell	T6-12	X			
R. A. Esch	T6-12	X			
R. K. Fuller	T6-12	X			
D. B. Hardy	T6-12	X			
K. L. Powell	S3-30	X			
F. H. Steen	T6-12	X			