PNWD-3215, Rev 1
WTP-RPT-048, Rev 1

Chemical Analysis and Physical Property Testing of 241-AZ-101 Tank Waste—Supernatant and Centrifuged Solids

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February 2004

Prepared for Bechtel National, Inc.
under Contract 24590-101-TSA-W000-0004

LEGAL NOTICE

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2/5/04
for W. Tamosaitis
ACCEPTED FOR
WTP PROJECT USE

February 2004

Prepared for Bechtel National, Inc.
under Contract 24590-101-TSA-W000-0004

Test specification: 24590-PTF-TSP-RT-01-001, Rev. 0
Test plan: TP-RPP-WTP-089, Rev 1
Test exceptions: 24590-WTP-TEF-RT-02-071, 24590-WTP-TEF-RT-02-073
R&T focus area: Waste Characterization
Test Scoping Statement(s): B-2a

Battelle - Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by 24590-PTF-TSP-RT-01-001 Rev. 0 and TP-RPP-WTP-089 Rev 1. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

2/4/04

Gordon H. Beeman, Manager *Date*
WTP R&T Support Project

Change History

Revision #	Revision	Purpose
Rev 1 February 2004	ICP-MS Tc-99 measurement uncertainty increased.	Clarification due to 'potential' calibration and verification standards bias.
Rev 1 February 2004	Np-237 and Pu-^{239,240} by ICP-MS same source standard for calibration and verification; added footnotes to Tables 8.1, 8.4, 9.1, and 9.4.	Document discrepancy with QA Plan requirement.
Rev 1 February 2004	Removed ICP-MS uncertainties Tables 8.1 and 8.3.	Clarification: Propogated uncertainties only provided for radchem results.
Rev 1 February 2004	Remove 'B' flags from process blanks in Tables 8.1 through 8.4.	"B" flags only appropriate for 'samples' impacted by high process blank.
Rev 1 February 2004	Added references to ASR revisions ASR 6193.01 through ASR 6193.06; ASRs added to appendix.	Included for completeness.
Rev 1 February 2004	Added uncertainties for radiochem results that were inadvertently omitted from Table 8.3.	Correction.
Rev 1 February 2004	Removed bold/outline >15% RSD if results were J-flagged (Tables 9.1 through 9.4).	Clarification: QC criterion of <15% only applies when results >EQL.
Rev 1 February 2004	Table 9.4 ICPAES acid and fusion results switched for As, Ce, Co, Mo, Pd, Rh, Ru, Sb, Se, Te, Th, Tl, V, W, and Y.	Correction. Note: all results are non-detects.
Rev 1 February 2004	Changed approach for reporting gluconate.	More appropriate to report gluconate as coeluting anion with glycolate (not as "no method available").
Rev 1 February 2004	Revised ^{127}I and ^{129}I results and text added throughout report.	Data correction required due to calibration and verification standards issue.
Rev 1 February 2004	Miscellaneous editorial changes.	Clarification.

Summary

Battelle – Pacific Northwest Division (PNWD) is conducting integrated process verification and waste-form qualification tests on Hanford waste from underground storage Tank 241-AZ-101 (AZ-101) in support of the River Protection Project-Waste Treatment Plant (RPP-WTP). Testing includes sample compositing, homogenization, and characterization; followed by pretreatment process testing and vitrification of the resulting low-activity waste (LAW) and high-level waste (HLW) streams.

To support this testing, portions from 18 segments of one core sample retrieved from Tank AZ-101 in August 2000 were provided to the PNWD. Following confirmation that none of the bottles containing liquid were contaminated with hydrostatic head fluid, these samples were homogenized into a single slurry composite and carefully separated into 15 sub-samples. Specified sub-samples were tested for physical properties, including density, percent solids, rheology, heat capacity, and particle size. Four composite sub-samples were selected for inorganic, radiochemical, and selected organic analysis. The sub-samples were phase separated by centrifuging into supernatant and wet centrifuged solids (WCS) and both phases characterized for numerous inorganic analytes, organic analytes, and radionuclides. Analyses were performed in triplicate on both the wet centrifuged solids and supernatant phases.

The characterization of the representative AZ-101 composite sub-samples for both supernatant and wet centrifuged solids included but was not limited to:

- inductively coupled plasma atomic emission spectrometry (ICP-AES)
- radiochemical analyses, including ^{99}Tc^{+7}
- inductively coupled plasma mass spectrometry (ICP-MS)
- total uranium by kinetic phosphorescence analysis (KPA)
- ion chromatography (IC, inorganic and organic anions)
- titration for hydroxide
- total inorganic carbon (TIC) and total organic carbon (TOC)
- selected organic analytes (e.g., organic acids and chelators)
- selected physical properties: density, weight percent solids, and weight percent oxide, heat capacity, particle size distribution (PSD), and rheology.

The homogenization and characterization activities were conducted per test plan TP-RPP-WTP-089 (Appendix D1) in accordance with the requirements set forth in test specification 24590-PTF-TSP-RT-01-001 (Appendix A), which was initially defined in Test Scoping Statement B-2. This report summarizes sample receipt, compositing, homogenization, and initial characterization activities of the AZ-101 tank waste. The results for all analytes of interest specified by the test specification are reported with the exception of bis-(2-ethylhexyl) phosphate (D2EHP) for which no reliable method was available, and polychlorinated biphenyls (PCBs) which were removed as an analyte of interest by BNI (Test Exception 24590-WTP-TEF-RT-02-071 and 24590-WTP-TEF-RT-02-073).

Table S.1 summarizes the physical properties measured on the AZ-101 material and Table S.2 and Table S.3 summarize the results of the radiochemical and chemical analysis of the composite supernatant and wet centrifuged solids. The results shown in the "WCS column" (Table S.2 and Table S.3) are the wet centrifuged solids concentrations, which contain a contribution from interstitially entrained supernatant. The results shown in the "UDS columns" are the resulting undissolved solids contents after the supernatant contribution is mathematically removed. The calculated undissolved solids results are reported on both a wet-weight and dry-weight basis. Because the centrifuged solids were not washed, they contain components that may be removed during pretreatment.

Table S.1. AZ-101 As-received Composite – Physical Properties

Matrix	Physical Property/Measurement	Average	%RSD
Supernatant	Density (g/mL)	1.233	0.4
	TDS (wt%)	26.7	4
Composite slurry	Density (g/mL)	1.25	1
	Wt% centrifuged solids	18	9
	Wt% undissolved solids	7.6	9
3-wt% undissolved solids slurry	Heat capacity (J/g-K) at 25°C	3.49	18
	Heat capacity (J/g-K) at 40 °C	3.48	19
13-wt% undissolved solids slurry	Heat capacity (J/g-K) at 25 °C	2.89	16
	Heat capacity (J/g-K) at 40 °C	2.89	17
20-wt% undissolved solids slurry	Heat capacity (J/g-K) at 25 °C	2.83	25
	Heat capacity (J/g-K) at 40 °C	2.82	26
14-wt% undissolved solids slurry	Apparent viscosity (cP) at 25 °C	2.9	14
	Yield point (Pa) at 25 °C	0.2	15
	Apparent viscosity (cP) at 40 °C	2.5	3
	Yield point (Pa) at 40 °C	0.1	29
45-wt% undissolved solids slurry	Apparent viscosity (cP) at 25 °C	13	25
	Yield point (Pa) at 25 °C	3.7	22
Slurry solids	PSD (%Vol) – Particles above 16.6 µm	14	n/a
	PSD (%Vol) – Particles between 4.4 and 16.6 µm	51	n/a
	PSD (%Vol) – Particles between 1.14 and 4.4 µm	32	n/a
	PSD (%Vol) – Particles between 0.27 and 1.14 µm	3	n/a

RSD: relative standard deviation

TDS: total dissolved solids

PSD: particle size distribution following 90 second sonication

Table S.2. AZ-101 As-received Composite – Radionuclide Summary

Radionuclide	Measurement Method	WCS (b)		Supernatant (b)		UDS	
		Average µCi/g	(a) DF	Average µCi/mL	(a) DF	Average µCi/g (Wet)	Average µCi/g (Dry)
^{3}H	H-3	7.08E-03		1.81E-02		1.06E-03	1.79E-03
^{14}C	C-14	1.04E-03	B	1.93E-03		4.04E-04	6.80E-04
^{60}Co	GEA	2.03E+00		4E-02	U	2.03E+00	3.42E+00
^{90}Sr	Sr-90	2.02E+04		5.01E-02 (c)		2.02E+04	3.40E+04
^{99}Tc (total)	ICP-MS	1.48E-01		3.27E-01		3.91E-02	6.59E-02
^{125}Sb	GEA	8.07E+00	J	NM		8.07E+00	1.36E+01
^{126}Sn	GEA	2E+00	U	2E+00	U	< 2E+00	< 4E+00
^{129}I	ICP-MS	1.09E-05	J	1.06E-06	J	1.05E-05	1.78E-05

Table S.2. (Cont'd)

Radionuclide	Measurement Method	WCS (b) Average µCi/g	(a) DF	Supernatant (b) Average µCi/mL	(a) DF	UDS Average µCi/g (Wet)	UDS Average µCi/g (Dry)
^{137}Cs	GEA	7.52E+02		1.64E+03		2.06E+02	3.47E+02
^{137}Cs	ICP-MS (Calc)	8.76E+02		1.40E+03		4.11E+02	6.92E+02
^{152}Eu	GEA	1E+00	U	NM		< 1E+00	< 2E+00
^{154}Eu	GEA	2.34E+01		2E-01	U	2.34E+01	3.95E+01
^{155}Eu	GEA	3.06E+01		2E+00	U	3.06E+01	5.15E+01
^{233}U	ICP-MS	1.61E-03		9E-06	U	1.61E-03	2.71E-03
^{235}U	ICP-MS	7.32E-05		4.73E-08	B	7.32E-05	1.23E-04
^{237}Np	ICP-MS	1.94E-02		3.77E-05	J	1.94E-02	3.26E-02
^{238}Pu	AEA	3.06E-01		3.44E-04	B	3.06E-01	5.16E-01
^{239,240}Pu	AEA	2.41E+00		1.81E-03		2.41E+00	4.06E+00
^{239}Pu	ICP-MS	1.14E+00		2.00E-03	JB	1.14E+00	1.92E+00
^{241}Pu	Pu-241	1.15E+01		NM		1.15E+01	1.93E+01
^{241}Am	AEA	3.75E+01		1.05E-04	B	3.75E+01	6.32E+01
^{241}Am (GEA)	GEA	4.49E+01		2E+00	U	4.49E+01	7.56E+01
^{242}Pu	AEA	9E-03	U	NM		< 9E-03	< 2E-02
^{242}Cm	AEA	3.91E-02	J	9E-06	U	3.91E-02	6.59E-02
^{243,244}Cm	AEA	9.48E-02	J	4.18E-05	JB	9.48E-02	1.60E-01
Alpha	Gross alpha	5.39E+01		5E-03	U	5.39E+01	9.08E+01
Radionuclides measured only in Supernatant							
^{79}Se	Se-79	NM		9.69E-04		NA	NA
^{99}Tc^{+7}	Tc-99	NM		3.77E-01		NA	NA
^{231}Pa	GEA	NM		2E+00	U	NA	NA
^{234}U	ICP-MS	NM		6E-06	U	NA	NA
^{236}U	ICP-MS	NM		9.32E-08	J	NA	NA
^{238}U	ICP-MS	NM		9.38E-07	B	NA	NA

WCS = wet centrifuged solids; UDS = undissolved solids; NM = not measured; NA= not applicable

(a) Data flags: B = analyte measured in blank above the EQL; J = estimated value; U= not detected above the reported MDL.

(b) For decay correction reference dates see Table 8.1and Table 8.3.

(c) Supernatant ^{90}Sr result is significantly lower than other published results; see discussion Section 8.3 and Section 9.4.4.

Table S.3. AZ-101 As-received Composite – Analyte Summary

Analyte	Measurement Method	WCS Average µg/g	(a) DF	Supernatant Average µg/mL	(a) DF	UDS Average µg/g (Wet)	UDS Average µg/g (Dry)
Ag	ICP-AES	5.50E+01	J	6E-01	U	5.50E+01	9.27E+01
Al	ICP-AES	1.36E+05		6.05E+03		1.34E+05	2.26E+05
Ammonia	none	NM		2.30E+00	J	NA	NA
As	ICP-MS	4.61E+01		9.90E+00	J	4.28E+01	7.21E+01
B	ICP-AES	3E+01	U	7.74E+01	BX	< 3E+01	< 5E+01

Table S.3. (Cont'd)

		WCS		Supernatant		UDS	
Analyte	Measurement Method	Average µg/g	(a) DF	Average µg/mL	(a) DF	Average µg/g (Wet)	Average µg/g (Dry)
Ba	ICP-AES	4.89E+02		2.50E-01	J	4.89E+02	8.24E+02
Be	ICP-AES	9.00E+00	J	3E-01	U	9.00E+00	1.52E+01
Bi	ICP-AES	6E+01	U	3E+00	U	<6E+01	< 1E+02
C as TIC (c)	C (Furn)	1.10E+03	J	5.83E+03		< 1E+03	< 2E+03
C as TIC (c)	C (HP)	8.38E+03		9.85E+03		5.11E+03	8.60E+03
C as TOC (c)	C (Furn)	9.90E+03		1.50E+03		9.40E+03	1.58E+04
C as TOC (c)	C (HP)	8.20E+02		5.10E+02		6.50E+02	1.10E+03
Ca	ICP-AES	2.40E+03		9.30E+00	J	2.40E+03	4.04E+03
Cd	ICP-AES	4.67E+03		7.70E-01	JB	4.67E+03	7.87E+03
Ce	ICP-MS	2.97E+02		5E+00	U	2.97E+02	5.00E+02
Cl	IC-Inorg	1.10E+02		2.40E+02		3.02E+01	5.09E+01
CN	CN	5.41E-01	X	2.04E+00	B	< 4E-01	< 7E-01
Co	ICP-MS/ICP-AES	3.83E+01	B	1E+00	U	3.83E+01	6.45E+01
Cr	ICP-AES	8.00E+02		6.86E+02		5.72E+02	9.64E+02
Cs (d)	ICP-MS	3.20E+01		5.18E+01		1.48E+01	2.49E+01
Cu	ICP-AES	2.38E+02		6E-01	U	2.38E+02	4.01E+02
F (b)	IC-Inorg	4.11E+03		2.01E+03		3.44E+03	5.80E+03
Fe	ICP-AES	6.68E+04		1.50E+00	JB	6.68E+04	1.13E+05
Hg	CVAA	3.15E+00		5.53E-02		3.13E+00	5.28E+00
K	ICP-AES	1.40E+03	J	4.46E+03		< 1E+03	< 2E+03
La	ICP-AES	1.79E+03		1E+00	U	1.79E+03	3.02E+03
Li	ICP-AES	7.00E+01	J	8.90E-01	J	6.97E+01	1.17E+02
Mg	ICP-AES	4.40E+02	J	3E+00	U	4.40E+02	7.41E+02
Mn	ICP-AES	1.48E+03		1E+00	U	1.48E+03	2.49E+03
Mo	ICP-MS	1.46E+02	B	8.88E+01		1.16E+02	1.96E+02
Na	ICP-AES	7.15E+04		1.11E+05		3.46E+04	5.83E+04
Nd	ICP-AES	1.26E+03		3E+00	U	1.26E+03	2.12E+03
Ni	ICP-AES	2.77E+03		1.00E+00	JB	2.77E+03	4.67E+03
NO_2	IC-Inorg	2.74E+04		6.13E+04		7.02E+03	1.18E+04
NO_3	IC-Inorg	2.29E+04		5.26E+04		5.41E+03	9.12E+03
P	ICP-AES	1.79E+03		5.04E+02		1.62E+03	2.73E+03
Pb	ICP-AES	4.90E+02	J	3.30E+00	J	4.89E+02	8.24E+02
Pd	ICP-MS	1.01E+02		2.00E+01	J	9.44E+01	1.59E+02
Pr	ICP-MS	2.64E+02		NM		2.64E+02	4.45E+02
Pu sum	ICP-MS	1.97E+01		NM		1.97E+01	3.32E+01
Rb	ICP-MS	2.62E+02	B	9.20E+00		2.59E+02	4.36E+02
Rh	ICP-MS/ICP-AES	9.08E+01		8E+00	U	9.08E+01	1.53E+02
Ru	ICP-MS/ICP-AES	1.15E+03		3E+01	U	1.15E+03	1.94E+03
Sb	ICP-MS	3.42E+00	B	1E+01	U	3.42E+00	5.76E+00
Se	ICP-MS	1E+00	UB	6E+00	U	< 1E+00	< 2E+00
Si	ICP-AES	3.80E+03	J	2.06E+02	BX	3.73E+03	6.29E+03
SO_4	IC-Inorg	2.40E+04		1.62E+04		1.86E+04	3.14E+04
Sr	ICP-AES	3.56E+02		4E-01	U	3.56E+02	6.00E+02
Ta	ICP-MS	3.65E+00	B	NM		3.65E+00	6.15E+00

Table S.3. (Cont'd)

		WCS		Supernatant		UDS	
	Measurement	Average	(a)	Average	(a)	Average	Average
Analyte	**Method**	**µg/g**	**DF**	**µg/mL**	**DF**	**µg/g (Wet)**	**µg/g (Dry)**
Tc	ICP-MS	8.69E+00		1.92E+01		2.30E+00	3.88E+00
Te	ICP-MS/ICP-AES	1.96E+02		4E+01	U	1.96E+02	3.30E+02
Th	ICP-MS/ICP-AES	1.09E+02		3E+01	U	1.09E+02	1.84E+02
Ti	ICP-AES	6.40E+01	J	6E-01	U	6.40E+01	1.08E+02
Tl	ICP-MS/ICP-AES	1.55E-01	B	1E+01	U	1.55E-01	2.61E-01
U	ICP-AES	3.60E+03	J	5E+01	U	3.60E+03	6.07E+03
U	KPA	3.17E+03		5.15E-01		3.17E+03	5.34E+03
U sum	ICP-MS	3.68E+03		2.81E+00	B	3.68E+03	6.20E+03
V	ICP-MS	2.34E+01	B	1.50E+00	J	2.29E+01	3.86E+01
W	ICP-MS	3.33E+01	B	5.80E+01	J	1.40E+01	2.36E+01
Y	ICP-MS/ICP-AES	1.17E+02		1E+00	U	1.17E+02	1.97E+02
Zn	ICP-AES	7.90E+01	J	1E+00	U	7.90E+01	1.33E+02
Zr	ICP-AES	1.95E+04		1E+00	U	1.95E+04	3.29E+04
Analytes measured only in Supernatant							
Acetate	IC-Org	NM		1E+02	U	NA	NA
Citrate	IC-Org	NM		4E+02	U	NA	NA
Citric acid	GC/FID	NM		6E+00	U	NA	NA
D2EHP (e)	GC/FID	NM		(e)		NA	NA
ED3A	GC/FID	NM		5E+00	U	NA	NA
EDTA	GC/FID	NM		5E+00	U	NA	NA
Formate	IC-Org	NM		3.70E+02	J	NA	NA
Gluconate (g)	IC-Org	NM		5E+02	U	NA	NA
Glycolate (g)	IC-Org	NM		2E+02	U	NA	NA
HEDTA	GC/FID	NM		9E+00	UX	NA	NA
IDA	GC/FID	NM		1E+01	U	NA	NA
NTA	GC/FID	NM		6E+00	U	NA	NA
OH	Titration	NM		1.14E+04		NA	NA
Oxalate	IC-Org	NM		1.00E+03		NA	NA
PCB (f)	GC/ECD	NM		(f)		NA	NA
Succinic acid	GC/FID	NM		5.00E+01	J	NA	NA

WCS = wet centrifuged solids; UDS = undissolved solids; NM= not measured; NA= not applicable
EDTA=ethylenediaminetetraacetic acid; HEDTA=N-(2-hydroxyethyl)ethylenediaminetriacetic acid;
ED3A=ethylenediaminetriacetic acid; NTA=nitrilotriacetic acid; IDA=iminodiacetic acid;
D2EHP= bis-(2 ethylhexyl) phosphate

(a) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria; X = quality control (QC) deficiency.

(b) Fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggests the presence of co-eluting anion(s).

(c) For TOC and TIC: HP = hot persulfate oxidation method; Furn = furnace oxidation method.

(d) Total Cs: Cesium on wet centrifuged solids calculated from sum of ^{133}Cs measured by ICP-MS and the calculated ^{135,137}Cs using the Cs isotope mass fractions from the supernatant results.

(e) Analyte not measured due to lack of reliable method.

(f) Measurement of PCBs deleted from test specification per test exception 24590-WTP-TEF-RT-02-071 and 24590-WTP-TEF-RT-02-073.

(g) Glycolate and gluconate results should be considered the upper bound MDL concentration, since glycolate and gluconate are not resolved by the IC measurement method used for the analysis. IC system calibrated using glycolate; gluconate estimate based on gluconate-to-glycolate response factor. Each result assumes 100% of response due to each analyte.

The AZ-101 analytical results for the supernatant and undissolved solids are compared to the WTP specifications. The supernatant composite was compared to the Contract Specification 7 LAW Envelope B Definition (see Table S.4); the undissolved solids component was compared to Contract Specification 8, HLW Definition (see Table S.5). The Specification 7 limits may have been exceeded for ^{60}Co and ^{154}Eu because these radionuclides could not be measured at the required minimum reportable quantity (MRQ) due to interference from the very high ^{137}Cs concentration. The Contract Specification 8 limits were exceeded for Al and S concentrations, with both the Al and S exceeding the limits at approximately 200%. One radionuclide, ^{126}Sn, could not be measured at a detection level sufficiently low enough to determine whether or not the radionuclide was within the Contract Specification 8 limit.

Table S.4. AZ-101 As-received Supernatant – Compared to Specification 7 Envelope B

Table TS-7.1 Analytes					
Analytes Measured Above MDL			**Analytes Not Detected Above MDL**		
Measurement Method	**Analyte**	**% of Contract Limit**	**Measurement Method**	**Analyte**	**% of Contract Limit**
IC	NO_2	73	ICP-AES	U	< 4
C [(a)]	C as TIC	57	ICP-AES	La	< 2
IC	SO_4	50			
C [(b)]	C as TIC	34			
IC	NO_3	22			
ICP-AES	Al	19			
ICP-AES	Cr	14			
ICP-AES	K	13			
IC	F	11			
C [(b)]	C as TOC	5			
IC	PO_4	3			
ICP-AES	P as PO_4	3			
C [(a)]	C as TOC	2			
IC	Cl	2			
ICP-AES	Pb, Ba, Ca, Ni, Fe, Cd	0.5 or less			
CVAA	Hg				
ICP-MS	U				
KPA	U (KPA)				
Table TS-7.2 Radionuclides					
Analytes Measured Above MDL			**Analytes Not Detected Above MDL**		
Measurement Method	**Radionuclide**	**% of Contract Limit**	**Measurement Method**	**Analyte**	**% of Contract Limit**
GEA	^{137}Cs	63	GEA	^{60}Co	**< 560**
ICP-MS	^{99}Tc	35	GEA	^{154}Eu	**< 130**
(Sum)	TRU	4			
Sr-90	^{90}Sr	1 [(c)]			

Outlined and bolded results may exceed Contract Specification 7 (Envelope B) criteria.

TRU (transuranic) = Z>92, alpha emitter, half-life >10yr (^{237}Np, ^{238,239,240}Pu, ^{241}Am, and ^{242,243,244}Cm)

(a) Carbon by hot persulfate oxidation method

(b) Carbon by furnace oxidation method

(c) Supernatant ^{90}Sr result is significantly lower than other published results; see discussion Section 8.3 and Section 9.4.4.

Table S.5. AZ-101 As-received Undissolved Solids – Compared to Specification 8

Analytes Measured Above MDL			Analytes Not Detected Above MDL		
Measurement Method	Analyte	% of Contract Limit	Measurement Method	Analyte	% of Contract Limit
Specification 8 Table TS-8.1 Analytes					
ICP-MS	Te	33	ICP-AES	B	< 0.5
ICP-MS	Rb	30	ICP-MS	Se	< 0.05
ICP-MS	Pr	16	ICP-MS	Tc	< 0.0001
ICP-AES	Nd	16			
ICP-MS	Y	16			
ICP-MS	V	15			
ICP-AES	La	15			
ICP-AES	Sr	15			
ICP-AES	Li	11			
ICP-AES	Cu	11			
Multiple	Ce, Pu, As, Mn, Zn, Mo, Be, Ta, Co, W, Hg, Cs, Sb, Tl	10 or less			
Specification 8 Table TS-8.2 Analytes					
None	Ammonia	NM	C(b)	C as CO_3	< 4
C(a)	C as CO_3	18	CN	CN	< 0.001
C(b)	C as TOC	19	None	NH_3	NM
Multiple	NO_2/NO_3, Cl, C as TOC(a)	10 or less			
Specification 8 Table TS-8.3 Radionuclides					
Sr-90	^{90}Sr	44	GEA	^{126}Sn	$\boxed{\mathbf{< 337}}$
ICP-MS	^{233}U	39	GEA	^{152}Eu	< 57
GEA	^{155}Eu	23			
AEA	^{238}Pu	19			
AEA	^{239,240}Pu	17			
Pu-241	^{241}Pu	11			
Multiple	^{154}Eu, ^{241}Am, ^{235}U, ^{237}Np, ^{125}Sb, ^{60}Co, ^{137}Cs, ^{14}C, ^{129}I, ^{243,244}Cm, ^{3}H, ^{99}Tc	10 or less			

Table S.5 (cont’d)

Analytes Measured Above MDL			Analytes Not Detected Above MDL		
Measurement Method	**Analyte**	**% of Contract Limit**	**Measurement Method**	**Analyte**	**% of Contract Limit**
Specification 8 Table TS-8.4 Analytes					
ICP-AES	Al	**207**	ICP-AES	K	< 23
IC-Inorg	SO_4 as S	**207**	ICP-AES	Bi	< 0.5
ICP-MS	Ru	71			
ICP-AES	Fe	50			
ICP-AES	Na	39			
ICP-AES	Zr	28			
ICP-AES	Ni	25			
ICP-AES	Cd	22			
IC-Inorg	F	21			
ICP-AES	P	21			
ICP-AES	Cr	18			
ICP-MS	Pd	16			
ICP-MS	Rh	15			
Multiple	Pb, Ca, U (ICP-MS, KPA, and ICP-AES), Mg, Si, Ba, Ag, Ti, Th	10 or less			

Outlined and bolded results exceed or may exceed Contract Specification 8 (Envelope D) criteria.

(a) carbon by hot persulfate oxidation method

(b) carbon by furnace oxidation method

NM: not measured

Terms and Abbreviations

AEA	alpha energy analysis
AMU	atomic mass unit
ASR	analytical service request
BNI	Bechtel National Inc.
BS	blank spike
CAR	corrective action report
CI	confidence interval
CVAA	cold vapor atomic absorption
COC	chain of custody
CUF	cells unit filter
D2EHP	bis-(2-ethylhexyl) phosphate
DF	data quality flag
DIW	distilled, deionized water
DPP	diphenylphosphate
DQO	data quality objective
DSC	differential scanning calorimeter
EDTA	ethylenediaminetetraacetic acid
ED3A	ethylenediaminetriacetic acid
EQL	estimated quantitation limit
FURN	furnace oxidation (TIC/TOC analysis)
GC/ECD	gas chromatography/electron capture detector
GC/FID	gas chromatography/flame ionization detector
GEA	gamma energy analysis
HASQARD	Hanford Analytical Services Quality Assurance Requirements Documents
HEDTA	N-(2-hydroxyethyl)ethylenediaminetriacetic acid
HLRF	High-Level Radiation Facility
HLW	high-level waste
HP	hot persulfate oxidation (TIC/TOC analysis)
HPIC	high-performance ion chromatography
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
IDA	iminodiacetic acid
IDL	instrument detection limit
ISE	ion specific electrode
KPA	kinetic phosphorescence
LAW	low-activity waste
LCS	laboratory control standard
LSC	liquid scintillation counter

MDA	minimum detectable activity
MDL	method detection limit
MRQ	minimum reportable quantity
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
NIDA	nitrosoiminodiacetic acid
NIST	National Institute of Standards and Technology
NM	not measured
NTA	nitrilotriacetic acid
%Diff	percent difference
PB	process blank
PCB	polychlorinated biphenyl
PNWD	Battelle - Pacific Northwest Division
PS	post spike
PSD	particle size distribution
QA	quality assurance
QC	quality control
RPL	Radiochemical Processing Laboratory
RPP	River Protection Project
RSD	relative standard deviation
SAL	Shielded Analytical Laboratory
SEM	scanning electron microscope
SRM	standard reference material
TC	total carbon
TDS	total dissolved solids
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
UDS	undissolved solids
UPA	Microtrac™ ultrafine particle size analyzer
WCS	wet centrifuged solids
WTP	Waste Treatment Plant
X100	Microtrac™ X-100 particle size analyzer

Unit Abbreviations

Bq	Becquerel
Ci	curie
cP	centipoise
°C	degrees Centigrade
g	gram
G	gravitational force
K	Kelvin
kg	kilogram
L	liter
μCi	microcurie
μg	microgram
μm	micrometer
M	molarity / mole
m	meter
mg	milligram
mL	milliliter
mPa	millipascal
N	normality
Pa	Pascal
rpm	revolutions per minute
s	second
vol%	volume percent
W	watt
wt%	weight percent

Definitions

Settled Solids – the solids layer that separated from the bulk slurry after gravity settling for a specified settling period (typically 3 to 7 days). The results may be reported in volume percent (vol%) and/or weight percent (wt%). The wt% may be reported on either a wet-weight basis (i.e., mass of settled solids contains interstitial liquid) or on a dry-weight basis (i.e., mass of settled solid dried at 105°C to a constant weight).

Centrifuged Solids – the solids layer that separates from the bulk slurry after centrifugation (typically for 1 hour at 1000 G). These results may be reported as vol% or wt%, with the wt% on either a wet-weight or dry-weight basis.

Dissolved Solids – the soluble solids in the liquid phase. The solids remaining after the liquid is dried at 105°C to a constant weight. Typically reported as wt% total dissolved solids (TDS). During drying, most mass loss is due to water but other volatile components (e.g., organics) may also be lost.

Undissolved Solids – solids excluding all interstitial liquid. The solids remaining if all the supernatant and dissolved solids associated with the supernatant could be removed from the bulk slurry. The calculated undissolved solids of the as-received tank waste generally include some materials that can be washed or dissolved during pretreatment.

Total Solids – the solids remaining after drying the bulk slurry at 105°C to a constant weight; includes dissolved and undissolved solids.

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1.0 Introduction

Battelle – Pacific Northwest Division is conducting physical property testing and inorganic, radiochemical, and selected organics characterization of waste from underground storage tank number 241-AZ-101 (hereafter designated as AZ-101) for Bechtel National, Inc. (BNI). This effort supports the planning and design of the RPP-WTP. Tank wastes must be compared to analyte and radioisotopic compositions described for Envelope B in Contract Specification 7 and for Envelope D in Contract Specification 8. It is important to characterize the compositions of tank wastes as specified in the envelopes to define pretreatment and separations processes before final stabilization (vitrification) of the waste. The RPP-WTP design flowsheets intend to use filtration to phase separate the liquid and solids fractions and to process the liquids and solids separately. Therefore, it is important to fully characterize both the liquids and solids in the tank waste before proceeding with final process design.

One core sample from Riser 59 was obtained from Tank AZ-101 between August 17 and August 28, 2000. The core sample consisted of eighteen 19-inch segments from the entire tank depth and two additional sludge segments from the bottom 39 inches of the tank. The core sample was obtained to satisfy requirements of ICD-23 (1) for process testing. The core sampling process required the addition of a hydrostatic head fluid (0.3 M LiBr solution) into the sampling system to displace the volume of waste sample removed. The AZ-101 core sample was obtained after tank mixing pumps were run in the tank in April and May of 2000(2). Data collected at the time of the mixer pump testing demonstrated that most of the sludge in the tank was suspended, and the sludge became well homogenized both vertically and horizontally. Although the solids settled to the tank bottom (forming the sludge layer) during the three to four months between mixing and sampling, the sludge in the core sample is considered representative of the tank sludge composition. The sludge (slurry) waste contained in AZ-101 is a candidate HLW feed and the liquid portion of the tank, after separation from the sludge, is a candidate LAW feed (Envelope B). Current characterization activity is to provide information for assessing AZ-101 waste for compliance with the LAW (Envelope B) and HLW feed specifications and to provide additional information for process testing.

Approximately 6.6 kg of AZ-101 waste, which was retrieved from the tank in August 2000, was used as starting material for this characterization task. Test specification 24590-PTF-TSP-RT-01-001, *Tank 241-AZ-101 Sample Composite, Homogeneity, and Analysis*, defined the work scope (see Appendix A). Test plan TP-RPP-WTP-089, *AZ-101 Homogenization and As-received Characterization*, was prepared by the PNWD to conduct the work scope defined in the test specification (see Appendix D1). The objectives of this work include:

- Receive and verify liquid samples were not corrupted with hydrostatic head fluid, which has a LiBr tracer.
- Composite liquid samples from segments 1 through 16 and sludge samples from segments 17, 17A, 18, and 18A and thoroughly mix them.
- Collect homogenized composite sub-samples in volume-graduated bottles.

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1 BNFL-5193-ID-23, Rev 4, Interface Control Document for Waste Treatability Samples, June 2000.

2 Preliminary Test 241-AZ-101 Mixer Pump Test, RPP-6548, Rev 0., July 2000.

- Verify the homogeneity of the sub-samples by measuring the volume of settled solids and supernatant in at least five sub-samples.
- Determine the wt% undissolved solids concentration of the composite.
- Perform PCB analysis per methods established for the Regulatory Data Quality Objective (DQO) task (deleted from scope per test exception 24590-WTP-TEF-RT-02-071 and 24590-WTP-TEF-RT-02-073)
- Adjust the undissolved solids content of sub-samples to 3±1 wt%, 13±2 wt%, and 20±2 wt%.
- Measure physical properties of the 3, 13, and 20 wt% sub-samples.
- Measure chemical and radioisotope concentrations listed in Tables 2 and 3 (of the test specification) in the wet centrifuged solids and supernatant fraction of the homogenized composite.
- Determine liquid fraction compliance to Contract Specification 7 (Envelope B) of DE-AC27-01RV14136 (Specification included in test specification, Appendix A).
- Determine solids fraction compliance to Contract Specification 8 of DE-AC27-01RV14136 (Specification included in test specification, Appendix A. Specification is in units of curies or grams per 100 grams non-volatile waste oxide. The solids results used for comparison to this specification are from the undissolved solids only; i.e., the solids remaining after mathematically removing the interstitial liquid contribution from the wet centrifuged solids.)
- Report supernatant and wet centrifuged solids analyses in accordance with Standard Electronic Format Specification for Tank Waste Characterization Data Loader.
- Provide a comprehensive technical report.

The PNWD Quality Assurance (QA) Program Plan "Conducting Analytical Work in Support of Regulatory Program" was used in support of all analytical operations and is compliant with the *Hanford Analytical Service Quality Assurance Requirements Documents* (HASQARD)(3). The inorganic, radioisotopic, and organic analytes tested were identified in the test plan. The analysis and quality control (QC) requirements for the evaluation of hydrostatic head fluid contamination and measurement of both the supernatant and wet centrifuged solids fractions were included in the test plan and transmitted to the laboratory staff via the Analytical Service Requests (ASR) 6132, 6193, and 6193.01 through 6193.06.

Results and data limitations, method modifications, and general observations are discussed in Section 8.0, Analytical Results. Initial sample processing, QC sample results, method detection limits (MDLs), and other QC indicators are described in the Section 9.0, Procedures, Quality Control, and Data Limitations.

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3 Hanford Analytical Services Quality Assurance Requirements Document; Volume 4: Laboratory Technical Requirements. DOE/RL-96-68, Rev 2, September 1998.

2.0 Sample Receiving

Following the tank sampling event in August 2000, the core segments were extruded and archived in glass bottles at the 222-S Laboratory. The core segment samples were archived for more than six months at 222-S prior to shipping to PNWD, and some evaporation of the contents may have occurred. To facilitate shipping of the core segments to PNWD, segments were combined into 500-mL shipping bottles as indicated in Table 2.1. Also, the 222-S Laboratory removed a small quantity of sludge from segments 17, 17A, 18, and 18A for testing. The combined core segment samples were shipped to PNWD between May 23 and May 31, 2001 under chain of custody (COC) (see Appendix B1).

Upon receipt at PNWD's High-Level Radiation Facility (HLRF), the AZ-101 waste samples were visually inspected, and the inspection was documented through test instruction TI-RPP-WTP-087, *AZ-101 Sample Inspection* (Appendix B2). Except for bottle 15958, which had a broken lid, all bottles and lids were in good condition. All liquid samples were similar in appearance, containing very small quantities (e.g., ¼ to 2 g) of white flaky solids in a clear single-phase liquid. The sludge samples were black in color with essentially no free-standing liquid. The results of the inspection are provided in Table 2.1.

Table 2.1. AZ-101 Waste Samples Received from 222-S Laboratory

Tank Segment Information				222-S Composite		PNWD Inspection	
Bottle Number	**Segment Number**	**Physical State**	**Net Weight (g)**	**Bottle Number**	**Net Weight (g)**	**Organic Layer**	**Appearance**
15958[(a)]	18	Solids	NA	15958	133.7	NA	About 5 mL of free clear liquid; black sludge; 50% full
17331	17ALH	Wet Sludge	129.4	16029	220.1	NA	No visible liquid; gooey black sludge
15888	17AUH	Sludge-Slurry	90.7				
17023	17	Solids	NA	17023	307.2	NA	Very small quantity of light liquid above black sludge
17025	18A	Solids	NA	17025	388.2	NA	Very small quantity of light liquid above black sludge
18396	1	Supernatant	344.6	18719	508.2	No	Clear liquid; very small amount of dark precipitate on bottom
18390	12	Supernatant	163.7				
18399	4	Supernatant	359.3	18720	522.2	No	Clear liquid; 1-2 g white and dark flaky solids on bottom
18408	13	Supernatant	163.1				
18397	2	Supernatant	352.1	18721	521.1	No	Clear liquid; 2 g of chunky white solids on bottom
18390	12	Supernatant	169.5				
18398	3	Supernatant	345.8	18722	521.9	No	Clear liquid; < ¼ g white fine solids on bottom
18390	12	Supernatant	30.4				
18408	14	Supernatant	146.4				

Table 2.1. (Cont'd)

Tank Segment Information				222-S Composite		PNWD Inspection	
Bottle Number	**Segment Number**	**Physical State**	**Net Weight (g)**	**Bottle Number**	**Net Weight (g)**	**Organic Layer**	**Appearance**
18394	5	Supernatant	366.6	18723	523.7	No	Clear liquid; < ¼ g white flakes on bottom
18408	13	Supernatant	40.5				
18400	14	Supernatant	116.8				
18391	6	Supernatant	361.8	18724	517.0	No	Clear liquid; < ½ g white flakes on bottom
18400	14	Supernatant	155.3				
18406	9	Supernatant	365.7	18725	516.4	No	Clear liquid; 1-2 g white solids on bottom
18401	15	Supernatant	106.5				
18413	16	Supernatant	44.2				
18403	11	Supernatant	365.3	18726	522.6	No	Clear liquid; < ½ g white flakes on bottom
18413	16	Supernatant	157.5				
18393	7	Supernatant	360.7	18727	519.9	No	Clear liquid; < ¼ g white fine solids on bottom
18400	14	Supernatant	58.0				
18401	15	Supernatant	102.1				
18407	8	Supernatant	366.1	18728	516.2	No	Clear liquid; < ¼ g white fine solids on bottom
18401	15	Supernatant	150.2				
18402	10	Supernatant	369.5	18729	521.1	No	Clear liquid; < ½ g white flakes on bottom
18413	16	Supernatant	151.7				
Sludge Solids Total (g)					**958.5**		
Supernatant Total (g)					**5800.9**		
Waste Total (g)					**6759.4**		

NA = not applicable

(a) Initial inspection determined lid to bottle 15958 was broken; lid was replaced prior to any further handling.

3.0 Evaluation for Hydrostatic Head Fluid Contamination

Prior to compositing, homogenization, and sub-sampling activities, the test specification requires all jars containing liquids received from 222-S to be evaluated for potential contamination from the hydrostatic head fluid used for extracting the core samples from Tank AZ-101. The hydrostatic head fluid contains a LiBr tracer; therefore, the liquids are analyzed for lithium by inductively-coupled plasma spectrometry (ICP-AES) and bromide by IC to determine if the liquids contain any appreciable quantity of hydrostatic head fluid. Per the test specification, any bottle with either the lithium or bromide concentration exceeding 0.003 molar was not to be included in the compositing process. However, after discussion with BNI, this exclusion criterion was changed to any bottle with the lithium and bromide concentration each exceeding 0.003 molar. Table 3.1 presents the results of the ICP-AES lithium analysis and the IC bromide analysis. The IC analysis was optimized specifically for bromide determination; however, the high nitrate concentration produced a significant interference and the reported results have a high uncertainty (e.g., ±25%). Since there is essentially no lithium in the liquids samples, the bromide concentration found in the liquids appears to be native to the AZ-101 tank waste. Based on the low lithium concentration, all the bottles in Table 3.1 and the remaining bottles containing solids were used for compositing.

Table 3.1. Lithium and Bromide Results on As-received Liquids

RPL Number	Bottle Number	Bromide		Lithium		Meets Acceptance Criteria [(a)]
		µg/mL	Molarity (M)	µg/mL	Molarity (M)	
01-1381	18729	1,200	0.015	< 1.2	< 0.0002	Yes
01-1382	18725	950	0.012	< 1.2	< 0.0002	Yes
01-1383	18726	1,200	0.015	< 1.2	< 0.0002	Yes
01-1384	18728	420	0.005	< 1.2	< 0.0002	Yes
01-1385	18727	920	0.012	< 1.2	< 0.0002	Yes
01-1386	18724	1,200	0.016	2.7	0.0004	Yes
01-1387	18723	200	0.003	< 1.2	< 0.0002	Yes
01-1388	18720	410	0.005	< 1.2	< 0.0002	Yes
01-1389	18722	1,10	0.014	< 1.2	< 0.0002	Yes
01-1390	18721	740	0.009	< 1.2	< 0.0002	Yes
01-1391	18719	230	0.003	< 1.2	< 0.0002	Yes

(a) Acceptance criteria: Both Br and Li concentration <0.003 M. The criterion of 0.003 M ensures that contamination is limited to 1 part hydrostatic head fluid in 100 parts tank waste.

4.0 Compositing and Sub-Sampling

The objective of compositing the AZ-101 samples is to provide homogeneous feed to tasks within the project. The homogenized feed is used to support the characterization task as well as process testing activities. The homogenization and sub-sampling activity was performed according to the test plan following the test instruction TI-RPP-WTP-095, "AZ-101 Sample Homogenization" (Appendix D2).

Following confirmation that the samples were not contaminated with hydrostatic head fluid, the contents of the 15 bottles of AZ-101 tank waste received from Hanford's 222-S Laboratory were homogenized and sub-sampled. The sample quantities transferred to the homogenization vessel are shown in Table 4.1.

Table 4.1. AZ-101 Samples Used for Preparing Composite

Initial Container ID (From 222-S)	Net Weight Mass Received (g)	Net Weight Transferred into Homogenization Vessel (g)
15958	133.7	127.5
16029	220.1	208.1
17023	307.2	274.9
17025	388.2	365.6
18719	508.2	501.6
18720	522.2	515.4
18721	521.1	511.6
18722	521.9	519.9
18723	523.7	518.0
18724	517.0	511.2
18725	516.4	512.0
18726	522.6	517.4
18727	519.9	516.6
18728	516.2	510.1
18729	521.1	512.9
Total:	**6759.4**	**6,622.8**
Loss on Transfer: 136.6 g (2.0%)		

The contents of each bottle were emptied into a homogenization vessel with a 0.125-inch screen placed over the top. Particles that accumulated on the screen were to be rinsed with supernatant and collected in a separate bottle labeled 'AZ-101>1/8'. However, no particles were observed to accumulate on the screen, and the 'AZ-101>1/8' sample was not collected. Once all the bottles were emptied, the lid was placed on the homogenization vessel, and the contents of the homogenization vessel were agitated via a dual-bladed impeller for approximately two hours at about 30°C (ambient hot cell temperature) to thoroughly homogenize the composite. The homogenization vessel 0.75-inch sampling valve was opened, and an initial sub-sample was extracted. This initial sub-sample was recycled back into the homogenization vessel to minimize the potential for collecting a large quantity of solids in the first sample due to dead-zones near the sampling valve. Sub-samples were collected in 18 pre-labeled bottles. All source bottles were weighed before and after the homogenization process and all sub-sampling bottles were weighed before and after sub-sampling. This allowed for a mass balance to be performed and sample losses due to residuals and evaporation quantified (see Table 4.1 and Table 4.2). About 6.8 kg of the sample were received from the 222-S Laboratory, about 6.6 kg of the sample was transferred into the homogenization vessel, and about 6.3 kg was extracted as 18 sub-samples. Sample losses due to evaporation and residuals in the source shipping bottles and the homogenization vessel are approximately 7%, which is consistent with previous homogenization efforts.

Table 4.2. AZ-101 Sub-sample Homogenization Information

Composite Sub-Sample ID	Net Weight (g)	Total Volume (mL)	Solids Volume (mL)	Vol % Solids (%)	Collection Order
AZ-101-PCB-1	167.3	130	24	18.5	3
AZ-101-PCB-2	157.8	127	25	19.7	4
AZ-101-PCB-3	162.4	128	25	19.1	5
AZ-101-CHEM-1	173.4	130	37	28.5	6
AZ-101-CHEM-2	156.0	123	23	18.7	7
AZ-101-CHEM-3	157.3	125	25	19.6	8
AZ-101-CHEM-4	176.8	130	25	19.2	9
AZ-101-RHEO-3%	154.3	123	23	18.7	10
AZ-101-RHEO-13%	307.9	242	43	17.7	2
AZ-101-RHEO-20%	486.3	381	68	17.8	1
AZ-101-AR-A	527.8	420	69	16.5	11
AZ-101-AR-B	567.9	458	68	14.8	12
AZ-101-AR-C	566.4	458	68	14.8	13
AZ-101-AR-D	580.8	458	62	13.5	14
AZ-101-AR-E	582.1	473	62	13.1	15
AZ-101-AR-F	586.9	473	59	12.5	16
AZ-101-AR-G	581.4	473	62	13.1	17
AZ-101-AR-H	208.3	174	24	13.6	18
Total Sub-sampled:	**6,301.1**	**5,026**			
Loss from Compositing [(a)]: 458.3 g (6.8%)					

(a) Total loss includes residuals retained in source shipping bottles, evaporations losses, and residual retained in homogenization vessel following sub-sampling.

The sub-samples settled for 24 hours and total volume and settled solids volume were measured (see Table 4.2). No floating organic layer was observed. The test specification has an administrative hold point requiring the evaluation of the vol% settled solids data and approval from BNI prior to using the sub-samples for characterization and process testing. A plot of vol% settled solids versus sub-sample collection order was presented to BNI and approval to proceed was obtained. Note that since additional sludge segments had to be obtained from Tank AZ-101 to meet the minimum quantity of solids needed for process verification testing, the homogenized composite does not have the same liquid-solids ratio as that of Tank AZ-101.

5.0 Physical Measurement

The AZ-101 material was tested for select physical and rheological properties using methodology defined in Sections 4.0 and 5.0, respectively, of *Guidelines for Performing Physical and Rheological Properties Measurements*.(4) The composite material from bottles AZ-101-RHEO-3%, AZ-101-RHEO-13%, and AZ-101-RHEO-20% were selected for further physical properties measurements, including density, vol% solids, and wt% solids, rheology, heat capacity, particle size, and scanning electron microscopy (SEM) for particle shape.

5.1 Sample Preparation for Physical Measurements

According to the test plan, analyses were to be carried out on samples prepared to 3-wt%, 13-wt%, and 20-wt% undissolved solids. Preparing these samples involved determining the wt% undissolved solids in the as-received material, using this value to calculate the mass of supernatant to be removed to prepare the 3-wt%, 13-wt%, and 20-wt% samples. A work flow diagram illustrating the steps performed to determine the amount of solids in each physical properties measurement is shown in Figure 5.1.

Initial physical properties measurements on as-received material from bottles AZ-101-CHEM-2 and AZ-101-AR-E produced a value of approximately 1.5-wt% undissolved solids. This undissolved solids value (sample A in Figure 5.1), later determined to be incorrect, was used to calculate the volume of supernatant that needed to be removed to achieve the target undissolved solids contents of 3 wt%, 13 wt% and 20 wt%. Based on this value it was determined that the settled solids layer in the as-received samples should correspond to 8-wt% undissolved solids. Consequently, 13-wt% and 20-wt% undissolved solids were believed to be unattainable and further instruction from BNI was sought. Direction was provided from BNI to proceed by decanting half of the supernatant from the as-received samples to reach an estimated value of 3-wt% undissolved solids. This corresponds to sample B in Figure 5.1. BNI also directed further analyses to be performed on the settled solids layer (sample C in Figure 5.1), which would be approximately 8-wt% undissolved solids based on the incorrect starting value of 1.5-wt% undissolved solids. Rheology measurements were performed on these two sets of material (i.e., half the supernatant removed and settled solids layer).

Additional physical properties measurements were performed on the material with half the supernatant removed and on the settled solids layer. The resulting wt% undissolved solids values for these samples did not correlate with the predicted values of 3-wt% and 8-wt% undissolved solids. Sub-sampling techniques were determined to be cause of this problem and a Corrective Action Report (CAR # 4181) was issued (see Appendix E). However, the rheology measurements had already been performed with sample B and sample C. These were later calculated to have been at approximately 14-wt% and 45-wt% undissolved solids, respectively.

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4 Procedure: Technology Development, *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*, Bechtel 24590-WTP-GPP-RTD-001, Rev 1, April 19, 2002.

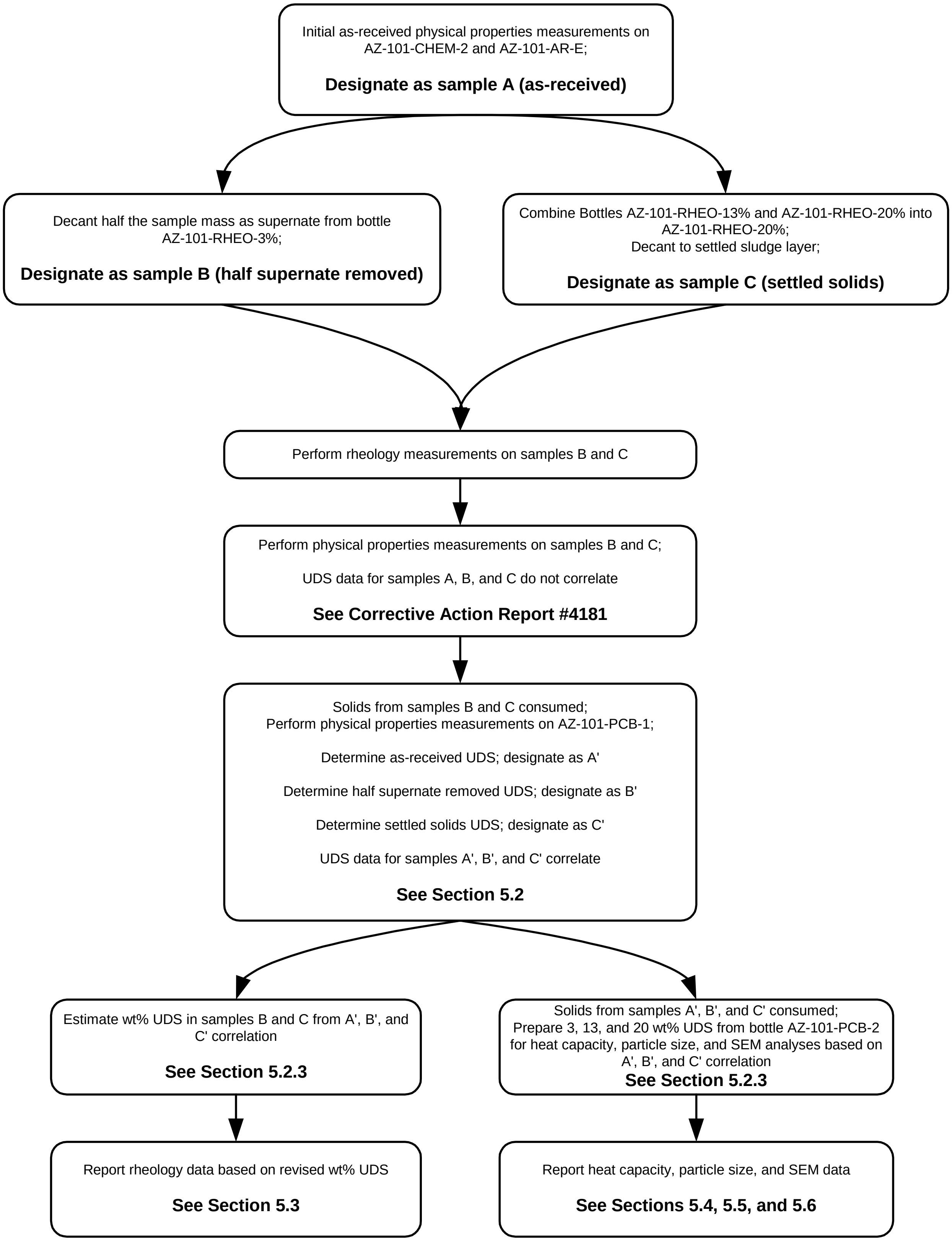


Figure 5.1. Physical Properties Sample Preparation Work Flow Diagram

With the sub-sampling problem addressed, further physical properties measurements were performed on as-received homogenized composite from bottle AZ-101-PCB-1. Weight percent undissolved solids was determined on the following material:

- As-received material (sample A′ in Figure 5.1)
- Half the supernate removed (sample B′ in Figure 5.1)
- Settled solids layer (sample C′ in Figure 5.1).

The results from these measurements appear to correlate with one another and can be found in Section 5.2.2. Based on this correlation, the wt% undissolved solids for the samples used for the rheology measurements were calculated. The correlation was also used to prepare sub-samples from bottle AZ-101-PCB-2 at 3-wt%, 13-wt%, and 20-wt% undissolved solids, which were used in heat capacity, PSD, and SEM. A discussion of the use of this correlation can be found in Section 5.2.3.

5.2 Selected Physical Properties

The physical properties listed in Table 5.1 were measured on the homogenized 'as-received' composite, a midrange slurry with approximately half the supernatant decanted, and a settled solids sample generated by decanting all standing supernatant (i.e., only interstitial liquid remained in the settled solids sample).

Table 5.1. Physical Properties Measurements

Physical Property Measured
Slurry density
Liquid density
Vol% centrifuged solids
Wt% centrifuged solids
Wt% total dried centrifuged solids
Wt% total dissolved solids
Wt% undissolved solids

5.2.1 Method of Analysis

Physical properties listed in Table 5.1 were measured on material taken from bottle AZ-101-PCB-1 sub-sampled from the homogenized as-received composite (see Section 4.0). The wt% oxide measurements required for comparison of the undissolved solids to Contract Specification 8 were conducted as part of the inorganic characterization testing (see Section 6.0).

The AZ-101 slurry sample was stirred using a magnetic stir bar while 5-mL to 10-mL sub-samples were transferred into three graduated centrifuge tubes. The mass and volume of material in each tube was recorded. The tubes were then centrifuged for one hour at approximately 1000 G. The total sample volume and volume of centrifuged solids were recorded. The standing supernatant was decanted into a graduated cylinder of known mass. The mass and volume of supernatant in the cylinders were recorded, as was the mass of centrifuged solids left in the centrifuge tubes. The supernatant was transferred to a preweighed vial and reweighed. The vials (containing primarily liquid) and centrifuge tubes (containing primarily solids with interstitial liquid) were transferred to an oven at 105°C for at least 24 hours. The vials and centrifuge tubes were reweighed periodically until a stable mass was reached (e.g., change in mass is less than 0.1% in 24 hours).

5.2.2 Results

Three samples were prepared (see Section 5.1), and the physical properties were analyzed. The first sample was the AZ-101-PCB-1 sample from the homogenized as-received composite. The results from the procedure described in Section 5.2.1 are shown in Table 5.2. Density and wt% total dissolved solids (TDS) measurements of the supernatant were not performed on this sub-sample, as these measurements were included as part of the inorganic analyses of the supernatant (see Table 6.2). The second sample was created by decanting approximately half of the supernatant from AZ-101-PCB-1. These results are shown in Table 5.3. The third sample was created by decanting and pipetting the supernatant from the AZ-101-PCB-1 bottle such that no further clear supernatant could be removed. However, a small volume of clear supernatant was observed in the AZ-101-PCB-1 source bottle prior to mixing and sub-sampling, and therefore contained a level of solids slightly below the actual settled sludge value. The results from the third sample are shown in Table 5.4.

Table 5.2. Physical Properties Measurement of As-received AZ-101 Composite

Description	Aliquot			Average [(a)]	RSD
	A	B	C		
Slurry: density (g/mL)	1.24	1.27	1.25	1.25	1%
Supernatant: density (g/mL)	--	--	--	--	--
Slurry: centrifuged solids (vol%)	15.3	15.6	13.9	15.0	6%
Slurry: centrifuged solids (wt%)	19.8	17.6	16.6	18.0	9%
Slurry: total dried solids (wt%)	33.4	32.8	32.4	32.8	2%
Supernatant: total dissolved solids (wt%)	--	--	--	--	--
Undissolved solids (wt%)	8.3	7.5	6.9	7.6	9%

(a) Average is calculated using more digits than presented for A, B, and C in this table.

Table 5.3. Physical Properties Measurement of AZ-101 Composite After Decanting Half the Supernatant

Description	Aliquot			Average [(a)]	RSD
	A	B	C		
Slurry: density (g/mL)	1.35	1.33	1.33	1.33	1%
Supernatant: density (g/mL)	1.27	1.25	1.24	1.25	1%
Slurry: centrifuged solids (vol%)	31.0	29.1	31.0	30.4	3%
Slurry: centrifuged solids (wt%)	38.5	38.1	38.7	38.4	1%
Slurry: total dried solids (wt%)	38.9	39.6	40.0	39.5	1%
Supernatant: total dissolved solids (wt%)	26.7	26.7	26.6	26.7	0%
Undissolved solids (wt%)	17.1	17.3	17.4	17.2	1%

(a) Average is calculated using more digits than presented for A, B, and C in this table.

Table 5.4. Physical Properties Measurement of AZ-101 Settled Solids

Description	Aliquot A	Aliquot B	Aliquot C	Average [(a)]	RSD
Slurry: density (g/mL)	1.54	1.59	1.60	1.58	2%
Supernatant: density (g/mL)	1.23	1.28	1.46	1.32	9%
Slurry: centrifuged solids (vol%)	66.3	63.2	68.3	65.9	4%
Slurry: centrifuged solids (wt%)	74.0	73.0	75.4	74.1	2%
Slurry: total dried solids (wt%)	55.1	56.1	56.1	55.8	1%
Supernatant: total dissolved solids (wt%)	28.4	27.4	28.2	28.0	2%
Undissolved solids (wt%)	37.7	39.4	40.2	39.1	3%

(a) Average is calculated using more digits than presented for A, B, and C in this table.

5.2.3 Discussion and Impacts on Sample Preparation

With the wt% undissolved solids determined at various solid loadings, a relationship between the amount of supernatant present in the sample and wt% undissolved solids can be determined. This relationship should be linear and behave according to Equation 5.1.

$$P_1 = \frac{P_0 M_0}{M_1} \tag{5.1}$$

where: P_1 is the final undissolved solids (wt%)
P_0 is the initial undissolved solids (wt%)
M_1 is the final decanted sample mass (g)
M_0 is the initial sample mass (g).

Using the nine points discussed in Table 5.2 through 5.4, the data were plotted, and a line of best fit was found. This plot can be seen in Figure 5.2. From this best fit line, the as-received wt% undissolved solids content ($M_0/M_1 = 1$) is approximately 7%, and the gravity-settled solids wt% undissolved solids content ($M_0/M_1 = 5.3$) is approximately 38%.

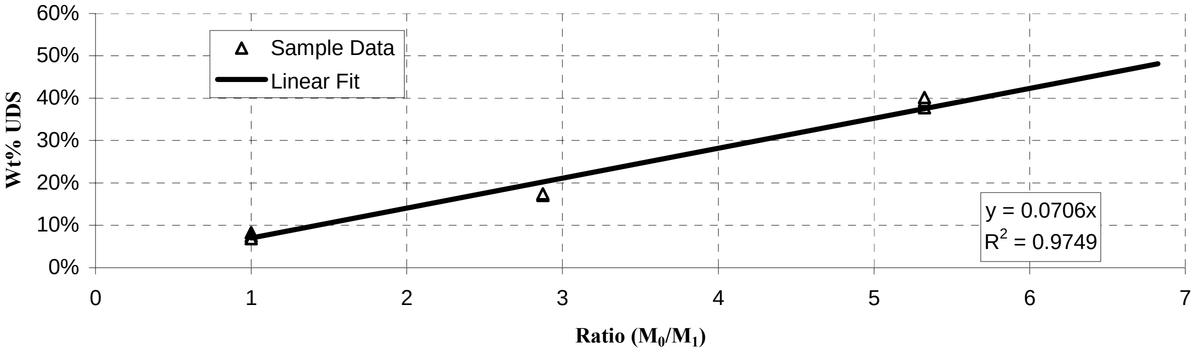


Figure 5.2. Wt% Undissolved Solids as a Function of Mass (Initial M_0 and After Supernatant Removal M_1)

Based on this correlation, the wt% undissolved solids of the rheology samples analyzed in Section 5.3 can be determined. The calculations for the rheology samples are shown in Table 5.5 and Table 5.6.

Table 5.5. Wt% Undissolved Solids Calculation for Rheology Sample (Half Supernatant Removed)

Description	Value	Units
Initial mass of AZ-101-RHEO-3% (M_0)	154.26	g
Mass after decant (M_1)	77.41	g
Ratio (M_0/M_1)	1.99	--
Estimated undissolved solids based on Figure 5.2	14	wt%

Table 5.6. Wt% Undissolved Solids Calculation for Rheology Sample (Settled Solids)

Description	Value	Units
Initial mass of sample AZ-101-RHEO-20%	416.18	g
Mass of sample AZ-101-RHEO-13% added to Sample AZ-101-RHEO-20%	296.97	g
Total initial sample mass (M_0)	713.15	g
Mass after decant (M_1)	112.5	g
Ratio (M_0/M_1)	6.34	--
Estimated undissolved solids based on Figure 5.2	45	wt%

The samples prepared for heat capacity, particle size, and SEM analyses were created by decanting an as-received sample (AZ-101-PCB-2) to 32-wt% undissolved solids. Various amounts of this high-solids sample and supernatant were combined to produce 3-wt%, 13-wt%, and 20-wt% undissolved solids subsamples. This calculation can be seen in Table 5.7.

Table 5.7. Wt% Undissolved Solids Calculation for Heat Capacity, Particle Size, and SEM Samples

Description	Value	Units
Preparation of 32-wt% Sample		
Initial mass of AZ-101-PCB-2 sample (M_0)	156.41	g
Mass after decant (M_1)	34.77	g
Ratio (M_0/M_1)	4.50	--
Wt% undissolved solids of Source AZ-101-PCB-2 Sample	32	wt%
Preparation of 3-wt% Sample		
Mass of 32-wt% undissolved solids sample added	1.49	g
Final mass after adding supernatant	16.08	g
Calculated wt% undissolved solids in sub-sample	2.9	wt%
Preparation of 13-wt% Sample		
Mass of 32-wt% undissolved solids sample added	3.49	g
Final mass after adding supernatant	8.69	g
Calculated wt% undissolved solids in sub-sample	13	wt%

Table 5.7 (Cont'd)

Description	Value	Units
Preparation of 20-wt% Sample		
Mass of 32-wt% undissolved solids sample added	4.52	g
Final mass after adding supernatant	7.20	g
Calculated wt% undissolved solids in sub-sample	20	wt%

5.3 Rheology

The rheology testing of the AZ-101 homogenized composite was performed in the HLRF A-cell using the Haake™ M5 rheometer. The primary tests were standard shear stress versus shear rate curves.

5.3.1 Background on Basic Rheology Testing

Viscosity is the internal resistance to flow of a fluid against external forces. Mathematically, viscosity is defined as the ratio of shear stress to shear rate. For a Newtonian fluid, this value is constant. For non-Newtonian fluids, this ratio can change based on flow conditions and shear history. The rheological data is most often presented as a rheogram. Rheograms provide flow data over a range of shear rates rather than at one shear rate. A rheometer ramps up the shear rate to a chosen value while measuring and recording the resulting shear stress. This is the primary difference between a rheometer and a viscometer. From rheogram viscosity data, yield stress data and flow curve information are obtained. Viscosity is usually reported in centipoise (cP). One cP is equal to a millipascal-second (mPa-s). There are several types flow curves that have been well studied and have defined mathematical curve fits assigned to them. These curve fits are usually used to describe and predict flow behaviors of fluids. Some materials have a yield point or minimal external force that must be applied before any flow is obtained. The four curve fits that best describe most slurries, and consequently tank waste, are as follows (Chhabra, R. 1999):

1) Newtonian ……………………………............................ $\tau = \eta * \gamma$

2) Bingham Plastic: ………………………………................. $\tau = \tau_0 + \eta_p * \gamma$

3) Ostwald (Pseudo-plastic or Power Law Fluid): ………..$\tau = \eta_p * \gamma^n$

4) Herschel-Bulkley (Yield Pseudo-plastic): ………………$\tau - \tau_0 = \eta_p * \gamma^n$

where: τ = Shear Stress (Pascal, Pa)

τ_0 = Yield Point (Pascal, Pa)

γ = Shear Rate (per second, s^{-1})

η = Viscosity (Pascal-seconds, Pa-s; reported in cP)

η_p = Coefficient related to flow resistance; i.e., apparent viscosity. $\eta_p = \eta$ for Newtonian fluid

n = Power law factor

$n = 1$ for Newtonian fluid

$n > 1$ for dilatant fluid

$n < 1$ for pseudo-plastic fluid.

Newtonian fluids are the classic fluids like water and honey. The viscosity is a constant over all shear conditions. A Bingham plastic is a fluid that contains a yield point. Once enough force has been applied to exceed the yield point, the material behaves in a Newtonian fashion over the rest of the shear rate range. A pseudoplastic, or power law fluid, has a viscosity that varies with stress in a non-linear fashion and is modeled by the Ostwald equation. A dilatant fluid has a viscosity that increases with shear rate. A yield pseudoplastic is a power law fluid with a yield point and is modeled by the Herschel-Bulkley equation.

5.3.2 Equipment Capabilities and Sensor Selection

A Haake™ M5 rheometer (modified for hot cell operation) was used for this work. The M5 system is a cup and bob (Serle) rotational system. The sensor, a cylinder of known geometry with a specified gap in the appropriate cup, is turned within a fluid. The resulting fluid resistance to the flow causes a small movement in a torsion bar mounted between the motor and the drive shaft that is measured by an electronic transducer. This signal is read and combined with the rate of spin information to produce the shear stress and shear rate data. The Haake™ M5 head design specifications give it a maximum deflection of 1% at full torque with sensitivity ranging to 0.001 degrees for low-viscosity fluids. It has a maximum toque range of 4.9 Newton-centimeters and rotational speed capability of 0.05-500 rpm. This combined with the sensor geometry determines the optimum apparent viscosity and shear rate ranges available for any given measurement. For the AZ-101 samples in this report, the MV1 sensor system was utilized. This sensor has a large available surface area for sensitivity but also a gap large enough to allow for fairly concentrated slurries to be measured. This measurement head and sensor combination has an optimum apparent viscosity range of $10\text{-}10^5$ cP and can measure over a shear rate range of approximately 0.1 to 1150 s^{-1}.

A 48.4 cP standard oil was used to validate the calibration of the rheometer. A value of 44.4 cP was measured, and the plot is shown in Figure 5.3.

5.3.3 Samples

Two AZ-101 samples were prepared at different wt% undissolved solids levels. The first sample was prepared by decanting half the total sample mass off as supernatant. This approximately doubles the as-received undissolved solids to 14 wt%. The second sample was prepared by removing the supernatant until the settled solids layer was reached (approximately 45-wt% undissolved solids). Additional information on the preparation of these samples can be found in Sections 5.1 and 5.2.3. As detailed in these Sections, the original target concentrations for testing were 3-wt%, 13-wt% and 20-wt% undissolved solids. However, due to an error in the original undissolved solids content measurements it was believed that these targets were unreachable with the amount of material on hand. So the rheology was performed on two samples: one with half the supernatant removed and one consisting only of the settled solids. Once corrective actions had been taken to obtain the correct undissolved solids content on the as-received material, there was insufficient material to repeat the rheology measurements. Thus, the data collected is being reported even though the measurements were not performed at the target wt% undissolved solids contents.

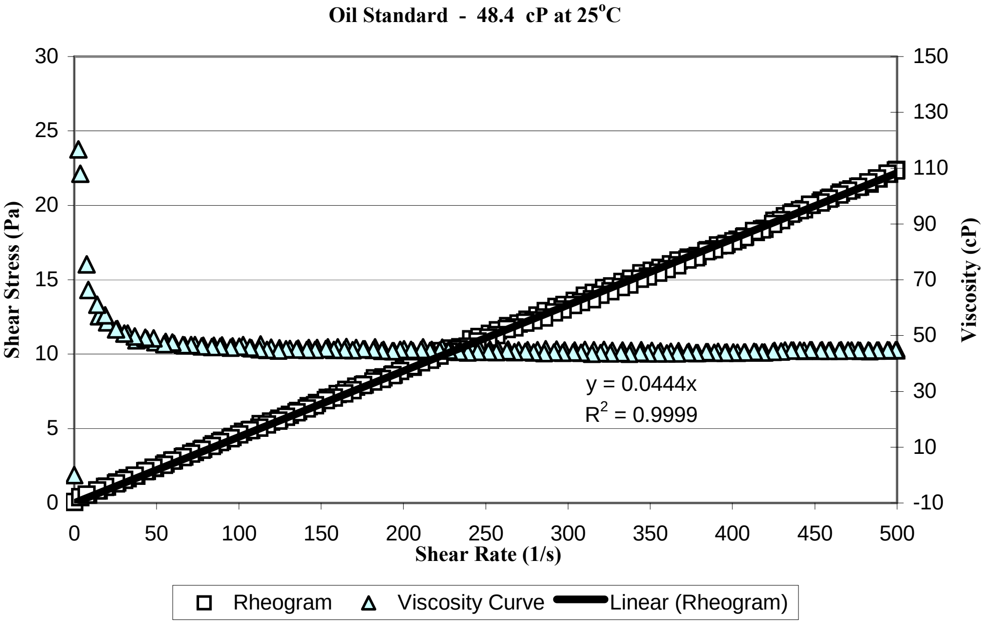


Figure 5.3. Haake™ M5 Viscosity Standard Calibration Check

The AZ-101 samples were loaded and then ramped up from 0 to 1000 s^{-1} in 5 minutes and then from 1000 to 0 s^{-1} in 5 minutes. This ramp cycle was repeated at least once for each sample. Therefore each sample was tested through a minimum of two complete ramp cycles from 0 to 1000 s^{-1} then 1000 to 0 s^{-1} over a total time of 20 minutes. If the second set of run data was a close overlay of the first set of run data, the testing for that sample was considered complete. If there was noticeable variation in the data, the sample was ramped through this cycle again until two consecutive similar data sets were obtained. The purpose of this repetition was to determine if rheological changes are made to the material while under the influence of shear. Shear history is often an important part of determining expected rheological behaviors.

Specific testing parameters were used to identify the rheological behavior and shear sensitivity of the AZ-101 material at different undissolved solids loadings. The first ramp cycle shows newly loaded or fresh sample behavior including breakdown of sample structure through hysteresis, if present. Hysteresis is when the ramp down curve is different from the ramp up curve. An immediate repeat allows little or no time for the sample to recover. The complete cycle repeat shows the effects of a shear history with a short time of recovery for the sample. Once two ramp cycles displayed consistent behavior, the sample was removed, and the analysis was repeated with another sample.

The 14-wt% material was tested at two separate temperatures (25°C and 40°C) to determine temperature effects on viscosity and flow curves at possible expected processing conditions. Due to sample limitations, the 45-wt% settled solids material could only be measured at 25°C.

5.3.4 Results

Each run of the AZ-101 14-wt% undissolved solids sample showed a strong Bingham plastic behavior with little or no yield point. The yield point is low enough ($\tau_0 < 0.3$ Pa) for the fluid to be considered nearly Newtonian in nature. The detection limit for yield stress data is 2 Pa. The apparent viscosity (η_p) ranged from 2 to 4 cP in the fully developed flow range. At lower shear rates (i.e., < 100 s^{-1}), some apparent viscosity (up to 10 cP) was observed. However, this apparent viscosity may be an artifact of the mechanical inertia of the measuring system, since the MV1 sensor is not designed for quantification below 10 cP. In all of the 14-wt% undissolved solids runs, Taylor Vortices developed at around 500 s^{-1}. Taylor Vortices are flow patterns that can develop in cup and bob systems with low viscosity fluids above certain shear rates (see Section 5.3.7). The mathematical basis used to create a rheogram is not valid for this flow regime and the data in this range are inaccurate. There was also no significant hysteresis in any of the runs. The sample reruns were consistent, showing good repeatability and little to no lasting shear effects on the material. Rheograms of these runs at 25°C and 40°C with corresponding Bingham plastic model fits are shown in Figure 5.4 and Figure 5.5, respectively.

The AZ-101 45-wt% undissolved solids samples also behaved as a Bingham plastic. Each sample produced reproducible repeat runs. Samples 1 and 2 also were consistent with one another. Sample 3 showed a higher yield and apparent viscosity (η_p), possibly due to drying or variations in sub-sampling. The 45-wt% undissolved solids AZ-101 appears to have a yield factor in the range of 4-5 Pa. The apparent viscosity (η_p) of the 45-wt% undissolved solids AZ-101 slurry ranged from about 10-20 cP. Rheograms of these runs with corresponding Bingham plastic model fit are shown in Figure 5.6.

5.3.5 Temperature and Concentration Effects

It would be expected that the fluid resistance and apparent viscosity would decrease with an increase in the fluid temperature. However, the 14-wt% undissolved solids material rheology was dominated by the water content of the slurry, and while it is known that water viscosity decreases with increasing temperature, this level of precision is beyond the capacity of the Haake™ M5 measuring system. Thus, no discernable temperature effects were seen. The change in temperature also did not change the flow profile. The increase of solids content to 45-wt% undissolved solids increased both the yield point and the apparent viscosity parameters. Taylor Vortices were not observed during these runs. However, the higher solids content caused several data spikes and some hysteresis, especially during the first run of each sample. In order to compare the temperature and solids content effects simultaneously, summaries of the Bingham plastic model fit under each of the run conditions are shown in Table 5.8.

Table 5.8. Bingham Plastic Model Fit for AZ-101 Slurries at Various Measurement Conditions

	14-wt% UDS at 25°C			14-wt% UDS at 40°C			45-wt% UDS at 25°C		
Sample	**Yield Point, τ_0 (Pa)**	**Apparent Viscosity, η_p (cP)**	**R^2**	**Yield Point, τ_0 (Pa)**	**Apparent Viscosity, η_p (cP)**	**R^2**	**Yield Point, τ_0 (Pa)**	**Apparent Viscosity, η_p (cP)**	**R^2**
Run 1-1	0.28	2.2	0.96	0.062	2.5	0.95	NM	NM	NM
Run 1-2	0.18	2.8	0.99	0.16	2.6	1.00	4.0	11	0.95
Run 1-3	NM	NM	NM	NM	NM	NM	4.0	11	0.95
Run 2-1	0.25	3.0	0.97	0.17	2.6	0.98	2.4	11	0.92
Run 2-2	0.21	3.0	0.99	0.17	2.6	0.99	4.0	10	0.86
Run 2-3	NM	NM	NM	NM	NM	NM	2.9	11	0.93
Run 3-1	0.22	3.5	0.99	0.16	2.5	0.99	4.7	17	0.93
Run 3-2	0.22	3.0	0.99	0.17	2.4	0.99	3.0	18	0.98
Run 3-3	NM	NM	NM	NM	NM	NM	4.4	16	0.95

UDS = undissolved solids; NM = not measured

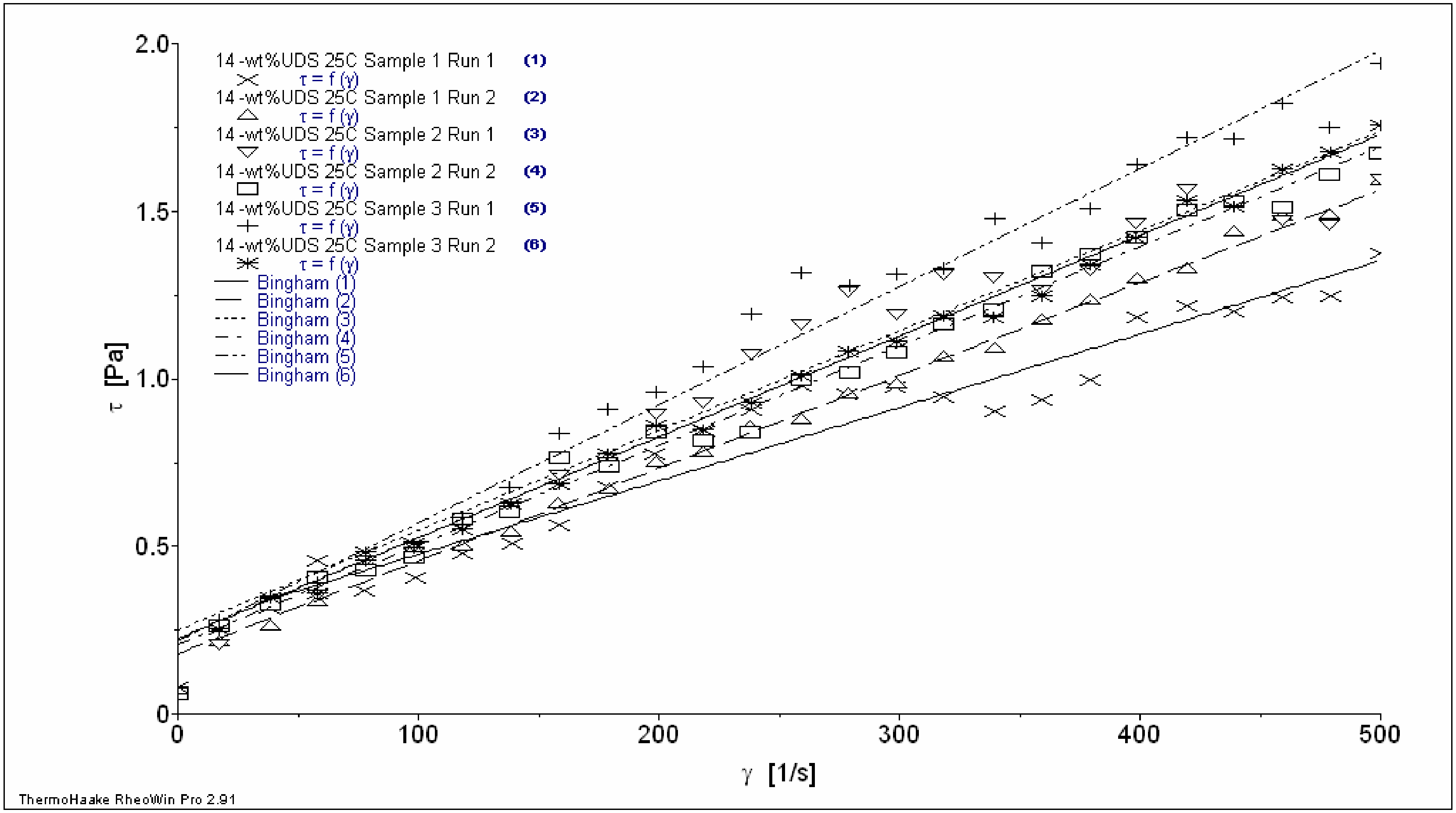


Figure 5.4. Rheograms of 14-wt% Undissolved Solids AZ-101 Slurry at 25°C

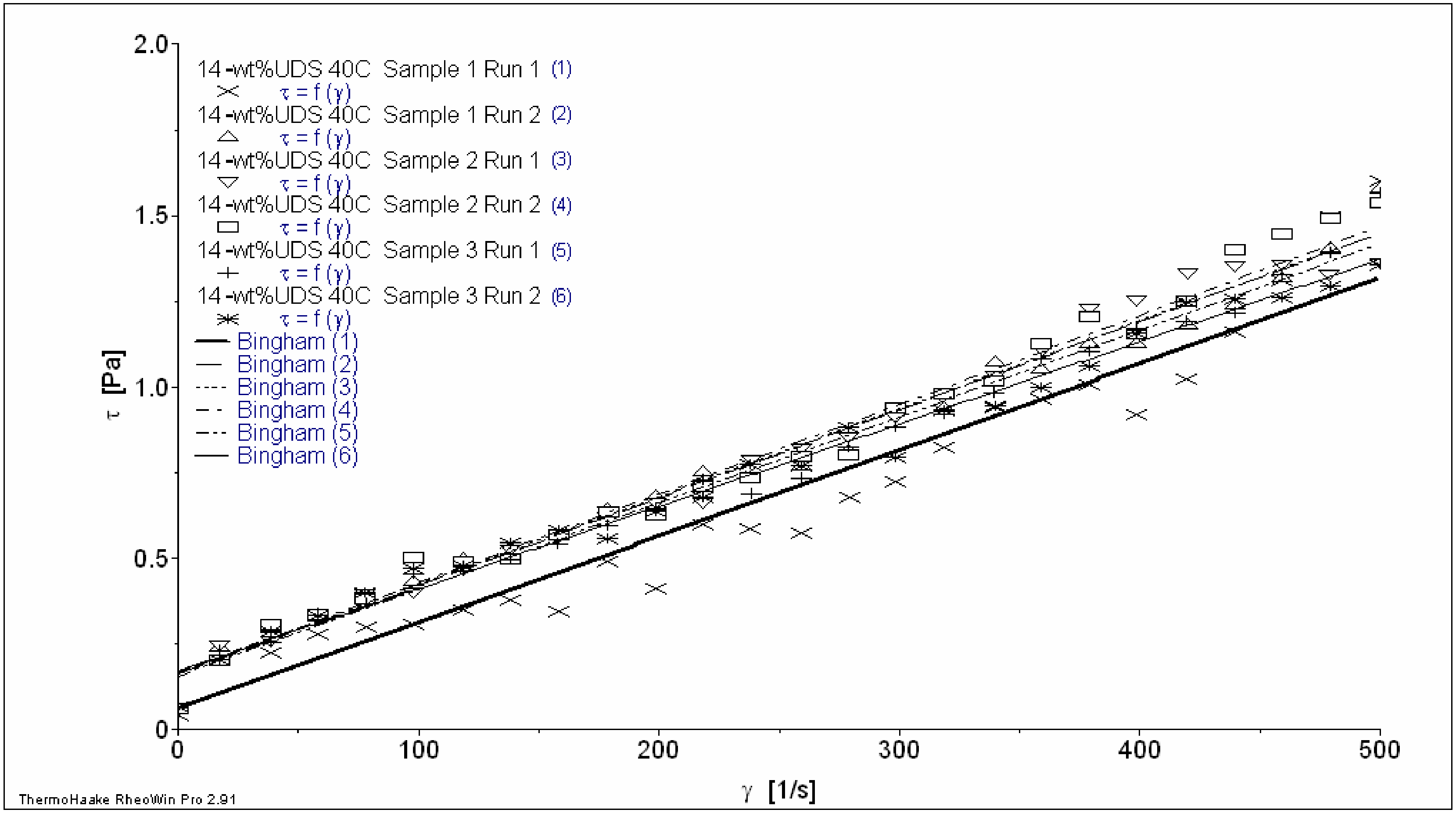


Figure 5.5. Rheograms of 14-wt% Undissolved Solids AZ-101 Slurry at 40°C

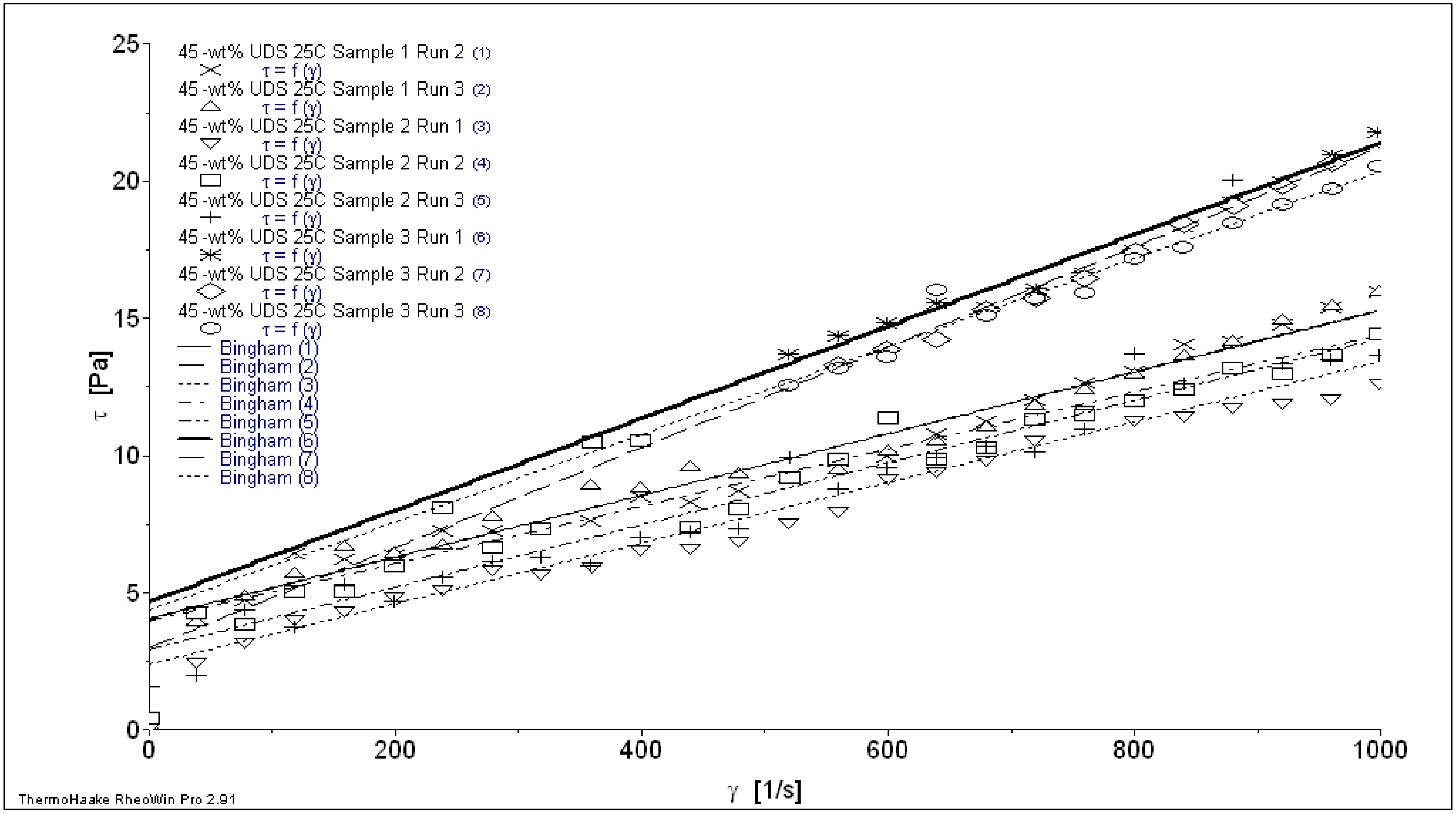


Figure 5.6. Rheograms of 45-wt% Undissolved Solids AZ-101 Slurry at 25°C

5.3.6 Data Limitations

The system has a mechanical 'start-up' resistance that can cause a false yield stress in very low viscosity fluids. This can be attributed to energy input required to overcome the inertial forces of the sensor rather than actual fluid resistance. It is likely that this contributed to the 'yield stress' seen in these samples. For the 14-wt% undissolved solids samples any yield stress is most likely a mechanical artifact. However, in the 45-wt% undissolved solids samples the nature of the curves indicates a real yield stress does exist, and the inertial forces only slightly affect the quantification in these samples.

There was some scatter, especially in the low shear ranges that can lead to misleading apparent viscosity numbers if only 'single points' are referenced. Therefore, it is best to use the curve equations obtained for the entire run to predict viscosities at any given shear rate rather than using singular data points, especially for the high solids data.

5.3.7 Taylor Vortices

Taylor Vortices are the result of a secondary flow that occurs as the inner cylinder of the concentric cylinder rheometer rotates. Taylor Vortices result from subjecting a material at too high a shear rate during analysis. All data collected above the onset of Taylor Vortices are invalid. Using the following set of variables, a criterion for the onset of Taylor vortices can be derived.

$$T_a = \mathrm{Re}\left(\frac{R_o - R_i}{R_o}\right)^{1/2} = \text{Talyor Number}$$

$$\mathrm{Re} = \frac{U_i \rho (R_o - R_i)}{\mu} = \text{Reynolds Number}$$

R_o = outer radius of cup

R_i = inner radius of cylinder

$U_i = \dot{\Omega}_i R_i$ = rotational velocity of the inner cylinder

$\dot{\Omega}_i$ = inner cylinder angular velocity

μ = viscosity

γ = shear rate

ρ = sample density

According to Schlichting (1979) flow instabilities arise when the following condition is met:

$$T_a \geq 41.3$$

The shear rate is defined as the derivative of the velocity profile of the fluid:

$$\gamma = -\frac{dv}{dr}$$

where:

v = velocity

r = radius

In the limit of a small gap ($\lim dr \to 0$) the flow between the concentric cylinders approaches the flow between two parallel plates. One of the plates is stationary and the other is moving at velocity U_i. In this case, the average shear across the gap can be approximated by an algebraic equation where $dv = \Delta v$ and $dr = \Delta r$. The change in velocity can be calculated by the difference in plate velocities ($\Delta v = 0 - U_i$). And the distance between these plates is the gap distance ($dr = R_o - R_i$). Combining these equations relates the rotational speed of the sensor system to the shear rate.

$$U_i = \gamma (R_o - R_i)$$

This equation can then be placed in the Reynolds Number (Equation 5.2).

$$\mathrm{Re} = \frac{\gamma (R_o - R_i)^2 \rho}{\mu} \tag{5.2}$$

Finally, this Equation 5.2 can be combined with the Taylor instability criterion to provide a criterion for data rejection.

$$\frac{\gamma (R_o - R_i)^{\frac{5}{2}} \rho}{41.3 \mu R_o^{\frac{1}{2}}} \geq 1 \tag{5.3}$$

When the criterion in Equation 5.3 is met, the data generated above this point should not be considered reliable. However, many assumptions and measurements are used in the application of this equation. Therefore, this equation should only be used to estimate when Taylor Vortices may occur. Typically the onset of this behavior can be seen in the rheograms and data above this observed value should be discarded.

This can be illustrated by examining a rheogram discussed in Section 5.3.4 (See Figure 5.5). Using the derived criterion, onset of Taylor Vortices is predicted at 430 s^{-1}. However, Taylor Vortices are observed at 500 s^{-1} and data above 500 s^{-1} were not used.

Rheogram: 14-wt% UDS AZ-101 at 40 deg C Sample 3 Run 2

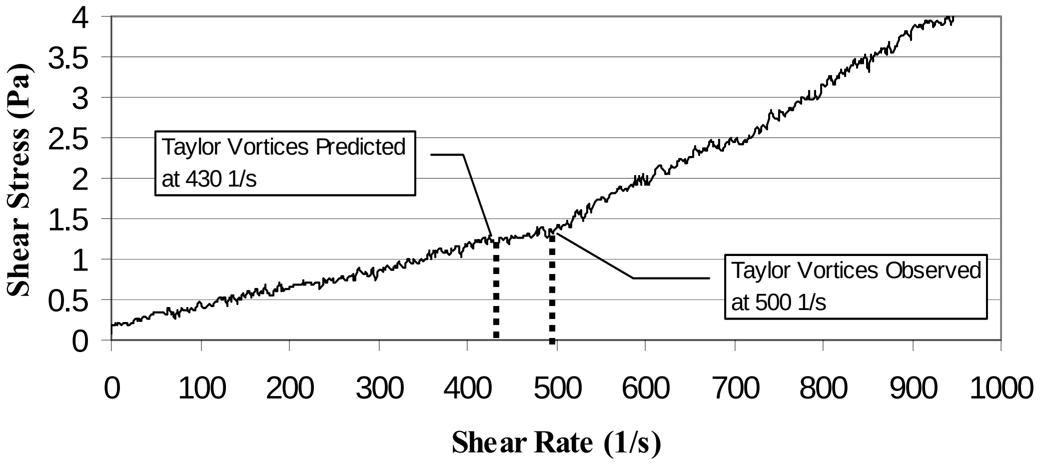


Figure 5.7. Onset of Taylor Vortices

5.4 Heat Capacity Measurement

A differential scanning calorimeter (DSC) was used to measure the heat capacity (i.e., amount of heat or enthalpy required to raise 1 g of a material 1°C) on AZ-101 slurries with varying wt% undissolved solids content. The three AZ-101 slurries analyzed were as-received AZ-101 composite adjusted to 3-wt%, 13-wt%, and 20-wt% undissolved solids; prepared as described in Section 5.2.3.

5.4.1 Background

For heat capacity measurement using the DSC, the instrument manufacturer prescribes a three-step temperature program. First the empty sample pan is heated or cooled to the starting temperature, held for 10 minutes, then heated over the temperature range of interest at a controlled rate, and held at the final temperature for 10 minutes. Second, the sample pan is filled with roughly the same amount of reference material as to be used for the sample and the same temperature program repeated. The third step is to remove and replace the reference material with the sample and then to repeat the same temperature program.

The heat capacity of the sample is calculated using Equation 5.4, which employs: 1) differences between the reference and the baseline, 2) differences between the sample and the baseline, and 3) differences in reference and sample masses. The reference material is selected to be similar in state and mass to the samples to be analyzed; e.g., the manufacturer suggests water for aqueous samples and sapphire or alumina for solid samples.

$$C_p^S = \frac{Y_S}{Y_R} \times \frac{M_R}{M_S} \times C_p^R \tag{5.4}$$

where: C_P^S – Heat capacity at constant pressure of the sample material
C_P^R – Heat capacity at constant pressure of the reference material (known)
Y_S – Curve difference between the sample and the empty container
Y_R – Curve difference between the reference and the empty container
M_S – Sample mass
M_R – Reference mass

The AZ-101 samples measured were aqueous slurries containing 3-wt%, 13-wt%, and 20-wt% undissolved solids. To prevent heat of vaporization due to water evaporation from masking heat absorption due to an increase in temperature, hermetically sealed 15-µL gold pans were used for the single 10-mg distilled, deionized water (DIW) reference and each of the 10-mg AZ-101 slurry samples. The use of hermetically sealed pans prevented the reuse of a pan for both the reference and sample. Since the sealed pans provide a constant volume, the measured heat capacity is actually at constant volume, C_V, rather than the C_P requested in the test specification.

Using a reference material similar in state and mass to the sample, the heat capacity ratio C_P/C_V of the sample and reference material should be approximately equal. With this assumption, C_P can be calculated as seen in Equation 5.5.

$$\gamma^S \cong \gamma^R$$

$$\frac{C_P^S}{C_V^S} = \frac{C_P^R}{C_V^R}$$

$$C_P^S = \left(\frac{C_V^S}{C_V^R}\right) \cdot C_P^R$$

$$C_P^S = \left(\frac{\Delta U^S}{\Delta U^R}\right) \cdot C_P^R \qquad (5.5)$$

where:
γ^S – Heat capacity ratio of the sample material
γ^R – Heat capacity ratio of the reference material
C_P^S – Heat capacity at constant pressure of the sample material
C_P^R – Heat capacity at constant pressure of the reference material (known)
C_V^S – Heat capacity at constant volume of the sample material
C_V^R – Heat capacity at constant volume of the reference material
ΔU^S – Change in internal energy of the sample material (measured)
ΔU^R – Change in internal energy of the reference material (measured)

The DIW reference was encapsulated in its own gold pan and used as the reference for each analysis; the mass was checked after each analysis to ensure that no water escaped. Each AZ-101 slurry sample was encapsulated in the same pan used for its baseline determination (i.e., empty pan). The pan used for the sample is assumed to be equivalent to the pan used for the DIW reference. For C_P^R in Equation 5.5, the heat capacities for water of 4.180 J/g-K at 298 K (25°C) and 4.179 J/g-K at 313 K (40°C) (Weast 1984) were used to calculate the sample material heat capacity.

Each AZ-101 slurry sample (i.e., 3-wt%, 13-wt%, and 20-wt% undissolved solids) was analyzed in triplicate, with an additional replicate analysis being performed on the 3-wt% undissolved solids sample. The AZ-101 slurry samples were homogenized by swirling. For each analysis, a 10-mg aliquot of the homogenized sample was drawn with a micropipette and transferred to the sample pan. The aliquots were weighed after each analysis to ensure no mass loss.

5.4.2 Heat Capacity Results

The temperature program used for the triplicate analysis of the AZ-101 slurry samples was to 1) cool to 10°C, 2) hold at 10°C for 10 minutes, 3) heat to 70°C at 2.5°C/min, and 4) hold at 70°C for 10 min. High purity DIW was used as a reference for the AZ-101 slurry sample analyses.

The heat capacities at 298 K (25°C) and 313 K (40°C) and their 955 confidence intervals (CI) are provided in Table 5.9. The 95% confidence interval reported for each replicate is based on a pooled variance calculated per Snedecor and Cochran (1980) across all of the repeated aliquot analyses. The overall mean heat capacity is calculated by averaging the replicate heat capacities, and the confidence interval for the overall mean is based on variance across the replicates. For example, for the 3-wt% undissolved solids sample, the variance is calculated using four replicates, thus providing 3 degrees of freedom to estimate the Student's t value.

Table 5.9. Measured Heat Capacity of AZ-101 Slurry Samples

AZ-101 Slurry Samples	Temperature, K (°C)	Replicate 1 C_p (95% CI) J/g-K	Replicate 2 C_p (95% CI) J/g-K	Replicate 3 C_p (95% CI) J/g-K	Replicate 4 C_p (95% CI) J/g-K	Overall Mean C_p (95% CI) J/g-K
3-wt% UDS	298 (25)	3.48 (±0.30)	3.50 (±0.25)	3.97 (±0.25)	3.01(±0.17)	3.49 (±0.62)
	313 (40)	3.44 (±0.29)	3.49 (±0.24)	4.01 (±0.24)	2.99(±0.17)	3.48 (±0.66)
13-wt% UDS	298 (25)	2.91 (±0.15)	3.06 (±0.15)	2.70 (±0.15)	NM	2.89 (±0.45)
	313 (40)	2.91 (±0.17)	3.08 (±0.17)	2.69 (±0.17)	NM	2.89 (±0.48)
20-wt% UDS	298 (25)	3.07 (±0.11)	2.51 (±0.16)	2.89 (±0.16)	NM	2.83 (±0.72)
	313 (40)	3.09 (±0.12)	2.50 (±0.16)	2.91 (±0.16)	NM	2.84 (±0.75)

CI = confidence interval; UDS = undissolved solids; NM = not measured

5.4.3 Evaluation of Heat Capacity Results

Table 5.9 shows a small to negligible temperature effect consistent with the small temperature effect between these two temperatures for water. The heat capacity of water at 298 K is 4.180 J/g-K and at 313 K is 4.179 J/g-K; i.e., there is no change in the second decimal for the heat capacity of water at the two temperatures. A comparison of heat capacities for the AZ-101 slurry samples, with heat capacities for water at 298 K and 313 K, indicates that the heat capacities of water are greater than those measured on the slurries. Also, the results show that as the undissolved solids content in the slurry increases, the heat capacity decreases. This decrease in heat capacity, with increasing undissolved solids content and lower heat capacity relative to pure water, is consistent with expectations since solids have a much lower heat capacity than liquids. For example, at 298 K alumina (Al_2O_3, corundum) has a heat capacity of 0.8 J/g-K compared to 4.18 J/g-K for water (Barin 1989).

The heat capacities measured on the various aliquots from each of the AZ-101 slurry samples demonstrate significant variability. This variability likely arises from 1) the necessity of using very small (i.e., 10 mg) samples and 2) the difference in the solids content between the separate aliquots, given the difficulties in obtaining a representative sample from a small slurry sample that is not being constantly agitated (i.e., simply stirred prior to aliquoting).

5.5 Particle Size

The source of the sample for the particle size measurements was AZ-101-PCB-2 (See Section 5.1). Supernatant was decanted from the AZ-101-PCB-2 source bottle in such a manner that three, 1-mL samples at 3-wt% undissolved solids were obtained (See Section 5.2.3 and Table 5.7). With the exception of changes resulting from field sampling and laboratory homogenization (e.g., particle fracturing), the solids measured should be representative of the solids in Tank AZ-101. A Microtrac™ X-100 particle analyzer (X100) and a Microtrac™ ultrafine particle analyzer (UPA) were both used to measure the PSD of these AZ-101 solids.

5.5.1 Instrument Description

The X100 measures particle diameter by scattered light from a laser beam projected through a stream of the sample particles diluted in a suspending medium. The amount and direction of light scattered by the particles are measured by an optical detector array and then analyzed to determine the size distribution of the particles. This measurement is limited to particles with diameters between 0.12 μm and 704 μm. The UPA measures particle diameter by Doppler shifted scattered light. This method is limited to particles with diameters between 0.003 μm and 6.5 μm.

5.5.2 Particle Size Distribution Data Reporting Details

When evaluation of a multi-variable system is required, measured or generated data can be grouped by ranges of values within specific variables. These groupings are called bins. These bins are then represented and plotted in one or two variable graphs called histograms. This grouping process results in a loss of some information in comparison to a standard XY scatter plot diagram, since each and every individual point is not shown. But it is a necessary and commonly used statistical method for evaluation of complex data. Histograms serve the purpose of showing the statistical properties of the data and allow for application of computational methods. Histograms are the standard format for the presentation of PSD data. The particle size results are saved in the form of a histogram with varying bin sizes. The upper range of each bin is determined by a geometric sequence shown in Equation 5.6.

$$\overset{\frown}{d}_{i+1} = \frac{\overset{\frown}{d}_i}{\sqrt[4]{2}} \tag{5.6}$$

where:

$\overset{\frown}{d}_1 = 704.0\,\mu\text{m for the X100}$

$\forall i = 1...50 \text{ for the X100}$

$\overset{\frown}{d}_1 = 6.541\,\mu\text{m for the UPA}$

$\forall i = 1...44 \text{ for the UPA}$

The lower range for each bin is determined by Equation 5.7

$$\breve{d}_i = \overset{\frown}{d}_{i+1} \tag{5.7}$$

where:

$$\breve{d}_{50} = \frac{\overset{\frown}{d}_{50}}{\sqrt[4]{2}} = 0.122\ \mu\text{m for the X100}$$

$$\breve{d}_{44} = \frac{\overset{\frown}{d}_{44}}{\sqrt[4]{2}} = 0.0032\ \mu\text{m for the UPA}$$

The bin centered values for this bin set is determined by Equation 5.8:

$$\bar{d}_i = \frac{\widehat{d}_i + \breve{d}_i}{2} \tag{5.8}$$

The PSD stored by the Microtrac™ instruments represent the volume percent of particles attributed to a particular bin. This is usually called the 'differential' volume distribution. For example, a value of 5 in the first bin of a volume distribution for the X100 indicates that 5% of the volume of the particles measured by the instrument are between $\widehat{d}_1 = 704.0\mu\text{m}$ and $\breve{d}_1 = 592.0\mu\text{m}$. The bin centered value, $\bar{d}_1 = 648.0\ \mu\text{m}$, would be used to display this data point on a graph. A similar calculation can be performed for the UPA data. The volume distribution data will be denoted as, V_i. These data can be represented in as a 'cumulative' distribution using Equation 5.9. If the differential distribution is properly normalized (i.e., to 100%), the range of the cumulative distribution will be between 0% and 100%. When displayed on a graph, the cumulative distribution uses the upper range of the bins such that a data point is represented by $\left(\widehat{d}_i, V_i^C\right)$. The resulting graph should be interpreted as V_i^C percent of volume of the sample has particles smaller than $\widehat{d}_i$.

$$\begin{aligned} V_{i+1}^C &= 100\% - \sum_{j=1}^{i} V_j \\ V_1^C &= 100\% \end{aligned} \tag{5.9}$$

If it is assumed that the particles are spherical with an equivalent diameter of $\bar{d}_i$, the differential distribution can be transformed from a volume basis to a number basis. The number basis represents the percent number or percent of the population of particles between a certain size range. For example, a value of 5 in the first bin of a number distribution for the X100 indicates that 5% of the population of the particles measured by instrument are between $\widehat{d}_1 = 704.0\mu\text{m}$ and $\breve{d}_1 = 592.0\mu\text{m}$. The bin centered value, $\bar{d}_1 = 648.0\ \mu\text{m}$, would be used to display this data point on a graph. The percent number distribution, N_i, can be calculated using Equation 5.10.

$$\begin{aligned} n_i &= \left(\frac{6 \cdot V_i}{\pi \bar{d}_i^3}\right) \\ N_i &= 100\% \times \left(\frac{n_i}{\sum_i n_i}\right) \end{aligned} \tag{5.10}$$

These data can be represented as a 'cumulative' distribution using Equation 5.11. If the differential distribution is properly normalized (i.e., to 100%), the range of the cumulative distribution will be between 0% and 100%. When displayed on a graph the cumulative distribution uses the upper range of the bins such that a data point is represented by $\left(\widehat{d}_i, N_i^C\right)$. The resulting graph should be interpreted as N_i^C percent of the population of particles in the sample is smaller than $\widehat{d}_i$.

$$N_{i+1}^C = 100\% - \sum_{j=1}^{i} N_j$$
$$N_1^C = 100\% \tag{5.11}$$

When comparing the volume and number distributions, the volume distribution is weighted cubically towards larger particles. For example, one 10-µm particle has the same volume as 1,000 1-µm particles.

Lastly, the data can be displayed on a surface area basis. If it is assumed that the particles are spherical, the surface area of the resulting sphere and the number distribution can be used to calculate the area distribution. As an example, a value of 5 in the first bin of an area distribution for the X100 indicates that 5% of the surface area of the particles in the slurry are between $\widehat{d}_1 = 704.0\mu\text{m}$ and $\breve{d}_1 = 592.0\mu\text{m}$. The bin centered value, $\overline{d}_1 = 648.0\ \mu\text{m}$, would be used to display this data point on a graph. The percent area distribution, A_i, can be calculated using Equation 5.12.

$$a_i = \pi \overline{d}_i^{\,2} N_i$$
$$A_i = 100\% \times \left(\frac{a_i}{\sum_i a_i} \right) \tag{5.12}$$

When comparing the area and number distributions, the area distribution is weighted to the second power towards larger particles. For example, one 10-µm particle has the same surface area as 100 1-µm particles.

These area data can be represented as a 'cumulative' distribution using Equation 5.13. If the differential distribution is properly normalized (i.e., to 100%), the range of the cumulative distribution will be between 0% and 100%. When displayed on a graph the cumulative distribution uses the upper range of the bins such that a data point is represented by $\left(\widehat{d}_i, A_i^C\right)$. The resulting graph should be interpreted as A_i^C percent of the surface area of particles in the sample is smaller than $\widehat{d}_i$.

$$A_{i+1}^C = 100\% - \sum_{j=1}^{i} A_j$$
$$A_1^C = 100\% \tag{5.13}$$

The mean value for the differential form of these distributions can be calculated by the Equation 5.14. This value represents the centroid of the distribution.

$$d_V = \frac{\sum_i V_i \bar{d}_i}{\sum_i V_i}$$

$$d_N = \frac{\sum_i N_i \bar{d}_i}{\sum_i N_i} \tag{5.14}$$

$$d_A = \frac{\sum_i A_i \bar{d}_i}{\sum_i A_i}$$

The median value of the cumulative form of these distributions is calculated using Equation 5.15. This value represents the diameter where 50% of the particles have a smaller volume, population, or surface area; and 50% of the particles have a larger volume, population, or surface area. Since this diameter rarely falls directly on the 50% value, this point is typically calculated from linear interpolation.

$$D_V = V_i^C\big|_{50\%}$$

$$D_N = N_i^C\big|_{50\%} \tag{5.15}$$

$$D_A = A_i^C\big|_{50\%}$$

5.5.3 Calibration Checks

Both instruments performance were checked against a range of National Institute of Standards and Technology (NIST) traceable standards from Duke Scientific Corporation. These standards are polystyrene microspheres dispersed in a 1 mM KCl solution. These standards were run prior to analysis of the sample. Results from these standard tests are presented in Table 5.10, Figure 5.8 and Figure 5.9. The percentile data represent the given percent of the volume (or mass if the specific gravity for all particles is the same) that is smaller than the indicated particle size. The mean diameter of the volume distribution represents the centroid of the distribution and is weighted in the direction of larger particles. To check the functionality of the instrument, a close fit of the number basis mean data is typically required. The number basis mean results were within approximately 10% of the NIST traceable values.

Table 5.10. Particle Size Calibration Check Standards

	X100 Instrument			UPA Instrument	
	Size (µm)	Size (µm)	Size (µm)	Size (µm)	Size (µm)
Standard mean size	5.0	50.4	500	0.096	0.895
Measured mean size [(a)]	4.48	45.7	543	0.094	0.894
10 number% < size	4.00	37.4	499	0.083	0.732
50 number% < size	4.40	44.8	543	0.093	0.876
90 number% < size	4.08	54.3	589	0.110	1.128

(a) Mean particle size calculated on a number basis.

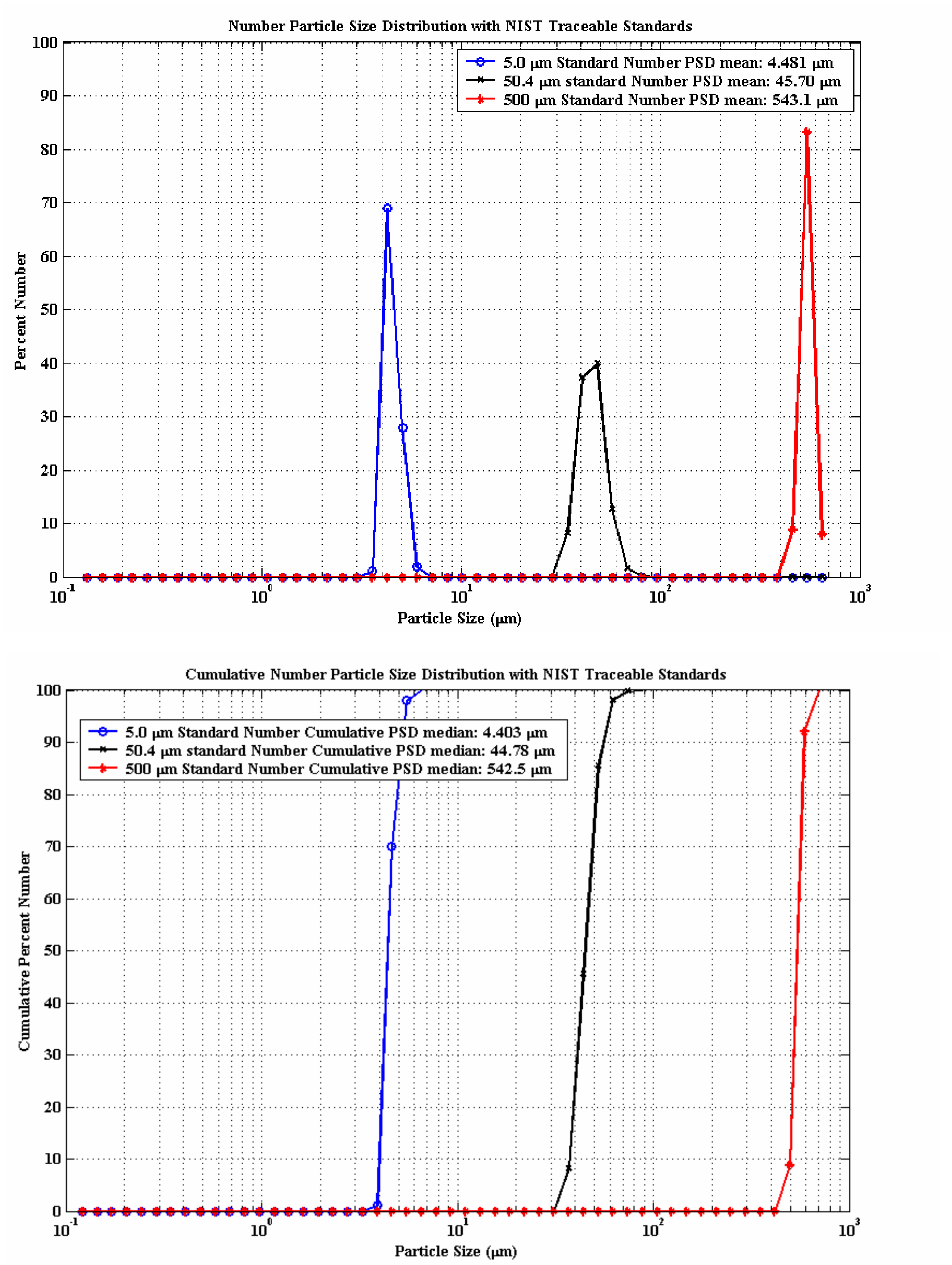


Figure 5.8. X-100 Cal Standards on a Number Basis (top: differential; bottom: cumulative)

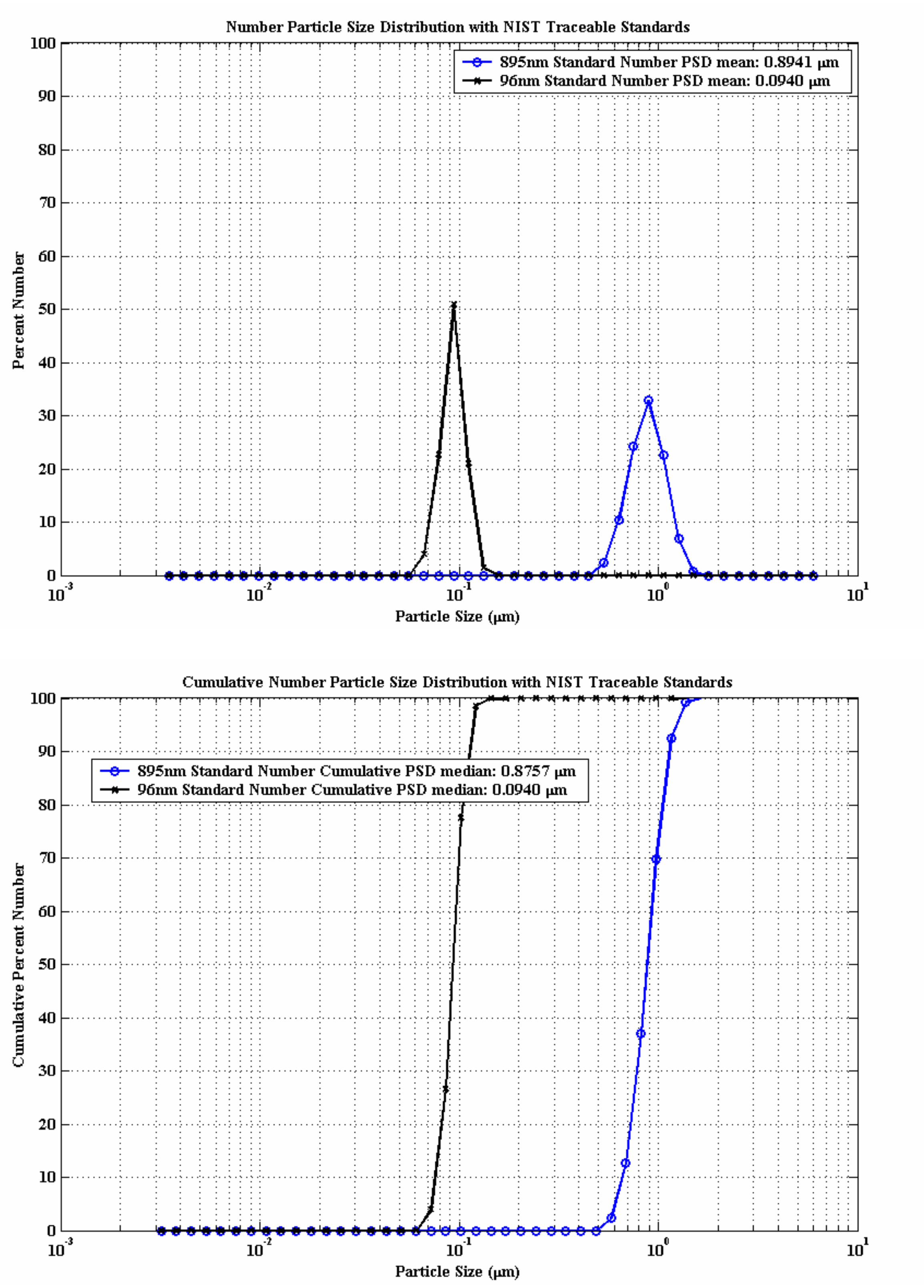


Figure 5.9. UPA Cal Standards on a Number Basis (top: differential; bottom: cumulative)

5.5.4 Operating Conditions

The PSD of the AZ-101 sample was measured in the X100 at a flow rate of 40 mL/s. The flow rate was then increased to 60 mL/s, and the PSD was measured. The samples were then sonicated with 40W ultrasonic waves for 90 seconds at a flow rate of 60 mL/s, and the PSD was measured. The sample was then sonicated a second time with 40W ultrasonic waves for 90 seconds at a flow rate of 60 mL/s, and the PSD was measured. The different flow rates and ultrasonic energy inputs are performed to determine the shear sensitivity of the slurry. The purpose of the shear variations is to investigate whether flocculation/deagglomeration is occurring. Analyses were performed in triplicate on each sample under all flow/sonication conditions.

No sonication or flow options are available for the UPA. Therefore, the sample is placed in the instrument, and the measurements are performed on the as-received, stationary material.

5.5.5 Suspending Medium

The suspending medium for the AZ-101 sample analyses was a surrogate supernatant based on the analytical laboratory data obtained for the AZ-101 supernatant. The composition of this surrogate supernatant liquid is listed in Table 5.11.

Table 5.11. Surrogate Supernatant Composition

Component	Concentration (M)
$NaNO_3$	0.338
NaOH	1.46
$Al(NO_3)_3 \bullet 9H_2O$	0.197
Na_2SO_4	0.167
$Na_2HPO_4 \bullet 7H_2O$	0.0171
NaCl	0.00634
$NaNO_2$	1.46
$NaCO_3$	0.680
$Na_2C_2O_4$	0.0111
NaF	0.105

5.5.6 Results

The average PSDs are presented in tabular form on a volume basis in Table 5.12. These data represent the separation of the particle size data into one or more peaks or modes (first column). The peak/mode particle size (second column) represents the value where 50% of the particles in this peak/mode are smaller than the given value. The peak/mode width (third column) provides a measure of the size variability within the peak/mode. The fourth column represents the percent contribution of each peak/mode to the entire distribution. The average PSDs are presented graphically on a volume, surface area, and number basis in Figure 5.10 through Figure 5.21. The AZ-101 sample designation used in all PSD Figures is AZ-AR and represents the 3-wt% undissolved solids sample prepared from bottle AZ-101-PCB-2. This set of figures compares the PSD of AZ-101 slurry at different rates of shear.

Table 5.12. Summary Particle Size Distribution (Volume) AZ-101 As-Received

Sample Conditions	Peak/Mode Number	Peak/ Mode Particle Size (μm)	Peak/Mode Width (μm)	Approximate Vol% of Particles in Peak/Mode (%)
X100 at 40 mL/s	1	16.69	9.96	14
	2	4.35	4.80	51
	3	1.14	0.95	32
	4	0.27	0.09	3
X100 at 60 mL/s	1	16.71	10.10	14
	2	4.35	4.81	51
	3	1.14	0.95	32
	4	0.27	0.09	3
X100 at 60 mL/s with 90 second sonication at 40 W (#1)	1	16.41	9.35	14
	2	4.34	4.81	50
	3	1.12	0.96	32
	4	0.26	0.11	4
X100 at 60 mL/s with 90 second sonication at 40 W (#2)	1	16.39	9.31	13
	2	4.34	4.84	50
	3	1.04	1.14	37
UPA	1	4.54	3.91	91
	2	0.31	0.19	9

On a volume basis (See Figure 5.10 and Figure 5.11), the AZ-101 solids analyzed by the X100 appear to consist of particles in the 0.2-μm to 30-μm range. As the shear increases due to increases in flowrate and sonication, slight changes to the PSD are observed. Particles in the 3-μm to 6-μm range appear to deagglomerate and the volume of particles in the 0.2-μm to 0.6-μm range increases slightly. On a surface area basis (See Figure 5.12 and Figure 5.13), the AZ-101 sample appears to consist of particles in the 0.2-μm to 30-μm range with peaks at 0.25 μm and 1.0 μm. As the shear increases, the 1.0-μm peak decreases and the peak at 0.25 μm broadens. On a number basis (See Figure 5.14 and Figure 5.15), the AZ-101 sample appears to consist of particles in the 0.2-μm to 30-μm range with a peak at 0.2-0.25 μm. As the shear increases due to sonication, the PSD shifts to slightly smaller particles. The shear sensitivity observed in these samples is most likely due to a small degree of flocculation in the original sample.

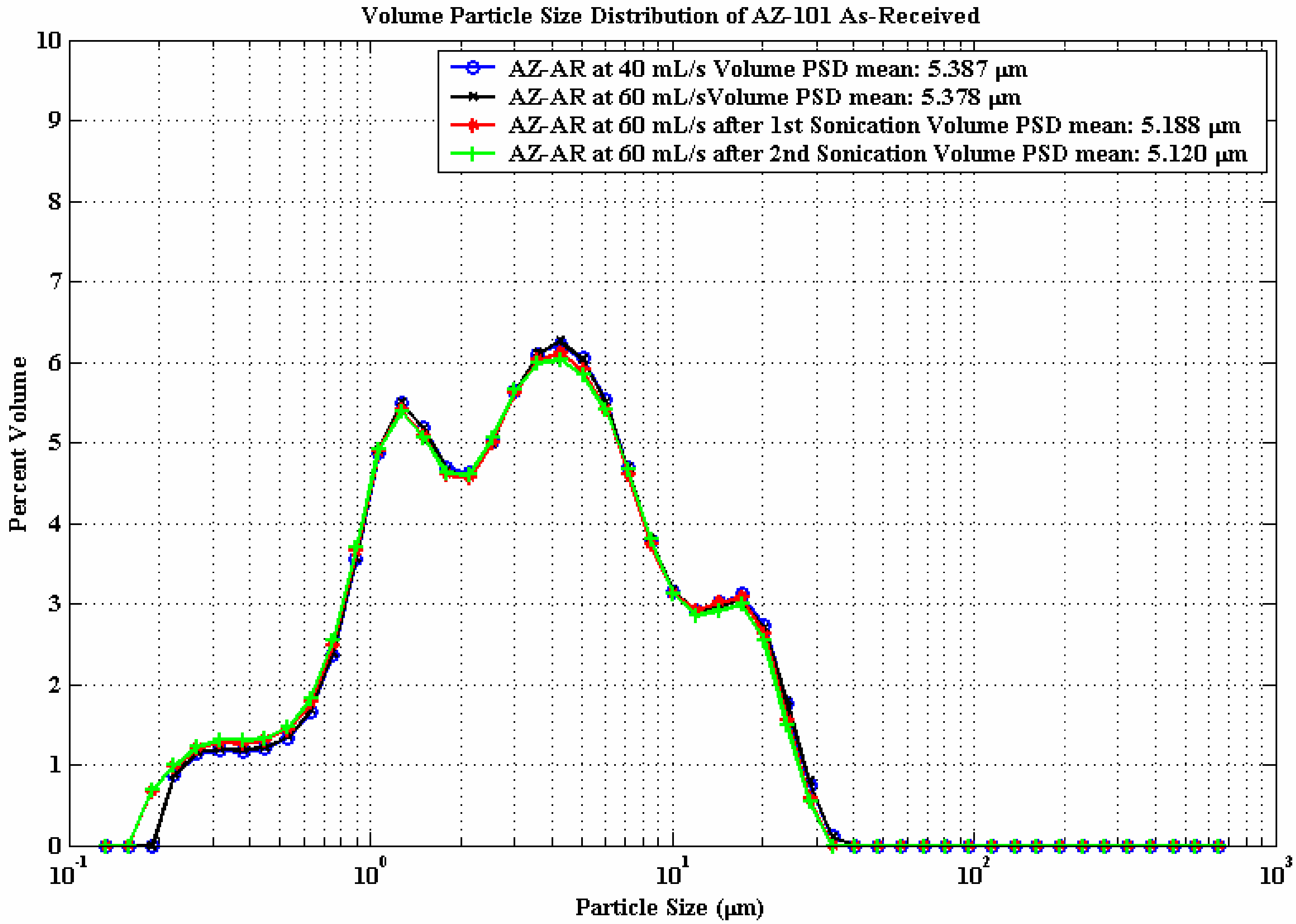


Figure 5.10. X100 Differential Particle Size Distribution of AZ-101 on a Volume Basis

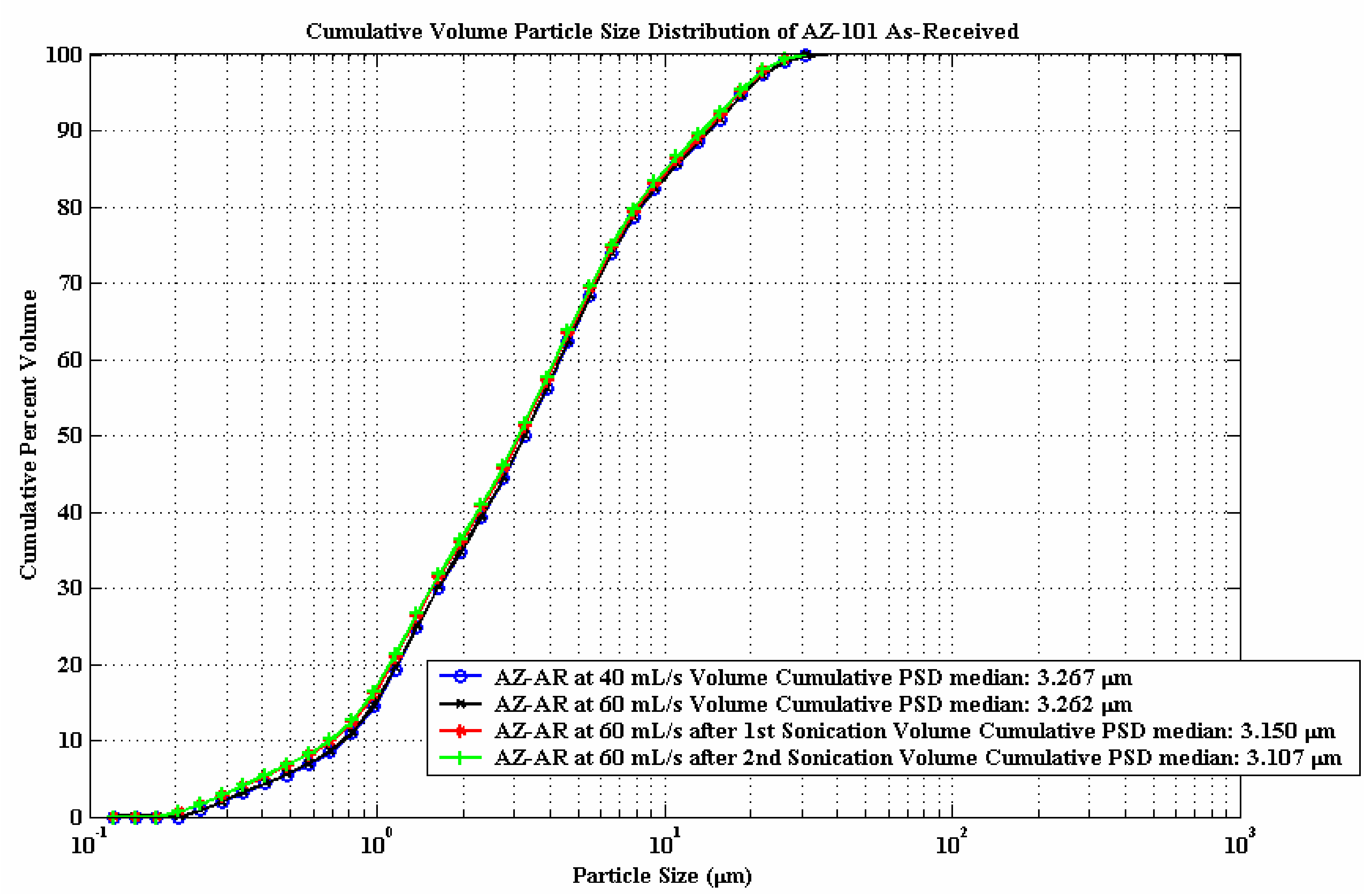


Figure 5.11. X100 Cumulative Particle Size Distribution of AZ-101 on a Volume Basis

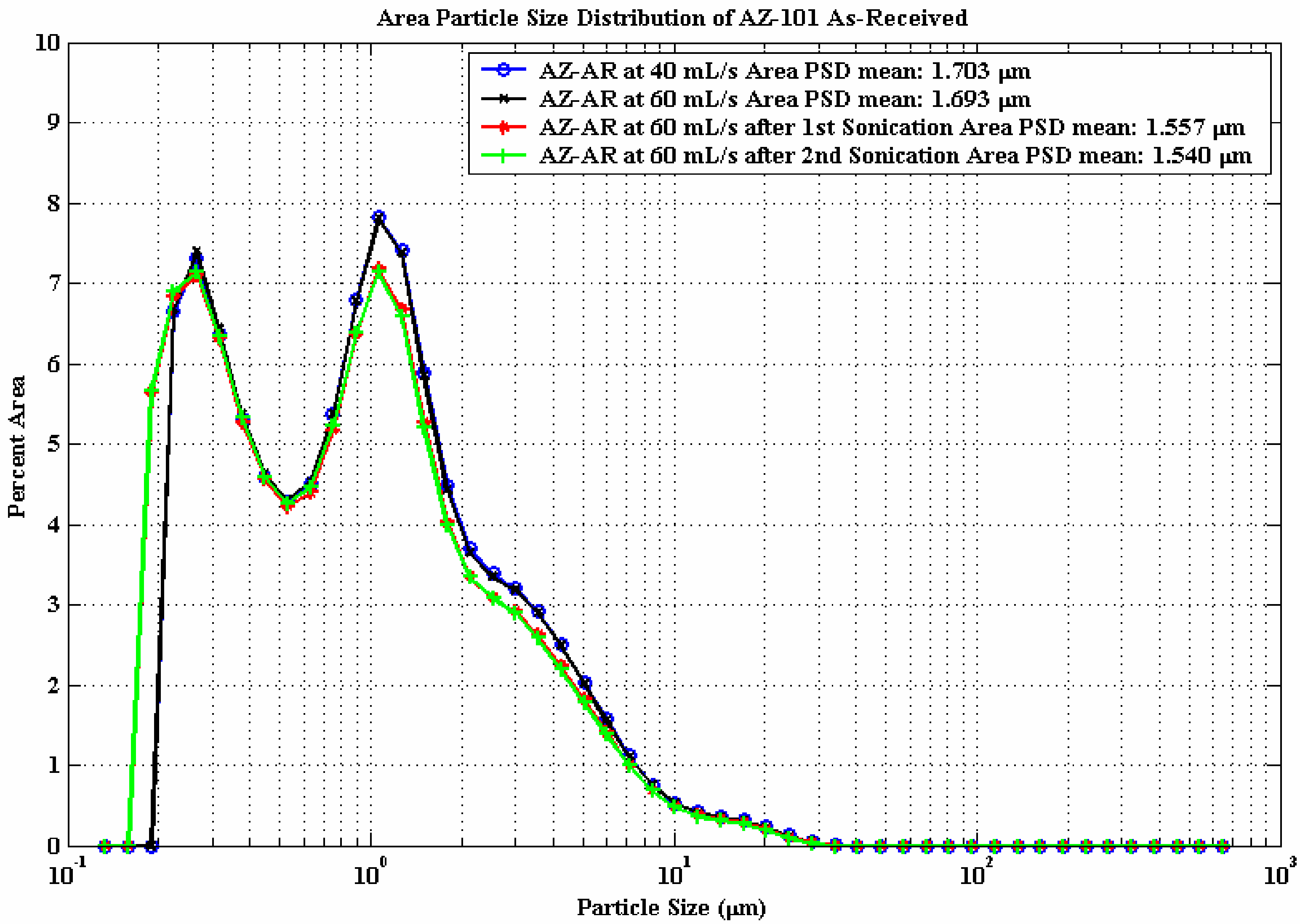


Figure 5.12. X100 Differential Particle Size Distribution of AZ-101 on a Surface Area Basis

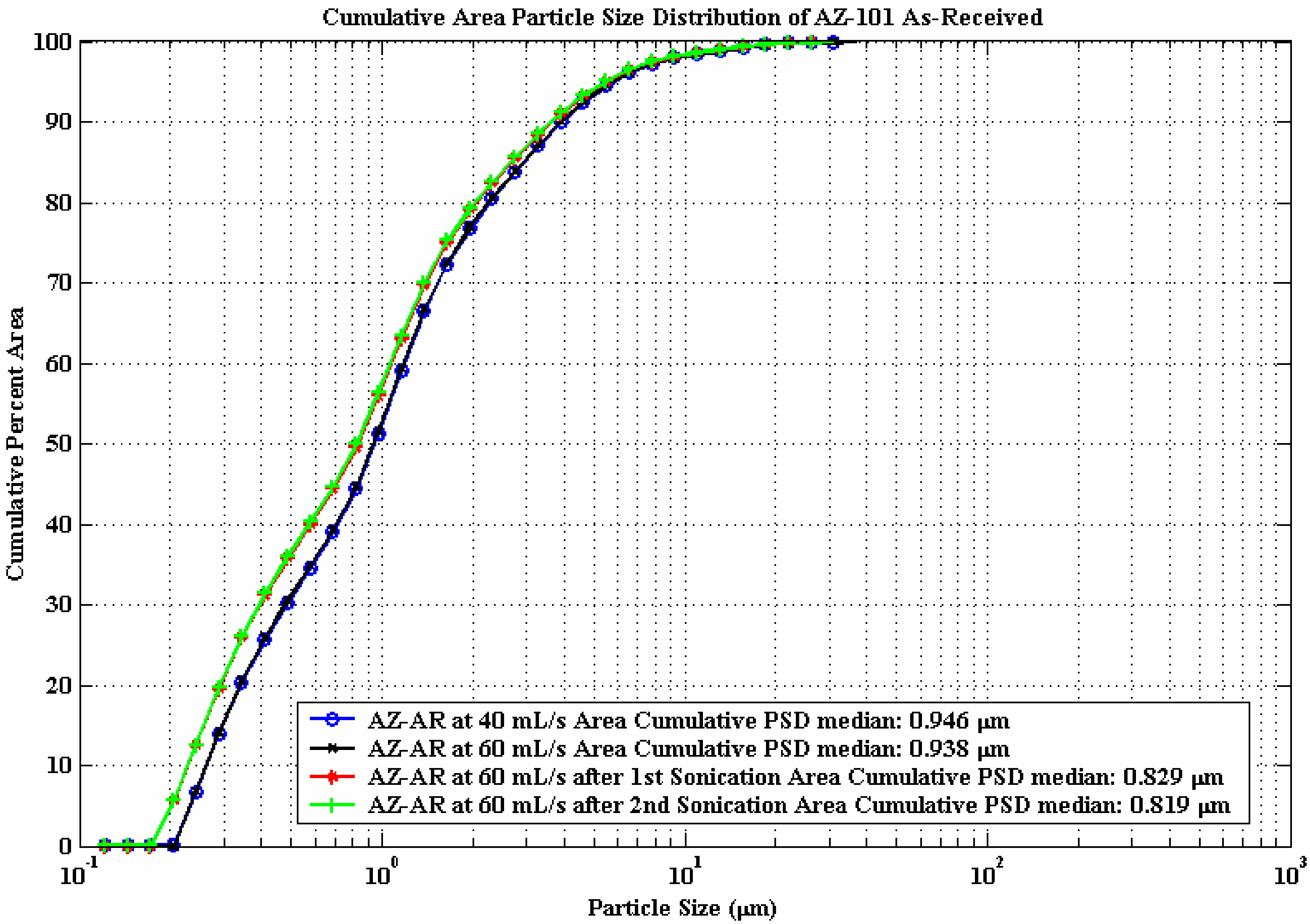


Figure 5.13. X100 Cumulative Particle Size Distribution of AZ-101 on a Surface Area Basis

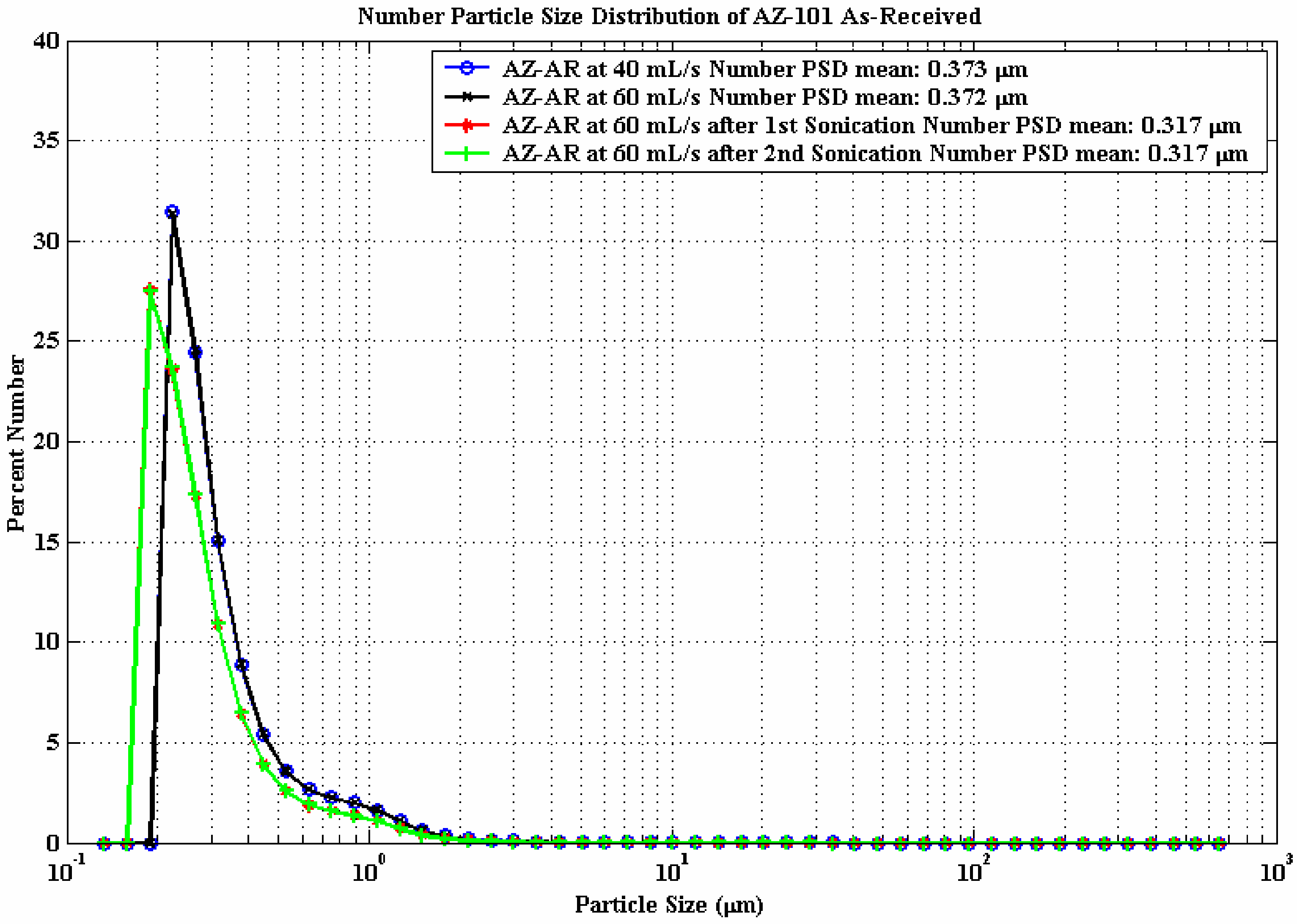


Figure 5.14. X100 Differential Particle Size Distribution of AZ-101 on a Number Basis

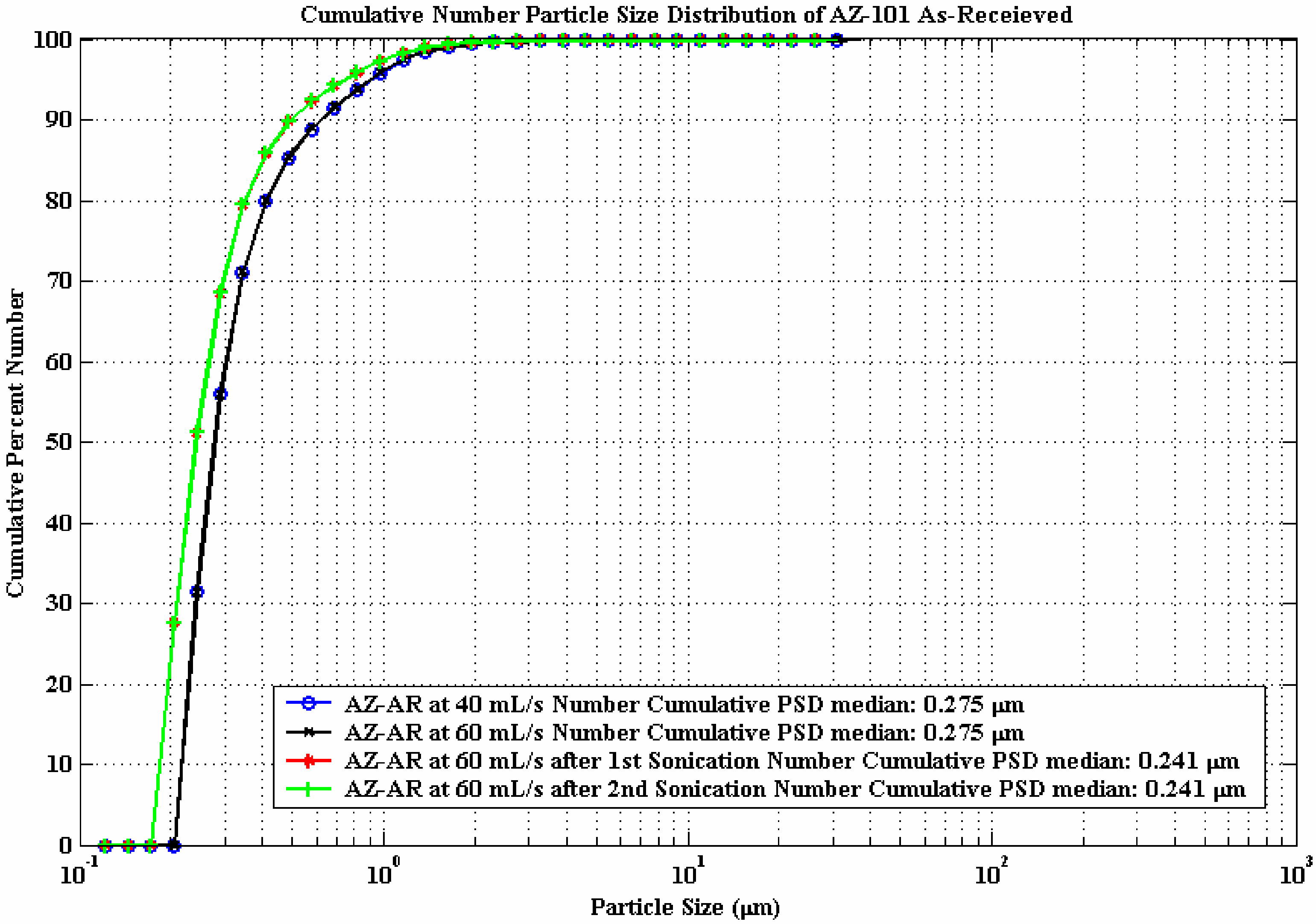


Figure 5.15. X100 Cumulative Particle Size Distribution of AZ-101 on a Number Basis

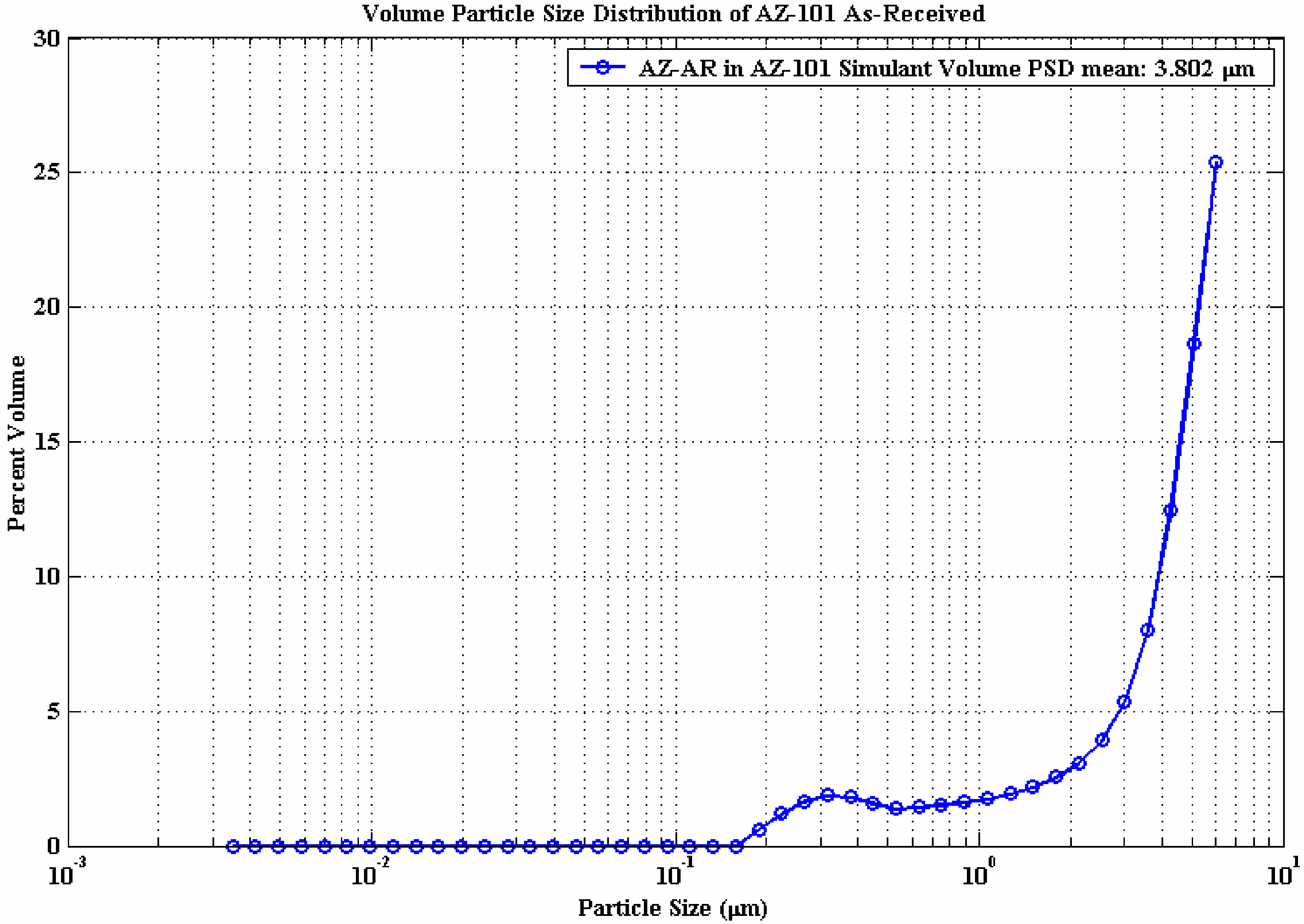


Figure 5.16. UPA Differential Particle Size Distribution of AZ-101 on a Volume Basis

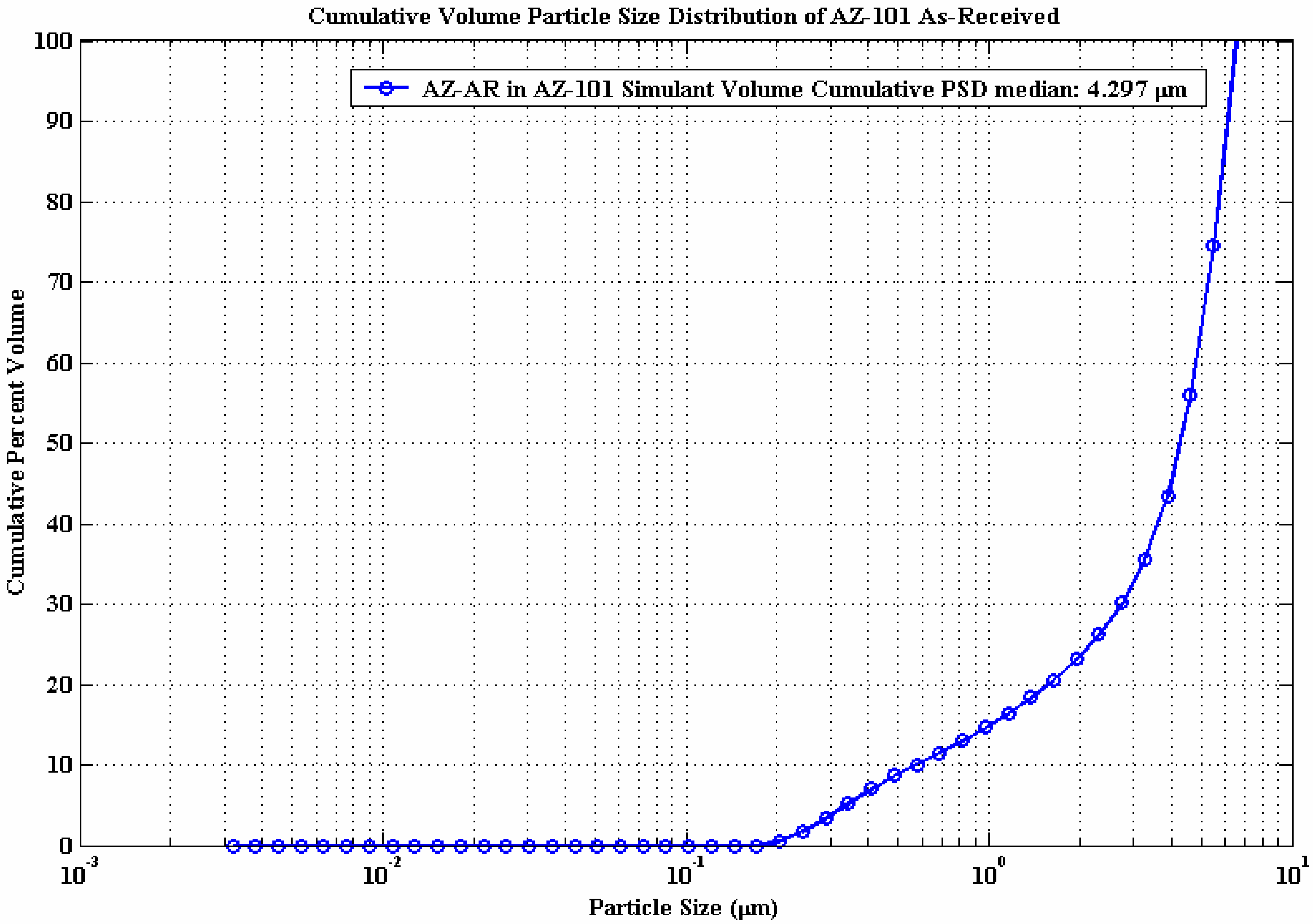


Figure 5.17. UPA Cumulative Particle Size Distribution of AZ-101 on a Volume Basis

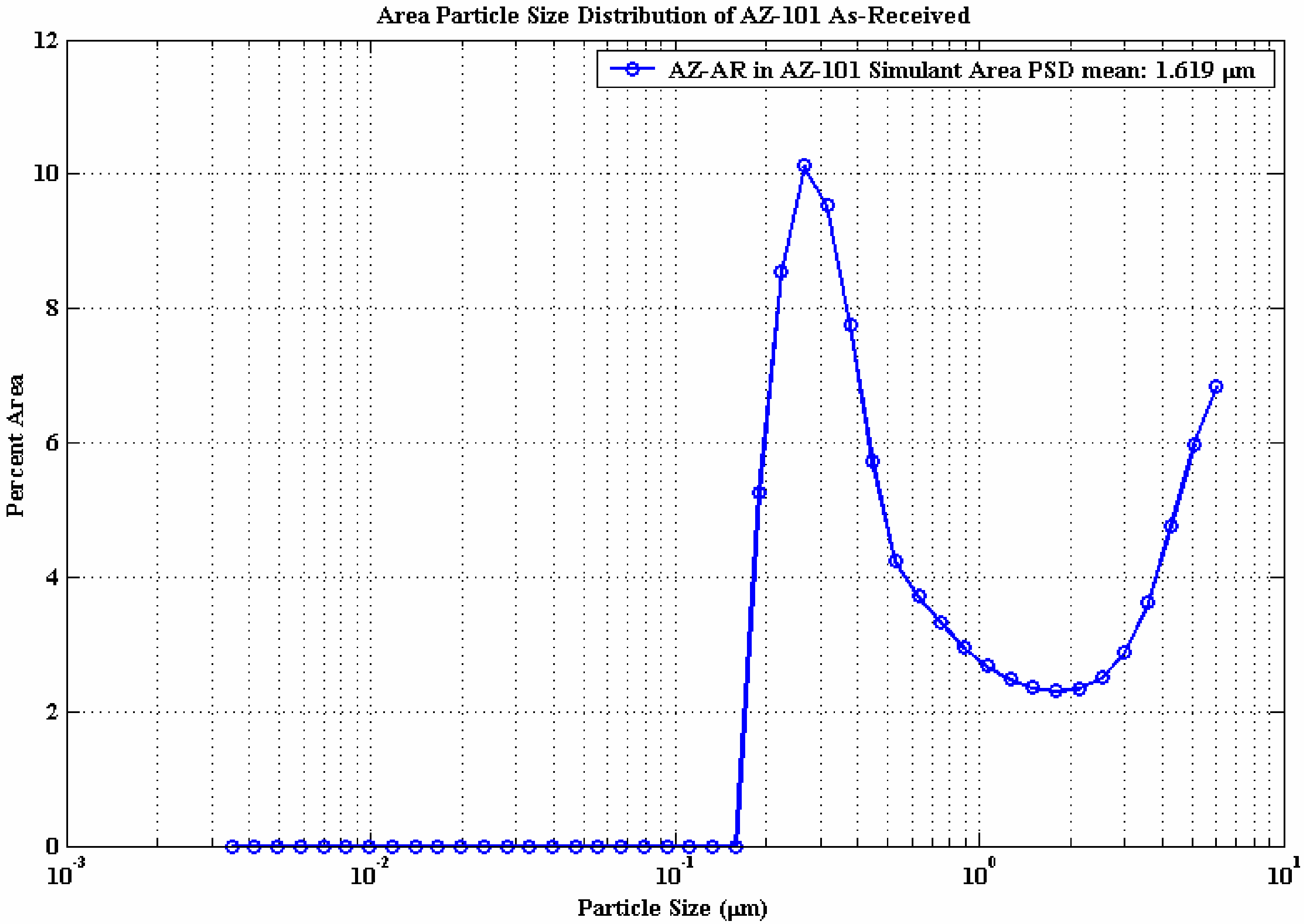


Figure 5.18. UPA Differential Particle Size Distribution of AZ-101 on a Surface Area Basis

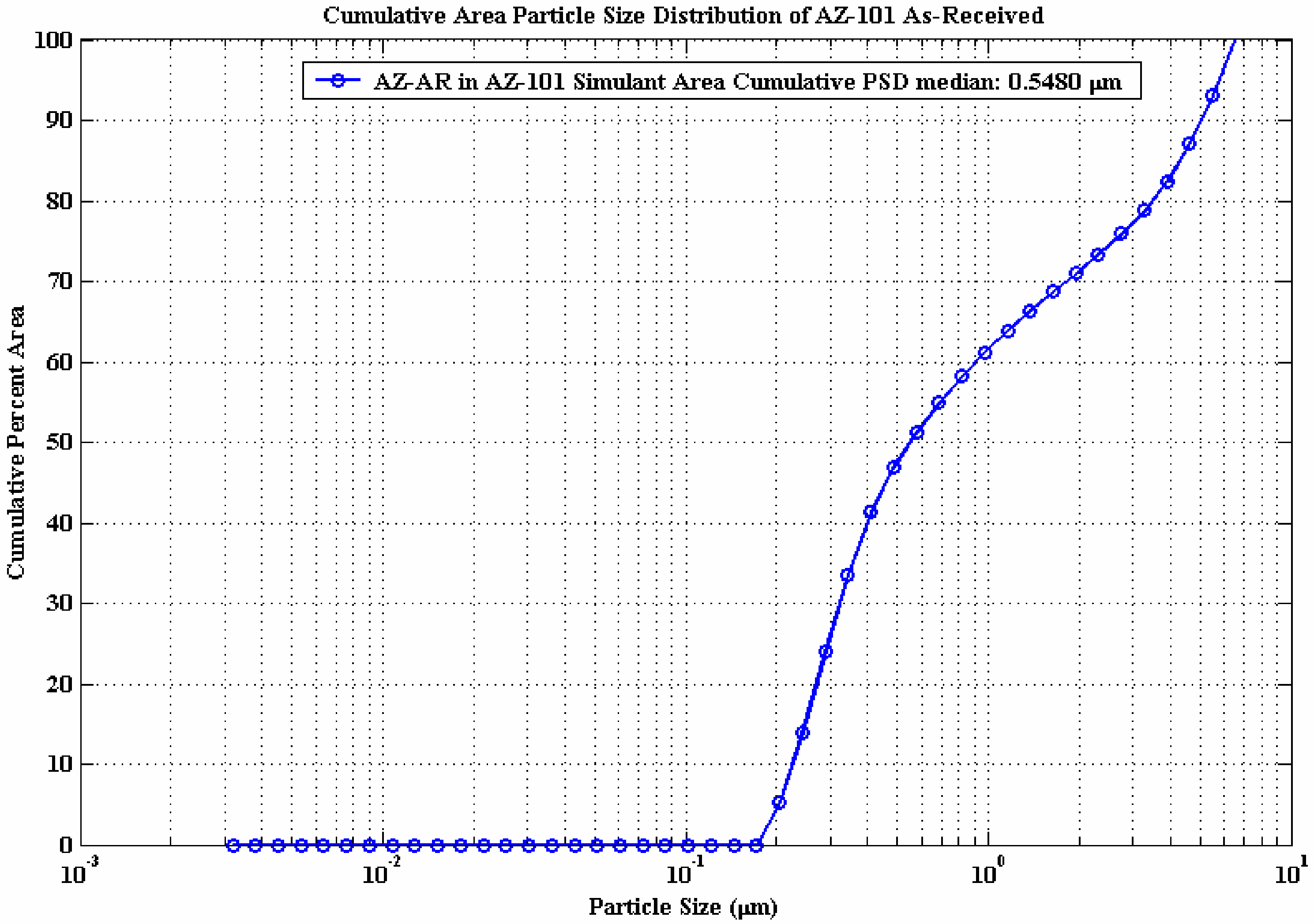


Figure 5.19. UPA Cumulative Particle Size Distribution of AZ-101 on a Surface Area Basis

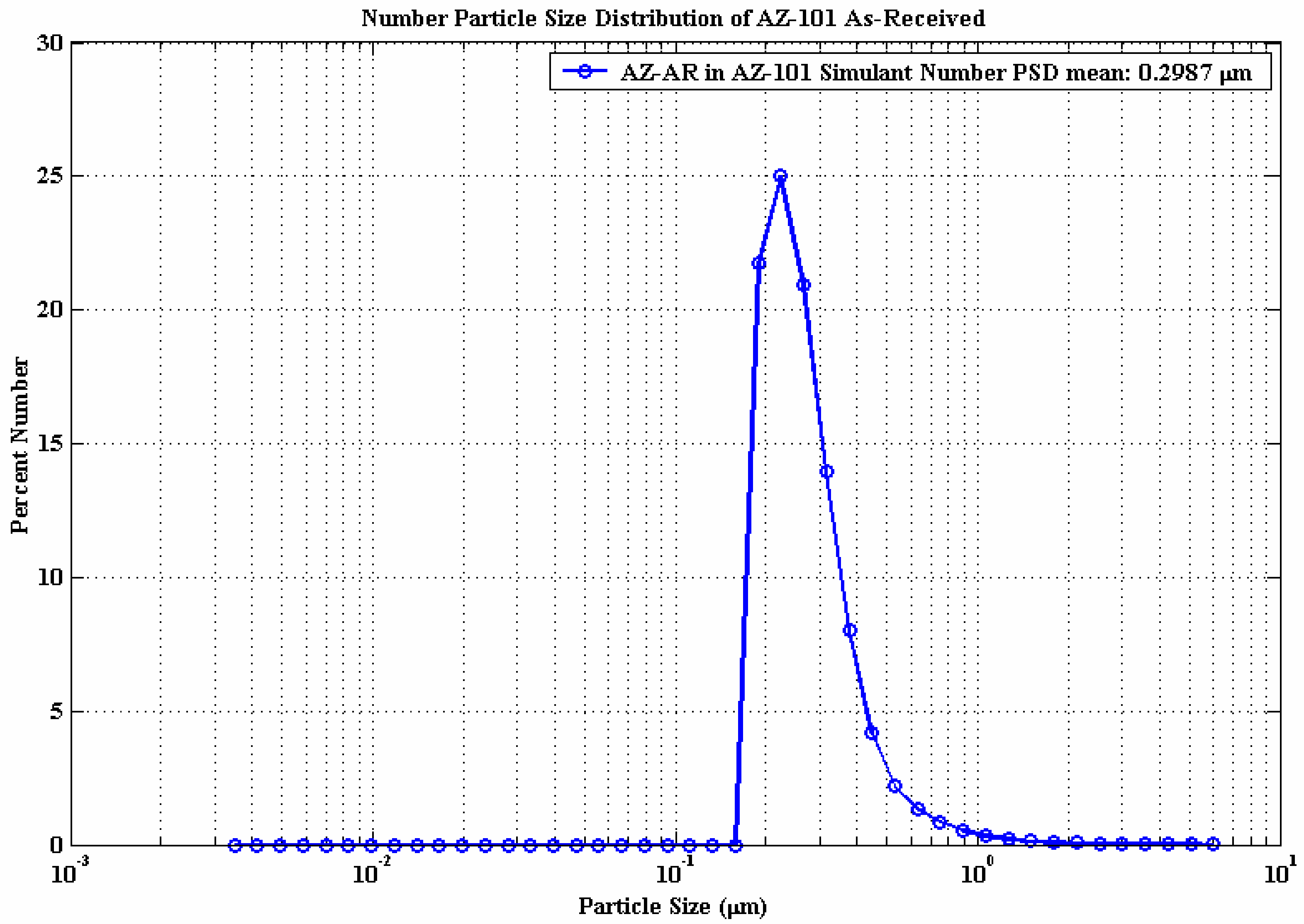


Figure 5.20. UPA Differential Particle Size Distribution of AZ-101 on a Number Basis

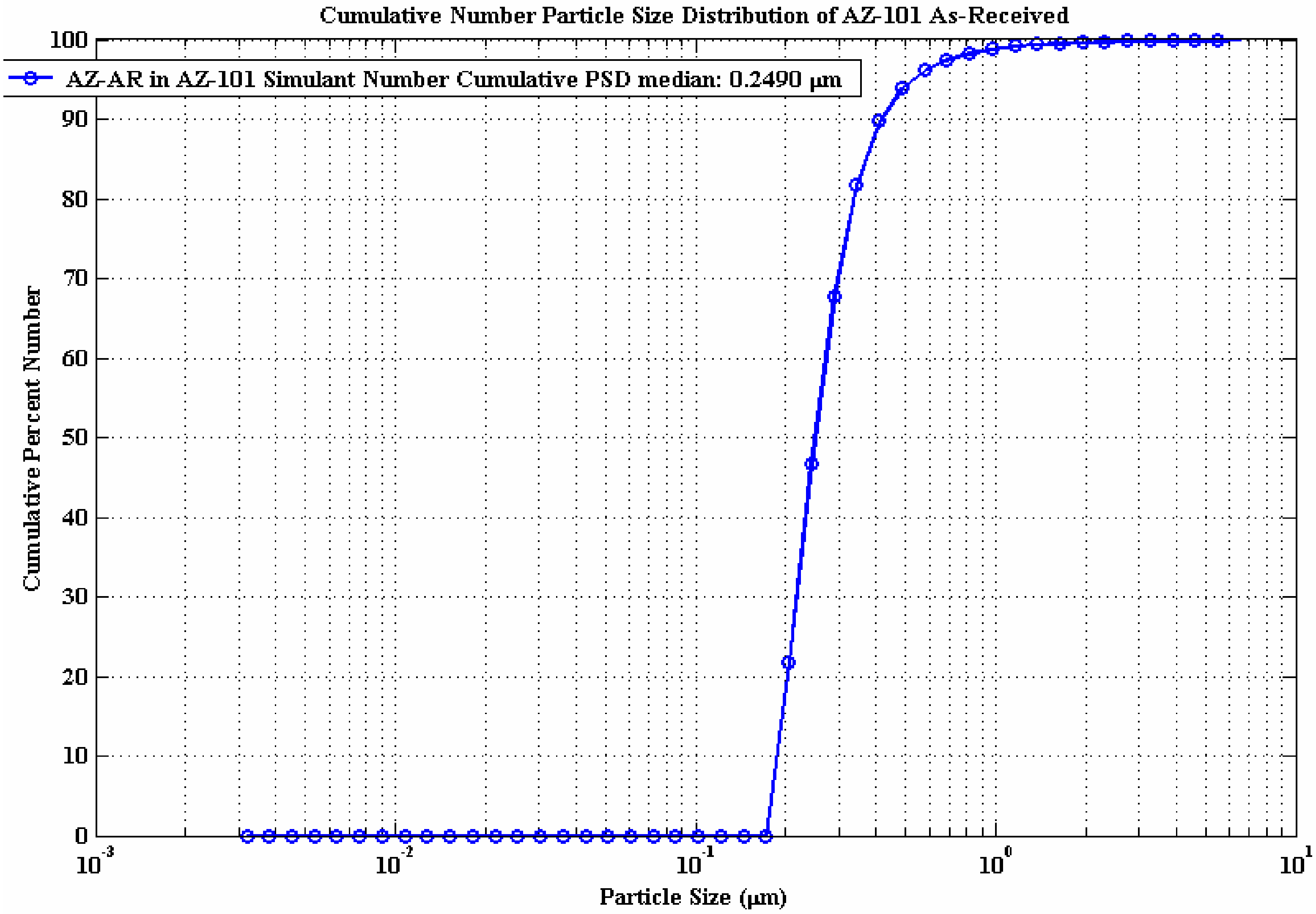


Figure 5.21. UPA Cumulative Particle Size Distribution of AZ-101 on a Number Basis

The AZ-101 solids were also analyzed in the UPA without flow or sonication. The particle size range that is common to both the X100 and UPA is 0.12 µm to 6.5 µm. The UPA data show no significant volume of particles outside this common range (See Figure 5.16). The shape of the cumulative distribution curves (See Figure 5.17) indicates that particles larger than 6.5 µm are likely to exist under no shear. The PSD on a surface area basis (See Figure 5.18 and Figure 5.19) is consistent with the X100 surface area data at 40 mL/sec (See Figure 5.12 and Figure 5.13). A large degree of particles exist in the 0.1-µm to 6.5-µm range. A peak on the PSD for the AZ-101 solids is observed for both the X100 and UPA data at approximately 0.2 µm to 0.3 µm. Shear rate differences and flocculation may explain the second peak observed at approximately 1.0 µm by the X100 but not by the UPA. Lastly, the UPA number distributions (See Figure 5.20 and Figure 5.21) correspond well with the X100 number distributions (See Figure 5.14 and Figure 5.15). Because no particles are observed below 0.1 µm with the UPA, the PSD measured by the X100 particle is considered a complete representation of the PSD over a range of 0.003 µm to 704 µm.

5.6 Scanning Electron Microscopy of As-Received AZ-101 Solids

Samples of as-received AZ-101 solids were prepared for SEM in the Radiochemical Processing Laboratory (RPL) hot cells (See Section 5.1 and Section 5.2.3); however, the radiation level from the samples was too high to allow the sample to be loaded into the microscope. Using a fine needle, a small quantity of the sample was transferred onto a SEM mount. This reduced the radiation level associated with the sample to acceptable levels; however, the particles may not be fully representative of the undissolved solids. The sample was transported to the SEM in Building 326 and examined in the JEOL840® SEM.

The as-received AZ-101 sample was extremely complex, revealing many different phases and shapes. Most of the particles appeared to be irregularly shaped agglomerates consisting of many phases with varied composition. This characteristic makes it difficult to determine the composition of individual phases. Micrographs from the AZ-101 solids are shown in Figure 5.22 (a through j).

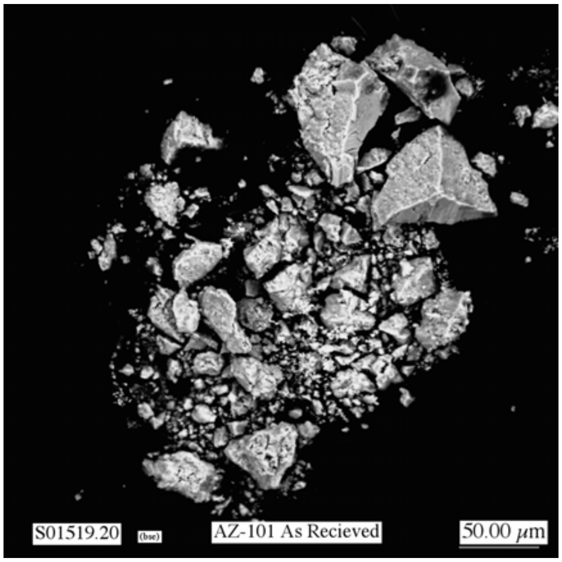


Figure 5.22a Overview of the As-Received AZ-101 Sample

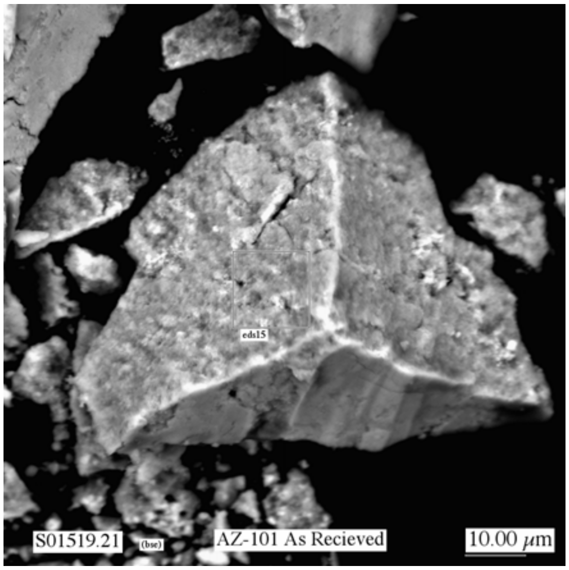


Figure 5.22b SEM image of a large AZ-101 particle

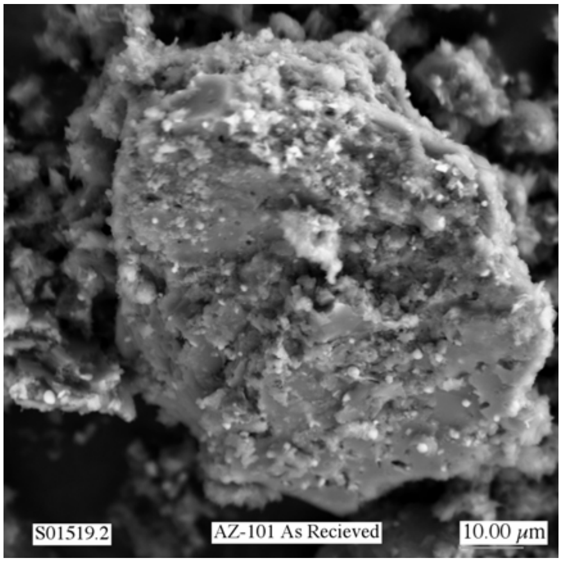


Figure 5.22c SEM image of AZ-101 Agglomerate Particle

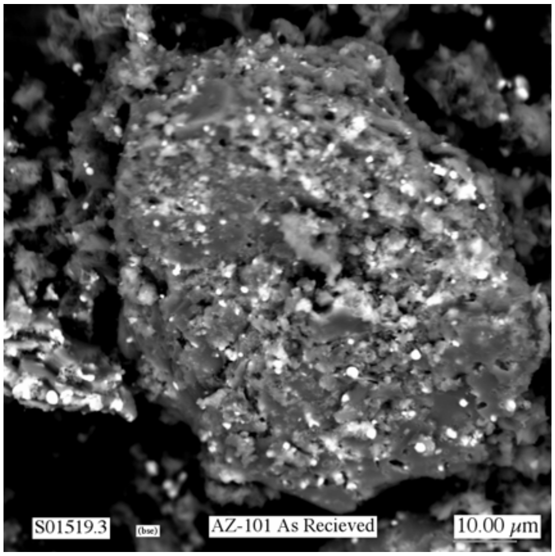


Figure 5.22d SEM image of AZ-101 Agglomerate Particle

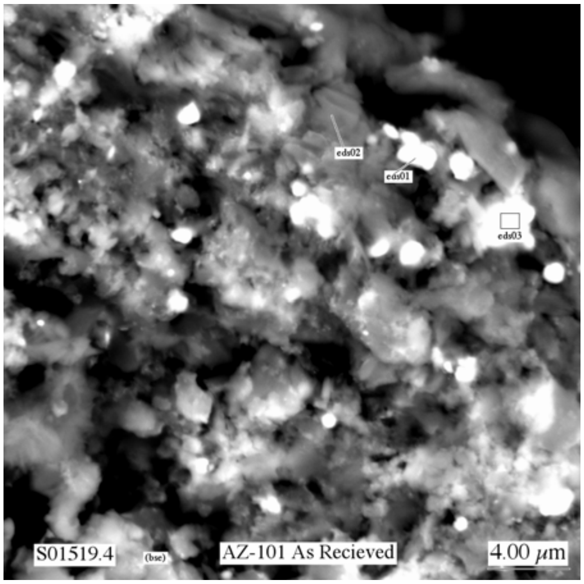


Figure 5.22e SEM image of Agglomerate Particle Surface

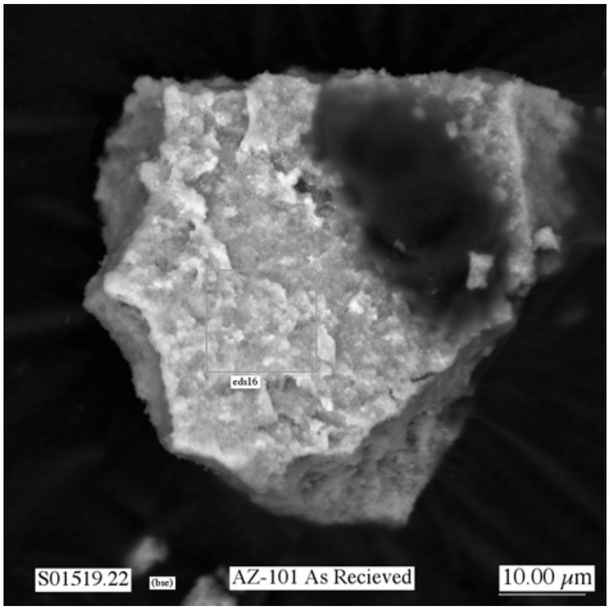


Figure 5.22f SEM image of irregular shaped As-Received AZ-101 Particle

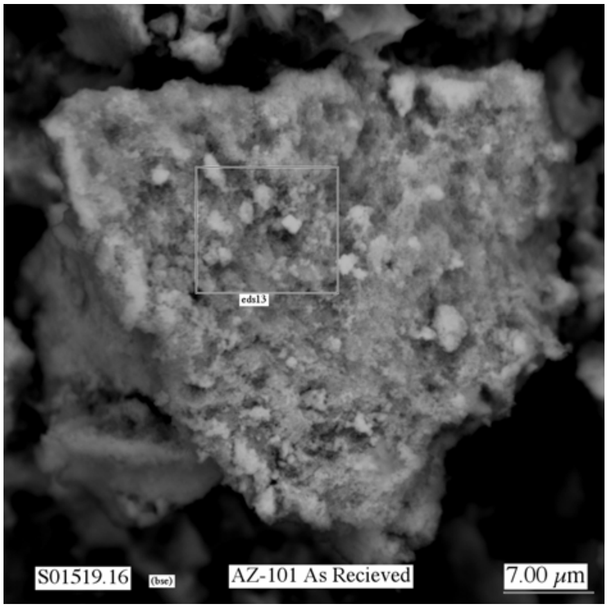


Figure 5.22g SEM image of AZ-101 Agglomerate Particle

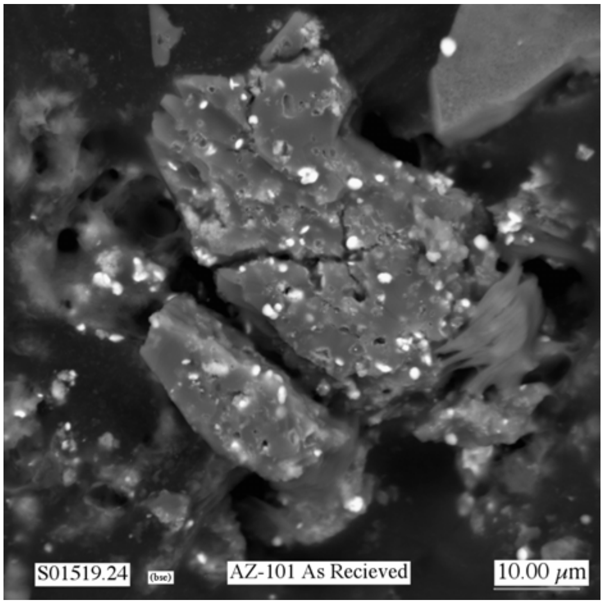


Figure 5.22h SEM image of AZ-101 Agglomerate Particle

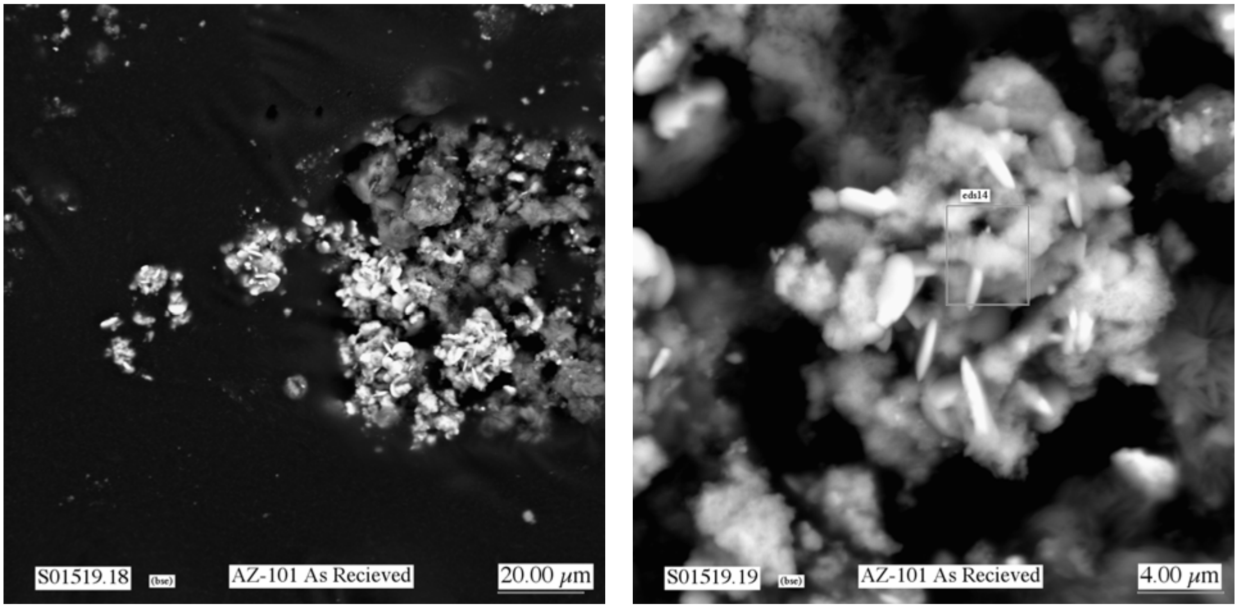


Figure 5.22i Overview of Smaller As-Received AZ-101 Particles

Figure 5.22j High Magnification of Smaller As-Received AZ-101 Particles

Figure 5.22. AZ-101 Solids – SEM Images

6.0 Phase Separation

Following the compositing, homogenization, and sub-sampling of the AZ-101 as-received slurry material, the bottles identified for chemical characterization were transferred from the HLRF to the Shielded Analytical Laboratory (SAL). Four bottles, AZ-101-CHEM-1 through AZ-101-CHEM-4, were allocated for chemical and radiochemical analyses and phase separated by centrifuging (in two batches) per instructions provided in ASR 6193 Addendum 1 and Addendum 2. The phase separation was performed by centrifuging the contents of the AZ-101-CHEM bottles at 1000 G for one hour (with secondary containment in case of breakage). The supernatant was then decanted into bottles labeled AZ-101 SUP1 AR (Addendum 1) or AZ-101 SUP2 AR (Addendum 2); the wet centrifuged solids were transferred by spatula into bottles labeled AZ-101 CS1 AR or AZ-101 CS2 AR. The decanting and transfer operations were performed as rapidly as possible with remote manipulators to minimize the time that the supernatant and wet centrifuged solids were exposed to the SAL environment (i.e., to reduce potential of cross contamination and weight change due to evaporation/drying). Table 6.1 identifies the phase-separated masses of supernatant and wet centrifuged solids collected for analytical characterization.

Table 6.1. Supernatant and Wet Centrifuged Solids Quantities after Phase Separation

AZ-101 Composite Characterization Sub-samples			Phase Separated Samples				
			Supernatant 01-01844		WCS 01-01845		
Bottle ID	Analysis Activity	Mass (g)	AZ-101 SUP1 AR Mass (g)	AZ-101 SUP2 AR Mass (g)	AZ-101 CS 1 AR Mass (g)	AZ-101 CS2 AR Mass (g)	Loss (%)
AZ-101-CHEM-1	ASR Addendum 1	173					
AZ-101-CHEM-2	ASR Addendum 1	120 (a)					
	Total	293	205		74		5
AZ-101-CHEM-3	ASR Addendum 2	157					
AZ-101-CHEM-4	ASR Addendum 2	177					
	Total	334		270		55	3
AZ-101-PCB-1	Archive (b)	111 (c)					
AZ-101-PCB-2	Archive (b)	149 (d)					
AZ-101-PCB-3	Archive (b)	162					
	Total	422					

WCS = wet centrifuged solids

(a) Mass after removing 36 g for slurry testing; initial mass was 156 g.

(b) AZ-101-PCB-1, -2, and -3 archived as slurry samples (i.e., not phase separated).

(c) Mass after removing 56 g to perform rheology measurements.

(d) Mass after removing 9 g removed to prepare samples for heat capacity, particle size, and SEM.

Following phase separation conducted under both ASR 6193 Addendum 1 and Addendum 2, the wt% TDS of the supernatant and the wt% total solids of the wet centrifuged solids were measured. The wt% total solids for the wet centrifuged solids performed under ASR 6193 Addendum 1 was determined on three different dates to cover the time period for the analytical sub-sampling activities. The supernatant density was performed only on the supernatant sample obtained from phase separation conducted under

ASR 6193 Addendum 1. To provide data for calculating the concentration of the undissolved solids, the wt% total oxide was determined on the supernatant and wet centrifuged solids obtained from the phase separation conducted under ASR 6193 Addendum 1. All physical property measurement conducted on the phase-separated supernatant and wet centrifuged solids were performed in triplicate; the results are presented in Table 6.2.

Table 6.2. Density and Percent Solids on Analytical Characterization Samples

RPL #	Sample ID[a]	Date	Analysis	Sample	Duplicate	Triplicate	Average [f]	RSD
01-01844	AZ-101 SUP1 AR	09/04/01	Density (g/mL)	1.220	1.226	1.224	1.223	0.2%
		08/28/01	Total dissolved solids (wt%) [b]	27.0	24.3	27.0	26.1 [d]	6%
		08/28/01	Total oxide (wt%) [c]	16.2	14.1	17.2	15.8	10%
	AZ-101 SUP2 AR	10/05/01	Total dissolved solids (wt%) [b]	27.5	26.8	27.2	27.2 [d]	1%
01-01845	AZ-101 CS1 AR	08/28/01	Total solids (wt%) [b]	69.5	69.3	70.2	69.7 [e]	0.7%
		09/14/01	Total solids (wt%) [b]	69.0	68.9	69.0	68.9 [e]	0.1%
		09/27/01	Total solids (wt%) [b]	72.8	72.9	72.9	72.9 [e]	0.1%
		08/28/01	Total oxide (wt%) [c]	52.2	52.0	53.5	52.6	1%
	AZ-101 CS2 AR	10/05/01	Total solids (wt%) [b]	62.9	64.0	62.6	63.2	1%

(a) SUP1 and CS1 from phase separation conducted under ASR 6193 Addendum 1; SUP2 and CS2 from phase separation conducted under ASR 6193 Addendum 2.

(b) After drying at 105°C to constant weight.

(c) After firing at 1050°C to constant weight.

(d) Average TDS = 26.7%; values used for determining undissolved solids analyte concentration (see Section 8.6).

(e) Average wt% solids AZ-101 CS1 AR = 70.2%; values used for determining undissolved solids analyte concentration (see Section 8.6). No chemical analyses were performed on wet centrifuged solids from AZ-101 CS2 AR.

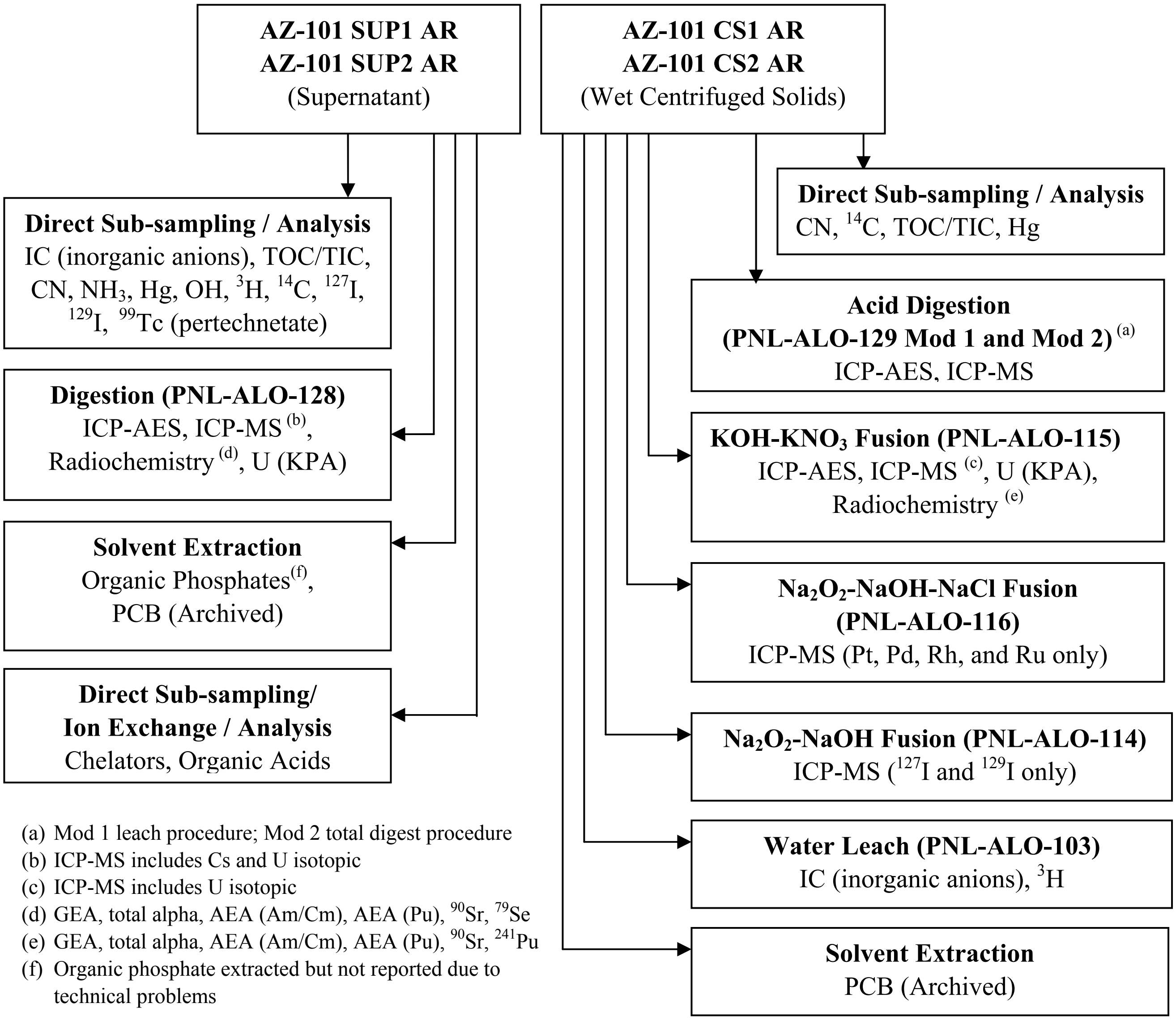
(f) Averages calculated using more digits than presented in the table.

Based on these physical measurements results in Table 6.2, which demonstrate good consistency, the supernatant and wet centrifuged solids phases separated in the SAL are considered representative of the supernatant and solids material sub-sampled for process testing. The separated supernatant and wet centrifuged solids phases should provide excellent baseline characterization results for process testing and good results for comparison of the waste phases to Specification 7 (Envelope B for the supernatant) and Specification 8 (Envelope D for the undissolved solids). The TDS for the supernatant and the wt% total solids for the wet centrifuged solids are used to calculate the concentration of the undissolved solids. Only wet centrifuged solids from AZ-101 CS1 AR were used for chemical and radiochemical characterization; therefore, the average wt% solids for this sample is used in calculating the undissolved solids analyte concentration. The average TDS results obtained from AZ-101 SUP1 AR and AZ-101 SUP2 AR is used in the undissolved solids analyte concentration calculation. See Section 8.6 for undissolved solids calculation.

Revision 1 of this report includes revised data for all ^{127}I and ^{129}I results. To support these analyses, additional sample was phase separated in May of 2003. Following phase separation, the TDS of the supernatant and wt% solids of the centrifuged solids were measured. The TDS (26.3%) amd wt% solids (69.3%) results are essentially identical to results obtained on the initial phase separations (see Table 6.2).

7.0 Analytical Sample Processing

Following the phase separation of the AZ-101 composite into supernatant and wet centrifuged solids, each phase was analyzed for the target analytes defined in the test plan. The analytical processing of the supernatant and wet centrifuged solids and distribution of the unprocessed and processed sample aliquots are detailed in Figure 7.1. ASR 6193 Addendum 1 and Addendum 2 and revisions 6193.01 throuugh 6193.06 provided instructions to the laboratory to successfully complete the analytical and QC requirements defined in the test plan.



(a) Mod 1 leach procedure; Mod 2 total digest procedure
(b) ICP-MS includes Cs and U isotopic
(c) ICP-MS includes U isotopic
(d) GEA, total alpha, AEA (Am/Cm), AEA (Pu), ^{90}Sr, ^{79}Se
(e) GEA, total alpha, AEA (Am/Cm), AEA (Pu), ^{90}Sr, ^{241}Pu
(f) Organic phosphate extracted but not reported due to technical problems

Figure 7.1. Flow Diagram for Analytical Processing of Samples

7.1 Supernatant

7.1.1 Direct Sub-Sampling

The AZ-101 supernatant was sub-sampled in the SAL hot cells and then delivered to the RPL analytical workstations for various measurements including inorganic anion, hydroxide, ammonia, cyanide, mercury, total organic and inorganic carbon (TOC/TIC), ^{3}H, ^{14}C, ^{99}Tc (pertechnetate), ^{127}I, and ^{129}I. For these sub-samples, the staff at the analytical workstation are responsible for ensuring that the appropriate batch and analytical QC samples are analyzed, as well as for providing any additional processing to the sub-samples that might be required (e.g., digestions for mercury analysis or distillations for cyanide analysis).

7.1.2 Direct Sub-sampling Followed by Ion Exchange for Dose Reduction

The AZ-101 supernatant was sub-sampled and subjected to an ion exchange procedure (i.e., procedure TPR-RPP-WTP-049, *Ion Exchange for Activity Reduction*) in the SAL to reduce the sample dose levels. The resulting effluents from the ion exchange procedure were delivered to the 329 Facility analytical workstations for measurements of organic acids and chelators. Besides a process blank (PB) and laboratory control sample/blank spike (LCS/BS), additional AZ-101 sample was processed through the ion exchange procedure to provide the analytical workstation with separate samples for the matrix spike (MS) and matrix spike duplicate (MSD). The staff at the analytical workstation are responsible for ensuring that the appropriate batch and analytical QC samples are analyzed as well as providing any additional processing to the sub-samples that might be required (e.g., derivatization for the chelators).

7.1.3 Acid Digestion

The AZ-101 supernatant was acid digested in the SAL according to procedure PNL-ALO-128, *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*. Aliquots of the digested sub-samples were delivered to the 329 Facility for inductively coupled plasma-mass spectrometry (ICP-MS) and to various RPL analytical workstations for ICP-AES, total U by KPA and the following radiochemical analyses: total alpha, gamma emitters by GEA, ^{79}Se, ^{90}Sr, ^{238}Pu, ^{239,240}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm.

The SAL processed 1-mL aliquots of the supernatant in triplicate. The acid extracted solutions were brought to a nominal 25-mL volume, and absolute volumes were determined based on final solution weights and densities. Along with the triplicate samples, the SAL processed a digestion PB, two LCS/BSs (one for ICP-AES and one for ICP-MS), and two MSs (one for ICP-AES and one for ICP-MS). Aliquots of the LCS/BS, MSs, and the PB were sent with aliquots of the triplicate samples for ICP-AES or ICP-MS analyses. For radiochemical analyses, only the PB was sent with aliquots of the triplicate samples for analysis. Post-digestion LCS/BS and MS samples were prepared at the time of radiochemical separation except for gamma energy analysis (GEA), which did not require any additional sample preparation.

7.1.4 Solvent Extraction for Organic Phosphates

The AZ-101 supernatant was sampled and extracted in the SAL for analysis of D2EHP according to the test plan TP-RPP-WTP-047, *Identification and Quantification of D2EHP in Tank Waste*.
Sub-samples consisted of triplicate aliquot samples of the supernatant (surrogate spike only) and duplicate

MS samples (surrogate and D2EHP spike) adjusted to pH <2. A PB consisting of DIW (surrogate spike only) and a LCS/BS consisting of DIW spiked with D2EHP were processed with the sample batch.

Five-mL aliquots of the samples were extracted three times with 25-mL portions of methylene chloride followed by three contacts with 25-mL portions of butanol. The extracts were transferred from the SAL in RPL to the 329 Facility analysis workstation. The methylene chloride extracts were concentrated to a volume of less than 1 mL, derivatized with diazomethane/ether solution, and concentrated to a final volume of 1 mL for analysis. The analysis was performed per test plan TP-RPP-WTP-047 using gas chromatography with a flame ionization detector (GC/FID).

7.2 Wet Centrifuged Solids

7.2.1 Direct Sub-Sampling/Analysis

The AZ-101 wet centrifuged solids were sub-sampled in the SAL and then delivered to the RPL analytical workstations for various measurements, including cyanide, mercury, TOC/TIC, and ^{14}C. For these sub-samples, the staff at the analytical workstation are responsible for ensuring that the appropriate batch and analytical QC samples are analyzed, as well as for providing any additional processing to the sub-samples that might be required (e.g., combustion for TOC/TIC and ^{14}C).

7.2.2 Water Leach

The AZ-101 wet centrifuged solids were leached with DIW in the SAL according to procedure PNL-ALO-103, *Water Leach of Sludges, Soils, and other Solids Samples*. The SAL prepared triplicate samples of the wet centrifuged solids for anions and tritium analysis on three separate occasions. Triplicate samples were prepared for 1) inorganic anions by leaching approximately 1 g of wet centrifuged solids with 10 mL of water and 2) tritium by leaching approximately 0.14 g of wet centrifuged solids with 16 mL of water. Besides the triplicate samples, the SAL prepared a PB, MS, and LCS/BS for each analysis. Aliquots of the leached samples, LCS/BS, MS and PB were delivered to RPL analytical workstation for analysis.

7.2.3 Acid Leach (PNL-ALO-129 Mod 1)

The AZ-101 wet centrifuged solids were acid leached in the SAL according to procedure PNL-ALO-129, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater (Mod 1)*. The 'Mod 1' modification(5) to the PNL-ALO-129 procedure slightly changes the quantities of HCl and HNO_3 used for leaching the samples. The SAL processed approximately 0.5-g aliquots of the wet centrifuged solids in triplicate. The acid-extracted solutions were brought to a nominal 25-mL volume, and absolute volumes were determined based on final solution weights and densities. Along with the samples, the SAL processed a PB, two LCS/BSs (one for ICP-AES and one for ICP-MS) and two MSs (one for ICP-AES and one for ICP-MS). Aliquots of the digested samples, LCS/BS, MS, and PB were delivered to the 329 Facility for ICP-MS analysis and to the ICP-AES analytical workstation for analysis.

The wet centrifuged solids processed by this acid leach procedure were not totally dissolved. A significant quantity of fine white flocculants and dark grainy particulates remained in the leaching vials after bringing the samples to final volume. No estimate could be made of the residual solids. Following sub-sampling of the digestion solutions for ICP-AES and ICP-MS analysis, the liquid was removed from the solids (to the extent possible). The residual solids were quantitatively transferred to Teflon™ digestion vials, assigned a different RPL number (i.e., 01-02273), and subjected to an additional acid digest using PNL-ALO-129 Mod 2 (see Section 7.2.4).

7.2.4 Acid Digestion (PNL-ALO-129 Mod 2)

The AZ-101 wet centrifuged solids were acid digested in the SAL according to procedure PNL-ALO-129, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater (Mod 2)*. The 'Mod 2' modification(6) to the PNL-ALO-129 procedure includes changes to the quantities of HCl and HNO_3 used, addition of HF, evaporation to dryness, and extended digestion temperatures and times. With addition of HF and evaporation to dryness, this digestion is not applicable to silicon or to metals that form volatile fluorides (this procedure is also not applicable to rare earths without additional modification). The SAL processed ~0.35-g aliquots of the wet centrifuged solids in triplicate. The digestion solutions were brought to a final volume of 100 mL. Along with the samples, the SAL processed a digestion PB, two LCS/BSs (one for ICP-AES and one for ICP-MS) and two MSs (one for ICP-AES and one for ICP-MS). Also, the residues remaining following the 'Mod 1' digestion were processed using the 'Mod 2' procedure. All samples processed by the 'Mod 2' digestion procedure appeared to be totally dissolved. Aliquots of the digested wet centrifuged solids samples, LCS/BS, MS, PB, and 'Mod 1' residue samples were delivered to the 329 Facility for ICP-MS analysis and to the ICP-AES analytical workstation for analysis.

7.2.5 Fusion Digestion (PNL-ALO-115)

The AZ-101 wet centrifuged solids were prepared in the SAL according to procedure PNL-ALO-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion*. Aliquots of the dissolved sub-samples were delivered to the 329 Facility for ICP-MS and to various RPL analytical workstations for ICP-AES, total U by KPA and the following radiochemical analyses: total alpha, gamma emitters by GEA, ^{90}Sr, ^{238}Pu, ^{241}Pu, ^{239,240}Pu, ^{242}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm.

The SAL processed 0.18-g to 0.26-g aliquots of the wet centrifuged solids in triplicate. The fusion digestions were brought to a final volume of 100 mL. A brown colloidal-like suspension was observed on the bottom of the volumetric flasks. Therefore, the contents of the flask were homogenized by vigorous shaking prior to aliquoting sub-samples for analysis. Along with the triplicate samples, the SAL processed duplicate fusion PBs, two LCS/BSs (one for ICP-AES and one for ICP-MS), a MS (for ICP-MS), and a solid LCS (NIST SRM-2710). Aliquots of the PBs, LSC/BSs, MSs, and the solid LCS were sent with aliquots of the triplicate samples for ICP-AES or ICP-MS analyses. For radiochemical analyses, only the PBs were sent with aliquots of the triplicate samples for analysis. Post-digestion LCS/BS and MS samples were prepared at the time of radiochemical separation except for GEA, which did not require any additional sample preparation.

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5 Modification documented in TP-RPP-WTP-023 Rev 0, Regulatory DQO Step 1: MDL/EQL Evaluation for Metals by ICP-AES in Tank Waste, JJ Wagner, 2001.

6 Modification documented in TP-RPP-WTP-023 Rev 0, Regulatory DQO Step 1: MDL/EQL Evaluation for Metals by ICP-AES in Tank Waste, JJ Wagner, 2001.

7.2.6 Fusion Digestion (PNL-ALO-116, Platinum Group Metals only)

The AZ-101 wet centrifuged solids were prepared in the SAL according to procedure PNL-ALO-116, *Solubilization of Platinum Group Metals from Solids Using a* Na_2O_2*-NaOH-NaCl Fusion*. The SAL processed approximately 0.2-g aliquots of the wet centrifuged solids in triplicate. The fusion digestions were brought to a final volume of 100 mL; a slight cloudiness was observed in the flask but cleared upon heating. Besides the triplicate samples, the SAL prepared duplicate PBs, a LCS/BS, and a MS. Aliquots of all digestions were delivered to the 329 Facility for analysis of Pt, Pd, Rh, and Ru by ICP-MS.

7.2.7 Fusion Digestion (PNL-ALO-114, Iodine only)

The AZ-101 wet centrifuged solids were prepared in the SAL for iodine analysis according to procedure PNL-ALO-114, *Solubilization of Metals from Solids Using a* Na_2O_2*-NaOH Fusion (Iodine Analysis Option)*. The SAL processed approximately 0.2-g aliquots of the wet centrifuged solids in triplicate. The fusion digestions were brought to a final volume of 100 mL. All samples (except PB and LCS/BS), exhibited brownish particulates on the bottom of the flask. The samples were allowed to settle over night and aliquots withdrawn without remixing. Along with the triplicate samples, the SAL prepared duplicate PBs, a LCS/BS, and a MS. Aliquots of all digestions were delivered to the 329 Facility for analysis of ^{127}I and ^{129}I by ICP-MS.

8.0 Analytical Results

8.1 Introduction

The inorganic, radioisotopic, and organic analytical results for the AZ-101 as-received supernatant and wet centrifuged solids samples are provided in Table 8.1 through Table 8.4. Results are reported in µg/mL, µg/g, µCi/mL, or µCi/g, as appropriate. For many radioisotope analyses, the nominal propagated uncertainties are provided as 1-σ, unless otherwise noted. For the inorganic and organic analyses, no uncertainties are included in the tables; the estimated uncertainty is 10-15% for results above the estimated quantitation limit (EQL) (See 8.3 Data Limitations for analyses that exceed this estimated uncertainty). Besides the triplicate sample results, the results obtained on the PBs are also reported, as appropriate.

The analyte concentrations reported in Tables 8.1 through 8.4 include a data flag column (i.e., a data qualifier code). The data flag is developed from the QC results (found in Table 9.1 through Table 9.4) and concentration results are flagged, as appropriate. The data flags are taken from the QA Plan and are defined below, as they relate to this report:

U	Undetected. Analyte was analyzed but not detected (e.g., no measurable instrument response) or response was less than the MDL. (Note: For some analyses, no results are reported below an EQL established by the lowest calibration standard adjusted for sample and analytical dilutions. In these cases, results less than the EQL are flagged with a U. Footnotes in the tables identify which analyses use the lowest calibration standard as the reporting level.)
J	Estimated value. The value reported is below the EQL and above the MDL. For radiochemical data, the J flag identifies results that have a propagated error of >10%, indicating that the results are typically within 10 times the minimum detectable activity (MDA).
B	Analyte found in associated PB above the QA plan acceptance criteria (i.e., the analyte concentration in the blank is greater than the EQL or exceeds 5% of sample concentration).
X	A QC deficiency is associated with the reported result. For this report the X flag is used for the following: a) LCS (LCS/BS) fails or was not analyzed, b) both the MS and the post spike (PS) fail, and c) serial dilution test (if required) fails for analytes with concentration greater than 0.1%.

The term MDL used in this report is an estimated MDL. That is, the MDLs have not been determined on the AZ-101 waste matrix per SW-846 (7) protocol. For most inorganic and organic methods, the estimated MDLs are based on an instrument detection limit (IDL) established from using reagents and/or low concentration high-purity standards as samples and evaluating instrument response near background levels. For mercury and cyanide the MDLs are based on the MDLs established from the Regulatory DQO work (Patello 2001) using samples from Tanks 241-AN-102 (supernatant) and 241-AY-102 (solids). For

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7 U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition, Office of Solid Waste and Emergency Response. Washington, D.C.

radiochemical methods, the MDA is calculated per the QA Plan and is based on the background counting statistics.

The EQL is typically set at 10 times the estimated MDL adjusted for dilution factors resulting from digestion or leaching processing. For a few methods (e.g., IC and ammonia), no ‘estimated’ MDL is determined and the EQL is based on the lowest calibration standard; no results are reported below the EQL for these methods. For radiochemical methods, no EQL is established; however, results are flagged with a ‘J’ when uncertainty exceeds 10%. Specific QC and QA discussions are given in Section 9.4.

8.2 Analyte List or Method Deviations

The supernatant analytes (i.e., liquid fraction analyses) and undissolved solids analytes (i.e., HLW solids analyses) and recommended analysis methods are defined by the test specification. A few modifications to the analyte list or procedures defined by the test specification are detailed below:

- The laboratory was directed to determine pertechnetate ($^{99}TcO_4^-$), as opposed to total ^{99}Tc, using separations and beta counting techniques. Procedure PNL-ALO-432, *Separation of Technetium by Cation Exchange and Solution Extraction Prior to Measurement by Beta Counting*, was modified slightly to exclude the sample oxidation step so that the non-pertechnetate fraction was not oxidized.
- Results for analytes not specified by the test specification are included in this report for information only. These additional analytes are measured as part of the method and may or may not have adequate QC for validating the results.
- The ^{135}Cs and ^{137}Cs concentration in the wet centrifuged solids was not measured by ICP-MS, but calculated based on the assumption the cesium isotopic ratio in the wet centrifuged solids is equivalent to the isotopic ratio in the supernatant. The ^{133}Cs in the wet centrifuged solids and supernatant was measured and the ^{135}Cs and ^{137}Cs are calculated based on the supernatant cesium isotope mass ratios.
- The wet centrifuged solids were prepared by using a modification to the acid leaching procedure PNL-ALO-129, HNO3-HCl Acid Extraction of Solids Using a Dry-Block Heater. The modification provided an acid digestion procedure that dissolved all the wet centrifuged solids material, allowing the results to be compared to those from the fusion digestion (see Section 7.2.4 and Appendix G).
- The supernatant uranium analysis by KPA required an ion exchange separation prior to analysis to mitigate interference effects (see Section 9.3).
- One analyte defined in the test specification (i.e., D2EHP) could not be measured due to the lack of reliable methods (see Section 9.13).
- The tritium in both the supernatant and wet centrifuged solids was very low and the ^{137}Cs beta activity very high. This required a minor modification to the analytical separations procedure (i.e., ion exchange to remove beta emitters other than tritium, and a double distillation) (see Section 9.4.6).
- For both the wet centrifuged solids and especially the supernatant, a violent reaction between the sample matrix and the cyanide releasing agent required a minor modification to the cyanide

procedure. However, the modification did not totally mitigate the problems and low recoveries were common (see Section 9.7).

- Per the test specification, ammonia was not an analyte of interest for the wet centrifuged solids analysis. Therefore, comparison of the solids fraction to the Contract Specification 8 (Envelope D) was not possible.

8.3 Data Limitations

- The fluoride results on both the supernatant and wet centrifuged solids represent the summation of fluoride, acetate, and formate (if present), as these anions cannot adequately be resolved using the IC procedure used for measuring inorganic anions. Thus, the fluoride results may be biased high.
- The ICP-MS result for the AZ-101 supernatant at atomic mass unit 241 (AMU-241) is ^{241}Am, ^{241}Pu, or a combination of both. However, no response was detected at AMU-241 above the ^{241}Am MDL of 1E-03 µg/mL, which is approximately an order of magnitude below the MRQ of 8.7E-03 µg/mL for ^{241}Pu/^{241}Am.
- Concentrations of numerous elements are reported by ICP-MS. Element concentrations are determined by comparison of a selected isotopic mass response for a given element to the calibration curve generated for that element. However, the calibration curve is based on natural abundance, and many of the analytes measured may not have a natural isotopic distribution. Elements such as Rb, Ru, Pd, Sb, Se, Mo, Ce, Te, and AMU-151 (Sm) likely have significantly altered isotopic ratios. For accurate analysis of elements with altered isotopic distributions, chemical separation of the element is required so that individual atomic masses can be quantified. Except for the Cs, U, and Pu, no chemical separations were performed prior to the ICP-MS analysis of the AZ-101 as-received sample.
- For ICP-MS analysis of ^{99}Tc the uncertainty is estimated at ±30%, versus the typical 10-15% for ICP-MS analyses. Approximately a year following the ^{99}Tc analysis, the calibration and verification standards used for the analysis were analyzed by liquid scintillation counting (LSC) and although the standards measured within acceptance criteria, the measured values for both standards were biased slightly high from the assigned standard value. To ensure that the ^{99}Tc results are bounded properly, the uncertainty has been increased to include the measured-to-assigned value differences.
- The average IC phosphate result of 1,630 µg/mL for the supernatant is consistent with the ICP-AES phosphorous results 504 µg/mL (i.e., 1,550 µg/mL as phosphate). However, the average IC phosphate result of 250 µg/g for the leached wet centrifuged solids does not compare well with the ICP-AES phosphorus result from either the KOH fusion (350 µg/g or 1,070 µg/g as phosphate) or the Mod 2 acid digestion (1,790 µg/g or 5,500 µg/g), indicating water insoluble phosphate. Additional phosphorus data was obtained from the Na_2O_2 fusions used for the ICP-MS platinum group metals analysis. At an average of about 1,900 µg/g, these results compare favorably with the Mod 2 results, suggesting that the KOH fusion results for phosphorus may be significantly biased low.

- There are few QC failures (i.e., failure of QC sample to meet the QC flagging criteria established by the test specification). These failures do not impact the usability of the reported results except in those cases where the results are flagged with an 'X', which indicates a significant QC failure. The following results are flagged with an 'X' and are considered qualitative at best.
  - > **AZ-101 Supernatant**: Boron, silicon, and HEDTA.
  - > **AZ-101 Wet Centrifuged Solids**: Silver, calcium, zinc, and bismuth from fusion preparation, silicon from acid digestion preparation, total cyanide, and oxalate from water leach.
- The TIC and TOC results obtained from the hot persulfate oxidation method and the furnace oxidation method are significantly different. The hot persulfate TOC result and furnace TOC result differ by factor of 10. The TOC results produced by the furnace oxidation method appear to be biased high. The best TOC results are most likely estimated from the hot persulfate results for the TIC and furnace results for total carbon (TC); i.e., TOC = 9,670 - 8,380 = 1,290 µg/g.
- The supernatant ^{90}Sr result is significantly lower than other published data for the AZ-101 supernatant; see Section 9.4.4 for full discussion. It is recommended that the ^{90}Sr result from the first liquid sampled from the cells unit filter (CUF) operation (i.e., 1.19 µCi/mL) be used for the supernatant ^{90}Sr concentration.

8.4 General Observations

- The average total ^{99}Tc concentration measured by ICP-MS (3.7E-01 µCi/mL) is within about 15% of the ^{99}Tc measured as pertechnetate ($^{99}TcO_4^-$) by separation and beta counting (3.77E-01 µCi/mL), indicating that all of the technetium is in the pertechnetate form.
- Two analytes (Ce and Y) were analyzed by both ICP-AES and ICP-MS on the wet centrifuged solids within the MDL of the ICP-AES. The agreement between the results is excellent. The average results from ICP-AES are 380 and 110 µg/g for Ce and Y, respectively. For ICP-MS, the average results were 300 and 117 µg/g for Ce and Y, respectively.
- The supernatant ^{137}Cs concentration determined by ICP-MS agreed within 19% of the concentration determined by GEA. And, the wet centrifuged solids ^{137}Cs concentration calculated from ^{133}Cs measured by ICP-MS and the supernatant Cs isotopic mass fraction result agreed within 12% of the concentration determined by GEA.
- The ^{239,240}Pu supernatant result from alpha energy analysis (AEA) compare reasonably well with the sum of the ^{239}Pu and ^{240}Pu from ICP-MS (i.e., within 10%). The supernatant average being 1.8E-03 µg/mL from AEA versus the sum of 2.0E-03 µg/mL from ICP-MS; the ICP-MS results are 'B' flagged. Per the test specification, Pu was not measured in the wet centrifuged solids by ICP-MS.
- For the wet centrifuged solids, the total alpha measurements do not agree well with the sum of measured alpha-emitting radioisotopes (^{237}Np, ^{238}Pu, ^{239,240}Pu, ^{241}Am, ^{243,244}Cm, and ^{242}Cm). The total alpha is approximately 33% higher than the sum of the alpha emitters. The total alpha in the supernatant was below the MDL and no comparison could be made to the sum of the alpha emitters.

8.5 Analytical Results Tables

The analytical results for the AZ-101 as-received supernatant are presented in Table 8.1 and Table 8.2 and the AZ-101 as-received wet centrifuged solids results are presented in Table 8.3 and Table 8.4. The undissolved solids results corrected for interstitial supernatant analyte contribution are presented in Section 8.6. The comparison of the supernatant results to Contract Specification 7 and undissolved solids results to Contract Specification 8 are presented in Section 8.7 and Section 8.8, respectively.

Table 8.1. AZ-101 As-Received Supernatant – Radionuclide Results (µCi/mL)

			Process Blank (PB)				Sample (01-01844)				Duplicate (01-01844D)				Triplicate (01-01844T)			
Method	Prep	Radionuclides	MDA/ MDL µCi/mL	Result µCi/mL	(a) 1-sigma µCi/mL	DF	MDA/ MDL µCi/mL	Result µCi/mL	(a) 1-sigma µCi/mL	DF	MDA/ MDL µCi/mL	Result µCi/mL	(a) 1-sigma µCi/mL	DF	MDA/ MDL µCi/mL	Result µCi/mL	(a) 1-sigma µCi/mL	DF
H-3	Direct	^{3}H	2E-05	2.79E-05	6.42E-06	J	2E-05	1.82E-02	7.28E-04		2E-05	1.80E-02	7.20E-04		2E-05	1.81E-02	7.24E-04	
C-14	Direct	^{14}C (f)		NA			4E-05	1.98E-03	8E-05		4E-05	1.95E-03	7.80E-05		4E-05	1.85E-03	7.4E-05	
GEA	Acid-128	**^{60}Co**	1E-04	1E-04		U	4E-02	4E-02		U	5E-02	5E-02		U	5E-02	5E-02		U
Se-79	Acid-128	^{79}Se	2E-05	2.84E-05	7.18E-06	J	2E-05	9.76E-04	3.32E-05		4E-05	8.01E-04	3.84E-05		1E-05	1.13E-03	3.62E-05	
Sr-90	Acid-128	**^{90}Sr** (g)	1E-03	1E-03		U	1E-03	4.97E-02	2.49E-03		1E-03	4.76E-02	2.38E-03		1E-03	5.30E-02	2.65E-03	
ICP-MS	Acid-128	**^{99}Tc (h)**	2E-04	3.03E-03			2E-04	3.32E-01			2E-04	3.31E-01			2E-04	3.18E-01		
Tc-99	Direct	^{99}Tc^{+7}	6E-04	6E-04		U	6E-04	3.80E-01	7.60E-03		6E-04	3.74E-01	7.48E-03		6E-04	3.76E-01	7.52E-03	
GEA	Acid-128	^{126}Sn	1E-04	1E-04		U	2E+00	2E+00		U	2E+00	2E+00		U	2E+00	2E+00		U
ICP-MS	Direct	^{129}I	4E-07	4E-07		U	4E-07	9.90E-07		J	4E-07	1.06E-06		J	4E-07	1.03E-06		J
ICP-MS	Acid-128	^{135}Cs-MS (b)	1E-05	1.33E-05		J	2E-05	1.06E-02			2E-05	1.09E-02			2E-05	1.03E-02		
ICP-MS	Acid-128	^{137}Cs-MS (b)	9E-01	9.06E-01		J	1E+00	1.40E+03			1E+00	1.43E+03			1E+00	1.37E+03		
GEA	Acid-128	**^{137}Cs**	1E-04	2.25E-03	9.00E-05		7E-01	1.66E+03	3.33E+01		7E-01	1.64E+03	3.28E+01		7E-01	1.63E+03	3.25E+01	
GEA	Acid-128	**^{154}Eu**	4E-04	4E-04		U	2E-01	2E-01		U	2E-01	2E-01		U	1E-01	1E-01		U
GEA	Acid-128	^{155}Eu	4E-04	4E-04		U	2E+00	2E+00		U	2E+00	2E+00		U	2E+00	2E+00		U
GEA	Acid-128	^{231}Pa	6E-03	6E-03		U	2E+00	2E+00		U	2E+00	2E+00		U	2E+00	2E+00		U
ICP-MS	Acid-128	^{233}U	9E-06	9E-06		U	9E-06	9E-06		U	9E-06	9E-06		U	9E-06	9E-06		U
ICP-MS	Acid-128	^{234}U	6E-06	6E-06		U	6E-06	6E-06		U	6E-06	6E-06		U	6E-06	6E-06		U
ICP-MS	Acid-128	^{235}U	3E-09	1.86E-08		J	3E-09	4.66E-08		B	3E-09	4.75E-08		B	3E-09	4.78E-08		B
ICP-MS	Acid-128	^{236}U	6E-08	6E-08		U	6E-08	9.15E-08		J	6E-08	9.69E-08		J	6E-08	9.11E-08		J
ICP-MS	Acid-128	^{238}U	2E-09	3.60E-07			2E-09	9.22E-07		B	2E-09	9.56E-07		B	2E-09	9.36E-07		B
ICP-MS	Acid-128	**^{237}Np** (i)	7E-06	7.4E-06		U	7E-06	3.87E-05		J	7E-06	3.88E-05		J	7E-06	3.55E-05		J
AEA	Acid-128	^{238}Pu	6E-07	2.89E-05	1.44E-06		2E-05	3.77E-04	2.26E-05	B	1E-05	3.40E-04	2.04E-05	B	2E-05	3.14E-04	2.51E-05	B
AEA	Acid-128	^{239,240}Pu	7E-07	9.77E-06	7.82E-07		2E-05	1.76E-03	5.28E-05		7E-06	1.82E-03	5.47E-05		2E-05	1.83E-03	7.34E-05	
ICP-MS	Acid-128	^{239}Pu (i)	6E-04	1.36E-03		J	6E-04	2.49E-03		JB	6E-04	2.00E-03		JB	5E-04	1.50E-03		JB
ICP-MS	Acid-128	^{240}Pu (i)	2E-03	2E-03		U	2E-03	2E-03		U	2E-03	2E-03		U	2E-03	2E-03		U
AEA	Acid-128	^{241}Am	9E-07	4.07E-05	1.63E-06		1E-05	1.02E-04	1.13E-05	JB	1E-05	1.43E-04	1.43E-05	B	2E-05	6.95E-05	1.11E-05	JB
GEA	Acid-128	^{241}Am-GEA(c)	4E-04	4E-04		U	2E+00	2E+00		U	2E+00	2E+00		U	2E+00	2E+00		U

Table 8.1. (Cont'd)

			Process Blank (PB)				Sample (01-01844)				Duplicate (01-01844D)				Triplicate (01-01844T)			
Method	**Prep**	**Radionuclides**	**MDA/ MDL µCi/mL**	**Result µCi/mL**	**(a) 1-sigma µCi/mL**	**DF**	**MDA/ MDL µCi/mL**	**Result µCi/mL**	**(a) 1-sigma µCi/mL**	**DF**	**MDA/ MDL µCi/mL**	**Result µCi/mL**	**(a) 1-sigma µCi/mL**	**DF**	**MDA/ MDL µCi/mL**	**Result µCi/mL**	**(a) 1-sigma µCi/mL**	**DF**
AEA	Acid-128	^{242}Cm	5E-07	5E-07		U	9E-06	9E-06		U	9E-06	9E-06		U	1E-05	1E-05		U
AEA	Acid-128	^{243,244}Cm	7E-07	1.83E-05	1.10E-06		1E-05	3.46E-05	6.92E-06	JB	1E-05	4.89E-05	8.32E-06	JB	2E-05	2E-05		U
Alpha	Acid-128	Gross Alpha	6E-05	1.43E-04	2.15E-05	J	5E-03	5E-03		U	6E-03	6E-03		U	5E-03	5E-03		U
		Alpha Sum (d)		9.77E-05	2.54E-06			2.27E-03	5.91E-05			2.35E-03	6.12E-05			2.22E-03	7.76E-05	
		TRU (e)		9.78E-05				2.31E-03				2.39E-03				2.25E-03		

Bolded radionuclides required for comparison to Contract Specification 7 (Envelope B)

MDA: minimum detectable activity (used with all radiochemical analysis results)

MDL: method detection limit (used with ICP-MS results)

DF: Data quality flag (for definition of flags used see Section 8.1)

NA: Not applicable

(a) 1-sigma: Nominal propagated uncertainty including preparation and counting for radiochemistry methods only.

(b) ^{135}Cs-MS and ^{137}Cs-MS determined by HPIC-ICP-MS.

(c) Opportunistic radionuclides analysis; not required by test specification or test plan.

(d) Alpha Sum = Summation of AEA results only (^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm).

(e) TRU = Z>92, alpha emitter, half-life >10yr (^{237}Np, ${}^{239/,40}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm).

(f) Results corrected for laboratory blanks and verification standard recoveries per procedure PNL-ALO-482. Supernatant analyzed without processing; no PB prepared.

(g) Supernatant ^{90}Sr result is significantly lower than other published results (e.g., first filtered liquid from CUF operation; ^{90}Sr = 1.19E+00 µCi/mL); see discussion Section 8.3 and Section 9.4.4.

(h) Uncertainty estimated at ±30%; see Section 6.4 for further details.

(i) Same certified source standard used to prepare calibration and verification standards for ICP-MS. Calibration and verification standards prepared approximately 1 year apart; prepared standards verified by independent analysis (i.e., LSC, AEA, or GEA).

Nominal decay correction reference dates:
ICP-MS: U (3/2002), Pu/Np (4/2002), Tc (2/2002) and I (5/2003)
Radchem: GEA, Gross Alpha, AEA Pu and Am/Cm (10/2001); ^{90}Sr (9/2002); ^{99}Tc (8/2002); ^{79}Se (11/2001); ^{3}H (2/2002); and ^{14}C (6/2002)

Table 8.2. AZ-101 As-Received Supernatant— Analyte Results (µg/mL)

			Process Blank (PB)			Sample (01-01844)			Duplicate (01-01844D)			Triplicate (01-01844T)		
Method	**Prep**	**Analyte**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**
IC-Org	Direct	Acetate (f)	140	140	U	140	140	U	140	140	U	140	140	U
ICP-AES	Acid-128	Ag (f)	0.7	0.7	U	0.7	0.7	U	0.6	0.6	U	0.6	0.6	U
ICP-AES	Acid-128	**Al**	1.6	7.1	J	1.6	6,090		1.5	6,050		1.5	6,020	
ISE	Direct	Ammonia	1.5	1.5	U	1.5	3.0	J	1.5	2.3	J	1.5	1.7	J
ICP-MS	Acid-128	AMU-241(e)	0.0011	0.0011	U	0.0011	0.0011	U	0.0011	0.0011	U	0.0011	0.0012	J
ICP-AES	Acid-128	As (f)	6.6	6.6	U	6.7	9.7	J	6.4	10.0	J	6.3	10.0	J
ICP-AES	Acid-128	B	1.3	60.6	X	1.3	66	BX	1.3	85	BX	1.3	82	BX
ICP-AES	Acid-128	**Ba**	0.3	0.3	U	0.3	0.3	U	0.3	0.3	U	0.3	0.3	J
ICP-AES	Acid-128	Be (f)	0.3	0.3	U	0.3	0.3	U	0.3	0.3	U	0.3	0.3	U
ICP-AES	Acid-128	Bi (f)	2.6	2.6	U	2.7	2.7	U	2.6	2.6	U	2.5	2.5	U
IC-Inorg	Direct	Br	130	130	U	130	620		130	660		130	660	
C (HP)	Direct	C as TC (d,f)		NA		61	10,400		61	10,300		61	10,400	
C (Furn)	Direct	C as TC (f)		NA		89	7,320		89	7,240		89	7,480	
C (HP)	Direct	**C as TIC**		NA		34	9,810		34	9,820		34	9,920	
C (Furn)	Direct	**C as TIC** (c)		NA		170	5,540		170	6,080		170	5,880	
C (HP)	Direct	**C as TOC**		NA		87	560		87	500		87	470	
C (Furn)	Direct	**C as TOC**		NA		250	1,780		250	1,160	J	250	1,600	
ICP-AES	Acid-128	**Ca**	6.6	6.6	U	6.7	9.3	J	6.4	6.4	U	6.3	6.3	U
ICP-AES	Acid-128	**Cd**	0.4	0.4	J	0.4	0.9	JB	0.4	0.8	JB	0.4	0.7	JB
ICP-AES	Acid-128	Ce	5.3	5.3	U	5.3	5.3	U	5.1	5.1	U	5.0	5.0	U
IC-Org	Direct	Citrate	410	410	U	410	410	U	410	410	U	410	410	U
GC/FID	Derivatize	Citric acid	5.8	5.8	U	5.8	5.8	U	5.8	5.8	U	5.8	5.8	U
IC-Inorg	Direct	**Cl** (k)	130	130	U	130	230		130	220		130	280	
CN	Distill-287	CN	0.055	0.099	J	0.057	1.53	B	0.053	1.19	B	0.054	3.40	
ICP-AES	Acid-128	Co (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
ICP-AES	Acid-128	**Cr**	0.5	0.5	U	0.5	689		0.5	685		0.5	683	
ICP-MS	Acid-128	^{133}Cs-MS (b)	0.013	0.013	U	0.016	27		0.016	28		0.015	26	
ICP-MS	Acid-128	^{135}Cs-MS (b,f)	0.011	0.011	J	0.013	8.8		0.014	9.1		0.014	8.6	

Table 8.2. (Cont'd)

			Process Blank (PB)			Sample (01-01844)			Duplicate (01-01844D)			Triplicate (01-01844T)		
Method	**Prep**	**Analyte**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**	**MDL µg/mL**	**Results (j) µg/mL**	**DF**
ICP-MS	Acid-128	^{137}Cs-MS (b,f)	0.01	0.01	J	0.013	16		0.015	16		0.013	16	
ICP-MS	Acid-128	^{133}Cs	0.031	0.065	J	0.0089	26.2		0.0092	26.8		0.0088	26.3	
ICP-AES	Acid-128	Cu (f)	0.7	0.7	U	0.7	0.7	U	0.6	0.6	U	0.6	0.6	U
None	None	D2EHP (i)		(i)			(i)			(i)			(i)	
ICP-AES	Acid-128	Dy (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
GC/FID	Derivatize	ED3A	4.9	4.9	U	4.9	4.9	U	4.9	4.9	U	4.9	4.9	U
GC/FID	Derivatize	EDTA	4.9	4.9	U	4.9	4.9	U	4.9	4.9	U	4.9	4.9	U
ICP-AES	Acid-128	Eu (f)	2.6	2.6	U	2.7	2.7	U	2.6	2.6	U	2.5	2.5	U
IC-Inorg	Direct	**F** (g,k)	125	125	U	125	1,960		125	2,020		125	2,040	
ICP-AES	Acid-128	**Fe**	0.7	0.8	J	0.7	2.1	JB	0.6	1.3	JB	0.6	1.1	JB
IC-Org	Direct	Formate	180	180	U	180	180	U	180	370	J	180	180	U
None	None	Gluconate (h)	450	450	U	450	450	U	450	450	U	450	450	U
IC-Org	Direct	Glycolate (h)	170	170	U	170	170	U	170	170	U	170	170	U
GC/FID	Derivatize	HEDTA	8.8	8.8	UX	8.8	8.8	UX	8.8	8.8	UX	8.8	8.8	UX
CVAA	Acid-131	**Hg**	0.00013	0.00064	J	0.00014	0.067		0.00011	0.030		0.00014	0.069	
ICP-MS	Direct	^{127}I	0.007	0.007	U	0.007	0.007	U	0.007	0.007	U	0.007	0.007	U
GC/FID	Derivatize	IDA	11	11	U	11	11	U	11	11	U	11	11	U
ICP-AES	Acid-128	**K**	53	53	U	53	4,500		51	4,460		50	4,420	
ICP-AES	Acid-128	**La**	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
ICP-AES	Acid-128	Li	0.8	0.8	U	0.8	0.9	J	0.8	0.9	J	0.8	0.9	J
ICP-AES	Acid-128	Mg	2.6	2.6	U	2.7	2.7	U	2.6	2.6	U	2.5	2.5	U
ICP-AES	Acid-128	Mn (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
ICP-AES	Acid-128	Mo (f)	1.3	1.3	U	1.3	89.2		1.3	88.7		1.3	88.4	
ICP-AES	Acid-128	Na	3.9	70.6		20	111,000		19	112,000		19	110,000	
ICP-AES	Acid-128	Nd (f)	2.6	2.6	U	2.7	2.7	U	2.6	2.6	U	2.5	2.5	U
ICP-AES	Acid-128	**Ni**	0.8	2.7	J	0.8	1.2	JB	0.8	1.0	JB	0.8	0.8	JB
IC-Inorg	Direct	**NO$_2$** (k)	2,500	2,500	U	2,500	60,700		2,500	61,600		2,500	61,600	
IC-Inorg	Direct	**NO$_3$** (k)	2,500	2,500	U	2,500	52,000		2,500	52,900		2,500	52,800	
GC/FID	Derivatize	NTA	5.6	5.6	U	5.6	5.6	U	5.6	5.6	U	5.6	5.6	U
Titration	Direct	OH	170	170	U	170	11,300		170	11,400		170	11,400	

Table 8.2. (Cont'd)

			Process Blank (PB)			Sample (01-01844)			Duplicate (01-01844D)			Triplicate (01-01844T)		
Method	**Prep**	**Analyte**	**MDL μg/mL**	**Results (j) μg/mL**	**DF**	**MDL μg/mL**	**Results (j) μg/mL**	**DF**	**MDL μg/mL**	**Results (j) μg/mL**	**DF**	**MDL μg/mL**	**Results (j) μg/mL**	**DF**
IC-Inorg	Direct	Oxalate (k)	250	250	U	250	930		250	1,030		250	1,030	
IC-Org	Direct	Oxalate	210	210	U	210	1,100		210	960		210	1,000	
ICP-AES	Acid-128	P	2.6	2.6	U	2.7	506		2.6	505		2.5	500	
ICP-AES	Acid-128	**Pb**	2.6	2.6	U	2.7	3.4	J	2.6	3.3	J	2.5	3.3	J
ICP-AES	Acid-128	Pd (f)	20	20	U	20	20	U	19	20	J	19	20	J
IC-Inorg	Direct	**PO_4** (k)	250	250	U	250	1,610		250	1,640		250	1,630	
ICP-MS	Acid-128	Rb	0.025	0.32		0.024	9.53		0.025	9.54		0.026	8.53	
ICP-AES	Acid-128	Rh (f)	7.9	7.9	U	8.0	8.0	U	7.7	7.7	U	7.5	7.5	U
ICP-AES	Acid-128	Ru (f)	29	29	U	29	29	U	28	28	U	28	28	U
ICP-AES	Acid-128	Sb (f)	13	13	U	13	13	U	13	13	U	13	13	U
ICP-AES	Acid-128	Se (f)	6.6	6.6	U	6.7	6.7	U	6.4	6.4	U	6.3	6.3	U
ICP-AES	Acid-128	Si (f)	13	138	X	13	194	BX	13	221	BX	13	204	BX
ICP-AES	Acid-128	Sn (f)	39	39	U	40	46	J	38	47	J	38	47	J
IC-Inorg	Direct	**SO_4** (k)	250	250	U	250	15,700		250	16,400		250	16,400	
ICP-AES	Acid-128	Sr (f)	0.4	0.4	U	0.4	0.4	U	0.4	0.4	U	0.4	0.4	U
GC/FID	Derivatize	Succinic acid	6.1	19	J	6.1	52	J	6.1	52	J	6.1	47	J
ICP-AES	Acid-128	Te (f)	39	39	U	40	40	U	38	38	U	38	38	U
ICP-AES	Acid-128	Th	26	26	U	27	27	U	26	26	U	25	25	U
ICP-AES	Acid-128	Ti (f)	0.7	0.7	U	0.7	0.7	U	0.6	0.6	U	0.6	0.6	U
ICP-AES	Acid-128	Tl (f)	13	13	U	13	13	U	13	13	U	13	13	U
ICP-AES	Acid-128	**U**	53	53	U	53	53	U	51	51	U	50	50	U
KPA	Acid-128	**U**	0.0004	0.0016	J	0.0004	0.50		0.0004	0.52		0.0004	0.52	
ICP-MS	Acid-128	**U** (a)	0.0044	1.08		0.0045	2.77	B	0.0047	2.87	B	0.0046	2.81	B
ICP-AES	Acid-128	V	1.3	1.3	U	1.3	1.5	J	1.3	1.5	J	1.3	1.5	J
ICP-AES	Acid-128	W	53	53	U	53	58	J	51	58	J	50	58	J
ICP-AES	Acid-128	Y (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
ICP-AES	Acid-128	Zn (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U
ICP-AES	Acid-128	Zr (f)	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U	1.3	1.3	U

Table 8.2. (Cont'd)

			Process Blank (PB)			Sample (01-01844)			Duplicate (01-01844D)			Triplicate (01-01844T)		
Method	Prep	Analyte	MDL µg/mL	Results [(j)] µg/mL	DF	MDL µg/mL	Results [(j)] µg/mL	DF	MDL µg/mL	Results [(j)] µg/mL	DF	MDL µg/mL	Results [(j)] µg/mL	DF

Bolded analytes required for comparison to Contract Specification 7 (Envelope B)

MDL: method detection limit (with all processing factors applied)
DF: data quality flag (for definition of flags used see Section 8.1)

AMU: atomic mass unit

D2EHP: bis-(2-ethylhexyl) phosphate

EDTA: ethylenediaminetetraacetic acid

ED3A: ethylenediaminetriacetic acid

HEDTA: N-(2-hydroxyethyl) ethylenediaminetriacetic acid

IDA: iminodiacetic acid

NTA: nitrilotriacetic acid

NA: not applicable

(a) U analysis is the sum of all measured isotopes of U by ICP-MS.

(c) TIC by difference (TC Furn - TOC Furn).

(d) TC by summation (TIC HP + TOC HP).

(e) AMU-241 calibrated using Am-241; AMU-241 is typically the sum of ^{241}Pu and ^{241}Am.

(f) Opportunistic analyte; not included in test specification or test plan.

(g) The fluoride results are upper bound concentration; peak shape and retention time suggests the presence of co-eluting anion(s), possibly formate.

(h) Glycolate and gluconate results should be considered the upper bound concentration, since glycolate and gluconate are not resolved by the IC measurement method used for the analysis. IC system calibrated using glycolate; gluconate estimate based on gluconate-to-glycolate response factor. Each result assumes 100% of response due to each analyte

(i) Analytes not measured due to lack of reliable method.

(j) Typical analysis precision/accuracy better than ±15% for results >10xMDL (i.e., results without a U or J flag).

(k) MDL is based on the lowest calibration standard adjusted for sample dilution; equivalent to SW-846 EQL definition.

Table 8.3. AZ-101 As-Received Wet Centrifuged Solids – Radionuclide Results (µCi/g)

			Process Blank (PB-1)				Process Blank (PB-2)				Sample (01-01845)				Duplicate (01-01845D)				Triplicate (01-01845T)			
Method	**Prep**	**Radionuclide**	**MDA/ MDL µCi/g**	**Result µCi/g**	**(a) 1-sigma µCi/g**	**DF**	**MDA/ MDL µCi/g**	**Result µCi/g**	**(a) 1-sigma µCi/g**	**DF**	**MDA/ MDL µCi/g**	**Result µCi/g**	**(a) 1-sigma µCi/g**	**DF**	**MDA/ MDL µCi/g**	**Result µCi/g**	**(a) 1-sigma µCi/g**	**DF**	**MDA/ MDL µCi/g**	**Result µCi/g**	**(a) 1-sigma µCi/g**	**DF**
H-3	Leach-103	**^{3}H**	2E-05	2E-05		U		n/a			4E-04	7.19E-03	3.60E-04		4E-04	7.05E-03	3.53E-04		4E-04	7.00E-03	3.50E-04	
C-14	Combust	**^{14}C** (e)	2E-04	4.21E-04	6.32E-05	J		n/a			2E-04	4.62E-04	5.54E-05	JB	2E-04	1.56E-03	9.36E-05	B	2E-04	1.11E-03	7.77E-05	B
GEA	Fusion-115	**^{60}Co**	2E-03	2E-03		U	2E-03	2E-03		U	3E-01	2.06E+00	1.65E-01		2E-01	1.97E+00	6.38E-01		3E-01	2.06E+00	1.44E-01	
Sr-90	Fusion-115	**^{90}Sr**	8E-03	6.80E-02	2.72E-03		1E-02	8.75E-02	3.50E-03		4E+02	2.12E+04	6.36E+02		3E+02	2.03E+04	6.09E+02		4E+02	1.91E+04	5.73E+02	
GEA	Fusion-115	**^{125}Sb**	7E-03	7E-03		U	8E-03	8E-03		U	6E+00	6E+00		U	4E+00	8.07E+00	1.29E+00	J	5E+00	5E+00		U
GEA	Fusion-115	**^{126}Sn**	3E-03	3E-03		U	3E-03	3E-03		U	2E+00	2E+00		U	2E+00	2E+00		U	3E+00	3E+00		U
ICP-MS	Fusion-114	**^{129}I**	2E-08	2E-08		U	2E-08	2E-08		U	7E-06	8.28E-06		J	8E-06	1.40E-05		J	9E-06	1.04E-05		J
GEA	Fusion-115	^{134}Cs	3E-03	3E-03		U	2E-03	2E-03		U	7E-01	7E-01		U	6E-01	6E-01		U	6E-01	6E-01		U
ICP-MS	Fusion-115	^{135}Cs (b)	1E-06	1E-06		U	1E-06	1E-06		U	1E-06	6.72E-03			1E-06	6.66E-03			1E-06	6.50E-03		
GEA	Fusion-115	**^{137}Cs**	3E-03	1.10E-01	4.40E-03		3E-03	2.39E-01	9.56E-03		2E+00	7.51E+02	2.25E+01		2E+00	7.64E+02	3.06E+01		2E+00	7.42E+02	2.97E+01	
ICP-MS	Fusion-115	**^{137}Cs** (b)	2E-01	2E-01		U	2E-01	2E-01		U	2E-01	8.88E+02			1E-01	8.81E+02			2E-01	8.60E+02		
ICP-MS	Fusion-115	^{151}AMU (Sm)(f)	1E-02	6.36E-01			1E-02	2.25E-01			1E-02	5.55E+02			9E-03	5.34E+02			1E-02	5.31E+02		
GEA	Fusion-115	**^{152}Eu**	8E-03	8E-03		U	9E-03	9E-03		U	2E+00	2E+00		U	8E-01	8E-01		U	1E+00	1E+00		U
GEA	Fusion-115	**^{154}Eu**	5E-03	5E-03		U	6E-03	6E-03		U	1E+00	2.27E+01	6.81E-01		9E-01	2.45E+01	7.35E-01		8E-01	2.31E+01	6.93E-01	
GEA	Fusion-115	**^{155}Eu**	8E-03	8E-03		U	8E-03	8E-03		U	8E+00	3.27E+01	2.62E+00		7E+00	3.38E+01	2.78E+00		6E+00	2.52E+01	2.27E+00	
ICP-MS	Fusion-115	^{234}U	7E-06	2.47E-05		J	7E-06	1.56E-05		J	9E-06	1.84E-03			6E-06	1.72E-03			7E-06	1.78E-03		
AEA	Fusion-115	^{236}Pu	8E-06	8E-06		U	1E-05	1E-05		U	4E-02	4E-02		U	3E-02	3E-02		U	3E-02	3E-02		U
ICP-MS	Fusion-115	^{236}U	1E-07	2.41E-07		J	1E-07	1.77E-07		J	1E-07	1.72E-04			8E-08	1.56E-04			9E-08	1.54E-04		
AEA	Fusion-115	**^{238}Pu**	1E-05	7.50E-04	3.00E-05		1E-05	2.20E-03	6.60E-05		3E-02	3.27E-01	3.60E-02	J	3E-02	2.99E-01	2.99E-02		4E-02	2.93E-01	2.93E-02	
ICP-MS	Fusion-115	^{238}U	3E-08	3.79E-07			3E-08	5.95E-07			4E-08	1.28E-03			3E-08	1.20E-03			3E-08	1.24E-03		
AEA	Fusion-115	**^{239,240}Pu**	8E-06	2.62E-04	1.83E-05		9E-06	6.00E-04	3.00E-05		3E-02	2.48E+00	9.92E-02		3E-02	2.41E+00	9.64E-02		3E-02	2.34E+00	9.36E-02	
AEA	Fusion-115	**^{241}Am**	1E-05	6.41E-04	2.56E-05		2E-05	1.85E-03	5.55E-05		4E-02	3.92E+01	7.84E-01		4E-02	3.56E+01	7.12E-01		5E-02	3.78E+01	7.56E-01	
GEA	Fusion-115	**^{241}Am GEA**	1E-02	1E-02		U	2E-02	2E-02		U	2E+01	5.93E+01	6.52E+0	J	9E+00	4.27E+01	3.94E+00		9E+00	3.26E+01	3.91E+00	J
Pu-241	Fusion-115	**^{241}Pu**	1E-03	1.40E-02	1.01E-03		1E-03	3.77E-02	2.56E-03		3E-03	1.20E+01	8.40E-01		2E-03	1.17E+01	8.19E-01		2E-03	1.12E+01	7.84E-01	
AEA	Fusion-115	^{242}Cm	7E-06	7E-06		U	1E-05	1E-05		U	4E-02	4E-02		U	3E-02	3.70E-02	1.22E-02	J	3E-02	4.11E-02	1.36E-02	J
AEA	Fusion-115	^{242}Pu	2E-05	2E-05		U	2E-05	2E-05		U	1E-02	1E-02		U	6E-03	6E-03		U	1E-02	1E-02		U
AEA	Fusion-115	**^{243,244}Cm**	8E-06	4.44E-04	2.22E-05		2E-05	2.58E-03	7.74E-05		4E-02	8.38E-02	2.10E-02	J	3E-02	7.76E-02	1.78E-02	J	3E-02	1.23E-01	2.34E-02	J
Alpha	Fusion-115	Gross alpha	8E-04	1.41E-03	2.82E-04	J	9E-04	6.22E-03	5.60E-04		1E+01	5.34E+01	5.87E+00	J	7E+00	5.35E+01	4.28E+00		1E+01	5.49E+01	4.94E+00	
		Alpha Sum (c)		2.10E-03	4.82E-05			7.23E-03	1.23E-04			4.21E+01	8.00E-01			3.84E+01	7.29E-01			4.06E+01	7.71E-01	
		TRU (d)		2.11E-03	5.39E-05			7.24E-03	1.26E-04			4.21E+01	8.00E-01			3.84E+01	7.29E-01			4.06E+01	7.71E-01	

Table 8.3. (Cont'd)

Method	Prep	Radionuclide	Process Blank (PB-1)				Process Blank (PB-2)				Sample (01-01845)				Duplicate (01-01845D)				Triplicate (01-01845T)			
			MDA/ MDL µCi/g	Result µCi/g	[a] 1-sigma µCi/g	DF	MDA/ MDL µCi/g	Result µCi/g	[a] 1-sigma µCi/g	DF	MDA/ MDL µCi/g	Result µCi/g	[a] 1-sigma µCi/g	DF	MDA/ MDL µCi/g	Result µCi/g	[a] 1-sigma µCi/g	DF	MDA/ MDL µCi/g	Result µCi/g	[a] 1-sigma µCi/g	DF

Bolded radionuclides required for comparison to Contract Specification 8 (Envelope D)

MDA: minimum detectable activity (used with all radiochemical analysis results)

MDL: method detection limit (used with ICP-MS results)

DF: Data quality flag (for definition of flags used see Section 8.1)

(a) 1-sigma: Nominal propagated uncertainty including preparation and counting for radiochemical methods; instrument analysis uncertainty for ICP-MS method.

(b) Calculated using ^{133}Cs results and isotopic mass distribution from Supernatant analysis; see Table 8.2.

(c) Alpha Sum = Summation of AEA results only (^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm).

(d) TRU = Z>92, alpha emitter, half-life >10yr (^{237}Np, ^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm).

(e) Results corrected for laboratory blanks and verification standard recoveries per procedure PNL-ALO-482. PB results from DIW transferred from SAL with the samples.

(f) AMU-151: Mass calibrated using ^{147}Sm; response converted to activity using ^{151}Sm specific activity; No chemical separation of samarium and europium performed and result may be bias high by ^{151}Eu

Nominal decay correction references dates

ICP-MS: ^{129}I (5/2003), all others (6/2002)

Radchem: GEA, AES Pu and Am/Cm, ^{90}Sr, gross alpha (10/2001); ^{3}H (2/2002); ^{14}C (3/2002); ^{241}Pu (6/2002), ^{242}Pu (6/2002)

Table 8.4. AZ-101 As-Received Wet Centrifuged Solids – Analyte Results (µg/g)

			Process Blank (PB-1)			**Process Blank (PB-2)**			**Sample (01-01845)**			**Duplicate (01-01845D)**			**Triplicate (01-01845T)**		
Method	**Prep**	**Analyte**	**MDL µg/g**	**Result µg/g**	**DF**	**MDL µg/g**	**Result (k) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**
ICP-MS	Fusion-115	**^{99}Tc**	0.002	0.21		0.002	0.18		0.002	9.0		0.001	8.4		0.002	8.7	
ICP-MS	Fusion-114	**^{127}I**	0.00008	0.00008	U	0.00008	0.00008	U	0.03	2.78		0.04	2.13		0.04	2.44	
ICP-MS	Fusion-115	**^{133}Cs**	0.003	0.003	U	0.003	0.003	U	0.004	16.8		0.003	16.7		0.003	16.3	
ICP-MS	Fusion-115	^{233}U	0.001	0.006	J	0.001	0.002	J	0.001	0.17		0.001	0.17		0.001	0.16	
ICP-MS	Fusion-115	^{235}U	0.002	0.024		0.002	0.018	J	0.003	34.5		0.002	32.3		0.002	33.1	
ICP-MS	Fusion-115	**^{237}Np** (l)	0.001	0.01		0.001	0.010		0.001	30.0		0.0005	26.6		0.0005	25.3	
ICP-MS	Fusion-115	**^{239}Pu** (l)	0.01	0.39		0.01	0.12		0.01	20.7		0.004	17.6		0.004	16.9	
ICP-MS	Fusion-115	^{240}Pu (l)	0.0003	0.024		0.0003	0.007		0.0002	1.5		0.0001	1.3		0.0001	1.2	
ICP-AES	Acid-129M2	**Ag**	14	14	U				16	59	J	14	49	J	14	58	J
ICP-AES	Fusion-115	**Ag**	55	55	UX	55	55	UX	68	230	JX	48	210	JX	54	330	JX
ICP-AES	Acid-129M2	**Al**	34	34	U				38	138,000		34	134,000		34	137,000	
ICP-AES	Fusion-115	**Al**	130	190	J	133	290	J	160	134,000		120	127,000		130	130,000	
ICP-MS	Fusion-115	**As**	0.1	0.1	U	0.1	0.1	U	0.1	49.3		0.1	45.7		0.1	43.4	
ICP-AES	Acid-129M2	**As** (a)	140	140	U				160	160	U	140	140	U	140	140	U
ICP-AES	Fusion-115	**As** (a)	550	550	U	550	550	U	680	680	U	480	480	U	540	540	U
ICP-AES	Acid-129M2	**B**	29	29	U				32	32	U	28	28	U	29	29	U
ICP-AES	Fusion-115	**B**	110	110	U	111	111	U	140	140	U	96	96	U	110	110	U
ICP-AES	Acid-129M2	**Ba**	5.7	5.7	U				6.4	516		5.7	463		5.7	489	
ICP-AES	Fusion-115	**Ba**	22	25	J	22	22	U	27	452	B	19	462	B	22	437	B
ICP-AES	Acid-129M2	**Be**	5.7	5.7	U				6.4	9.6	J	5.7	8.5	J	5.7	8.9	J
ICP-AES	Fusion-115	**Be**	22	22	U	22	22	U	27	27	U	19	19	U	22	22	U
ICP-AES	Acid-129M2	**Bi**	57	57	U				64	64	U	57	57	U	57	57	U
ICP-AES	Fusion-115	**Bi**	220	220	UX	221	221	UX	270	270	UX	190	190	UX	220	220	UX
IC-Inorg	Leach-103	Br (i)	1.3	1.3	U				130	350		130	360		130	350	
C (HP)	Direct	C as TC (a,c)		NA					83	9,770		104	9,510		98	8,410	
C (Furn)	Direct	C as TC		NA					350	10,500		500	9,000		850	9,500	
C (HP)	Direct	**C as TIC**		NA					47	8,780		59	8,600		56	7,750	
C (Furn)	Direct	**C as TIC** (c)		NA					750	1,100	J	1,300	1,300	U	950	950	U

Table 8.4. Cont'd)

			Process Blank (PB-1)			Process Blank (PB-2)			Sample (01-01845)			Duplicate (01-01845D)			Triplicate (01-01845T)		
Method	**Prep**	**Analyte**	**MDL µg/g**	**Result µg/g**	**DF**	**MDL µg/g**	**Result (k) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**
C (HP)	Direct	**C as TOC**		NA					120	960		150	880		140	630	J
C (Furn)	Direct	**C as TOC**		NA					1,100	9,300		2,000	10,800		1,000	9,600	
ICP-AES	Acid-129M2	**Ca**	140	140	U				160	2,580		140	2,230		140	2,390	
ICP-AES	Fusion-115	**Ca**	550	550	UX	554	554	UX	680	2,300	JX	480	2,500	JX	540	2,200	JX
ICP-AES	Acid-129M2	**Cd**	8.6	8.6	U				9.6	5,040		8.5	4,380		8.6	4,600	
ICP-AES	Fusion-115	**Cd**	33	33	U	33	33	U	41	4,470		29	4,400		32	4,270	
ICP-MS	Fusion-115	**Ce**	0.04	4.07		0.04	5.12		0.04	301		0.03	289		0.04	301	
ICP-AES	Acid-129M2	**Ce** (a)	110	110	U				130	410	J	110	350	J	120	370	J
ICP-AES	Fusion-115	**Ce** (a)	440	440	U	440	440	U	540	540	U	380	500	J	430	550	J
IC-Inorg	Leach-103	**Cl** (i)	1.3	1.3	U				13	50		13	130		13	160	
CN	Distill-287	**CN**	0.0400	0.040	U				0.042	0.891	X	0.039	0.039	UX	0.039	0.69	X
ICP-MS	Fusion-115	**Co**	0.01	11.4		0.01	9.8		0.01	46.7	B	0.01	33.9	B	0.01	34.4	B
ICP-AES	Acid-129M2	**Co** (a)	29	29	U				32	32	U	28	33	J	29	29	U
ICP-AES	Fusion-115	**Co** (a)	110	110	U	110	110	U	140	140	U	96	96	U	110	110	U
ICP-AES	Acid-129M2	**Cr**	11	11	U				13	638		11	1,180		11	583	
ICP-AES	Fusion-115	**Cr**	44	44	U	44	44	U	54	551		38	591		43	688	
ICP-AES	Acid-129M2	**Cu**	14	14	U				16	259		14	224		14	231	
ICP-AES	Fusion-115	**Cu**	55	55	U	55	55	U	68	68	U	48	48	U	54	54	U
ICP-AES	Acid-129M2	Dy (a)	29	29	U				32	32	U	28	28	U	29	29	U
ICP-AES	Fusion-115	Dy (a)	110	110	U	110	110	U	140	140	U	96	96	U	110	110	U
ICP-AES	Acid-129M2	Eu (a)	57	57	U				64	64	U	57	57	U	57	57	U
ICP-AES	Fusion-115	Eu (a)	220	220	U	220	220	U	270	270	U	190	190	U	220	220	U
IC-Inorg	Leach-103	**F** (d,i)	1.3	1.3	U				130	4,490		130	3,850		130	4,000	
ICP-AES	Acid-129M2	**Fe**	14	14	U				16	70,100		14	63,900		14	66,300	
ICP-AES	Fusion-115	**Fe**	55	130	J	55	86	J	68	62,900		48	63,500		54	58,400	
CVAA	Acid-131	**Hg**	0.0008	0.002	J				0.007	2.4		0.0075	3.4		0.0092	3.6	
ICP-AES	Acid-129M2	**K**	1,100	1,100	U				1,300	1,700	J	1,100	1,200	J	1,100	1,200	J
ICP-AES	Acid-129M2	**La**	29	29	U				32	1,900		28	1,700		29	1,780	
ICP-AES	Fusion-115	**La**	110	110	U	111	111	U	140	1,600		96	1,590		110	1,550	
ICP-AES	Acid-129M2	**Li**	17	17	U				19	75	J	17	66	J	17	70	J

Table 8.4. (Cont'd)

			Process Blank (PB-1)			Process Blank (PB-2)			Sample (01-01845)			Duplicate (01-01845D)			Triplicate (01-01845T)		
Method	Prep	Analyte	MDL µg/g	Result µg/g	DF	MDL µg/g	Result [k] µg/g	DF	MDL µg/g	Result [f] µg/g	DF	MDL µg/g	Result [f] µg/g	DF	MDL µg/g	Result [f] µg/g	DF
ICP-AES	Fusion-115	**Li**	66	66	U	66	66	U	82	82	U	57	69	J	65	70	J
ICP-AES	Acid-129M2	**Mg**	57	57	U				64	460	J	57	430	J	57	430	J
ICP-AES	Fusion-115	**Mg**	220	220	U	221	221	U	270	530	J	190	580	J	220	470	J
ICP-AES	Acid-129M2	**Mn**	29	29	U				32	1,560		28	1,480		29	1,400	
ICP-AES	Fusion-115	**Mn**	110	110	U	111	111	U	140	1,500		96	1,570		110	1,380	
ICP-MS	Fusion-115	**Mo**	0.03	9.48		0.03	4.00		0.03	147	B	0.02	146	B	0.03	145	B
ICP-AES	Acid-129M2	**Mo** [a]	29	29	U				32	45	J	28	110	J	29	36	J
ICP-AES	Fusion-115	**Mo** [a]	110	110	U	110	110	U	140	140	U	96	96	U	108	108	U
ICP-AES	Acid-129M2	**Na**	86	86	U				96	72,600		85	65,400		86	72,300	
ICP-AES	Fusion-115	**Na**	330	2,400	J	332	2,400	J	410	65,400		290	73,500		320	75,600	
ICP-AES	Acid-129M2	**Nd**	57	57	U				64	1,340		57	1,200		57	1,250	
ICP-AES	Fusion-115	**Nd**	220	220	U	221	221	U	270	1,300	J	190	1,200	J	220	1,200	J
ICP-AES	Acid-129M2	**Ni**	17	17	U				19	2,750		17	2,980		17	2,570	
IC-Inorg	Leach-103	$\mathbf{NO_2}$ [i]	2.5	2.5	U				260	27,200		260	27,900		260	27,200	
IC-Inorg	Leach-103	$\mathbf{NO_3}$ [i]	2.5	2.5	U				260	22,800		260	23,300		260	22,700	
IC-Inorg	Leach-103	Oxalate [a,e,i]	2.5	2.5	UX				26	2,180	X	26	4,010 [h]	X	26	3,260	X
ICP-AES	Acid-129M2	**P**	57	57	U				64	1,910		57	1,700		57	1,750	
ICP-AES	Fusion-115	**P**	220	220	U	221	221	U	270	410	J	190	290	J	220	340	J
ICP-AES	Acid-129M2	**Pb**	57	57	U				64	540	J	57	450	J	57	480	J
ICP-AES	Fusion-115	**Pb**	220	220	U	221	221	U	270	520	J	190	460	J	220	560	J
ICP-MS	Fusion-116	**Pd**	0.01	0.55		0.01	0.76		0.003	110		0.003	142		0.003	50	
ICP-AES	Acid-129M2	**Pd** [a]	430	430	U				480	530	J	430	450	J	430	500	J
ICP-AES	Fusion-115	**Pd** [a]	1,700	1,700	U	1,700	1,700	U	2,000	2,000	U	1,400	1,400	U	1,600	1,600	U
IC-Inorg	Leach-103	PO_4 [i]	2.5	2.5	U				26	210		26	270		26	280	
ICP-MS	Fusion-115	**Pr**	0.004	0.40		0.004	0.03	J	0.01	273		0.004	259		0.004	259	
ICP-MS	Fusion-116	**Pt**	0.02	0.27		0.02	0.59		0.01	0.6	B	0.01	0.60	B	0.01	0.94	B
ICP-MS	Fusion-115	**Pu sum** [g,l]	0.01	0.41		0.01	0.12		0.01	22.1		0.004	18.9		0.004	18.2	
ICP-MS	Fusion-115	**Rb**	0.02	287		0.02	271		0.03	323	B	0.02	217	B	0.02	247	B
ICP-MS	Fusion-116	**Rh**	0.001	0.044		0.001	0.035		0.001	94.9		0.001	94.7		0.001	82.8	
ICP-AES	Acid-129M2	**Rh** [a]	170	170	U				190	190	U	170	170	U	170	170	U

Table 8.4. (Cont'd)

			Process Blank (PB-1)			**Process Blank (PB-2)**			**Sample (01-01845)**			**Duplicate (01-01845D)**			**Triplicate (01-01845T)**		
Method	**Prep**	**Analyte**	**MDL µg/g**	**Result µg/g**	**DF**	**MDL µg/g**	**Result (k) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**	**MDL µg/g**	**Result (f) µg/g**	**DF**
ICP-AES	Fusion-115	**Rh** (a)	660	660	U	660	660	U	820	820	U	570	570	U	650	650	U
ICP-MS	Fusion-116	**Ru**	0.3	0.3	U	0.3	0.3	U	0.2	1183		0.2	1185		0.2	1070	
ICP-AES	Acid-129M2	**Ru** (a)	630	630	U				700	700	U	620	620	U	630	630	U
ICP-AES	Fusion-115	**Ru** (a)	2,400	2,400	U	2,400	2,400	U	3,000	3,000	U	2,100	2,100	U	2,400	2,400	U
ICP-MS	Fusion-115	**Sb**	0.006	0.18		0.006	0.16		0.007	3.8		0.005	3.2	B	0.005	3.2	B
ICP-AES	Acid-129M2	**Sb** (a)	290	290	U				320	320	U	280	280	U	290	290	U
ICP-AES	Fusion-115	**Sb** (a)	1,100	1,100	U	1,100	1,100	U	1,400	1,400	U	960	960	U	1,100	1,100	U
ICP-MS	Fusion-115	**Se**	1	1	U	1	2	J	1	1	UB	1	1	UB	1	1	UB
ICP-AES	Acid-129M2	**Se** (a)	140	140	U				160	160	U	140	140	U	140	140	U
ICP-AES	Fusion-115	**Se** (a)	550	550	U	550	550	U	680	680	U	480	480	U	540	540	U
ICP-AES	Acid-129M2	**Si** (j)	290	290	UX				320	990	JX	280	820	JX	290	1,100	J
ICP-AES	Fusion-115	**Si**	1,100	1,100	U	1,107	1,107	U	1,400	3,700	J	960	4,200	J	1,100	3,500	J
ICP-AES	Acid-129M2	Sn (a)	860	860	U				960	1,000	J	850	850	U	860	860	U
ICP-AES	Fusion-115	Sn (a)	3,300	3,300	U	3,300	3,300	U	4,100	4,100	U	2,900	2,900	U	3,200	3,200	U
IC-Inorg	Leach-103	SO_4 (i)	2.5	2.5	U				260	26,300		260	22,400		260	23,300	
ICP-AES	Acid-129M2	**Sr**	8.6	8.6	U				9.6	376		8.5	336		8.6	356	
ICP-AES	Fusion-115	**Sr**	33	33	U	33	33	U	41	330	J	29	323		32	310	J
ICP-MS	Fusion-115	**Ta**	0.001	0.39		0.001	0.46		0.001	9.2	B	0.001	0.45	B	0.001	1.31	B
ICP-MS	Fusion-115	**Te**	0.2	0.7	J	0.2	0.4	J	0.3	206		0.2	193		0.2	189	
ICP-AES	Acid-129M2	**Te** (a)	860	860	U				960	960	U	850	850	U	860	860	U
ICP-AES	Fusion-115	**Te** (a)	3,300	3,300	U	3,300	3,300	U	4,100	4,100	U	2,900	2,900	U	3,200	3,200	U
ICP-MS	Fusion-115	**Th**	0.005	0.14		0.005	0.092		0.004	116		0.003	106		0.003	104	
ICP-AES	Acid-129M2	**Th** (a)	570	570	U				640	640	U	570	570	U	570	570	U
ICP-AES	Fusion-115	**Th** (a)	2,200	2,200	U	2,200	2,200	U	2,700	2,700	U	1,900	1,900	U	2,200	2,200	U
ICP-AES	Acid-129M2	**Ti**	14	14	U				16	50	J	14	91	J	14	52	J
ICP-AES	Fusion-115	**Ti**	55	55	U	55	55	U	68	75	J	48	69	J	54	60	J
ICP-MS	Fusion-115	**Tl**	0.002	0.053		0.002	0.016	J	0.002	0.13	B	0.002	0.12	B	0.002	0.22	B
ICP-AES	Acid-129M2	**Tl** (a)	290	290	U				320	320	U	280	280	U	290	290	U
ICP-AES	Fusion-115	**Tl** (a)	1,100	1,100	U	1,100	1,100	U	1,400	1,400	U	960	960	U	1,100	1,100	U
ICP-AES	Acid-129M2	**U**	1,100	1,100	U				1,300	3,800	J	1,100	3,500	J	1,100	3,400	J

Table 8.4. (Cont'd)

			Process Blank (PB-1)			Process Blank (PB-2)			Sample (01-01845)			Duplicate (01-01845D)			Triplicate (01-01845T)		
Method	Prep	Analyte	MDL µg/g	Result µg/g	DF	MDL µg/g	Result (k) µg/g	DF	MDL µg/g	Result (f) µg/g	DF	MDL µg/g	Result (f) µg/g	DF	MDL µg/g	Result (f) µg/g	DF
ICP-AES	Fusion-115	**U**	4,400	4,400	U	4,400	4,400	U	5,400	5,600	J	3,800	4,900	J	4,300	5,400	J
KPA	Fusion-115	**U**	0.20	0.38	J	0.20	2.26		0.20	3130		0.20	3400		0.20	2970	
ICP-MS	Fusion-115	**U sum** (b)	0.1	1.2		0.1	1.8		0.1	3800		0.1	3560		0.1	3680	
ICP-MS	Fusion-115	**V**	0.03	6.97		0.03	6.17		0.03	24.0	B	0.02	23.9	B	0.03	22.2	B
ICP-AES	Acid-129M2	**V** (a)	29	29	U				32	32	U	28	28	U	29	29	U
ICP-AES	Fusion-115	**V** (a)	110	110	U	110	110	U	140	140	U	96	96	U	110	110	U
ICP-MS	Fusion-115	**W**	0.01	1.53		0.01	1.12		0.02	38.3		0.01	32.2		0.01	29.4	B
ICP-AES	Acid-129M2	**W** (a)	1,100	1,100	U				1,300	1,300	U	1,100	1,100	U	1,100	1,100	U
ICP-AES	Fusion-115	**W** (a)	4,400	4,400	U	4,400	4,400	U	5,400	5,400	U	3,800	3,800	U	4,300	4,300	U
ICP-MS	Fusion-115	**Y**	0.01	0.39		0.01	0.02	J	0.01	118		0.01	115		0.01	117	
ICP-AES	Acid-129M2	**Y** (a)	29	29	U				32	120	J	28	110	J	29	110	J
ICP-AES	Fusion-115	**Y** (a)	110	110	U	110	110	U	140	140	U	96	100	J	110	110	U
ICP-AES	Acid-129M2	**Zn**	29	29	U				32	81	J	28	73	J	29	83	J
ICP-AES	Fusion-115	**Zn**	110	110	UX	111	111	UX	140	140	JX	96	110	JX	110	130	JX
ICP-AES	Acid-129M2	**Zr**	29	29	U				32	20,600		28	18,300		29	19,500	
ICP-AES	Fusion-115	**Zr**	110	110	U	111	111	U	140	12,600		96	14,100		110	13,400	

Bolded analytes required for comparison to Contract Specification 8 (Envelope D)

MDL: method detection limit (with all processing factors applied)

DF: data quality flag (for definition of flags used see Section 8.1)

NA: not applicable

Prep: See Section 7.0 for preparation information for PNL-ALO-114, -115, -116 fusion and PNL-ALO-129 Mod 2 acid digestion

(a) Opportunistic analytes; not included in test specification or test plan.

(b) Total uranium (U Sum) based on the sum of all U isotopes measured by ICP-MS.

(c) TC is sum of TIC and TOC by HP; TIC is difference between measured TC Furn and TOC Furn.

(d) The fluoride results should be considered the upper bound concentration for the fluoride, since the fluoride peak shape and retention time suggests the presence of co-eluting anion(s).

(e) Oxalate not recovered in the hot cell BS/LCS, apparently due to precipitation (see Section 9.5).

(f) Typical analysis precision/accuracy better that ±15% for results >10xMDL (i.e., results without a U or J flag).

(g) Total plutonium (Pu Sum) based on sum of Pu isotopes measured by ICP-MS.

(h) Result calculated from response approximately 5% above the highest calibration standard.

(i) MDL is based on the lowest calibration standard adjusted for sample dilution; equivalent to SW-846 EQL definition.

Table 8.4. (Cont'd)

			Process Blank (PB-1)			Process Blank (PB-2)			Sample (01-01845)			Duplicate (01-01845D)			Triplicate (01-01845T)		
Method	Prep	Analyte	MDL µg/g	Result µg/g	DF	MDL µg/g	Result [(k)] µg/g	DF	MDL µg/g	Result [(f)] µg/g	DF	MDL µg/g	Result [(f)] µg/g	DF	MDL µg/g	Result [(f)] µg/g	DF

(j) Most silicon from PNL-ALO-129 Mod 2 lost in processing; see Section 7.2.4.

(k) Two PBs prepared only for fusion preparations.

(l) Same certified source standard used to prepare calibration and verification standards for ICP-MS. Calibration and verification standards prepared approximately 1 year apart; prepared standards verified by independent analysis (i.e., LSC, AEA, or GEA).

8.6 Undissolved Solids Results

Table 8.5 and Table 5.6 present the calculated undissolved solids results derived from subtracting the contribution of the interstitial liquid from the wet centrifuged solids results. When the concentration of the analyte in the wet centrifuged solids is above the MDL, the density and wt% solids data from Table 6.2 have been used to calculate the concentration of each analyte in the undissolved solids per Equation 8.1.

$$X = C - \left(\left(\frac{S}{D} \right) * \left(\frac{1-W}{1-T} \right) \right) \tag{8.1}$$

where: X = undissolved solids concentration (µg/g or µCi/g) on a per g of wet centrifuged solids (i.e., concentration on a wet-weight basis)

C = average measured concentration of analyte in wet centrifuged solids (µg/g or µCi/g)

S = average measured concentration of analyte in supernatant (µg/mL or µCi/L)

D = density of supernatant (1.223 g/mL, see Table 6.2)

W = fractional solids weight in wet centrifuged solids after drying at 105°C (0.702, see Table 6.2 and Section 6.0).

T = fractional solids weight in supernatant after drying at 105°C (0.267, see Table 6.2 and Section 6.0).

Also reported in Table 8.5 and Table 5.6 are the analyte concentrations calculated on a dry-weight basis. This is calculated by subtracting the supernatant from the wet centrifuged solids fraction according to Equation 8.2.

$$Y = \frac{X}{F}, \text{ where } F = 1 - \left(\frac{1-W}{1-T} \right) \tag{8.2}$$

where: Y = undissolved solids concentration (µg/g or µCi/g) on a per gram of undissolved solids (i.e., concentration on a dry-weight basis).

X = undissolved solids concentration (µg/g or µCi/g) on a per g of wet centrifuged solids (See Equation 8.1)

F = undissolved solids weight fraction (g undissolved solids per g of wet centrifuged solids).

The following apply to the both undissolved solids calculations:

a) If the analyte is not measured on the supernatant, then the undissolved solids concentration '*X*' is set equal to the wet centrifuged solids concentration '*C*' (i.e., assumes no analyte contribution from the supernatant).
b) If the analyte is measured on the supernatant but not detected above the MDL, then the average concentration in the supernatant '*S*' is set to zero (0).
c) If the analyte is measured on the wet centrifuged solids but is not detected, the undissolved solids concentration '*X*' is set to the wet centrifuged solids MDL, even if the analyte is measured in the supernatant.
d) If the calculated undissolved solids concentration is less than or equal to zero, the analyte is assumed to come only from the supernatant within uncertainty of the

analysis method. The undissolved solids concentration ‘*X*’ is set to the wet centrifuged solids MDL.

e) The wet centrifuged solids average concentration ‘*C*’ and the supernatant average concentration ‘*S*’ are based only on results >MDL.

Table 8.5. AZ-101 Undissolved Solids Radionuclide Concentration (µCi/g)

	WCS				Supernatant (a)			UDS (b)	
Radionuclide	**Measurement Method**	**MDL/MDA µCi/g**	**Average µCi/g**	**(e) DF**	**Method**	**Average µCi/g**	**(e) DF**	**Average µCi/g (Wet)**	**Average µCi/g (Dry)**
^{3}H	H-3	4E-04	7.08E-03		H-3	1.81E-02		1.06E-03	1.79E-03
^{14}C	C-14	2E-04	1.04E-03	B	C-14	1.93E-03		4.04E-04	6.80E-04
^{60}Co	GEA	3E-01	2.03E+00		GEA	0	U	2.03E+00	3.42E+00
^{90}Sr	Sr-90	4E+02	2.02E+04		Sr-90	5.01E-02 (c)		2.02E+04	3.40E+04
^{99}Tc (d)	ICP-MS	3E-05	1.48E-01		ICP-MS	3.27E-01		3.91E-02	6.59E-02
^{125}Sb	GEA	5E+00	8.07E+00	J	GEA	0	U	8.07E+00	1.36E+01
^{126}Sn	GEA	2E+00	2E+00	U		NM		< 2E+00	< 4E+00
^{129}I	ICP-MS	8E-06	1.09E-05	J	ICP-MS	1.06E-06	J	1.05E-05	1.78E-05
^{137}Cs	GEA	2E+00	7.52E+02		ICP-MS	1.64E+03		2.06E+02	3.47E+02
^{137}Cs	ICP-MS (Calc)	8E-02	8.76E+02		GEA	1.40E+03		4.11E+02	6.92E+02
^{152}Eu	GEA	1E+00	1E+00	U		NM		< 1E+00	< 2E+00
^{154}Eu	GEA	9E-01	2.34E+01		GEA	0	U	2.34E+01	3.95E+01
^{155}Eu	GEA	7E+00	3.06E+01		GEA	0	U	3.06E+01	5.15E+01
^{233}U	ICP-MS	9E-06	1.61E-03		ICP-MS	0	U	1.61E-03	2.71E-03
^{235}U	ICP-MS	5E-09	7.32E-05		ICP-MS	4.73E-08	B	7.32E-05	1.23E-04
^{237}Np	ICP-MS	4E-07	1.94E-02		ICP-MS	3.77E-05	J	1.94E-02	3.26E-02
^{238}Pu	AEA	3E-02	3.06E-01		AEA	3.44E-04	B	3.06E-01	5.16E-01
^{239,240}Pu	AEA	3E-02	2.41E+00		AEA	1.81E-03		2.41E+00	4.06E+00
^{239}Pu	ICP-MS	3E-04	1.14E+00		ICP-MS	2.00E-03	JB	1.14E+00	1.92E+00
^{241}Pu	Pu-241	2E-03	1.15E+01			NM		1.15E+01	1.93E+01
^{241}Am	AEA	4E-02	3.75E+01		AEA	1.05E-04	B	3.75E+01	6.32E+01
^{241}Am (GEA)	GEA	1E+01	4.49E+01		GEA	0	U	4.49E+01	7.56E+01
^{242}Pu	AEA	9E-03	9E-03	U		NM		< 9E-03	< 2E-02
^{242}Cm	AEA	3E-02	3.91E-02	J	AEA	0	U	3.91E-02	6.59E-02
^{243,244}Cm	AEA	3E-02	9.48E-02	J	AEA	4.18E-05	JB	9.48E-02	1.60E-01
Alpha	Gross Alpha	9E+00	5.39E+01		Gross Alpha	0	U	5.30E+01	9.08E+01

WCS = wet centrifuged solids; UDS = undissolved solids; NM = not measured

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured (NM), the supernatant is assumed to have no contribution to the wet centrifuged solids results; the average field indicates ‘NM’ and the supernatant concentration is set to 0 (zero) when calculating the undissolved solids concentration.

(b) If analyte is measured in the wet centrifuged solids, but is not above the MDL, the undissolved solids results is set to < MDL of the wet centrifuged solids. When the calculated undissolved solids results is <0 or =0, the undissolved solids results is set to <MDL of the wet centrifuged solids.

(c) Supernatant ^{90}Sr result is significantly lower than other published results; see discussion Section 8.3 and Section 9.4.4. However, the WCS ^{90}Sr concentration is so high that the low supernatant ^{90}Sr concentration has no impact on the UDS concentration.

(d) Uncertainty estimated at ±30%; see Section 8.3 for further details.

(e) data flag: B = analyte measured in blank above EQL; T = estimated value, U = not detected above the reported MDL.

Table 8.6. AZ-101 Undissolved Solids Analyte Concentration (μg/g)

	WCS				Supernatant (a)			UDS (b)	
Analyte	Measurement Method	MDL μg/g	Average μg/g	(e) DF	Measurement Method	Average μg/g	(e) DF	Average μg/g (Wet)	Average μg/g (Dry)
Ag	ICP-AES	2E+01	5.50E+01	J	ICP-AES	0	U	5.50E+01	9.27E+01
Al	ICP-AES	4E+01	1.36E+05		ICP-AES	6.05E+03		1.34E+05	2.26E+05
Ammonia (d)	none		NM		ISE	2.30E+00	J	NM	NM
As	ICP-MS	1E-01	4.61E+01		ICP-AES	9.90E+00	J	4.28E+01	7.21E+01
B	ICP-AES	3E+01	3E+01	U	ICP-AES	7.74E+01	BX	< 3E+00	<5E+00
Ba	ICP-AES	6E+00	4.89E+02		ICP-AES	2.50E-01	J	4.89E+02	8.24E+02
Be	ICP-AES	6E+00	9.00E+00	J	ICP-AES	0	U	9.00E+00	1.52E+01
Bi	ICP-AES	6E+01	6E+01	U	ICP-AES	0	U	< 6E+01	< 1E+02
C as TIC	C (Furn)	1E+03	1.10E+03	J	C (HP)	5.83E+03		< 1E+03	< 2E+03
C as TIC	C (HP)	5E+01	8.38E+03		C (Furn)	9.85E+03		5.11E+03	8.30E+03
C as TOC	C (Furn)	1E+03	9.90E+03		C (HP)	1.50E+03		9.40E+03	1.58E+04
C as TOC	C (HP)	1E+02	8.20E+02		C (Furn)	5.10E+02		6.50E+02	1.10E+03
Ca	ICP-AES	2E+02	2.40E+03		ICP-AES	9.30E+00	J	2.40E+03	4.04E+03
Cd	ICP-AES	9E+00	4.67E+03		ICP-AES	7.70E-01	JB	4.67E+03	7.87E+03
Ce	ICP-MS	4E-02	2.97E+02		ICP-AES	0	U	2.97E+02	5.00E+02
Cl	IC-Inorg	1E+01	1.10E+02		IC-Inorg	2.40E+02		3.02E+01	5.09E+01
CN	CN	4E-02	5.41E-01	X	CN	2.04E+00		< 4E-01	< 7E-01
Co	ICP-MS	6E-03	3.83E+01	B	ICP-AES	0	U	3.83E+01	6.45E+01
Cr	ICP-AES	1E+01	8.00E+02		ICP-AES	6.86E+02		5.72E+02	9.64E+02
Cs (c)	ICP-MS	3E-03	3.20E+01		ICP-MS	5.18E+01		1.48E+01	2.92E+01
Cu	ICP-AES	2E+01	2.38E+02		ICP-AES	0	U	2.38E+02	4.01E+02
F	IC-Inorg	1E+02	4.11E+03		IC-Inorg	2.01E+03		3.44E+03	5.80E+03
Fe	ICP-AES	2E+01	6.68E+04		ICP-AES	1.50E+00	JB	6.68E+04	1.13E+05
Hg	CVAA	8E-03	3.15E+00		CVAA	5.53E-02		3.13E+00	5.28E+00
K	ICP-AES	1E+03	1.40E+03	J	ICP-AES	4.46E+03		< 1E+03	< 2E+03
La	ICP-AES	3E+01	1.79E+03		ICP-AES	0	U	1.79E+03	3.02E+03
Li	ICP-AES	2E+01	7.00E+01	J	ICP-AES	8.90E-01	J	6.97E+01	1.17E+02
Mg	ICP-AES	6E+01	4.40E+02	J	ICP-AES	0	U	4.40E+02	7.41E+02
Mn	ICP-AES	3E+01	1.48E+03		ICP-AES	0	U	1.48E+03	2.49E+03
Mo	ICP-MS	3E-02	1.46E+02	B	ICP-AES	8.88E+01		1.16E+02	1.96E+02
Na	ICP-AES	3E+02	7.15E+04		ICP-AES	1.11E+05		3.46E+04	5.83E+04
Nd	ICP-AES	6E+01	1.26E+03		ICP-AES	0	U	1.26E+03	2.12E+03
Ni	ICP-AES	2E+01	2.77E+03		ICP-AES	1.0	JB	2.77E+03	4.67E+03
NO_2	IC-Inorg	3E+02	2.74E+04		IC-Inorg	6.13E+04		7.02E+03	1.18E+04
NO_3	IC-Inorg	3E+02	2.29E+04		IC-Inorg	5.26E+04		5.41E+03	9.12E+03
P	ICP-AES	6E+01	1.79E+03		ICP-AES	5.04E+02		1.62E+03	2.73E+03
Pb	ICP-AES	6E+01	4.90E+02	J	ICP-AES	3.30E+00	J	4.89E+02	8.24E+02
Pd	ICP-MS	3E-03	1.01E+02		ICP-AES	2.00E+01	J	9.44E+01	1.59E+02
Pr	ICP-MS	4E-03	2.64E+02			NM		2.64E+02	4.45E+02
Pu sum	ICP-MS	4E-03	1.97E+01			NM		1.97E+01	3.32E+01
Rb	ICP-MS	2E-02	2.62E+02	B	ICP-MS	9.20E+00		2.59E+02	4.36E+02
Rh	ICP-MS	6E-04	9.08E+01		ICP-AES	0	U	9.08E+01	1.53E+02

Table 8.6. (Cont'd)

	WCS				Supernatant (a)			UDS (b)	
Analyte	**Measurement Method**	**MDL µg/g**	**Average µg/g**	**(e) DF**	**Measurement Method**	**Average µg/g**	**(e) DF**	**Average µg/g (Wet)**	**Average µg/g (Dry)**
Ru	ICP-MS	2E-01	1.15E+03		ICP-AES	0	U	1.15E+03	1.94E+03
Sb	ICP-MS	6E-03	3.42E+00	B	ICP-AES	0	U	3.42E+00	5.76E+00
Se	ICP-MS	1E+00	1E+00	UB	ICP-AES	0	U	< 1E+00	< 2E+00
Si	ICP-AES	1E+03	3.80E+03	J	ICP-AES	206	BX	3.73E+03	6.29E+03
SO_4	IC-Inorg	3E+02	2.40E+04		IC-Inorg	1.62E+04		1.86E+04	3.14E+04
Sr	ICP-AES	9E+00	3.56E+02		ICP-AES	0	U	3.56E+02	6.00E+02
Ta	ICP-MS	7E-04	3.65E+00	B		NM		3.65E+00	6.15E+00
Te	ICP-MS	2E-01	1.96E+02		ICP-AES	0	U	1.96E+02	3.30E+02
Th	ICP-MS	3E-03	1.09E+02		ICP-AES	0	U	1.09E+02	1.84E+02
Ti	ICP-AES	2E+01	6.40E+01	J	ICP-AES	0	U	6.40E+01	1.08E+02
Tl	ICP-MS	2E-03	1.55E-01	B	ICP-AES	0	U	1.55E-01	2.61E-01
U	ICP-AES	1E+03	3.60E+03	J	ICP-AES	0	U	3.60E+03	6.07E+03
U	KPA	2E-01	3.17E+03		KPA	5.15E-01		3.17E+03	5.34E+03
U sum	ICP-MS	1E-01	3.68E+03		ICP-MS	2.81E+00	B	3.68E+03	6.20E+03
V	ICP-MS	3E-02	2.34E+01	B	ICP-AES	1.50E+00	J	2.29E+01	3.86E+01
W	ICP-MS	1E-02	3.33E+01	B	ICP-AES	5.80E+01	J	1.40E+01	2.36E+01
Y	ICP-MS	9E-03	1.17E+02		ICP-AES	0	U	1.17E+02	1.97E+02
Zn	ICP-AES	3E+01	7.90E+01	J	ICP-AES	0	U	7.90E+01	1.33E+02
Zr	ICP-AES	3E+01	1.95E+04		ICP-AES	0	U	1.95E+04	3.29E+04

WCS = wet centrifuged solids; UDS = undissolved solids; NM = not measured

C as TIC and TOC: HP = hot persulfate oxidation method; Furn = Furnace oxidation method

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured (NM), the supernatant is assumed to have no contribution to the wet centrifuged solids results; the average field indicates 'NM' and the supernatant concentration is set to 0 (zero) when calculating the undissolved solids concentration.

(b) If the analyte is measured in the wet centrifuged solids, but is not above the MDL, the undissolved solids results is set to < MDL of the wet centrifuged solids. When the calculated undissolved solids results is <0 or =0, the undissolved solids results is set to <MDL of the wet centrifuged solids.

(c) Total Cs: Cesium on wet centrifuged solids calculated from sum of ^{133}Cs measured by ICP-MS and the calculated $^{135/137}$Cs using the Cs isotope mass fractions from the supernatant results.

(d) Ammonia analysis of wet centrifuged solids not specified by test specification or test plan.

(e) data flag: B = analyte measured in blank above EQL; T = estimated value, U = not detected above the reported MDL; X = QC deficiency.

8.7 Comparison of Supernatant Results to Specification 7 Limits

Specification 7 for Envelope B defines limits for several analytes relative to sodium concentration (moles analyte per mole Na or Bq analyte per mole Na). Table 8.7 presents the Specification 7 ratio limits and compares them to the AZ-101 as-measured ratios. For analytes measured above the MDL or MDA, the mole or Bq analyte to mole Na ratio does not exceed the limits defined in the specification. However, two analytes (^{60}Co and ^{154}Eu) had MDAs that exceeded the threshold necessary to evaluate whether or not Specification 7 limits were met. The ^{60}Co and ^{154}Eu were measured by GEA with an extended counting time of 14 hours. However, due the very high ^{137}Cs activity, the MDA for ^{60}Co and ^{154}Eu are significantly higher than normal. For the sodium molarity measured, the Bq to mole of sodium ratio based on the ^{60}Co and ^{154}Eu MDAs are 5.6 and 1.3 times the Specification 7 Envelop B limit, respectively.

Table 8.7. AZ-101 As-Received Supernatant Compared to Specification 7

Method	Prep	Analyte	MRQ µg/mL	MDL(a) µg/mL	Average µg/mL	(g) DF	Average Moles (M)	Average M/M Na	'B Spec 7' M/M Na	% of Limit	Meet Spec 7
ICP-AES	Acid-128	Al	75	1.5	6,050		2.24E-01	4.64E-02	2.5E-01	19	Yes
ICP-AES	Acid-128	Ba	2.3	0.3	0.3	J	1.82E-06	3.77E-07	1.0E-04	0.4	Yes
ICP-AES	Acid-128	Ca	150	6.4	9.3	J	2.32E-04	4.81E-05	4.0E-02	0.1	Yes
ICP-AES	Acid-128	Cd	2.3	0.4	0.8	JB	6.88E-06	1.42E-06	4.0E-03	0.04	Yes
IC-Inorg	Direct	Cl	300	125	243		6.86E-03	1.42E-03	8.9E-02	2	Yes
ICP-AES	Acid-128	Cr	15	0.5	686		1.32E-02	2.73E-03	2.0E-02	14	Yes
IC-Inorg	Direct	F	150	125	2,010		1.06E-01	2.19E-02	2.0E-01	11	Yes
ICP-AES	Acid-128	Fe	150	0.6	1.5	JB	2.69E-05	5.56E-06	1.0E-02	0.1	Yes
CVAA	Acid-131	Hg	1.5	0.00013	0.055		2.76E-07	5.71E-08	1.4E-05	0.4	Yes
ICP-AES	Acid-128	K	75	52	4,460		1.14E-01	2.36E-02	1.8E-01	13	Yes
ICP-AES	Acid-128	La	35	1.3	1.3	U	<9.27E-06	<1.92E-06	8.3E-05	< 2	Yes
ICP-AES	Acid-128	*Na*	*75*	*19*	*111,000*		*4.83E+00*	*1.00E+00*			
ICP-AES	Acid-128	Ni	30	0.8	1.0	JB	1.71E-05	3.54E-06	3.0E-03	0.1	Yes
IC-Inorg	Direct	NO_2	3,000	2,500	61,300		1.33E+00	2.76E-01	3.8E-01	73	Yes
IC-Inorg	Direct	NO_3	3,000	2,500	52,600		8.48E-01	1.76E-01	8.0E-01	22	Yes
ICP-AES	Acid-128	Pb	300	2.6	3.3	J	1.61E-05	3.33E-06	6.8E-04	0.5	Yes
ICP-AES(b)	Acid-128	P as PO_4	7,700	7.9	1,540		1.62E-02	3.36E-03	1.3E-01	3	Yes
IC-Inorg	Direct	PO_4	7,700	250	1,630		1.68E-02	3.49E-03	1.3E-01	3	Yes
IC-Inorg	Direct	SO_4	7,500	250	16,200		1.69E-01	3.49E-02	7.0E-02	50	Yes
C (HP)	Direct	C as TIC	150	34	9,850		8.21E-01	1.70E-01	3.0E-01	57	Yes
C (Furn)	Direct	C as TIC	150	170	5,830		4.86E-01	1.01E-01	3.0E-01	34	Yes
C (HP)	Direct	C as TOC	1,500	87	510		4.25E-02	8.80E-03	5.0E-01	2	Yes
C (Furn)	Direct	C as TOC	1,500	250	1,500		1.26E-01	2.59E-02	5.0E-01	5	Yes
ICP-AES	Acid-128	U	600	52	52	U	<2.16E-04	<4.48E-05	1.2E-03	< 4	Yes
KPA	Acid-128	U	780	0.0004	0.52		2.17E-06	4.48E-07	1.2E-03	0.04	Yes
Method	**Prep**	**Radio-nuclide**	**MRQ µCi/mL**	**MDA µCi/mL**	**Average µCi/mL(c)**	**(g) DF**	**Average Bq/mL(c)**	**Average Bq/M Na**	**'B Spec 7' Bq/M Na**	**% of Limit**	**Meet Spec 7**
ICP-MS	Acid-128	^{99}Tc	1.5E-03	2E-04	3.27E-01		1.21E+04	2.50E+06	7.1E+06	35	Yes
Sr-90	Acid-128	^{90}Sr	1.5E-01	1E-03	5.01E-02		1.85E+03	3.84E+05	4.4E+07	1 (f)	Yes
GEA	Acid-128	^{60}Co	2.1E-03	4E-02	4E-02	U	<1.66E+03	**<3.44E+05**	6.1E+04	**< 560** (d)	No?
GEA	Acid-128	^{137}Cs	9.0E+00	7E-01	1.64E+03		6.08E+07	1.26E+10	2.0E+10	63	Yes
GEA	Acid-128	^{154}Eu	2.0E-03	2E-01	2E-01	U	<7.54E+03	**<1.56E+06**	1.2E+06	**< 130** (d)	No?
		TRU (e)			2.32E-03		8.58E+01	1.78E+04	4.8E+05	4	Yes

Outlined and bolded results may exceed Specification 7 (Envelope B) criteria.

MDL: method detection limit

MDA: minimum detectable activity

No?: Absolute evaluation can not be made, since analyte was not detected above the analysis MDL or MDA.

(a) Value represents EQL for F, Cl, NO_2, NO_3, PO_4, and SO_4, since anions only reported above the EQL.

(b) Phosphate based on ICP-AES average total phosphorus result of 504 µg/mL.

(c) Nominal decay correction reference dates: ^{99}Tc (June 2002), ^{90}Sr (9/2002), and GEA radionuclides (10/2001).

(d) ^{60}Co and ^{154}Eu not detected from 14 hour extended time GEA, and due to high activity from ^{137}Cs the MDAs exceed the Specification limit by 5.6 times and 1.3 times, respectively.

(e) Z>92, alpha emitter, half-life >10yr (^{237}Np, ^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm); Pu, Am, and Cm from AES and Np from ICP-MS.

(f) Supernatant ^{90}Sr result is significantly lower than other published results; see discussion Sections 8.3 and 9.4.4. At a concentration of 1.19 µCi/mL (CUF result), the Bq/M Na = 9.12E+06 (21% of limit).

(g) data flag: B = analyte measured in blank above EQL; T = estimated value, U = not detected above the reported MDL.

8.8 Comparison of Undissolved Solids Results to Specification 8

Specification 8 for Envelope D defines limits for several analyte concentrations and radionuclide activities per 100 g equivalent non-volatile waste oxides (e.g., sodium oxide and silicon oxide). The analyte mass (g) per 100 g of waste oxide is calculated according to Equation 8.3 when starting from wet centrifuged solids (i.e., wet-weight basis assuming only water is the supernatant), and according to Equation 8.4 when starting from an undissolved dry solid (i.e., dry-weight basis). The radionuclide activity (Ci) per 100 g of waste oxide is calculated in a similar manner.

$$Z = X * F_1 * \left(\frac{1-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * (1-W)\right)} \right) * F_2 \tag{8.3}$$

$$Z = Y * F_1 * \left(\frac{W-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * (1-W)\right)} \right) * F_2 \tag{8.4}$$

where: Z = analyte concentration per mass of oxide (g/100g)
X = undissolved solids analyte concentration in µg/g (wet-weight basis, see Section 8.6)
Y = undissolved solids analyte concentration in µg/g (dry-weight basis, see Section 8.6)
F_1 = mass conversion factor (g/10^6 µg)
W_{ocs} = fractional oxide mass of the wet centrifuged solids (0.526, see Table 6.2)
W_{os} = fraction oxide mass of the supernatant (0.158, see Table 6.2)
W = fractional dried mass of solids in the wet centrifuged solids (e.g., 0.702, see Table 6.2 and Section 6.0)
T = fractional dried mass of solids in supernatant (0.267, see Table 6.2)
F_2 = 100-g oxide mass conversion factor, 100.

The calculated results are based on the unwashed solids (i.e., as received from tank AZ-101) analysis results. The HLW solids feed for vitrification will be washed, removing soluble solids present in the AZ-101 sludge. The calculated 'Ci radionuclide per 100 g waste oxide' results are shown in Table 8.8 and the 'g analyte per 100 g waste oxide' results are presented in Table 8.9. One radionuclide (^{126}Sn) could not be measured at a low enough detection level to be able to determine whether or not the radionuclide met the specification. All other radionuclides met the Table TS-8.3 criteria. Of the analytes measured, aluminum and sulfur are significantly above the Table TS-8.4 criteria. The sulfur comparison to the specification is made from the SO_4 results from DIW leaching of the solids and the reported result is most likely lower than the actual SO_4 concentration.

Table 8.8. AZ-101 Undissolved Solids – Ci Radionuclide / 100 g Waste Oxide

			UDS (b) Average		(d)	Spec 8 Evaluation			
						Ci/100 g waste oxide			
Radionuclide	**Measurement Method**	**MDL/MDA µCi/g (a)**	**µCi/g (Wet)**	**µCi/g (Dry)**	**DF**	**UDS**	**Spec Limit**	**% of Limit**	**Meets Spec**
			Specification 8 Table TS-8.3 Radionuclides (Isotopes)						
^{3}H	H-3	4E-04	1.06E-03	1.79E-03		2.30E-07	6.5E-05	0.4	Yes
^{14}C	C-14	2E-04	4.04E-04	6.80E-04	B	8.74E-08	6.5E-06	1	Yes
^{60}Co	GEA	3E-01	2.03E+00	3.42E+00		4.40E-04	1.0E-02	4	Yes
^{90}Sr	Sr-90	4E+02	2.02E+04	3.40E+04		4.37E+00	1.0E+01	44	Yes
^{99}Tc (c)	ICP-MS	3E-05	3.91E-02	6.59E-02		8.47E-06	1.5E-02	0.1	Yes
^{125}Sb	GEA	5E+00	8.07E+00	1.36E+01	J	1.75E-03	3.2E-02	5	Yes
^{126}Sn	GEA	2E+00	< 2E+00	< 4E+00	U	**< 5E-04**	1.5E-04	**< 340**	No?
^{129}I	ICP-MS	8E-06	1.05E-05	1.78E-05	J	2.27E-09	2.9E-07	0.8	Yes
^{137}Cs	GEA	2E+00	2.06E+02	3.47E+02		4.47E-02	1.5E+00	3.0	Yes
^{152}Eu	GEA	1E+00	< 1E+00	< 2E+00	U	< 3E-04	4.8E-04	< 57	Yes
^{154}Eu	GEA	9E-01	2.34E+01	3.95E+01		5.07E-03	5.2E-02	10	Yes
^{155}Eu	GEA	7E+00	3.06E+01	5.15E+01		6.62E-03	2.9E-02	23	Yes
^{233}U	ICP-MS	9E-06	1.61E-03	2.71E-03		3.48E-07	9.0E-07	39	Yes
^{235}U	ICP-MS	5E-09	7.32E-05	1.23E-04		1.59E-08	2.5E-07	6	Yes
^{237}Np	ICP-MS	4E-07	1.94E-02	3.26E-02		4.19E-06	7.4E-05	6	Yes
^{238}Pu	AEA	3E-02	3.06E-01	5.16E-01		6.63E-05	3.5E-04	19	Yes
^{239,240}Pu	AEA	3E-02	2.41E+00	4.06E+00		5.22E-04	3.1E-03	17	Yes
^{241}Pu	Pu-241	2E-03	1.15E+01	1.93E+01		2.48E-03	2.2E-02	11	Yes
^{241}Am	AEA	4E-02	3.75E+01	6.32E+01		8.13E-03	9.0E-02	9	Yes
^{243,244}Cm	AEA	3E-02	9.48E-02	1.60E-01	J	2.05E-05	3.0E-03	0.7	Yes

UDS = undissolved solids

Outlined and bolded results may exceed Specification 8 (Envelope D).

No?: Absolute evaluation can not be made, since analyte was not detected above the analysis MDA.

(a) Method detection limit (MDL) is presented for ICP-MS and minimum detectable activity (MDA) is presented for all radiochemical analyses.

(b) For decay correction reference dates, see Table 8.1 and Table 8.3.

(c) Uncertainty estimated at ±30%; see Section 8.3 for further details.

(d) data flag: B = analyte measured in blank above EQL; T = estimated value, U = not detected above the reported MDL.

Table 8.9. AZ-101 Undissolved Solids – g Analyte / 100 g Waste Oxide

Analyte	Measurement Method	MDL µg/g	UDS Average µg/g Wet	UDS Average µg/g Dry	[d] DF	Spec 8 Evaluation g/100 g waste oxide UDS	Spec 8 Evaluation g/100 g waste oxide Spec Limit	Spec 8 Evaluation % of Limit	Spec 8 Evaluation Meets Spec
Specification 8 Table TS-8.1 Analytes									
As	ICP-MS	1E-01	4.28E+01	7.21E+01		9.27E-03	1.6E-01	6	Yes
B	ICP-AES	3E+01	< 3E+01	< 5E+01	U	< 6E-03	1.3E+00	< 0.5	Yes
Be	ICP-AES	6E+00	9.00E+00	1.52E+01	J	1.95E-03	6.5E-02	3	Yes
Ce	ICP-MS	4E-02	2.97E+02	5.00E+02		6.43E-02	8.1E-01	8	Yes
Co	ICP-MS	6E-03	3.83E+01	6.45E+01	B	8.29E-03	4.5E-01	2	Yes
Cs [a]	ICP-MS	3E-03	1.48E+01	2.49E+01		3.20E-03	5.8E-01	0.6	Yes
Cu	ICP-AES	2E+01	2.38E+02	4.01E+02		5.15E-02	4.8E-01	11	Yes
Hg	CVAA	8E-03	3.13E+00	5.28E+00		6.78E-04	1.0E-01	1	Yes
La	ICP-AES	3E+01	1.79E+03	3.02E+03		3.88E-01	2.6E+00	15	Yes
Li	ICP-AES	2E+01	6.97E+01	1.17E+02	J	1.51E-02	1.4E-01	11	Yes
Mn	ICP-AES	3E+01	1.48E+03	2.49E+03		3.21E-01	6.5E+00	5	Yes
Mo	ICP-MS	3E-02	1.16E+02	1.96E+02	B	2.52E-02	6.5E-01	4	Yes
Nd	ICP-AES	6E+01	1.26E+03	2.12E+03		2.73E-01	1.7E+00	16	Yes
Pr	ICP-MS	4E-03	2.64E+02	4.45E+02		5.72E-02	3.5E-01	16	Yes
Pu sum	ICP-MS	4E-03	1.97E+01	3.32E+01		4.27E-03	5.4E-02	8	Yes
Rb	ICP-MS	2E-02	2.59E+02	4.36E+02	B	5.61E-02	1.9E-01	30	Yes
Sb	ICP-MS	6E-03	3.42E+00	5.76E+00	B	7.41E-04	8.4E-01	0.1	Yes
Se	ICP-MS	1E+00	< 1E+00	< 2E+00	UB	< 3E-04	5.2E-01	< 0.05	Yes
Sr	ICP-AES	9E+00	3.56E+02	6.00E+02		7.71E-02	5.2E-01	15	Yes
Ta	ICP-MS	7E-04	3.65E+00	6.15E+00	B	7.90E-04	3.0E-02	3	Yes
Tc	ICP-MS	3E-05	< 2E-03	< 3E-03		< 3E-07	2.6E-01	< 0.0001	Yes
Te	ICP-MS	2E-01	1.96E+02	3.30E+02		4.24E-02	1.3E-01	33	Yes
Tl	ICP-MS	2E-03	1.55E-01	2.61E-01	B	3.36E-05	4.5E-01	0.01	Yes
V	ICP-MS	3E-02	2.29E+01	3.86E+01	B	4.96E-03	3.2E-02	15	Yes
W	ICP-MS	1E-02	1.40E+01	2.36E+01	B	3.04E-03	2.4E-01	1	Yes
Y	ICP-MS	9E-03	1.17E+02	1.97E+02		2.53E-02	1.6E-01	16	Yes
Zn	ICP-AES	3E+01	7.90E+01	1.33E+02	J	1.71E-02	4.2E-01	4	Yes
Specification 8 Table TS-8.2 Analytes									
Ammonia [c]	none		NM	NM		NM	1.6E+00	NM	NM
C as CO_3	C (Furn)	1E+03	< 6E+03	< 9E+03	J	< 1E+00	3.0E+01	< 4	Yes
C as CO_3	C (HP)	5E+01	2.55E+04	4.30E+04		5.53E+00	3.0E+01	18	Yes
C as TOC	C (Furn)	1E+03	9.40E+03	1.58E+04		2.04E+00	1.1E+01	19	Yes
C as TOC	C (HP)	1E+02	6.50E+02	1.10E+03		1.41E-01	1.1E+01	1	Yes
Cl	IC-Inorg	1E+01	3.02E+01	5.09E+01		6.54E-03	3.3E-01	2	Yes
CN	CN	4E-02	< 4E-02	< 7E-02		< 9E-06	1.6E+00	< 0.001	Yes
NO_2	IC-Inorg	3E+02	9.32E+03	1.57E+04		2.02E+00	3.6E+01	9	Yes
NO_3	IC-Inorg	3E+02	5.41E+03	9.12E+03		1.17E+00			

Table 8.9. (Cont'd)

			UDS Average			Spec 8 Evaluation			
Analyte	Measurement Method	MDL µg/g	µg/g Wet	µg/g Dry	[d] DF	g/100 g waste oxide UDS	Spec Limit	% of Limit	Meets Spec
Specification 8 Table TS-8.4 Analytes									
Ag	ICP-AES	2E+01	5.50E+01	9.27E+01	J	1.19E-02	5.5E-01	2	Yes
Al	ICP-AES	4E+01	1.34E+05	2.26E+05		**2.90E+01**	1.4E+01	**207**	No
Ba	ICP-AES	6E+00	4.89E+02	8.24E+02		1.06E-01	4.5E+00	2	Yes
Bi	ICP-AES	6E+01	< 6E+01	< 1E+02	U	< 1E-02	2.8E+00	< 0.5	Yes
Ca	ICP-AES	2E+02	2.40E+03	4.04E+03		5.19E-01	7.1E+00	7	Yes
Cd	ICP-AES	9E+00	4.67E+03	7.87E+03		1.01E+00	4.5E+00	22	Yes
Cr	ICP-AES	1E+01	5.72E+02	9.64E+02		1.24E-01	6.8E-01	18	Yes
F	IC-Inorg	1E+02	3.44E+03	5.80E+03		7.45E-01	3.5E+00	21	Yes
Fe	ICP-AES	2E+01	6.68E+04	1.13E+05		1.45E+01	2.9E+01	50	Yes
K	ICP-AES	1E+03	< 1E+03	< 2E+03	J	< 3E-01	1.3E+00	< 23	Yes
Mg	ICP-AES	6E+01	4.40E+02	7.41E+02	J	9.53E-02	2.1E+00	5	Yes
Na	ICP-AES	3E+02	3.46E+04	5.83E+04		7.49E+00	1.9E+01	39	Yes
Ni	ICP-AES	2E+01	2.77E+03	4.67E+03		6.00E-01	2.4E+00	25	Yes
P	ICP-AES	6E+01	1.62E+03	2.73E+03		3.51E-01	1.7E+00	21	Yes
Pb	ICP-AES	6E+01	4.89E+02	8.24E+02	J	1.06E-01	1.1E+00	10	Yes
Pd	ICP-MS	3E-03	9.44E+01	1.59E+02		2.04E-02	1.3E-01	16	Yes
Rh	ICP-MS	6E-04	9.08E+01	1.53E+02		1.97E-02	1.3E-01	15	Yes
Ru	ICP-MS	2E-01	1.15E+03	1.94E+03		2.49E-01	3.5E-01	71	Yes
Si	ICP-AES	1E+03	3.73E+03	6.29E+03	J	8.08E-01	1.9E+01	4	Yes
SO_4 as S[b]	IC-Inorg	3E+02	6.21E+03	1.05E+04		**1.35E+00**	6.5E-01	**207**	No
Th	ICP-MS	3E-03	1.09E+02	1.84E+02		2.36E-02	5.0E+00	0.5	Yes
Ti	ICP-AES	2E+01	6.40E+01	1.08E+02	J	1.39E-02	1.3E+00	1	Yes
U	ICP-AES	1E+03	3.60E+03	6.07E+03	J	7.80E-01	1.4E+01	6	Yes
U	KPA	2E-01	3.17E+03	5.34E+03		6.86E-01	1.4E+01	5	Yes
U sum	ICP-MS	1E-01	3.68E+03	6.20E+03		7.97E-01	1.4E+01	6	Yes
Zr	ICP-AES	3E+01	1.95E+04	3.29E+04		4.22E+00	1.5E+01	28	Yes

Outlined and bolded results exceed or may exceed Specification 8 (Envelope D).

MDL: method detection limit; UDS: undissolved solids; NM: not measured

C as TIC and TOC: HP = hot persulfate oxidation method; Furn = furnace oxidation method

(a) Cs total calculated from measured ^{133}Cs by ICP-MS and ^{135,137}Cs calculated from supernatant Cs isotope mass fractions.

(b) Sulfur not measured. Sulfate from IC analysis converted to sulfur and compared to the specification.

(c) Ammonia analysis on wet centrifuged solids not specified in test specification or test plan.

(d) data flag: B = analyte measured in blank above EQL; T = estimated value, U = not detected above the reported MDL.

9.0 Procedures, Quality Control, and Data Evaluation

A discussion of procedures, data quality, and QC is provided below for each analytical method. Analytical instrument calibration and calibration verification were performed in accordance with the QA plan, *Conducting Analytical Work in Support of Regulatory Programs*, which is in compliance with HASQARD(8). Raw data including bench sheets, instrument printouts, data reduction, and calibration files are maintained or cross-referenced in the Project 42365 file (by ASR number).

The sample average, MRQ, data flags, and QC results (including QC flagging criteria) are presented in Table 9.1 through Table 9.4. The QC flagging criteria are defined in the test plan for both the supernatant analysis (i.e., Test plan Table 4, QC Parameters for Liquid Analysis) and wet centrifuged solids analysis (i.e., Test plan Table 5, QC Parameters for Solids Analysis). Where the result for one (or two) of the triplicate analysis is reported at <MDL (i.e., flagged with a U) and remaining sample result(s) is(are) >MDL (i.e., either not flagged or flagged with a J), the average is based only on the result(s) >MDL.

The QC and results evaluations provided in the following sections are limited to the analytes of interest defined by the test plan. Analytes other than those specified by the test plan are considered 'opportunistic' and are provided for information only. Some of these 'opportunistic' analytes have been measured per the requirements stated in the governing QA Plan or test plan; however, the data may not have been fully evaluated against the QC flagging criteria.

9.1 Inductively Coupled Plasma-Atomic Emission Spectroscopy

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F4

The AZ-101 supernatant samples were prepared by acid digesting per procedure PNL-ALO-128. The AZ-101 wet centrifuged solids were prepared using four different preparation methods; PNL-ALO-129 Mod 1 (acid leach), PNL-ALO-129 Mod 2 (acid digest), PNL-ALO-115 (KOH fusion), and PNL-ALO-116 (Na_2O_2 fusion). However, only the results from the PNL-ALO-129 Mod 2 and PNL-ALO-115 are discussed; a summary comparison of the results between the four different preparative methods is presented in Appendix G. Once dissolved, all samples were analyzed according to PNL-ALO-211, *Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry*. The detected analytes at or above the EQL (equivalent to ten times the MDL) were reported with an uncertainty of ±15% (2-σ). As the MDL was approached, uncertainty increases to 100%.

Quality control for the ICP-AES analysis consisted of triplicate samples, PBs (one for the acid digestions and two for the fusions), MSs, LCS/BS (or solid LCS), PS, serial dilution, calibration verification check standards, interference check standards, and linear range check standards. Matrix spike recovery, LCS/BS (or solid LCS) recovery, and precision (based triplicate RSD) QC flagging criteria are defined by the test plan.

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[8] Hanford Analytical Services Quality Assurance Requirements Document; Volume 4: Laboratory Technical Requirements. DOE/RL-96-68, Rev 2, September 1998.

9.1.1 Acid Digest (PNL-ALO-128) – Supernatant

The QC flagging criteria were met except for the sodium LCS/BS recovery and the MS recoveries for boron, cerium, thorium, and tungsten. The sodium LCS/BS recovery of 121% exceeds the LCS flagging criterion by 11%; the LSC/BS criterion for sodium is significantly more restrictive that either the PNWD QA Plan or SW-846 [(9)] QC requirements. This QC failure is highlighted in the QC tables. Other sodium QC analyses were acceptable and the failure is considered restricted to the LCS/BS; thus, the analysis results are not flagged with an 'X' as detailed in Section 8.1. The boron MS recovery (136%) significantly exceeds the MS flagging criterion. The PB had significant boron contamination (as well as silicon contamination), most likely from contamination from glassware. Moderate variability (e.g., ±20%) of the PB boron concentration impacts the MS recovery at the concentrations measured. Based on the processing dilutions and the spiking concentrations, cerium, thorium, and tungsten were at final concentrations below the EQL, and the MS recoveries were not calculated; PSs were not prepared. Cerium, thorium, and tungsten were not detected above the EQL in any sample.

9.1.2 Acid Digest (PNL-ALO-129 Mod 2) – Wet Centrifuged Solids

The QC flagging criteria were met except the RSD criterion for chromium, potassium, titanium, and sodium and the MS recovery criterion for a number of analytes. The MSs were not recovered on a number of analytes because the spiking concentration was low with respect to the sample concentration (i.e., spike <20% of sample), and a few MSs were not calculated since the MS were below the EQL following processing dilution. A batch PS (sample 01-00955, AW-101 simulant) was performed for all analytes specified in the test plan and all PS recoveries were well within the QC flagging criterion. The poor RSD for potassium and titanium is likely due to the measured concentration being near the MDL. For chromium and sodium, the RSD appears to be biased by the duplicate results, with the duplicate chromium being nearly twice the concentration of the sample and triplicate. The chromium results for the sample and triplicate correspond very well with the chromium results obtained from the KOH fusion digestion. The 3.5% RSD criterion for sodium is difficult to meet by ICP-AES, especially on solids where sample heterogeneity from processing of small sample sizes contributes significantly to the variability of the results. The results from both the acid digestion and KOH fusion digestion of the wet centrifuged solids produced similar RSD for sodium (6.0% and 7.5%, respectively). Since the acid digestion procedure used for processing the wet centrifuged solids uses HF and evaporates the samples to dryness during processing, silicon cannot be measured by this method.

9.1.3 Fusion Digestion (PNL-ALO-115) – Wet Centrifuged Solids

The QC flagging criteria were met except the RSD criterion for silver, molybdenum, and phosphorus and the LCS/BS recovery criterion for silver, bismuth, calcium, and zinc. The poor RSD for silver and phosphorus is likely due to the measured concentration being near the MDL. The slightly high LCS/BS recovery for calcium and zinc (i.e., 122% and 128%, respectively) is not considered significant, since the solid LCS (NIST SRM-2710 Montana Soil), which better represents the fusion digestion processing, demonstrated calcium and zinc recoveries at 99% and 98%, respectively. The LCS/BS recoveries for silver and bismuth were approximately 50% higher than expected; the reason is unknown. (However, it should be noted that the measured concentration silver is about 3 times lower than the MRQ and bismuth

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9 U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition, Office of Solid Waste and Emergency Response. Washington, D.C.

is 30 times lower.) All other batch and analysis QC (e.g., PS, calibration check standard, linear range checks) produced results well within acceptance criteria, suggesting that the reported results are correct and that there may be difficulties in processing ‘acid-based’ spiking solution through the caustic fusion process. Both the silver results and bismuth results have been flagged as suspect data (i.e., X flag). It can be noted that the fusion results produced similar results to the acid digestion (PNL-ALO-129 Mod 2), except that the aluminum, iron, and particularly zirconium appear slightly lower.

9.2 Inductively Coupled Plasma-Mass Spectrometry

Table 8.1 through Table 8.4 and Table 9.1 through Table 9.4. Appendix F13

The PNL-ALO-128 acid-digested samples of the supernatant and direct dilutions of the supernatant (for ^{127}I and ^{129}I), as well as the PNL-ALO-114, PNL-ALO-115, and PNL-ALO-116 fusion samples of the wet centrifuged solids, were submitted for ICP-MS analysis and analyzed according to procedure PNL-SC-01, *Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*. Except for the MS and LCS, the acid digest solutions and the fusion digestions from PNL-ALO-115 were from the same as those analyzed by ICP-AES.

Quality control for the ICP-MS analysis consisted of sample triplicates, PSs, MS, LCS/BSs (solid LCSs), PSs, and calibration verification check standards and blanks. Matrix spike recoveries, LCS recoveries, and precision (based on duplicate analyses) QC criteria are defined by the test specification. Radionuclides were not spiked into the LCS/BS or the MS samples during digestion processing. The quantities of isotopics required would be extremely large given the large dilutions necessary to perform the analysis. Therefore, radionuclides are post-spiked into samples following digestion.

9.2.1 Direct Dilution – Supernatant

Both ^{127}I and ^{129}I were determined from direct dilutions of the AZ-101 superantant; i.e., no processing was performed on the supernatant. Revision 1 of this report replaces of all ^{127}I and ^{129}I results and related discussion of results presented in Revision 0. Updating of the iodine results was necessary due to standards verification issues; i.e., potential biases were discovered in ^{129}I standards used to calibrate and verify the ICP-MS.

No ^{127}I was detected in the AZ-101 supernatant and the ^{129}I concentration was below the EQL and is J-flagged. All QC met acceptance criteria.

9.2.2 Acid Digest (PNL-ALO-128) – Supernatant

Except for the poor RSD for ^{239}Pu, analytes and radionuclides analyzed by ICP-MS met the QC flagging criteria. The poor precision is attributed to the low concentrations of the ^{239}Pu in the AZ-101 samples. The failure of the MS may be due to loss from the acid digestion processing.

The Cs isotopic mass (^{133}Cs, ^{135}Cs, and ^{137}Cs) was determined following separation of the Cs from isobaric interferences using high-performance ion chromatography (HPIC). Relative abundances of the cesium isotopes were measured and mass concentrations determined relative to the measured ^{133}Cs. QC acceptance criteria were not specified for cesium isotopic distribution. Triplicate samples results agreed within 3% RSD, and the computed ^{137}Cs compared within 15% with the GEA result.

9.2.3 Fusion Digestion (PNL-ALO-115) – Wet Centrifuged Solids

In general the QC performance for the analytes and radionuclides measured by ICP-MS was good. However, a number of analytes demonstrate poor precision (i.e., RSD >15%). In each such case, the samples have significant blank contribution (i.e., B flagged), and a variable blank contribution is the most likely reason for the poor RSDs. Also, there are a few failures of the LCS/BS and MS analytes and radionuclides. The majority of these failures are due to the spiking levels selected versus the final dilutions analyzed. For all LCS/BS failures a post-spiked PB was analyzed and for all MS failures a PS was analyzed. The post spiked PB (laboratory LCS/BS) and sample PS produced recoveries within the QC flagging criteria.

9.2.4 Fusion Digestion (PNL-ALO-114, Iodine only) – Wet Centrifuged Solids

^{127}I and ^{129}I were determined by ICP-MS following fusion of the wet centrifuged solids (using the iodine preparation option of the fusion procedure). Revision 1 of this report replaces of all ^{127}I and ^{129}I results and related discussion of results presented in Revision 0. Updating of the iodine results was necessary due to standards verification issues; i.e., potential biases were discovered in ^{129}I standards used to calibrate and verify the ICP-MS.

For the reanalysis of ^{127}I and ^{129}I, all QC met acceptance criteria. However, the MDL for ^{129}I is higher than the MRQ defined by the test plan.

9.2.5 Fusion Digestion (PNL-ALO-116, Platinum Group Metals only) – Wet Centrifuged Solids

Poor precision (i.e., RSD > 15%) was evident for platinum and palladium. Since the MDL for these analytes is significantly lower than the measured concentrations and since the QC samples show good to excellent recoveries, poor precision may be due to either heterogeneity of the sample (i.e., only 0.2 g samples processed) or the fusion digestion produced non-reproducible results. Since the method has been evaluated for quantitative dissolution of the platinum group metals from various matrices, sample heterogeneity is the most likely reason for the poor precision.

9.3 U Analysis by Kinetic Phosphorescence Analysis (KPA)

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F12

Total uranium was measured on the AZ-101 supernatant and wet centrifuged solids according to procedure RPG-CMC-4014, *Uranium by Kinetic Phosphorescence Analysis (KPA)*. For the total uranium analysis by KPA the supernatant sample was prepared in the SAL per procedure PNL-ALO-128 (HNO_3-HCl digestion) and the wet centrifuged solids per procedure PNL-ALO-115 (KOH fusion).

Prior to analysis by KPA, the supernatant and wet centrifuged solids preparations were evaporated to dryness several times with nitric acid to eliminate halides and any residual organics, then reconstituted in 0.5 M nitric acid. These reconstituted solutions, free of halides and organics, were used for the KPA uranium measurement. The wet centrifuged solids analysis met all QC flagging criteria; i.e., the LCS/BS, MS, and triplicate RSD are well within the QC flagging criteria and the PBs have concentrations <1% of the sample concentrations. However, the SAL supernatant digestion preparations ran poorly on the KPA system and produced results that failed QC flagging criteria (e.g., the linearity was poor, the lifetimes were short, and the MS failed).

Measurement of the uranium (by KPA) in the supernatant required an alternate digestion and separation of the uranium by anion exchange in order to obtain solutions suitable for KPA. This anion exchange separation is an abbreviated form of the uranium separation in procedure RPG-CMC-4017, *Analysis of Environmental Water Samples for Actinides and Strontium-90*. This separation is very fast, gives quantitative recovery of the uranium, and eliminates all interferences in the KPA. Following the anion exchange separation, the supernatant samples ran very well by KPA; i.e., the LCS/BS, MS, and triplicate RSD are well within the QC flagging criteria and the PB has a concentration <0.5% of the sample concentrations. Highlights of the digestion and anion exchange method modifications for preparation of the supernatant are:

- Supernatant samples heated in a beaker with HNO_3 to destroy organics, evaporated to dryness, then dissolved in HCl.
- Each sample solution passed through an anion exchange column (approximately 1 mL of strong base anion exchanger in concentrated hydrochloric acid; e.g., AG MP-1, 50-100 mesh, chloride form) and the effluent discarded.
- Anion exchange column washed with HCl solution and the effluent discarded.
- Uranium eluted from the column into a collection beaker using 0.5 M HNO_3.
- The HNO_3 effluent evaporated to dryness to eliminate the chloride ion, then re-constituted in a known volume of 0.5 M HNO_3.
- The 0.5 M HNO_3 solution measured for uranium concentration per procedure RPG-CMC-4014.

9.4 Radiochemical Analyses

Table 8.1, Table 8.3, Table 9.1, and Table 9.3. Appendix F12

For most radiochemical analyses, the supernatant samples were prepared per procedure PNL-ALO-128 (HNO_3-HCl digestion), and the wet centrifuged solids samples were prepared by PNL-AOL-115 (KOH fusion). The supernatant digests were analyzed for gamma emitters, total alpha, ^{79}Se, ^{90}Sr, ^{238}Pu, ^{239,240}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm. The wet centrifuged solids fusion digestions were analyzed for gamma emitters, total alpha, ^{90}Sr, ^{238}Pu, ^{239,240}Pu, ^{241}Pu, ^{242}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm. Some radiochemical analyses were performed using as-received material (i.e., samples processed by neither PNL-ALO-128 or PNL-ALO-115). These analyses include 1) ^{3}H, ^{14}C, and ^{99}Tc (as pertechnetate) on the supernatant, 2) ^{14}C on the wet centrifuged solids, and 3) ^{3}H on the wet centrifuged solids following DIW leaching per procedure PNL-ALO-103. For nearly all radiochemical analyses, the LCS/BSs and MSs were prepared at the analytical workstation; the exception being ^{3}H for the wet centrifuged solids where the LCS/BS and MS are processed through the leaching process. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate.

9.4.1 Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Photon Spectrometry*. No LCS/BSs or MSs are required for this analysis; the measurement is a direct reading of the gamma energy and is not subject to matrix interferences; laboratory control standards analyses and background counts were performed. The samples were counted for up to 14 hours to give extended-time MDAs. Supernatant and wet centrifuged solids triplicate results were in good agreement taking into account the relative uncertainties in the measurements. Negligible levels of ^{137}Cs were detected in both the supernatant and wet centrifuged solids PBs. Due to the very high concentration of ^{137}Cs in the supernatant, no other gamma emitting isotopes were detected. The supernatant MDA values for all gamma emitters, except ^{137}Cs, exceed the MRQ values defined in the test plan. For the wet centrifuged solids, all radionuclides specified in the test plan, except ^{126}Sn, ^{134}Cs, and ^{152}Eu, were detected; however, the MDAs for all radionuclides exceed the MRQ values defined in the test plan. The decay correction reference date for radionuclides reported by GEA is October 9, 2001.

9.4.2 Total Alpha Activity

The total alpha activity was determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001, *Source Requirements for Gross Alpha and Gross Beta Analysis*. The samples were counted on Ludlum detectors according to RPG-CMC-408, *Low Background Alpha and Beta Counting - Proportional*. Alpha activity was not detected in the supernatant by this method, and the MDA is much lower than the MRQ defined in the test plan. Summing the individual alpha emitters from plutonium, americium, and curium provides the best estimate of the total alpha activity. The total alpha activities determined in the wet centrifuged solids samples are significantly higher (approximately 35%) than the sum of the wet centrifuged solids alpha emitters for reasons that are not understood. There is negligible alpha activity in the PB and the LCS/BS and MS recoveries are acceptable. The total alpha activities for the wet centrifuged solids are significantly ($>10^4$ times) higher than the MRQ value. The decay correction reference date for the total alpha activity analysis is October 8, 2001.

9.4.3 Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417, *Separation of Am and Pu and Actinide Screen by Extraction Chromatography*. The separated fractions were precipitation plated according to PNL-ALO-496, *Precipitation Plating of Actinides for High-Resolution Alpha Spectrometry*, and counted by alpha spectrometry according to PNL-ALO-422, *Solution Analysis: Alpha Spectrometry*. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium chemistry and both of these elements were traced with ^{243}Am. For both the supernatant and wet centrifuged solids samples, the plutonium and americium radiochemical yields were acceptable, averaging about 95% for plutonium and 80% for americium/curium. The decay correction reference date for the plutonium isotopes (except ^{241}Pu and ^{242}Pu) is October 17, 2001, for ^{241}Pu is June 27, 2002, for ^{242}Pu is June 20, 2002, and for americium and curium isotopes is October 18, 2001.

The supernatant and wet centrifuged solids LCS/BS and MS recoveries are acceptable, ranging from 86% to 112% recovery, and triplicate results for all radionuclides measured are in good agreement, except for the measurement uncertainties. The RSD for ^{243,244}Cm in both the supernatant and wet centrifuged solids exceeded the QC flagging criterion of <15%; however, the ^{243,244}Cm concentration is only 2-3 times the MDA and poor precision is not unexpected. The PB for the supernatant demonstrated significant contamination with ^{238}Pu, ^{241}Am, and ^{243,244}Cm at levels up to 50% of the activities in the supernatant samples; thus, the results have been flagged with a 'B'. Most of the total alpha activity in the supernatant is due to ^{239,240}Pu (i.e., about 70%) and the supernatant PB activity for ^{239,240}Pu meets the acceptance criteria of <EQL or <5% of the sample activity. Per discussions with BNI, re-processing and re-analysis of the supernatant was not performed since the alpha activities for ^{238}Pu, ^{241}Am, and ^{243,244}Cm are well below the MRQ values (i.e., 30-300 times). The alpha activities in the SAL PBs for the wet centrifuged solids, as well as the laboratory PB, were negligible. As indicated in Section 9.4.2, 1) the sum of the alpha emitters detected in the supernatant is the best estimate of the total alpha activity, since the measured total activity results were below the MDA; 2) the sum of the alpha emitters detected in the wet centrifuged solids is about 35% lower than the measured total alpha activity for reasons unknown.

The ^{241}Pu activity in the wet centrifuged solids samples was determined by performing a liquid scintillation beta count on the precipitation mount that was prepared for the alpha energy analyses, as described above. The samples were analyzed using procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. A blank filter was used to determine the background counting rate, and a blank filter was spiked with a ^{241}Pu standard to determine the beta counting efficiency. The LCS/BS analyzed with the batch had a recovery of 113%, well within the QC flagging criterion. The batch MS was prepared from an AZ-101 sample from ASR 6403 (02-02246, AZ-101-Cs-Eluate) and had an acceptable recovery of 114%. The ^{241}Pu activity in the two PBs and the laboratory reagent blank were negligible with respect to the activity in the samples. The triplicate results were in excellent agreement with an RSD of 3%. At an average 11.8 µCi/g, the sample activity is approximately ten times greater than the MRQ of 1.2 µCi/g.

Since ^{242}Pu was used as a tracer for the initial plutonium measurements, it could not be measured. A second plutonium separation was performed without using any tracer. The ratio of the counts in the ^{242}Pu peak to the ^{239,240}Pu peak times the ^{239,240}Pu activity measured in the initial run (using the ^{242}Pu tracer) was used to determine the absolute ^{242}Pu activity. No peak was observed for ^{242}Pu in the triplicate samples or the two PBs, and the ^{242}Pu MDA values were calculated from the background on the tail of the ^{239,240}Pu alpha peak. A ^{242}Pu MS gave a recovery of 98%.

9.4.4 Strontium-90

The strontium separation was performed on aliquots of the digestions according to PNL-ALO-476, *Strontium Determination using Sr-SPEC*, and radiochemical yields were traced with ^{85}Sr. The separated fractions were beta-counted according to RPG-CMC-408, *Low Background Alpha and Beta Counting – Proportional* (for ^{90}Sr determination). Following beta counting the samples were gamma counted according to PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Photon Spectrometry* (for ^{85}Sr yield determination and ^{137}Cs impurity assessment). The supernatant and wet centrifuged solids samples were processed and analyzed in separate batches. The LCS/BS recovered at 94% for the supernatant and 111% for the wet centrifuged solids, well within the QC flagging criterion. At 95% and 107% for the supernatant and wet centrifuged solids, respectively, the MS recoveries were also well within the QC flagging criterion. ^{90}Sr was not detected in either the supernatant or the wet centrifuged solids PB above the MDA, and no ^{137}Cs was detected in the separated strontium fraction. The ^{90}Sr activity in the wet centrifuged solids is significantly greater than 100 times the MRQ value; whereas the ^{90}Sr in the supernatant was below the MRQ. The decay correction reference date for the wet centrifuged solids ^{90}Sr is October 15, 2001 and for the supernatant is September 4, 2002.

The supernatant analysis for ^{90}Sr was performed three different times because of QA/QC problems. The data from the final analysis (average 5.0E-02 µCi/mL) is reported in the data tables. The first result (<MDA, which was 5.0E-01 µCi/mL) and the second result (average 7.9E-02 µCi/mL) were consistent with the final analysis. These results are significantly lower than the value currently assigned to the tank

AZ-101 supernatant[10] (9.3E-01 μCi/mL). These results are also lower than those obtained on the first filtrate following separation from the solids by the CUF[11] (1.19E+00 μCi/mL). The sample material used for the CUF run was from the same composite as the characterization sample (different composite sub-samples), but the CUF filtrate was analyzed immediately following the CUF phase separation. This suggests that some of the strontium in the archived phase-separated characterization sub-sample could have precipitated and adhered to the container walls prior to sub-sampling for the analysis.

It is recommended that the ^{90}Sr results from the CUF run (i.e., 1.19E+00 μCi/mL) be used as the best estimate of the supernatant ^{90}Sr concentration. This concentration agrees with other reported values for the tank supernatant.

9.4.5 Technetium-99 (as Pertechnetate)

The radiochemical ^{99}Tc determination was requested to measure only technetium in the +7 oxidation state (as pertechnetate); therefore, all sample processing was conducted so as not to alter the original technetium oxidation state. Small aliquots from the as-received supernatant (no digestion) was taken for analysis according to procedure PNL-ALO-432, *Separation of Technetium by Cation Exchange and Solution Extraction Prior to Measurement by Beta Counting*. This procedure normally requires the use of a sodium dichromate addition to oxidize all technetium to the +7 oxidation state. The sodium dichromate addition was omitted and otherwise the procedure was performed as written. The separated fraction was then counted according to RPG-CMC-408, *Low Background Alpha and Beta Counting - Proportional*. The sample was also counted by liquid scintillation counting according to PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*, to confirm that the beta energy spectra matched that of ^{99}Tc and that no other beta emitters were present. The decay correction reference date for ^{99}Tc by this method is August 5, 2002.

An initial analysis for ^{99}Tc^{+7} produced results that indicated that only about 25% of the total ^{99}Tc was pertechnetate. These results were inconsistent with information available on the AZ-101 supernatant; therefore, the sample analyses were rerun. Only the results from the rerun are reported. The triplicate analyses are in excellent agreement with a RSD of 1%. The level of ^{99}Tc in the PB was negligible. The LCS/BS recovery was 95% and the MS recovery was 94%, both well within the QC flagging criteria. The measured sample activities were well above the requested MRQ value of 1.50E-03 mCi/L, while the MDA levels are slightly greater than a third of the MRQ value.

9.4.6 Tritium

The wet centrifuged solids sample was prepared in triplicate for tritium analysis using a water leach method PNL-ALO-103, *Water Leach of Sludges, Soils and Other Solid Samples*. The wet centrifuged solids leachate solutions as well as the as-received composite supernatant samples were distilled using procedure PNL-ALO-418, *Tritium Determination in Soil and Water Using a Lachat Micro-Dist™ System*. The tritium was measured by liquid scintillation counting (LSC) according to procedure PNL-ALO-474,

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[10] Tank Waste Information Network System (August 2002): ^{90}Sr (liquid phase) concentration equal to 7.5E-01 μCi/g at density of 1.24 g/mL.

[11] PNWD analysis: ASR 6284 Sample AZ-A (first filtered supernatant sample from cells unit filter, chemically unaltered).

Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry. The decay correction reference date for tritium is February 2, 2002.

The samples have very high beta activity (typically from ^{137}Cs contamination) and a low tritium concentration; a combination that has caused trouble in previous tritium analyses using distillation procedure PNL-ALO-418. Therefore, prior to measuring the tritium by LSC, two procedure modifications were made to eliminate the other beta emitters that would interfere in tritium measurement; 1) after distillation, a cation exchange column (Dowex® 50-WX8, 0.25-mL, excess water removed) was used to remove any remaining beta emitters, and 2) the sample was distilled a second time. These modification are being incorporated in the next revision of procedure PNL-ALO-418.

These modifications resulted in very clean beta energy spectra with no trace of other beta emitters. The SAL PBs did not show any significant tritium activities. The supernatant and wet centrifuged solids samples demonstrated excellent reproducibility (i.e., RSD values of 1%). The SAL MS activity for the supernatant was very low compared to the sample activity; thus, the calculated recovery (57%) has a very high uncertainty. The SAL MS for the wet centrifuged solids recovered at 90%. The SAL LCS/BSs recovered at 92% and 91% for the supernatant and wet centrifuged solids, respectively. A laboratory LCS/BS recovered at 89%, both indicating that the method modifications do not impact the tritium measurement. Tritium was detected near the MRQ for the supernatant and about 50% below the MRQ for the wet centrifuged solids.

9.4.7 Carbon-14

The supernatant and wet centrifuged solids samples were sub-sampled in the SAL and prepared in triplicate for ^{14}C analysis according to Method A in procedure PNL-ALO-482, *Determination of Carbon-14 in Radioactive Liquids, Soils, and Sludges*. The trap solution generated from PNL-ALO-482 was measured by LSC according to procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. Beside the triplicate samples, an LCS/BS, MS, and PB were analyzed. Like the TIC and TOC results measured by PNL-ALO-381, the samples, duplicate, LCS, PB, and MSs were corrected for the recovery obtained on calibration standards that were processed and analyzed with the samples. The decay correction reference date for ^{14}C in the wet centrifuged solids is March 5, 2002 and in the supernatant is June 19, 2002.

The LCS for the supernatant and wet centrifuged solids analysis were recovered at 101% and 94%, respectively and are within the QC flagging criterion. The MSs demonstrated good recoveries; 112% for the supernatant and 100% for the wet centrifuged solids. The ^{14}C measured in the supernatant is approximately three times the MRQ and RSD results (i.e., 4%) are well within the QC flagging criterion of <15%. However, the triplicate results for the wet centrifuged solids had very poor agreement; an RSD of 53%. The ^{14}C measured in the wet centrifuged solids at only five times the MDA, and the average result is approximately 60% of the MRQ. The PB exhibits significant ^{14}C contamination. Contamination, as evidenced by the PB, and the low ^{14}C concentration are the primary reasons for the poor precision demonstrated for the wet centrifuged solids. Although the PB contamination may be a factor in the poor precision, the samples were not re-prepared because the results were less than the MRQ.

9.4.8 Selenium-79

The AZ-101 supernatant sample was sub-sampled in the SAL and analyzed in triplicate for ^{79}Se according to Method A in procedure PNL-ALO-440, *Selenium-79 by Ion Exchange and Distillation Separation prior to Measurement by Liquid Scintillation Counting*. This procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a selenium bromide distillation and reduction of selenium to elemental form.

Since ^{79}Se is not available as a radioactive standard, a selenium carrier was used in the analysis for establishing the yield and ^{14}C was used to establish the instrument efficiency since it has a very similar beta maximum energy (156 keV for ^{14}C versus 149 keV for Se-79). Direct 2-mL aliquots of the diluted acid digest supernatant were analyzed. The gravimetric recoveries for the PB and triplicate samples ranged from 25% to 80%. The ^{79}Se activities were measured by LSC according to procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. Peaks were observed in the ^{79}Se region of interest in the beta energy spectra; no other higher energy beta contaminants were observed. The PB had no discernable peak in the beta energy spectrum, and the counts above background (i.e., PB reported result) are most likely not due to ^{79}Se. Since ^{79}Se is not available as a standard, no LCS/BS or MS was analyzed. The average results for the ^{79}Se are approximately ten times higher than the MRQ and approximately 40 times the MDA. At 17%, the RSD does not meet the QC flagging criteria; this is primarily due to the low concentration and the very low carrier recovery for the duplicate.

9.5 Inorganic Anions

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F5

The wet centrifuged solids samples were prepared for inorganic anion analysis in the SAL by water leach procedure PNL-ALO-103, *Water Leach of Sludges, Soils, and Other Solid Samples*, at a leaching ratio of approximately 10:1 (DIW : wet centrifuged solids). The supernatants and the wet centrifuged solids leach solutions were prepared for analysis at the IC workstation by diluting with the stock IC mobile phase solution (eluent). The anion analysis was conducted according to method PNL-ALO-212, *Determination of Inorganic Anions by Ion Chromatography*. Prior to analysis, the wet centrifuged solids leach solutions required 10-fold to 100-fold dilution and the supernatant samples required 1,000-fold to 10,000-fold dilution to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the wet centrifuged solids leachates at dilutions less than 10 fold and the supernatant at dilutions less than 1,000 fold.

Quality control for the anions analysis consisted of sample triplicates, PBs, MSs, LCS/BSs, PSs, and calibration verification check standards and blanks. Matrix spike recovery, LCS/BS recovery, and precision (based on triplicate RSD) QC flagging criteria are defined by the test plan.

All supernatant QC analyses produced results within the QC flagging criteria. However, the RSD for the chloride results is near the RSD criterion and the initial MS preparation did not provide useful spike recoveries for nitrate and nitrite (i.e., spike concentrations were significantly less than 20% of the sample concentrations). The chloride results are only about two times the EQL, leading to the poorer precision. The initial MS was prepared at a sample dilution of 2,000 fold; at this dilution, nitrate and nitrite produced results that exceeded the highest calibration standard. A MS prepared from a 20,000-fold dilution of the sample demonstrated recoveries well within the QC flagging criterion.

Most of the wet centrifuged solids QC analyses produced results within the QC flagging criteria. The exceptions are the chloride and oxalate RSD results, the initial MS recoveries for all anions, and the LCS/BS result for oxalate. The phosphate RSD is at the QC flagging limit of 15%. The poor precision (i.e., high RSD) is mostly demonstrated on anions that may be present as solids (e.g., fluoride, chloride, phosphate, sulfate, and oxalate) and is most likely attributed to variability of the leaching process. The MS prepared and processed through the solids leaching procedure could not be recovered since the dilution required for the analysis of the AZ-101 wet centrifuged solids leachates diluted the spikes to below the EQL. Post spikes were prepared on the leachate, and all PS recoveries were within the QC MS flagging criterion. The oxalate in the LCS/BS processed through the SAL was not recovered (i.e., recovery = 0%). The LCS/BS standard solution was analyzed and confirmed to have oxalate at the expected concentration. The loss of the oxalate is most likely due to precipitation of the oxalate as sodium oxalate (sodium being the only cation in the LCS/BS standard solution), but this has not been confirmed. The oxalate results from the inorganic IC analysis are for information only; oxalate is measured and reported as part of the organic acids analysis (See Section 9.11).

9.6 TOC/TIC by Hot Persulfate and Furnace

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F6

The AZ-101 as-received supernatant and wet centrifuged solids were analyzed in triplicate for TOC and TIC by two different procedures: Procedure PNL-ALO-381, *Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method*, and PNL-ALO-380, *Determination of Carbon in Solids Using the Coulometric Carbon Dioxide Coulometer*.

9.6.1 Hot Persulfate Method (PNL-ALO-381)

The hot persulfate wet oxidation method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. All sample results are corrected for average percent recovery of system calibration check standards and are also corrected for contribution from the system blanks, as per calculations defined in procedure PNL-ALO-381.

For both the supernatant and wet centrifuged solids analyses, pure chemical solid compounds are used for system calibration check standards as well as for the LCS and MSs. The TIC analysis uses two calcium carbonate compounds and the TOC analysis uses two α-Glucose compounds. The QC for the method involves, sample triplicates, LCS, and MS. Although PBs are analyzed, they are not reported since their average is subtracted from the sample instrument result prior to calculating the reported results.

The supernatant and wet centrifuged solids samples were analyzed in separate analytical batches. For the supernatant analysis; three calibration check blanks and three calibration check standards were run at the beginning and end of the analysis run. The blanks averaged 22 μgC TIC and 59 μgC TOC, which is about typical for the method. The calibration check standards for the supernatant analysis averaged 96% for TIC analysis and 99% for the TOC analysis. For the wet centrifuged solids analysis, five calibration check blanks and four calibration check standards were run at the beginning and end of the analysis run. Although the TOC blank is slightly higher than normal, the average TOC blank (101 μgC) and average TIC blank (18 μgC) are considered acceptable for the concentration of TIC and TOC measured in the samples. The standard deviation for the TOC blanks was outside the historical pooled standard deviation used to establish the MDL; this indicates that there is more variability in the blank than normal. The calibration check standards for the wet centrifuged solids analysis averaged 97% for TIC analysis and 101% for the TOC analysis.

Except for the TOC results on the wet centrifuged solids, the TIC and TOC RSDs for both the supernatant and wet centrifuged solids meet the QC flagging criterion of <15%. Since the average wet centrifuged solids TOC result of 820 μg/g is less than five times the average MDL of 140 μg/g for the wet centrifuged solids TOC measurements, a RSD of 21% is not unreasonable. The MS and LCS recoveries for both the supernatant and wet centrifuged solids are well within the QC flagging criterion of 75% to 125%.

9.6.2 Furnace Oxidation Method (PNL-ALO-380)

The furnace oxidation method determines TOC by oxidizing organic carbon in oxygen at temperatures between 600°C and 750°C and TC by oxidizing all carbon species at 1000°C. By the furnace oxidation method, TIC is determined by difference. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the system blank, as per calculations defined in procedure PNL-ALO-380. The temperature selected for the TOC analysis for the AZ-101 samples was 700°C.

For both the supernatant and wet centrifuged solids analyses, pure chemical solid compounds are used for system calibration check standards as well as for the LCS and MSs. The TC analysis (1000°C) uses two calcium carbonate compounds and the TOC analysis (700°C) uses two α-Glucose compounds. The QC for the method involves, sample triplicates, LCS, and MS. Although PBs are analyzed, they are not reported since their average is subtracted from the sample instrument result prior to calculating the reported results.

The supernatant and wet centrifuged solids TOC samples were analyzed in the same analysis batch, as were the supernatant and wet centrifuged solids TC samples. For both the TOC and TC analyses, the performance of the coulometer analysis system is checked by analyzing calibration check standards and calibration check blanks at the beginning and end of the analysis run. The average recovery for TOC analysis was 101% and for the TC analysis was 95%, well within the procedure requirements of 90% to 110%. The average TOC blank (22 μgC) and TC blanks (17 μgC) generally represent less than 5% of the sample concentration.

Except for the TOC on the supernatant, which has a RSD of 21%, the TC and TOC RSDs for both the supernatant and wet centrifuged solids meet the QC flagging criterion of <15%. The reason for the poor precision on the supernatant TOC is unknown; however, the duplicate analysis appears to be significantly lower than the sample and triplicate analysis. The MS and LCS recoveries for both the supernatant and wet centrifuged solids are within the QC flagging criteria of 75% to 125%.

9.6.3 Comparison of TIC/TOC by Hot Persulfate and Furnace Oxidation Methods

Table 8.2 presents the TOC and TIC results obtained from the hot persulfate method and the furnace oxidation method for the AZ-101 as-received supernatant and wet centrifuged solids. The TIC results from the furnace method are obtained by difference (TC – TOC), with the analysis being performed on two independent sample aliquots. The TC for the hot persulfate method is the summation of the TIC and TOC, with the analyses being performed on the same aliquot under different oxidation conditions.

9.6.3.1 Wet Centrifuged Solids Results

The two methods appear to produce similar results for TC for the wet centrifuged solids samples; however, there are significant differences between the TIC and TOC results. It appears that the nearly all the carbon (both inorganic and organic compounds) combusts at the 700°C used for the furnace method and produces a TOC result that is biased high; some metal carbonates, such as iron, magnesium, and nickel, will typically fully or partially oxidize at 700 °C. It is unlikely that the TIC result from the hot persulfate acid wet oxidation is biased high; i.e., organic compounds typically present in the tank waste should not be oxidized. Conclusion: Best results are most likely the hot persulfate results for the TIC and furnace results for TC, with the TOC being the difference (i.e., 9,670 - 8,380 = 1,290 µg/g).

9.6.3.2 Supernatant Results

For the supernatant sample, the trend is similar (i.e., inorganic carbon compounds combusting during the 700°C furnace analysis) but not as pronounced. The analysis time for the furnace method TOC analysis is 10 minutes; extending the analysis time by 20 minutes produced TOC results approximately twice as high as those reported. Typically, this effect is either from difficult to oxidize organic compounds or inorganic compounds that wholly or partially oxidize at 700°C. The fact that the furnace TC results are significantly lower than the hot persulfate TC results is very unusual and is difficult to evaluate. However, the hydroxide titration (2nd and 3rd inflection points equivalent to 0.88 and 0.70 molar OH, respectively) results suggest that the carbon from carbonate should be closer to the hot persulfate TIC results (see Section 9.9). Based on the hydroxide supporting data, the TIC furnace result for the supernatant (i.e., average 5,830 µg/mL) is highly suspect.

9.7 Cyanide (CN) Analysis

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F7

The AZ-101 as-received supernatant and wet centrifuged solids were distilled and analyzed in triplicate. The supernatant and wet centrifuged solids samples were micro-distilled according to PNL-ALO-287, *Midi and Micro Distillation of Cyanide in Liquid and Solid Samples*, with the addition of sulfamic acid to minimize interference from high nitrates present in the sample. The distillates were analyzed by automated spectrophotometry for CN concentration according to PNL-ALO-289, *Total Cyanide Determination with Spectrophotometry (Manual or Automated) or Argentometric Titration*. Due to the dose levels, the wet centrifuged solids samples were distilled in the SAL; the supernatant samples were distilled at the cyanide workstation. Quality control for the cyanide analysis consisted of sample triplicates, PBs, MSs, LCS (BS for supernatant and solids LCS for wet centrifuged solids), and calibration verification check standards and blanks.

Three LCSs were prepared, distilled, and analyzed. A liquid LCS prepared from a dilution of the highest calibration standard, which was prepared from sodium cyanide (97% assay, from Aldrich), meets the QC flagging criterion. Two solid LCSs (ERA Priority PollutnT® reference materials), identified in this report as LCS solid-old and LCS solid-new, were distilled and analyzed. The LCS solid-old is an expired solid standard, which has consistently produced satisfactory results and the recovery meets the QC flagging criterion of 80% to 120%. And at 161 µg/g, the result is well within the vendor's advisory range of 71 to 301 μg/g. The LCS solid-new is from a newly procured solid standard, which has a significantly larger mesh size than the LCS solid-old. The LCS solid-new is a portion of the newly procured solid standard that has been ball-milled in the laboratory to a powder. The LCS solid-new recovery of 37% is significantly below the QC flagging criterion. And at 75 μg/g, the result is slightly outside the vendor's advisory range of 80 to 322 μg/g. Previous analyses of this ball-milled material have yielded widely varying recoveries. Since very small sample sizes are analyzed, the larger mesh size and the inconsistency of the mill powder is the most likely cause of the poor recoveries for this LCS solid-new material.

The PBs for both the supernatant and wet centrifuged solids analyses meet the QA Plan's acceptance criteria of ≤EQL or ≤5% of sample concentration; the EQL being establish by multiplying the lowest calibration standard (i.e., 0.01 μg/mL) times the appropriate processing dilution factors. However, the RSDs for the supernatant (i.e., 58%) and wet centrifuged solids (i.e., 82%) do not meet the QC success criteria of <15%. When the releasing agent was added to the samples, a vigorous reaction occurred. In other samples where this reaction has been observed, poor yields were often the result, and it was assumed that cyanide was being volatilized and lost prior to placing the distillation tube on the sample tube. To minimize this effect, the releasing agent was pipetted into small vials, which were then added to the sample tubes. The small vials floated on the surface of the sample while the distillation tube was capped onto the sample tube. The distillation assembly was then inverted to mix the releasing agent into the sample. However, the reaction was so vigorous that the cyanide may have been released too rapidly for the trapping solution to capture all of the cyanide or was lost through leaking created by over-pressurization. In addition, when the distillation assembly was being inverted for mixing, there were some areas in the cross section of the distillation tube where the off-gases, for a brief time, did not contact the trapping solution. For samples that react vigorously with the releasing agent, additional investigations and/or method modification are warranted.

For the supernatant, the MS recovery averages 94%, with both the MS recovery (87%) and MSD recovery (101%) meeting the QC flagging criterion of 75% to 125%. However, for the wet centrifuged solids, the MS recovery averages only 15%, with both the MS recovery (27%) and MSD recovery (3%) being significantly below the QC flagging criterion. The poor MS recovery of the wet centrifuged solids is most likely due to either sample heterogeneity or the same cause as the RSD failures; i.e., vigorous reactions resulting in loss of cyanide.

9.8 Mercury (Hg) Analysis

Table 8.2, Table 8.4, Table 9.2, and Table 9.4. Appendix F8

The AZ-101 supernatant and wet centrifuged solids samples and associated batch QC samples were digested for Hg analysis per procedure RPG-CMC-131, *Mercury Digestion*, and analyzed by cold vapor atomic absorption (CVAA) spectroscopy for inorganic mercury according to procedure RPG-CMC-201, *Mercury Analysis*. Due to the dose levels, the wet centrifuged solids samples were digested in the SAL; the supernatant samples were digested at the mercury workstation. Quality control for the Hg analysis consisted of sample triplicates, preparation blanks, MSs, LCS (BS for supernatant and solids LCS for wet centrifuged solids), and calibration verification check standards and blanks.

The liquid LCS (NIST SRM-1641d) for the supernatant and the solids LCS (NIST SRM 2709) for the wet centrifuged solids analyses meet the required QC flagging criteria. Also, the PBs for both the supernatant and wet centrifuged solids analyses meet the QA Plan's acceptance criteria of ≤EQL or ≤5% of sample concentration. However, the RSD for both the supernatant (40% RSD) and wet centrifuged solids (21% RSD), as well as the MS and MSD recoveries for the wet centrifuged solids (131% and 175%, respectively), failed to meet the QC flagging criterion. Based on the excellent results from all the other QC samples and since the typical precision and accuracy is ±15% (2-σ) or better for non-complex aqueous samples that are free of interference, the failure of the supernatant and wet centrifuged solids to meet the RSD (i.e., precision) criterion and the failure of the wet centrifuged solids to meet the MS recovery (i.e., accuracy) criterion is considered to be a matrix effect.

The supernatant demonstrated a vigorous reaction during the addition of the nitric acid, the first of the digestion reagents. Although the oxidizing strength of the supernatant digests was maintained, this vigorous reaction may have caused inconsistent loss of mercury. It should be noted that the average supernatant mercury concentration is 15 times lower than the MRQ. The reason for the wet centrifuged solids RSD and MS failure is unknown. However, three factors may have contributed to the failures: 1) an inconsistent loss of mercury may be attributed to a slight foaming reaction (similar to the supernatant, but less vigorous), 2) the oxidizing strength of the digests may not have been maintained during the digestion of each sample (i.e., color consistency of the digest is difficult to determine through the SAL hot-cell leaded windows), and 3) the wet centrifuged solids may not be homogeneous relative to the small sample quantities used for analysis. The wet centrifuged solids contained an average concentration of mercury approximately two times the MRQ.

9.9 Hydroxide (OH) Titration

Table 8.2 and Table 9.2. Appendix F9

The AZ-101 supernatant was analyzed in triplicate for free hydroxide content following procedure PNL-ALO-228, *Determination of Hydroxyl and Alkalinity of Aqueous Solutions, Leachates & Supernates*. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N sodium hydroxide solution was prepared for use as a standard and spiking solution. The titrant was 0.2040 M hydrochloric acid. Triplicate results gave an average hydroxide molarity of 0.67 with a 1% RSD for the triplicate measurements. This result is equivalent to 11,400 µg/mL, well below the MRQ of 75,000 µg/mL. The LCS/BS recovery of 93% is well within the QC flagging criterion. Although not required by the test plan, a MS was prepared and analyzed; the MS recovered at 94%. No hydroxide was detected in the SAL hot cell DIW PB.

For information only – the second and third inflection points were detected in the samples at an average of 0.88 molar (2% RSD) and 0.70 molar (2% RPD), respectively. The second inflection point is primarily from both carbonate and aluminate and the third inflection point from carbonate (i.e., second equivalent point). Weak acids such as acetate, oxalate, formate, citrate, etc. also contribute to these inflection points, but are too low in concentration relative to the carbonate and aluminate to be detected separately. Based on these inflection points, the upper bound (i.e., inflection points due solely to carbonate) for TIC is approximately 14,000 µg/mL; the hot persulfate carbon method produced an average TIC result of approximately 10,000 µg/mL.

9.10 Ammonia Analysis

Table 8.2 and Table 9.2. Appendix F12

Triplicate samples of the AZ-101 supernatant were diluted with water (to reduce dose) in the SAL and preserved by acidifying with sulfuric acid. The preserved samples were stirred to allow carbon dioxide to evolve, then transferred to clean vials and removed from the SAL. (The supernatant sample is high in carbonate and evolves carbon dioxide upon acidification.) The supernatant samples, PB, MS, and LCS/BS were transferred to the ammonia analytical workstation. The samples were analyzed for ammonia by ion selective electrode (ISE) procedure RPG-CMC-226, *Measurement of Ammonia in Aqueous Solutions*. The ISE was calibrated over the range of 1E-01 molar to 1E-05 molar ammonia.

The 28% RSD of the triplicate samples for the supernatant exceeds the QC flagging criterion of <15%. However, the ammonia concentration is J-flagged and is below the linear range of the ISE, leading to very high uncertainty. The average ammonia result is only about 2% of the MRQ; thus the high RSD is not considered significant. Ammonia was not detected in the PB. The LCS/BS and MS recoveries at 95% and 85%, respectively, meet the QC flagging criteria.

9.11 Organic Acids

Table 8.2 and Table 9.2. Appendix F11

Triplicate samples of the AZ-101 supernatant were sub-sampled in the SAL and subjected to an ion exchange procedure to reduce the sample dose, such that the resulting samples could be analyzed in the 329 Facility organic IC workstation. Following the ion exchange dose reduction in the SAL which diluted the samples about 25 fold, the samples were further diluted from 63 fold to 125 fold at the IC workstation, then analyzed for the organic acids glycolate, acetate, formate, oxalate, and citrate by IC procedure TP-RPP-WTP-046, *Method for the Analysis and Quantification of Organic Acids in Simulated and Actual Hanford Tank Waste by Ion Chromatography*. A PB, LCS/BS, MS, and MSD were also prepared for analysis. The LCS/BS was subjected to the ion exchange process to provide assurances that the ion exchange processing did not impact the analytes of interest. The MS and MSD were prepared following the ion exchange processing. All measured organic acids meet the <15% RSD QC flagging criterion defined in the test plan. No organic acids were detected in the SAL PB above the MDL, which is estimated at about 30% of the lowest calibration standard adjusted for process dilution factors. The LCS/BS recoveries and MS/MSD recoveries for the organic acids meet the QC flagging criteria of 80% to 120% and 75% to 125%, respectively, for all organic acids measured.

The organic acids analyses were accomplished by using different columns to provide separation and quantitation for the anions specified in the test plan, except for gluconate and glycolate. A Dionex™ AS-15 column was used for separation of glycolate and acetate, and a Dionex™ AS-11 column used for the analysis of formate, oxalate, and citrate (glycolate and acetate co-elute on AS-11 column). This multiple column approach provides good analyses for acetate, formate, oxalate, and citrate. However, glycolate and gluconate co-elute on the AS-15 column and gluconate elutes very close to the unretained volume of the AS-11 column and can not be resolved from early eluting anions (e.g., fluoride), making both gluconate and glycolate analyses difficult.

Ion chromatography with conductivity detection is not well suited for analysis of gluconate in the presence of glycolate (or vice versa), due to low relative response and lack of retention and resolution from other anions. Alternate detection methods that provide better response are available; however, this provides little benefit since current IC columns suffer from either poor retention of gluconate or significant overlap/co-elution with other anions. With the current IC methods available, gluconate can be measured using the either the AS-11 and AS-15 column but only in the known absence of either fluoride or glycolate, respectively. For the AS-15 column, the MDL stated for glycolate is based on a calibration using glycolate standards; a MDL for the co-eluting gluconate is estimated at 450 µg/mL, since the detector response factor for gluconate is approximately 40% that of glycolate.

9.12 Chelator Analysis and Degradation Products

Table 8.2 and Table 9.2. Appendix F10

The analysis of the AZ-101 supernatant samples for chelators and chelator-degradation products was performed on a best-effort basis. The chelators, ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl) ethylenediaminetriacetic acid (HEDTA), ethylenediaminetriacetic acid (ED3A), iminodiacetic acid (IDA), succinic acid, and nitrilotriacetic acid (NTA) have low volatility and high polarity precluding direct analysis by GC/FID. Derivatizing the chelators with a boron trifluoride/methanol mixture produces a methyl ester that is amenable to GC/FID separation and analysis. The derivatization process and analysis are still considered experimental; i.e., the method has not been fully qualified for complex matrices such as Hanford tank waste. Additional work is required to provide a reliable, robust technique for the analysis of chelators in tank waste.

Triplicate 5-mL (nominal) sub-samples of the AZ-101 supernatant were diluted with 5-mL of DIW and subjected to an ion exchange procedure, TPR-RPP-WTP-049, *Ion Exchange for Activity Reduction*, to reduce the sample dose. Besides the samples a PB, LCS/BS, MS, and MSD were prepared for analysis and subjected to the ion exchange process to provide assurances that the ion exchange processing did not impact the analytes of interest. Following ion exchange, the samples were transferred to the 329 Facility organic analysis workstation for analysis according to procedure TP-RPP-WTP-048, *Derivatization GC/FID Analysis of Chelators and Degradation Products*. Adipic acid was added to 2-mL aliquots of each sample (prior to the derivatization step) as a derivatization monitor. An LCS/BS was prepared by spiking DIW with citric acid, succinic acid, EDTA, NTA and HEDTA. The MS and MSD for the analytical batch were prepared by spiking an AP-104 tank waste sample from ASR 6378 (prepared and analyzed in the same batch as the AZ-101 supernatant samples) with all analytes of interest except ED3A. Since no standard is available for ED3A, no LCS/BS or MSs was prepared and the reported results are based on the EDTA calibration.

Only succinic acid was detected (albeit at a very low concentration) above the EQL. The sample EQL is defined as the lowest calibration standard adjusted for any preparative and analysis dilutions. The MDL is set as one-tenth the EQL. The LCS/BS recovery met the QC flagging criterion with the exception of HEDTA, which was significantly high (170%). Of the seven MS analytes, the MS recovery for EDTA and HEDTA exceeded the QC flagging criterion but the MSD was acceptable. For the IDA MS/MSD recovery, it is assumed that all of the IDA spiked into the sample is converted to NIDA (the measured compound) in the presence of nitrite found in the tank waste.

Because of the failures observed with the MS/MSD and LCS/BS data, the QC samples were reprepared in the SAL. A different analyst performed the derivatization of the samples to confirm or refute original observed results. The reprepared data confirmed the trends observed in the original results; with the MS EDTA producing a low recoveries and the LCS/BS HEDTA producing high recoveries. One difference between the original and the confirmation results is the HEDTA recovery result for the MS and MSD. Originally, the recoveries for the MS/MSD were 70% and 103% respectively; the confirmation results produced slightly higher results for the MS/MSD (135% and 155%, respectively). The QC samples from both runs are included in Table 9.2.

9.13 Organic Phosphates Analysis

Following extraction of the AZ-101 supernatant in the SAL for the organic phosphate analysis, the resulting extracts (both methylene chloride and butanol) were transferred to the Building 329 Facility for analysis. The butanol extracts were processed in case the recoveries demonstrated from the methylene chloride were very poor but were not analyzed.

A five-point calibration curve was constructed for both diphenylphosphate (DPP - surrogate compound and D2EHP). The MDL for D2EHP was based on the concentration of the lowest calibration standard adjusted for the sample volume extracted (about 5 mL). The supernatant samples and QC samples were analyzed per test plan TP-RPP-WTP-047, *Identification and Quantification of D2EHP in Tank Wastes*. This method describes a derivatization technique with diazomethane. The products are then measured using GC/FID.

The sample and QC data obtained from this method were unacceptable and no results are reported. Similar unacceptable results were obtained for supernatant samples from Tank 241-AP-104. The problems encountered are potentially due to critical pH adjustment, poor extraction, and/or incomplete and variable derivatization, and they are compounded by the necessity of performing the pH adjustment and extraction operations in a remote handling facility (i.e., SAL). Following the failure of this method to reliably analyze for D2EHP, this analyte was deleted from the analyte list by BNI and no further work was undertaken to evaluate the failure.

Table 9.1. AZ-101 As-Received Supernatant – Radionuclide QC Results

QC Flagging Criterion >>							<15%	80% - 120%	75% - 125%
Method	**Radionuclide**	**MRQ µCi/mL**	**MDA/ MDL µCi/mL**	**MRQ > 3xMDL**	**Average µCi/mL**	**DF**	**RSD(g) %**	**Blank Spike (LCS) %Rec**	**Matrix Spike (MS) %Rec**
H-3	^{3}H	2.1E-02	2E-05	Yes	1.81E-02		1	92	**57** (f,h)
C-14	^{14}C	7.2E-04	4E-05	Yes	1.93E-03		4	101	112
GEA	**^{60}Co**	2.1E-03	4E-02	No	4E-02	U			
Se-79	^{79}Se	9.0E-05	2E-05	Yes	9.69E-04		**17**		
Sr-90	**^{90}Sr** (i)	1.5E-01	1E-03	Yes	5.01E-02		5	94 (c)	95 (h)
ICP-MS	**^{99}Tc** (j)	1.5E-03	2E-04	Yes	3.27E-01		2	105	106
Tc-99	^{99}Tc^{+7}	1.5E-03	6E-04	No	3.77E-01		1	95	94 (b)
GEA	^{126}Sn	6.0E-03	2E+00	No	2E+00	U			
ICP-MS	^{129}I	1.8E-05	4E-07	Yes	1.03E-06	J	4	90	97 (b)
ICP-MS	^{135}Cs - MS	1.5E+00	2E-05	Yes	1.06E-02		3	(e)	(e)
ICP-MS	^{137}Cs - MS	1.5E+00	1E+00	No	1.40E+03		2	(e)	(e)
GEA	**^{137}Cs**	9.0E+00	7E-01	Yes	1.64E+03		1		
GEA	**^{154}Eu**	2.0E-03	2E-01	No	2E-01	U			
GEA	^{155}Eu	9.0E-02	2E+00	No	2E+00	U			
GEA	^{231}Pa	7.9E-05	2E+00	No	2E+00	U			
ICP-MS	^{233}U	4.2E-04	9E-06	Yes	9E-06	U		(d)	(d)
ICP-MS	^{234}U	1.2E-04	6E-06	Yes	6E-06	U		(d)	(d)
ICP-MS	^{235}U	4.5E-08	3E-09	Yes	4.73E-08	B	1	(d)	(d)
ICP-MS	^{236}U	1.4E-06	6E-08	Yes	9.32E-08	J	3	(d)	(d)
ICP-MS	^{238}U	7.2E-09	2E-09	Yes	9.38E-07	B	2	(d)	(d)
ICP-MS	**^{237}Np**(k)	2.7E-02	7E-06	Yes	3.77E-05	J	5	101	101 (b)
AEA	^{238}Pu	1.0E-02	2E-05	Yes	3.44E-04	B	9		
AEA	^{239,240}Pu	3.0E-02	2E-05	Yes	1.81E-03		2	102 (h)	112 (h)
ICP-MS	^{239}Pu(k)	3.0E-02	6E-04	Yes	2.00E-03	JB	25	102	99 (b)
ICP-MS	^{240}Pu(k)	1.0E-02	2E-03	Yes	2E-03	U		101	99 (b)
AEA	^{241}Am	3.0E-02	2E-05	Yes	1.05E-04	B	**35**	91 (h)	86 (h)
GEA	^{241}Am - GEA	3.0E-02	2E+00	No	2E+00	U			
AEA	^{242}Cm	1.5E-01	9E-06	Yes	9E-06	U			
AEA	^{243,244}Cm	1.5E-02	2E-05	Yes	4.18E-05	JB			
Alpha	Gross Alpha (a)	2.3E-01	5E-03	Yes	5E-03	U		104	103
	Alpha Sum				2.28E-03				
	TRU				2.32E-03				

Outlined and bolded results exceed QC flagging criteria

Blank fields indicate QC not required or not defined in the test plan or test specification

Bolded radionuclides required for comparison to Contract Specification 7 (Envelope B)

Alpha Sum: Summation of AEA results only (^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm)

TRU: Z>92, alpha emitter, half-life >10yr (^{237}Np, ^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm); Pu, Am, and Cm from AES and Np from ICP-MS

Table 9.1. (Cont'd)

QC Flagging Criterion >>							<15%	80% - 120%	75% - 125%
Method	Radionuclide	MRQ µCi/mL	MDA/ MDL µCi/mL	MRQ > 3xMDL	Average µCi/mL	DF	RSD[g] %	Blank Spike (LCS) %Rec	Matrix Spike (MS) %Rec

MRQ: minimum reportable quantity

MDA: minimum detectable activity (used with all radiochemical analysis results)

MDL: method detection limit (used with ICP-MS results)

DF: data quality flag (for definition of flags used see Section 8.1)

RSD: relative standard deviation (in percent)

(a) LCS and MS QC flagging criteria 70% to 130%.

(b) MS QC flagging criterion 70% to 130%.

(c) LCS QC flagging criterion 75% to 125%.

(d) For U LCS and MS performance, see ICP-MS ^{238}U in Table 9.2.

(e) ^{135}Cs-MS and ^{137}Cs-MS calculated from HPIC/ICP-MS isotopic data and ICP-MS ^{133}Cs result; For Cs performance ^{133}Cs Table 9.2

(f) Spike concentration significantly less than 20% of sample concentration; high uncertainty.

(g) RSD only calculated if results >MDA/MDL for sample, duplicate, and triplicate.

(h) Not required by test plan or test specification; performed as part of laboratory QC.

(i) Although both the batch QC (i.e., LCS, MS, PB) and analysis QC (i.e., reference counting standards) produced excellent results, the supernatant ^{90}Sr result is significantly lower than other published results; see discussion Section 8.3 and Section 9.4.4.

(j) Uncertainty estimated at ±30%; see Section 8.3 for further details.

(k) Same certified source standard used to prepare calibration and verification standards for ICP-MS. Calibration and verification standards prepared approximately 1 year apart; prepared standards verified by independent analysis (i.e., LSC, AEA, or GEA).

Nominal decay correction reference dates:
ICP-MS: U (3/2002), Pu/Np (4/2002), Tc (2/2002) and I (5/2003)
Radchem: GEA, Gross Alpha, AEA Pu and Am/Cm (10/2001); ^{90}Sr (9/2002); ^{99}Tc (8/2002); ^{79}Se (11/2001); ^{3}H (2/2002); and ^{14}C (6/2002)

Table 9.2. AZ-101 As-Received Supernatant – Analyte QC Results

						QC Flagging Criteria >>	(e) <15%	80% - 120%	75% - 125%	75% - 125%	75%- 125%	< ±10%
Method	**Analyte**	**MRQ µg/mL**	**MDL µg/mL**	**MRQ >3xMDL**	**Average Result µg/mL**	**DF**	**RSD %**	**Blank Spike (LCS) %Rec**	**Matrix Spike (MS) %Rec**	**Matrix Spike Dup (MSD) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
IC-Org	Acetate (r)	1,500	140	Yes	140	U		102	82	87		
ICP-AES	Ag (r)		0.6		0.6	U		102	101			
ICP-AES	**Al**	75	1.5	Yes	6,050		1	97	(i)			2.2
ISE	Ammonia	140	1.5	Yes	2.3	J	28	95	85			
ICP-MS	AMU-241(q)	0.0087	0.0011	Yes	0.0012	J		92 (a)	NM		91 (d)	
ICP-AES	As (r)		6.4		9.9	J	2					
ICP-AES	B	2.3	1.3	No	77	BX	13	109	**136**			6.3
ICP-AES	**Ba**	2.3	0.26	Yes	0.3	J		98	92			
ICP-AES	Be (r)		0.3		0.3	U		95	97			
ICP-AES	Bi (r)		2.6		2.6	U		98	(j)			
IC-Inorg	Br	300	130	No	650		4	98	98			
C (HP)	C as TC (q,r)		61		10,400							
C (Furn)	C as TC (r)		89		7,350		2	97	88			
C (HP)	**C as TIC**	150	34	Yes	9,850		1	100	105			
C (Furn)	**C as TIC** (q)	150	170	No	5,830		9	(b)	(b)			
C (HP)	**C as TOC**	1,500	87	Yes	510		9	97	103			
C (Furn)	**C as TOC**	150	250	No	1,500		**21**	102	81			
ICP-AES	**Ca**	150	6.4	Yes	9.3	J		100	95			
ICP-AES	**Cd**	2.3	0.39	Yes	0.8	JB	10	97	98			
ICP-AES	Ce	7.5	5.2	No	5.2	U		95	(j)			
IC-Org	Citrate		410		410	U		95	86	82		
GC/FID	Citric acid (r)	1,500	5.8	Yes	5.8	U		88/93 (o)	92/82 (o)	88/83 (o)		
IC-Inorg	**Cl**	300	130	No	240		13	95	96			
CN	CN	3.0	0.055	Yes	2.04	B	**58**	105	87	101		
ICP-AES	Co		1.3		1.3	U						

Table 9.2. (Cont'd)

						QC Flagging Criteria >>	(e) <15%	80% - 120%	75% - 125%	75% - 125%	75%- 125%	< ±10%
Method	**Analyte**	**MRQ µg/mL**	**MDL µg/mL**	**MRQ >3xMDL**	**Average Result µg/mL**	**DF**	**RSD %**	**Blank Spike (LCS) %Rec**	**Matrix Spike (MS) %Rec**	**Matrix Spike Dup (MSD) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
ICP-AES	**Cr** (r)	15	0.52	Yes	686		0.4	102	(i)			4.7
ICP-MS	^{133}Cs – MS (h)	0.00070	0.016	No	26.5		2	(h)	(h)			
ICP-MS	^{135}Cs – MS (h)		0.013	No	8.8		3	(h)	(h)			
ICP-MS	^{137}Cs – MS (h)		0.014	No	16.1		2	(h)	(h)			
ICP-MS	^{133}Cs	0.00070	0.0090	No	26.5		1	107	98 (d)		111 (d)	0.8
ICP-AES	Cu (r)		0.6		0.6	U		106	**66**			
None	D2EHP (p)	1,500										
ICP-AES	Dy (r)		1.3		1.3	U						
GC/FID	ED3A	1,500	4.9	Yes	4.9	U		(m)	(m)	(m)		
GC/FID	EDTA	1,500	4.9	Yes	4.9	U		103/111 (o)	**61/65** (o)	85/**74** (o)		
ICP-AES	Eu (r)		2.6		2.6	U						
IC-Inorg	**F**	150	130	No	2,010		2	95	96			
ICP-AES	**Fe**	150	0.64	Yes	1.5	JB	35	107	94			
IC-Org	Formate	1,500	180	Yes	370	J		104	97	82		
IC-Org	Gluconate (t)	1,500	450	Yes	450	U						
IC-Org	Glycolate (t)	1,500	170	Yes	170	U		109	95	84		
GC/FID	HEDTA	1,500	8.8	Yes	8.8	UX		**170/221** (o)	**70/135** (o)	103/**155** (o)		
CVAA	**Hg**	1.5	0.00013	Yes	0.055		**40**	88	104	105	100	
ICP-MS	^{127}I	1.5	0.007	Yes	0.007	U		(d,f)	(d,f)			
GC/FID	IDA	1,500	5.4	Yes	5.4	U		(n)	121/110 (o)	**131**/111 (o)		
ICP-AES	**K**	75	52	No	4,460		1	97	80			3.9
ICP-AES	**La**	35	1.3	Yes	1.3	U		96	96			
ICP-AES	Li	2.3	0.77	No	0.89	J	1	102	94			
ICP-AES	Mg	300	2.6	Yes	2.6	U		103	102			
ICP-AES	Mn (r)		1.3		1.3	U		101	97			
ICP-AES	Mo (r)		1.3		88.8		0.5	98	91			4.8
ICP-AES	Na	75	19	Yes	111,000		1 (k)	**121** (k)	(i, k)			4.6

Table 9.2. (Cont'd)

						QC Flagging Criteria >>	(e) <15%	80% - 120%	75% - 125%	75% - 125%	75%- 125%	< ±10%
Method	**Analyte**	**MRQ µg/mL**	**MDL µg/mL**	**MRQ >3xMDL**	**Average Result µg/mL**	**DF**	**RSD %**	**Blank Spike (LCS) %Rec**	**Matrix Spike (MS) %Rec**	**Matrix Spike Dup (MSD) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
ICP-AES	Nd (r)		2.6		2.6	U		96	95			
ICP-AES	**Ni**	30	0.77	Yes	1.0	JB	19	101	97			
IC-Inorg	**NO$_2$**	3,000	2,500	No	61,300		1	99	102			
IC-Inorg	**NO$_3$**	3,000	2,500	No	52,600		1	92	95			
GC/FID	NTA	1,500	5.6	Yes	5.6	U		111/118 (o)	78/80 (o)	81/89 (o)		
Titration	OH	75,000	170	Yes	11,400		1	93	94 (s)			
IC-Inorg	Oxalate	1,500	250	Yes	1,000		6	101	101			
IC-Org	Oxalate	1,500	210	Yes	1,000		5	103	97	75		
ICP-AES	P	600	2.6	Yes	504		1	97	(i)			0.4
ICP-AES	**Pb**	300	2.6	Yes	3.3	J	2	102	100			
ICP-AES	Pd (r)		19		20	J	0.1					
IC-Inorg	**PO$_4$**	7,700	250	Yes	1,600		1	95	95			
ICP-MS	Rb	1.0	0.025	Yes	9.20		6	103	128 (d)		95 (d)	5.3
ICP-AES	Rh (r)		7.7		7.7	U						
ICP-AES	Ru (r)		28		28	U						
ICP-AES	Sb (r)		13		13	U						
ICP-AES	Se (r)		6.4		6.4	U						
ICP-AES	Si (r)		13		206	BX	7	$\boxed{\mathbf{122}}$	$\boxed{\mathbf{132}}$			4.1
ICP-AES	Sn (r)		39		47	J	1					
IC-Inorg	**SO$_4$**	7,500	250	Yes	16,200		2	95	96			
ICP-AES	Sr (r)		0.4		0.4	U		113	$\boxed{(j)}$			
GC/FID	Succinic acid	1,500	50	Yes	50	J	6	97/104 (o)	90/94 (o)	91/96 (o)		
ICP-AES	Te (r)		39		39	U						
ICP-AES	Th	2.3	26	No	26	U		99	$\boxed{(j)}$			
ICP-AES	Ti (r)		0.6		0.6	U		95	92			
ICP-AES	Tl (r)		13		13	U						
ICP-AES	**U**	600	52	Yes	52	U		96	90			

Table 9.2. (Cont'd)

						QC Flagging Criteria >>	(e) <15%	80% - 120%	75% - 125%	75% - 125%	75%- 125%	< ±10%
Method	**Analyte**	**MRQ µg/mL**	**MDL µg/mL**	**MRQ >3xMDL**	**Average Result µg/mL**	**DF**	**RSD %**	**Blank Spike (LCS) %Rec**	**Matrix Spike (MS) %Rec**	**Matrix Spike Dup (MSD) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
KPA	**U**	780	0.0004	Yes	0.52		2	103	98			
ICP-MS	**U** (g,r)	600	0.0046	Yes	2.81	B	2	105	106 (d)		101 (d)	0.8
ICP-AES	V	2.3	1.3	No	1.5	J	0.0	94	91			
ICP-AES	W	2.3	52	No	58	J	0.0	**(j)**	**(j)**			
ICP-AES	Y (r)		1.3		1.3	U						
ICP-AES	Zn (r)		1.3		1.3	U		103	97			
ICP-AES	Zr (r)		1.3		1.3	U		104	101			

Outlined and bolded results exceed QC flagging criteria

Blank fields indicate QC not required or not defined in the test plan or test specification.

Bolded analytes required for comparison to Contract Specification 7 (Envelope B)

MRQ: minimum reportable quantity

MDL: method detection limit

DF: data quality flag (See Section 8.1 for definitions)

RSD: relative standard deviation (in percent)

D2EHP: bis-(2-ethylhexyl) phosphate

EDTA: ethylenediaminetetraacetic acid

ED3A: ethylenediaminetriacetic acid

HEDTA: N-(2-hydroxyethyl) ethylenediaminetriacetic acid

IDA: iminodiacetic acid

NTA: nitrilotriacetic acid

(a) AMU-241: ^{241}Am and/or ^{241}Pu; ICP-MS calibrated using ^{241}Am; post spiked as ^{241}Am.

(b) Not applicable, TIC by difference (TC-TOC); see TC (Furn) for QC performance.

(c) Not applicable, TC by summation (TIC+TOC); see TIC (HP) and TOC (HP) for QC performance.

(d) MS/PS QC flagging criteria 70% to 130%.

(e) RSD only calculated if sample, duplicate, and triplicate >MDL.

(f) For QC performance for uranium by ICP-MS, see ^{129}I Table 9.1.

(g) U is the sum of all measured isotopes of U by ICP-MS.

(h) ^{133}Cs-MS, ^{135}Cs-MS, and ^{137}Cs-MS calculated from HPIC/IC-MS isotopic data and ICP-MS ^{133}Cs result; see ^{133}Cs for LCS and MS performance.

Table 9.2. (Cont'd)

QC Flagging Criteria >>							(e) <15%	80% - 120%	75% - 125%	75% - 125%	75%- 125%	< ±10%
Method	Analyte	MRQ µg/mL	MDL µg/mL	MRQ >3xMDL	Average Result µg/mL	DF	RSD %	Blank Spike (LCS) %Rec	Matrix Spike (MS) %Rec	Matrix Spike Dup (MSD) %Rec	Post Spike %Rec	Serial Dilution %Diff

(i) Sodium MS concentration <20% of sample concentration; serial dilution used to evaluate matrix interferences.

(j) Analyte not included in LCS or MS, or diluted below EQL and no PS performed.

(k) Sodium RSD requirement <3.5% and LCS/MS requirement 90% to 110%.

(m) ED3A not added to LCS or MS; unavailability of standards.

(n) IDA not added to LCS; LCS matrix does not convert IDA to NIDA for measurement.

(o) Second set of analyses performed to confirm low EDTA and high HEDTA; both sets of LCS and MS reported.

(p) Analytes not measured due to lack of reliable method.

(q) TC is sum of TIC and TOC by HP; TIC is difference between measured TC Furn and TOC Furn.

(r) Opportunistic analytes; not include in test specification or test plan.

(s) Not required by test plan or test specification; performed as part of laboratory QC.

(t) Glycolate and gluconate results should be considered the upper bound concentration, since glycolate and gluconate are not resolved by the IC measurement method used for the analysis. IC system calibrated using glycolate; gluconate estimate based on gluconate-to-glycolate response factor. Each result assumes 100% of response due to each analyte

Table 9.3. AZ-101 As-Received Wet Centrifuged Solids – Radionuclide QC Results

QC Flagging Criteria >>							<15%	80% - 120%	80% - 120%	70%-130%	70%-130%	< +/-10%
Method	**Analyte (l)**	**MRQ µCi/g**	**MDA/ MDL µCi/g**	**MRQ > 3xMDL**	**Average µCi/g**	**DF**	**RSD(b) %**	**Blank Spike (LCS) %Rec**	**SRM-2710 (LCS) (h) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Rec**
H-3	**^{3}H**	1.5E-02	4E-04	Yes	7.08E-03		1	91		90 (m)		
C-14	**^{14}C**	1.8E-03	2E-04	Yes	1.04E-03	B	**53**	94		100 (k)		
GEA	**^{60}Co**	1.2E-02	3E-01	No	2.03E+00		3					
Sr-90	**^{90}Sr**	7.0E+01	4E+02	No	2.02E+04		5	111 (i)		107 (m)		
GEA	**^{125}Sb**	6.0E+00	5E+00	No	8.07E+00	J						
GEA	**^{126}Sn**	6.0E-02	2E+00	No	2E+00	U						
ICP-MS	**^{129}I**	1.80E-05	8E-06	No	1.09E-05	J	27	82		83		
GEA	^{134}Cs	9.0E-01	6E-01	No	6E-01	U						
ICP-MS	^{135}Cs (a)	5.30E-03	1E-06	Yes	6.63E-03		2	(e)		(e)	(e)	(e)
GEA	**^{137}Cs**	6.0E-02	2E+00	No	7.52E+02		1					
ICP-MS	**^{137}Cs** (a)	9.00E-02	2E-01	No	8.76E+02		2	(e)		(e)	(e)	(e)
ICP-MS	^{151}AMU (Sm)	TBD	1E-02	--	5.40E+02		2	105 (f)	**124**		105	1
GEA	**^{152}Eu**	6.0E-02	1E+00	No	1E+00	U						
GEA	**^{154}Eu**	6.0E-02	9E-01	No	2.34E+01		4					
GEA	**^{155}Eu**	6.0E-02	7E+00	No	3.06E+01		15					
ICP-MS	^{234}U	3.70E-03	7E-06	Yes	1.78E-03		4	(c)		(c)	(c)	(c)
AEA	^{236}Pu		NA		3E-02	U						
ICP-MS	^{236}U	3.80E-04	1E-07	Yes	1.61E-04		6	(c)		(c)	(c)	(c)
AEA	**^{238}Pu**	6.0E-02	3E-02	No	3.06E-01		6					
ICP-MS	^{238}U	2.00E-06	4E-08	Yes	1.24E-03		3	**124**/102 (g)	109	(d)	106	9
AEA	**^{239,240}Pu**	6.0E+00	3E-02	Yes	2.41E+00		3	102 (m)		103 (m)		
AEA	**^{241}Am**	1.8E-02	4E-02	No	3.75E+01		5	91 (m)		89 (m)		
GEA	**^{241}Am -GEA**	6.0E+00	1E+01	No	4.49E+01		**30**					
Pu-241	**^{241}Pu**	1.2E+00	2E-03	Yes	1.15E+01		3	113 (m)		114 (m)		
AEA	^{242}Cm	1.2E-02	3E-02	No	3.91E-02	J	7					
AEA	^{242}Pu	3.36E-01	9E-03	Yes	9E-03	U				98		
AEA	**^{243,244}Cm**	1.2E-01	3E-02	No	9.48E-02	J	26					

Table 9.3. (Cont'd)

QC Flagging Criteria >>							<15%	80% - 120%	80% - 120%	70%-130%	70%-130%	< +/-10%
Method	**Analyte [(l)]**	**MRQ µCi/g**	**MDA/ MDL µCi/g**	**MRQ > 3xMDL**	**Average µCi/g**	**DF**	**RSD[(b)] %**	**Blank Spike (LCS) %Rec**	**SRM-2710 (LCS) [(h)] %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Rec**
Alpha	Gross alpha	1.0E-03	9E+00	No	5.39E+01		2	104 [(j)]		108		
	Alpha Sum				4.04E+01							
	TRU				4.04E+01							

Outlined and bolded results exceed QC flagging criteria

Blank fields indicate QC not required or not defined in the test plan or test specification

Bolded radionuclides required for comparison to Contract Specification 8 (Envelope D)

Alpha Sum: Summation of AEA results only (^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm)

TRU: Z>92, alpha emitter, half-life >10Y (^{237}Np, ^{239,240}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and ^{243,244}Cm); Pu, Am, and Cm from AES and Np from ICP-MS

MRQ: minimum reportable quantity

MDA: minimum detectable activity (used with all radiochemical analysis results)

MDL: method detection limit (used with ICP-MS results)

DF: data quality flag (for definition of flags used see Section 8.1)

RSD: relative standard deviation (in percent)

NA: not applicable

TBD: to be determined

(a) Calculated using ^{133}Cs results and isotopic mass distribution from Supernatant analysis; see Table 8.2.

(b) RSD only calculated if results >MDA/MDL for sample, duplicate, and triplicate.

(c) For QC performance for uranium by ICP-MS see ^{238}U.

(d) MS not recovered; sample concentration significantly greater than spike concentration or spiking level too low after required analytical dilution.

(e) For cesium QC performance, see ^{133}Cs Table 9.4.

(f) Post spiked Blank used as LCS/BS for analyte.

(g) Results of both the LCS/BS (first results) and post spiked LCS/BS (second result).

(h) Solids LCS NIST 2710 for ICP-MS prepared by total digestion method and compared to certified results based on a leaching preparation; high recoveries for some analytes not unexpected.

(i) LCS recovery criterion 75% to 125%.

(j) LCS recovery criterion 70% to 130%.

(k) MS/PS recovery criterion 75% to 125%.

(l) Prep method PNL-ALO-115, except ^{3}H leached by PNL-ALO-103, ^{129}I by PNL-ALO-114, and ^{14}C by combustion.

(m) Not required by test plan or test specification; performed as part of laboratory QC.

Table 9.3. (Cont'd)

							<15%	80% - 120%	80% - 120%	70%-130%	70%-130%	< +/-10%
						QC Flagging Criteria >>	**<15%**	**80% - 120%**	**80% - 120%**	**70%-130%**	**70%-130%**	**< +/-10%**
Method	**Analyte [l]**	**MRQ µCi/g**	**MDA/ MDL µCi/g**	**MRQ > 3xMDL**	**Average µCi/g**	**DF**	**RSD[b] %**	**Blank Spike (LCS) %Rec**	**SRM-2710 (LCS) [h] %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Rec**

Nominal decay correction references dates

ICP-MS: ^{129}I (5/2003), all others (6/2002)

Radchem: GEA, AES Pu and Am/Cm, ^{90}Sr, gross alpha (10/2001); ^{3}H (2/2002); ^{14}C (3/2002); ^{241}Pu (6/2002), ^{242}Pu (6/2002)

Table 9.4. AZ-101 As-Received Wet Centrifuged Solids – Analyte QC Results

							QC Criteria >>	<15%	80-120% (i)	80-120% (i)	75-125% (h)	75-125% (h)	< +/-10%
Method	**Prep**	**Analyte**	**MRQ µg/g**	**MDL µg/g**	**MRQ > 3xMDL**	**Average µg/g**	**DF**	**RSD(g) %**	**Blank Spike (LCS) %Rec**	**Solid LCS (c,l) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
ICP-MS	Fusion-115	**^{99}Tc**	6.0	0.002	Yes	8.69		3	105 (j)			100	0
ICP-MS	Fusion-114	**^{127}I**	1.5	0.04	Yes	2.45		13	(y)		(y)		
ICP-MS	Fusion-115	**^{133}Cs**	0.00070	0.003	No	16.6		2	103	105	$\boxed{53}$	106	1
ICP-MS	Fusion-115	**^{233}U**	60	0.001	Yes	0.17		4	(v)	(v)		(v)	$\boxed{13}$
ICP-MS	Fusion-115	**^{235}U**	6.0	0.002	Yes	33.3		3	(v)	(v)		(v)	8
ICP-MS	Fusion-115	**^{237}Np**(z)	1.8	0.001	Yes	27.3		9	106 (j)			111	5
ICP-AES	Acid-129M2	**Ag**	900	15	Yes	55	J	10	94		95	98 (a)	
ICP-AES	Fusion-115	**Ag**	900	57	Yes	260	JX	25	$\boxed{142}$			100	
ICP-AES	Acid-129M2	**Al**	330	36	Yes	136,000		2	101	100	(b)	108 (a)	2
ICP-AES	Fusion-115	**Al**	330	140	No	130,000		3	93	96		105	2
ICP-AES	Acid-129M2	**As** (o)		150		150	U					103(a)	
ICP-AES	Fusion-115	**As** (o)		570		570	U					100	
ICP-MS	Fusion-115	**As**	3.0	0.1	Yes	46.1		6	96	94	$\boxed{23}$	103	$\boxed{45}$
ICP-AES	Acid-129M2	**B**	3.0	30	No	30	U		91			104 (a)	
ICP-AES	Fusion-115	**B**	3.0	120	No	110	U		100			105	
ICP-AES	Acid-129M2	**Ba**	60	5.9	Yes	489		5	101	99	92	101 (a)	2
ICP-AES	Fusion-115	**Ba**	60	23	No	450	B	3	91	93		101	
ICP-AES	Acid-129M2	**Be**	3.0	5.9	No	9	J	6	98		93	100 (a)	
ICP-AES	Fusion-115	**Be**	3.0	23	No	23	U		91			101	
ICP-AES	Acid-129M2	**Bi**	6,000	59	Yes	59	U		98			100 (a)	
ICP-AES	Fusion-115	**Bi**	6,000	230	Yes	230	UX		$\boxed{153}$			99	
IC-Inorg	Leach-103	Br (t)	450	130	Yes	350		2	98		(b)	94	
C (Furn)	Direct	C as TC (o,q)		570		9,670		8	97		104		
C (HP)	Direct	C as TC (o)		95		9,230		8			93		
C (Furn)	Direct	**C as TIC** (q)	30	1,000	No	1,100	J						

Table 9.4. (Cont'd)

						QC Criteria >>		<15%	80-120% (i)	80-120% (i)	75-125% (h)	75-125% (h)	< +/-10%
Method	**Prep**	**Analyte**	**MRQ µg/g**	**MDL µg/g**	**MRQ > 3xMDL**	**Average µg/g**	**DF**	**RSD(g) %**	**Blank Spike (LCS) %Rec**	**Solid LCS (c,l) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
C (HP)	Direct	**C as TIC**	30	54	No	8,380		7	101		96		
C (Furn)	Direct	**C as TOC** (x)	60	1,400	No	9,900		8	102		81		
C (HP)	Direct	**C as TOC** (x)	60	140	No	820		$\boxed{21}$	93		89		
ICP-AES	Acid-129M2	**Ca**	180	150	No	2,400		7	105	101	90	103 (a)	
ICP-AES	Fusion-115	**Ca**	180	570	No	2,300	JX	7	$\boxed{122}$	99		102	
ICP-AES	Acid-129M2	**Cd**	11	8.9	No	4,670		7	104		(b)	103 (a)	2.4
ICP-AES	Fusion-115	**Cd**	11	34	No	4,380		2	98			103	1.9
ICP-AES	Acid-129M2	**Ce** (o)		120		380	J	8	98			99 (a)	
ICP-AES	Fusion-115	**Ce** (o)		450		530	J		117			109	
ICP-MS	Fusion-115	**Ce**	6.0	0.04	Yes	297		2	88	101	$\boxed{11}$	102	2
IC-Inorg	Leach-103	**Cl** (t)	230	13	Yes	110		$\boxed{50}$	105		(b)	95	
CN	Distill-287	**CN**	3.0	0.040	Yes	0.54	X	$\boxed{82}$		91/37 (f)	$\boxed{15\ (e)}$		
ICP-AES	Acid-129M2	**Co** (o)		30		33	J					104 (a)	
ICP-AES	Fusion-115	**Co** (o)		120		110	U					109	
ICP-MS	Fusion-115	**Co**	3.0	0.006	Yes	38.3	B	$\boxed{19}$	98	$\boxed{144}$	75	101	$\boxed{11}$
ICP-AES	Acid-129M2	**Cr**	120	12	Yes	800		$\boxed{41}$	107		(b)	104 (a)	2.3
ICP-AES	Fusion-115	**Cr**	120	45	No	610		12	99			102	
ICP-AES	Acid-129M2	**Cu**	18	15	No	238		8	104	97	101	99 (a)	
ICP-AES	Fusion-115	**Cu**	18	57	No	57	U			88		104	
ICP-AES	Acid-129M2	Dy (o)		30		30	U					101 (a)	
ICP-AES	Fusion-115	Dy (o)		120		110	U					96	
ICP-AES	Acid-129M2	Eu (o)		59		59	U					108 (a)	
ICP-AES	Fusion-115	Eu (o)		230		230	U					103	
IC-Inorg	Leach-103	**F** (r,t)	7,500	130	Yes	4,110		8	104		(b)	99	
ICP-AES	Acid-129M2	**Fe**	140	15	Yes	66,800		5	109	100	(b)	104 (a)	3.2
ICP-AES	Fusion-115	**Fe**	140	57	No	61,600		5	107	98		105	2.5
CVAA	Acid-131	**Hg**	1.5	0.0079	Yes	3.2		$\boxed{21}$		101	$\boxed{155\ (d)}$	93	

Table 9.4. (Cont'd)

							QC Criteria >>	<15%	80-120% (i)	80-120% (i)	75-125% (h)	75-125% (h)	< +/-10%
Method	**Prep**	**Analyte**	**MRQ µg/g**	**MDL µg/g**	**MRQ > 3xMDL**	**Average µg/g**	**DF**	**RSD (g) %**	**Blank Spike (LCS) %Rec**	**Solid LCS (c,l) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
ICP-AES	Acid-129M2	**K**	200	1,200	No	1,400	J	21	97			102 (a)	
ICP-AES	Acid-129M2	**La**	60	30	No	1,790		6	100		(b)	97 (a)	1.1
ICP-AES	Fusion-115	**La**	60	120	No	1,580		2	97			96	
ICP-AES	Acid-129M2	**Li**	30	18	No	70	J	6	106		87	104 (a)	
ICP-AES	Fusion-115	**Li**	30	68	No	74	J	10	100			100	
ICP-AES	Acid-129M2	**Mg**	540	59	Yes	440	J	4	112	103	107	109 (a)	
ICP-AES	Fusion-115	**Mg**	540	230	No	530	J	10	107	106		109	
ICP-AES	Acid-129M2	**Mn**	300	30	Yes	1,480		5	105	103	(b)	110 (a)	2.4
ICP-AES	Fusion-115	**Mn**	300	120	No	1,480		6	120	103		106	
ICP-AES	Acid-129M2	**Mo** (o)		30		64	J	63	103		98	101 (a)	
ICP-AES	Fusion-115	**Mo** (o)		110		110	U		99			103	
ICP-MS	Fusion-115	**Mo**	30	0.03	Yes	146	B	1	106	$\boxed{178}$	$\boxed{53}$	101	2.0
ICP-AES	Acid-129M2	**Na**	150	89	No	70,100		$\boxed{5.8^{(m)}}$	103	101	(b)		7.4
ICP-AES	Fusion-115	**Na**	150	340	No	71,500		$\boxed{7.5^{(m)}}$	97	94		101	2.4
ICP-AES	Acid-129M2	**Nd**	600	59	Yes	1,260		6	99		95	98 (a)	
ICP-AES	Fusion-115	**Nd**	600	230	No	1,200	J	5	102			96	
ICP-AES	Acid-129M2	**Ni**	160	18	Yes	2,770		7	106		(b)	104 (a)	2.7
IC-Inorg	Leach-103	**NO_2** (t)	450	260	No	27,400		1	90		(b)	102	
IC-Inorg	Leach-103	**NO_3** (t)	450	260	No	22,900		1	104		(b)	98	
IC-Inorg	Leach-103	Oxalate (o,t)		26	Yes	3,150	X	$\boxed{29}$	$\boxed{0^{(n)}}$		(b)	100	
ICP-AES	Acid-129M2	**P**	600	59	Yes	1,790		6	103	90	100	101 (a)	
ICP-AES	Fusion-115	**P**	600	230	No	350	J	17	102			101	
ICP-AES	Acid-129M2	**Pb**	600	59	Yes	490	J	9	109	99	103	100 (a)	
ICP-AES	Fusion-115	**Pb**	600	230	No	510	J	10	108	97		104	
ICP-AES	Acid-129M2	**Pd** (o)		450		490	J	8				92 (a)	
ICP-AES	Fusion-115	**Pd** (o)		1,700		1,700	U					102	
ICP-MS	Fusion-116	**Pd**	300	0.003	Yes	101		$\boxed{47}$	84		(b)	104	3

Table 9.4. (Cont'd)

							QC Criteria >>	<15%	80-120% (i)	80-120% (i)	75-125% (h)	75-125% (h)	< +/-10%
Method	**Prep**	**Analyte**	**MRQ µg/g**	**MDL µg/g**	**MRQ > 3xMDL**	**Average µg/g**	**DF**	**RSD(g) %**	**Blank Spike (LCS) %Rec**	**Solid LCS (c,l) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
IC-Inorg	Leach-103	PO_4 (t)	1,840	26	Yes	250		15	86		(b)	93	
ICP-MS	Fusion-115	**Pr**	6.0	0.004	Yes	264		3	$\boxed{\textbf{135}/105^{(k)}}$		$\boxed{\textbf{163}}$	99	1
ICP-MS	Fusion-116	**Pt**	3.0	0.01	Yes	0.70	B	$\boxed{\textbf{30}}$	92		97	94	$\boxed{\textbf{24}}$
ICP-MS	Fusion-115	**Pu sum** (s,z)	6.0	0.004	Yes	19.7		11	(w)			(w)	(w)
ICP-MS	Fusion-115	**Pu-239(z)**		0.004		18.4		11	106 (j)			114	5
ICP-MS	Fusion-115	Pu-240(z)		0.0002		1.3		9	110 (j)			118	8
ICP-MS	Fusion-115	**Rb**	6.0	0.02	Yes	262	B	$\boxed{\textbf{21}}$	111 (j)	107	(b)	102	9
ICP-AES	Acid-129M2	**Rh** (o)		180		180	U					95 (a)	
ICP-AES	Fusion-115	**Rh** (o)		680		680	U					97	
ICP-MS	Fusion-116	**Rh**	300	0.001	Yes	90.8		8	94		$\boxed{\textbf{66}}$	95	3
ICP-AES	Acid-129M2	**Ru** (o)		650		650	U						
ICP-AES	Fusion-115	**Ru** (o)		2,500		2,500	U						
ICP-MS	Fusion-116	**Ru**	300	0.2	Yes	1150		6	88		(b)	106	2
ICP-AES	Acid-129M2	**Sb** (o)		300		300	U					101 (a)	
ICP-AES	Fusion-115	**Sb** (o)		1,200		1,100	U					99	
ICP-MS	Fusion-115	**Sb**	12	0.01	Yes	3.4	B	10	$\boxed{\textbf{69}/102^{(k)}}$	$\boxed{\textbf{39}}$	$\boxed{\textbf{68}}$	103	$\boxed{\textbf{29}}$
ICP-AES	Acid-129M2	**Se** (o)		150		150	U					103 (a)	
ICP-AES	Fusion-115	**Se** (o)		570		570	U					100	
ICP-MS	Fusion-115	**Se**	300	1	Yes	1	UB		106		77	96	
ICP-AES	Acid-129M2	**Si** (u)	3,000	300	Yes	970	JX	15	(u)	(u)	(u)	107 (a)	
ICP-AES	Fusion-115	**Si**	3,000	1,200	No	3,800	J	9	99	89		106	
ICP-AES	Acid-129M2	Sn (o)		890		1,000	J						
ICP-AES	Fusion-115	Sn (o)		3,400		3,400	U						
IC-Inorg	Leach-103	SO_4 (t)	1,800	260	Yes	24,000		9	94		(b)	100	
ICP-AES	Acid-129M2	**Sr**	300	8.9	Yes	356		6	118	100	(b)	103 (a)	
ICP-AES	Fusion-115	**Sr**	300	34	Yes	320	J	3	119			101	
ICP-MS	Fusion-115	**Ta**	6.0	0.001	Yes	3.65	B	$\boxed{\textbf{132}}$	101 (j)		(b)	97	$\boxed{\textbf{39}}$

Table 9.4. (Cont'd)

							QC Criteria >>	<15%	80-120% (i)	80-120% (i)	75-125% (h)	75-125% (h)	< +/-10%
Method	**Prep**	**Analyte**	**MRQ µg/g**	**MDL µg/g**	**MRQ > 3xMDL**	**Average µg/g**	**DF**	**RSD (g) %**	**Blank Spike (LCS) %Rec**	**Solid LCS (c,l) %Rec**	**Matrix Spike (MS) %Rec**	**Post Spike %Rec**	**Serial Dilution %Diff**
ICP-AES	Acid-129M2	**Te** (o)		890		890	U						
ICP-AES	Fusion-115	**Te** (o)		3,400		3,400	U						
ICP-MS	Fusion-115	**Te**	6.0	0.2	Yes	196		4	**73**/93 (k)		**45**	90	8
ICP-AES	Acid-129M2	**Th** (o)		590		590	U		104			102 (a)	
ICP-AES	Fusion-115	**Th** (o)		2,300		2,300	U					97	
ICP-MS	Fusion-115	**Th**	600	0.003	Yes	109		6	**50**/107 (k)	111	(b)	110	5
ICP-AES	Acid-129M2	**Ti**	150	15	Yes	64	J	36	98	95	110	99 (a)	
ICP-AES	Fusion-115	**Ti**	150	57	No	68	J	11	95	89		100	
ICP-AES	Acid-129M2	**Tl** (o)		300		300	U					96 (a)	
ICP-AES	Fusion-115	**Tl** (o)		1,200		1100	U					98	
ICP-MS	Fusion-115	**Tl**	600	0.002	Yes	0.16	B	**38**	82	111	80	95	**50**
ICP-AES	Acid-129M2	**U**	150	1,200	No	3,600	J	6	98		96	94 (a)	
ICP-AES	Fusion-115	**U**	150	4,500	No	5300	J	7	108			97	
KPA	Fusion-115	**U**	150	0.20	Yes	3,170		7	100		95		
ICP-MS	Fusion-115	**U sum** (p)	6.0	0.1	Yes	3680		3	(v)	(v)		(v)	(v)
ICP-AES	Acid-129M2	**V** (o)		30		30	U		97		95	98 (a)	
ICP-AES	Fusion-115	**V** (o)		120		110	U		100			98	
ICP-MS	Fusion-115	**V**	6.0	0.03	Yes	23.4	B	4	115	110	75	101	**11**
ICP-AES	Acid-129M2	**W** (o)		1,200		1,200	U				(b)		
ICP-AES	Fusion-115	**W** (o)		4,500		4,500	U						
ICP-MS	Fusion-115	**W**	6.0	0.01	Yes	33.3	B	14	106	103	**54**	101	1
ICP-AES	Acid-129M2	**Y** (o)		30		110	J	5				99 (a)	
ICP-AES	Fusion-115	**Y** (o)		120		100	J					102	
ICP-MS	Fusion-115	**Y**	6.0	0.009	Yes	117		1	**129**/107 (k)	96	106	101	2
ICP-AES	Acid-129M2	**Zn**	6.0	30	No	79	J		114	99	97	105 (a)	
ICP-AES	Fusion-115	**Zn**	6.0	120	No	130	JX	12	**128**	98		104	
ICP-AES	Acid-129M2	**Zr**	600	30	Yes	19,500		6	105		(b)	103 (a)	1.2

Table 9.4. (Cont'd)

						QC Criteria >>		<15%	80-120% [(i)]	80-120% [(i)]	75-125% [(h)]	75-125% [(h)]	< +/-10%
Method	Prep	Analyte	MRQ µg/g	MDL µg/g	MRQ > 3xMDL	Average µg/g	DF	RSD[(g)] %	Blank Spike (LCS) %Rec	Solid LCS [(c,l)] %Rec	Matrix Spike (MS) %Rec	Post Spike %Rec	Serial Dilution %Diff
ICP-AES	Fusion-115	**Zr**	600	120	Yes	13,400		6	109			105	1.8

Bolded analytes required for comparison to Contract Specification 8 (Envelope D)

Outlined and bolded results exceed QC flagging criteria

Blank fields indicate QC not required or not defined in the test plan or test specification.

Prep: See Section 7.0 for preparation information for PNL-ALO-114, 115, and 116 fusion methods and PNL-ALO-129 Mod 2 acid digestion method

MRQ: minimum reportable quantity

MDL: method detection limit

DF: data quality flag (See Section 8.1 for definitions)

RSD: relative standard deviation (in percent)

(a) Batch QC PS for ICP-AES performed on sample 01-0955 ASR 6104 (AW-101 Simulant).
(b) LCS or MS not recovered; sample concentration >> spike concentration (MS) or spiking level too low after required analytical dilution (MS and/or LCS).
(c) Solid LCS: ICP-AES/ICP-MS NIST SRM-2710; Hg NIST SRM-2709; CN ERA Priority PollutnT reference material (see Section 9.1, 9.7 and 9.8).
(d) MS recovery average of MS 131% and MSD 175%.
(e) MS recovery average of MS 27% and MSD 3%.
(f) Solids LCS recovery 91% from expired standard; see Section 9.7 for explanation.
(g) RSD only calculated when sample, duplicate, and triplicate exceed MDL.
(h) MS and PS QC flagging criteria for Na = 90% to 110%, all ICP-MS analytes 70% to 130%.
(i) Sodium QC flagging criterion: 90% to 110%.
(j) Post spiked blank used as LCS/BS for analyte.
(k) Results include both the LCS/BS recovery (first result) and a post spiked LCS/BS recovery (second result).
(l) Solids LCS for ICP-AES and ICP-MS prepared by total digestion method and compared to certified results based on a leaching preparation; high recoveries for some analytes not unexpected.
(m) RSD QC criterion for sodium: 3.5%.
(n) Oxalate LCS recovery = 0%; reason unknown (see Section 9.5).
(o) Opportunistic analytes; not include in test specification or test plan.
(p) Total uranium (U Sum) based on the sum of all U isotopes measured by ICP-MS.
(q) TC is sum of TIC and TOC by HP; TIC is difference between measured TC Furn and TOC Furn.
(r) The fluoride results considered the upper bound concentration for the fluoride, since the fluoride peak shape and retention time suggest the presence of co-eluting anion(s).
(s) Total plutonium (Pu Sum) based on sum of Pu isotopes measured by ICP-MS.
(t) MDL is based on the lowest calibration standard adjusted for sample dilution; equivalent to SW-846 EQL definition.

Table 9.4. (Cont'd)

QC Criteria >>								<15%	80-120% [i]	80-120% [i]	75-125% [h]	75-125% [h]	< +/-10%
Method	Prep	Analyte	MRQ µg/g	MDL µg/g	MRQ > 3xMDL	Average µg/g	DF	RSD[g] %	Blank Spike (LCS) %Rec	Solid LCS [c,l] %Rec	Matrix Spike (MS) %Rec	Post Spike %Rec	Serial Dilution %Diff

(u) Most silicon from PNL-ALO-129 Mod 2 lost in processing; see Section 7.2.4.

(v) For QC performance for uranium by ICP-MS, see ^{238}U Table 9.3.

(w) For QC performance for plutonium by ICP-MS, see ^{239}Pu and ^{240}Pu by ICP-MS.

(x) Best estimate for TOC is about 1,300 µg/g; See Section 9.6.3.1.

(y) For QC performance for iodine by ICP-MS, see ^{129}I Table 9.3.

(z) Same certified source standard used to prepare calibration and verification standards for ICP-MS. Calibration and verification standards prepared approximately 1 year apart; prepared standards verified by independent analysis (i.e., LSC, AEA, or GEA).

10.0 References

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CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDICES

Prepared By: **MW Urie**

2004

Battelle - Pacific Northwest Division

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APPENDICES: Primary Analytical Data

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX A

TEST SPECIFICATION

2004

Battelle - Pacific Northwest Division

TEST SPECIFICATION

TSP-24590-01-00006, Rev. 0

A00-002

River Protection Project
Waste Treatment Plant

RPP-WTP

Tank 241-AZ-101 Sample Composite, Homogeneity, and Analysis

Test Specification

Revision 0

July 2, 2001

Ernest Lee
Characterization, R&T
BNI/WGI

Approval	Date
Ernest Lee, Characterization R&T	7/16/01
Roger Roosa, Characterization R&T Manager	7/16/01
Todd Wright, R&T Manager	7/16/2001

ISSUED BY
RPP-WTP PDC
7-27-01
INIT DATE

A00-003

Revision Summary

Revision Number	Task	Purpose
DRAFT A	N/A	First, draft of document.
DRAFT B	General revision effecting all sections	To incorporate comments received from the review of Draft A
0		Initial Issue
24590-PTF-TSP-RT-01-001 Rev 0	Changed Document Number Section 1.5, added sentence 1.6.1.1 added sentence Added footnotes to Tables 1, 2 & 3 Added comments in Section 1.6.1 (f), (g), and 1.6.1.2	Formerly TSP-24590-01-00006 Clarify when QARD is applied Clarify when QARD is applied Clarify when QARD is applied. Requested documentation of hold point approval to continue

Purpose

This specification provides instructions to Battelle Laboratories personnel for:

- Preparing composite samples of waste from multiple waste samples from tank 241-AZ-101
- Verifying the homogeneity of the composite samples
- Measuring physical properties of the composite samples at three undissolved solids concentrations that are expected to occur during processing in the waste pretreatment facility
- Analyzing both the liquid and the sludge composites
- Comparing analytical results with the low-activity waste (LAW) and high-level waste (HLW) feed specifications for the waste treatment plant (WTP)
- Reporting analytical results

Testing performed in accordance with this specification is to support process verification testing. Environmental/regulatory testing is limited to PCB analysis only. PCB testing is to address urgent waste management issues in a timely fashion that cannot be addressed soon enough by implementation of the Regulatory DQO (Wiemers, et al., PNNL 12040).

Background

Between August 17 and 28, 2000, Hanford site personnel obtained one core sample [Core 283] consisting of eighteen segments (19 inch high) from the entire tank depth and two additional sludge segments from the bottom 39 inches of tank 241-AZ-101. The prefix "241" is common to all Hanford Site tanks and will not be used further. This sample was obtained to satisfy requirements in ICD-23 for process verification testing[1]. Attachment 2 contains recent core profiles taken from AZ-101 and includes Core 283. The core sampling process requires the addition of a hydrostatic fluid into the sampling system to displace the sample volume removed. The hydrostatic fluid is a 0.3 M LiBr solution. Both lithium and bromine are not normally found in Hanford waste. Attachment 3 provides additional information collected at the time the core sediments were extruded in building 222-S. The 283 core sample was taken after tank mixing pumps were run in tank AZ-101 in April and May 2000[2]. Evidence collected during the mixer pump testing program demonstrated that most of the tank's sludge was suspended. At that time, the sludge became well homogenized both vertically and horizontally, therefore, the sludge in the core sample taken after the sludge suspension event is representative of the tank's entire sludge composition.

However, compositing all core samples is not likely to produce a composite sample that has the same liquid-solids ratio of tank AZ-101. This is the case since additional segments were taken for segments 17 and 18. The additional cores were necessary to obtain the minimum quantity of solids needed for process verification testing. During the three month lag between mixing and sampling, the readily gravity separable solids settled to the tank bottom. The near complete sediment of the solids is evident by the data shown in Attachment 3. As a result of the earlier tank pump test, the supernate zone is likely to contain entrained solids (non-gravity separable solids) more closely representative of the entrained solids expected during Low Activity Waste (LAW) transfers to WTP.

After sampling the tank, the core segments were extruded or poured into glass bottles. These sample bottles have been stored in 222-S building since the sampling event. It is likely that some evaporation of the sample contents has occurred. The tank AZ-101 waste samples are expected to be shipped to Battelle by way of sample trucks. Shipments are expected to occur in May or June 2001. Prior to shipment of the core to Battelle, 222-S personnel are planning to sub-sample the solids in segments 17, 17A, 18, and 18A. A total of about 6 grams of solids are expected to be removed from the entire core. The slurried waste contained in tank AZ-101 is candidate high level waste (HLW) for the River Protection Project Waste Treatment Plant (RPP-WTP). The liquid portion of the tank waste, after separation from the sludge in the RPP-WTP will become low-activity waste (LAW) feed (envelope B) for RPP-WTP.

Battelle personnel were initially going to perform a crossflow ultrafiltration test using waste from core segment 1 through 16. However, after BNI R&T and PNNL representatives inspected the core segments that containing liquid proposed for crossflow ultrafiltration, it was determined that there was insufficient solids present to perform, crossflow ultrafiltration testing. Battelle personnel shall composite the waste using the sludge containing segments

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[1] BNFL-5193-ID-23, Rev 4, Interface Control Document for Waste Treatability Samples, June 2000.

[2] Preliminary Test 241-AZ-101 Mixer Pump Test, RPP-6548, Rev. 0, July 2000.

and liquid core segments that have not been contaminated with hydrostatic fluid. The entire composite will be sub-sampled. The homogeneity of sub-samples will be measured. Physical properties listed in Table 1 will be measured at three different solids concentrations. The concentration of elements and radionuclides listed in Tables 2 and 3 will be measured in the liquid and centrifuged solids. Separate test specifications will be issued for the process verification testing and waste form qualification test activities conducted with the AZ-101 sample and will include the following tests:

- Crossflow ultrafiltration of HLW slurry to provide filter flux for WTP design verification
- Undissolved solids washing
- Caustic leaching of the undissolved solids
- The filtrate will be used for multiple load and elution tests with SuperLig 644 (Cs) and SuperLig 639 (Tc) ion exchange columns (~10-ml resin per column) to determine useful operating life of these resins and variability in resin performance
- Evaporation of pretreated sample to determine saturation end-point, partitioning of compounds of potential regulatory concern, and provide information to verify process models
- Melter feed rheology measurements
- Lab-scale radioactive melter process test to determine partitioning of radionuclides and inorganic compounds, determine if hazardous compounds (e.g., dioxins) form in melter system, verify ability to process glass formulation, demonstrate compliance for both LAW and HLW glasses with land disposal restrictions and other contract specifications

In 2000, the Tank Farm contractor performed mixer pump tests in AZ-101 to demonstrate the applicability of the mixer pump system to prepare high level waste for retrieval. During the mixer pump operation, the tank waste was sampled.[3,4,5]

The sodium concentration of the liquid fraction of tank AZ-101 waste is ~4.8M as reported in Tank Waste Information Network System [TWINS], "Best Basis Inventory". Battelle personnel will not need to dilute the tank 241-AZ-101 sample for process verification testing and waste form qualification activities unless considerable loss of liquid occurred during sample storage.

Tank AZ-101 contains 865,000 gallons of supernate and 52,000 gallons of settled sludge.[6] The tank waste is scheduled to be mixed and transferred to WTP to become the first batches of waste to the HLW Pretreatment Facility.[7]

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[3] HNF-1706,Tank 241-AZ-101, Grab samples for the Mixer Pump Test Event 1 Analytical Results for the Final Report.

[4] HNF-1708,Tank 241-AZ-101, Grab samples for the Mixer Pump Test Event Two, Three and Four Analytical Results for the Final Report.

[5] HNF-6052, Tank 241-AZ-101 Grab Samples for the Mixer Pump Test Event Six Analytical Results for the Final Report.

[6] HNF-EP-0182, Rev. 155, Waste Tank Summary Report for Month Ending February 28, 2001.

[7] HNF-SD-WM-SP-012, Rev. 2, Tank Farm Contractor Operation and Utilization Plan, April 19, 2000.

BNI R&T Plan Reference

Section 1.0 of the Research and Technology Plan identifies characterization requirements for low-activity waste (LAW) and high-level waste (HLW) samples.

Schedule Reference

The RPP-WTP Research and Technology schedule for fiscal year 2001 identifies this activity as R20250, Characterization (PNNL). This activity is listed in the Battelle schedule for fiscal year 2001.

BNI/WGI Statement of Work / RPP-WTP Request for Proposal Reference

Characterization of tank waste samples is identified in the Design and Construction Contract for the Hanford Tank Waste Treatment and Immobilization Plant, standard 2, item (a)(3)(i).

1.1 Justification

This task provides information for assessing tank AZ-101 waste for compliance with the HLW and LAW feed specifications. Measurement of the physical properties of the AZ-101 feed and concentrated slurries provides an opportunity to underpin the pretreatment facility design basis. Additionally, it is critical that the AZ-101 waste samples shipped to Battelle are homogeneous before sub-sampling to ensure the analysis of the sub-samples provides information representative of the bulk sample.

1.2 Objectives

The objectives of these tasks are to:

- Receive and verify liquid samples were not corrupted with hydrostatic head fluid
- Composite all low LiBr liquid samples and undissolved solids from cores 17, 17A, 18, and 18A from CUF if performed by thoroughly mixing in a compositing vessel,
- While operating the vessel agitator, extract all of the material from the compositing vessel into volume graduated jars
- Verify the homogeneity of the sub-samples by measuring the volume of settled solids and supernate in at least five sub-samples
- Determine the wt% undissolved solids concentration in composite
- Perform PCB analysis per methods established for the Regulatory DQO
- Adjust the concentration of sub-samples to 3±1 wt%, 13±2 wt% and 20±2 wt% undissolved solids
- Measure the physical properties of the 3, 13 and 20 wt% sub-samples
- Measure the chemical and radioisotope concentrations listed Tables 2 and 3 in the solid and liquid fractions of the original composite.
- Determine liquid fraction compliance to specification 7 of contract DE-AC27-01RV14136
- Determine solid fraction compliance to specification 8 of contract DE-AC27-01RV14136
- Report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1)
- Provide a draft comprehensive technical report within 90-days of completing all analyses

1.3 Test Plan

Battelle shall prepare a general or specific test plan containing detailed information needed to implement this test specification. The test plan shall include a table that lists the expected amount of sample needed for each characterization step and the expected amount of sample remaining and available for process verification testing.

The test plan shall provide specific direction for the designated use volumes and preparation method for each sub-sample. All non-corrupted excess sample material shall be retained for use for process verification testing.

The test plan shall provide in tabular form; the methods chosen for sample preparation, whether an actual analyte, surrogate, or tracer shall be used for Laboratory Control Standards (LCS), matrix spikes and the instrument calibration requirements. The estimated minimum detection limit for each analyte in Tables 2 and 3 shall be provided. If the target minimum reportable quantity for an analyte in Tables 2 and 3 are below the estimated minimum detection limit, they shall be flagged. The QA target requirements listed in Tables 1, 4, and 5 shall be reviewed and any requirements that are not expected to be met should be identified and an the expected delta from the criteria be identified. The test plan shall provide the technical basis for calibration and demonstration methods for continued performance where LCS and spike recovery are not performed for a specific analyte.

Battelle may propose and use alternate analytical methods and QC requirements than those listed in this test specification provided justification is provided in the approved test plan. If analytical procedures developed for the Regulatory DQO (Wiemers et al., PNNL-12040) have been successfully demonstrated, then the use of these procedures shall be considered. However, modification of these procedures may be needed to account for smaller sample volumes.

A draft of the test plan shall be submitted to BNI R&T for review and comment. Comments labeled "Required" under the "significance" column require a disposition. That is, the comment should be incorporated into the document to the degree that it is technically correct if it is not negated by other document revision. Comments that are "acknowledged" or "noted" require written justification for not incorporating the comment to some degree.

The final test plan must be approved by BNI R&T representative. Prior to performing testing for this work, the approved test plan must be submitted to BNI's Project Document Control.

1.4 Success Criteria

The analytes and physical properties listed in this test plan were obtained. The QC target criteria in Tables 1, 4, and 5 along with the QC requirements for the approved QA plan for the project were also met. The target criteria presented are goals for demonstrating reliable method performance. However, the analytical data may be acceptable for its intended purpose even if some of the QA criteria is not met. It is understood that the laboratory will follow its internal QC system for required actions whenever QC failures occur. If more than 5 QC failures occur, or if all analysis cannot be performed (e.g., insufficient sample), analysts shall consult with BNI R&T representative 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.

For PCB measurements, success criteria will be identification/quantification of detectable PCBs with the QA/QC requirements specified in the "Regulatory DQO Test Plan for Determining Method Detection Limits, Estimated Quantitation Limits, and Quality Assurance Criteria for Specified Analytes", PNNL-13429, and the associated PCB test plan, TP-RPP-WTP-30.

1.5 Quality Assurance

The RPP-WTP *Quality Assurance Program,* BNFL-5193-QAP-01 revision 8, requires the designation of a quality level for all work. The sample analysis results may be used directly for assessing the performance of important to safety equipment. Therefore, the work performed under this test specification is designated as important to safety (quality level QL-1 or QL-2 per the RPP-WTP *Quality Assurance Program,* BNFL-5193-QAP-01 revision 8).

Battelle implements the RPP-WTP quality requirements in a quality assurance plan (QAP) as approved by the RPP-WTP QA organization. Battelle shall perform this task in compliance with 10 CFR 830.120, "Quality Assurance Requirements" and any additional quality assurance requirements based on nationally recognized standards incorporated in the Battelle QAP for important to safety items and activities.

Additionally, BNI/WGI personnel may use the PCB analysis results for regulatory submittals (i.e., environmental data). For the PCB analysis, the requirements specified in PNNL-13429 and the associated test plan, TP-RPP-WTP-30 shall be met.

The *Quality Assurance Requirements and Description* (DOE/RW-00333P), the principal quality assurance document for the Civilian Radioactive Waste Management Program applies to certain activities conducted as part of this task. Specifically, the formation of the composite, the homogeneity check of the composite fractions and the characterization of the tank composite centrifuged solids should be performed to the QARD. The QARD does not apply to the characterization of the liquid fraction of the tank composite, the process testing of the expected receipt, mid-range and maximum slurry concentration or the lithium bromide segment analyses.

1.6 Test Conditions

Battelle personnel are to inspect and weigh the "as received samples" to determine what degree of sample loss has occurred during sample storage and transport. Battelle personnel are to report the visual appearance of the liquid and solid / crystalline phases present in each AZ-101 sample bottles.

Battelle personnel are to maintain a material balance for the AZ-101 samples throughout the process steps defined by this test specification. Items typically to be recorded include sample bottle identification number, bottle tare weight (provided by 222-S), the mass (or volume) of sample received in each bottle, loss of sample due to residual sample left in each bottle, mass (or volume) of composite AZ-101 sample, mass and volume of chemical additions, mass (or volume) of sample removed for analysis, and any other significant activities that add or remove mass (or volume) from the AZ-101 sample.

Battelle personnel are to monitor the condition of all samples that are archived for extended periods. Actions shall be taken to prevent samples from drying out. At a minimum actions shall include; keeping the samples in sealed jars, replacing evaporated liquid before solids are exposed, and delay separating the liquid from solids until ready to proceed with testing or analysis.

1.6.1.1 Compositing Tank Samples

Battelle personnel are to record the temperature of the cell during sample compositing and sub-sampling activity.

Battelle personnel shall sub-sample the liquid in each sample jar that contains a separate liquid phase and analyze the sub-samples for lithium and bromide (only single analysis per sub-sample is required). The purpose of the analysis is to determine if the sample contains hydrostatic head fluid from the sampling process. If the concentration of either lithium or bromide is greater than 0.003 M, then the sample shall not be included in the compositing process. The criteria of 0.003 M was selected to limit the amount of hydrostatic head fluid to 1 part in 100 parts. Notify BNI R&T of actual lithium and bromine concentrations before proceeding . QARD does not apply to the above activity.

All sample bottles containing liquid that meet the criteria of ≤0.003 M lithium and / or bromide and sample bottles containing undissolved solid shall be transferred into a clean vessel that contains an 1/8 to 3/32" screen, and in addition agitation and sampling (e.g., re-circulation line from pump) systems.

Once the material is transferred into the vessel, remove the screen and place a cover on the mixing vessel. If any material remains on the sample screen after flushing the screen with the liquid samples, determine weight of material and archive material. Notify BNI representative with a proposed path forward for this material.

The vessel agitation system shall be operated continuously at least an hour prior to performing sub-sampling and shall continue to operate during sub-sampling.

QARD applies to the following steps. The sub-sampling method and homogeneity verification method shall be based on the methods and criteria described in PNNL Test Plan for "Regulatory Data Quality Objective: Sample Compositing Strategy," TP-41500-001, Rev. 0 and shall be consistent with the following. While operating the vessel agitation system, Battelle personnel are to extract sub-samples from the AZ-101 slurry into volume graduated glass jars. The volume of the composite samples will range in size from 100 to 500 ml and be based on the anticipated volume needed for sample preparation and analysis, that is documented in the test plan. The order the sub-samples pulled must be recorded. The entire contents of the compositing vessel shall be extracted as sub-samples.

a. These sub-samples are to be tightly sealed and sit undisturbed for a minimum of 24-hours.

b. Battelle personnel are to inspect the sub-samples for floating organic layers and record volume or thickness and appearance on any found layer.

c. **Administrative Hold Point:** If an organic layer is observed in any of the composite batches, then BNI's R&T representative must be consulted before work continues.

d. Battelle personnel are to record the volume of settle solids/crystalline material and supernate present in each of the sub-samples after the 24 hour undisturbed period. The volume measurement shall be reported to an accuracy of 1% of the container volume for containers > 100 mls and ±1.0-ml for containers < 100 mls.

e. Battelle personnel are to calculate the relative volume percentage of settle solids/crystalline material present in each of the sub-samples selected for homogeneity verification and compare the volume percent settled solids versus the order of sample collection.

f. **Administrative Hold Point:** If the liquid sub-samples contain an average of less than 5 vol% settled solids, then the homogenization is complete. The volume settled solids data on the composite liquid sub-samples are to be presented to BNI R&T for approval before proceeding. Once this approval has been received, proceed to compositing sludge samples. Documentation of approval shall be recorded and included in the data package.

g. **Administrative Hold Point:** If the liquid sub-samples contain an average of greater than 5 vol% but less than 60% settled solids, then the following two criteria must be met before the composite sub-samples are to be presented to the BNI R&T for approval before proceeding to composing sludge samples. The data package submitted for BNI R&T approval is to include information on any outlier eliminated from the data set reconciled against the criteria. If the composite sub-samples fail the criteria, then the composite sub-samples will be returned to the mixing vessel and rehomogenized and sub-sampled again at a higher agitation rate and/or other modifications deemed necessary to obtain improved mixing. If the second homogenization fails, BNI R&T will be consulted. Documentation of approval to continue shall be recorded and included in the data package.

   1. Calculate a standard deviation for the entire vol% settled solids data set. If there are a few outliers, eliminate up to 10% of the samples from the data set, and then recalculate the standard deviation. This standard deviation must be no greater than 5 vol%. Note, after eliminating outliers, there must be at least 5 sub-samples remaining in the data set.

   2. Calculate a best fit line for the data set (vol% settled solids versus collection order). This best fit line must not show a trend of greater than 5 Vol% over the range. For example, if the linear best fit line is at 50 vol% for the first sample, then it cannot exceed 55 vol% or be below 45 vol% for the last sample (these are the values for the best fit line, not samples).

h. If the liquid sub-samples contain an average of greater than 60% settled solids, then proceed to do the following with the samples selected for homogeneity testing. A minimum of five sub-samples shall be used for determination of sub-sample homogeneity. These sub-samples for homogeneity determination shall include the second, middle and next to the last sub-samples removed from the compositing vessel. Two other sub-samples will be chosen at random. Mix the selected sub-sample and transfer triplicate aliquots of the selected homogeneity sub-samples into volume graduated centrifuge cones. Allow the triplicates to settle for 3 days. Record the total mass and volume and the volume of the settled solids. Calculate the bulk slurry density, and the volume percent settled solids. If sufficient clarified liquid is present following settling, transfer a portion to a volumetric container and determine supernate density. Centrifuge the settled slurries at approximately 1000 times the force of gravity for 1 hour. Transfer all of the centrifuged supernate into a graduated cylinder and measure its mass and volume. Measure and record the mass and volume of the centrifuged solids. Calculate density of centrifuge solids. Dry the centrifuged solids and supernate at 105 to constant weight. Determine wt% solids, wt% dissolved solids and wt% undissolved solids. Record the volume of centrifuged solids/crystalline material and supernate present in each of the sub-samples after centrifuging. Plot the data against the sub-sample removal sequence. Submit the data Package to BNI R&T as confirmation that the sub-sampling was homogenized. The data package submitted for BNI R&T approval is to include information on any outlier eliminated from the data set reconciled against the criteria. If the composite sub-samples fail homogenization criteria, then all the composite sub-samples will be returned to the mixing vessel and rehomogenized and sub-sampled again at a higher agitation rate and/or other modifications deemed necessary to obtain improved mixing. If the second homogenization fails, BNI R&T will be consulted.

1.6.1.2 Chemical and Radiological Characterization of Sub-Sample

If step 1.6.1.2 h was not done then Battelle personnel are to rehomogenizing one sub-sample of the initial slurry composite and then measure in triplicate the slurry density, wt% total solids, wt% dissolved solids, wt% undissolved solids (by way of calculation) and supernate density.

The remaining material from the sub-sample shall be centrifuged for 1 hr at ~ 1000 x g. to separate damp solids from liquid. Decant the liquid from the solids. Measure the volumes and weights of the liquid and centrifuge solids. Calculate the density of the damp centrifuge solids, the density of the centrifuged liquid and the vol% and wt% centrifuged solids.

For Decanted Liquid Analysis:

- Dry an aliquot of the decanted centrifuged liquid to a constant weight at 105 °C. Measure and record % wt residual, this is the wt% dissolved solids.
- Determine the concentration of the analytes listed in Table 2. All analysis shall be performed in triplicate.
- Quality Control parameters are defined in Table 4. Note extended GEA count time will be required to obtain minimum reportable quantities for some isotopes.

For Centrifuge solids analysis:

- Dry an aliquot of the centrifuged solids at 105 °C to a constant weight. Measure and record % wt loss.
- Calculate the wt% undissolved solids and wt% soluble solids in the centrifuged solids and in the original sub-samples, using an equation equivalent to Section 1.6.1.7, Equation (1).
- Prepare the centrifuged solids for analysis by using laboratory sludge dissolution methods that are specified in the test plan. Note: Sample dissolution may be done on the centrifuge solids rather than dried solids as long as the results of the analysis are reported on a equivalent undissolved solids basis. If after the dissolution process, the solids aren't completely dissolved, then archive the solids analysis. After reviewing the results from all dissolution methods, contact the BNI R&T representative if there is concern that the undissolved material was not dissolved by either dissolution method used. Document any issues and the approval to continue and include in the data package.
- Determine the concentration of the analytes listed in Table 3. All analysis shall be performed in triplicate. Quality Control parameters are defined in Table 5. Note extended GEA count time will be required to obtain minimum reportable quantities for some isotopes.
- Report concentration on a dried solids and a undissolved solids basis. Reporting on a undissolved basis will require adjusting the results for the contribution of the dissolved solids, using a method equivalent to that provided Section 1.6.1.7 equation (3).

1.6.1.3 PCB Characterization of Sub-Sample

One sub-sample shall be designated for PCB analysis. This sub-sample shall be archived without separating the solids from the liquid until the analytical laboratory is ready to analyze the sample. The sample shall be analyzed along with the Reg. DQO Step 1 Analysis or after the Reg. DQO Step 1 analysis is complete. The analysis shall be performed using the same approved procedures and reporting methods required by the Reg. DQO. The archived sample condition shall be monitored and controlled in order to preserve the sample integrity.

1.6.1.4 Physical Characterization at the Expected Receipt, Mid-Range and Maximum Concentration

Settle and decant supernate from one sub-sample designated for the maximum-range concentration sub-sample until a final concentration of 20±2 wt% undissolved solids is reached. Note the 20 wt% undissolved solids is the design basis for concentrating HLW. To reach this concentration, it may be necessary to augment settling by centrifuging the sub-sample. Save the supernate. The amount of supernate to be decanted shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample (Section 1.6.1.3).

Settle and decant supernate from sub-sample designated to be the mid-range concentration sub-sample until a final concentration of 13±2 wt% undissolved solids is reached. Note the 13 wt% undissolved solids is mid range for design basis for concentrating HLW. Save the supernate. The amount of supernate to be decant shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample (Section 1.6.1.3).

Adjust the concentration of the sub-sample designated for the receipt concentration by adding liquid to the sub-sample to reach a concentration of 3±1 wt% undissolved solids. Note 3 wt% undissolved solids is the expected feed concentration for tank AZ-101[8]. The amount of supernate to be added shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample (Section 1.6.1.3).

Battelle personnel are to mix the above three sub-samples after adjusting the wt% solids. Then the three sub-samples shall be analyze in triplicate to determine slurry density, wt% total solids, wt% dissolved solids, wt% undissolved solids, yield strength, shear stress Vs shear rate, and heat capacity per Table 1.

Only one of the sub-samples shall require particle size distribution and SEM analysis.

1.6.1.5 Compare Analysis to WPT Contract Specification 7 (LAW)

Battelle personnel are to compare and report the centrifuged liquid analytical results for the slurry feed concentration composite sample to the limits listed in Specification 7, Low-Activity Waste Envelopes in *WTP Contract No. DE-AC27-01RV14136* (see attachment 1*)*.

1.6.1.6 Compare Analysis to WPT Contract Specification 8 (HLW)

Battelle personnel are to compare and report the analytical results for the centrifuged solids to the limits listed in Specification 8, High-Level Waste Envelope in *WTP Contract No. DE-AC27-01RV14136* (see attachment 1*)*.

Specification 8 limits are specified in units of curies or grams per 100 grams non-volatile waste oxides (unwashed solids). It is necessary to limit the waste oxides to those available from the undissolved solids.

The characterization of sludge solids is done on samples that contain both dissolved and undissolved solids. Therefore the results of the analysis must be adjusted to an undissolved basis, by calculation. The undissolved solids basis does not include the contribution of the analyte and dried mass in the interstitial liquid. The results are determined from a calculation rather that analysis of the undissolved solids only. A method that accomplishes this is:

- determine wt of centrifuged solids sub-sample [w_{cs}]
- determine wt of dried (105 °C for 24±1-hour) centrifuged solids sub-sample [w_{dcs}]
- determine wt of a sub-sample of liquid [w_{ls}]
- determine wt of dried sub-sample of liquid [w_{dls}]

w_{ds} = weight fraction of dissolved solids, $w_{dls}/w_{ls,}$ (wt dissolved solids/ wt of supernate)
w_{tcs} = weight fraction of total dried centrif. solids, w_{dcs}/w_{cs}, (wt total dried centrif. solids/wt of centrif. solids)
w_{uds} = weight fraction undissolved solids, (wt of undissolved solids/ wt of centrifuged solids)
w_{ss} = weight fraction of soluble solids, (wt dissolved solids/wt of centrifuged solids)

$$w_{uds} = (w_{tcs} - w_{ds})/(1 - w_{ds}) \qquad \text{Equation (1)}$$

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[8] HNF-SD-WM-SP-012, Rev. 2, Tank Farm Contractor Operation and Utilization Plan, April 19, 2000.

$W_{ss} = W_{tcs} - W_{uds}$ Equation (2)

i_x =analyte i concentration on dried centrifuged solids wt basis (mg/Kg or mCi/Kg)
i_y =analyte i concentration in liquid, on liquid volume basis (mg/L or mCi/L)
i_z =analyte i concentration on undissolved solids basis (mg/Kg or mCi/Kg)
S_pg_l = specific gravity of liquid

$i_z = ((i_x * w_{tcs}) - (i_y /(s_pg_l) * (w_{ds}))/(1-w_{ds})$ Equation (3)

Once the concentrations are reported on a undissolved solids basis, then the concentrations can be reported on a oxide basis by converting the concentration of the elements present to their oxide form and summing the oxides.

1.7 Reporting

Battelle personnel are to report all characterization results in metric units, in accordance with section 6.6.3, *Convention of Units of Measure*, of the DOE-ORP statement of work to BNI/WGI.
Battelle personnel are to issue the draft test results to RPP-WTP within 90 calendar days after completing the analyses identified in this test specification. Battelle personnel are to issue a final test report within 30 calendar days after receiving comments on the draft report from RPP-WTP. This report shall be issued as a document approved for public release and available to all Hanford contractors.
Battelle personnel shall also report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1), to the extent data field information is available.

Table 1[1]

Slurry Analyses for Feed Composition, Mid Range Composition and Concentrated Composition(a)

Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Physical Property (a)	**Expected Range**	
Slurry Density(b)	1 to 1.6 gm/cm;	Gravimetric
Liquid Density(b)	1 to1.6 gm/cm	Gravimetric
Vol% Centrifuged solids(b)	0.1 to 50 vol%	Volumetric
Wt% Centrifuged solids(b)	0.1 to 30 wt%	Gravimetric
Wt% Total Dried solids(b)	1 to 80 wt%	Gravimetric
Wt% Dissolved solids(b)	1 to 25 wt%	Gravimetric
Wt% Undissolved solids(b)	1 to 25 wt%	Calculation
Heat Capacity(c, d)	0.5 to 2.0 J/g-°C	Calorimeter
Shear Stress Vs Shear Rate(c, f)	0 to 1000(e) sec^{-1}	Viscometer
Yield Strength(c, f)	1 to 1000 Pa	Viscometer
Particle size(g)	0.1 to 500 micron	Laser Scattering
Particle (size & shape)	0.1 to 500 micron	SEM

[1] QARD does not apply when these analyses are performed on the process testing samples. Any physical property measurements on the original composite should be performed according to QARD

Footnotes:

(a) Triplicate measurements are to be made for each Physical Property at specified concentration and temperature. Acceptable precision is <15% RSD. RSD = (standard deviation/mean) * 100

(b) Measurements are to be made using vendor calibration of glassware and laboratory balances.

(c) Measurements are to be made at cell ambient temperature (normally ambient between 28-35 °C), and 40±3 °C.

(d) Results are calculated based on behavior of sample relative to a standard (water or sapphire depending on sample range).

(e) or the upper limit of selected sensor/viscometer configuration.

(f) Perform calibration check every 30 days using a NIST traceable standard.

(g) Perform calibration check every 30 days using instrument manufactures recommended method.

Table 2 [1]
Liquid Fraction Analyses

Analyte	**Target Minimum Reportable Quantity**	**Recommended Analysis Method[(c)]**
	mg/L	
Al	7.5E+01	ICP-AES (AA may be used for Na, K)
B	2.3E+00	
Ba	2.3E+00	
Ca	1.5E+02	
Ce	2.3E+00	
Cd	7.5E+00	
Cr	1.5E+01	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Li	2.3E+00	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Th	2.3E+00	
V	2.3E+00	
W	2.3E+00	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TOC	1.5E+03 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
Br	3.0E+02	
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03 (as P)	
SO_4	2.3E+03 (as S)	
	mCi/L (except as noted)	ICP-MS (AA may be used for Cs determination)
Rb	1.0E+00 (mg/L)	
^{127}I	1.5E+00 (mg/L)	
^{129}I	1.8E-05	
^{133}Cs	7.0E-04 (mg/L)	
^{135}Cs	1.5E+00	
^{137}Cs	1.5E+00	
^{237}Np	2.7E-02	
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	8.7E-03(mg/L)	
^{99}Tc	1.5E-03	
^{233}U	4.2E-04	
^{234}U	1.2E-04	
^{235}U	4.5E-08	
^{236}U	1.4E-06	
^{238}U	7.2E-09	
^{99}Tc(pertechnetate)	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{3}H	2.1E-02	Separations / Liquid Scintillation
^{14}C	7.2E-04	
^{79}Se	9.0E-05	
^{90}Sr	1.5E-01	
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}$Pu	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}$Cm	1.5E-02	

Table 2 Cont.

^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	2.1E-03	
^{126}Sn	6.0E-03	
^{137}Cs	9.0E+00	
^{231}Pa	7.89E-05	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha (TRU)	N/A	Summation(b) of: Pu-238, Pu-239+Pu-240 (or Pu-239, Pu-240 ICP/MS) and Am-241
Total and Free OH	7.5E+04 mg/L	Titration
CN	3.0E+00 mg/L	CN Analysis
Ammonia	1.4E+02 mg/L	ISE or IC
Organic Analytes	**mg/L**	
Oxalate	1.5E+03	Ion Chromatograph
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
EDTA(a)	1.5E+03	Derivatization/ GC-MS
HEDTA(a)	1.5E+03	
D2EHPA(a)	1.5E+03	
NTA(a)	1.5E+03	
IDA(a)	1.5E+03	
Succinic Acid(a)	1.5E+03	
ED3A(a)	1.5E+03	
	Expected Range	
Density	0.95 –1.5 (gm/mL)	Gravimetric
Dissolved solids	1 to 50 (gm solids/gm supernate)	Gravimetric

[1] QARD does not apply to these analyses

Footnote:

(a) MRQs are target values, measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters.

(b) Guidance for reporting summation of isotopics and reporting isotopic values derived by different methods will be provided later after an agreement is obtained with DOE.

(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 3. HLW Solids Analyses [1]

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method(c)
	mg/Kg(a)	
Ag	9.0E+02	ICP-AES (AA may be used for Na & K)
Al	3.3E+02	
B	3.0E+00	
Ba	6.0E+02	
Be	3.0E+00	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
K	2.0E+02	
La	6.0E+01	
Li	3.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
U	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
Br	4.5E+02	
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	
CN	3.0E+00	CN analysis
	mg/Kg	ICP-MS
As	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Mo	3.0E+01	
Pd	3.0E+02	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Rh	3.0E+02	
Ru	3.0E+02	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	

Table 3. Continued, HLW Solids Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analytical Methods(c)
W	**mg/Kg** 6.0E+00	ICP-MS
Y	6.0E+00	
^{99}Tc	6.0E+00	
^{127}I	1.5E+00	
^{133}Cs	7.0E-04	
^{233}U	6.0E+01	
^{235}U	6.0E+00	
^{237}Np	1.8E+00	
^{129}I	**mCi/Kg** 1.8E-05	
^{135}Cs	5.3E-03	
^{137}Cs	9.0E-02	
^{151}Sm	TBD	
^{234}U	3.7E-03	
^{236}U	3.8E-04	
^{238}U	2.0E-06	
^{3}H	1.5E-02	Separations / Liquid Scintillation
^{14}C	1.8E-03	
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}$Pu	6.0E+00	
^{242}Pu	3.36E-01	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}$Cm	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
^{125}Sb	6.0E+00	
^{126}Sn	6.0E-02	
^{134}Cs	9.0E-01	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha (TRU)	N/A	Summation(b) of: Pu-238, Pu-239, Pu-240, and Am-241
^{241}Pu	1.2E+00 mCi/Kg	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	–
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetric
Density	0.9 to 2.0 gm/ml	Gravimetric
Wt% Undissolved Solids	10 to 50 wt%	Calculation
Wt% Soluble Solids	1 to 50 wt%	Calculation
^{1} QARD does apply to these analyses		

Footnote:

(a) MRQs are based on dried solids weighs.

(b) Guidance for reporting summation of isotopics that are derived by different methods will be provided later after an agreement is obtained with DOE

(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Recommended Analytical Technique[f]	QC Flagging Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Triplicate RSD[c]
Al, B, Ba, Ca, Cd, Ce, Cr, Fe, K, La, Li, Mg, Ni, P, Pb, Th, U, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
U	Kin. Phosphorescence	80 - 120%	75 - 125%	<15%
Rb, ^{127}I, ^{129}I, ^{133}Cs, Cs^{135}, Cs^{137}, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Pu / ^{241}Am, ^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cl$^-$, Br$^-$, F$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{-3}$, SO$_4^{-2}$	IC	80 - 120%	75 - 125%	<15%
CN$^-$	CN Analysis	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH$_3$/NH$_4^+$	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH- (total and Free)	Potentiometric titration /precipitation	80 - 120%	N/A	<15%
TIC	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[e]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[i], ^{126}Sn[j], ^{231}Pa	Extended GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[e]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[e]	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A[g]	<15%
^{154}Eu[i]	GEA	NP	N/A[g]	<15%
^{155}Eu[i]	GEA	NP	N/A[g]	<15%
^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm	Separation/AEA	NP	N/A[e]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Sum of Alpha[h] [TRU]	Calculation	N/A	N/A	N/A
Density	Gravimetric	N/A	N/A	<20%
Wt% dissolved solids	Gravimetric	N/A	N/A	<20%
EDTA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
HEDTA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Oxalate	IC	80 - 120%	75 - 125%	<15%
Citrate	IC	80 - 120%	75 - 125%	<15%
Formate	IC	80 - 120%	75 - 125%	<15%
Gluconate	IC	80 - 120%	75 - 125%	<15%
Glycolate	IC	80 - 120%	75 - 125%	<15%
D2EPHA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr, use Sr in the standard for determining the total mass-90 concentration.

(e) 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.

(f) 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.

(g) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(h) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting 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 that 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 MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination. If any of the isotopes are below the MRQ, then the method of summation will be agreed by BNI prior to reporting values.

(i) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(j) Calibrate with Sn-117.

(k) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

(l) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 5. Quality Control Parameters for Solids Analysis

Solids Fraction	Recommended Analytical Technique[e]	QC Flagging Criteria		
		LCS % Recovery[a]	Spike % Recovery[b]	Triplicate RSD[c]
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Cs, Fe, K, La, Mg, Mn, Nd, Ni, P, Pb, Pd, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, U, V, W, mass unit 90[d], ^{99}Tc, ^{127}I, ^{133}Cs, ^{233}U, ^{235}U, ^{237}Np, ^{129}I, ^{135}Cs, ^{137}Cs, ^{151}Sm, ^{234}U, ^{236}U, ^{238}U.	ICP/MS	80 - 120%	70 - 130%	<15%
Cl$^-$, Br$^-$, F$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{-3}$, SO$_4^{-2}$	IC	80 - 120%	75 - 125%	<15%
CN$^-$	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Hg	CVAA	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%
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{60}Co[i]	Extended count GEA	NP	N/A[k]	<15%
^{90}Sr[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{125}Sb[m]	Extended Count GEA	NP	N/A	<15%
^{126}Sn[n]	Extended Count GEA	NP	N/A	<15%
^{134}Cs[h]	Extended Count GEA	NP	N/A	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{152}Eu[i]	Extended Count GEA	NP	N/A[k]	<15%
^{154}Eu[i]	Extended Count GEA	NP	N/A[k]	<15%
^{155}Eu[i]	Extended Count GEA	NP	N/A[k]	<15%
^{241}Am	Extended Count GEA	NP	N/A[k]	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, $^{239/240}$Pu, ^{242}Pu	Separation/AEA	NP	N/A[g]	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[g]	<15%
^{242}Cm	Separation/AEA	NP	N/A[g]	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A[g]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Bulk density	Gravimetric	N/A	N/A	<20%
Wt% solids	Gravimetric	N/A	N/A	<20%

Acronyms:
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 Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.

(e) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(f) Not used.

(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 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 WAPS justification.

(i) 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 ^{152}Eu, ^{154}Eu, ^{155}Eu.

(j) Combined analysis of ^{90}Sr and ^{90}Y.

(k) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(l) Combined analysis with ^{93m}Nb.

(m) Combined analysis with ^{125m}Te.

(n) Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

(o) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

ATTACHMENT 1, CONTRACT SPECIFICATIONS
(Excerpt from Contract No. DE-AC27-O1RV14136)

Specification 7: Low-Activity Waste Envelopes Definition

7.1 Scope: This Specification establishes three LAW feed envelopes, Waste Envelopes A, B, and C; and defines how a unit of LAW is determined for each LAW envelope. Each waste envelope provides the compositional limits for chemical and radioactive constituents in the waste feed to be provided to the WTP. The WTP shall be designed to treat the waste envelopes.

7.2 Requirements:

7.2.1 References:

7.2.1.1 HNF-SD-WM-SAR-067, Rev. 1-I. March 2000. *Tank Waste Remediation System Final Safety Analysis Report.* CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.2 HNF-SD-WM-TSR-006, Rev. 1-HE. March 2000. *Tank Waste Remediation System Technical Safety Requirements*, CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.3 OSD-T-151-00007, Rev. H-22. June 14, 2000. *Operating Specification for 241-AN, AP, AW, AY, AZ, and SY Tank Farms.* CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.4 DOE/RL-88-21, Rev. 10. December 21, 1999. *Double Shell Tank Unit Permits Application.* U.S. Department of Energy, Richland Operations Office, Richland, Washington.

7.2.2 Envelope Requirements:

7.2.2.1 Composition: This specification lists the concentration limits for the LAW Envelopes A, B, and C feed to be transferred by DOE to the Contractor for LAW services in Tables TS-7.1, *Low-Activity Waste Chemical Composition, Soluble Fraction Only*, and TS-7.2, *Low-Activity Waste Radionuclide Content, Soluble Fraction Only*. The concentration limits apply to the soluble fraction only. The Na concentration limits for the LAW feeds are identified below.

1.7.1.1.1 Waste Feed	Na (mole per liter)
Envelope A, B, C	4 – 10
AZ-101 and AZ-102 Supernatant	2 – 5
HLW Slurry and other HLW Liquids (Defined in Specification 8, *High-Level Waste Envelope Definition*)	0.1 – 10

The LAW feeds may contain up to two weight percent solids. Solids are defined as the product of centrifuging the LAW feed, separating and drying the solids, and removing the dissolved solids contribution. The insoluble fraction characterization will include measurements of Al, Cr, Fe, Mn, Na, P, S, Si, U, TIC, TOC, ^{60}Co, ^{90}Sr, ^{99}Tc, ^{137}Cs, ^{154}Eu, $^{239/240}$Pu, ^{241}Am, and total alpha concentrations. Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed.

All LAW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report*, and *Technical Safety Requirements*, as applicable.

The radiochemical inventory of the LAW feed at the time of delivery shall be compared to the specification limits to assess compliance. The specifications for ^{60}Co, and ^{154}Eu shall apply at the time of delivery for ILAW immobilization.

The LAW feed provided shall not contain a visible separate organic phase.

The LAW feed provided will generate gases, including hydrogen and ammonia, at a nearly constant rate and a nearly uniform composition.

The maximum ^{137}Cs concentration equivalent in the transferred Envelope A, Envelope B, and Envelope C wastes feeds shall not exceed 1.2 Ci/l. The maximum ^{137}Cs concentration equivalent in the liquid fraction of Tank AZ-101 and AZ-102 feeds shall not exceed 3.0 Ci/l.

Dangerous waste codes are identified in the Double-Shell Tank System Unit Permit Application (DOE/RL-88-21, December 21, 1999). Multi-source leachate (F039) is included as a waste derived from non-specific source wastes F001 through F005.

7.2.3 Units of Low-Activity Waste: Units of LAW shall be defined as follows:

(a) Envelope A: The quantity of Waste Envelope A containing one metric ton of waste sodium shall equal one unit.

(b) Envelope B: The quantity of Waste Envelope B containing one metric ton of waste sodium shall be the lesser of the following number of units:

(1) 2.6 units; or

(2) $\frac{X}{Y}$ units

where X is equal to 18-weight percent sodium oxide loading in the ILAW glass and Y is equal to the achievable waste sodium oxide loading, for the particular waste feed. The waste loading limitations shall be based solely upon effects of chlorine, chromium, phosphate, and sulfate.

(c) Envelope C: The quantity of Waste Envelope C containing one metric ton of waste sodium shall be the lesser of the following number of units:

(1) 1.15 units; or

(2) $\frac{X}{Y}$ units

where X and Y are defined above. The waste loading limitations shall be based solely upon sodium additions required for cesium, technetium, strontium and TRU removal from Envelope C for the particular waste feed.

Table TS-7.1 Low-Activity Waste Chemical Composition, Soluble Fraction Only

Chemical Analyte	Maximum Ratio, analyte (mole) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
Al	2.5E-01	2.5E-01	2.5E-01
Ba	1.0E-04	1.0E-04	1.0E-04
Ca	4.0E-02	4.0E-02	4.0E-02
Cd	4.0E-03	4.0E-03	4.0E-03
Cl	3.7E-02	8.9E-02	3.7E-02
Cr	6.9E-03	2.0E-02	6.9E-03
F	9.1E-02	2.0E-01	9.1E-02
Fe	1.0E-02	1.0E-02	1.0E-02
Hg	1.4E-05	1.4E-05	1.4E-05
K	1.8E-01	1.8E-01	1.8E-01
La	8.3E-05	8.3E-05	8.3E-05
Ni	3.0E-03	3.0E-03	3.0E-03
NO_2	3.8E-01	3.8E-01	3.8E-01
NO_3	8.0E-01	8.0E-01	8.0E-01
Pb	6.8E-04	6.8E-04	6.8E-04
PO_4	3.8E-02	1.3E-01	3.8E-02
SO_4	1.0E-02	7.0E-02	2.0E-02
TIC[1]	3.0E-01	3.0E-01	3.0E-01
TOC[2]	5.0E-01	5.0E-01	5.0E-01
U	1.2E-03	1.2E-03	1.2E-03

Notes:

[1] Mole of inorganic carbon atoms/mole sodium

[2] Mole of organic carbon atoms/mole sodium

Table TS-7.2 Low-Activity Waste Radionuclide Content, Soluble Fraction Only

Radionuclide	Maximum Ratio, radionuclide (Bq) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
TRU[2]	4.8E+05	4.8E+05	3.0E+06
^{137}Cs	4.3E+09	2.0E+10	4.3E+09
^{90}Sr	4.4E+07	4.4E+07	8.0E+08
^{99}Tc	7.1E+06	7.1E+06	7.1E+06
^{60}Co	6.1E+04	6.1E+04	3.7E+05
^{154}Eu	1.2E+06	1.2E+06	4.3E+06

Notes:

[1] The activity limit shall apply to the feed certification date.

[2] TRU is defined as: Alpha-emitting radioncludes with an atomic number greater than 92 with half-life greater than 10 years.

Some radionuclides, such as ^{90}Sr and ^{137}Cs, have daughters with relatively short half-lives. These daughters have not been listed in this table. However, they are present in concentrations associated with the normal decay chains of the radionuclides.

Specification 8: High-Level Waste Envelope Definition

8.1 Scope: This Specification establishes the HLW slurry composition and the unwashed solids composition (Envelope D). This waste envelope provides the compositional limits for chemical and radioactive constituents and physical properties in the waste feed to be provided to the WTP. The WTP shall be designed to treat the feed envelopes.

8.2 Requirements:

8.2.1 References:

8.2.1.1 HNF-SD-WM-SAR-067, Rev. 1-I. March 2000. *Tank Waste Remediation System Final Safety Analysis Report*. CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.2 HNF-SD-WM-TSR-006, Rev. 1-HE. March 2000. *Tank Waste Remediation System Technical Safety Requirements*, CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.3 OSD-T-151-00007, Rev. H-22. June 14, 2000. *Operating Specification for 241-AN, AP, AW, AY, AZ, and SY Tank Farms*. CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.4 DOE/RL-88-21, Rev. 10. December 21, 1999. *Double Shell Tank Unit Permits Application*. U.S. Department of Energy, Richland Operations Office, Richland, Washington.

8.2.2 High-Level Waste Slurry Description and Envelope Requirements:

8.2.2.1 Composition: The HLW slurry will contain a mixture of liquids (Envelopes A, B, or C) and solids (Envelope D). The compositional range of the liquid fraction is defined in Specification 7, *Low-Activity Waste Envelopes Definition*. For liquid fractions with a sodium molarity of less than three, the liquid shall be treated as if 3 molar sodium were present for feed certification purposes. Specification 7.2.2.4, *Radioactive Material Concentration*, does not apply to Envelope A, B, or C liquids. The composition range of the Envelope D unwashed solids is given in Tables TS-8.1, TS-8.2, TS-8.3, and TS-8.4. The feed concentration will be between 10 and 200 grams of unwashed solids/liter, except for feeds from waste Tanks AZ-101 and AZ-102, where minimum solids content does not apply.

Compositions for Envelope D unwashed solids (Tables TS-8.1, TS-8.2, TS-8.3, and TS-8.4) are defined in terms of elemental or anion concentrations and radionuclide activities per 100 grams equivalent non-volatile waste oxides. The non-volatile waste oxides include sodium oxide and silicon oxide.

The HLW feed components identified in Tables TS-8.1, TS-8.2 and TS-8.3 are waste components important to establishing the waste oxide loading in the HLW glass. Only these components have concentration limits, which will be used to provide the basis for certification that the HLW feed is within specification limits.

The HLW feed components identified in Table TS-8.4 are also important to HLW glass production. The concentrations of these components in the waste are not expected to exceed the maximum values listed in Table TS-8.4. Information on these components will be provided to support product and process qualification but will not be used as a basis for determining if the feed meets specification requirements.

All HLW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report* (HNF-SD-WM-SAR-067), and *Technical Safety Requirements* (HNF-SD-WM-TSR-006, Revision 1-D) as applicable. The radiochemical inventory of the waste feed at the time of delivery shall be compared to the specification limits to assess compliance.

Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed. Feed will be delivered by pipeline in batches. Limits apply to the total retrievable contents of waste from a feed tank. Some elements, components, and isotopes are determined by calculation and not analytic measurement.

The HLW feed provided will not contain a visible separate organic layer.

The HLW waste provided will generate gases due to radiolysis including hydrogen and ammonia at a nearly constant rate and nearly uniform composition. The Contractor is responsible for the management of changes in gas release rate and distribution resulting from their waste processing activities.

Applicable dangerous waste codes are identified in the Double-Shell Tank System Unit Permit Application (DOE/RL-88-21, December 21, 1999). Multi-source leachate (F039) is included as a waste derived from non-specific source wastes F001 through F005.

**Table TS-8.1 High-Level Waste Feed Unwashed Solids Maximum Non-Volatile Component Composition
(grams per 100 grams non-volatile waste oxides)**

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
As	0.16	Pu	0.054
B	1.3	Rb	0.19
Be	0.065	Sb	0.84
Ce	0.81	Se	0.52
Co	0.45	Sr	0.52
Cs	0.58	Ta	0.03
Cu	0.48	Tc	0.26
Hg	0.1	Te	0.13
La	2.6	Tl	0.45
Li	0.14	V	0.032
Mn	6.5	W	0.24
Mo	0.65	Y	0.16
Nd	1.7	Zn	0.42
Pr	0.35		

**Table TS-8.2 High-Level Waste Feed Unwashed Solids Maximum Volatile Component Composition
(grams per 100 grams non-volatile waste oxides)**

Volatile Components	Maximum (grams / 100 grams waste oxides)
Cl	0.33
CO_3^{-2}	30
NO_2	36 (total NO_2/NO_3) as NO_3
NO_3	
TOC	11
CN	1.6
NH_3	1.6

Table TS-8.3 High-Level Waste Feed Unwashed Solids Maximum Radionuclide Composition (Curies per 100 grams non-volatile waste oxides)

Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)
^{3}H	6.5E-05	^{129}I	2.9E-07	^{237}Np	7.4E-05
^{14}C	6.5E-06	^{137}Cs	1.5E00	^{238}Pu	3.5E-04
^{60}Co	1E-02	^{152}Eu	4.8E-04	^{239}Pu	3.1E-03
^{90}Sr	1E+01	^{154}Eu	5.2E-02	^{241}Pu	2.2E-02
^{99}Tc	1.5E-02	^{155}Eu	2.9E-02	^{241}Am	9.0E-02
^{125}Sb	3.2E-02	^{233}U	9.0E-07	$^{243+244}$Cm	3.0E-03
^{126}Sn	1.5E-04	^{235}U	2.5E-07		

Table TS-8.4 Additional High-Level Waste Feed Unwashed Composition for Non-Volatile Components (grams per 100 grams non-volatile waste oxides)

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
Ag	0.55	Ni	2.4
Al	14	P	1.7
Ba	4.5	Pb	1.1
Bi	2.8	Pd	0.13
Ca	7.1	Rh	0.13
Cd	4.5	Ru	0.35
Cr	0.68	S	0.65
F	3.5	Si	19
Fe	29	Ti	1.3
K	1.3	U	14
Mg	2.1	Zr	15
Na	19		
Th	5.0		

Attachment 2

AZ-101 CORE PROFILE

Attachment 3

CORE SEGMENT EXTRUSION INFORMATION

AZ-101 Core 283 Segment 1 Riser 59

Date Extruded:	09/12/00
Sample Number:	283-01
Cask SN:	24G
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001800

2 EXTRUSION INFORMATION

Liner Liquid: < 5 mL, did not retain.

Drainable Liquid: Collected approximately 285 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solids: No solids were observed.

3 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	346.2 g	**Jar:**	18396 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	346.2 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 2 Riser 59

Date Extruded:	9/12/00
Sample Number:	283-02
Cask SN:	6G
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001801

4 EXTRUSION INFORMATION

Liner Liquid: < 10 mLs /did not retain.

Drainable Liquid: Collected approximately 290 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solids: Trace brown/white, flaky solids were present. The solids were retained with the drainable liquid.

5 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	354.8 g	**Jar:**	18397 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	354.8 g		

Reviewed by Rob Schroeder 9/12/00

AZ-101 Core 283 Segment 3 Riser 59

Date Extruded:	09/12/00
Sample Number:	283-03
Cask SN:	C2004
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001802

6 EXTRUSION INFORMATION

Liner Liquid: < 5 mL, did not retain.

Drainable Liquid: Collected approximately 295 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solids: Observed a few brown/white, flaky, suspended solids. The solids were retained with the drainable liquid.

7 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	364.2 g	**Jar:**	18398 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	364.2 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 4 Riser 59

Date Extruded: 09/12/00

Sample Number: 283-04

Cask SN: SN-77

Logbook : WHC-N-1126-2

LABCORE ID: S00T001803

8 EXTRUSION INFORMATION

Liner Liquid: None observed.

Drainable Liquid: Collected approximately 310 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solids: A small quantity of brown/white solids was observed. The solids were retained with the drainable liquid.

9 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	362.2 g	**Jar:**	18399 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	362.2 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 5 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-05
Cask SN:	15G
Logbook :	WHC-N-1126
LABCORE ID:	S00T001786

10 EXTRUSION INFORMATION

Liner Liquid: There was no liner liquid.

Drainable Liquid: Collected approximately 290 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solids: Observed a few white, flaky, suspended solids. The solids were retained with the drainable liquid.

11 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	370.4 g	**Jar:**	18394 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	370.4 g		

Reviewed by Robert Schroeder 09/07/00

AZ-101 Core 283 Segment 6 Riser 59

Date Extruded:	9/07/00
Sample Number:	283-06
Cask SN:	C-2010
Logbook :	WHC-N-1126
LABCORE ID:	S00T001787

12 EXTRUSION INFORMATION

Liner Liquid: <5 mLs /did not retain.

Drainable Liquid: Collected approximately 290 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solids: Trace white, flaky solids were present. The solids were retained with the drainable liquid.

13 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	369.6 g	**Jar:**	18391 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	369.6 g		

Reviewed by Rob Schroeder 9/07/00

AZ-101 Core 283 Segment 7 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-07
Cask SN:	C-1047
Logbook :	WHC-N-1126
LABCORE ID:	S00T001788

14 EXTRUSION INFORMATION

Liner Liquid: < 5 mL, did not retain.

Drainable Liquid: Collected approximately 298 mLs of drainable liquid (L1). The liquid was yellow and slightly opaque.

Solids: Observed a small amount of white, flaky, suspended solids. The solids were retained with the drainable liquid.

15 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	364.8 g	**Jar:**	18393 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	364.8 g		

Reviewed by Rob Schroeder 09/07/00

AZ-101 Core 283 Segment 8 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-08
Cask SN:	14G
Logbook :	WHC-N-1126
LABCORE ID:	S00T001789

16 EXTRUSION INFORMATION

Liner Liquid: <5 mLs /did not retain.

Drainable Liquid: Collected approximately 305 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solid Sample: Observed a small quantity of white, flaky, suspended solids. The solids were retained with the drainable liquid.

17 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	371.0 g	**Jar:**	18407 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	371.0 g		

Reviewed by Rob Schroeder 09/07/00

AZ-101 Core 283 Segment 9 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-09
Cask SN:	C-1002
Logbook :	WHC-N-1126
LABCORE ID:	S00T001790

18 EXTRUSION INFORMATION

Liner Liquid: <5 mLs /did not retain.

Drainable Liquid: Collected approximately 300 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solid Sample: Observed a small quantity of white, flaky, suspended solids. The solids were retained with the drainable liquid.

19 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	372.0 g	**Jar:**	18406 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	372.0 g		

Reviewed by Annette Barnes 09/08/00

AZ-101 Core 283 Segment 10 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-10
Cask SN:	1012
Logbook :	WHC-N-1126
LABCORE ID:	S00T001791

20 EXTRUSION INFORMATION

Liner Liquid: <5 mls / did not retain.

Drainable Liquid: Collected approximately 310 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solid Sample: Observed a small quantity of white and brown, suspended solids. The solids were retained with the drainable liquid.

21 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	374.8 g	**Jar:**	18402 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	374.8 g		

Reviewed by Annette Barnes 09/07/00

AZ-101 Core 283 Segment 11 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-11
Cask SN:	76
Logbook :	WHC-N-1126
LABCORE ID:	S00T001792

22 EXTRUSION INFORMATION

Liner Liquid: <5 mLs /did not retain.

Drainable Liquid: Collected approximately 305 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solid Sample: Observed a small quantity of white, flaky, suspended solids. The solids were retained with the drainable liquid.

23 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	370.9 g	**Jar:**	18403 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	370.9 g		

Reviewed by Annette Barnes 09/07/00

AZ-101 Core 283 Segment 12 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-12
Cask SN:	72
Logbook :	WHC-N-1126
LABCORE ID:	S00T001793

24 EXTRUSION INFORMATION

Liner Liquid: < 10 mLs /did not retain.

Drainable Liquid: Collected approximately 320 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solid Sample: Observed a small quantity of white, flaky, suspended solids. The solids were retained with the drainable liquid.

25 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	368.5 g	**Jar:**	18390 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	368.5 g		

Reviewed by Annette Barnes 09/07/00

AZ-101 Core 283 Segment 13 Riser 59

Date Extruded:	09/07/00
Sample Number:	283-013
Cask SN:	29
Logbook :	WHC-N-1126
LABCORE ID:	S00T001794

26 EXTRUSION INFORMATION

Liner Liquid: < 10 mLs /did not retain.

Drainable Liquid: Collected approximately 300 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solid Sample: Observed a small quantity of white, flaky, suspended solids. The solids were retained with the drainable liquid.

27 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	366.0 g	**Jar:**	18408 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	366.0 g		

Reviewed by Annette Barnes 09/07/00

AZ-101 Core 283 Segment 14R Riser 59

Date Extruded:	09/12/00
Sample Number:	283-14R
Cask SN:	73
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001804

28 EXTRUSION INFORMATION

Liner Liquid: < 10 mL, did not retain.

Drainable Liquid: Collected approximately 270 mLs of drainable liquid (L1). The liquid was yellow in color and clear.

Solids: No solids were observed.

29 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	333.3 g	**Jar:**	18400 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	333.3 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 15 Riser 59

Date Extruded:	09/12/00
Sample Number:	283-15
Cask SN:	20-G
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001805

30 EXTRUSION INFORMATION

Liner Liquid: < 5 mL, did not retain.

Drainable Liquid: Collected approximately 295 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solids: There was a trace amount of white solids observed. The solids were retained with the drainable liquid.

31 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	364.3 g	**Jar:**	18401 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	364.3 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 16 Riser 59

Date Extruded:	9/12/00
Sample Number:	283-16
Cask SN:	1009-C
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001806

32 EXTRUSION INFORMATION

Liner Liquid: <5 mLs /did not retain.

Drainable Liquid: Collected approximately 295 mLs of drainable liquid (L1). The liquid was yellow in color and slightly opaque.

Solids: Trace brown/white, flaky solids were present. The solids were retained with the drainable liquid.

33 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	358.3 g	**Jar:**	18413 (500 mL)
Whole Segment Solids:	n/a	**Jar:**	
Total Sample Mass:	358.3 g		

Reviewed by Rob Schroeder 9/12/00

AZ-101 Core 283 Segment 17 Riser 59

Date Extruded:	09/12/00
Sample Number:	283-17
Cask SN:	27-G
Logbook :	WHC-N-1126
LABCORE ID:	S00T001807

34 EXTRUSION INFORMATION

Liner Liquid: Less than 30 mL liner liquid present, did not retain.

Drainable Liquid: There was no drainable liquid observed.

Solids: Extruded approximately 8 inches of solids. The solids were dark brown in color and the texture resembled a sludge slurry (D2).

35 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	n/a	**Jar:**	
Whole Segment Solids:	309.0 g	**Jar:**	17023 (250 mL)
Total Sample Mass:	309.0 g		

Reviewed by Robert Schroeder 09/12/00

AZ-101 Core 283 Segment 17A Riser 59

Date Extruded:	9/12/00
Sample Number:	283-017A
Cask SN:	62
Logbook :	WHC-N-1126
LABCORE ID:	S00T001808

36 EXTRUSION INFORMATION

Liner Liquid: <10 mLs /did not retain.

Drainable Liquid: Collected approximately 60 mLs of drainable liquid (L1). The liquid was dark brown in color and opaque. A few mL (<5) of drainable liquid was lost during sampling.

Solids: Extruded approximately 10 inches of solids. The upper segment solids, 4 inches, were dark brown in color and the texture resembled a sludge slurry (D2). The lower segment solids, 6 inches, were dark brown in color and the texture resembled a wet sludge (D3).

37 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	69.1 g	**Jar:**	18344 (60 mL)
Upper Segment Solids:	110.9 g	**Jar:**	15888 (125 mL)
Lower Segment Solids:	155.6 g	**Jar:**	17331 (125 mL)
Total Sample Mass:	335.6 g		

Reviewed by Rob Schroeder 9/12/00

AZ-101 Core 283 Segment 18 Riser 59

Date Extruded:	09/12/00
Sample Number:	283-18
Cask SN:	SN-64
Logbook :	WHC-N-1126-2
LABCORE ID:	S00T001809

38 EXTRUSION INFORMATION

Liner Liquid: < 40 mL, did not retain.

Drainable Liquid: Collected approximately 50 mLs of drainable liquid (L1). The liquid was dark brown in color and opaque.

Solids: Extruded approximately 4 inches of solids. The solids were dark brown in color and the texture resembled a wet sludge (D3).

39 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	54.4 g	**Jar:**	18345 (60 mL)
Whole Segment Solids:	133.7 g	**Jar:**	15958 (250 mL)
Total Sample Mass:	188.1 g		

Reviewed by Rob Schroeder 09/12/00

AZ-101 Core 283 Segment 18A Riser 59

Date Extruded: 09/12/00

Sample Number: 283-018A

Cask SN: 16G

Logbook : WHC-N-1126-2

LABCORE ID: S00T001810

40 EXTRUSION INFORMATION

Liner Liquid: < 10 mLs /did not retain.

Drainable Liquid: Observed approximately 5 mLs of drainable liquid (L1). The liquid was dark brown in color and opaque. The liquid was retained with the segment solids.

Solid Sample: Extruded approximately 11 inches of solids. The solids were dark brown in color and the texture resembled a wet sludge (D3).

41 SUBSAMPLE INFORMATION

Liner Liquid:	n/a	**Jar:**	
Drainable Liquid:	n/a	**Jar:**	
Whole Segment Solids:	390.1 g	**Jar:**	17025 (250 mL)
Total Sample Mass:	390.1 g		

Reviewed by Rob Schroeder 09/12/00

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX B

CHAIN OF CUSTODY AND INSPECTION

2004

Battelle - Pacific Northwest Division

B1

CHAIN OF CUSTODY

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

F

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.15	17025		5/31/01	1400	1EA/500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
R+Steele	RHSteele	5/31/01 1500	DP Wohlwend	DP Wohlwend	5-31-01 1515
DP Wohlwend	DP Wohlwend	5-31-01 2000	RE Thornhill		5-31-01 2000

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquid	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION — Disposal Method (e.g., Return to customer, per lab procedure, used in process) — Disposed By — Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

BC-6000-828 (04/

B01-002

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No

Page 1 of 1

Collector
Rob Shroeder

Contact/Requestor

Telephone No. 373-5810
MSIN T6-50
FAX 373-1438

SAF No.

Sample Origin
11A Hotcell

WORK ORDER#BE4742 CACN114728

Logbook No.

Ice Chest No.

Temp.

Project Title
AZ-101 ICD23 Shipment to PNNL

Method of Shipment

Bill of Lading/Air Bill No.

Shipped To (Lab)
325 Laboratory PNNL

Data Turnaround

Offsite Property No.

Protocol

Sample No.	Lab ID	•	Date	Time	No./Type Container	Sample Analysis	Preservative
460.14	17023		5/31/01	1400	1EA/500ml/Glass		

Hold Time

B

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS
Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Relinquished By (Print / Sign)	Date/Time	Received By (Print / Sign)	Date/Time
RFSteele	5/31/01 1500	DPWohlwend / DP Wohlwend	5-31-01 1505
DPWohlwend / DP Wohlwend	5-31-01 2000	RE Thornhill	5-31-01 2000

Matrix*
S = Soil
SE = Sediment
SO = Solid
SL = Sludge
W = Water
O = Oil
A = Air
DS = Drum Sc
DL = Drum Li
T = Tissue
WI = Wipe
L = Liquid
V = Vegetat
X = Other

FINAL SAMPLE DISPOSITION

Disposal Method (e.g., Return to customer, per lab procedure, used in process)

Disposed By

Date/Time

ll be picked up by requestor and returned to parent container or site of origin

BC-6000-828

B01-003

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460-22	18228				1EA/500ml glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By (Print / Sign)	Date/Time	Received By (Print / Sign)	Date/Time
RTSTEELE / RHStl 5/31/01	2055	BCEkstrom / BCElk	5/31/01 2055
BCEkstrom / BCElk 5/31/01	2229	RE Thornhill / RE Thornhill	5-31-01 2230

Matrix*

S = Soil		DS = Drum Solid	
SE = Sediment		DL = Drum Liquid	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION — Disposal Method (e.g., Return to customer, per lab procedure, used in process) Disposed By Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rod Shreeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.4	18729				1EA) 500ml glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RTSteele	5/31/01	2050	DP Wohlwend	DP Wohlwend	5-31-01 2050
DP Wohlwend	DP Wohlwend 5-31-01	2235	RE Thornhill	RE	5-31-01 2235

Matrix*

S	= Soil	DS	= Drum Solid
SE	= Sediment	DL	= Drum Liquid
SO	= Solid	T	= Tissue
SL	= Sludge	WI	= Wipe
W	= Water	L	= Liquid
O	= Oil	V	= Vegetation
A	= Air	X	= Other

FINAL SAMPLE DISPOSITION

Disposal Method (e.g., Return to customer, per lab procedure, used in process)

Disposed By

Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/9

B01-005

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.7	18721		5/23/01	1500	1EA 500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
R T STEELE	Rt Steele	5/22/01 5/23/01 1545	JA Johnston	Julie A Johnston	5-23-01 1559
Julie A Johnston		5/23/01 1810	BC Ekstrom	BC Ekstrom	5/23/01 1810
BC Ekstrom	BC EKSTROM	5/23/01 2021	RE Thornhill	RE Thornhill	EOC 5/23/01

Matrix*

S	= Soil	DS	= Drum Solids
SE	= Sediment	DL	= Drum Liquids
SO	= Solid	T	= Tissue
SL	= Sludge	WI	= Wipe
W	= Water	L	= Liquid
O	= Oil	V	= Vegetation
A	= Air	X	= Other

FINAL SAMPLE DISPOSITION — Disposal Method (e.g., Return to customer, per lab procedure, used in process) — Disposed By — Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/98)

B01-006

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 1CD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.24	18719		5/23/01	1500	1EA-500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention Ron L. Myers 372-1323 or Randy Thornhill 376-6769.

Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RTSteele		5/23/01 1545	DP Wohlwend	DP Wohlwend	5-23-01 1547
DP Wohlwend	DP Wohlwend	5-23-01 2013	RE Thornhill		5/23/01 2013

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquids	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION — Disposal Method (e.g., Return to customer, per lab procedure, used in process) Disposed By Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/98)

B01-007

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.23	18720		5/23/01	1500	1EA-500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By (Print)	Sign	Date/Time	Received By (Print)	Sign	Date/Time
RLSteele	RL Steele	5/23/01 1930	BCEkstrom	BCEk	5/23/01 2124
BCEkstrom	BCEk	5/23/01 2228	RE Thornhill	RE	5/23/01 2230

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquids	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION | Disposal Method (e.g., Return to customer, per lab procedure, used in process) | Disposed By | Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/98)

B01-008

B01-009

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector
Rod Shroeder

Contact/Requestor

Telephone No. 373-5810
MSIN T6-50
FAX 373-1438

SAF No.

Sample Origin
11A Hotcell

Purchase Order/Charge Code
WORK ORDER#BE4742 CACN114728 COAEF21

Project Title
AZ-101 ICD23 Shipment to PNNL

Logbook No.

Ice Chest No.

Temp.

Shipped To (Lab)
325 Laboratory PNNL

Method of Shipment

Bill of Lading/Air Bill No.

Protocol

Data Turnaround

Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.16	16029		5/31/01	1400	1EA/Sound/Glass		

B

Hold Time

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS
Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RA Steele	RASteele	5/31/01 1530	BEkstrom		5/31/01 1830
BEkstrom		5/31/01 2004	RE Thornhill	RE[illegible]	5-31-01 2004

Matrix*
S = Soil
SE = Sediment
SO = Solid
SL = Sludge
W = Water
O = Oil
A = Air
DS = Drum Sol
DL = Drum Liq
T = Tissue
WI = Wipe
L = Liquid
V = Vegetati
X = Other

FINAL SAMPLE DISPOSITION
Disposal Method (e.g., Return to customer, per lab procedure, used in process)
Disposed By
Date/Time

* ...ples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

BC-6000-828 (

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rod Schroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
400,26	15958		5/31/01	1400	1 EA/500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RF STEELE	RFSteele	5/31/01 1545	BCEkstrom	BCEkstrom	5/31/01 1545
BC Ekstrom	BCEkstrom	5/31/01 2004	RE Thornhill	RETh	5-31-01 2004

Matrix*

S = Soil
SE = Sediment
SO = Solid
SL = Sludge
W = Water
O = Oil
A = Air
DS = Drum Solid
DL = Drum Liquid
T = Tissue
WI = Wipe
L = Liquid
V = Vegetation
X = Other

FINAL SAMPLE DISPOSITION

Disposal Method (e.g., Return to customer, per lab procedure, used in process)

Disposed By

Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

BC-6000-828 (04/

B01-010

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.21	18723				1EA/500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS Hold Time

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Relinquished By Print Sign	Date/Time	Received By Print Sign	Date/Time
RLChambers	5-30-01 1610	B Campbell	5-30-01 1610
B Campbell	5-30-01 2010	RE Thornhill	5-30-01 2010

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquids	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION — Disposal Method (e.g., Return to customer, per lab procedure, used in process) — Disposed By — Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/98)

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shreeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.20	18722				1EA/Scand/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Hold Time

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RL Chambers	RL Chambers	1610 5-30-01	B Campbell	B Campbell	1610 5-30-01
B Campbell	B Campbell	5-30-01 2010	RE Th[illegible]	[illegible]	2010 5-30-01

Matrix*

S = Soil
SE = Sediment
SO = Solid
SL = Sludge
W = Water
O = Oil
A = Air
DS = Drum Solids
DL = Drum Liquids
T = Tissue
WI = Wipe
L = Liquid
V = Vegetation
X = Other

FINAL SAMPLE DISPOSITION

Disposal Method (e.g., Return to customer, per lab procedure, used in process)

Disposed By

Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

BC-6000-828 (04/98)

B01-013

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.3	18726				1EA/500ml/glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Hold Time

Ship to 325 Laboratory PNNL attention Ron L. Myers 372-1323 or Randy Thornhill 376-6769.

Deliver to North East Door (HRLF Door).

Relinquished By Print / Sign	Date/Time	Received By Print / Sign	Date/Time
RL Chambers/RL Chambers	2105 5-30-01	B Campbell / B Campbell	2105 5-30-01
B Campbell / B Campbell		RE Thornhill	2210 5/30/01

Matrix*
S = Soil
SE = Sediment
SO = Solid
SL = Sludge
W = Water
O = Oil
A = Air
DS = Drum Solids
DL = Drum Liquids
T = Tissue
WI = Wipe
L = Liquid
V = Vegetation
X = Other

FINAL SAMPLE DISPOSITION | Disposal Method (e.g., Return to customer, per lab procedure, used in process) | Disposed By | Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

B01-014

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	•	Date	Time	No./Type Container	Sample Analysis	Preservative
460.18	18725				1EA/500ml glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Hold Time

Ship to 325 Laboratory PNNL attention Ron L. Myers 372-1323 or Randy Thornhill 376-6769.
Deliver to North East Door (HRLF Door).

Relinquished By (Print / Sign)	Date/Time	Received By (Print / Sign)	Date/Time
RL Chambers / RLChambers	2105 5-30-01	B Campbell / B Campbell	2105 5-30-01
B Campbell / B Campbell	2210 5-30-01	RE Thornhill / RE Thornhill	2210 5/30/01

Matrix*

S	= Soil	DS	= Drum Solid
SE	= Sediment	DL	= Drum Liquid
SO	= Solid	T	= Tissue
SL	= Sludge	WI	= Wipe
W	= Water	L	= Liquid
O	= Oil	V	= Vegetation
A	= Air	X	= Other

FINAL SAMPLE DISPOSITION

Disposal Method (e.g., Return to customer, per lab procedure, used in process)

Disposed By

Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

BC-6000-828 (04/

B01-015

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.30	18727				1EA/500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By (Print)	Sign	Date/Time	Received By (Print)	Sign	Date/Time
RT Steele		5/30/01 1500	V. Matthews	V. Matthews	1500 5-30-01
V. Matthews	V. Matthews	2000 5-30-01	RE Thornhill	RE	5/30/01 2000

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquid	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION | Disposal Method (e.g., Return to customer, per lab procedure, used in process) | Disposed By | Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

Color: Customer

BC-6000-828 (04/

B01-016

CHAIN OF CUSTODY/SAMPLE ANALYSIS REQUEST

C.O.C. No.

Page 1 of 1

Collector Rob Shroeder	Contact/Requestor	Telephone No. 373-5810 MSIN T6-50 FAX 373-1438
SAF No.	Sample Origin 11A Hotcell	Purchase Order/Charge Code WORK ORDER#BE4742 CACN114728 COAEF21
Project Title AZ-101 ICD23 Shipment to PNNL	Logbook No.	Ice Chest No. Temp.
Shipped To (Lab) 325 Laboratory PNNL	Method of Shipment	Bill of Lading/Air Bill No.
Protocol	Data Turnaround	Offsite Property No.

Sample No.	Lab ID	*	Date	Time	No./Type Container	Sample Analysis	Preservative
460.28	18724				1EA/500ml/Glass		

POSSIBLE SAMPLE HAZARDS/REMARKS (List all known wastes) MSDS ☐ Yes ☒ No

SPECIAL INSTRUCTIONS

Ship to 325 Laboratory PNNL attention
Ron L. Myers 372-1323 or Randy Thornhill
376-6769.
Deliver to North East Door (HRLF Door).

Hold Time

Relinquished By Print	Sign	Date/Time	Received By Print	Sign	Date/Time
RL Chambers	RL Chambers	5/30/01 1545	V. Mathews	V. Mathews	5-30-01 1545
V. Mathews	V. Mathews	2000 5-30-01	RE Thornhill		5/30/01 2000
Relinquished By		Date/Time	Received By		Date/Time
Relinquished By		Date/Time	Received By		Date/Time

Matrix*

S = Soil		DS = Drum Solids	
SE = Sediment		DL = Drum Liquids	
SO = Solid		T = Tissue	
SL = Sludge		WI = Wipe	
W = Water		L = Liquid	
O = Oil		V = Vegetation	
A = Air		X = Other	

FINAL SAMPLE DISPOSITION	Disposal Method (e.g., Return to customer, per lab procedure, used in process)	Disposed By	Date/Time

All samples containing hazardous materials shall be picked up by requestor and returned to parent container or site of origin.

DISTRIBUTION: White - Remain with Samples Color - Customer

B2

RECEIPT INSPECTION TEST INSTRUCTION

TI-RPP-WTP-087, Rev. 0

[redacted]

Battelle Test Instruction	**Document No.: TI-RPP-WTP-087** **Rev. No.: 0**
Title: AZ-101 Sample Inspection	
Work Location: HLRF/SAL	**Page** 1 of 8
Author: Paul Bredt	**Effective Date: Upon final signature** **Supersedes Date: New**
Identified Hazards: ___Radiological ___Hazardous Materials ___Physical Hazards ___Hazardous Environment ___Other:	**Required Reviewers:** _X_Author _X_Technical Reviewer ___Quality Engineer ___Project Manager ___Client

Approval	**Signature**	**Date**
Author	Paul B	6/11/01
Technical Reviewer		6/11/01

Completed TASK 6-12-01

Applicability

This test instruction describes work to be performed under Project 42365 Tasks 2.01. Samples of actual Hanford waste from Tank AZ-101 were received in the 325 HLRF for testing. Information on these samples is provided in Attachment 1. This test instruction provides a method for documenting the condition of the as-received samples in accordance with Technical Procedure 41500-004, "Sample Compositing". This document is a mechanism for the cognizant scientist to communicate to technical staff specifics on procedure implementation. Therefore, this Test Instruction qualifies as a Test Instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction a cognizant scientist.

Work with actual tank material will be performed in radiological hot cells.

Justification/Test Objectives/Success Criteria

The initial objective of this test instruction is to document the condition of the samples shipped from the Hanford 222-S laboratory to the 325 HLRF. Information to be documented includes the following:

1) Jar numbers
2) Condition of the jars and lids
3) Jar masses
4) Appearance of the solids (approximate volume, color, texture, gel, and/or other observations)
5) Appearance of the liquids (approximate volume, color, turbidity, and/or other observations)
6) Inspection for a separable organic layer

The second objective of test instruction is to obtain a subsample of AZ-101 supernatant for quantification of LiBr content. Supernatant (~2ml) will be removed from each bottle containing material from segments 1 through 16. The bottles will be weighed before and after removal of this supernatant.

Finally, lids on the jars will be replaced. Lids currently on the jars have shown poor radiation resistance leading to cracking or loss of sealing ability. If radiation tolerant lids (currently on order) are available at the time of this work, the old lids will be replaced and the bottles will be reweighed.

Quality Control

This work is to be conducted under the quality requirements of PNNL's Standards-Based Management System (SBMS). Changes to the test instructions can be made only by the cognizant scientist and will be documented by crossing out the original information with a single line, and recording, initialing, and dating the changes. The results of all measurements will be recorded in this Test Instruction, a Laboratory Record Book (LRB), or bench sheet. Copies of the completed Test Instruction, bench sheets, and LRBs will be transferred to the project file upon completion of the project.

Spill Mitigation

When working with liquids and slurries, there is a risk of sample loss through inadvertent spills. During hot cell work, spills primarily result from dropped or tipped sample bottles, broken glassware, and the failure of transfer equipment. In most cases, spills can be controlled and material losses minimized through the use of secondary containment and other good laboratory practices. The cognizant scientist is responsible for working with hot cell staff to reduce the potential and programmatic impact of spills. Specific examples include:

1) Bottle holders to stabilize jars during sample transfers
2) Catch pans below homogenization vessels and primary sample containers
3) Safety coated jars and bottles (when this does not interfere with analytical requirements)
4) Plastic centrifuge liners

If a spill does occur, every practical effort will be made to recover as much of the sample as possible.

M&TE List

Balance: Calibration ID 111319277

Expiration Date 2-14-02

Balance Location A cell North

Record daily balance check information below:

Date	Actual (g)	Measured (g)	Tolerance (g)	Acceptable (Y/N)
06-11-01	1000.01	1000.02	±.01	yes
06-12-01	1000.01	1000.01	±.01	yes

6/12/01

Work Instructions

1) The following table contains a list of samples received from the 222-S laboratory. Video tape each of the bottles and complete the table.
Tape Label: A2-101 Sample Inspect. Signature rg Swoboda Date 6/11/01

Jar ID	Gross Mass (g)	Bottle and Lid in good Condition (a)? ✱	Organic Layer? (b)	Appearance of Solids (approximate volume, color, texture, gel, other observations)	Appearance of Liquid (approximate volume, color, turbidity, other observations)
18729	808.75	y	No	~ < ½ g very few white flakes on bottom	Clear Liquid
18725	810.21	y	No	Lots more ~1-2g of white solids	Clear Liquid .
18726	808.45	y	No	< ½ g very few white solids	Clear Liquid
18728	804.47	y	No	< ¼ very few white fine solids	Clear Liquid
18727	805.70	y	No	< ¼ g very few white solids (fine)	Clear liquid
18724	805.98	y	No	< ½ g of white solid like snow flakes	Clear Liquid
18723	818.77	y	No	< ¼ g of white solids like snow flakes	Clear liquid
18720	824.09	Good Lid y	No	white & dark flacky ppt. on Bottom ~1-2g	Clear liquid
18722	818.08	y	No	< ¼ g of white solids (fine)	Clear Liquid
18721	813.09	y	No	approx. 2g of chunky white solids -	Bottle Leaked - white solids on side Clear liquid
18719	797.20	y	No	very Small amt of dark ppt. on Bottoms	Clear liquid

All bottles Essentially full to top.
rgs

✱ - current Lid is OK but will be replaced with Thermal set Lid (Green) 6/12/01 rgs

The following 4 Bottles (250mL wide mouth JARS) all have heavy Dark Sludge constituent.

Jar ID	Gross Mass (g)	Bottle and Lid in good Condition wt (a)?	Organic Layer? (b)	Appearance of Solids (approximate volume, color, texture, gel, other observations)	Appearance of Liquid (approximate volume, color, turbidity, other observations)
16029	436.46	with New lid 449.46	not possible	~½ full - didn't Leak, heavy black greasey Sludge	No visible free liquid - just gooey black sludge.
17023	500.91	New lid 513.07	↓	Bottle Leaked - very dark, hard to c	There is some light liquid above the black sludge but not possible to determine amt,
* 15958	with Busted lid 347.76	New Lid 364.81	↓	Lid busted & fell inside along with Liner.	< 5mL of free Liquid remaining, mostly sludge remaining < 50% full,
17025	588.76	[illegible] 601.62	↓	Bottle Leaked; Probably heavy Sludge	Some Liquid on top of black Sludge but Not possible to determine amt.

(a) If bottle and lid are not in good condition, describe the condition below including sample ID. Replace lids as needed, and transfer material to new bottles if needed. Label any new lids or bottles with the old sample number and document the new tare for the bottle.

(b) If organic layer is found, describe below. Include depth of layer, appearance, and bottle ID

Comments and Additional Observations (add additional pages if necessary):

* 15958 – the poly propyline lid was brittle from radiolysis of sample. Replaced this black lid with green thermal set lid. Also replaced lids on other three 250mL jars.

2) If radiation tolerant lids are available, replace the lids on each of the bottles and reweigh. Record the new gross mass below.

Signature ______ Date 6/12/01

Jar ID	Gross Mass with new lid (g)
18729	819.20
18725	819.63
18726	818.26
18728	814.59
18727	816.24
18724	816.10
18723	828.47
18720	824.14
18722	827.92
18721	822.80
18719	[illegible] 6/13/01 ~~807.62~~ 807.62
16029	449.46
17023	513.07
15958	364.81
17025	601.62

3) Obtain 11 small clean vials from Mike Urie.
   i. Label the small vials as indicated in the table below
   ii. Weigh the small vials (with caps) to the nearest 0.01 g. Record the mass data in the table below.
   iii. Transfer ~2ml of supernatant from each of the respective as-received jars to the respective small vial
   iv. Weight the loaded small vials to the nearest 0.01 g. Record the mass data in the table below.
   v. Reweigh the as-received (parent) jar to the nearest 0.01 g. Record the mass data in the table below.

Signature ______ Date 6/12/01

ASR 6132	As-Received Jar ID	Small Vial ID	Small Vial Tare (g)	Small Vial with Sample (g)	Sample mass in Small Vial (g)	New As-Received Gross Mass (g)
DI-01381	18729	18729 LiBr	6.49	8.59	2.10	817.08
2	18725	18725 LiBr	6.53	8.63	2.10	817.00
3	18726	18726 LiBr	6.44	8.61	2.17	815.90
4	18728	18728 LiBr	6.49	9.43	2.94	811.16
5	18727	18727 LiBr	6.53	8.49g	1.96g	814.16
6	18724	18724 LiBr	6.51	8.74	2.23	813.81
7	18723	18723 LiBr	6.51	9.93	3.42	824.99
8	18720	18720 LiBr	6.48	8.72 ~~8.67~~ rgs	2.24 ~~2.19~~ rgs 6/13/01	821.90
9	18722	18722 LiBr	6.51	8.96	2.45	825.35
90	18721	18721 LiBr	6.45	8.67	2.22	820.47
1	18719	18719 LiBr	6.52	8.13	2.21	805.33
	~~16029~~	~~16029 LiBr~~				
	17023	17023 LiBr				
	15958	~~15958 LiBr~~				
	~~17025~~	17025 LiBr				

Not required on sludge sample fractions rgs

PRB 6/12/01

Attachment 1

Sample information received from staff at 222-S

Composite Jar				Content Jar				
Number	Size (mL)	Tare Weight (g)	Net Weight (g)	Number	Segment	Tare Weight (g)	Net Weight (g)	Physical State
18729	500	288.66	521.05	18402	10	304.84	369.45	Supernatant
				18413	16	456.89 a	151.69	Supernatant
18725	500	294.6	516.37	18406	9	305.49	365.74	Supernatant
				18401	15	303.41	106.47	Supernatant
				18413	16	608.58 a	44.22	Supernatant
18726	500	286.48	522.64	18403	11	303.36	365.33	Supernatant
				18413	16	299.42	157.47	Supernatant
18728	500	288.94	516.16	18407	8	301.12	366.05	Supernatant
				18401	15	409.88 a	150.22	Supernatant
18727	500	286.57	519.89	18393	7	298.65	360.69	Supernatant
				18400	14	301.5	58.03	Supernatant
				18401	15	560.1 a	102.08	Supernatant
18724	500	289.79	516.99	18391	6	301.17	361.81	Supernatant
				18400	14	359.53 a	155.29	Supernatant
18723	500	295.92	523.69	18394	5	300.49	366.57	Supernatant
				18408	13	303.44	40.5	Supernatant
				18400	14	514.9 a	116.83	Supernatant
18720	500	302.6	522.2	18399	4	298.04	359.3	Supernatant
				18408	13	343.94 a	163.06	Supernatant
18722	500	296.95	521.86	18398	3	304.08	345.82	Supernatant
				18390	12	302.76	30.44	Supernatant
				18408	14	507 a	146.43	Supernatant
18721	500	296.26	521.13	18397	2	302.22	352.1	Supernatant
				18390	12	333.2 a	169.45	Supernatant
18719	500	290.46	508.24	18396	1	299.7	344.6	Supernatant
				18390	12	502.64 a	163.7	Supernatant
16029	250	218.07	220.09	17331	17ALH	122.86	129.4	Wet Sludge
				15888	17AUH	130.71	90.69	Sludge-Slurry
17023	250	217.7	307.2	same	17		NA	Solids
* 15958	250	216.7	133.7	same	18		NA	Solids
17025	250	217.07	388.2	same	18A		NA	Solids
Total			**958.5** **5800.91**					**Solids** **Supernate**

Note: For core segment 17, the upper half (17AUH) was counted as liquid and the lower half (17ALH) as solids.

a-Denotes the weight of a bottle that is not completely empty; not a tare weight.

* - See attached e-mail on tare wts. rjs

From: Myers, Ronald L
Sent: Tuesday, June 12, 2001 2:27 PM
To: Bredt, Paul R
Cc: Kurath, Dean E
Subject: AZ-101 Tare weights

Paul,

The tare weights you requested are:

Bottle # 15958	216.7 g
# 17023	217.7 g
# 17025	217.07 g

Ron

Ronald L. Myers
Sr. Research Engineer
Radiochemical Science & Engineering
Mail Stop P7-28
372-1323

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX C

LITHIUM BROMIDE TESTING

2004

Battelle - Pacific Northwest Division

C1

ASR 6132

~~Record Copy~~
KS 7/31/02

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature MW Urie
Print Name M. W. URIE
Phone 376-9454 MSIN A7-22

PNNL Project #: 42365
Charge Code: SEE SPCL INST
Date Required: 6-29-01

Matrix Type Information

- **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ___ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ___
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) ✓ (EXIST QC ONLY)
- **Additional QA Requirements?** No ✓
  or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ✓ Yes ___
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ___
  or Reference Doc # ___
  or Previous ASR # ___
  or Previous RPL ID # 01-01392 TO 01-01402

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ **Yes** ___

Additional or Special Instructions WTP

Send Report To M. URIE **Phone** 376 9454
Phone ___

Preliminary results requested, as available? No ___ Yes ✓ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: TO BE MOVED FROM
Delivered By (optional) HERF TO SAL 6/14/01
Time Delivered (optional) ___
Group ID (optional) ___
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: SAL
ASR Number: 6132
RPL Numbers: (01-01381)-(01-01391)

RPG/CMC Work Accepted By: MW Urie **Signature/Date:** MW Ur 6-14-01

ASR FY2000 - RPG1.doc

FVH, MJS, JJW, DRS, MWU, KNP

C01-002

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
01-01381	18729 LiBr		Li, Br		
82	18725 LiBr			See	
83	18726 LiBr	AZ-101 As-		Special Instructions	
84	18728 LiBr	received			
85	18727 LiBr	Supernatant			
86	18724 LiBr				
87	18723 LiBr				
88	18720 LiBr				
89	18722 LiBr				
90	18721 LiBr				
91	18719 LiBr				

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6132

Page 1 of 1

Special Instructions: ASR 6132
Lithium and Bromide Analyses for AZ-101 As-received Supernatants

AZ-101 Samples 01-01381 through 01-01391 – LIMS Tests

Digestion-128	SAL	-- Use W57930
ICP-211-CMC	LAB	-- Use CMC K88407
Sub-Sample	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406

NEED VERY RAPID TURNAROUND TIMES FOR BOTH PREPARATION AND ANALYSIS. PROVIDE PRELIMINARY RESULTS TO M.W. URIE.

1. SAL receives from HLRF a 2 mL sub-sample from each of the AZ-101 as received jars containing only liquid for Lithium and Bromide Analysis.
2. Obtain ICP spiking solution from J. Wagner
3. SAL – For each sample.

   ICP Analysis:
   a. Digest 0.25 mL per method ALO-128.
   b. Digestion preparation blank and LCS with batch
   c. Select one of the samples and digest as a duplicate
   d. Select one of the samples, add a matrix spike, and digest
   e. Contact J. Wagner for quantity of digestate to transfer to ICP lab.

   IC Analysis:
   f. Obtain water diluent from IC lab staff.
   g. Aliquot 0.5 mL into a 25-ml Volumetric flask and dilute with water diluent
   h. Select one of the samples and dilute for a matrix spike (the MS solution will be added by the lab)
   i. Select one of the samples and dilute as a duplicate
   j. Transfer 0.5 mL of each diluted sample to IC lab
   k. Transfer 5 mL of water diluent back to IC lab

4. Lab – For each sample
   a. Target MRQ is 0.003M lithium or bromide
   b. QC requirements: process/diluent blank, LCS, matrix spike, and instrument QC criteria meeting the on-line QA Plan for lithium and bromide.
   c. Report all analytes measured; however, preparation QC is required only for lithium and bromide

C2

IC ANION RESULTS - ASR 6132

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	M. Urie	Charge Code/Project:	W57932/42365
ASR Number:	6132	Sample Receipt Date:	06/14/2001
Sample Prep Date:	N/A	Sample Analysis Date:	07/05/2001
Analyst:	MJ Steele		

Preparation Procedure: N/A
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Sample (01-01381 through 01-1391) Final Results:

RPL Number	Sample ID	Br EQL ug/mL	Br ug/ml	Exceed Target?
Target: 0.003 M Br			240	
01-1381 IC PB	IC Prep Blank	0.13	< 0.13	n/a
01-1381 Hot Cell PB	Hot Cell Prep Blank	0.14	< 0.13	n/a
01-1381	18729 LiBr	126	1,160	Y
01-1382	18725 LiBr	126	950	Y
01-1383	18726 LiBr	126	1,200	Y
01-1384	18728 LiBr	126	420	Y
01-1384 Dup	18728 LiBr Dup (Hot Cell)	126	440	Y
	RPD		5%	
01-1385	18727 LiBr	126	920	Y
01-1386	18724LiBr	196	1,240	Y
01-1387	18723LiBr	84	200	N
01-1388	18720LiBr	84	410	Y
01-1389	18722 LiBr	126	1,090	Y
01-1390	18721LiBr	126	740	Y
01-1391	18719 LiBr	84	230	N
01-1391 Dup	18719 LiBr Dup (Lab)	84	230	N
	RPD		0%	
01-1390 MS(PS) % Rec	18721LiBr MS(PS)		102%	
LCS % Rec			101%	

The samples were prepared for ion chromatography anion analysis by diluting to a level to allow separation of the bromide and nitrate peak. The typical dilution was from 670-fold to 1570-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the bromide results are provided in the table. The target bromide concentration is 0.003 M or about 240 μg/mL. Values in excess of this concentration indicate that sample could have been contaminated by the hydrostatic head fluid used for obtaining the AZ-101 core sample. Based on the IC results only, contamination is possible; however, an evaluation of the IC and ICP results will be required before contamination is verified.

Q.C. Comments:

Duplicates: No duplicate was provided. However, the sample 18728 LiBr (01-01384) was split in the hot cell and sample 18719 LiBr (01-01391) was split in the laboratory and analyzed in duplicate. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20%.

Matrix Spike (HCV 010328): A matrix spike was prepared and the bromide recovery was within the 75% to 125% recovery acceptance criteria.

Laboratory Control Sample -LSC (Blank Spike): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared and measured at the same time as the Matrix Spike sample and demonstrated recoveries for bromide within the 90% to 110% acceptance criteria.

System Blank/Processing Blanks: Six system blanks were processed during the analysis of the liquid sample. No bromide was detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards: Six mid-range verification standards were analyzed throughout the analysis runs. Bromide recoveries were within the acceptance criteria from 90% to 110% for the verification standard.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: MW Ur Date 7-11-01

Review/Approval: MJ Steele Date 7-17-01

Archive Information:

Files: ASR 6132 Urie.doc ASR 6132, 6155.xls

C3

ICP-AES RESULTS - ASR 6132

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Project / WP#:	42365 / K88407
ASR#:	6132
Client:	M. Urie
Total Samples:	11

	From	To
RPL#:	01-01381	01-01391
Client ID:	"18729 LiBr"	"18719 LiBr"
Sample Preparation: PNL-ALO-128/SAL/vh (~0.25mL/25mL) [AZ-101 as received supernatant]		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 06-29-01 (A0691)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1 (Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

Jerry Wagner 7-16-01
Reviewed by

MW Urie 7-17-01

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Concur

Eleven AZ-101 "as received supernatant" samples, **18729-LiBr** through **18719-LiBr** (RPL# 01-01381 through 01-01391), was processed in two batches by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-128 acid digestion of liquids. Samples were prepared using approximately 0.250mL (~0.30g) of liquid supernatant material. After digestion the digestate was diluted to a final volume of approximately 25mL. All sample aliquots and digestate solutions were weighed. Digestate solution volume was determined by dividing the weight of digestate by its density. Density of sample was measured therefore the volume is assumed to be 0.25mL. A 1mL pipette was used to determine the density of each digestate solution. The total digestate volume was calculated by dividing the weight of digestate by its density. Supernatant density of samples is estimated to be approximately 1.20 g/mL. Density of digestate solutions is about 1.05 g/mL.

Lithium was the only analyte requested. Lithium was detected in only one sample, **18724-LiBr** (RPL# 01-01386) and was below EQL (EQL=12.5 µg/mL) and Target MRQ (0.003M or 20.82 µg/mL). All other analytes reported are for information only.

ICPAES results are reported in µg/mL and have been corrected for analytical dilution and sample processing. Analytes that were generally above EQL include Al, B, Cr, K, Mo, Na, P, and Si. The process blanks appear to have a small amount of B, Na and Si. The B, Na, and Si may come from glassware used to digest the samples or from the digestion reagents (nitric acid or hydrochloric acid). The Na concentration in the process blanks is far below that found in the samples (< 0.3%). Boron and Si in the samples is approximately the same concentration as found in the process blanks.

See attached ICPAES Data Report for final results.

Quality control check-standard results met tolerance requirements for analyte of interest (Li). Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. LCS/BS, Matrix-spikes and duplicates were prepared with each batch of samples and analyzed in this report for lithium only.

Matrix-spike and LCS/blank-spiked samples were prepared using 1mL and 3mL respectively of a 5µg/mL lithium solution per 25mL sample digestate volume.

Five fold serial dilution:

All analytes were within tolerance limit of 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 20% RPD for all analytes greater than EQL except B and Si which are not an analytes of interest.

Post-Spiked Samples (Group A):

All post-spiked analytes were recovered within tolerance of 75% to 125% except Si, which is not an analytes of interest.

Post-Spiked Samples (Group B):

All post-spiked analytes were recovered within tolerance of 75% to 125%.

Blank Spike:

Blank-Spike recovery for Li (97% and 103%) was within tolerance of 80% to 120%.

Matrix Spiked Sample:

Matrix-Spike recovery for Li (95% and 96%) was within tolerance of 75% to 125%.

Quality Control Check Standards:

Concentration of Li and all other analytes are within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV with the following exceptions.
Palladium measured (-12% to -26%) lower than expected in QC_MCVB.
Tin measured (-11% to -34%) lower than expected in QC_MCVB.
Europium measured (10% to 14%) higher than expected in QC_MCVB.
All analytes are within tolerance limit of ± 10% accuracy in QCSSTMCV.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except for potassium. Potassium measurements were about 6% to 7% high in the QC_SST standard. All other analytes measured were within tolerance.

Process Blanks:

Concentration of Li and all other analytes detected is within tolerance limit of ≤EQL or less than ≤5% of the sample except B, Na, and Si (not analytes of interest).

Laboratory Control Standard (LCS):

See "LCS/blank spike" above.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	83.1	82.6	85.0	88.2	85.9
	RPL/LAB #=	01-1381-B	01-1381	01-1381-DUP	01-1382	01-1383
	Client ID=		*18729-LiBr*	*18729-LiBr*	*18725-LiBr*	*18726-LiBr*
Det. Limit	*Run Date=*	6/29/2001	6/29/2001	6/29/2001	6/29/2001	6/29/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	[15]	5,860	5,760	5,740	7,090
0.250	As	--	--	--	--	--
0.050	B	132	165	193	196	200
0.010	Ba	--	--	[1.1]	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	[22]	[22]	[21]	[26]	[18]
0.250	Ca	--	--	[23]	--	--
0.015	Cd	[3.1]	[1.7]	--	[1.3]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	577	567	555	684
0.025	Cu	[4.4]	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[3.7]	[16]	--	--	--
2.000	K	--	3,570	3,530	3,420	4,240
0.050	La	--	--	--	--	--
0.015	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	75.4	74.4	72.7	89.7
0.150	Na	174	87,500	87,600	85,600	>94544
0.100	Nd	--	--	--	--	--
0.030	Ni	--	[2.6]	--	--	--
0.100	P	--	372	367	359	443
0.100	Pb	[10]	[11]	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	422	521	511	[420]	761
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[5.9]	[5.8]	--	[4.7]	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Data (1) from ~A0691 M.URIE ASR-6132.XLS

7/16/2001 @ 5:18 PM

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	85.1	83.6	83.5	84.1	88.3
	RPL/LAB #=	01-1384	01-1385	01-1386	01-1387	01-1388
	Client ID=	*18728-LiBr*	*18727-LiBr*	*18724-LiBr*	*18723-LiBr*	*18720-LiBr*
Det. Limit	*Run Date=*	6/29/2001	6/29/2001	6/29/2001	6/29/2001	6/29/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	6,240	5,640	6,170	6,840	6,140
0.250	As	--	--	--	--	--
0.050	B	134	133	168	130	200
0.010	Ba	--	--	--	[1.6]	[1.1]
0.010	Be	--	--	--	--	--
0.100	Bi	[21]	[22]	[17]	[20]	[21]
0.250	Ca	--	--	--	--	--
0.015	Cd	[1.3]	--	--	[1.3]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	572	544	574	641	558
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[6.3]	--	--	93.8	[3.0]
2.000	K	3,500	3,320	3,590	3,840	3,570
0.050	La	--	--	--	--	--
0.015	Li	--	--	[2.7]	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	[5.4]	--
0.050	Mo	74.4	71.0	75.2	83.1	74.0
0.150	Na	88,200	84,300	88,800	>92507	90,900
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	46.9	--
0.100	P	369	348	374	405	360
0.100	Pb	--	--	--	[8.5]	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	661	525	483	460	556
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[4.7]	--	--	[5.0]	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit	Multiplier=	87.7	80.3	82.2	83.4	76.3
	RPL/LAB #=	01-1389	01-1389-B	01-1389-DUP	01-1390	01-1391
	Client ID=	*18722-LiBr*		*18722-LiBr*	*18721-LiBr*	*18719-LiBr*
	Run Date=	6/29/2001	6/29/2001	6/29/2001	6/29/2001	6/29/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	5,940	[11]	5,940	5,890	6,200
0.250	As	--	--	--	--	--
0.050	B	271	141	209	153	194
0.010	Ba	[1.1]	[1.9]	--	--	[0.92]
0.010	Be	--	--	--	--	--
0.100	Bi	[18]	[22]	[17]	[16]	[19]
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	571	--	573	555	551
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	--	[4.4]
2.000	K	3,350	--	3,360	3,230	3,540
0.050	La	--	--	--	--	--
0.015	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	74.6	--	75.0	72.4	73.3
0.150	Na	90,400	214	>90427	88,100	>83934
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	367	--	365	355	361
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	703	519	576	520	503
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Data (1) from ~A0691 M.URIE ASR-6132.XLS

7/16/2001 @ 5:18 PM

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX D

HOMOGENIZATION AND SUB-SAMPLING

2004

Battelle - Pacific Northwest Division

D1

COMPOSITING TEST PLAN:

TP-RPP-WTP-089, Rev. 1

Battelle Test Plan	Document No.: TP-RPP-WTP-089 Rev. No.: 1

Title: AZ-101 Homogenization and As-Received Characterization

Work Location: RPL	Page 1 of 29
Author: Paul Bredt	Effective Date: Upon Final Signature Supersedes Date: New
Identified Hazards: ___Radiological ___Hazardous Materials ___Physical Hazards ___Hazardous Environment ___Other:	Required Reviewers: _X_Author _X_Technical Reviewer _X_Quality Engineer _X_Project Manager _X_Client

Approval	Signature	Date
Author	Paul Bredt	8/9/01
Technical Reviewer		8/10/01
Quality Engineer	Almeida	8/9/01
Project Manager	DE Kurath	8/9/01
Bechtel	ED Lee Ernest Lee R&T	8/14/01

1 Applicability

This Test Plan describes work to be performed under Project 42365, WTP-RPP Support. Samples of actual waste from Hanford tank AZ-101 were received at Battelle's Radiochemical Processing Laboratory (RPL) for testing under this project. This Test Plan describes how these materials will be composited, homogenized, and sub-sampled for process testing and characterization tasks. This Test Plan also provides details on a select set of physical, rheological, chemical and radiochemical tests.

Client requirements for successful achievement of project data needs have been established via Task Specification TSP-24590-01-00006, "Tank 241-AZ-101 Sample Composite, Homogeneity, Analysis". This Test Plan is a mechanism for communicating Battelle's testing scheme for meeting these client expectations.

Work is to be performed by RPL technicians under the supervision of a cognizant scientist or additional technical staff. The cognizant scientist shall be responsible for implementation and adherence to this test plan. As needed, test instructions may be issued by the cognizant scientist to communicate details on implementation of existing procedures.

2 Justification

Characterization of the AZ101 feed and concentrated slurries provides an opportunity to underpin the pretreatment facility design basis. Additionally, it will provide data to the WPT to validate assumptions in the flowsheet and to support process verification testing and waste form qualification. Additionally, it is critical that the AZ-101 waste samples shipped to Battelle are homogenous before sub-sampling to ensure the analysis of the sub-samples provides information representative of the bulk sample.

3 Objectives

The objectives of these tasks are to:

- Receive and verify liquid samples were not corrupted with hydrostatic head fluid
- Composite all low LiBr liquid samples and undissolved solids from cores 17, 17A, 18, and 18A by thoroughly mixing in a compositing vessel. The compositing scheme implement within this test plan is not consistent with the compositing requirements of the Regulatory DQO, "Regulatory Data Quality Objectives Supporting Tanks Waste Remediation System Privatization Project", PNNL-12040.
- While operating the vessel agitator, extract all of the material from the compositing vessel into volume graduated jars
- Verify the homogeneity of the sub-samples by measuring the volume of settled solids and supernatant in at least five sub-samples
- Determine the wt% undissolved solids concentration in composite
- Perform PCB analysis per methods established for the Regulatory DQO
- Adjust the concentration of sub-samples to 3±1 wt%, 13±2 wt% and 20±2 wt% undissolved solids
- Measure the physical properties of the 3, 13 and 20 wt% sub-samples (see Attachment 3)
- Measure the chemical and radioisotope concentrations listed in Tables 2 and 3 of the Test Specification (see Attachment 1) in the solid and liquid fractions of the original composite
- Determine liquid fraction compliance to specification 7 of contract DE-AC27-01RV14136 (see Attachment 2)
- Determine solid fraction compliance to specification 8 of contract DE-AC27-01RV14136 (see Attachment 2)
- Report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1)
- Provide a draft comprehensive technical report within 90-days of completing all analyses

4 Success Criteria

- The analytes' analyses listed in this test plan were obtained.
- The QC target criteria in Tables 1, 4, and 5 of Attachment 1. The target criteria presented are goals for demonstrating reliable method performance. In the event of QC failures, the laboratory will follow internal procedures to resolve the issues. If the failures can not be resolved, the BNI R&T representative will be notified as soon as the inability to resolve the failures at the bench level is identified. All QC failures and limitations on the associated data shall be discussed in the narrative of the data report.
- For PCB measurements, success criteria will be identification/quantification of detectable PCBs with the QA/QC requirements specified in the "Regulatory DQO Test Plan for Determining Method Detection Limits, Estimated Quantitation Limits, and Quality Assurance Criteria for Specified Analytes", PNNL-13429, and the associated PCB test plan, TP-RPP-WTP-30.

5 Description of Test

5.1 General Instructions

5.1.1 A material balance for the AZ-101 samples will be maintained throughout the test steps defined by this Test Plan. Items to be recorded include sample bottle identification number, bottle tare weight (provided by 222-S), the mass (or volume) of sample received in each bottle, loss of sample due to residual sample left in each bottle, mass (or volume) of composite AZ-101 sample, mass and volume of chemical additions, mass (or volume) of sample removed for analysis, and any other significant activities that add or remove mass (or volume) from the AZ-101 sample. Sample identification must be traceable to the original sampling event through ASR, Test Instructions, Laboratory Record Books, etc. Depending on conditions, it is best to record both mass and volumes, although measurement of both properties may not be possible in all situations during this testing.

5.1.2 Several administrative hold points in this Test Plan require client consultation, concurrence or approval before proceeding. The client is to respond with approval by email or letter before proceeding past the hold points. This documentation is to be included in the data package delivered to the client as part of the final report.

5.1.3 Battelle personnel are to record the temperature of the cell during any data collection that could be effected solids solubility. At a minimum, temperature shall be recorded at the time of vol% settled solids measurements, and decanting of liquid from settled or centrifuged solids.

5.1.4 As appropriate, any unused material collected for characterization, will be made available for use in process testing. The task leader responsible for the as-received characterization is considered the sample custodian and will ensure that the unused material is transferred to the appropriate task leader(s).

5.1.5 Samples requiring archiving per this test plan shall be archived under an approved archiving/monitoring procedure. [Note: An archiving procedure is to be developed to support this and other RPP-WTP archiving needs in the RPL.) The purpose of the archiving procedure will be to insure sample security and integrity.

5.1.6 The following table list estimated sample volumes required for each analysis detailed in this Test Plan. Volume for physical and rheological testing assumes the initial composite sample contains at least 5 wt% undissolved solids. If the sample contains less than 5 wt% undissolved solids, the client will be contacted prior to using a greater volume of material for work described under section 5.4. Total material available is estimated to be 958 g of sludge and 5800 g of supernatant.

Volume of Initial Composite Required for Characterization Testing

Testing	Section	Bulk Slurry (mL)	Liquid Fraction (mL)	Wet Centrifuged Solids(g wet)

Chemical & Radiochem	5.5	NA	120-150	30-35
PCB Archive	5.6	NA	300-400	20
Physical Properties	5.3/5.4	3 wt% ⇒ 60 13 wt% ⇒ 260 20 wt% ⇒ 400	NA	NA
Total	NA	720	420-550	50-55

5.2 Compositing Tank Samples

5.2.1 The "as-received samples" will be inspected and weighed to determine what degree of sample loss has occurred during sample storage and transport. Report will include visual appearance of the liquid and solid /crystalline and floating phases present in each AZ-101 sample bottles. Video images of the samples are to be collected at this time as well.

5.2.2 Battelle personnel shall sub-sample the liquid in each sample jar that contains a separate liquid phase and analyze the sub-samples for lithium and bromide (only single analysis per sub-sample is required). The purpose of the analysis is to determine if the sample contains hydrostatic head fluid from the sampling process. If the concentration of either lithium or bromide is greater than 0.003 M, then the sample shall not be included in the compositing process. The criteria of 0.003 M was selected to limit the amount of hydrostatic head fluid to 1 part in 100 parts.

5.2.3 **<u>Administrative Hold Point:</u>** BNI R&T will be notified of the measured lithium and bromine concentrations before proceeding. Email or letter of concurrence from BNI R&T is to be received before proceeding.

5.2.4 All sample bottles containing liquid that meet the criteria of ≤0.003 M lithium and/or bromide and sample bottles containing undissolved solids shall be transferred to the 7.5L mixing vessel through screen with openings sized between 1/8 x 1/8 inches and 3/32 x 3/32 inches in diameter.

5.2.5 If any material remains on the sample screen after flushing the screen with the liquid samples, determine weight of material and archive material.

5.2.6 **<u>Administrative Hold Point:</u>**

5.2.6.1 If greater than 2 wt% of the settled solids content of the composite (20g for AZ-101) are collected on the screen, notify BNI representative with a proposed path forward for this material. Email or letter of concurrence from BNI R&T is to be received before proceeding.

5.2.6.2 If 2 wt% or less of the settled solids content of the composite (20g for AZ-101) are collected on the screen, testing may continue without BNI concurrence. Retain this material separate from the composite and proceed with testing. Notify BNI of the solids mass at the earliest convenience.

5.2.7 The vessel agitation system shall be operated continuously at least an hour prior to performing sub-sampling and shall continue to operate during sub-sampling.

5.2.8 The sub-sampling method and homogeneity verification method shall be based on the methods and criteria described in PNNL Test Plan for "Regulatory Data Quality Objective: Sample Compositing Strategy," TP-41500-001, Rev. 0 and shall be consistent with the following. While operating the vessel agitation system, Battelle personnel are to extract sub-samples from the AZ-101 slurry into volume graduated glass jars. Jar volumes will range from 100 to 500 mL. The order the sub-samples pulled shall be recorded. The entire contents of the compositing vessel shall be extracted as sub-samples.

5.2.9 Sub-samples are to be tightly sealed and sit undisturbed for a minimum of 24-hours.

5.2.10 Sub-samples will be inspected for floating layers. If found, the record volume or thickness and appearance will be noted and if possible, video images will be collected.

5.2.11 **<u>Administrative Hold Point:</u>** If an organic layer is observed in any of the composite batches, then BNI's R&T representative must be consulted before work continues. Email or letter of concurrence from BNI R&T is to be received before proceeding.

5.2.12 Battelle personnel are to record the volume of settled solids/crystalline material and supernatant present in each of the sub-samples after the 24 hour undisturbed period (settling times may exceed 24 hours). The volume measurement shall be reported to an accuracy of 1% of the container volume for containers >100 ml and ±1.0-ml for containers <100 ml. Record settling time and temperature at the time of the measurements.

5.2.13 Battelle personnel are to calculate the relative volume percentage of settled solids/crystalline material present in each of the sub-samples selected for homogeneity verification and compare the volume percent settled solids versus the order of sample collection.

Note:
10 vol% was chosen as a criteria over the vol% listed in the task specification. Volume graduations on typical commercially available volume graduated jars start at 10% of full scale. Approximately 11 vol% of the material received at the RPL is settled sludge. Assuming the solids will be less compacted after the homogenization activities, the final composite should contain greater than 10 vol% settled solids.

5.2.14 **Administrative Hold Point:** If the liquid sub-samples contain an average of less than 10 vol% settled solids, then the homogenization is complete. The volume settled solids data on the composite liquid sub-samples are to be presented to BNI R&T for approval before proceeding. Once this approval has been received, proceed to compositing sludge samples.

5.2.15 **Administrative Hold Point:** If the liquid sub-samples contain an average of greater than 10 vol% but less than 60% settled solids, then the following two criteria must be met before the composite sub-samples are considered homogenous. If the composite sub-samples fail the criteria, then the composite sub-samples will be returned to the mixing vessel and rehomogenized and sub-sampled again at a higher agitation rate and/or other modifications deemed necessary to obtain improved mixing. If the second homogenization fails, BNI R&T will be consulted. A data package is to be presented to the BNI R&T for approval before proceeding. The data package submitted for BNI R&T approval is to include information on any outlier eliminated from the data set reconciled against the criteria. Email or letter of concurrence from BNI R&T is to be received before proceeding.

1) Calculate a standard deviation for the entire vol% settled solids data set. This standard deviation must be no greater than 5 vol%. (Note: Outliers may be eliminated using any standard outlier test, e.g., Dixon Test; however, at least 5 sub-samples must be used for calculating the standard deviation.)
2) Calculate a best fit line for the data set (vol% settled solids versus collection order). This best fit line must not show a trend of greater than 5 vol% over the range. For example, if the linear best fit line is at 50 vol% for the first sample, then it cannot exceed 55 vol% or be below 45 vol% for the last sample (these are the values for the best fit line, not samples).

5.2.16 **Administrative Hold Point:** If the liquid sub-samples contain an average of greater than 60% settled solids, then perform the following physical properties analysis with the samples selected for homogeneity testing. Attachment 3 provides further detail on this protocol and associated calculations. Submit a data Package to BNI R&T containing the results of the following as confirmation that the sub-sampling was homogenized. The data package submitted for BNI R&T approval is to include information on any outlier eliminated from the data set reconciled against the criteria. If the composite sub-samples fail homogenization criteria, then all the composite sub-samples will be returned to the mixing vessel and rehomogenized and sub-sampled again at a higher agitation rate and/or other modifications deemed necessary to obtain improved mixing. If the second homogenization fails, BNI R&T will be consulted. Email or letter of concurrence from BNI R&T is to be received before proceeding.

5.2.16.1. A minimum of five sub-samples shall be used for determination of sub-sample homogeneity. These sub-samples for homogeneity determination shall include the second, middle and next to the last sub-samples removed from the compositing vessel. Two other sub-samples will be chosen at random.
5.2.16.2. Mix the selected sub-samples and transfer triplicate aliquots of the selected homogeneity sub-samples into volume graduated centrifuge cones (a total of 15 cones).
5.2.16.3. Allow the triplicates to settle for 3 days. Record the total mass and volume and the volume of the settled solids.

> Note:
> Measurement of the settled supernatant density requested in the Test Specification has been omitted. Supernatant density will be determined under step 5.2.16.7 following centrifugation. More supernatant is available after centrifugation, and supernatant loss during step 5.2.16.4 could effect vol% centrifuged solids calculations.

5.2.16.4. Calculate the volume percent settled solids and bulk density.
5.2.16.5. Centrifuge the settled slurries at approximately 1000 times the force of gravity for 1 hour. Measure and record the mass and volume of the centrifuged solids and volume of centrifuged liquid. Calculate the solids density.
5.2.16.6. Plot the vol% centrifuged solids against the sub-sample removal sequence.
5.2.16.7. Transfer all of the centrifuged supernatant into a graduated cylinder and measure its mass and volume. Calculate the supernatant density.
5.2.16.8. Dry the centrifuged solids and supernatant at 105°C to constant weight. Determine wt% solids, wt% dissolved solids and wt% undissolved solids as detailed in attachment 3.

5.3 Chemical and Radiological Characterization of Composite Sub-Sample

5.3.1 If the composite contains less than 60 vol% settled solids, a subsample will be mixed and sampled in triplicate to determine the slurry density, supernate density, wt% total solids, wt% dissolved solids and wt% undissolved solids. See Attachment 3 for details on the physical properties protocol and associated calculations.

5.4 Physical and Rheological Characterization at the Expected Receipt, Mid-Range and Maximum Concentration

Administrative Hold Point: It is estimated that the testing detailed in this section will required 720 ml of as-received composite. This estimate is based on an assumption of 5 wt% undissolved solids in the as-received composite (analyses will be conducted under section 5.3 or 5.2.16). If the undissolved solids content is less than 5wt%, then client will be contacted prior to using more than 750 ml of the as-received composite for this work (In this case, Email or letter of concurrence from BNI R&T is to be received before proceeding on testing with more material). At least 75% of this material will be returned for process testing.

5.4.1 Settle and decant supernatant from one sub-sample designated for the maximum-range concentration sub-sample until a final concentration of 20±2 wt% undissolved solids is reached. To reach this concentration, it may be necessary to augment settling by centrifuging the sub-sample. Archive the supernatant. The amount of supernatant to be decanted shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample.

5.4.2 Settle and decant supernatant from sub-sample designated to be the mid-range concentration sub-sample until a final concentration of 13±2 wt% undissolved solids is reached. Archive the supernatant. The amount of supernatant to be decanted shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample.

5.4.3 Adjust the concentration of the sub-sample designated for the receipt concentration by adding liquid to the sub-sample to reach a concentration of 3±1 wt% undissolved solids. The amount of supernatant to be added shall be determined by calculation based on the undissolved solids concentration in the original composite sub-sample.

5.4.4 The three sub-samples prepared in steps 5.4.1, 5.4.2, and 5.4.3 (20, 13, 3 wt% respectively) shall be analyzed in triplicate to determine slurry density, wt% total solids, wt% dissolved solids, wt% undissolved solids, yield strength, shear stress vs shear rate, and heat capacity per Table 1 in attachment 1. Particle size distribution and SEM analysis need only be conducted on one of the three samples.

5.5 Chemical and Radiological Characterization of Sub-Sample

The chemical and radiological characterization of the sub-sample shall be initiated by a "standard" laboratory Analytical Service Request with special instructions to direct laboratory staff on quantity of sub-sample to process, phase separation requirements, analysis requirement for the liquid and wet centrifuged solid phases, and the QC criteria for the analyses. All analyses will be performed on triplicate aliquots from one homogeneous sub-sample. **Steps 5.5.1 through 5.5.12 summarize the ASR documentation.**

5.5.1 Sub-sample is to be phase separated by centrifuging for 1 hour at approximately 1000x G and decanting the liquid phase.

5.5.2 Determine the volume and weights of the liquid and wet centrifuged solids, and calculated the density of the centrifuged liquid, the density, vol%, and wt% of the wet centrifuged solids.

For Decanted Liquids

5.5.3 Perform a Total Dissolved Solids (TDS) measurement (i.e., dry liquid at 105 °C to constant weight) and Wt% Oxide measurement (i.e., following TDS, heat in air at 1050 °C to constant weight.)

5.5.4 Determine concentration of analytes in Table 2 using QC parameters defined in Table 4. (Note: Extend GEA counting times necessary to obtain MRQs for some analytes.)

For Wet Centrifuged Solids

5.5.5 Perform a Wt% Solids measurement (i.e., dry wet centrifuged solids at 105 °C to constant weight) and Wt% Oxide measurement (i.e., following Wt% Solids, heat in air at 1050 °C to constant weight.)

5.5.6 Using the TDS data from the Decant Liquid analysis and the Wt% Solids data from the Centrifuged Solids analysis, calculate the weight fraction of undissolved solids and the weight fraction of soluble solids in the Centrifuged Solids per the equations provided in the Test Specification TSP-24590-01-00006. .

5.5.7 Prepare centrifuged solids for analysis by both an acid digestion and a fusion method.

5.5.8 **Administrative Hold Point:** Review the results from all dissolution methods, contact the BNI R&T representative if there is concern that the sample material was not completely dissolved by either dissolution method used.

5.5.9 If after the either the acid digestion or fusion dissolution process the solids aren't completely dissolved, then archive the undissolved solids for possible future analysis.

5.5.10 Determine concentration of analytes in Table 3 using QC parameters defined in Table 5. (Note: Extended GEA counting times necessary to obtain MRQs for some analytes)

5.5.11 Using the Wt% solids result from the wet centrifuged solids, calculate and report the centrifuged solids results on a 'dried' solids basis.

5.5.12 Calculate and report the centrifuged solids results on an 'undissolved' solids basis per the equations provided in the Test Specification TSP-24590-01-00006..

5.5.13 Using the Wt% oxide results from the decanted liquid and the wet centrifuged solids, calculate and report the centrifuged solids results on an 'undissolved' oxide basis (g / 100 g oxide). Provide details of the calculation in the final report.

5.6 PCB Characterization of Sub-Sample

The PCB characterization of the sub-sample shall be initiated by a same "standard" laboratory Analytical Service Request with special instructions as used for the Chemical and Radiological Characterization (Step 5.5). In summary,

5.6.1 One sub-sample shall be archived without separating the solids from the liquid until the analytical laboratory is ready to analyze the sample. The sample shall be analyzed along with the Reg. DQO Step 1 Analysis or after the Reg. DQO Step 1 analysis is complete. The analysis shall be performed using the same QA/QC, approved procedures, and reporting methods required by the Reg. DQO.

5.6.2 The archived sample condition shall be monitored and controlled in order to preserve the sample integrity.

5.7 Compare Analysis to WPT Contract Specification 7 (LAW)

The analytical results for the centrifuged liquid are to be reported and compared to the limits listed in Specification 7, Low-Activity Waste Envelopes in *WTP Contract No. DE-AC27-01RV14136* (See attachment 2, Tables TS-7.1 and TS-7.2).

5.8 Compare Analysis to WPT Contract Specification 8 (HLW)

The analytical results for the 'undissolved' centrifuged solids are to be reported and compared to the limits listed in Specification 8, High-Level Waste Envelope in *WTP Contract No. DE-AC27-01RV14136* (See Attachment 2, Table TS-8.1 through TS-8.4).

5.9 Reporting

5.9.1 All characterization results are to be reported in metric units, in accordance with section 6.6.3, Convention of Units of Measure, of the DOE-ORP statement of work to BNI/WGI.

5.9.2 Draft test results shall be reported to RPP-WTP within 90 calendar days after completing the analyses identified in this test plan and a final test report shall be issued within 30 calendar days after receiving comments on the draft report from RPP-WTP. The final report shall be issued as a document approved for public release and available to all Hanford contractors. The sample manipulations representative of each analyzed sample shall be detailed in the report.

5.9.3 Test results for the liquids and solids shall be reported in a format compatible with Standard Electronic Format Specification for Tank Waste Characterization *Data Loader: Version 3.0*, (HNF-3638 revision 1), to the extent data field information is available.

6 Quality Assurance

Battelle implements the RPP-WTP quality requirements in a quality assurance plan (QAP) as approved by the RPP-WTP QA organization. Battelle shall perform this task in compliance with 10 CFR 830.120, "Quality Assurance Requirements".

Additionally, BNI/WGI personnel may use the PCB analysis results for regulatory submittals (i.e., environmental data). For the PCB analysis, the requirements specified in PNNL-13429 and the associated test plan, TP-RPP-WTP-30 shall be met.

The *Quality Assurance Requirements and Description* (DOE/RW-00333P), the principal quality assurance document for the Civilian Radioactive Waste Management Program applies to all activities conducted under this Test Plan with the exception of sections 5.4, 5.5.3 and 5.5.4. Activities in sections 5.4, 5.5.3 and 5.5.4 are to be conducted under the quality requirements of PNNL's Standards-Based Management System (SBMS).

7 Definitions

None

Attachment 1

Tables from TSP24590-01-00006 Rev.0
"Tank 241-AZ-101 Sample Composite, Homogeneity, and Analysis Test Specification".

Table 1
Slurry Analyses for Feed Composition, Mid Range Composition and Concentrated Composition(a)

Analyte	Minimum Reportable Quantity	Analysis Method
Physical Property (a)	Expected Range	
Slurry Density(b)	1 to 1.6 gm/cm;	Gravimetric
Liquid Density(b)	1 to1.6 gm/cm	Gravimetric
Vol% Centrifuged solids(b)	0.1 to 50 vol%	Volumetric
Wt% Centrifuged solids(b)	0.1 to 30 wt%	Gravimetric
Wt% Total Dried solids(b)	1 to 80 wt%	Gravimetric
Wt% Dissolved solids(b)	1 to 25 wt%	Gravimetric
Wt% Undissolved solids(b)	1 to 25 wt%	Calculation
Heat Capacity(c, d)	0.5 to 2.0 J/g-°C	Calorimeter
Shear Stress Vs Shear Rate(c, f)	0 to 1000(e) sec^{-1}	Viscometer
Yield Strength(c, f)	1 to 1000 Pa	Viscometer
Particle size(g)	0.1 to 500 micron	Laser Scattering
Particle (size & shape)	0.1 to 500 micron	SEM

Footnotes:

(a) Triplicate measurements are to be made for each Physical Property at specified concentration and temperature. Acceptable precision is <15% RSD. %RSD = (standard deviation/mean) * 100

(b) Measurements are to be made using vendor calibration of glassware and laboratory balances.

(c) Measurements are to be made at cell ambient temperature (approximately 32°C), and also at 40±1°C. If the hot cell temperature is greater than 35°C, BNI will be contacted to verify the intent to perform two sets of rheological measurements with a temperature differential of less than 4°C.

(d) Results are calculated based on behavior of sample relative to a standard (water or sapphire depending on sample range).

(e) or the upper limit of selected sensor/viscometer configuration.

(f) Perform calibration check every 30 days using a NIST traceable standard.

(g) Perform calibration check every 30 days using instrument manufactures recommended method.

Table 2
Liquid Fraction Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method[c]
	mg/L	
Al	7.5E+01	
B	2.3E+00	ICP-AES (AA may be used for Na, K)
Ba	2.3E+00	
Ca	1.5E+02	
Ce	2.3E+00	
Cd	7.5E+00	
Cr	1.5E+01	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Li	2.3E+00	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Th	2.3E+00	
V	2.3E+00	
W	2.3E+00	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TOC	1.5E+03 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
Br	3.0E+02	
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03 (as P)	
SO_4	2.3E+03 (as S)	
	mCi/L (except as noted)	
Rb	1.0E+00 (mg/L)	ICP-MS (AA may be used for Cs determination)
^{127}I	1.5E+00 (mg/L)	
^{129}I	1.8E-05	
^{133}Cs	7.0E-04 (mg/L)	
^{135}Cs	1.5E+00	
^{137}Cs	1.5E+00	
^{237}Np	2.7E-02	
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	8.7E-03(mg/L)	
^{99}Tc	1.5E-03	
^{233}U	4.2E-04	
^{234}U	1.2E-04	
^{235}U	4.5E-08	
^{236}U	1.4E-06	
^{238}U	7.2E-09	
^{99}Tc(pertechnetate)	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{3}H	2.1E-02	Separations / Liquid Scintillation
^{14}C	7.2E-04	
^{79}Se	9.0E-05	
^{90}Sr	1.5E-01	
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}$Pu	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}$Cm	1.5E-02	

Table 2 Cont.

^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	2.1E-03	
^{126}Sn	6.0E-03	
^{137}Cs	9.0E+00	
^{231}Pa	7.89E-05	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha (TRU)	N/A	Summation(b) of: Pu-238, Pu-239+Pu-240 (or Pu-239, Pu-240 ICP/MS) and Am-241
Total and Free OH	7.5E+04 mg/L	Titration
CN	3.0E+00 mg/L	CN Analysis
Ammonia	1.4E+02 mg/L	ISE or IC
Organic Analytes	mg/L	
Oxalate	1.5E+03	Ion Chromatograph
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
EDTA(a)	1.5E+03	Derivatization/ GC-MS
HEDTA(a)	1.5E+03	
D2EHPA(a)	1.5E+03	
NTA(a)	1.5E+03	
IDA(a)	1.5E+03	
Succinic Acid(a)	1.5E+03	
ED3A(a)	1.5E+03	
Density Dissolved solids	Expected Range 0.95 - 1.5 (gm/mL) 1 to 50 (gm solids/gm supernate)	Gravimetric Gravimetric

Footnote:

(a) MRQs are target values, measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters.

(b) Guidance for reporting summation of isotopics and reporting isotopic values derived by different methods will be provided later after an agreement is obtained with DOE.

(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 3. HLW Solids Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method(c)
	mg/Kg(a)	
Ag	9.0E+02	
Al	3.3E+02	ICP-AES (AA may be used for Na & K)
B	3.0E+00	
Ba	6.0E+02	
Be	3.0E+00	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
K	2.0E+02	
La	6.0E+01	
Li	3.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
U	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
Br	4.5E+02	
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	
CN	3.0E+00	CN analysis
	mg/Kg	
As	3.0E+00	ICP-MS
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Mo	3.0E+01	
Pd	3.0E+02	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Rh	3.0E+02	
Ru	3.0E+02	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	

Table 3. Continued, HLW Solids Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analytical Methods[c]
W	**mg/Kg** 6.0E+00	ICP-MS
Y	6.0E+00	
^{99}Tc	6.0E+00	
^{127}I	1.5E+00	
^{133}Cs	7.0E-04	
^{233}U	6.0E+01	
^{235}U	6.0E+00	
^{237}Np	1.8E+00	
^{129}I	**mCi/Kg** 1.8E-05	
^{135}Cs	5.3E-03	
^{137}Cs	9.0E-02	
^{151}Sm	TBD	
^{234}U	3.7E-03	
^{236}U	3.8E-04	
^{238}U	2.0E-06	
^{3}H	1.5E-02	Separations / Liquid Scintillation
^{14}C	1.8E-03	
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{151}Sm	TBD	Beta Counting
^{238}Pu	6.0E-02	
$^{239/240}$Pu	6.0E+00	Separations / AEA
^{242}Pu	3.36E-01	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}$Cm	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
^{125}Sb	6.0E+00	
^{126}Sn	6.0E-02	
^{134}Cs	9.0E-01	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha (TRU)	N/A	Summation[b] of: Pu-238, Pu-239, Pu-240, and Am-241
^{241}Pu	1.2E+00 mCi/Kg	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetric
Density	0.9 to 2.0 gm/ml	Gravimetric
Wt% Undissolved Solids	10 to 50 wt%	Calculation
Wt% Soluble Solids	1 to 50 wt%	Calculation

Footnote:

[a] MRQs are based on dried solids weighs.

[b] Guidance for reporting summation of isotopics that are derived by different methods will be provided later after an agreement is obtained with DOE

[c] If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Recommended Analytical Technique[l]	QC Flagging Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Triplicate RSD[c]
Al, B, Ba, Ca, Cd, Ce, Cr, Fe, K, La, Li, Mg, Ni, P, Pb, Th, U, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
U	Kin. Phosphorescence	80 - 120%	75 - 125%	<15%
Rb, ^{127}I, ^{129}I, ^{133}Cs, Cs^{135}, Cs^{137}, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Pu / ^{241}Am, ^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cl$^-$, Br$^-$, F$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{-3}$, SO$_4^{-2}$	IC	80 - 120%	75 - 125%	<15%
CN$^-$	CN Analysis	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH$_3$/NH$_4^+$	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH- (total and Free)	Potentiometric titration /precipitation	80 - 120%	N/A	<15%
TIC	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	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[f], ^{126}Sn[i], ^{231}Pa	Extended GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[m]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 – 130%	<15%
^{137}Cs	GEA	NP	N/A[g]	<15%
^{154}Eu[f]	GEA	NP	N/A[g]	<15%
^{155}Eu[f]	GEA	NP	N/A[g]	<15%
^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm	Separation/AEA	NP	N/A[e]	<15%
Total Alpha	Proportional counter	70 - 130%	70 – 130%	<15%
Sum of Alpha[h] [TRU]	Calculation	N/A	N/A	N/A
Density	Gravimetric	N/A	N/A	<20%
Wt% dissolved solids	Gravimetric	N/A	N/A	<20%
EDTA[k]	Derivatization/GC-MS	80 - 120%	75 – 125%	<15%
HEDTA[k]	Derivatization/GC-MS	80 - 120%	75 – 125%	<15%
Oxalate	IC	80 - 120%	75 – 125%	<15%
Citrate	IC	80 - 120%	75 – 125%	<15%
Formate	IC	80 - 120%	75 – 125%	<15%
Gluconate	IC	80 - 120%	75 – 125%	<15%
Glycolate	IC	80 - 120%	75 – 125%	<15%
D2EPHA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A[k]	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr, use Sr in the standard for determining the total mass-90 concentration.

(e) 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.

(f) 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.

(g) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(h) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting 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 that 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 MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination. If any of the isotopes are below the MRQ, then the method of summation will be agreeed by BNI prior to reporting values.

(i) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(j) Calibrate with Sn-117.

(k) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

(l) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

(m) Matrix spike analyses are not required for this method. No standards are available for Se-79. Instead, metal Se is added during processing and the recovery is determined gravimetrically".

Table 5. Quality Control Parameters for Solids Analysis

Solids Fraction	Recommended Analytical Technique(o)	QC Flagging Criteria		
		LCS % Recovery(a)	Spike % Recovery(b)	Triplicate RSD(c)
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Cs, Fe, K, La, Mg, Mn, Nd, Ni, P, Pb, Pd, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, U, V, W, mass unit 90(d), ^{99}Tc, ^{127}I, ^{133}Cs, ^{233}U, ^{235}U, ^{237}Np, ^{129}I, ^{135}Cs, ^{137}Cs, ^{151}Sm, ^{234}U, ^{236}U, ^{238}U.	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, Br^-, F^-, NO_2^-, NO_3^-, PO_4^{-3}, SO_4^{-2}	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Hg	CVAA	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%
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A(g)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{60}Co(i)	Extended count GEA	NP	N/A(k)	<15%
^{90}Sr(j)	Isotopic specific separation/beta count	75 - 125%	N/A(g)	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{125}Sb(m)	Extended Count GEA	NP	N/A	<15%
^{126}Sn(n)	Extended Count GEA	NP	N/A	<15%
^{134}Cs(h)	Extended Count GEA	NP	N/A	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{152}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{154}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{155}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{241}Am	Extended Count GEA	NP	N/A(k)	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, $^{239/240}$Pu, ^{242}Pu	Separation/AEA	NP	N/A(g)	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A(g)	<15%
^{242}Cm	Separation/AEA	NP	N/A(g)	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A(g)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Bulk density	Gravimetric	N/A	N/A	<20%
Wt% solids	Gravimetric	N/A	N/A	<20%

Acronyms:
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 Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.

(e) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(f) Not used.

(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 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 WAPS justification.

(i) 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 ^{152}Eu, ^{154}Eu, ^{155}Eu.

(j) Combined analysis of ^{90}Sr and ^{90}Y.

(k) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(l) Combined analysis with ^{93m}Nb.

(m) Combined analysis with ^{125m}Te.

(n) Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

(o) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

CONTRACT SPECIFICATIONS
(Excerpt from Contract No. DE-AC27-O1RV14136)

Specification 7: Low-Activity Waste Envelopes Definition

7.1 Scope: This Specification establishes three LAW feed envelopes, Waste Envelopes A, B, and C; and defines how a unit of LAW is determined for each LAW envelope. Each waste envelope provides the compositional limits for chemical and radioactive constituents in the waste feed to be provided to the WTP. The WTP shall be designed to treat the waste envelopes.

7.2 Requirements:

7.2.1 References:

7.2.1.1 HNF-SD-WM-SAR-067, Rev. 1-I. March 2000. *Tank Waste Remediation System Final Safety Analysis Report*. CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.2 HNF-SD-WM-TSR-006, Rev. 1-HE. March 2000. *Tank Waste Remediation System Technical Safety Requirements*, CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.3 OSD-T-151-00007, Rev. H-22. June 14, 2000. *Operating Specification for 241-AN, AP, AW, AY, AZ, and SY Tank Farms*. CH2M HILL Hanford Group, Inc., Richland, Washington.

7.2.1.4 DOE/RL-88-21, Rev. 10. December 21, 1999. *Double Shell Tank Unit Permits Application*. U.S. Department of Energy, Richland Operations Office, Richland, Washington.

7.2.2 Envelope Requirements:

7.2.2.1 Composition: This specification lists the concentration limits for the LAW Envelopes A, B, and C feed to be transferred by DOE to the Contractor for LAW services in Tables TS-7.1, *Low-Activity Waste Chemical Composition, Soluble Fraction Only*, and TS-7.2, *Low-Activity Waste Radionuclide Content, Soluble Fraction Only*. The concentration limits apply to the soluble fraction only. The Na concentration limits for the LAW feeds are identified below.

– Waste Feed	Na (mole per liter)
Envelope A, B, C	4 – 10
AZ-101 and AZ-102 Supernatant	2 – 5
HLW Slurry and other HLW Liquids (Defined in Specification 8, *High-Level Waste Envelope Definition*)	0.1 – 10

The LAW feeds may contain up to two weight percent solids. Solids are defined as the product of centrifuging the LAW feed, separating and drying the solids, and removing the dissolved solids contribution. The insoluble fraction characterization will include measurements of Al, Cr, Fe, Mn, Na, P, S, Si, U, TIC, TOC, ^{60}Co, ^{90}Sr, ^{99}Tc, ^{137}Cs, ^{154}Eu, $^{239/240}$Pu, ^{241}Am, and total alpha concentrations. Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed.

All LAW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report*, and *Technical Safety Requirements*, as applicable.

The radiochemical inventory of the LAW feed at the time of delivery shall be compared to the specification limits to assess compliance. The specifications for ^{60}Co, and ^{154}Eu shall apply at the time of delivery for ILAW immobilization.

The LAW feed provided shall not contain a visible separate organic phase.

The LAW feed provided will generate gases, including hydrogen and ammonia, at a nearly constant rate and a nearly uniform composition.

The maximum ^{137}Cs concentration equivalent in the transferred Envelope A, Envelope B, and Envelope C wastes feeds shall not exceed 1.2 Ci/l. The maximum ^{137}Cs concentration equivalent in the liquid fraction of Tank AZ-101 and AZ-102 feeds shall not exceed 3.0 Ci/l.

Dangerous waste codes are identified in the Double-Shell Tank System Unit Permit Application (DOE/RL-88-21, December 21, 1999). Multi-source leachate (F039) is included as a waste derived from non-specific source wastes F001 through F005.

7.2.3 Units of Low-Activity Waste: Units of LAW shall be defined as follows:

(a) Envelope A: The quantity of Waste Envelope A containing one metric ton of waste sodium shall equal one unit.

(b) Envelope B: The quantity of Waste Envelope B containing one metric ton of waste sodium shall be the lesser of the following number of units:

(1) 2.6 units;or

(2) $\frac{X}{Y}$ units

where X is equal to 18-weight percent sodium oxide loading in the ILAW glass and Y is equal to the achievable waste sodium oxide loading, for the particular waste feed. The waste loading limitations shall be based solely upon effects of chlorine, chromium, phosphate, and sulfate.

(c) Envelope C: The quantity of Waste Envelope C containing one metric ton of waste sodium shall be the lesser of the following number of units:

(1) 1.15 units; or

(2) $\frac{X}{Y}$ units

where X and Y are defined above. The waste loading limitations shall be based solely upon sodium additions required for cesium, technetium, strontium and TRU removal from Envelope C for the particular waste feed.

Table TS-7.1 Low-Activity Waste Chemical Composition, Soluble Fraction Only

Chemical Analyte	Maximum Ratio, analyte (mole) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
Al	2.5E-01	2.5E-01	2.5E-01
Ba	1.0E-04	1.0E-04	1.0E-04
Ca	4.0E-02	4.0E-02	4.0E-02
Cd	4.0E-03	4.0E-03	4.0E-03
Cl	3.7E-02	8.9E-02	3.7E-02
Cr	6.9E-03	2.0E-02	6.9E-03
F	9.1E-02	2.0E-01	9.1E-02
Fe	1.0E-02	1.0E-02	1.0E-02
Hg	1.4E-05	1.4E-05	1.4E-05
K	1.8E-01	1.8E-01	1.8E-01
La	8.3E-05	8.3E-05	8.3E-05
Ni	3.0E-03	3.0E-03	3.0E-03
NO_2	3.8E-01	3.8E-01	3.8E-01
NO_3	8.0E-01	8.0E-01	8.0E-01
Pb	6.8E-04	6.8E-04	6.8E-04
PO_4	3.8E-02	1.3E-01	3.8E-02
SO_4	1.0E-02	7.0E-02	2.0E-02
TIC[1]	3.0E-01	3.0E-01	3.0E-01
TOC[2]	5.0E-01	5.0E-01	5.0E-01
U	1.2E-03	1.2E-03	1.2E-03

Notes:

1 Mole of inorganic carbon atoms/mole sodium

2 Mole of organic carbon atoms/mole sodium

Table TS-7.2 Low-Activity Waste Radionuclide Content, Soluble Fraction Only

Radionuclide	Maximum Ratio, radionuclide (Bq) to sodium (mole)		
	Envelope A	Envelope B	Envelope C
TRU[2]	4.8E+05	4.8E+05	3.0E+06
^{137}Cs	4.3E+09	2.0E+10	4.3E+09
^{90}Sr	4.4E+07	4.4E+07	8.0E+08
^{99}Tc	7.1E+06	7.1E+06	7.1E+06
^{60}Co	6.1E+04	6.1E+04	3.7E+05
^{154}Eu	1.2E+06	1.2E+06	4.3E+06

Notes:

[1] The activity limit shall apply to the feed certification date.

[2] TRU is defined as: Alpha-emitting radioncludes with an atomic number greater than 92 with half-life greater than 10 years.

Some radionuclides, such as ^{90}Sr and ^{137}Cs, have daughters with relatively short half-lives. These daughters have not been listed in this table. However, they are present in concentrations associated with the normal decay chains of the radionuclides.

Specification 8: High-Level Waste Envelope Definition

8.1 Scope: This Specification establishes the HLW slurry composition and the unwashed solids composition (Envelope D). This waste envelope provides the compositional limits for chemical and radioactive constituents and physical properties in the waste feed to be provided to the WTP. The WTP shall be designed to treat the feed envelopes.

8.2 Requirements:

8.2.1 References:

8.2.1.1 HNF-SD-WM-SAR-067, Rev. 1-I. March 2000. *Tank Waste Remediation System Final Safety Analysis Report.* CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.2 HNF-SD-WM-TSR-006, Rev. 1-HE. March 2000. *Tank Waste Remediation System Technical Safety Requirements*, CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.3 OSD-T-151-00007, Rev. H-22. June 14, 2000. *Operating Specification for 241-AN, AP, AW, AY, AZ, and SY Tank Farms.* CH2M HILL Hanford Group, Inc., Richland, Washington.

8.2.1.4 DOE/RL-88-21, Rev. 10. December 21, 1999. *Double Shell Tank Unit Permits Application.* U.S. Department of Energy, Richland Operations Office, Richland, Washington.

8.2.2 High-Level Waste Slurry Description and Envelope Requirements:

8.2.2.1 Composition: The HLW slurry will contain a mixture of liquids (Envelopes A, B, or C) and solids (Envelope D). The compositional range of the liquid fraction is defined in Specification 7, *Low-Activity Waste Envelopes Definition.* For liquid fractions with a sodium molarity of less than three, the liquid shall be treated as if 3 molar sodium were present for feed certification purposes. Specification 7.2.2.4, *Radioactive Material Concentration*, does not apply to Envelope A, B, or C liquids. The composition range of the Envelope D unwashed solids is given in Tables TS-8.1, TS-8.2, TS-8.3, and TS-8.4. The feed concentration will be between 10 and 200 grams of unwashed solids/liter, except for feeds from waste Tanks AZ-101 and AZ-102, where minimum solids content does not apply.

Compositions for Envelope D unwashed solids (Tables TS-8.1, TS-8.2, TS-8.3, and TS-8.4) are defined in terms of elemental or anion concentrations and radionuclide activities per 100 grams equivalent non-volatile waste oxides. The non-volatile waste oxides include sodium oxide and silicon oxide.

The HLW feed components identified in Tables TS-8.1, TS-8.2 and TS-8.3 are waste components important to establishing the waste oxide loading in the HLW glass. Only these components have concentration limits, which will be used to provide the basis for certification that the HLW feed is within specification limits.

The HLW feed components identified in Table TS-8.4 are also important to HLW glass production. The concentrations of these components in the waste are not expected to exceed the maximum values listed in Table TS-8.4. Information on these components will be provided to support product and process qualification but will not be used as a basis for determining if the feed meets specification requirements.

All HLW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report* (HNF-SD-WM-SAR-067), and *Technical Safety Requirements* (HNF-SD-WM-TSR-006, Revision 1-D) as applicable. The radiochemical inventory of the waste feed at the time of delivery shall be compared to the specification limits to assess compliance.

Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed. Feed will be delivered by pipeline in batches. Limits apply to the total retrievable contents of waste from a feed tank. Some elements, components, and isotopes are determined by calculation and not analytic measurement.

The HLW feed provided will not contain a visible separate organic layer.

The HLW waste provided will generate gases due to radiolysis including hydrogen and ammonia at a nearly constant rate and nearly uniform composition. The Contractor is responsible for the management of changes in gas release rate and distribution resulting from their waste processing activities.

Applicable dangerous waste codes are identified in the Double-Shell Tank System Unit Permit Application (DOE/RL-88-21, December 21, 1999). Multi-source leachate (F039) is included as a waste derived from non-specific source wastes F001 through F005.

Table TS-8.1 High-Level Waste Feed Unwashed Solids Maximum Non-Volatile Component Composition (grams per 100 grams non-volatile waste oxides)

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
As	0.16	Pu	0.054
B	1.3	Rb	0.19
Be	0.065	Sb	0.84
Ce	0.81	Se	0.52
Co	0.45	Sr	0.52
Cs	0.58	Ta	0.03
Cu	0.48	Tc	0.26
Hg	0.1	Te	0.13
La	2.6	Tl	0.45
Li	0.14	V	0.032
Mn	6.5	W	0.24
Mo	0.65	Y	0.16
Nd	1.7	Zn	0.42
Pr	0.35		

Table TS-8.2 High-Level Waste Feed Unwashed Solids Maximum Volatile Component Composition (grams per 100 grams non-volatile waste oxides)

Volatile Components	Maximum (grams / 100 grams waste oxides)
Cl	0.33
CO_3^{-2}	30
NO_2	36 (total NO_2/NO_3) as NO_3
NO_3	
TOC	11
CN	1.6
NH_3	1.6

Table TS-8.3 High-Level Waste Feed Unwashed Solids Maximum Radionuclide Composition
(Curies per 100 grams non-volatile waste oxides)

Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)
^{3}H	6.5E-05	^{129}I	2.9E-07	^{237}Np	7.4E-05
^{14}C	6.5E-06	^{137}Cs	1.5E00	^{238}Pu	3.5E-04
^{60}Co	1E-02	^{152}Eu	4.8E-04	^{239}Pu	3.1E-03
^{90}Sr	1E+01	^{154}Eu	5.2E-02	^{241}Pu	2.2E-02
^{99}Tc	1.5E-02	^{155}Eu	2.9E-02	^{241}Am	9.0E-02
^{125}Sb	3.2E-02	^{233}U	9.0E-07	$^{243+244}$Cm	3.0E-03
^{126}Sn	1.5E-04	^{235}U	2.5E-07		

Table TS-8.4 Additional High-Level Waste Feed Unwashed Composition for Non-Volatile Components
(grams per 100 grams non-volatile waste oxides)

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
Ag	0.55	Ni	2.4
Al	14	P	1.7
Ba	4.5	Pb	1.1
Bi	2.8	Pd	0.13
Ca	7.1	Rh	0.13
Cd	4.5	Ru	0.35
Cr	0.68	S	0.65
F	3.5	Si	19
Fe	29	Ti	1.3
K	1.3	U	14
Mg	2.1	Zr	15
Na	19		
Th	5.0		

Attachment 3

Physical Testing Protocol

At PNNL we have developed a single physical testing protocol. This protocol was designed to satisfy the all of the needs of multiple tasks within the project while at the same time ensure opportunistic data and data between tasks could be compared on a common basis. This protocol allows for the measurement of

1) density of the bulk slurries, settled solids, centrifuged solids, and centrifuged supernatant.
2) The weight percent (wt%) and volume percent (vol%) settled solids, wt% and vol% centrifuged solids, wt% total solids, and wt% undissolved solids.

Under this protocol, settled solids are defined as the solids layer that separates from the bulk slurry after 3 days of gravity settling. Centrifuged solids are defined as the solids layer that separates from the bulk slurry after 1 hour of centrifugation at 1000 gravities.

For this testing, a known mass of each slurry is placed in triplicate in volume graduated centrifuge cones. The triplicates are then allowed to settle for 3 days. The total mass (M_B) and volume (V_B) of the settled are recorded and the density of the bulk slurry are calculated ($D_B=M_B/V_B$). These results can be biased low due to entrained gas as well as an inability to clearly measure the total sample volume due to material smeared on the sides of the centrifuge tubes. Therefore, the bulk slurry densities are recalculated latter in the work using volumes recorded following centrifugation. Following settling, the volume of the settled solids (V_{ss}) and volume of settled supernatant (V_{sl}) are recorded. The vol% settled solids are then calculated ($Vol\%_{ss}=V_{ss}/V_B$ x 100%).

If sufficient clarified liquid is present following settling, a portion is transferred to a graduated cylinder for determination of supernatant density. This value should correlate to the supernatant density measured following centrifugation. The advantage of this measurement is that this supernatant density can be measured for samples equilibrated at multiple temperatures. Centrifuge densities are generally limited to slurries equilibrated at ambient temperatures.

The settled slurries are then centrifuged at approximately 1000 times the force of gravity for 1 hour. All of the centrifuged supernatant is then transferred to a graduated cylinder and its mass (M_{cl}) and volume (V_{cl}) recorded, and the density is calculated ($D_{cl}=M_{cl}/V_{cl}$). The mass (M_{cs}) and volume (V_{cs}) of the centrifuged solids are also recorded, and the density is calculated ($D_{cs}=M_{cs}/V_{cs}$). In addition, the wt% centrifuged solids ($Wt\%_{cs}=M_{cs}/M_B$ x 100%), and vol% centrifuged solids ($Vol\%_{cl}=V_{cl}/V_B$ x 100%) is calculated.

In many cases, centrifugation can result in the release of gas in the form of bubbles or foams. Evidence of trapped gas was observed during some rheological testing. Therefore, comparison of the bulk density measurements before and after centrifugation are very important in understanding the rheology of some samples including SY-103 and AZ-102. In addition, it is possible that not all of the gas is released from the slurry by centrifugation, so the density results following centrifugation may be biased low.

Prior to centrifugation, if the supernatant could not be removed from the centrifuge cone without disturbing the settled solids, the mass of the settled solids (M_{ss}) can not be measured directly. Therefore, the mass of the settled solids is calculated. This is done by first calculating the mass of the settled supernatant in the centrifuge cone using the measured centrifuged supernatant density and volume ($M_{sl} = D_{cl} \times V_{sl}$), then subtracting this mass for the mass of the bulk slurry to get the mass of the settled solids ($M_{ss}=M_B-M_{sl}$). The density of the settled solids is now calculated ($D_{ss}=M_{ss}/V_{ss}$) as well as the wt% settled solids ($Wt\%_{ss}=M_{ss}/M_B$ x 100%).

The centrifuged solids and supernatants are now dried separately at 105°C for 24 hours. The mass of the dried centrifuged supernatant (M_{dcl}) and dried centrifuged solids (M_{dcs}) are then measured. Assuming all mass lost during the drying process is water and not another volatile component, the weight percent total solids in the bulk slurry is calculated (Wt% total solids = $(M_{dcs}+M_{dcl})/(M_{cs}+M_{cl})$ x 100%). Waters of hydration or volatile organics can lead to low bias M_{dcs}. Waters of hydration led to a low bias in during the analysis of the AW-101 entrained solids.

An additional calculation is then performed to determine the wt% solids in the samples excluding all interstitial liquid. We have referred to this as "wt% undissolved solids". This wt% undissolved can be thought of as the solids left if all the supernatant could be drained from the bulk slurry. The following equation is used:

$$Wt\%\ undissolved\ solids = \left(1 - \frac{1 - \frac{M_{dcs}}{M_{cs}}}{1 - \frac{M_{dcl}}{M_{cl}}}\right) \times \frac{M_{cs}}{M_B} \times 100\%$$

This calculation assumes 1) that the supernatant and the interstitial liquid have the same composition, and 2) that all mass loss during the drying of the centrifuged solids is water loss from interstitial liquid.

D2

COMPOSITING, HOMOGENIZATION, SUB-SAMPLING TEST INSTRUCTION

TI-RPP-WTP-095, Rev. 0

Battelle Test Instruction	**Document No.: TI-RPP-WTP-095** **Rev. No.: 0** Urie
Title: AZ-101 Sample Homogenization	
Work Location: 325/HLRF	**Page** 1 of 14
Author: PR Bredt	**Effective Date: Upon final signature** **Supersedes Date: New**
	Required Reviewers: _X_Author _X_Technical Reviewer ___Quality Engineer ___Project Manager ___Client

Approval **Signature** **Date**

Author Paul Br 7/16/01

Technical Reviewer 7/16/01

Applicability

This Test Instruction describes work to be performed under Project 42365, WTP-RPP Phase B2 Support. Samples of actual waste from Hanford tank AZ-101 were received at Battelle's Radiochemical Processing Laboratory (RPL) for testing under this project. This Test Instruction describes how these materials will be composited, homogenized, and sub-sampled for process testing and characterization tasks.

Client expectations for successful achievement of project data needs have been established via Task Specification TSP-24590-01-00006, "Tank 241-AZ-101 Sample Composite, Homogeneity, Analysis", and Test Plan TP-RPP-WTP-089, "AZ-101 Homogenization and As-Received Characterization". This work is to be performed under procedure PRO-41500-04, "Sample Compositing", and PRO-29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges". This document is a mechanism for the cognizant scientist to communicate to technical staff the specifics on procedure implementation and client expectations. Therefore, this document qualifies as a Test Instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction of cognizant project staff.

Work with actual tank material will be performed in radiological hot cells.

Justification/Test Objectives/Success Criteria

The objective of this test instruction is to provide homogeneous sub-samples to tasks within the RPP-WTP project. Material from Hanford tank AZ-101 were transferred to the Radiochemical Processing Laboratory in 15 separate shipping containers. Under this test instruction, the contents of the shipping containers will be recombined into a tank composite based on the results of Li and Br analyses. The material will be homogenized using a bladed impeller and then sub-sampled into bottles.

An administrative hold point has been incorporated in the work instructions. This hold point is contained is steps 18 and 19. At this point in the homogenization, a composite will have been prepared and sub-sampled. Data on the volume percent settled solids, used as a gauge of successful homogenization, will be presented to the client for concurrence.

Quality Control

This work is to be conducted to the quality requirements of the Nuclear Quality Assurance Requirements and Description (NQARD) Manual and to the current approved Quality Assurance Project Plan.

Changes to this test instructions can be made only by the cognizant scientist and will be documented by crossing out the original information with a single line, and recording, initialing, and dating the changes.

The results of all measurements will be recorded in an LRB, test instruction, or bench sheet. Copies of the completed test instructions, bench sheets, and LRBs will be transferred to the project file upon completion of the project.

Spill Mitigation

When working with liquids and slurries, there is a risk of sample loss through inadvertent spills. During hot cell work, spills primarily result from dropped or tipped sample bottles, broken glassware, and the failure of transfer equipment. In most cases, spills can be controlled and material losses minimized through the use of secondary containment and other good laboratory practices. The cognizant scientist is responsible for working with hot cell staff to reduce the potential and programmatic impact of spills. Specific examples include:

1) Bottle holders to stabilize jars during sample transfers
2) Catch pans below homogenization vessels and primary sample containers
3) Safety coated jars and bottles (when this does not interfere with analytical requirements)
4) Plastic centrifuge liners

If a spill does occur, every practical effort will be made to recover as much of the sample as possible.

BALANCE # 1 - 200g cal. wt = 200.00g rgs 7/17/01
BALANCE # 2 - 200g " " = 199.98 g rgs 7/23/01
Balance # 3 -- 200g " " = 199.94 g - 7/27/01 @ 9 Am

M&TE List:

Balance 1: - used for TARE Wts ON Bottles
Calibration ID i113120467
Calibration Expiration 2/2002
Location 325B1dg Rm 302

Balance 2: (if needed) HLRF CELL C used to obtain tare wts on Empty original Bottles
Calibration ID 388-06-01-020
Calibration Expiration 2/2002
Location HLRF Cell C

~~Thermocouple:~~ Balance # 3 used for Final wts on 500mL Bottles
Calibration ID L 90599 Type Pm4000
Calibration Expiration 2/2002
Location HLRF Cell B

~~Digital Thermometer~~: Thermo couple Type K
Calibration ID 02885
Calibration Expiration 11/2
Location C-Cell

BALANCE 3 needed because Balance # 2 max is 400g rgs 8/13/01

Work Instructions

1) Confirm that the samples have been inspected under test instruction TI-RPP-WTP-087, "AZ-101 Sample Inspection".

2) Table 1 lists the samples shipped from 222-S to the RPL for this task. Weigh the full sample jars to ±0.01 g and record the data in Table 1.

3) **Administrative Hold Point**: Contact the client and confirm that the first 11 samples in Table 1 had acceptable Li-Br concentrations. The last 4 samples in the table (16029, 17023, 15958, 17025) were not tested for Li-Br. Attach a copy of an email from the client listing the samples that may be used in the composite.

4) Install the 1/8" (1/8" to 3/32" is acceptable) screen on the 7 L stainless steel mixing vessel. The modified food mill may be used provided the mesh size is 1/8" to 3/32".

> Note:
> While 1/8 inch is a fairly course mesh, samples with a high solids content may have a tendency to pile on the mesh. If this happens, rinse the material through the screens using the low solids content samples or using standing supernatant in the mixing vessel.

5) Transfer all material from the jars with acceptable LiBr concentrations to the 7L mixing vessel using the 1/8" screen to remove the large particle. If necessary, use supernatant from the jars or mixing vessel to rinse the solids into the kettle.

6) Remove the screen and place an airtight cover on the mixing vessel.

7) Reweigh the empty jars and record the empty masses to ± 0.01 g in Table 1.

8) Transfer any large particles retained on the screen to an appropriately sized glass jar of known mass labeled **"AZ-101 >1/8"**. Weigh the jar to ±0.01g. Record the approximate time that elapsed between transferring the solids to the screen and this weighing.

Signature ______ Date 7/23/01 Started @ 07:30
Finished @ 10:30

Elapse time 3hr

AZ-101 >1/8

Total ______ g
Tare 62.72 g
Wet Solids ______ g

After oberving food mill after processing, not any oversize particles were noticed, the holes in bottom were open & SS surface was clear. - Some dried on sludge ~5-10 g was observed on the sides of mill.

Video taped part of this process - recorded 1 of the Liquid bottle fractions transferred to see bottom was reasonably clean (18729, 18725), also solids bottles 17025 & 15958 were recorded.

After transfer of each solids bottle the food mill was cranked, in addition the mill was cranked a few turns between transfers of the last 5 aqueous bottles. The video was hard to collect good picture due to not having clearance with manipulator. rgs.

9) **Administrative Hold Point**:
   a. If greater than 20 g of material are collected on the screen, notify BNI representative with a proposed path forward for this material. Attach a supplemental test instruction to this test instruction documenting the process for resolution.
   b. If less than 20 g of material are collected on the screen, testing may continue without BNI concurrence. Retain this material separate from the composite and proceed with testing. Notify BNI of the solids mass at the earliest convenience.

Pouring Sequence

Table 1. AZ-101 Sample Information

Best Rinsed

See attached PRD 12/17/01

	Sample ID	Mass on 6/12/01[a]	Full Mass (g) used 6/12/01	Empty Mass (g) having some sample residual	Mass Transferred / (g)
6	18729	817.08	masses	304.20	
7	18725	817.00	rgs 7/23/01	305.01	
10	18726	815.90		298.46	
	18728	811.16		301.10	
4	18727	814.16		297.57	
10	18724	813.81		302.61	
2.	18723	824.99		306.45	
8	18720	821.90		306.50	
9	18722	825.35		308.44	
	18721	820.47		308.84	
	18719	805.33		303.78	
	16029	449.46		241.32	
3	17023	513.07		238.15	
5	15958	364.81		237.27	
1	17025	601.62		236.03	

Last 4 ★ mostly solids.

[a] Mass on 6/19/01 was collected after removal of sample for Li-Br analysis. This information is recorded in TI-RPP-WTP-087, "AZ-101 Sample Inspection". - See copy attached of pertinent pages# 6 & 7

Signature Date 7/23/01

Started Compositing @ 7:30
Finished compositing @ 10:3

10) Retain the empty bottle for one month. If no direction is given, then dispose of them as waste.

11) The goal of this step is to homogenize the sample using as little force as possible. Stir the sample by slowly increasing the motor speed until the solids are mobilized. Stir for a minimum of one hour. Record time, date, sample temperature to ±1.1°C and impeller speed (RPM or controller setting).

Time ~~0730~~ 11:15 rgs 7/23/01 Date 7/23/01 Temperature 37.9 °C Speed Propeller Set @ 5.5

★ Solids were very pasty & hard to break loose - much time was spent trying to get sample out of jar & rinsed so we could see glass.
17025 was particularly bad since solids were nearly dry & very chunky.
15958 - was dry due to busted lid - see TI-RPP-WTP-087 - some small lid fragments & liner (from orig bottle lid) were dumped into food mill on top of mixing vessel. Also lost a little in (~10-15ml) swirling the transfer rinse on this bottle.

AZ-101 Date: 07/26/01 test plan: TI-RPP-WTP-095 Rev 0

c Paul B 12/17/01

SAMPLE TRACKING AND MATERIAL BALANCE

Initial (222S Lab) Container ID	Mass (Gross) (g)	Empty Weight with residue (g)	Net Weight (g)	After Compositing and Mixing- Sub- Sample ID	Current Loc	Mass (Gross) (g)	Bottle Tare Weight (g)	Net Weight (g)	Approx Density g/mL	Volume % Settled solids after 1-day Total Vol (oz)	Solids Vol (oz)	Total Volume (mL)	Solids Volume (mL)	Vol % Solids	Collect order
15958	364.81	237.27	127.54	AZ-101-PCB-1	HLRF	300.14	132.81	167.33	1.29			130	24	18.5%	3
16029	449.46	241.32	208.14	AZ-101-PCB-2	HLRF	290.63	132.84	157.79	1.24			127	25	19.7%	4
17023	513.07	238.15	274.92	AZ-101-PCB-3	HLRF	295.34	132.95	162.39	1.27			128	25	19.1%	5
17025	601.62	236.03	365.59	AZ-101-CHEM-1	HLRF	305.74	132.34	173.40	1.33			130	37	28.5%	6
18719	805.33	303.78	501.55	AZ-101-CHEM-2	HLRF	289.23	133.28	155.95	1.27			123	23	18.7%	7
18720	821.90	306.50	515.40	AZ-101-CHEM-3	HLRF	290.59	133.34	157.25	1.26			125	25	19.6%	8
18721	820.47	308.89	511.58	AZ-101-CHEM-4	HLRF	309.52	132.72	176.80	1.36			130	25	19.2%	9
18722	825.35	305.44	519.91	AZ-101-RHEO-3%	HLRF	287.54	133.22	154.32	1.25			123	23	18.7%	10
18723	824.99	306.95	518.04	AZ-101-RHEO-13%	HLRF	628.5	320.66	307.87	1.27	8.2	1.5	242	43	17.7%	2
18724	813.81	302.61	511.20	AZ-101-RHEO-20%	HLRF	805.3	318.97	486.34	1.28	12.9	2.3	381	68	17.8%	1
18725	817.00	305.01	511.99	AZ-101-AR-A	HLRF	845.93	318.09	527.84	1.26	14.2	2.4	420	69	16.5%	11
18726	815.90	298.46	517.44	AZ-101-AR-B	HLRF	888.82	320.94	567.88	1.24	15.5	2.3	458	68	14.8%	12
18727	814.16	297.57	516.59	AZ-101-AR-C	HLRF	885.13	318.72	566.41	1.24	15.5	2.3	458	68	14.8%	13
18728	811.16	301.10	510.06	AZ-101-AR-D	HLRF	900.67	319.85	580.82	1.27	15.5	2.1	458	62	13.5%	14
18729	817.08	304.20	512.88	AZ-101-AR-E	HLRF	901.47	319.35	582.12	1.23	16.0	2.1	473	62	13.1%	15
all depleted	10916.1		6622.8	AZ-101-AR-F	HLRF	905.35	318.48	586.87	1.24	16.0	2.0	473	59	12.5%	16
				AZ-101-AR-G	HLRF	902.18	320.83	581.35	1.23	16.0	2.1	473	62	13.1%	17
				AZ-101-AR-H	HLRF	528.85	320.57	208.28	1.19	5.9	0.8	174	24	13.6%	18
						10561.0		6301.01	1.26				Ave	17.2%	
													STD	3.8%	

Rejecting High and Low (Chem 1 and AR-F)

Ave	16.8%
STD	2.5%

Material Balance	
Start wt	6622.8
Recovered	6301.0
wt. lost	321.8
% loss	4.86%

1oz = 29.56 mL

D02-007

Page 5A of 14

Checked Paul B 8/8/01

Started Pulling Samples @ 13:10

12) While operating the vessel agitator, extract one sub-sample of sufficient size (minimum of 100 ml) through the sample valve to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel. Then extract all of the material in the vessel into pre-weighed volume graduated glass jars. The volume and designations for these samples are listed below. Use the necessary spaces below to record the sample masses.

Stopped Mixing & Pulling Samples @ 13:40 hr End 13:56

Signature ry swen Date 7/23/01 @

Elapsed Time
46 min
to collect

Total Target
Volume = ~ 943 ml

AZ-101 PCB1
3) Jar Size 125ml @13:16
Target 120ml

Total 300.14 g
Tare 132.81 g
Slurry 167.33 g

AZ-101 PCB2
4) Jar Size 125ml @13:19
Target 120ml

Total 290.63 g
Tare 132.84 g
Slurry 157.79 g

AZ-101 PCB3
5) Jar Size 125ml @ 13:22
Target 120ml

Total 295.34 g
Tare 132.95 g
Slurry 162.39 g

AZ-101 CHEM1 *
6) Jar Size 125ml @ 13:24
Target 120ml

Total 305.74 g
Tare 132.34 g
Slurry 173.40 g

AZ-101 CHEM2
7) Jar Size 125ml @ 13:26
Target 120ml

Total 289.23 g
Tare 133.28 g
Slurry 155.95 g

AZ-101 CHEM3
8) Jar Size 125ml @ 13:29
Target 120ml

Total 290.59 g
Tare 133.34 g
Slurry 157.25 g

AZ-101 CHEM4
9) Jar Size 125ml @13:31
Target 120ml
Total 309.52 g
Tare 132.72 g
Slurry 176.80 g

AZ-101 RHEO 3%
10) Jar Size 125ml @ 13:33
Target 120ml
Total 287.54 g
Tare 133.22 g
Slurry 154.32 g

2) **AZ-101 RHEO 13%**
Jar Size 500ml @13:14
Target 260ml
Total 628.53 g
Tare 320.66 g
Slurry 307.87 g

AZ-101 RHEO 20%
1) Jar Size 500ml @13:10
Target 400ml

Total 805.31 g
Tare 318.97 g
Slurry 486.34 g

AZ-101 AR-A
11) Jar Size 500ml 13:35
Target 460ml

Total 845.93 g
Tare 318.09 g
Slurry 527.84 g

AZ-101 AR-B
12) Jar Size 500ml 13:38
Target 460ml

Total 888.82 g
Tare 320.94 g
Slurry 567.88 g

AZ-101 AR-C
13) Jar Size 500ml @13:41
Target 460ml

Total 885.13 g
Tare 318.72 g
Slurry 566.41 g

AZ-101 AR-D
14) Jar Size 500ml @13:44
Target 460ml

Total 900.67 g
Tare 319.85 g
Slurry 580.82 g

AZ-101 AR-E
15) Jar Size 500ml @13:47
Target 460ml

Total 901.47 g
Tare 319.35 g
Slurry 582.12 g

* Some residual sample remained in this bottle from sub-sampling pour-back usage. Might expect slightly higher amt. of solids here.

AZ-101 AR-F 16) Jar Size 500ml @ 13:50 Target 460ml Total 905.35 g Tare 318.48 g Slurry 586.87 g	**AZ-101 AR-G** 17) Jar Size 500ml @ 13:53 Target 460ml Total 902.18 g Tare 320.83 g Slurry 581.35 g	**AZ-101 AR-H** 18) Jar Size 500ml @ 13:56 Target 460ml Total 528.85 g Tare 320.57 g Slurry 208.28 g only 1/2 full END of Sub-Sampling
AZ-101 AR-I Jar Size 500ml Target 460ml Total ____ g Tare 320.85 g Slurry ____ g	**AZ-101 AR-J** Jar Size 500ml Target 460ml Total ____ g Tare 319.85 g Slurry ____ g	**AZ-101 AR-K** Jar Size 500ml Target 460ml Total ____ g Tare 321.42 g Slurry ____ g Not used rgs
AZ-101 AR-L Jar Size 500ml Target 460ml Total ____ g Tare 321.55 g Slurry ____ g	**AZ-101 AR-M** Jar Size 500ml Target 460ml Total ____ g Tare 321.41 g Slurry ____ g	**AZ-101 AR-N** Jar Size 500ml Target 460ml Total ____ g Tare 321.19 g Slurry ____ g Not needed
AZ-101 AR-O Jar Size 500ml Target 460ml Total ____ g Tare 319.51 g Slurry ____ g	**AZ-101 AR-P** Jar Size 500ml Target 460ml Total ____ g Tare 318.60 g Slurry ____ g	**AZ-101 AR-Q** Jar Size 500ml Target 460ml Total ____ g Tare 320.07 g Slurry ____ g Not Needed

13) Record the date and time and allow the sample collected in the previous step to settle for a minimum of 24 hours.

Day 7/24/01 Time 13:58

14) Visually inspect the samples for a floating organic layer. Record observations below.

Signature rg Swoboda Date 7/24/01

Observations:

No organic Layer observed Liquid fraction was very clean and light yellow in color

15) <u>Administrative Hold Point</u>: If a floating organic layer was observed in the previous step, contact the client prior to proceeding past step 16. N/A

16) Record the date and time, and total volume of the slurries and volume of the settled solids. Calculate the vol% of settled solids in the samples.

Day 7/24/01 Time 1400

AZ-101 PCB1

Total 130 ml
Solids 24 ml
Vol % Solids 18.5

AZ-101 PCB2

Total 127 ml
Solids 25 ml
Vol % Solids 19.7

AZ-101 PCB3

Total 128 ml
Solids 25 ml
Vol % Solids 19.1

AZ-101 CHEM1

Total 130 ml
Solids 37 ml – Best Guess sloped & not level
Vol % Solids 28.5

AZ-101 CHEM2

Total 123 ml
Solids 23 ml
Vol % Solids 18.7

AZ-101 CHEM3

Total 125 ml
Solids 25 ml
Vol % Solids 19.6

AZ-101 CHEM4

Total 130 ml
Solids 25 ml
Vol % Solids 19.2

AZ-102 RHEO 3%

Total 123 ml
Solids 23 ml
Vol % Solids 18.7

AZ-102 RHEO 13%

Total 8.2 oz ~~ml~~
Solids 1.5 oz ~~ml~~
Vol % Solids 17.7

1 oz = 29.56 mL
rgb

AZ-102 RHEO 20%

Total 12.9 oz ~~ml~~
Solids 2.3 oz ~~ml~~
Vol % Solids 17.8

AZ-102 AR-A

Total 14.2 oz ~~ml~~
Solids 2.4 oz ~~ml~~
Vol % Solids 16.5

AZ-102 AR-B

Total 15.5 oz ~~ml~~
Solids 2.3 oz ~~ml~~
Vol % Solids 14.8

AZ-102 AR-C

Total 15.5 oz ~~ml~~
Solids 2.3 oz ~~ml~~
Vol % Solids 14.8

AZ-102 AR-D

Total 15.5 oz ~~ml~~
Solids 2.1 oz ~~ml~~
Vol % Solids 13.5

AZ-102 AR-E

Total 16.0 oz ~~ml~~
Solids 2.1 oz ~~ml~~
Vol % Solids 13.1

AZ-102 AR-F

Total 16.0 oz ~~ml~~
Solids 2.0 oz ~~ml~~
Vol % Solids 12.5

AZ-102 AR-G

Total 16.0 oz ~~ml~~
Solids 2.1 oz ml
Vol % Solids 13.1

AZ-102 AR-H

Total 5.9 oz ~~ml~~
Solids 0.8 oz ~~ml~~
Vol % Solids 13.6

See attached Excel File (AZ-101-inventory 7-26-01.xls) for Vol % solids data as well as Net Bottle wt data.

7/25/01

AZ-101 AR-I

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-J

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-K

Total ________ml
Solids ________ml
Vol % Solids _____

Bottles Not used
PRB
12/17/01

AZ-101 AR-L

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-M

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-N

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-O

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-P

Total ________ml
Solids ________ml
Vol % Solids _____

AZ-101 AR-Q

Total ________ml
Solids ________ml
Vol % Solids _____

17) **Administrative Hold Point**: If the samples contain an average of less than 10 vol% settled solids, then the homogenization is complete. The vol% settled solids data on the subsamples is to be presented to the client for approval before proceeding. Once this approval has been received, proceed to 18.

18) **Administrative Hold Point**: If the samples contain an average of greater than 10 vol% settled solids but less than 60 vol% settled solids, then the following two criteria must be met before the sub-samples will be considered homogeneous. If these two criteria are met, data on the subsamples is to be presented to the client for approval before proceeding. If the sub-samples fail the criteria, then the subsamples will be returned to the mixing vessel and re-homogenized and sub-sampled under steps 11-16 at a higher agitation rate. If the second homogenization fails, the client will be consulted.

See attached letter from Ernie Lee on approval to proceed PRB 12/17/01

a. Calculate a standard deviation for the entire vol% settled solids data set. If there are a few outliers, eliminate up to 10% of the samples from the data set, and then recalculate the standard deviation. This standard deviation must be no greater than 5 vol%.

b. Calculate a best fit line for the data set (vol% settled solids versus collection order). This best fit line must not show a trend of greater than 5 vol% over the range. For example, if the linear best fit line is at 80% for the first sample, then it cannot be exceed 85% or be below 75% for the last sample (these are the values for the best fit line, not the samples).

19) **Administrative Hold Point**: If the samples contain greater than 60 vol% settled solids, contact the cognizant scientist prior to proceeding.

Balance #2 Cell C
100g = 100.001g 7/31/01

20) Transfer three ~8-9 ml sub-samples from one 125mL jar collected in step 12 into pre-weighed glass graduated centrifuge cones labeled AZ-101 A-C. Also transfer three ~8-9 ml sub-samples from one 500 mL jar into pre-weighed glass graduated centrifuge cones and label AZ-101 D-F . The cognizant scientist will select the jar to use. Record the mass data to ±0.001 g.

used CHEM-2 Bottle on A → C

CHEM-2 AZ-101 A		CHEM-2 AZ-101 B		CHEM-2 AZ-101 C	
Total	31.013 g	Total	32.127 g	Total	32.164 g
Tare	19.387 g	Tare	19.821 g	Tare	19.690 g
Slurry	11.626 g	Slurry	12.366 g	Slurry	12.474 g

used AR-E Bottle on D-F

AR-E AZ-101 D		AR-E AZ-101 E		AR-E AZ-101 F	
Total	30.279 g	Total	29.874 g	Total	31.167 g
Tare	18.879 g	Tare	18.697 g	Tare	19.265 g
Slurry	11.400 g	Slurry	11.177 g	Slurry	11.902 g

21) Agitate the cones and record the time and date.

Time 11:39 Date 7/31/01

Note:
i) The volume graduated centrifuge cones are to be read to ±0.1ml.
ii) The timing of volume measurements during the settling study need not be taken at the intervals listed. However, the samples must be left undisturbed for a minimum of 3 days and the final volumes must be recorded before the solid and liquid phases are separated.

22) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date 7/31/01 Time 12:54

AZ-101 A		AZ-101 B		AZ-101 C	
Total	9.5 ml	Total	10.0 ml	Total	10.2 ml
Solids	2.1 ml	Solids	2.4 ml	Solids	2.3 ml
Liquids	7.4 ml	Liquids	7.6 ml	Liquids	7.9 ml

AZ-101 D		AZ-101 E		AZ-101 F	
Total	9.35 ml	Total	9.10 ml	Total	9.8 ml
Solids	1.5 ml	Solids	1.5 ml	Solids	1.6 ml
Liquids	7.85 ml	Liquids	7.6 ml	Liquids	8.2 ml

23) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date 7/31/01 Time 15:18

AZ-101 A

Total 9.55 ml
Solids 1.5 ml
Liquids 8.0 ml

AZ-101 B

Total 10.0 ml
Solids 1.6 ml
Liquids 8.4 ml

AZ-101 C

Total 10.2 ml
Solids 1.7 ml
Liquids 8.5 ml

AZ-101 D

Total 9.35 ml
Solids 1.05 ml
Liquids 8.3 ml

AZ-101 E

Total 9.1 ml
Solids 1.15 ml
Liquids 8.0 ml

AZ-101 F

Total 9.8 ml
Solids 1.2 ml
Liquids 8.6 ml

24) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 8/1/01 Time 8:00 am

AZ-101 A

Total 9.50 ml
Solids 1.0 ml
Liquids 8.5 ml

AZ-101 B

Total 10.0 ml
Solids 1.05 ml
Liquids 9.0 ml

AZ-101 C

Total 10.2 ml
Solids 1.2 ml
Liquids 9.0 ml

AZ-101 D

Total 9.40 ml
Solids 0.7 ml
Liquids 8.7 ml

AZ-101 E

Total 9.1 ml
Solids 0.80 ml
Liquids 8.3 ml

AZ-101 F

Total 9.8 ml
Solids 0.80 ml
Liquids 9.0 ml

25) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 8/1/01 Time 3:00 PM

AZ-101 A

Total 9.50 ml
Solids 1.0 ml
Liquids 8.5 ml

AZ-101 B

Total 10.0 ml
Solids 1.1 ml
Liquids 8.9 ml

AZ-101 C

Total 10.2 ml
Solids 1.2 ml
Liquids 9.0 ml

AZ-101 D

Total 9.40 ml
Solids 0.75 ml
Liquids 8.7 ml

AZ-101 E

Total 9.10 ml
Solids 0.80 ml
Liquids 8.3 ml

AZ-101 F

Total 9.8 ml
Solids .90 ml
Liquids 8.9 ml

26) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 8/2/01 Time 8:45

AZ-101 A

Total 9.5 ml
Solids 1.0 ml
Liquids 8.5 ml

AZ-101 B

Total 10.0 ml
Solids 1.1 ml
Liquids 8.9 ml

AZ-101 C

Total 10.2 ml
Solids 1.2 ml
Liquids 9.0 ml

8/2/01 @ 8:45

	AZ-101 D	AZ-101 E	AZ-101 F
Total	9.4 ml	9.1 ml	9.8 ml
Solids	0.75 ml	.80 ml	0.9 ml
Liquids	8.7 ml	8.3 ml	8.9 ml

27) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 8/2/01 Time 15:00

	AZ-101 A	AZ-101 B	AZ-101 C
Total	9.5 ml	10.0 ml	10.2 ml
Solids	1.0 ml	1.1 ml	1.2 ml
Liquids	8.5 ml	8.9 ml	9.0 ml

	AZ-101 D	AZ-101 E	AZ-101 F
Total	9.4 ml	9.1 ml	9.8 ml
Solids	0.75 ml	0.8 ml	0.9 ml
Liquids	8.7 ml	8.3 ml	8.9 ml

28) At the beginning of the next day (day 4), record the volume of the total sample and the volume of settled solids.

Date 8-3-01 Time 9 Am

	AZ-101 A	AZ-101 B	AZ-101 C
Total	9.5 ml	10.0 ml	10.2 ml
Solids	1.0 ml	1.1 ml	1.2 ml
Liquids	8.5 ml	8.9 ml	9.0 ml

	AZ-101 D	AZ-101 E	AZ-101 F
Total	9.4 ml	9.1 ml	9.8 ml
Solids	0.75 ml	0.8 ml	0.9 ml
Liquids	8.7 ml	8.3 ml	8.9 ml

29) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

Cones

AZ-101 A

Total 31.011 g
Tare ________ g
Slurry ________ g

Total 9.5 ml
Solids 0.45 ml
Liquids ________ ml

AZ-101 B

Total 32.116 g
Tare ________ g
Slurry ________ g

Total 10.0 ml
Solids 0.5 ml
Liquids ________ ml

AZ-101 C

Total 32.156 g
Tare ________ g
Slurry ________ g

Total 10.2 ml
Solids 0.5 ml
Liquids ________ ml

AZ-101 D

Total 30.026 g
Tare ________ g
Slurry ________ g

Total 9.3 ml
Solids 0.35 ml
Liquids ________ ml

AZ-101 E

Total 29.759 g
Tare ________ g
Slurry ________ g

Total 9.1 ml
Solids 0.35 ml
Liquids ________ ml

AZ-101 F

Total 31.148 g
Tare ________ g
Slurry ________ g

Total 9.75 ml
Solids 0.45 ml
Liquids ________ ml

Calculations on spreadsheet
PRB 8/8/01

30) Decant as much of the centrifuged supernatant as possible into a pre-weighed graduated cylinder and record the mass and volume of the supernatant. Weigh the solids left in the centrifuge cone.

AZ-101 A

Liquid+Grad 46.344 g
Grad 35.424 g
Liquid 10.920 g
Liquid 9.00 ml
ρ = 1.2133 g/mL
Solids+cone 20.055 g
Cone ________ g
Solids ________ g

AZ-101 B

Liquid+Grad 47.068 g
Grad 35.424 g
Liquid 11.644 g
Liquid 9.60 ml
ρ = 1.2124
Solids+cone 20.562 g
Cone ________ g
Solids ________ g

AZ-101 C

Liquid+Grad 47.218 g
Grad 35.424 g
Liquid 11.794 g
Liquid 9.70 ml
ρ = 1.2159
Solids+cone 20.417 g
Cone ________ g
Solids ________ g

AZ-101 D

Liquid+Grad 34.535 g
Grad 24.311 g
Liquid 10.224 g
Liquid 8.40 ml
ρ = 1.217
Solids+cone 19.469 g
Cone ________ g
Solids ________ g

AZ-101 E

Liquid+Grad 34.742 g
Grad 24.311 g
Liquid 10.431 g
Liquid 8.50 ml
ρ = 1.227
Solids+cone 19.304 g
Cone ________ g
Solids ________ g

AZ-101 F

Liquid+Grad 35,350 g
Grad 24.311 g
Liquid 11.039 g
Liquid 9.10 ml
ρ = 1.213
Solids+cone 19,971 g
Cone ________ g
Solids ________ g

8/3/01 → Balance CK - C-cell Balance 100g = 100.001 g OK

8-3-01 rgs

31) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded vials.

AZ-101 A-L
Total 27.429 g
Tare 16.645 g
Liquid ______ g

AZ-101 B-L
Total 28.372 g
Tare 16.852 g
Liquid ______ g

AZ-101 C-L
Total 28.482 g
Tare 16.806 g
Liquid ______ g

AZ-101 D-L
Total 26.884 g
Tare 16.783 g
Liquid ______ g

AZ-101 E-L
Total 27.054 g
Tare 16.753 g
Liquid ______ g

AZ-101 F-L
Total 27.540 g
Tare 16.678 g
Liquid ______ g

32) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.
Air dried over the weekend rjs/8/6/01

33) Transfer the solids and liquids to an oven at 105°C for 24 hours. Record the time and date.

Time 14:30 Date 8/6/01

34) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh. Record the time and date.

Time 08:50 Date 8/7/01

AZ-101 A
Total 19.737 g
Tare ______ g
Liquid ______ g

AZ-101 B
Total 20.284 g
Tare ______ g
Liquid ______ g

AZ-101 C
Total 20,131 g
Tare ______ g
Liquid ______ g

AZ-101 AL
Total 20.342 g
Tare ______ g
Liquid ______ g

AZ-101 BL
Total 20.898 g
Tare ______ g
Liquid ______ g

AZ-101 CL
Total 20.842 g
Tare ______ g
Liquid ______ g

Calculations not needed
Paul Br
5/28/02

AZ-101 D

Total 19.142 g
Tare ______ g
Liquid ______ g

AZ-101 E

Total 19.016 g
Tare ______ g
Liquid ______ g

AZ-101 F

Total 19.579 g
Tare ______ g
Liquid ______ g

AZ-101 DL

Total 20.312 g
Tare ______ g
Liquid ______ g

AZ-101 EL

Total 20.298 g
Tare ______ g
Liquid ______ g

AZ-101 FL

Total 20.450 g
Tare ______ g
Liquid ______ g

calculations not needed
PRB
5/28/02

35) Remove the caps and transfer the solids and liquids to an oven at 105°C for 24 hours. Record the time and date.

Time ______ Date ______

36) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh. Record the time and date.

See step 37
PRB 12/17/01

Time ______ Date ______

AZ-101 A

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 B

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 C

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 D

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 E

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 F

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 AL

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 BL

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 CL

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 DL

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 EL

Total ______ g
Tare ______ g
Liquid ______ g

AZ-101 FL

Total ______ g
Tare ______ g
Liquid ______ g

37) If needed, repeat steps 35 and 36 until the samples loose no more than ~~5~~ 1-2 rgs 8/1/01 % of their mass during over a 24 hour period at 105°C. Record the total wt. data approaching a constant mass below:.

	ID AZ-101-	Date/Time 9:35AM 8/7/01 Wt (g)	Date/Time 8/8/01 Wt (g)	Date/Time 8/9/01 Wt (g)	Date/Time 8/10/01 Wt (g)	Date/Time 8/13/01 Wt (g)
Aliquoted from CHEM-2 Bottle	A	19.737	19.708	19.711	—	—
	B	20.284	20.170	20.167	—	—
	C	20.131	20.030	20.023	—	—
AR-E Bottle	D	19.142	19.140	19.141	—	—
	E	19.016	18.986	18.982	—	—
	F	19.579	19.579	19.579	—	—
CHEM 2	A-L	20.392	19.668	19.558	19.528	19.523
	B-L	20.848	20.520	20.092	19.971	19.968
	C-L	20.842	20.449	20.020	19.940	19.934
AR-E	D-L	20.312	19.961	19.598	19.529	19.519
	E-L	20.298	20.064	19.666	19.539	19.531
	F-L	20.450	19.980	19.701	19.586	19.585
	Balance chk wt. 100.002 gram		100.001	100.000	100.002	100.003

wts. taken by Jeff Chenault, 8/13/01
in my Absense/Vacation rg3

ATTACHMENT: TI-RPP-WTP-095 PAGE 1 of ~~15~~ 10
KS 7/10/02

Test Plan: TI-RPP-WTP-095 Rev. 0

Physical Testing: Feed Characterization Summary Report.
Tank and Test Matrix ---- **AZ-101**

Date: 08/07/2001

Step #	Description	units	**AZ-101-CHEM-2-A**	**AZ-101-CHEM-2-B**	**AZ-101-CHEM-2-C**
3	Centrifuge cone tare	g	19.387	19.821	19.690
5a	Total Wt after transfer	g	31.013	32.127	32.164
5b	Total Initial Volume	mL	9.50	10.00	10.20
7a	72 hr total Vol	mL	9.50	10.00	10.20
7b	72 hr total settled solids	mL	1.00	1.10	1.20
8a	Total Wt after centrifuging	g	31.011	32.116	32.156
8b	Total Volume after centrifuging	ml	9.50	10.00	10.20
8c	Solids volume mark after cent.	ml	0.45	0.50	0.50
9a	Tare Wt of Graduate	g	35.424	35.424	35.424
9b	Liquid plus Graduate wt.	g	46.344	47.068	47.218
9c	Liquid Volume in graduate	mL	9.00	9.60	9.70
10	Total Wet Solids Wt. + cone	g	20.055	20.562	20.417
11a	Tare Wt of 20mL Vial	g	16.645	16.852	16.806
11b	Liquid plus 20mL Vial wt.	g	27.429	28.372	28.482
16a	Final Dry Wt. of Cone	g	19.711	20.167	20.023
16b	Final Dry Wt. of 20mL Vial	g	19.523	19.968	19.934

Calc. Step	Description	units	A	B	C	AVE	std.dev
1	Bulk Density	g/mL	1.224	1.231	1.223	1.226	0.00
2	Vol % Settled Solids	%	10.5%	11.0%	11.8%	11.1%	0.6%
3	Density of Centrifuged Solids	g/mL	1.484	1.482	1.454	1.473	0.02
4	Vol % Centrifuged Solids	%	4.7%	5.0%	4.9%	4.9%	0.1%
5	Wt. % Centrifuged Solids	%	5.7%	6.0%	5.8%	5.9%	0.1%
6	Supernatant Density	g/mL	1.213	1.213	1.216	1.214	0.00
7	Density of Settled Solids	g/mL	1.313	1.374	1.276	1.321	0.05
8	Wt. % Settled Supernatant	%	88.7%	87.7%	87.7%	88.1%	0.6%
9	Wt.% Total Solids	%	28.0%	28.2%	27.9%	28.0%	0.2%
10	Wt. % Undissolved Solids	%	1.7%	1.6%	1.5%	1.6%	0.1%

Analyst: 8/13/01

reviewed by 8/15/01

Attachment: TI-RPP-WTP-095 Page 2 of ~~15~~ 10
KS 7/10/02

Test Plan: TI-RPP-WTP-095 Rev. 0

Physical Testing: Feed Characterization Summary Report.

Tank and Test Matrix ---- **AZ-101**

Date: 08/07/2001

Step #	Description	units	**AZ-101-AR-E** D	**AZ-101-AR-E** E	**AZ-101-AR-E** F
3	Centrifuge cone tare	g	18.879	18.697	19.265
5a	Total Wt after transfer	g	30.279	29.874	31.167
5b	Total Initial Volume	mL	9.35	9.10	9.80
7a	72 hr total Vol	mL	9.40	9.10	9.80
7b	72 hr total settled solids	mL	0.75	0.80	0.90
8a	Total Wt after centrifuging	g	30.026	29.759	31.148
8b	Total Volume after centrifuging	ml	9.30	9.10	9.75
8c	Solids volume mark after cent.	ml	0.35	0.35	0.45
9a	Tare Wt of Graduate	g	24.311	24.311	24.311
9b	Liquid plus Graduate wt.	g	34.535	34.742	35.350
9c	Liquid Volume in graduate	mL	8.40	8.50	9.10
10	Total Wet Solids Wt. + cone	g	19.469	19.304	19.971
11a	Tare Wt of 20mL Vial	g	16.783	16.753	16.678
11b	Liquid plus 20mL Vial wt.	g	26.884	27.054	27.540
16a	Final Dry Wt. of Cone	g	19.141	18.982	19.579
16b	Final Dry Wt. of 20mL Vial	g	19.519	19.531	19.585

Calc. Step	Description	units	D	E	F	AVE	std.dev
1	Bulk Density	g/mL	1.226	1.228	1.221	1.225	0.00
2	Vol % Settled Solids	%	8.0%	8.8%	9.2%	8.7%	0.6%
3	Density of Centrifuged Solids	g/mL	1.686	1.734	1.569	1.663	0.09
4	Vol % Centrifuged Solids	%	3.8%	3.8%	4.6%	4.1%	0.5%
5	Wt. % Centrifuged Solids	%	5.3%	5.5%	5.9%	5.6%	0.3%
6	Supernatant Density	g/mL	1.217	1.227	1.213	1.219	0.01
7	Density of Settled Solids	g/mL	1.162	1.239	1.228	1.210	0.04
8	Wt. % Settled Supernatant	%	92.4%	91.1%	90.7%	91.4%	0.9%
9	Wt.% Total Solids	%	28.0%	28.1%	27.8%	28.0%	0.1%
10	Wt. % Undissolved Solids	%	1.2%	1.5%	1.4%	1.4%	0.1%

Analyst: 8/13/01

reviewed by: 8/15/01

Attachment: TI-RPP-WTP-095 Page 3 of ~~15~~ 10
KS 7/10/02

Composite Jar				Content Jar				
Number	Size (mL)	Tare Weight (g)	Net Weight (g)	Number	Segment	Tare Weight (g)	Net Weight (g)	Physical State
18729	500	288.66	521.05	18402	10	304.84	369.45	Supernate
				18413	16	456.89 a	151.69	Supernate
18725	500	294.6	516.37	18406	9	305.49	365.74	Supernate
				18401	15	303.41	106.47	Supernate
				18413	16	608.58 a	44.22	Supernate
18726	500	286.48	522.64	18403	11	303.36	365.33	Supernate
				18413	16	299.42	157.47	Supernate
18728	500	288.94	516.16	18407	8	301.12	366.05	Supernate
				18401	15	409.88 a	150.22	Supernate
18727	500	286.57	519.89	18393	7	298.65	360.69	Supernate
				18400	14	301.5	58.03	Supernate
				18401	15	560.1 a	102.08	Supernate
18724	500	289.79	516.99	18391	6	301.17	361.81	Supernate
				18400	14	359.53 a	155.29	Supernate
18723	500	295.92	523.69	18394	5	300.49	366.57	Supernate
				18408	13	303.44	40.5	Supernate
				18400	14	514.9 a	116.83	Supernate
18720	500	302.6	522.2	18399	4	298.04	359.3	Supernate
				18408	13	343.94 a	163.06	Supernate
18722	500	296.95	521.86	18398	3	304.08	345.82	Supernate
				18390	12	302.76	30.44	Supernate
				18408	14	507 a	146.43	Supernate
18721	500	296.26	521.13	18397	2	302.22	352.1	Supernate
				18390	12	333.2 a	169.45	Supernate
18719	500	290.46	508.24	18396	1	299.7	344.6	Supernate
				18390	12	502.64 a	163.7	Supernate
16029	250	218.07	220.09	17331	17ALH	122.86	129.4	Wet Sludge
				15888	17AUH	130.71	90.69	Sludge-Slurry
17023	250		307.2	same	17		NA	Solids
15958	250		133.7	same	18		NA	Solids
17025	250		388.2	same	18A		NA	Solids
Total			**958.5**					**Solids**
			5800.91					**Supernate**

Note: For core segment 17, the upper half (17AUH) was counted as liquid and the lower half (17ALH) as solids

a Denotes the weight of a bottle that is not completely empty; not a tare weight.

liquid jars 11 → 5.5 l

solids 1045g $\xrightarrow{1.5 g/ml}$ 698 ml

6.2 l

Attachment to
TI-RPP-WTP-~~087~~ ~~95~~ ~~110~~ 095
Paul B
12/17/01

page 4 of ~~8~~ 10

KS 7/10/02

Bredt, Paul R

From: Lee, Ernest
Sent: Thursday, September 20, 2001 1:27 PM
To: Bredt, Paul R
Cc: Kurath, Dean E; Urie, Michael W; Burgeson, Ingrid E; Roosa, Roger K; Graves, William; Slaathaug, Eric J
Subject: RE: AZ-101 Characterization Physical Properties Measurement Scope Reduction

Subject: AZ-101 Characterization Physical Properties Measurement Scope Reduction

References: 1) TP-RPP-WTP-089, Rev. 1, AZ-101 Homogenization and As-Received Characterization, Battelle Test Plan, 8/14/2001.

2) 24590-PTF-TSP-RT-01-001, Rev. 0, Tank 241-AZ-101 Sample composite, Homogeneity, and Analysis, 7/2/2001.

WPT R&T understands that only 1 wt% undissolved solids are present in the composited AZ-101 sample rather than 3 wt% undissolved solids that was expected. The WTP R&T acknowledges and accepts the requirement to use 950 grams of composited sample for physical property measurement rather than the original projected amount of 420-550 grams. .

WTP R&T also agrees that the scope of the physical properties testing must be reduced, because of the lack of undissolved solids. After conferring with WTP Process Engineering, WPT R&T is providing the following direction to Battelle

Original Test Specification Requirement (Ref. 2)

Remove Paragraph 5.4.2

Revise Paragraph 5.4.5 in Reference 2 to the following

Adjust the wt% undissolved solids to 3 wt%, and 20 wt% and analyze to determine slurry density, wt% total solids, wt% dissolved solids, Wt% undissolved solids, yield strength, shear stress Vs shear rate, and heat capacity. The yield strength, shear stress versus shear rate and heat capacity is to be done at two temperature (25°C and 40°C) for 3 wt% undissolved solids in triplicate at 25°C and up to triplicate at 40°C depending on material availability. The yield strength, shear stress versus shear rate and heat capacity is to be done at one temperature (approximately 25°C) for 20 wt% undissolved solids in triplicate.

additional changes to test specification: Remove all references to performing physical property measurements on 13 wt% undissolved solids.

This change will be reflected in the next revision of the AZ-101 Test.

Ernie Lee
Research Engineer
Research & Technology

Attachment to
Test Instruction
TI-RPP-WTP-~~040~~ 095
Page 5 of ~~15~~ 10

KD
7/10/02

Bredt, Paul R

From: Lee, Ernest [edlee@bechtel.com]
Sent: Thursday, October 11, 2001 1:35 PM
To: DEAN E KURATH (E-mail); Lynette Jagoda (E-mail)
Cc: Paul Bredt (E-mail); Arakali, Aruna
Subject: Ref. AZ-101 Physical Property Testing

This message is in response to today's telecom regarding rheology measurements on 20 wt% undissolved solids. I have discussed the issue with BNI Process Engineering. It was agreed that instead of performing the analysis on 20 wt% centrifuged solids, to decant the supernate from the gravity settled solids and perform the measurement on the remaining sludge. It is my understanding that the wt% undissolved solids will be 14 wt% or higher after decanting.

If additional AZ-101 solids are located in the 222-S Lab, then an additional measurement should be made another solids concentration that is higher than 12% undissolved solids.

Ernie

Attachment: TI-RPP-WTP-095 page 6 of ~~15~~ 10

KS 7/10/02

Bredt, Paul R

From: Lee, Ernest
Sent: Monday, July 16, 2001 4:55 PM
To: Urie, Michael W; Lee, Ernest
Cc: Bredt, Paul R; Kurath, Dean E; Beeman, Gordon H
Subject: RE: Lithium and Bromide analysis of AZ-101

I plan to send you the following message in a letter. Can you start with compositing before you receive it?

Ernie

Reference: 1) TP- RPP-WTP-089, Rev. 0, from Battelle "AZ-101

Homogenization and AS-Received Characterization," July 16, 2001.

I have reviewed the attached results for lithium and bromide analysis from analyzing individual sub-samples from the "as-received" sample jars. The concentration of bromide are above the criteria in reference 1 and lithium concentration is well below the criteria. However, if sample contamination occurred from the hydrostatic head fluid both Lithium and bromide would be present in about the same molar concentration.

It is concluded that individual samples were not contaminated with hydrostatic head fluid and the tank waste contains bromide.

BNI approves compositing all the sample jars into a single composite sample as specified in reference 1.

Very truly yours,

Ernest Lee
Lead Engineer, Characterization
WPT R&T

edl

-----Original Message-----

From: **Urie, Michael W [mailto:mike.urie@pnl.gov]**
Sent: Monday, July 16, 2001 2:21 PM
To: Lee, Ernest
Cc: Bredt, Paul R; Kurath, Dean E; Beeman, Gordon H
Subject: Lithium and Bromide analysis of AZ-101

Ernie, The lithium and bromide results are in the table below. I have extracted only the results (e.g., I have provide no QC information, detection limits, etc.) As you can see, the samples appear to have bromide (at various levels) but no lithium. After you have evaluated the data, we need to have your approval to proceed with compositing.

RPL Number	Sample ID	Br ug/mL	Li ug/mL
Target: 0.003 M		240	20.7
01-1381	18729 LiBr	1160	< 1.2
01-1381 Dup	18729 LiBr Dup		< 1.3
RPD		n/a	

Attachment: TI-RPP-WTP-095 page 7 of ~~15~~ 10

KD 7/10/02

01-1382	18725 LiBr	950	< 1.3
01-1383	18726 LiBr	1200	< 1.3
01-1384	18728 LiBr	420	< 1.3
01-1384 Dup	18728 LiBr Dup	440	
RPD	5%		
01-1385	18727 LiBr	920	< 1.3
01-1386	18724LiBr	1240	2.7
01-1387	18723LiBr	200	< 1.3
01-1388	18720LiBr	410	< 1.3
01-1389	18722 LiBr	1090	< 1.3
01-1389 Dup	18722 LiBr Dup		< 1.3
RPD	n/a		
01-1390	18721LiBr	740	< 1.3
01-1391	18719 LiBr	230	< 1.3
01-1391 Dup	18719 LiBr Dup	230	
RPD	0%		

Mike

Michael W. Urie
Radiochemical Processing Group
Pacific Northwest National Laboratory
509-376-9454
mike.urie@pnl.gov

attachment to Test Instruction
TI-RPP-WTP-095 page 8 of ~~15~~ 10
Paul Bredt
12/17/01
KD 1/10/02

Bredt, Paul R

From: Lee, Ernest
Sent: Monday, July 30, 2001 4:32 PM
To: Bredt, Paul R; Lee, Ernest
Cc: Kurath, Dean E; Beeman, Gordon H; Roosa, Roger K; Graves, William; Harbour, John R
Subject: RE: AZ-101 Sub Sampling Data, Concurrence to Proceed

The Test Plan and Test Specification for AZ-101 contains administrative hold points for R&T to review the data package resulting from measuring sub-sample vol% solids after compositing AZ-101. I have reviewed the data package the Battelle as sent to me via E-mail. The criteria for compositing provided in step 5.2.15 1 of TP-RPP-WTP-089 rev 0, is demonstrated to be meet. However, the criteria in Step 5.2.15 2 (5% vol solids change for best fit line) was exceeded, 8 vol% solids change for best fit line.

The vol % solids change best fit line criteria was not met. The criteria was likely flawed because it did not account for different sub-sample container sizes (125 mL and 500 mL sample bottles) and different bottle aspect ratios. The difference in sample bottle geometry will cause identically slurry to settle at different rates and show a difference vol% settled solids.

Therefore, WTP R&T is providing provisional concurrence to proceed with the next step of characterization accordance with the test plan provided the following minor change occurs. Proceed with determining the wt% solids in triplicate. However, instead for determining the wt% solids on a single sample, perform the triplicate wt% solids analysis on both a 125 ml sub-sample bottle and a 500 mL sub-sample bottle. WTP R&T request to review the results of the above analysis before confirming that compositing of AZ-101 is adequate. It recommended to use the previously designated 125 mL sub-sample for performing the wt solids and either the 15 or 16 sample vial collected (based on the sequence that they were collected) for the 500 mL bottle wt% solids analysis.

Ernie Lee

-----Original Message-----

From: **Bredt, Paul R [mailto:paul.bredt@pnl.gov]**
Sent: Monday, July 30, 2001 1:36 PM
To: Lee, Ernest
Cc: Kurath, Dean E; Beeman, Gordon H
Subject: RE: AZ-101 Subsampling Data

Ernie,

Attaches is the file modified with the latest AZ-101 data that has been checked by Dean. At this point we would like your approval to proceed with testing the solids content of two of the bottles in triplicate (one 125 ml bottle and one 500 ml bottle). This testing will only consume ~50ml of the slurry. An email message will be sufficient based on our test plan.

<<AZ101-inventory 073001.xls>>

Thanks,
Paul

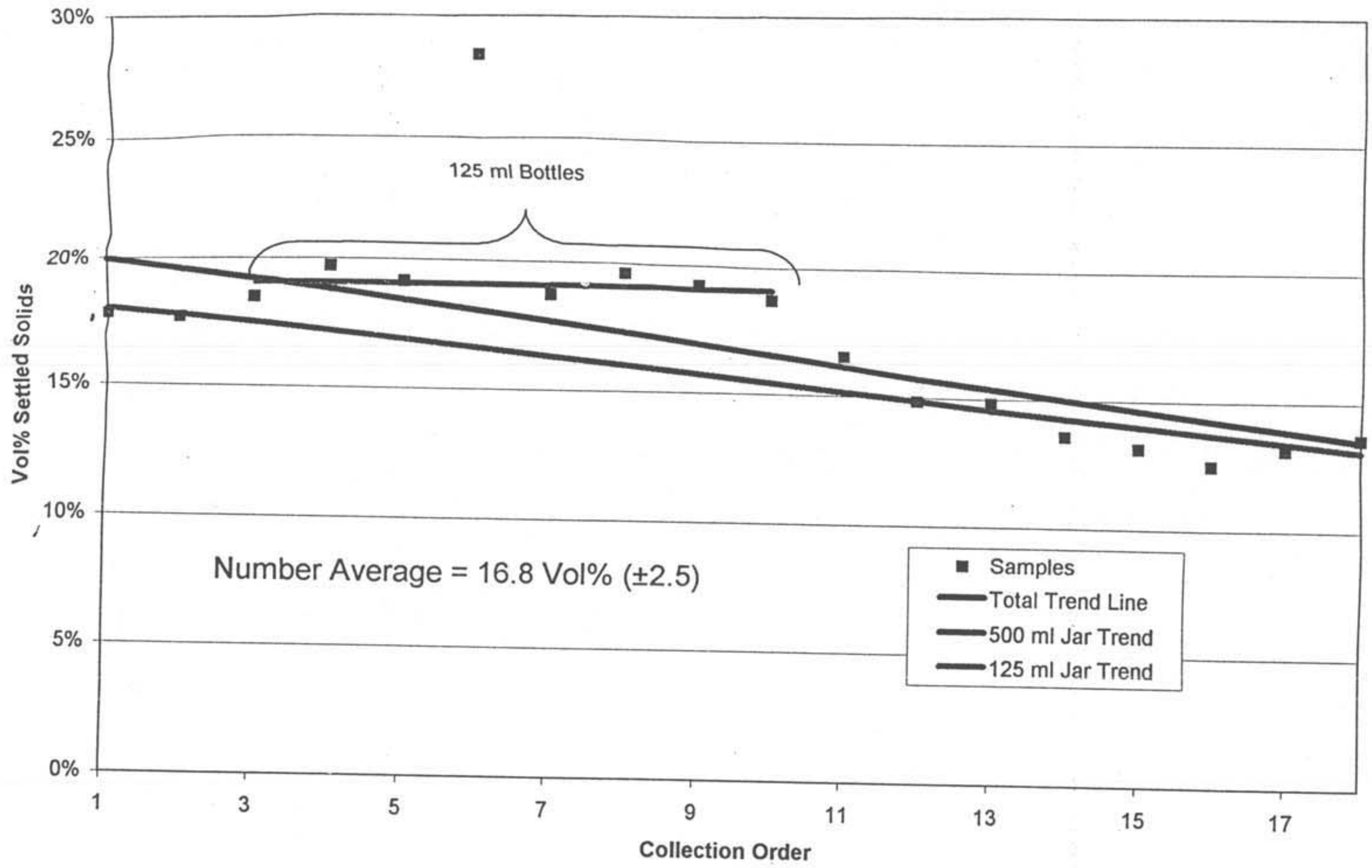
PS- As you noted, the first two bottles were 500ml.

-----Original Message-----

From: **Bredt, Paul R**

Attachment to
TI-RPP-WTP-095

AZ-101 Volume Percent Settled Solids Versus Collection Order



AZ101-inventory 073001.xls

page 9 of ~~15~~ 10
KD 7/10/02

D02-028

[illegible]

Attachment: TI-RPP-WTP-095 Page 10 of 15 10

KS 1/10/02

Raw Data **AZ-101**

SAMPLE TRACKING AND MATERIAL BALANCE

Date: 07/25/01

test plan: TI-RPP-WTP-095 Rev 0

As Received Samples

Initial Container ID	Mass (Gross) (g)	Empty Weight with residue (g)	Net Weight (g)
15958	364.81	237.27	127.54
16029	449.46	241.32	208.14
17023	513.07	238.15	274.92
17025	601.62	236.03	365.59
18719	805.33	303.78	501.55
18720	821.90	306.50	515.40
18721	820.47	308.89	511.58
18722	825.35	305.44	519.91
18723	824.99	306.95	518.04
18724	813.81	302.61	511.20
18725	817.00	305.01	511.99
18726	815.90	298.46	517.44
18727	814.16	297.57	516.59
18728	811.16	301.10	510.06
18729	817.08	304.20	512.88
all depleted	10916.1		6622.8

Composite Samples

After Compositing and Mixing- Sub- Sample ID	Current Loc	Mass (Gross) (g)	Bottle Tare Weight (g)	Net Weight (g)	Approx Density g/mL	Total Vol (oz)	Solids Vol (oz)	Total Volume (mL)	Solids Volume (mL)	Vol % Solids	Collect order
AZ-101-PCB-1	HLRF	300.14	132.81	167.33	1.29			130	24	18.5%	3
AZ-101-PCB-2	HLRF	290.63	132.84	157.79	1.24			127	25	19.7%	4
AZ-101-PCB-3	HLRF	295.34	132.95	162.39	1.27			128	25	19.1%	5
AZ-101-CHEM-1	HLRF	305.74	132.34	173.40	1.33			130	37	28.5%	6
AZ-101-CHEM-2	HLRF	289.23	133.28	155.95	1.27			123	23	18.7%	7
AZ-101-CHEM-3	HLRF	290.59	133.34	157.25	1.26			125	25	19.6%	8
AZ-101-CHEM-4	HLRF	309.52	132.72	176.80	1.36			130	25	19.2%	9
AZ-101-RHEO-3%	HLRF	287.54	133.22	154.32	1.25			123	23	18.7%	10
AZ-101-RHEO-13%	HLRF	628.5	320.66	307.87	1.27	8.2	1.5	242	43	17.7%	2
AZ-101-RHEO-20%	HLRF	805.3	318.97	486.34	1.28	12.9	2.3	381	68	17.8%	1
AZ-101-AR-A	HLRF	845.93	318.09	527.84	1.26	14.2	2.4	420	69	16.5%	11
AZ-101-AR-B	HLRF	888.82	320.94	567.88	1.24	15.5	2.3	458	68	14.8%	12
AZ-101-AR-C	HLRF	885.13	318.72	566.41	1.24	15.5	2.3	458	68	14.8%	13
AZ-101-AR-D	HLRF	900.67	319.85	580.82	1.27	15.5	2.1	458	62	13.5%	14
AZ-101-AR-E	HLRF	901.47	319.35	582.12	1.23	16.0	2.1	473	62	13.1%	15
AZ-101-AR-F	HLRF	905.35	318.48	586.87	1.24	16.0	2.0	473	59	12.5%	16
AZ-101-AR-G	HLRF	902.18	320.83	581.35	1.23	16.0	2.1	473	62	13.1%	17
AZ-101-AR-H	HLRF	528.85	320.57	208.28	1.19	5.9	0.8	174	24	13.6%	18
		10561.0		6301.01					Ave	17.2%	
									STD	3.8%	

Rejecting High and Low (Chem 1 and AR-F)

Ave	16.8%
STD	2.5%

1oz = 29.56 mL

Material Balance	
Start wt	6622.8
Recovered	6301.0
wt. lost	321.8
% loss	4.86%

(Attached)

OK, but it seems like you should provide a basis for rejecting these.

Calcs OK. Some formatting comments.

BEST COPY AVAILABLE

D02-029

filename =

AZ101-inventory 073001.xls

add sheet & page x of y name

7/30/01

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX E

AZ-101 PHYSICAL PROPERTIES

2004

Battelle - Pacific Northwest Division

E1

CORRECTIVE ACTION REPORT # 4181

CORRECTIVE ACTION REPORT

CAR NUMBER: 4181	DATE ISSUED: 5/16/02	QA PLAN NUMBER: RPP-WTP-QAPjP, Rev. 0	PROJECT TITLE AND NUMBER: 42365 River Protection Program - Waste Treatment Plant Technical Support Project

REQUIREMENT AND SOURCE OF REQUIREMENT (Document Title, No., and Revision):

Test Specification number TSP-24590-01-00006 Rev. 0, Tank 241-AZ-101 Sample Composite, Homogenization, and Analysis
Test Plan number TP-RPP-WTP-089, Rev. 0, AZ-101 Homogenization and As-Received Characterization
Test Instruction number TI-RPP-WTP-095, Rev. 0, AZ-101 Sample Homogenization
Test Instruction number TI-RPP-WTP-110, Rev. 0, AZ-101 As-Received Rheological Measurements

CONDITION ADVERSE TO QUALITY:

The physical properties measurements were performed on materials that were not at the weight percent solids requested in the Test Specification and as stated in the Test Plan for AZ-101. Direction from the client was to analyze the AZ-101 feed for slurry density, wt% total solids, wt% undissolved solids, wt% dissolved solids, yield strength, shear stress vs shear rate, and heat capacity at 3, 8wt% undissolved solids. Section 1.6.1.4 of the Test Specification requested this work at 3, 13, and 20wt%, but later client direction modified this to 3, and 8wt%.

However, inhomogeneous subsamples were collected from the as-received material for the initial solids analysis. These initial analyses led researchers to believe the solids concentration in the as-received material was only 1.5wt% undissolved solids when in fact it was 7.6 wt%. As a result, all analysis with the exception of heat capacity were performed at 14 and 45wt% undissolved solids instead of 3 and 8 wt%.

A meeting was held with the client on 3/11/02 to discuss the problem and arrive at a path forward. It was agreed that further analysis at 3 and 8 wt% were not needed, but that the heat capacity analysis which had not been initiated should be conducted at 3, 13, and 20 wt% undissolved solids.

Review by the task lead and technical staff determined that inhomogeneous subsampling was the cause of the problem. The primary sample was stirred with a magnetic stir bar. However, the hot cell where the work was performed has a very cloudy window. As a result of the visibility issue, hot cell staff were not able to confirm that the sample was effectively mixing. In spite of the cloudy window, staff could have confirmed if mixing was effective by using a remote video camera that was present in the hot cell.

SIGNIFICANT CONDITION ADVERSE TO QUALITY (SCAQ)? YES [ ] NO [x]
STOP WORK REQUIRED? YES [ ] NO [x]

CORRECTIVE ACTION RESPONSE DUE DATE: June 3, 2002

REFERENCED REPORTING DOCUMENT: (e.g., Surveillance No.)
N/A

Issued by:
(TL Almeida) TL Almeida 5/16/02
Signature and Date

REMEDIAL ACTIONS - CAQ

Determination of the extent of the adverse condition: This condition only affected analyses listed in section 1.6.1.4 of the test specification. No other activities or data were impacted.

Remedial actions: Future Test Instructions will include direction that mixing operation in the effected hot cell be monitored using a remote video camera.

Estimated completion date: N/A

REMEDIAL ACTIONS - SCAQ

N/A

5/23/02
Approval: Project Manager Signature and Date

TL Almeida 5/23/02
Concurrence: Project QE Signature and Date

CAR NUMBER: 4181	DATE ISSUED: 5/16/02	QA PLAN NUMBER: RPP-WTP-QAPjP, Rev. 0	PROJECT TITLE AND NUMBER: 42365 River Protection Program - Waste Treatment Plant Technical Support Project

CLOSEOUT:

Corrective action has been verified as complete.

Verified by: N/A - No actions
Project QE Signature/Date

DISTRIBUTION:

Project Mgr.: GH Beeman	Project QE: TL Almeida
PR Bredt	AP Poloski
QA Org. (K8-53)	Client

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX F

AZ-101 CHEMICAL/RADIOCHEMICAL CHARACTERIZATION

2004

Battelle - Pacific Northwest Division

F1

ASR 6193
(Addendum 1 and Addendum 2)

Addendum 1

KD

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor — Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature MW Urie
Print Name M W URIE
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: MULTIPLE (See Attached) W57395 FOR LOGGING
Date Required: 10-1-01

Matrix Type Information

- **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ✓ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ASR6193-42365
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) *Sections 4 and 5 apply*
- **Additional QA Requirements?** No ___
  or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ___ Yes ✓
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ✓
  or Reference Doc # ___
  or Previous ASR # ___
  or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ Yes ___

Additional or Special Instructions *The requirements of NQARD apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To M W URIE **Phone** 376-9454
Phone ___

Preliminary results requested, as available? No ___ Yes ✓ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: KJ 8-14-01 8 ~~10~~ 13 TO SAL
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: SAL
ASR Number: 6193
RPL Numbers: (01-1844)-(01-1845)

PAGE 1 of 2 + Addendum 1 (15 pages)

KNP, FVH, JAC, EWH, MLT, MJS, JJW, DRS, LPD, CZS, KKT, TTL, LRG
OTF, JPB

page 1 of 3

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

RPG/CMC Work Accepted By: MW Urie **Signature/Date:** MW Urie ~~10~~ 8/13/01 KN 8-14-01

~~PAGE 1~~

page 2 of 3

F01-003

F01–004

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Library
01-1844	AZ101 SUP AR	SUPERNATANT AZ101	SEE SPECIAL INSTRUCTIONS		
01-1845	AZ101 CS AR	Centrifuged SOLIDS AZ101			
	SEE SPECIAL INSTRUCTIONS				

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193

Page ~~2~~13 of ~~2~~13
KS 7/3/02 KS 7/3/02

Special Test Instructions: ASR 6193 SAL Addendum 1
As Received Analyses for AZ-101 Supernatant Sample 01-1844 and Centrifuged Solids Sample 01-1845

Objective of this Addendum: SAL is to phase separate only 2 of the 4 AZ-101 bottles identified for characterization, and perform only those preparative and distribution activities highlighted below.

Bottles Labeled AZ-101-PCB-1, AZ-101-PCB-2, and AZ-101-PCB-3		
Archive (for PCB bottles)	SAL	-- Use W57930

AZ-101 SUP AR (Supernatant Sample) 01-1844 – LIMS Tests

Density/Solution	SAL	-- Use W57930
(Sample used for this measurement can be used for other analyses)		
Wt% Solids/TDS	SAL	-- Use W57930
Wt% Oxide	SAL	-- Use W57930
Ext-Solvent (for PCBs)	SAL	-- Use W57930 (Hold)
PCB/Pesticides	ORG	-- Use W57930 (Hold)
Digestion-128	SAL	-- Use W57930
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use W57932
(ICP/MS to include Cs and U isotopic)		
GEA-450-CMC	RAD	-- Use CMC K88414
(GEA extended count time to obtain trace gamma emitters)		
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr90-476/408-CMC	RAD	-- Use CMC K88419
Se79-440/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC	RAD	-- Use CMC K88421
(Tc-99 w/o oxidation – pertechnetate)		
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
NH3-ISE	LAB	-- Use W57932
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408
H3-418/474-CMC	RAD	-- Use CMC K88422
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use W57933
Acids/Organic	ORG	-- Use W57933
Phosphates/Organic	ORG	-- Use W57933

AZ-101 CS AR (Centrifuged Solids Sample) 01-1845 – LIMS Tests

Wt% Solids	SAL	-- Use W57930
Wt% Oxide	SAL	-- Use W57930
Ext-Solvent (for PCBs)	SAL	-- Use W57930 (Hod)
PCB/Pesticides	ORG	-- Use W57930 (Hold)
Digestion-129	SAL	-- Use W57930
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use W57932

F01-005

(ICP/MS all analytes except I, Pt, Pd, Rh, Ru)		
Fusion-115	SAL	-- Use W57930
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use W57932
(ICP/MS all analytes except I, Pt, Pd, Rh, Ru and to include Cs and U isotopic)		
GEA-450-CMC	RAD	-- Use CMC K88414
(GEA extended count time to obtain trace gamma emitters)		
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr90-476/408-CMC	RAD	-- Use CMC K88419
Fusion 114 (Iodine option)	SAL	-- Use W57930
ICP/MS (I-127/129 only)	ADV INORG	-- Use W57932
Fusion-116	SAL	-- Use W57930
ICP/MS (Pt,Pd,Rh,Ru only)	ADV INORG	-- Use W57932
Leach/Water-103	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
NH3-ISE	LAB	-- Use W57932
H3-418/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use W57930
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408

QC Information

The analyses are to be conducted per PNNL's web-based Quality Assurance Planning Subject Area, "Conducting Analytical Work in Support of Regulatory Programs". The acceptance QC criteria for the supernatant and wet centrifuged solids are attached in Table 3 and Table 4, respectively.

All bottles containing AZ-101 as received composite are to be received from the HLRF under chain of custody (COC), and any analytical samples transferred from the RPL to other building shall be transferred under a COC.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in **TRIPLATE**.

Preparative or sample analysis QC is to include, preparation blank (**in duplicate for acid digestion and fusion preparations**), sample, duplicate, triplicate, matrix spike, matrix spike duplicate where appropriate, and a LCS. If possible, the matrix spikes and LCS are to include all the analytes of interest to be reported for the specific analysis. The matrix spikes and LCS are to be provided to the SAL by the appropriate cognizant scientist.

SAL Archive Samples:

The bottles labeled AZ-101-PCB-1 through AZ-101-PCB-3 are to be archived for future PCB analysis. Prior to archiving the bottle weights are to be recorded by the SAL. These bottle weights are to be maintained with the archived bottles and filed in the Project file.

Date 8-22-01

SAL operator

Balance ID 1120100979

FH

AZ-101-PCB-1 weight (g) 300.060

AZ-101-PCB-2 weight (g) 290.080

AZ-101-PCB-3 weight (g) 293.579

Phase Separation

The bottles labeled AZ-101-CHEM-1 through AZ-101-CHEM-2 are to be phase separated to obtain an AZ-101 supernatant composite sample and an AZ-101 centrifuged solids composite sample. Bottles AZ-101-CHEM-1 through AZ-101-CHEM-2 are to be centrifuged (in secondary containment) at approximately 1000 time the force of gravity for 1 hour. Transfer all the centrifuged supernatant to tared glass 250 to 500 mL bottle labeled **AZ-101 SUP1 AR (01-1844)** and transfer the wet centrifuged solids to a tared glass 50 to 125 mL bottle labeled **AZ-101 CS1 AR (01-1845)**.

The weights of the supernatant and centrifuged solids samples are to be recorded and sent to the Project file and to M. W. Urie.

Date 8-22-01 SAL operator Balance ID 1120100979

AZ-101 SUP1 AR gross (g) 399.019 AZ-101 CS1 AR gross (g) 206.075
AZ-101-SUP1 AR tare (g) 194.218 AZ-101 CS1 AR tare (g) 132.340
Net (g) 204.801 Net (g) 73.735

Note: Bottles labeled AZ-101-CHEM-3 and AZ-101-CHEM-4 are to be stored. Another SAL Addendum will be written to process the contents of these bottles.

Note: The phase separation process may be conducted by using one of the original bottles as the final container for the solids if the tare weight of the bottle is known. For example, centrifuge bottle AZ-101-CHEM-1, remove supernatant and add slurry from AZ-101-CHEM-2 and centrifuge. If this approach is used, the original bottle must be relabeled **AZ-101 CS1 AR (01-1845)**.

SAL Preparation/Analysis

During all sub-sampling activities, minimize the time that the AZ-101 SUP1 AR and AZ-101 CS1 AR containers are open to the hot cell atmosphere and ensure that they are tightly closed when not in use. The supernatant is to be thoroughly mixed and the wet centrifuged solids are to be thoroughly homogenized (mechanically or manually) prior to any sub-sampling activities. See QC Information Section for QC sample requirements. Table 5 and Table 6 provide the "nominal" quantities to be processed in the SAL for each preparation method and the "nominal" quantities to be distributed to the laboratory for those sub-samples not being processed in the SAL. These tables also identify when the MS, LCS/BS, and Blanks are required in the SAL.

Note: Following any daily sub-sampling activities, the sample bottles AZ-101 SUP1 AR and AZ-101 CS1 AR are to be weighed and the weights recorded and sent to the Project file and to M. W. Urie.

Note: The mass and volume of the supernatant sub-samples process under this Addendum are to be recorded on the distribution benchsheets or preparation benchsheets in both units of mass (g) and volume (mL).

1. Perform the Wt% Total Solids on the wet centrifuged solids sample and TDS on the supernatant per Appendix 1.
2. Following the Wt% Total Solids and the TDS, perform Wt% Oxide on both the Wt% Total Solids and TDS dried samples per Appendix 1.

3. Perform acid digestion and multiple fusion SAL sample preparations (See Table 5 and Table 6) and distribute analytical sub-samples. The preferred processing order is:

   - Fusions (114 Iodine only, 115, and 116)

     Note: If any residual solids remain after any of the fusion processings, note on preparation benchsheet (e.g., estimated quantity, color, texture, etc.) and contact M. W. Urie for further instructions prior to distribution.

     Note: Fusions do not require Matrix Spike for ICP and ICP/MS, but require LCS whenever possible

   - Acid Digestions (both supernatant and wet centrifuged solids)

     Note: If any residual solids remain after acid digestion, note on preparation benchsheet (e.g., estimated quantity, color, texture, etc.) and contact M. W. Urie for further instructions prior to distribution.

     Note: Acid digestions require both Matrix Spikes and LCS/BS for ICP and ICP/MS

Table 2
Liquid Fraction Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method[c]
	mg/L	
Al	7.5E+01	ICP-AES (AA may be used for Na, K)
B	2.3E+00	
Ba	2.3E+00	
Ca	1.5E+02	
Ce	2.3E+00	
Cd	7.5E+00	
Cr	1.5E+01	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Li	2.3E+00	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Th	2.3E+00	
V	2.3E+00	
W	2.3E+00	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TOC	1.5E+03 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
Br	3.0E+02	
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03 (as P)	
SO_4	2.3E+03 (as S)	
	mCi/L (except as noted)	ICP-MS (AA may be used for Cs determination)
Rb	1.0E+00 (mg/L)	
^{127}I	1.5E+00 (mg/L)	
^{129}I	1.8E-05	
^{133}Cs	7.0E-04 (mg/L)	
^{135}Cs	1.5E+00	
^{137}Cs	1.5E+00	
^{237}Np	2.7E-02	

Table 2
Liquid Fraction Analyses

Analyte	**Target Minimum Reportable Quantity**	**Recommended Analysis Method(c)**
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	8.7E-03(mg/L)	
^{99}Tc	1.5E-03	
^{233}U	4.2E-04	
^{234}U	1.2E-04	
^{235}U	4.5E-08	
^{236}U	1.4E-06	
^{238}U	7.2E-09	
^{99}Tc(pertechnetate)	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{3}H	2.1E-02	Separations / Liquid Scintillation
^{14}C	7.2E-04	
^{79}Se	9.0E-05	
^{90}Sr	1.5E-01	
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}$Pu	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}$Cm	1.5E-02	
^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	2.1E-03	
^{126}Sn	6.0E-03	
^{137}Cs	9.0E+00	
^{231}Pa	7.89E-05	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha (TRU)	N/A	Summation(b) of: Pu-238, Pu-239+Pu-240 (or Pu-239, Pu-240 ICP/MS) and Am-241
Total and Free OH	7.5E+04 mg/L	Titration
CN	3.0E+00 mg/L	CN Analysis
Ammonia	1.4E+02 mg/L	ISE or IC
Organic Analytes	**mg/L**	
Oxalate	1.5E+03	Ion Chromatograph
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
EDTA(a)	1.5E+03	Derivatization/ GC-MS
HEDTA(a)	1.5E+03	
D2EHPA(a)	1.5E+03	
NTA(a)	1.5E+03	
IDA(a)	1.5E+03	
Succinic Acid(a)	1.5E+03	
ED3A(a)	1.5E+03	
	Expected Range	
Density	0.95 –1.5 (gm/mL)	Gravimetric
Dissolved solids	1 to 50 (gm solids/gm supernate)	Gravimetric

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Footnote:

(a) MRQs are target values, measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters.

(b) Guidance for reporting summation of isotopics and reporting isotopic values derived by different methods will be provided later after an agreement is obtained with DOE.

(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 3. HLW Solids Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method(c)
	mg/Kg(a)	
Ag	9.0E+02	ICP-AES (AA may be used for Na & K)
Al	3.3E+02	
B	3.0E+00	
Ba	6.0E+02	
Be	3.0E+00	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
K	2.0E+02	
La	6.0E+01	
Li	3.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
U	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
Br	4.5E+02	
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	
CN	3.0E+00	CN analysis
	mg/Kg	ICP-MS
As	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Mo	3.0E+01	
Pd	3.0E+02	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Rh	3.0E+02	
Ru	3.0E+02	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	

Table 3. Continued, HLW Solids Analyses		
Analyte	**Target Minimum Reportable Quantity**	**Recommended Analytical Methods[c]**
W	mg/Kg 6.0E+00	ICP-MS
Y	6.0E+00	
^{99}Tc	6.0E+00	
^{127}I	1.5E+00	
^{133}Cs	7.0E-04	
^{233}U	6.0E+01	
^{235}U	6.0E+00	
^{237}Np	1.8E+00	
^{129}I	mCi/Kg 1.8E-05	
^{135}Cs	5.3E-03	
^{137}Cs	9.0E-02	
^{151}Sm	· TBD	
^{234}U	3.7E-03	
^{236}U	3.8E-04	
^{238}U	2.0E-06	
^{3}H	1.5E-02	Separations / Liquid Scintillation
^{14}C	1.8E-03	
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}$Pu	6.0E+00	
^{242}Pu	3.36E-01	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}$Cm	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
^{125}Sb	6.0E+00	
^{126}Sn	6.0E-02	
^{134}Cs	9.0E-01	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha (TRU)	N/A	Summation[b] of: Pu-238, Pu-239, Pu-240, and Am-241
^{241}Pu	1.2E+00 mCi/Kg	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetric
Density	0.9 to 2.0 gm/ml	Gravimetric
Wt% Undissolved Solids	10 to 50 wt%	Calculation
Wt% Soluble Solids	1 to 50 wt%	Calculation

Footnote:

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[a] MRQs are based on dried solids weighs.

[b] Guidance for reporting summation of isotopics that are derived by different methods will be provided later after an agreement is obtained with DOE

[c] If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Recommended Analytical Technique(i)	QC Flagging Criteria		
		LCS %Recovery(a)	Spike %Recovery(b)	Triplicate RSD(c)
Al, B, Ba, Ca, Cd, Ce, Cr, Fe, K, La, Li, Mg, Ni, P, Pb, Th, U, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
U	Kin. Phosphorescence	80 - 120%	75 - 125%	<15%
Rb, ^{127}I, ^{129}I, ^{133}Cs, Cs^{135}, Cs^{137}, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Pu / ^{241}Am, ^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cl$^-$, Br$^-$, F$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{-3}$, SO$_4^{-2}$	IC	80 - 120%	75 - 125%	<15%
CN$^-$	CN Analysis	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH$_3$/NH$_4^+$	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH- (total and Free)	Potentiometric titration /precipitation	80 - 120%	N/A	<15%
TIC	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(e)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co(f), ^{126}Sn(j), ^{231}Pa	Extended GEA	NP	N/A(g)	<15%
^{79}Se	Liq. scintillation	NP	N/A(e)	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A(e)	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A(g)	<15%
^{154}Eu(f)	GEA	NP	N/A(g)	<15%
^{155}Eu(f)	GEA	NP	N/A(g)	<15%
^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm	Separation/AEA	NP	N/A(e)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Sum of Alpha(h) [TRU]	Calculation	N/A	N/A	N/A
Density	Gravimetric	N/A	N/A	<20%
Wt% dissolved solids	Gravimetric	N/A	N/A	<20%
EDTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
HEDTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Oxalate	IC	80 - 120%	75 - 125%	<15%
Citrate	IC	80 - 120%	75 - 125%	<15%
Formate	IC	80 - 120%	75 - 125%	<15%
Gluconate	IC	80 - 120%	75 - 125%	<15%
Glycolate	IC	80 - 120%	75 - 125%	<15%
D2EPHA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr, use Sr in the standard for determining the total mass-90 concentration.

(e) 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.

(f) 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.

(g) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(h) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting 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 that 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 MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination. If any of the isotopes are below the MRQ, then the method of summation will be agreeed by BNI prior to reporting values.

(i) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(j) Calibrate with Sn-117.

(k) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

(l) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 5. Quality Control Parameters for Solids Analysis

Solids Fraction	Recommended Analytical Technique(o)	QC Flagging Criteria		
		LCS % Recovery(a)	Spike % Recovery(b)	Triplicate RSD(c)
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Cs, Fe, K, La, Mg, Mn, Nd, Ni, P, Pb, Pd, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, U, V, W, mass unit 90(d), ^{99}Tc, ^{127}I, ^{133}Cs, ^{233}U, ^{235}U, ^{237}Np, ^{129}I, ^{135}Cs, ^{137}Cs, ^{151}Sm, ^{234}U, ^{236}U, ^{238}U.	ICP/MS	80 - 120%	70 - 130%	<15%
Cl$^-$, Br$^-$, F$^-$, NO$_2^-$, NO$_3^-$, PO$_4^{-3}$, SO$_4^{-2}$	IC	80 - 120%	75 - 125%	<15%
CN$^-$	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Hg	CVAA	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%
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A(g)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{60}Co(i)	Extended count GEA	NP	N/A(k)	<15%
^{90}Sr(j)	Isotopic specific separation/beta count	75 - 125%	N/A(g)	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{125}Sb(m)	Extended Count GEA	NP	N/A	<15%
^{126}Sn(n)	Extended Count GEA	NP	N/A	<15%
^{134}Cs(h)	Extended Count GEA	NP	N/A	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{152}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{154}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{155}Eu(i)	Extended Count GEA	NP	N/A(k)	<15%
^{241}Am	Extended Count GEA	NP	N/A(k)	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, $^{239/240}$Pu, ^{242}Pu	Separation/AEA	NP	N/A(g)	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A(g)	<15%
^{242}Cm	Separation/AEA	NP	N/A(g)	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A(g)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Bulk density	Gravimetric	N/A	N/A	<20%
Wt% solids	Gravimetric	N/A	N/A	<20%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.

(e) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(f) Not used.

(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 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 WAPS justification.

(i) 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 ^{152}Eu, ^{154}Eu, ^{155}Eu.

(j) Combined analysis of ^{90}Sr and ^{90}Y.

(k) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(l) Combined analysis with ^{93m}Nb.

(m) Combined analysis with ^{125m}Te.

(n) Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

(o) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 5: SAL Supernatant "Nominal" Sample Quantities and In-cell QC

Supernatant	Sample	Duplicate	Triplicate	MS	LCS/BS	Blank
Density	10 ml minimum	10 ml minimum	10 ml minimum	N/A	N/A	N/A
TDS	5 ml	5 ml	5 ml	N/A	N/A	N/A
Wt% Oxide	From TDS	From TDS	From TDS	N/A	N/A	N/A
Digest 128 (1)	1 ml	1 ml	1 ml	1 ml	Required for ICP and ICP/MS (3)	Required
IC	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
Tc-99	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
TOC/TIC PerSul	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
TOC/TIC Furnace	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	(2)	(2)	(2)
C-14	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
Ammonia	1 ml	1 ml	1 ml	(2)	(2)	(2)
Mercury	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
CN	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
H3	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
OH	1 ml	1 ml	1 ml	1 ml	(2)	N/A
Chelators (4)	5 ml	5 ml	5 ml	5 ml Required (3)	Required (3)	Required (5)
Organic Acids (4)	1 ml	1 ml	1 ml	1 ml Required (3)	Required (3)	Required (5)
Organic Phosphates (4)	5 ml	5 ml	5 ml	5 ml Required (3)	Required (3)	Required (5)

(1) Digestate distributed for all analyses identified in the LIMS Tests under digestion heading

(2) MS, LCS/BS, and Blank are part of the laboratory preparations/analysis, not the SAL preps.

(3) MS and LCS/BS obtained from cognizant scientists (Wagner ICP, Farmer ICP-MS, Campbell Chelators and Organic Phosphates, Mong Organic Acids)

(4) SAL preparation activities only involve IX processing for dose reduction per test plan provided by Campbell.

(5) Blanks are to be processed through the IX process. The cognizant scientists are to define the blank matrix (e.g., water, dilute hydroxide, etc.)

Table : SAL Wet Centrifuged Solids Nominal Sample Quantities and In-cell QC

Solids	Sample	Duplicate	Triplicate	MS	LCS/BS	Blank
Wt% Solids	3 g	3 g	3 g	N/A	N/A	N/A
Wt% Oxide	From Wt% Solids	From Wt% Solids	From Wt% Solids From TDS	N/A	N/A	N/A
Digest 129 (1)	0.5 g	0.5 g	0.5 g	0.5 g	Required for ICP and ICP/MS (3)	Required
Fusion 115 (1)	0.2 g	0.2 g	0.2 g	N/A	Required (3)	Required
Fusion 116 (1)	0.2 g	0.2 g	0.2 g	N/A	Required (3)	Required
Fusion 114 (1)	0.2 g	0.2 g	0.2 g	N/A	Required (3)	Required
Water Leach for IC, H3 and ammonia	1 g	1 g	1 g	1 g IC and ammonia [H3 (2)]	Required IC and ammonia [H3 (2)]	Required
TOC/TIC PerSul	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
TOC/TIC Furnace	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	(2)	(2)	(2)
C-14	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
Mercury	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)
CN	0.5 ml	0.5 ml	0.5 ml	(2)	(2)	(2)

(1) Dissolutions distributed for all analyses identified in the LIMS Tests under each dissolution method.

(2) MS, LCS/BS, and Blank are part of the laboratory preparations/analysis, not the SAL preps.

(3) MS and LCS/BS obtained from cognizant scientists (Wagner ICP, Farmer ICP-MS, Campbell Chelators and Organic Phosphates, Mong Organic Acids)

Appendix 1:
Determination of Wt% Total Solids (centrifuged solids), TDS (supernatant), Wt% Oxide (centrifuged solids), and Wt% Oxide (supernatant).

Balance Calibration ID J8914AW
Calibration Expiration Date 2/02
Balance Location Cell #2

Furnace Thermocouple Calibration ID 14088 (oxides) / 3118 (wt% Solids)
Calibration Expiration Date 7/02 (Oxides) / 4/02 (wt% Solids)
Furnace Location Cell #3

1) Transfer about 5 mL of Supernatant (01-1844) into preweighed ceramic crucibles. Weigh the loaded crucibles.
Signature Date 8-27-01 Cell Temperature 29 °C

LA	LB	LC
Total 23.7150 g	Total 22.6155 g	Total 23.3540 g
Tare 17.6844 g	Tare 16.5463 g	Tare 17.2723 g
Liquid 6.0306 g	Liquid 6.0692 g	Liquid 6.0817 g

2) Transfer about 3 g of Wet Centrifuged Solids (01-1845) into preweighed ceramic crucibles. Weigh the loaded crucibles.
Signature Date 8-27-01 Cell Temperature 29 °C

SA	SB	SC
Total 21.0494 g	Total 19.2622 g	Total 19.6466 g
Tare 17.4387 g	Tare 16.4673 g	Tare 16.8688 g
Solids 3.6107 g	Solids 2.7949 g	Solids 2.7778 g

3) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult cognizant scientist on the use of a heat lamp or other drying technique to speed up the preliminary drying, if necessary.

4) Transfer the solids and liquids to an oven at 105 ±3 °C for 24 hours. Record the time and date when place in the oven.
Signature Time 9:00 Date 8-28-01

5) Remove the solids and liquids from the oven. Allow the crucible to cool in desiccator to room temperature (typically about 1 hr) and reweigh. Record the time and date.
Signature Time 9:00 Date 8-29-01

LA	LB	LC
Total 19.3095 g	Total 18.0179 g	Total 18.9143 g
SA	SB	SC
Total 19.9524 g	Total 18.4069 g	Total 18.8178 g

6) Return the solids and liquids to the oven at 105 ±3 °C for 24 hours. Record the time and date.
Signature ________ Time *10:00* Date *8-29-01*

7) Remove the solids and liquids from the oven. Allow the crucible to cool in a desiccator to room temperature (typically about 1 hr) and reweigh. Record the time and date.
Signature ________ Time *9:00* Date *8-30-01* *106° C*

LA Total *19.3190* g	LB Total *18.0250* g	LC Total *18.9215* g
SA Total *19.9502* g	SB Total *18.4075* g	SC Total *18.8208* g

8) As directed by the cognizant scientist, repeat steps 6 and 7 until samples have reached a constant mass. Record the mass data below.

10:00 8-31-01

	3rd Set	4th Set	5th Set	6th Set
Date	*8-31-01*			
LA	*19.3100*			
LB	*18.0180*			
LC	*18.9136*			
SA	*19.9481*			
SB	*18.4048*			
SC	*18.8190*			

9) Place crucibles in furnace.

10) Heat the furnace and hold for 30 minutes at between 1000 and 1050 °C.

11) Turn off the furnace and cool to about 150 °C (typically about 1 hour).

12) Transfer the crucibles using tongs to a desiccator and cool to room temperature (typically 1 to 2 hours).

13) Reweigh the crucibles and record any observations.
Signature ________ Date *8-31-01*

LA Total *18.6590* g	LB Total *17.4036* g	LC Total *18.~~6592~~* *3200* g *8-31-01*

Observations *All solids had a blueish TINT after heating.*

SA Total *19.3239* g	SB Total *17.9204* g	SC Total *18.3536* g

Observations ________

Addendum 2

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor — Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature
Print Name M W URIE
Phone 376-4454 MSIN P7-22

PNNL Project #: 42365
Charge Code: MULTIPLE (See Attached)
W57395 FOR LOGGING
Date Required: ~~10-1-01~~ 11-15-01 MWU

Matrix Type Information

- **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ✓ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ASR6193-42365
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) *Sections 4 and 5 apply*
- **Additional QA Requirements?** No ___
  or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ___ Yes ✓
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ✓
  or Reference Doc # ___
  or Previous ASR # ___
  or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ **Yes** ___

Additional or Special Instructions *The requirements of NQARD apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To M W URIE **Phone** 376-4454
___ **Phone** ___

Preliminary results requested, as available? No ___ Yes ✓ (requesting preliminary results may increase cost)

Receiving and Login Information (*to be completed by laboratory staff*)

Date Delivered: 10-13 TO SAL
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: ___

ASR Number: 6193
RPL Numbers: (01-1844)-(01-1895)

Page 1 of 2
+ Addendum 2 (12 pages)

F01-020

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

RPG/CMC Work Accepted By: MW Thi **Signature/Date:** MW Thi ~~10~~ 9/13/01

Addendum 2
MW Thi 10-04-01

Page 1 (cont)

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only				Lab Staff Use Only	
RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Test	Library
01-1844	AZ101 SUP AR	SUPERNATANT AZ101	SEE SPECIAL INSTRUCTIONS		
01-1845	AZ101 CS AR	Centrifuged SOLIDS AZ101			

SEE SPECIAL INSTRUCTIONS

Addendum 2 (12 pages)

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193

page 2 of 2

F01-022

Special Test Instructions: ASR 6193 SAL Addendum 2
As Received Analyses for AZ-101 Supernatant Sample 01-1844 and Centrifuged Solids Sample 01-1845

Objective of this Addendum: SAL is to phase separate last two bottles of AZ-101 bottles identified for characterization, and perform only those preparative and distribution activities detailed below.

AZ-101 SUP AR (Supernatant Sample) 01-1844 – LIMS Tests

Sub-Sample	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC	RAD	-- Use CMC K88421
(Tc-99 w/o oxidation – pertechnetate)		
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
NH3-ISE	LAB	-- Use W57932
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408
H3-418/474-CMC	RAD	-- Use CMC K88422
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use W57933
Acids/Organic	ORG	-- Use W57933
Phosphates/Organic	ORG	-- Use W57933

AZ-101 CS AR (Centrifuged Solids Sample) 01-1845 – LIMS Tests

Leach/Water-103	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
NH3-ISE	LAB	-- Use W57932
H3-418/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use W57930
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408

QC Information

The analyses are to be conducted per PNNL's web-based Quality Assurance Planning Subject Area, "Conducting Analytical Work in Support of Regulatory Programs". The acceptance QC criteria for the supernatant and wet centrifuged solids are attached in Table 4 and Table 5, respectively.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in **TRIPLATE**.

Preparative or sample analysis QC is to include, preparation blank, sample, duplicate, triplicate, matrix spike, matrix spike duplicate where appropriate, and a LCS. If possible, the matrix spikes and LCS are to include all the analytes of interest to be reported for the specific analysis. The matrix spikes and LCS are to be provided to the SAL by the appropriate cognizant analysis lead.

Phase Separation

The bottles labeled AZ-101-CHEM-3 through AZ-101-CHEM-4 are to be phase separated to obtain an AZ-101 supernatant composite sample and an AZ-101 centrifuged solids composite sample. Bottles AZ-101-CHEM-3 through AZ-101-CHEM-4 are to be centrifuged (in secondary containment) at approximately 1000 time the force of gravity for 1 hour. Transfer all the centrifuged supernatant to tared glass 250 to 500 mL bottle labeled **AZ-101 SUP2 AR (01-1844)** and transfer the wet centrifuged solids to a tared glass 50 to 125 mL bottle labeled **AZ-101 CS2 AR (01-1845)**.

The weights of the supernatant and centrifuged solids samples are to be recorded and sent to the Project file and to M. W. Urie.

Date 10-5-01 SAL operator ______ Balance ID 1120100979

AZ-101 SUP2 AR gross (g)	489.343	AZ-101 CS2 AR gross (g)	188.806
AZ-101-SUP2 AR tare (g)	219.694	AZ-101 CS2 AR tare (g)	133.340
Net (g)	269.649	Net (g)	55.466

Note: The phase separation process may be conducted by using one of the original bottles as the final container for the solids if the tare weight of the bottle is known. For example, centrifuge bottle AZ-101-CHEM-3, remove supernatant and add slurry from AZ-101-CHEM-4 and centrifuge. If this approach is used, the original bottle must be relabeled **AZ-101 CS2 AR (01-1845)**.

SAL Preparation/Analysis

During all sub-sampling activities, minimize the time that the AZ-101 SUP~~1~~2 AR and AZ-101 CS~~1~~2 AR containers are open to the hot cell atmosphere and ensure that they are tightly closed when not in use. The supernatant is to be thoroughly mixed and the wet centrifuged solids are to be thoroughly homogenized (mechanically or manually) prior to any sub-sampling activities. See QC Information Section for QC sample requirements. Table 5 and Table 6 provide the "nominal" quantities to be processed in the SAL for each preparation method and the "nominal" quantities to be distributed to the laboratory for those sub-samples not being processed in the SAL. These tables also identify when the MS, LCS/BS, and Blanks are required in the SAL.

Note: Table 6 and Table 7 are "nominal" sample quantities typically used for the specified analysis. However, prior to sub-sampling the actual target sample quantities (and any special sub-sampling or preservation instructions) need to be provided by the cognizant analysis lead for each analysis.

Note: Following any daily sub-sampling activities, the sample bottles AZ-101 SUP2 AR and AZ-101 CS2 AR are to be weighed and the weights recorded and sent to the Project file and to M. W. Urie.

Note: The mass and volume of the supernatant sub-samples processed under this Addendum are to be recorded on the distribution benchsheets or preparation benchsheets in both units of mass (g) and volume (mL). This requirement applies only when the SAL mass or volume is to be used in calculation of the analyte concentrations (i.e., some sub-samples may not need to be accurately measured since they will be further sub-sampled at the analysis work station).

1. Perform water leach of solids (AZ-101 CS2 AR) using PNL-ALO-103. Use 2.5 g of sample per 25 mL of water.

2. Sub-sample all supernatant (AZ-101 SUP2 AR), solids (AZ-101 CS2 AR), and solids leachate inorganic and radiochemical sub-samples that are to be distributed directly to the laboratories.
3. Perform any additional in-cell inorganic or radiochemistry preparations identified by the cognizant analysis leads (e.g., CN distillations may need to be performed in the SAL) on the supernatant and/or solids and distribute.
4. Perform the IX dose reduction for all the organic analyses and distribute the sub-samples.

Laboratory Analysis

1. The cognizant analysis lead for each of the analytical methods is to review the sub-sampling quantities identified in Table 6 and Table 7. If these quantities are insufficient to achieve the target minimum reportable quantity listed in Table 2 for supernatant and Table 3 for solids, contact M Urie prior to authorizing the SAL to provide the sub-samples. (Note: the centrifuged solids are approximately 70% solids and the MRQ as based on dried solids.)
2. For any preparative work conducted in the SAL (e.g., IX dose reduction, CN distillation), the cognizant analysis lead are to provide matrix spikes and laboratory control samples to the SAL for processing. For the IC, ammonia, and tritium on the solids, the "wet" solids will be leached at a ratio of 1:10 with a final volume being 25 mL.
3. All analyses are to be conducted with strict adherence to "Conducting Analytical Work in Support of Regulatory Programs" Sections 4 and 5. See URL --- http://quality.pnl.gov/Guidance/qaplan/

Table 2 Liquid Fraction Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method$^{(c)}$
	mg/L	
Al	7.5E+01	ICP-AES (AA may be used for Na, K)
B	2.3E+00	
Ba	2.3E+00	
Ca	1.5E+02	
Ce	2.3E+00	
Cd	7.5E+00	
Cr	1.5E+01	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Li	2.3E+00	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Th	2.3E+00	
V	2.3E+00	
W	2.3E+00	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TOC	1.5E+03 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
Br	3.0E+02	
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03 (as P)	
SO_4	2.3E+03 (as S)	
	mCi/L (except as noted)	
Rb	1.0E+00 (mg/L)	ICP-MS (AA may be used for Cs determination)
^{127}I	1.5E+00 (mg/L)	
^{129}I	1.8E-05	
^{133}Cs	7.0E-04 (mg/L)	
^{135}Cs	1.5E+00	
^{137}Cs	1.5E+00	
^{237}Np	2.7E-02	
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	8.7E-03(mg/L)	
^{99}Tc	1.5E-03	
^{233}U	4.2E-04	
^{234}U	1.2E-04	
^{235}U	4.5E-08	
^{236}U	1.4E-06	
^{238}U	7.2E-09	
^{99}Tc(pertechnetate)	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{3}H	2.1E-02	Separations / Liquid Scintillation
^{14}C	7.2E-04	
^{79}Se	9.0E-05	
^{90}Sr	1.5E-01	
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}$Pu	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}$Cm	1.5E-02	
^{154}Eu	2.0E-03	

F01-026

Table 2 Liquid Fraction Analyses

Analyte	**Target Minimum Reportable Quantity**	**Recommended Analysis Method[c]**
^{155}Eu	9.0E-02	Extended Counting Time GEA
^{60}Co	2.1E-03	
^{126}Sn	6.0E-03	
^{137}Cs	9.0E+00	
^{231}Pa	7.89E-05	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha (TRU)	N/A	Summation[b] of: Pu-238, Pu-239+Pu-240 (or Pu-239, Pu-240 ICP/MS) and Am-241
Total and Free OH	7.5E+04 mg/L	Titration
CN	3.0E+00 mg/L	CN Analysis
Ammonia	1.4E+02 mg/L	ISE or IC
Organic Analytes	**mg/L**	
Oxalate	1.5E+03	Ion Chromatograph
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
EDTA[a]	1.5E+03	Derivatization/ GC-MS
HEDTA[a]	1.5E+03	
D2EHPA[a]	1.5E+03	
NTA[a]	1.5E+03	
IDA[a]	1.5E+03	
Succinic Acid[a]	1.5E+03	
ED3A[a]	1.5E+03	
	Expected Range	
Density	0.95 –1.5 (gm/mL)	Gravimetric
Dissolved solids	1 to 50 (gm solids/gm supernate)	Gravimetric

Footnote:

[a] MRQs are target values, measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters.

[b] Guidance for reporting summation of isotopics and reporting isotopic values derived by different methods will be provided later after an agreement is obtained with DOE.

[c] If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 3. HLW Solids Analyses

Analyte	Target Minimum Reportable Quantity	Recommended Analysis Method[c]
	mg/Kg[a]	
Ag	9.0E+02	ICP-AES (AA may be used for Na & K)
Al	3.3E+02	
B	3.0E+00	
Ba	6.0E+02	
Be	3.0E+00	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
K	2.0E+02	
La	6.0E+01	
Li	3.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
U	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01 (as C)	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01 (as C)	
Hg	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
Br	4.5E+02	
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	
CN	3.0E+00	CN analysis
	mg/Kg	ICP-MS
As	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Mo	3.0E+01	
Pd	3.0E+02	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Rh	3.0E+02	
Ru	3.0E+02	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	

Table 3. Continued, HLW Solids Analyses		
Analyte	Target Minimum Reportable Quantity	Recommended Analytical Methods(c)
	mg/Kg	ICP-MS
W	6.0E+00	
Y	6.0E+00	
^{99}Tc	6.0E+00	
^{127}I	1.5E+00	
^{133}Cs	7.0E-04	
^{233}U	6.0E+01	
^{235}U	6.0E+00	
^{237}Np	1.8E+00	
	mCi/Kg	
^{129}I	1.8E-05	
^{135}Cs	5.3E-03	
^{137}Cs	9.0E-02	
^{151}Sm	TBD	
^{234}U	3.7E-03	
^{236}U	3.8E-04	
^{238}U	2.0E-06	
^{3}H	1.5E-02	Separations / Liquid Scintillation
^{14}C	1.8E-03	
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}$Pu	6.0E+00	
^{242}Pu	3.36E-01	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}$Cm	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
^{125}Sb	6.0E+00	
^{126}Sn	6.0E-02	
^{134}Cs	9.0E-01	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha (TRU)	N/A	Summation(b) of: Pu-238, Pu-239, Pu-240, and Am-241
^{241}Pu	1.2E+00 mCi/Kg	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetric
Density	0.9 to 2.0 gm/ml	Gravimetric
Wt% Undissolved Solids	10 to 50 wt%	Calculation
Wt% Soluble Solids	1 to 50 wt%	Calculation

Footnote:

(a) MRQs are based on dried solids weighs.

(b) Guidance for reporting summation of isotopics that are derived by different methods will be provided later after an agreement is obtained with DOE

(c) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 4. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Recommended Analytical Technique(f)	QC Flagging Criteria		
		LCS %Recovery(a)	Spike %Recovery(b)	Triplicate RSD(c)
Al, B, Ba, Ca, Cd, Ce, Cr, Fe, K, La, Li, Mg, Ni, P, Pb, Th, U, V, W	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
U	Kin. Phosphorescence	80 - 120%	75 - 125%	<15%
Rb, ^{127}I, ^{129}I, ^{133}Cs, Cs^{135}, Cs^{137}, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Pu / ^{241}Am, ^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, Br^-, F^-, NO_2^-, NO_3^-, PO_4^{-3}, SO_4^{-2}	IC	80 - 120%	75 - 125%	<15%
CN^-	CN Analysis	80 - 120%	75 - 125%	<15%
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH- (total and Free)	Potentiometric titration /precipitation	80 - 120%	N/A	<15%
TIC	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(e)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co(f), ^{126}Sn(j), ^{231}Pa	Extended GEA	NP	N/A(g)	<15%
^{79}Se	Liq. scintillation	NP	N/A(e)	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A(e)	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A(g)	<15%
^{154}Eu(f)	GEA	NP	N/A(g)	<15%
^{155}Eu(f)	GEA	NP	N/A(g)	<15%
^{238}Pu, $^{239/240}$Pu, ^{241}Am, ^{242}Cm, $^{243/244}$Cm	Separation/AEA	NP	N/A(e)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Sum of Alpha(h) [TRU]	Calculation	N/A	N/A	N/A
Density	Gravimetric	N/A	N/A	<20%
Wt% dissolved solids	Gravimetric	N/A	N/A	<20%
EDTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
HEDTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Oxalate	IC	80 - 120%	75 - 125%	<15%
Citrate	IC	80 - 120%	75 - 125%	<15%
Formate	IC	80 - 120%	75 - 125%	<15%
Gluconate	IC	80 - 120%	75 - 125%	<15%
Glycolate	IC	80 - 120%	75 - 125%	<15%
D2EPHA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
NTA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
IDA(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
Succinic Acid(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%
ED3A(k)	Derivatization/GC-MS	80 - 120%	75 - 125%	<15%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr, use Sr in the standard for determining the total mass-90 concentration.

(e) 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.

(f) 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.

(g) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(h) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting 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 that 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 MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination. If any of the isotopes are below the MRQ, then the method of summation will be agreeed by BNI prior to reporting values.

(i) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(j) Calibrate with Sn-117.

(k) Measurement of chelators and organic phosphates are best effort only, since there is insufficient method data available to set QC parameters, QC acceptance criteria are target values.

(l) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceding.

Table 5. Quality Control Parameters for Solids Analysis

Solids Fraction	Recommended Analytical Technique(e)	QC Flagging Criteria		
		LCS % Recovery(a)	Spike % Recovery(b)	Triplicate RSD(c)
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Cs, Fe, K, La, Mg, Mn, Nd, Ni, P, Pb, Pd, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	90 - 110%	90 - 110%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, U, V, W, mass unit 90(d), ^{99}Tc, ^{127}I, ^{133}Cs, ^{233}U, ^{235}U, ^{237}Np, ^{129}I, ^{135}Cs, ^{137}Cs, ^{151}Sm, ^{234}U, ^{236}U, ^{238}U.	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, Br^-, F^-, NO_2^-, NO_3^-, PO_4^{-3}, SO_4^{-2}	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Hg	CVAA	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%
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A(g)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{60}Co(i)	Extended count GEA	NP	N/A(k)	<15%
^{90}Sr(j)	Isotopic specific separation/beta count	75 - 125%	N/A(g)	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{125}Sb(m)	Extended Count GEA	NP	N/A	<15%
^{126}Sn(n)	Extended Count GEA	NP	N/A	<15%
^{134}Cs(h)	Extended Count GEA	NP	N/A	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{152}Eu(l)	Extended Count GEA	NP	N/A(k)	<15%
^{154}Eu(l)	Extended Count GEA	NP	N/A(k)	<15%
^{155}Eu(l)	Extended Count GEA	NP	N/A(k)	<15%
^{241}Am	Extended Count GEA	NP	N/A(k)	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, $^{239/240}$Pu, ^{242}Pu	Separation/AEA	NP	N/A(g)	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A(g)	<15%
^{242}Cm	Separation/AEA	NP	N/A(g)	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A(g)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Bulk density	Gravimetric	N/A	N/A	<20%
Wt% solids	Gravimetric	N/A	N/A	<20%

Acronyms:

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 Spectroscopy
LSC	– Laboratory Control Standard
N/A	– Not applicable
NP	– Not performed
RSD	– Relative Standard Deviation
Wt%	– Weight percent

Footnotes:

(a) 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.

(b) 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.

(c) 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 /mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{90}Zr.

(e) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.

(f) Not used.

(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 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 WAPS justification.

(i) 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 ^{152}Eu, ^{154}Eu, ^{155}Eu.

(j) Combined analysis of ^{90}Sr and ^{90}Y.

(k) The measurement is a direct reading of the energy and the sample matrix does not affect the analysis; therefore, a matrix spike is not required.

(l) Combined analysis with ^{93m}Nb.

(m) Combined analysis with ^{125m}Te.

(n) Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

(o) If analytical method changes result in full attainment of desired QC and MRQs, then method substitution shall be documented in Test Plan or in final test report. Method changes that don't satisfy desired QC/MRQ targets shall be communicated to BNI R&T representative for approval before proceeding.

Table 5: SAL Supernatant "Nominal" Sample Quantities and In-cell QC						
Supernatant	Sample	Duplicate	Triplicate	MS	LCS/BS	Blank
IC	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
Tc-99	0.3 ml	0.3 ml	0.3 ml	0.3 ml (1)	(1)	(1)
TOC/TIC PerSul	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
TOC/TIC Furnace	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml	0.5 ml + 0.5 ml (1)	(1)	(1)
C-14	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
Ammonia	1 ml	1 ml	1 ml	1 ml (1)	(1)	(1)
Mercury	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
CN	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
H3	0.5 ml	0.5 ml	0.5 ml	0.5 ml (1)	(1)	(1)
OH	1 ml	1 ml	1 ml	1 ml (1)	(1)	N/A
Chelators	5 ml	5 ml	5 ml	5 ml (1)(2)	(2)	(2)
Organic Acids	1 ml	1 ml	1 ml	1 ml (1)(2)	(2)	(2)
Organic Phosphates	5 ml	5 ml	5 ml	5 ml (1)(2)	(2)	(2)

(1) MS, LCS/BS, and Blank are part of the laboratory preparations/analysis, not the SAL preps; however, sub-sample need to be provided for preparing the MS in the laboratory. If the sample is diluted in the SAL, a SAL diluent blank should be provided with the samples.

(2) SAL preparation activities only involve IX processing for dose reduction per test plan provided by Campbell. The MS and LCS are to be provided by Campbell for Chelators and Organic Phosphates and Mong Organic Acids. Blanks are to be processed through the IX process. The cognizant analysis leads are to define the blank matrix (e.g., water, dilute hydroxide, etc.)

Table 7 : SAL Wet Centrifuged Solids "Nominal" Sample Quantities and In-cell QC						
Solids	Sample	Duplicate	Triplicate	MS	LCS/BS	Blank
Water Leach for IC, H3 and ammonia	2.5 g (per 25 mL)	2.5 g (per 25 mL)	2.5 g (per 25 mL)	2.5 g (per 25 mL) – IC, NH$_3$, H-3 spiked	BS (in 25 mL) for IC, NH$_3$, H-3	Required
TOC/TIC PerSul	0.1 g	0.1 g	0.1 g	0.1 g (1)	(1)	(1)
TOC/TIC Furnace	0.1 g + 0.1 g	0.1 g + 0.1 g	0.1 g + 0.1 g	0.1 g + 0.1 g (1)	(1)	(1)
C-14	0.1 g	0.1 g	0.1 g	0.1 g (1)	(1)	(1)
Mercury (2)	0.1 g	0.1 g	0.1 g	0.1 g (1)	(1)	(1)
CN (2)	0.1 g	0.1 g	0.1 g	0.1 g (1)	(1)	(1)

(1) MS, LCS/BS, and Blank are part of the laboratory preparations/analysis, not the SAL preps; however, sub-sample need to be provided for preparing the MS in the laboratory. If any of the analyses require preparation in the SAL (e.g., Hg digests, CN distillations, the MS, LCS and blank are required to be performed with the sample preparations.) MS and LCS/BS obtained from cognizant analysis leads – Steele IC, Soderquist NH$_3$, Swoboda H-3, and if needed Thomas Hg and CN.

(2) If Hg or CN preparation are performed in the SAL, the nominal samples sizes should be increase to 0.2 g for Hg and 1 g for CN.

F2

ASR 6193.01

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature Mike Ulric
Print Name Mike Ulric
Phone 376 9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W57930
Date Required: 10/1/01

Matrix Type Information

- **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ✓ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ASR 6193-42365
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) *Sections 4 and 5 apply*
- **Additional QA Requirements?** No ___
  or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ✓ Yes ___
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ___
  or Reference Doc # ___
  or Previous ASR # 6193.CC
  or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ Yes ___

Additional or Special Instructions *The requirements of NQARD apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To Mike Ulric **Phone** 376-9454
___ **Phone** ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 8/13/01
Delivered By (optional) HLCF
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [2.01]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: SAL
ASR Number: 6193.01
RPL Numbers: 01-02273

ASR Template for BNI NQARD Tasks.doc

AKNP, FVH, PRS, JJW, JPB, OTF

F02-002

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

RPG/CMC Work Accepted By: MWUrie **Signature/Date:** 9-24-05

Page 2 of 2

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only				Lab Staff Use Only	
RPL ID #	**Client Sample ID**	**Sample Description (& Matrix, If Varies)**	**Analysis Requested (1)**	**Test**	**Library**
01-02273	AZ101-129-Residual Solids)		ALC-129, Mod 2, ICP-MS;	Digest-129	SAL
	(from 129 MOD 1)		ICP-AES	ICP-211-CMC	labAnaly.
				ICP-MS	AdvInorg.

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ____]

ASR # 6193.01

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F02-004

F3

ASR 6193.02 Through ASR 6193.06

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature MW Urie 6-12-02
Print Name MWUrie
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W57930
Date Required: 6-19-02

Matrix Type Information

◆ **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
◆ **Solids:** ___ Soil [✓ Sludge] ___ Sediment
___ Glass ___ Filter ___ Metal
___ Smear ___ Organic ___ Other
◆ **Other:** ___ Solid/Liquid Mixture, Slurry
___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

◆ **Disposition of Virgin Samples:**
Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
If archiving, provide:
Archiving Reference Doc # ___
◆ **Disposition of Treated Samples:**
Dispose ✓ Return ___

QA/Special Requirements

◆ **QA Plan:**
SBMS ___
HASQARD (CAWSRP) *Sections 4 and 5 apply*
◆ **Additional QA Requirements?** No ✓
or Reference Doc # ___
◆ **Field COC?** No ✓ Yes ___
◆ **Lab COC Required?** No ✓ Yes ___
◆ **Hold Time:** None ✓
or RCRA ___ CERCLA ___
Other, Specify ___
& Date Sampled ___
Time Sampled ___
◆ **Special Storage Requirements:**
None ✓ Refrigerate (4°C) ___
or Other, specify ___
◆ **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

◆ **Sample Information Check List Attached?** Yes ___
or Reference Doc # ___
or Previous ASR # 6193.00
or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ **Yes** ___

Additional or Special Instructions *The requirements of the WTPSP QA Manual apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To MW Urie **Phone** 376-9454
___ **Phone** ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 8-13-01
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: SAL
ASR Number: 6193.02
RPL Numbers: (01-01845)

RPG/CMC Work Accepted By: KN Pool **Signature/Date:** 6/13/02

page 1 of 1

KNP

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only				Lab Staff Use Only	
RPL ID #	**Client Sample ID**	**Sample Description** (& Matrix, If Varies)	**Analysis Requested (1)**	**Test**	**Library**
01-01845	AZ101 CS AR	Centrifuged Solids AZ101	Pu-241	Pu-241	Radchem

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193.02

Page 1 of 1

ASR Template for BNI NQARD Tasks Rev. 1.doc

VBD

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature MW Urie 7/29/02
Print Name MW Urie
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W57930
Date Required: 8/18/02

Matrix Type Information

◆ **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
◆ **Solids:** ___ Soil ___ Sludge ___ Sediment
___ Glass ___ Filter ___ Metal
___ Smear ___ Organic ___ Other
◆ **Other:** ___ Solid/Liquid Mixture, Slurry
___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

◆ **Disposition of Virgin Samples:**
Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
If archiving, provide:
Archiving Reference Doc # ___
◆ **Disposition of Treated Samples:**
Dispose ✓ Return ___

QA/Special Requirements

◆ **QA Plan:**
SBMS ___
HASQARD (CAWSRP) *Sections 4 and 5 apply*
◆ **Additional QA Requirements?** No ✓
or Reference Doc # ___
◆ **Field COC?** No ✓ Yes ___
◆ **Lab COC Required?** No ✓ Yes ___
◆ **Hold Time:** None ___
or RCRA ___ CERCLA ___
Other, Specify ___
& Date Sampled ___
Time Sampled ___
◆ **Special Storage Requirements:**
None ✓ Refrigerate (4°C) ___
or Other, specify ___
◆ **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

◆ **Sample Information Check List Attached?** Yes ___
or Reference Doc # ___
or Previous ASR # 6193.00
or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ Yes ___

Additional or Special Instructions *The requirements of the WTPSP QA Manual apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To MW Urie **Phone** 376-9454
___ **Phone** ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 7-19-02 - Reg. Date
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: C. Soderquist
ASR Number: 6193.03
RPL Numbers: (01-01844-R-TC)

RPG/CMC Work Accepted By: KNPool **Signature/Date:** 8/29/02

KNP, LPD, CZS, LRG, MWU

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only				Lab Staff Use Only	
RPL ID #	**Client Sample ID**	**Sample Description** (& Matrix, If Varies)	**Analysis Requested (1)**	**Test**	**Library**
01-01844-R-Tc	AZ101 SUP AR		TcO4	TC99	Radchem
				SUBSAMPLE	SAL
			"		

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193.03

Page 1 of 2

ASR FY2000 - RPG1.doc

F03-005

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature
Print Name MW Urie
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W57930
Date Required: 9-15-02

Matrix Type Information

- **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ___ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ___
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) *Sections 4 and 5 apply*
- **Additional QA Requirements?** No ___
  or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ✓ Yes ___
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ___
  or Reference Doc # ___
  or Previous ASR # 6193.00
  or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ Yes ___

Additional or Special Instructions *The requirements of the WTPSP QA Manual apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To MW Urie **Phone** 376-9454
___ **Phone** ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 8-15-02
Delivered By (optional) Inlab
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: C. Soderquist
ASR Number: 6193.04
RPL Numbers: 01-01844

RPG/CMC Work Accepted By: KN Pool **Signature/Date:** 9/9/02

KNP

F03-006

F03-007

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
01-01844	AZ101 SUPAR		Sr90 in triplicate	SR90	Radchem
			reanalysis		
			"		

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193.04 Page 1 of 1

✓KP

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature
Print Name MW Urie
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W57930
Date Required: 9-15-02

Matrix Type Information

◆ **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
◆ **Solids:** ___ Soil ___ Sludge ___ Sediment
___ Glass ___ Filter ___ Metal
___ Smear ___ Organic ___ Other
◆ **Other:** ___ Solid/Liquid Mixture, Slurry
___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

◆ **Disposition of Virgin Samples:**
Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
If archiving, provide:
Archiving Reference Doc # ___
◆ **Disposition of Treated Samples:**
Dispose ✓ Return ___

QA/Special Requirements

◆ **QA Plan:**
SBMS ___
HASQARD (CAWSRP) *Sections 4 and 5 apply*
◆ **Additional QA Requirements?** No ___
or Reference Doc # ___
◆ **Field COC?** No ✓ Yes ___
◆ **Lab COC Required?** No ✓ Yes ___
◆ **Hold Time:** None ✓
or RCRA ___ CERCLA ___
Other, Specify ___
& Date Sampled ___
Time Sampled ___
◆ **Special Storage Requirements:**
None ✓ Refrigerate (4°C) ___
or Other, specify ___
◆ **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

◆ **Sample Information Check List Attached?** Yes ___
or Reference Doc # ___
or Previous ASR # 6193.00
or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ **Yes** ___

Additional or Special Instructions *The requirements of the WTPSP QA Manual apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To MW Urie **Phone** 376-9454
___ **Phone** ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 8-29-02
Delivered By (optional) SAL
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: C. Soderquist
ASR Number: 6193.05
RPL Numbers: 01-01844

RPG/CMC Work Accepted By: ~~MW Urie~~ KP 9/9/02 KN Pool **Signature/Date:** 9/9/02

ASR Template for BNI NQARD Tasks Rev. 1.doc

KNP

F03-008

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
01-01844	AZ101 SupAR		Sr90 re-analysis in triplicate	SR90	Radchem

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____ ; On Attachment ___]

ASR # 6193.05

Page 1 of 1

F03-009

✓P

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature
Print Name MW Urie
Phone 376-9454 MSIN P7-22

PNNL Project #: 42365
Charge Code: W65128
Date Required: 5/21/03

Matrix Type Information

◆ **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
◆ **Solids:** ___ Soil ___ Sludge ___ Sediment
___ Glass ___ Filter ___ Metal
___ Smear ___ Organic ___ Other
◆ **Other:** ___ Solid/Liquid Mixture, Slurry
___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

◆ **Disposition of Virgin Samples:**
Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
If archiving, provide:
Archiving Reference Doc # ___
◆ **Disposition of Treated Samples:**
Dispose ✓ Return ___

QA/Special Requirements

◆ **QA Plan:**
SBMS ___
HASQARD (CAWSRP) *Sections 4 and 5 apply*
◆ **Additional QA Requirements?** No ✓
or Reference Doc # ___
◆ **Field COC?** No ✓ Yes ___
◆ **Lab COC Required?** No ___ Yes ✓
◆ **Hold Time:** None ✓
or RCRA ___ CERCLA ___
Other, Specify ___
& Date Sampled ___
Time Sampled ___
◆ **Special Storage Requirements:**
None ✓ Refrigerate (4°C) ___
or Other, specify ___
◆ **Data Quality Review Required?** No ✓ Yes ___

Waste Designation Information

◆ **Sample Information Check List Attached?** Yes ___
or Reference Doc # ___
or Previous ASR # 6193
or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ Yes ___

Additional or Special Instructions *The requirements of the WTPSP QA Manual apply to this work. Administrative requirements prior to and following analyses are the responsibility of the Task Manager, Project Manager, and Quality Engineer. Task-specific Quality Control criteria are attached.*
Reference Document (i.e., TP-RPP-WTP-XXX):

Send Report To MW Urie **Phone** ___
Phone ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 5-16-03
Delivered By (optional) F. Hoopes
Time Delivered (optional) ___
Group ID (optional) _RPP-WTP / Task No: [ ]
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: T. Farmer
ASR Number: 6193.06
RPL Numbers: 01-01844-R2 + 01-01845-R2

RPG/CMC Work Accepted By: KN Pool **Signature/Date:** 5/16/03

KNP, OTF

ASR Template for BNI NQARD Tasks Rev. 1.doc

F03-010

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Lab Staff Use Only Library
01-01844-R2	AZ101 SUPAR-R2	①	1-129 (ICP-MS), ①	ICP-MS	Adv Inorg
01-01845-R2	AZ101 CS AR-R2	②	Wt% Solids, TDS	TDS	SAL
				SUBSAMPLE	SAL
			②	ICP-MS	Adv Inorg
				Wt% Solids	SAL
	Perform in triplicate			SUBSAMPLE	SAL

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ___]

ASR # 6193.06

Page 1 of 1

F03-011

Special Test Instructions: ASR 6193 SAL Addendum 2
As Received Analyses for AZ-101 Supernatant Sample 01-1844 and Centrifuged Solids Sample 01-1845

Objective of this Addendum: SAL is to phase separate last two bottles of AZ-101 bottles identified for characterization, and perform only those preparative and distribution activities detailed below.

AZ-101 SUP AR (Supernatant Sample) 01-1844 – LIMS Tests

Sub-Sample	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC	RAD	-- Use CMC K88421
(Tc-99 w/o oxidation – pertechnetate)		
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
NH3-ISE	LAB	-- Use W57932
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408
H3-418/474-CMC	RAD	-- Use CMC K88422
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use W57933
Acids/Organic	ORG	-- Use W57933
Phosphates/Organic	ORG	-- Use W57933

AZ-101 CS AR (Centrifuged Solids Sample) 01-1845 – LIMS Tests

Leach/Water-103	SAL	-- Use W57930
IC-212-CMC	LAB	-- Use CMC K88406
NH3-ISE	LAB	-- Use W57932
H3-418/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use W57930
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
C14-381/474-CMC	RAD	-- Use CMC K88410
CN/Total	LAB	-- Use W57932
Hg-131/201-CMC	LAB	-- Use CMC K88408

QC Information

The analyses are to be conducted per PNNL's web-based Quality Assurance Planning Subject Area, "Conducting Analytical Work in Support of Regulatory Programs". The acceptance QC criteria for the supernatant and wet centrifuged solids are attached in Table 4 and Table 5, respectively.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in **TRIPLATE**.

Preparative or sample analysis QC is to include, preparation blank, sample, duplicate, triplicate, matrix spike, matrix spike duplicate where appropriate, and a LCS. If possible, the matrix spikes and LCS are to include all the analytes of interest to be reported for the specific analysis. The matrix spikes and LCS are to be provided to the SAL by the appropriate cognizant analysis lead.

Phase Separation

The bottles labeled AZ-101-CHEM-3 through AZ-101-CHEM-4 are to be phase separated to obtain an AZ-101 supernatant composite sample and an AZ-101 centrifuged solids composite sample. Bottles AZ-101-CHEM-3 through AZ-101-CHEM-4 are to be centrifuged (in secondary containment) at approximately 1000 time the force of gravity for 1 hour. Transfer all the centrifuged supernatant to tared glass 250 to 500 mL bottle labeled **AZ-101 SUP2 AR (01-1844)** and transfer the wet centrifuged solids to a tared glass 50 to 125 mL bottle labeled **AZ-101 CS2 AR (01-1845)**.

The weights of the supernatant and centrifuged solids samples are to be recorded and sent to the Project file and to M. W. Urie.

Date __________ SAL operator ______________ Balance ID__________________

AZ-101 SUP2 AR gross (g) ________ AZ-101 CS2 AR gross (g) _________

AZ-101-SUP2 AR tare (g) ________ AZ-101 CS2 AR tare (g) _________

Net (g) ________ Net (g) _________

Note: The phase separation process may be conducted by using one of the original bottles as the final container for the solids if the tare weight of the bottle is known. For example, centrifuge bottle AZ-101-CHEM-3, remove supernatant and add slurry from AZ-101-CHEM-4 and centrifuge. If this approach is used, the original bottle must be relabeled **AZ-101 CS2 AR (01-1845)**.

SAL Preparation/Analysis

During all sub-sampling activities, minimize the time that the AZ-101 SUP~~1~~2 AR and AZ-101 CS~~1~~2 AR containers are open to the hot cell atmosphere and ensure that they are tightly closed when not in use. The supernatant is to be thoroughly mixed and the wet centrifuged solids are to be thoroughly homogenized (mechanically or manually) prior to any sub-sampling activities. See QC Information Section for QC sample requirements. Table 5 and Table 6 provide the "nominal" quantities to be processed in the SAL for each preparation method and the "nominal" quantities to be distributed to the laboratory for those sub-samples not being processed in the SAL. These tables also identify when the MS, LCS/BS, and Blanks are required in the SAL.

Note: Table 6 and Table 7 are "nominal" sample quantities typically used for the specified analysis. However, prior to sub-sampling the actual target sample quantities (and any special sub-sampling or preservation instructions) need to be provided by the cognizant analysis lead for each analysis.

Note: Following any daily sub-sampling activities, the sample bottles AZ-101 SUP2 AR and AZ-101 CS2 AR are to be weighed and the weights recorded and sent to the Project file and to M. W. Urie.

Note: The mass and volume of the supernatant sub-samples processed under this Addendum are to be recorded on the distribution benchsheets or preparation benchsheets in both units of mass (g) and volume (mL). This requirement applies only when the SAL mass or volume is to be used in calculation of the analyte concentrations (i.e., some sub-samples may not need to be accurately measured since they will be further sub-sampled at the analysis work station).

1. Perform water leach of solids (AZ-101 CS2 AR) using PNL-ALO-103. Use 2.5 g of sample per 25 mL of water.

2. Sub-sample all supernatant (AZ-101 SUP2 AR), solids (AZ-101 CS2 AR), and solids leachate inorganic and radiochemical sub-samples that are to be distributed directly to the laboratories.

3. Perform any additional in-cell inorganic or radiochemistry preparations identified by the cognizant analysis leads (e.g., CN distillations may need to be performed in the SAL) on the supernatant and/or solids and distribute.

4. Perform the IX dose reduction for all the organic analyses and distribute the sub-samples.

Laboratory Analysis

1. The cognizant analysis lead for each of the analytical methods is to review the sub-sampling quantities identified in Table 6 and Table 7. If these quantities are insufficient to achieve the target minimum reportable quantity listed in Table 2 for supernatant and Table 3 for solids, contact M Urie prior to authorizing the SAL to provide the sub-samples. (Note: the centrifuged solids are approximately 70% solids and the MRQ as based on dried solids.)

2. For any preparative work conducted in the SAL (e.g., IX dose reduction, CN distillation), the cognizant analysis lead are to provide matrix spikes and laboratory control samples to the SAL for processing. For the IC, ammonia, and tritium on the solids, the "wet" solids will be leached at a ratio of 1:10 with a final volume being 25 mL.

3. All analyses are to be conducted with strict adherence to "Conducting Analytical Work in Support of Regulatory Programs" Sections 4 and 5. See URL --- http://quality.pnl.gov/Guidance/qaplan/

F4

ICP-AES RESULTS
ASR 6193 AND 6193.01

PNL-ALO-128 - Acid Digestion

Project / WP#:	42365 / W57395
ASR#:	6193
Client:	M. Urie
Total Samples:	1 (liquid)

	First	Last
RPL#:	01-01844	–
Client ID:	"AZ101 Sup AR"	–
Sample Preparation: PNL-ALO-128 (~1mL/25mL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 09-21-2001 (A0719)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

MW Urie 4/1/02
Reviewed by

4/1/02
Concur

One liquid sample from Analytical Service Request (ASR) 6193 was prepared in triplicate by acid digestion per PNL-ALO-128 in the Shielded Analytical Laboratory (SAL). The samples were prepared using a nominal 1 mL of sample and diluting to a final volume of about 25 mL. The final volumes were calculated using the mass and the density of the resulting digestate.

Analytes of interest (AOI) were specified in Table 2 attached to the ASR and included Al, B, Ba, Ca, Ce, Cd, Cr, Fe, K, La, Li, Mg, Na, Ni, P, Pb, Th, V, W, and U. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than identified as analytes of interest are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (2 pages). All results are reported as µg/mL. The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan.

Process Blank:

Except for boron, concentrations of all AOIs measured in the process blanks were within the acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample. Boron was observed in the process blank at ~60 µg/mL, approximately the same as observed in the sample. Since silicon was also observed in the process blank, the boron is likely from leaking from the glass beaker used in the digestion process

Relative Standard Deviation (RSD):

All AOIs measured above the EQL were within the acceptance criteria of <15% (<3.5% for Na).

Blank Spike (laboratory control sample):

The blank spike for the acid digestion was prepared using 3 mL each of multi-element standard solutions AZ101 Spike Part 1 and AZ101 Spike Part 2. This spike includes all of the analytes of interest defined by the ASR. Except for Na with a recovery of 121%, all AOIs measured above the EQL demonstrated spike recoveries within the acceptance criteria of 80% to 120%. Although tungsten was present in the spiking solution, the final tungsten concentration was below the EQL following the preparative dilutions, and recovery is not reported.

Matrix Spiked Sample:

The matrix spike was prepared by adding 1 mL each of multi-element standard solutions AZ101 Spike Part 1 and AZ101 Spike Part 2 to approximately 1 mL of sample. For all AOIs measured above the EQL (except boron), the spike recoveries were within the acceptance criteria of 80% to 120%. Boron was slightly over recovered at 136%. The over recovery was likely due to the variability of the boron leached from the glassware used for the digestion process. The recoveries for Al, Cr, Na, and P were not calculated since the spike concentrations were less than 20% of the sample concentrations. Spiking at concentrations less than 20% of the sample concentration typically produces unreliable results, and serial dilution is used to assess potential matrix interferences. The final concentrations of Ce, Th, and W were below the EQL following the preparative dilutions, and recovery is not reported.

Post-Spiked Samples (Spike A Elements):

Due to an oversight in setting up the analysis run, no post-spiking of the sample was performed.

Post-Spiked Samples (Spike B Elements):

Due to an oversight in setting up the analysis run, no post-spiking of the sample was performed.

Five fold serial dilution:

All AOIs above the EQL, following the five-fold serial dilution, were in agreement within the acceptance criteria of ±10%.

Other QC:

All required instrument-related QC tests for the analytes of interest passed within tolerance limits.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICPAES measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis.... ICPASE Report

	Run Date=	9/21/2001	9/21/2001	9/21/2001	9/21/2001	9/21/2001	9/21/2001	9/21/2001
	Multiplier=	26.3	26.6	133.0	25.6	127.9	25.1	125.5
	RPL/LAB #=	01-01844-PB	01-01844	01-01844 @5	01-01844-DUP	01-01844-DUP @5	01-01844-TRIP	01-01844-TRIP @5
Det. Limit	*Client ID=*	*process blank*	*AZ-101 Sup AR*		*AZ-101 Sup AR-Dup*		*AZ-101 Sup AR-Trip*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	[7.1]	6,090		6,050		6,020	
0.050	B	60.6	65.9		84.7		81.6	
0.010	Ba	--	--		--		[0.25]	
0.250	Ca	--	[9.3]		--		--	
0.015	Cd	[0.40]	[0.85]		[0.77]		[0.70]	
0.200	Ce	--	--		--		--	
0.020	Cr	--	689		685		683	
0.025	Fe	[0.78]	[2.1]		[1.3]		[1.1]	
2.000	K	--	4,500		4,460		4,420	
0.050	La	--	--		--		--	
0.030	Li	--	[0.88]		[0.90]		[0.90]	
0.100	Mg	--	--		--		--	
0.150	Na	70.6	over range	111,000	over range	112,000	over range	110,000
0.030	Ni	[2.7]	[1.2]		[0.98]		[0.83]	
0.100	P	--	506		505		500	
0.100	Pb	--	[3.4]		[3.3]		[3.3]	
1.000	Th	--	--		--		--	
2.000	U	--	--		--		--	
0.050	V	--	[1.5]		[1.5]		[1.5]	
2.000	W	--	[58]		[58]		[58]	
Other Analytes								
0.025	Ag	--	--		--		--	
0.250	As	--	[9.7]		[10]		[10]	
0.010	Be	--	--		--		--	
0.100	Bi	--	--		--		--	
0.050	Co	--	--		--		--	
0.025	Cu	--	--		--		--	
0.050	Dy	--	--		--		--	
0.100	Eu	--	--		--		--	
0.050	Mn	--	--		--		--	
0.050	Mo	--	89.2		88.7		88.4	
0.100	Nd	--	--		--		--	
0.750	Pd	--	--		[20]		[20]	
0.300	Rh	--	--		--		--	
1.100	Ru	--	--		--		--	
0.500	Sb	--	--		--		--	
0.250	Se	--	--		--		--	
0.500	Si	138	194		221		204	
1.500	Sn	--	[46]		[47]		[47]	
0.015	Sr	--	--		--		--	
1.500	Te	--	--		--		--	
0.025	Ti	--	--		--		--	
0.500	Tl	--	--		--		--	
0.050	Y	--	--		--		--	
0.050	Zn	--	--		--		--	
0.050	Zr	--	--		--		--	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 9/21/2001

Criteria>	<15% (a)	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-01844 & Dup & Trip	01-01844 LCS/BS	01-01844 MS	Post Spike A (none)	Post Spike B (none)	01-01844 @1/@5 Serial Dil	01-01844-D @1/@5 Serial Dil	01-01844-T @1/@5 Serial Dil
Analytes	RSD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff
Al	0.6	97	nr			2.2	3.3	2.8
B	13	109	136			6.3	4.7	4.3
Ba		98	92					
Ca		100	95					
Cd		97	98					
Ce		95	nc					
Cr	0.5	102	nr			4.7	5.7	5.2
Fe		107	94					
K	0.9	97	80			3.9	4.2	4.3
La		96	96					
Li		102	94					
Mg		103	102					
Na	0.6	121	nr			4.6 (b)	4.9 (b)	4.2 (b)
Ni		101	97					
P	0.6	97	nr			0.4	1.3	1.2
Pb		102	100					
Th		99	nc					
U		96	90					
V		94	91					
W			nc					
Other Analytes								
Ag		102	101					
As								
Be		95	97					
Bi		98	nc					
Co								
Cu		106	66					
Dy								
Eu								
Mn		101	97					
Mo	0.5	98	91			4.8	5.7	5.2
Nd		96	95					
Pd								
Rh								
Ru								
Sb								
Se								
Si	6.6	122	132			4.1	-3.9	-6.1
Sn								
Sr		113	nc					
Te								
Ti		95	92					
Tl								
Y								
Zn		103	97					
Zr		104	101					

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration. nc = not calculated; result < EQL.

(a) = RPD <3.5% for Na (only); (b) data from 5x/25x dilutions.

PNL-ALO-115 - KOH-KNO_3/Ni Fusion

Project / WP#:	42365 / W57395
ASR#:	6193
Client:	M. Urie
Total Samples:	1 (solid)

	First	Last
RPL#:	01-01845-Ni	–
Client ID:	"AZ101 Centrifuged Solids"	–
Sample Preparation: PNL-ALO-115 (0.18-0.26g/100mL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 10-08-2001 (A0726)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

MW Urie 4/1/02
Reviewed by

B.M. Oliver 4/1/02
Concur

F04-009

One solid sample submitted under Analytical Service Request (ASR) 6193 was prepared in triplicate by KOH-KNO_3 fusion per PNL-ALO-115. The fusions were performed in the Shielded Analytical Laboratory, and used from ~0.18 to ~0.26 grams of sample diluted to a final volume of 100 mL.

Table 3 in the ASR identified the analytes of interest as being Ag, Al. B, Ba, Be, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Li, Mg, Mn, Na, Nd, Ni, P, Pb, Si, Sr, Ti, U, Zn, and Zr. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified as analytes of interest are reported for information only, since they have not been fully evaluated for QC performance.

The attached ICPAES Results (2 pages) presents the final results. The ICPAES measurement results are reported in µg/g of as-received wet centrifuged solids and have been corrected for all dilutions resulting from sample processing.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the fusion processing, two processing blanks, a laboratory control sample (SRM-2710), a blank spike, a duplicate, and a triplicate were prepared along with the sample.

Process Blank:

Concentrations of all analytes of interest measured in the process blanks were within the acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike:

The blank spike for the fusion preparation was prepared using two multi-element standard solutions (AZ101 Spike Part 1 and AZ101 Spike Part 2). This spike includes all of the analytes of interest defined by the ASR. For all included analytes measured above the EQL, the spike recoveries were within the acceptance criteria of 80% to 120%. Copper was included in the spike but not detected above the instrument detection limit.

Laboratory Control Sample (LCS):

The LCS for the fusion preparation was prepared from SRM-2710 (Montana Soil). For the AOI's present in the LCS detected above the EQL, the recoveries were within the acceptance criteria of 80% to 120%.

Relative Standard Deviation (RSD):

All AOI's measured above the EQL were within the acceptance criteria of <15% (<3.5% for Na) except for sodium which showed an RSD of 7.6%. For sodium, the result from the original sample appears to be significantly lower than for the duplicate or the triplicate, and this results in the higher than normal RSD. This effect was not noted, however, in other major analytes detected (e.g., Al and Fe) in the sample.

Matrix Spiked Sample:

No matrix-spiked sample was provide for analysis.

Post-Spiked Samples (Spike A Elements):

All post-spiked AOI's above the EQL were recovered within the acceptance criteria of 75% to 125%.

Post-Spiked Samples (Spike B Elements):

All post-spiked AOI's above the EQL were recovered within the acceptance criteria of 75% to 125%.

Serial dilution:

All AOI's above the EQL, following the five-fold serial dilution, were in agreement within the acceptance criteria of ±10%.

Other QC:

All required instrument-related QC tests for the analytes of interest passed within the acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) 3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis.... ICPAES Report

	Run Date=	10/8/2001	10/8/2001	10/8/2001	10/8/2001	10/8/2001
	Multiplier=	2214.2	2214.2	2720.8	1913.3	2151.8
	RPL/LAB #=	01-01845-B1-Ni	01-01845-B2-Ni	01-01845-Ni	01-01845-DUP-Ni	01-01845-TRIP-Ni
Det. Limit	*Client ID=*	*Process Blank 1*	*Process Blank 2*	*AZ101 CS AR*		
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	[230]	[210]	[330]
0.060	Al	[190]	[290]	134,000	127,000	130,000
0.050	B	--	--	--	--	--
0.010	Ba	[25]	--	452	462	437
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	[2,300]	[2,500]	[2,200]
0.015	Cd	--	--	4,470	4,400	4,270
0.200	Ce	--	--	--	[500]	[550]
0.020	Cr	--	--	551	591	688
0.025	Cu	--	--	--	--	--
0.025	Fe	[130]	[86]	62,900	63,500	58,400
2.000	K	na	na	na	na	na
0.050	La	--	--	1,600	1,590	1,550
0.030	Li	--	--	--	[69]	[70]
0.100	Mg	--	--	[530]	[580]	[470]
0.050	Mn	--	--	1,500	1,570	1,380
0.150	Na	[2,400]	[2,400]	65,400	73,500	75,600
0.100	Nd	--	--	[1,300]	[1,200]	[1,200]
0.030	Ni	na	na	na	na	na
0.100	P	--	--	[410]	[290]	[340]
0.100	Pb	--	--	[520]	[460]	[560]
0.500	Si	--	--	[3,700]	[4,200]	[3,500]
0.015	Sr	--	--	[330]	323	[310]
0.025	Ti	--	--	[75]	[69]	[60]
2.000	U	--	--	[5,600]	[4,900]	[5,400]
0.050	Zn	--	--	[140]	[110]	[130]
0.050	Zr	--	--	12,600	14,100	13,400
Other Analytes						
0.250	As	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
1.500	Sn	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.500	Tl	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	[100]	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

n/a = not applicable; KOH flux and Ni crucible used for preparing samples

QC Performance 10/8/01

Criteria>	<15%$^{(a)}$	80% - 120%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-01845-Ni & 01-01845-Ni & Dup & Trip	01-01845-Ni BS	01-01845-Ni LCS (2710)	MS (none)	01-01845-Ni + Post Spike A	01-01845-Ni + Post Spike B	01-01845-Ni @1/@5 Serial Dil
Analytes	RSD (%)	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Ag		**142**			100		
Al	2.9	93	96		105		1.9
B		**100**			105		
Ba	2.8	91	93		101		
Be		**91**			**101**		
Bi		**153**			99		
Ca		**122**	99		102		
Cd	2.3	**98**			103		1.9
Ce		**117**				**109**	
Cr	12	**99**			102		
Cu			88		104		
Fe	4.5	107	98		105		2.5
K	na	na	na		na		2.9
La	1.7	**97**				96	
Li		**100**			100		
Mg		107	106		109		
Mn	6.3	**120**	103		106		
Na	7.6	97	94		101		2.4
Nd		**102**				96	
Ni	na	na	na		na		3.3
P		**102**			101		
Pb		**108**	97		104		
Si		**99**	89		106		
Sr		**119**			101		
Ti		**95**	89		100		
U		**108**				**97**	
Zn		**128**	98		104		
Zr	5.7	**109**			105		1.8
Other Analytes							
As					100		
Co					109		
Dy						96	
Eu						103	
Mo		**99**			103		
Pd						**102**	
Rh						**97**	
Ru							
Sb					**99**		
Se					**100**		
Sn							
Te							
Th						**97**	
Tl					**98**		
V		**100**			**98**		
W							
Y					102		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration. nc = not calculated; result < EQL.

(a) = RPD <3.5% for Na (only)

na = not applicable; KOH flux and Ni crucible used for preparing samples.

ASR 6193 Final - ~A0726 M. Urie ASR-6193 ICP98 hi.XLS

PNL-ALO-129, Mod 1 and Mod 2
Acid Digestion

F04-014

Project / WP#: 42365 / W57395
ASR#: 6193 and 6193.01
Client: M. Urie
Total Samples: 2 (solids)

RPL#:	01-01845	01-02273
Client ID:	"AZ101 CS AR"	"AZ101-129 Residual Solids"
Sample Preparation: 01-01845 PNL-ALO-129 Mod. 1 and Mod. 2 01-02273 PNL-ALO-129 Mod 2		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File):
09-19-2001 (A0717)
09-25-2001 (A0722)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number:
WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

MW Urie 4/1/02
Reviewed by

B.M. Oliver 3/29/02
Concur

The AZ-101 wet centrifuged solids (AZ-101 CS AR) sample submitted under Analytical Service Requests (ASR) 6193 were prepared by acid digestion per PNL-ALO-129 (Mod 1 and Mod2). Also, the residual solids remaining following the digestion of AZ-101 CS AR per PNL-ALO-129 (Mod 1) were submitted for analysis under ASR 6193.01 and acid digested per PNL-ALO-129 (Mod 2). The digestions were performed in the Shielded Analytical Laboratory.

Solids sample AZ-101 CS AR was digested in triplicate per PNL-ALO-129 Mod 1 using approximately 0.5 g of sample diluted to a final volume of about 25 mL. Following digestion, each of the triplicate samples was further diluted by a factor of 6x prior to the ICPAES analysis. The PNL-ALO-129 Mod 1 acid digestion did not completely dissolve all the solids material; thus, the residual solids remaining from the digestion were subjected to further acid digestion using PNL-ALO-129 Mod. 2. At the same time the residual solids were being processed per PNL-ALO-129 Mod 2, triplicate samples of the original AZ-101 CS AR sample were also prepared for ICPAES analysis per PNL-ALO-129 Mod 2 in an effort to obtain higher total analyte recoveries. Approximately 0.35 g of AZ-101 CS AR was used for processing per PNL-ALO-129 Mod 2 and diluted to a final volume of 100 mL. The residual solids processed per PNL-ALO-129 Mod 2 were also diluted to a final volume of 100 mL; however, no attempt was made to obtain weights of the residual solids.

Table 3 in ASR 6193 identified the analytes of interest (AOI'S) as being Ag, Al, B, Ba, Be, Bi, Ca, Cd, Cr, Cu, Fe, K, La, Li, Mg, Mn, Na, Ni, Nd, P, Pb, Si, Sr, Ti, U, Zr, and Zn. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified as analytes of interest are reported for information only, since they have not been fully evaluated for QC performance.

The attached ICPAES Results (4 pages) presents the final results. For the AZ-101 CS AR sample submitted under ASR 6193 and prepared for analysis by both Mod 1 and Mod 2 of PNL0ALO-129, the results are reported in µg/g. For the PNL-ALO-129 Mod 1 residual solids submitted under ASR-6193.01, the results are reported as total µg, as no sample weights were obtained. All results have been corrected for dilutions resulting from sample processing.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the acid digestion processing, a processing blank, laboratory control sample (blank spike for samples processed per PNL-ALO-129 Mod 1 and blank spike and solids LCS for samples processed per PNL-ALO-129 Mod 2), matrix spike, duplicate, and triplicate were prepared along with the sample.

Analysis Run September 19, 2001 - PNL-ALO-129 Mod 1

Process Blank:

Concentrations of all AOI's measured in process blank were within acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike (BS):

The blank spike was prepared using two multi-element standard solutions (TW Spike Part 1 and TW Spike Part 2). This spike includes all of the analytes of interest defined by the ASR except for B, Be, and Li. For all included analytes except Si, the spike recoveries were within the acceptance criteria of 80% to 120%. The Si was slightly over-recovered at 141%.

Relative Standard Deviation (RSD):

All AOI'S measured above the EQL were within the tolerance limits of <15% (<3.5% for Na), except Na (at 8.3%) and Zr (at 21%). The results from the duplicate appear to be significantly different than the sample or triplicate, and this results in the higher than normal RSD.

Matrix Spiked Sample:

Except for Ag, all AOI's with spike concentrations greater than 20% of the sample concentration, and with measured results >EQL (following analysis dilution), met the matrix spike recovery criteria of 75% to 125%. Silver was recovered at only a 14% level. The analytes present in the spike that were at concentrations less than 20% of the concentration in the sample are labeled as "nr" and the analytes in the spike the were at a concentration <EQL (following dilution) are labeled as "nc". Post spiking or serial dilution are used to verify QC performance of these analytes.

Post-Spiked Samples (Spike A Elements):

All post-spiked AOI's above the EQL were recovered within the tolerance of 75% to 125%. All of those analytes present in the post spike that had failed the matrix spike because of low spike concentrations were recovered within tolerance in the post spike.

Post-Spiked Samples (Spike B Elements):

All post-spiked AOI's above the EQL were recovered within the tolerance limits of 75% to 125%.

Serial dilution:

All AOI'S above the EQL following the five-fold serial dilution were within the tolerance of ±10%.

Analysis Run September 25, 2001 – PNL-ALO-129 Mod 2

Process Blank:

Concentrations of all AOI's measured in process blank were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike (BS):

The blank spike was prepared using two multi-element standard solutions (AZ101 Spike Part 1 and AZ101 Spike Part 2). This spike includes all of the analytes of interest defined by the ASR. For all included analytes except Si, the spike recoveries were within the acceptance criteria of 80% to 120%. The Si was lost from the blank spike due to the addition of HF as part of the PNL-ALO-129 Mod 2 procedure.

Laboratory Control Sample (LCS):

A Laboratory Control Sample (SRM-2710 - Montana Soil) was processed with the samples prepared by PNL-ALO-129 Mod 2. For the AOI'S present in SRM-2710, the recoveries were within the acceptance criteria of 80% to 120%. As was noted in the blank spike, Si was not detected due to removal by the HF used in the digestion process.

Relative Standard Deviation (RSD):

All AOI'S measured above the EQL were within the tolerance limits of <15% (<3.5% for Na), except Na (at 8.3%) and Cr (at 45%). For Na and Cr, the results from the duplicate appear to be significantly different than the sample or triplicate, and this results in the higher than normal RSD.

Matrix Spiked Sample:

All AOI's with spike concentrations greater than 20% of the sample concentration, and with measured results >EQL (following analysis dilution), met the matrix spike recovery criteria of 75% to 125%. Silicon was not detected due to removal by HF added in the sample processing. The analytes present in the spike that were at concentrations less than 20% of the concentration in the sample are labeled as "nr" and the analytes in the spike the were at a concentration <EQL (following dilution) are labeled as "nc". Post spiking or serial dilution are used to verify QC performance of these analytes.

Post-Spiked Samples (Spike A Elements):

No post-spikes were analyzed for either AZ-101 CS AR or AZ-101 129 Residual Solids. Post spiking was, however, done for another sample run in the same ICPAES batch.

Post-Spiked Samples (Spike B Elements):

No post-spikes were analyzed for either AZ-101 CS AR or AZ-101 129 Residual Solids. As mentioned, post spiking was, however, done for another sample run in the same ICPAES batch.

Serial dilution:

All AOI'S above EQL following the five-fold serial dilution were within the tolerance of ±10%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis.... ICPASE Report

	Run Date=	9/19/2001	9/19/2001	9/19/2001	9/19/2001	9/19/2001
PNL-ALO-129	*Multiplier=*	300.9	293.4	1466.9	313.5	304.8
MOD. 1	*RPL/LAB #=*	01-1845-B@6	01-1845@6	01-1845@6	01-1845@6 - Dup	01-1845@6 - Trip
Det. Limit	*Client ID=*	*process blank*	*AZ101 CS AR*		*AZ101 CS AR-DUP*	*AZ101 CS AR-TRIP*
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	93.9		114	90.6
0.060	Al	--	113,000		94,200	106,000
0.050	B	[120]	[140]		[110]	[120]
0.010	Ba	--	487		478	438
0.010	Be	--	[9.2]		[9.0]	[8.2]
0.100	Bi	--	[55]		[45]	[48]
0.250	Ca	--	2,410		2,410	2,100
0.015	Cd	--	4,660		4,650	4,110
0.020	Cr	--	483		466	471
0.025	Cu	--	230		225	205
0.025	Fe	--	over range	70,700	64,700	60,900
2.000	K	--	[1,700]		[1,700]	[1,600]
0.050	La	--	1,750		1,810	1,580
0.030	Li	--	[68]		[75]	[64]
0.100	Mg	--	414		402	374
0.050	Mn	--	1,430		1,390	1,270
0.150	Na	[150]	64,800		57,400	67,600
0.100	Nd	--	1,240		1,270	1,120
0.030	Ni	--	2,520		2,630	2,290
0.100	P	--	[85]		[33]	[54]
0.100	Pb	--	505		454	481
0.500	Si	[280]	[1,000]		[1,200]	[830]
0.015	Sr	--	368		375	325
0.025	Ti	--	[47]		[42]	[42]
2.000	U	--	[3,500]		[3,600]	[3,300]
0.050	Zn	--	[70]		[71]	[64]
0.050	Zr	--	9,490		6,570	9,850
Other Analytes						
0.250	As	--	--		--	--
0.200	Ce	--	[430]		[420]	[400]
0.050	Co	--	[26]		[27]	[23]
0.050	Dy	--	--		--	--
0.100	Eu	--	--		--	--
0.050	Mo	--	[31]		[25]	[32]
0.750	Pd	--	[510]		[520]	[500]
0.300	Rh	--	[100]		[100]	--
1.100	Ru	--	[450]		[450]	[400]
0.500	Sb	--	--		--	--
0.250	Se	--	--		--	--
1.500	Sn	--	[950]		[820]	[750]
1.500	Te	--	--		--	--
1.000	Th	--	--		--	--
0.500	Tl	--	--		--	--
0.050	V	--	--		--	--
2.000	W	--	--		--	--
0.050	Y	--	[110]		[100]	[99]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

ASR 6193 Final - ~A0717 M. Urie ASR-6193 G. Patello ASR-6210 ICP98 hi.XLS

QC Performance 9/19/2001

Criteria>	<15% (a)	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-01845 & Dup & Trip	01-01845 LCS/BS	01-01845 & 01-01845-MS	01-01845 + Post Spike A	01-01845 + Post Spike B	01-10814 @1/@5 Serial Dil	01-10814-D @1/@5 Serial Dil	01-10814-T @1/@5 Serial Dil
Analytes	RSD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff
Ag	13	106	14	98				
Al	9.1	112	nr	107		0.5	-0.8	0.4
B				108				
Ba	5.5	108	nr	105		1.5	0.1	1.4
Be				107				
Bi		108	102	104				
Ca	7.7	112	100	106				
Cd	7.0	109	nr	107		2.7	1.2	1.8
Cr	1.8	111	77	108		1.5	0.6	2.5
Cu	6.1	111	101	108				
Fe	5.4 (b)	115	nr	108		0.1 (c)	3.1	4.1
K		94	86	105				
La	7.1	104	nr		87	0.8	-0.4	0.3
Li				105				
Mg	5.1	118	109	112				
Mn	6.2	113	nr	111		2.7	1.2	2.4
Na	8.3	114	nr	104		1.2	0.2	2.2
Nd	6.3	103	nr		94			
Ni	7.1	114	nr	109		3.6	2.4	3.0
P		109	nc	106				
Pb	5.4	111	85	105				
Si		141	nc	110				
Sr	7.6	109	nr	105		0.6	-0.5	0.7
Ti		106	85	102				
U		108	108		**99**			
Zn		109	104	107				
Zr	21	109	nr	111		0.5	-1.6	-0.3
Other Analytes								
As				**107**				
Ce					**101**			
Co				110				
Dy					95			
Eu					102			
Mo				108				
Pd		**104**	nc		**127**			
Rh		**106**	100		**101**			
Ru		**105**	nc		**111**			
Sb				**104**				
Se				**110**				
Sn								
Te								
Th					**96**			
Tl				**103**				
V				102				
W								
Y				102				

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration. nc = not calculated; result < EQL

(a) = RSD <3.5% for Na (only); (b) data from 5x dilution; (c) value for 5x/10x dilutiions

ASR 6193 Final - ~A0717 M. Urie ASR-6193 G. Patello ASR-6210 ICP98 hi.XLS

PNL-ALO-129	*Run Date=*	9/25/2001	9/25/2001	9/25/2001	9/25/2001	9/25/2001	9/25/2001	9/25/2001
	Multiplier=	570.8	637.7	567.0	573.8	200.8	200.8	200.8
MOD. 2	*RPL/LAB #=*	01-01845-B	01-01845	01-01845-DUP	01-01845-TRIP	01-02273	01-02273-DUP	01-02273-TRIP
Det. Limit	*Client ID=*	*process blank*	*AZ-101 CS AR*			*AZ-101-129 RS*		
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	(total ug)	(total ug)	(total ug)
0.025	Ag	--	[59]	[49]	[58]	[18]	[27]	[17.0]
0.060	Al	--	138,000	134,000	137,000	11,400	12,400	19,100
0.050	B	--	--	--	--	--	--	--
0.010	Ba	--	516	463	489	[13]	[13.0]	[16.0]
0.010	Be	--	[9.6]	[8.5]	[8.9]	--	--	--
0.100	Bi	--	--	--	--	--	--	--
0.250	Ca	--	2,580	2,230	2,390	[52]	--	[94]
0.015	Cd	--	5,040	4,380	4,600	61.9	50.8	119.0
0.020	Cr	--	638	1,180	583	67.2	61.9	51.5
0.025	Cu	--	259	224	231	--	--	[8.2]
0.025	Fe	--	70,100	63,900	66,300	929	842	1,450
2.000	K	--	[1,700]	[1,200]	[1,200]	--	--	--
0.050	La	--	1,900	1,700	1,780	[21]	[19.0]	[37]
0.030	Li	--	[75]	[66]	[70]	--	--	--
0.100	Mg	--	[460]	[430]	[430]	--	--	[30]
0.050	Mn	--	1,560	1,480	1,400	[32]	[32]	[39]
0.150	Na	--	72,600	65,400	72,300	790	800	1,160
0.100	Nd	--	1,340	1,200	1,250	--	--	[28]
0.030	Ni	--	2,750	2,980	2,570	[33]	[28]	[57]
0.100	P	--	1,910	1,700	1,750	831	678	759
0.100	Pb	--	[540]	[450]	[480]	--	--	--
0.500	Si	--	[990]	[820]	[1,100]	--	--	--
0.015	Sr	--	376	336	356	[4.7]	[4.1]	[7.5]
0.025	Ti	--	[50]	[91]	[52]	--	--	[5.5]
2.000	U	--	[3,800]	[3,500]	[3,400]	--	--	--
0.050	Zn	--	[81]	[73]	[83]	--	--	--
0.050	Zr	--	20,600	18,300	19,500	4,870	3,390	5,850
Other Analytes								
0.250	As	--	--	--	--	--	--	--
0.200	Ce	--	[410]	[350]	[370]	--	--	--
0.050	Co	--	--	[33]	--	--	--	--
0.050	Dy	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--
0.050	Mo	--	[45]	[110]	[36]	--	--	--
0.750	Pd	--	[530]	[450]	[500]	--	--	--
0.300	Rh	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--
1.500	Sn	--	[1,000]	--	--	--	--	--
1.500	Te	--	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--
0.050	Y	--	[120]	[110]	[110]	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 9/25/2001

Criteria>	<15% (a)	80% - 120%	80% - 120%	75%-125%	< +/-10%
QC ID=	01-01845 & Dup & Trip	01-01845 LCS/BS	01-01845 SRM2710	01-01845 & 01-01845-MS	01-01845 @1/@5 Serial Dil
Analytes	RSD (%)	%Rec	%Rec	%Rec	%Diff
Ag		94		95	
Al	5.1	101	100	nr	2.4
B		91		nc	
Ba	3.2	101	99	92	2.1
Be		98		93	
Bi		98		nc	
Ca	3.4	105	101	90	
Cd	2.5	104		nr	2.4
Cr	45	107		nr	2.3
Cu	1.9	104	97	101	
Fe	2.7	109	100	nr	3.2
K		97		nc	
La	2.7	100		nr	1.1
Li		106		87	
Mg		112	103	107	
Mn	3.2	105	103	nr	2.4
Na	6.0	103	101	nr	7.4
Nd	2.5	99		95	
Ni	10	106		nr	2.7
P	1.6	103	90	100	
Pb		109	99	103	
Si		(b)	(b)	(b)	
Sr	3.3	118	100	nr	
Ti		98	95	110	
U		98		96	
Zn		114	99	97	
Zr	3.4	105		nr	1.2
Other Analytes					
As					
Ce		98		nc	
Co					
Dy					
Eu					
Mo		103		98	
Pd					
Rh					
Ru					
Sb					
Se					
Sn					
Te					
Tl					
Th		104		nc	
V		97		95	
W				nr	
Y					

Shaded results exceed acceptance criteria

nr = not recovered; spike concentration less than 20% of sample concentration.

nc = not calculated; result < EQL.

(a) = RSD <3.5% for Na (only); (b) Si not detected in spike (removed by HF)

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IC ANION RESULTS - ASR 6193

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	M. Urie (WTP Char.)	Charge Code/Project:	W57932/ 42365
ASR Number:	6193	Sample Receipt Date:	10/13/2001
Sample Prep Date:	N/A	Sample Analysis Date:	11/13/2001
Analyst:	MJ Steele		

Preparation Procedure: For solids, PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples"
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Sample Results:

RPL #	**Liquid Sample ID**	**F(1) ug/ml**	**Cl ug/ml**	**NO_2 ug/ml**	**Br ug/ml**	**NO_3 ug/ml**	**PO_4 ug/ml**	**SO_4 ug/ml**	**C_2O_4 ug/ml**
	EQL	*0.13*	*0.13*	*0.25*	*0.13*	*0.25*	*0.25*	*0.25*	*0.25*
01-01844 PB	Water Blank	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	MRQ	*150*	*300*	*3,000*	*300*	*3,000*	*7,700*	*7,500*	*1,500*
	EQL	*125*	*125*	*2,500*	*125*	*2,500*	*250*	*250*	*250*
01-01844	AZ101 SUP AR	1,960	230	60,700	620	52,000	1,610	15,700	930
01-01844 DUP	AZ101 SUP AR	2,020	220(2)	61,600	660	52,900	1,640	16,400	1,030
01-01844 TRIP	AZ101 SUP AR	2,040	280	61,600	660	52,800	1,630	16,400	1,030
	RSD	2	13	1	4	1	1	2	6
	RPD	3	4	1	6	2	2	4	10
	LCS/BS 1113 %Rec	95	95	99	98	92	95	95	101
	LCS/BS 1113 %Rec	96	97	99	98	93	95	95	101
01-01844 MS (20,000)	Matrix Spike %Rec	96	96	102	98	95	95	96	101
01-01844 MS (2,000)	Matrix Spike %Rec	96	91	OvrRng	93	OvrRng	93	104	100
RPL #	**Solids Sample ID**	**F(1) ug/g**	**Cl ug/g**	**NO_2 ug/g**	**Br ug/g**	**NO_3 ug/g**	**PO_4 ug/g**	**SO_4 ug/g**	**C_2O_4 ug/g**
	EQL	*1.3*	*1.3*	*2.5*	*1.3*	*2.5*	*2.5*	*2.5*	*2.5*
01-01845 PB	Water Blank	< 1.3	< 1.3	< 2.5	< 1.3	< 2.5	< 2.5	< 2.5	< 2.5
	MRQ	*7,500*	*230*	*450*	*450*	*450*	*1,840*	*1,800*	--
	EQL	*130*	*13*	*260*	*130*	*260*	*26*	*260*	*26*
01-01845	AZ101 CS AR	4,490	50	27,200	350	22,800	210	26,300	2,180
01-01845 Dup	AZ101 CS AR Dup	3,850	130	27,900	360	23,300	270	22,400	4,010(3)
01-01845 Trip	AZ101 CS AR Trip	4,000	160	27,200	350	22,700	280	23,300	3,260
	RSD	8	50	1	2	1	15	9	29
	RPD	15	89	3	3	2	25	16	59
	Blank Spike/LCS %Rec	104	105	90	98	104	86	94	0(4)
	Blank Spike/LCS %Rec	106	105	90	98	104	91	94	0(4)
01-01845 MS (10)	Matrix Spike %Rec	n.r.	n.r.	n.r.	n.r.	n.r.	n.r.	n.r.	n.r.
01-01845 PS (200)	Post Spike %Rec	99	95	102	94	98	93	100	100

OvrRng = over range; EQL = estimated quantitation limit; MRQ = minimum reportable quantity; RSD = relative standard deviation; RPD = relative percent difference; n.r. = not recovered; matrix spike concentration less than 20% of sample concentration.

(1) The fluoride results should be considered the upper bound concentration for the fluoride, since the fluoride peak shape and retention time suggests the presence of co-eluting anion(s), possibly formate or acetate.

(2) Results based on response approximately 5% below the lowest calibration standard.

(3) Results based on response approximately 5% above the highest calibration standard.

(4) Oxalate not recovered in the hot cell BS/LCS, apparently due to precipitation (See QC Comments).

The liquid sample was prepared for ion chromatography anion analysis by diluting to ensure that the anions were measured within the calibration range and that the IC column was not overloaded during the analysis. Liquid samples required dilutions from 1,000-fold to 10,000-fold.

Solids samples were leached in the Shielded Analytical Laboratory hot cells at approximately 10 to 1 with distilled, deionized water. Following the leaching processing, the samples required additional 10-fold to 100-fold dilution to ensure that the anions were measured within the calibration range.

The estimated quantitation levels (EQL) are provided in the above table and are based on the lowest calibration standard and the dilutions used for reporting the results.

Q.C. Comments:

Liquid Sample Analysis:

Duplicates/Triplicates: The AZ-101 SUP AR sample was sub-sampled in triplicate for analysis in the laboratory. The sample/duplicate relative percent difference (RPD) meets the acceptance criteria of <20% of the laboratory's QA plan. Although the chloride triplicate relative standard deviation (RSD) is near the limit of <15% specified by the ASR attachment Table 4, all anions meet the RSD criteria.

Laboratory Control Sample/Blank Spike - (HCV 010912 @2x [BS 1113]): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared and measured at the same time as the Matrix Spike sample and demonstrate recoveries within the 80% to 120% acceptance criteria.

Matrix Spike (HCV 010912 @2x): A matrix spike was prepared from a sub-sample of AZ-101 SUP AR and all anion recoveries are within the 75% to 125% recovery acceptance criteria. At the 2,000-fold dilution of the sample, nitrite and nitrate recoveries could not be calculated since the measured concentrations were significantly above the highest calibration standard.

Solids Sample Analysis:

Duplicates/Triplicates: Three portions of sample AZ-101 CS AR were leached and analyzed in the laboratory. Chloride, phosphate, and oxalate do not meet the duplicate relative percent difference (RPD) acceptance criteria of <20% from the laboratory's QA plan. The chloride concentration is near the EQL and precision is expected to be poor. The poor precision demonstrated by the phosphate and oxalate may be due to solids sample heterogeneity or from solubility issues. Chloride and oxalate also fail the triplicate RSD of <15% from the ASR attachment Table 5.

Laboratory Control Sample/Blank Spike (MS 010912 halide + MS 010912 oxy-anions @~12.2x): A Blank Spike was prepared in the hot cells from two leach matrix spike standards (halides and oxy-anions). The blank spike was processed identically to the samples (i.e., leached/stirred) and, except for oxalate, demonstrates recoveries meeting the 80% to 120% acceptance criteria. The loss of the oxalate was unexpected and can not be explained; however, this loss is under further investigation.

Matrix Spike (MS 010912 halide + MS 010912 oxy-anions @~12.1x): A matrix spike was prepared from the AZ-101 CS AR wet centrifuges solids assuming that no additional dilution would be required following the leaching of the solids. The solids matrix spike was unusable due to 1) the spiking levels were <20% of the sample concentration or 2) the additional dilutions required for the sample analysis diluted the matrix spike to below the EQL.

Post Spike (HCV 010912 @2x): Following the failure to recover the matrix spikes, a post spike was prepared from the AZ-101 CS AR leachate. All anion post spike recoveries are within the acceptance criteria of 75% to 125%.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: ____________ Date 1-7-02

Review/Approval: MJ Steele Date 1-8-02

Excel Archive Information: ASR 6193 Urie.xls

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TOC/TIC RESULTS - ASR 6193

Hot Persulfate

Battelle PNNL/RSE/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client:	M. Urie	**Charge Code/Project:**	W57933 / 42365
RPL Numbers:	01-1844 and 01-1845	**ASR Number:**	6193
Analyst:	MJ Steele	**Analysis Date:**	May 9-10, 2002

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023)

Analysis Results

Liquids RPL Number	Sample ID	TIC MDL µgC/mL	TIC Results µgC/mL	TIC RSD	TOC MDL µgC/mL	TOC Results µgC/mL	TOC RSD	TC Results µgC/mL	TC RSD
	MRQ		150			1,500			
01-01844	AZ-101-SUP2 AR	34	9,810		87	560		10,400	
01-01844 D	AZ-101-SUP2 AR Dup	34	9,820		87	500		10,300	
01-01844 T	AZ-101-SUP2 AR Trip	34	9,920	1%	87	470	9%	10,400	1%
01-01844 MS	%Recovery		105%			103%		104%	
Blank Spike/LCS	%Recovery		100%			97%			

Wet Solids RPL Number	Sample ID	TIC MDL µgC/g	TIC Results µgC/g	TIC RSD	TOC MDL µgC/g	TOC Results µgC/g	TOC RSD	TC Results µgC/g	TC RSD
	MRQ		30			60			
01-01845	AZ-101-CS2	47	8,780		120	960		9,770	
01-01845 D	AZ-101-CS2 AR Dup	59	8,600		150	880		9,510	
01-01845 T	AZ-101-CS2 AR Trip	56	7,750	7%	140	630	21%	8,410	8%
01-01845 MS	Recovery		96%			89%		93%	
Blank Spike/LCS	Recovery		101%			93%			

MRQ = minimum required quantity; RSD = Relative Standard Deviation; MDL = method detection limit; na = not applicable

The TOC/TIC analyses of the supernatant and wet centrifuged solids samples submitted under ASR 6193 are to be performed by both the hot persulfate and furnace methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

Quality Control Comments:

The TIC analysis uses calcium carbonate and the TOC analysis uses α-D-Glucose as the calibration, laboratory control, and matrix spike standards. (The JT Baker, Aldrich, Sigma, and Mallinckrodt Chemical Measurement System numbers are provided on the raw data benchsheets for traceability).

The QC for the method involves calibration blanks, sample triplicates, laboratory control sample, and matrix spikes per analysis batch. The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs"; the performance of the QC samples is compared to this Plan. The ASR provided a more stringent precision acceptance criteria than the QA Plan (i.e,. <15% RSD versus <20% relative percent difference).

Supernatant (01-1844 QC)

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyzed with the batch of samples. At 100% TIC and 97% TOC, the LCS/BS recovery is well within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from the sample, an inorganic standard, and an organic standard. The TIC and TOC matrix spike recoveries are well within the acceptance criteria of 75% to 125% recovery. At 104%, the TC recovery (TIC + TOC standard) indicates that all the carbon added as a matrix spike was recovered.

Triplicates: The TIC and TOC measurement precision is demonstrated by the RSD of the triplicate analyses. Although the TOC RSD is slightly elevated, both the TIC and TOC RSDs meet the acceptance criteria of <15%. The 9% RSD for the TOC is essentially a result of the low TOC concentration, which is only about 6 times the MDL.

Wet Centrifuged Solids (01-1845 QC)

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyzed with the batch of samples. At 101% TIC and 93% TOC, the LCS/BS recovery is well within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from the sample, an inorganic standard, and an organic standard. The TIC and TOC matrix spike recoveries are well within the acceptance criteria of 75% to 125% recovery. At 93%, the TC recovery (TIC + TOC standard) indicates that nearly all of the carbon added as a matrix spike was recovered.

Triplicates: The TIC and TOC measurement precision is demonstrated by the RSD of the triplicate analyses. The TIC RSD meets the acceptance criteria of <15%, whereas the TOC RSD exceeds the criteria. The 20% RSD for the TOC is essentially a result of the low TOC concentration, which is only about 8 times the MDL. At these TOC concentrations, higher RSD are expected.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data. The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: MW Urie **Date** 6/4/02

Review/Approval by: DE Baldwin **Date** 6-5-02

Excel Archive File: ASR 6193 Urie L&S HP.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie	Analyzer M&TE:	WC01713 -- 701
Project :		Balance M&TE:	360-06-01-023
Work Pkg:	CMC	TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS)	40.00% Carbon <<[G]
Analyzed:	May 9, 2002	TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS)	11.99% Carbon <<[C]
ASR:	6193		

		Raw TIC (ug C)	Raw TOC (ug C)	TIC	TOC		Is Blank Std Dev < Method Det Limit?	
				17.5	101.2	**<<< Blank Average (ug C)**		
Blanks:	Calibration blank (start of batch)	21.6	116.8	3.4	32.4	**<<< Blank Std Dev (ug C)**	TIC	Yes
	Calibration blank (start of batch)	16.5	142.0	*2.16*	*5.8*	***<<< Pooled Std Dev (ug C)***	TOC	No
	Calibration blank (start of batch)	14.3	110.0					
	Calibration blank (start of batch)	14.6	75.0					
	Calibration blank (end of batch)	20.6	62.0	*6.5*	*17.3*	***<<< Method Det. Limit (ug C)***		

		Total Inorganic Carbon (TIC)					**Total Organic Carbon (TOC)**				
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch)	2783	18	0.0232	99.4		1411	101	0.0031	105.6	
	Calibration Standard (start of batch)	1778	18	0.0157	93.5		1484	101	0.0033	104.8	
	Calibration Standard (start of batch)	1601	18	0.0135	97.8		1435	101	0.0035	95.3	
	Calibration Standard (end of batch)	1740	18	0.0146	98.4		1160	101	0.0027	98.0	
		[L] Average TIC % Rec >>>>			**97.3**	**<<[L]**	**[P] Average TOC % Rec >>>>**			**100.9**	**<<[P]**
QC	Blank Spike/LCS	1564	18	0.0131	101.2		2052	101	0.0052	92.9	

Formulas:

Standard TIC % Recovery = ((A-B)/((C/100)*D))*10^{-6}*100

Standard TOC % Recovery = ((E-F)/((G/100)*H))*10^{-6}*100

Sample TIC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

Sample TOC (ug C/ml or ug C/g)= (M-N)/(O*P/100)

Matrix Spike Recoveries:

TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U

TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

TIC and TOC are measured; TC is the sum of the TIC and TOC results.

F06-006

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie	Analyzer M&TE: WC01713 -- 701
Project :		Balance M&TE: 360-06-01-023
Work Pkg:	CMC	TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS) 40.00% Carbon <<[G]
Analyzed:	May 9, 2002	TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS) 11.99% Carbon <<[C]
ASR:	6193	

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam wt (g)	TIC (ug C/g)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam wt (g)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
01-01845	AZ-101-CS2	1237	18	0.1428	8,778		240	101	0.1428	963		9,741	
01-01845 D	AZ-101-CS2 AR Dup	972	18	0.1141	8,598	2	202	101	0.1141	876	10	9,474	3
01-01845 T	AZ-101-CS2 AR Trip	916	18	0.1191	7,754		177	101	0.1191	631		8,385	
01-01845 MS	AZ-101-CS2 AR MS	1660	18	0.1302	see below		544	101	0.1302	see below		see below	

(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K]

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/g)	[T] MS Sam wt (g)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
01-01845 MS	TIC Recovery	1660	18	7754	0.1302	1010	0.0059	707	95.9	TIC
	TOC Recovery	544	101	631	0.1302	82	0.0010	400	89.2	TOC
	Total Carbon Recovery (TIC + TOC)							1107	93.5	TC

prepared by MJ Steele 6-4-02

Reviewer/date: DL Baldwin 6-5-02

F06-007

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie
Project :	
Work Pkg:	CMC
Analyzed:	May 10, 2002
ASR:	6193

Analyzer M&TE: WC01713 -- 701

Balance M&TE: 360-06-01-023

TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS) 40.00% Carbon <<[G]

TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS) 11.99% Carbon <<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch)	24.6	61.7
	Calibration blank (start of batch)	19.1	59.2
	Calibration blank (end of batch)	23.3	57.3

TIC	TOC	
22.3	59.4	<<< Blank Average (ug C)
2.9	2.2	<<< Blank Std Dev (ug C)
2.16	*5.8*	*<<< Pooled Std Dev (ug C)*
6.5	*17.3*	*<<< Method Det. Limit (ug C)*

Is Blank Std Dev < Method Det Limit?	
TIC	Yes
TOC	Yes

		Total Inorganic Carbon (TIC)					Total Organic Carbon (TOC)				
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch)	2079	22	0.0177	96.9		976	59	0.0023	99.6	
	Calibration Standard (start of batch)	2279	22	0.0195	96.5		909	59	0.0021	101.1	
	Calibration Standard (end of batch)	1825	22	0.0157	95.8		985	59	0.0024	96.4	
		[L] Average TIC % Rec >>>>			96.4	<<[L]	[P] Average TOC % Rec >>>>			99.1	<<[P]
QC	Blank Spike/LCS 1	1064	22	0.0090	100.1		1410	59	0.0035	97.4	

Formulas:

Standard TIC % Recovery = $((A-B)/((C/100)*D))*10^{-6}*100$

Standard TOC % Recovery = $((E-F)/((G/100)*H))*10^{-6}*100$

Sample TIC (ug C/ml or ug C/g) = $(I-J)/(K*L/100)$

Sample TOC (ug C/ml or ug C/g)= $(M-N)/(O*P/100)$

Matrix Spike Recoveries:

TIC % Recovery = $(((Q-R)/(L/100))-S*T)*100/U$

TOC % Recovery = $(((Q-R)/(P/100))-S*T)*100/U$

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

TIC and TOC are measured; TC is the sum of the TIC and TOC results.

F06-008

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie	Analyzer M&TE: WC01713 -- 701		
Project :		Balance M&TE: 360-06-01-023		
Work Pkg:	CMC	TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS)	40.00% Carbon	<<[G]
Analyzed:	May 10, 2002	TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS)	11.99% Carbon	<<[C]
ASR:	6193			

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

ACL Number	Client Sample ID (Liquids)	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam Vol (ml)	TIC (ug C/ml)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam Vol (ml)	TOC (ug C/ml)	TOC RPD (%)	TC (ug C/ml)	TC RPD (%)
01-01844	AZ-101-SUP2 AR	1914	22	0.20	9,812		170	59	0.20	558		10,370	
01-01844 D	AZ-101-SUP2 AR Dup	1915	22	0.20	9,817	0	158	59	0.20	498	11	10,315	1
01-01844 T	AZ-101-SUP2 AR Trip	1934	22	0.20	9,916		152	59	0.20	467		10,383	
01-01844 MS	AZ-101-SUP2 AR MS	3023	22	0.20	see below		946	59	0.20	see below		see below	

(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K])

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/ml)	[T] MS Sam Vol (ml)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
01-01844 MS	TIC Recovery	3023	22	9812	0.20	1962	0.0091	1091	105.4	TIC
	TOC Recovery	946	59	558	0.20	112	0.0019	760	103.1	TOC
	Total Carbon Recovery (TIC + TOC)							1851	104.5	TC

Reviewer/date: 6-4-02

prepared by MJ Steele 6-4-02

F06-009

Furnace

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client	M Urie	**Charge Code/Project:**	W57932 / 42365
RPL Numbers:	01-1844 to 01-1845	**ASR Number:**	6193
Analyst:	MJ Steele	**Analysis Date:**	May 21-22, 2002

Procedure: PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"

M&TE: Carbon System (WD13071); Balance (360-06-01-023)

TOC/TIC/TC Results

Supernatant RPL Number	Sample ID	TIC (a) Results µgC/mL	TIC RSD	TOC MDL µgC/mL	TOC Results µgC/mL	TOC RSD	TC MDL µgC/mL	TC Results µgC/mL	TC RSD
01-1844	AZ-101 CS AR	5,540		250	1,780		89	7,320	
01-1844 D	AZ-101 CS AR Dup	6,080		250	1,160		89	7,240	
01-1844 T	AZ-101 CS AR Trip	5,880	5%	250	1,600	21%	89	7,480	2%
MS 01-1845	%Recovery				81%			88%	
Blank Spike/LCS	%Recovery				102%			97%	

Wet Solids RPL Number	Sample ID	TIC Results µgC/g	TIC RSD	TOC MDL µgC/g	TOC Results µgC/g	TOC RSD	TC MDL µgC/g	TC Results µgC/g	TC RSD
01-1845	AZ-101 CS AR	1,200		1,100	9,300		350	10,500	
01-1845 D	AZ-101 CS AR Dup	< 1,300		2,000	10,800		500	9,000	
01-1845 T	AZ-101 CS AR Trip	< 1,000	(a)	1,000	9,600	8%	850	9,500	8%
01-1845 MS	%Recovery				81%			104%	
Blank Spike/LCS	%Recovery				102%			97%	

MRQ = minimum required quantity; RSD = Relative Standard Deviation; MDL = method detection limit

(a) RSD not calculated, since results not >5x MDL
(b) TIC is determined by difference (TC - TOC)
(c) TOC for 01-1844 continued to increase with time, suggesting decomposition of carbonate based on Hot Persulfate results

The TOC/TIC analyses of the supernatant and wet centrifuged solids samples submitted under ASR 6193 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 700 °C for 10 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 10 minutes. The total inorganic carbon is obtained by difference.

The table above shows the results, rounded to two or three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

Q.C. Comments:

The calibration and QC standards for TC and TOC analysis are solid carbon pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt. The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The coulometer analysis system calibration is checked by analyzing calibration standards at the beginning, middle, and end of each day's run. The supernatant and wet solids were analyzed for TOC as a batch and for TC as a batch; i.e., the batch analytical QC applies to both phases for each batch. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the TOC was 95% and for TC was 101%.

System carbon blanks were analyzed, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The system blanks are used to calculate an estimated method detection limit (MDL) based on the standard deviation of the system blanks and the number of system blanks processed (See attached calculation work sheets).

The QC for the analyses include sample triplicates, blank spikes (as a laboratory control sample), and matrix spikes. The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs", Sections 4 and 5. The performance of the QC samples is compared to this Plan. The exception being the precision acceptance criteria; the ASR establishes a more stringent precision criteria than the QA Plan (i.e,. <15% RSD versus <20% relative percent difference).

Analysis Run May 21: TC QC

Blank Spike/Laboratory Control Sample: A TC LCS/BS (inorganic standard) was analyzed with the batch of samples. At 97% TC, the LCS/BS recovery is well within acceptance criteria of 80% to 120%.

Triplicates: The TC measurement precision is demonstrated by the RSD of the triplicate analyses. The TC RSDs for both the supernatant and wet solids meet the acceptance criteria of <15%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from both the supernatant and wet solids sample by adding a know quantity of an inorganic standard. The TC matrix spike recoveries for both the supernatant (88%) and wet solids (104%) are well within the acceptance criteria of 75% to 125% recovery.

Analysis Run May 22: TOC QC

Blank Spike/Laboratory Control Sample: A TOC LCS/BS was analyzed with the batch of samples. At 102% TOC, the LCS/BS recovery is well within acceptance criteria of 80% to 120%.

Triplicates: The TOC measurement precision is demonstrated by the RSD of the triplicate analyses. The TOC RSD for the wet solids meets the acceptance criteria of <15%. The supernatant TOC results are only slightly above 5-times the MDL for the sample sized used, and at these low concentrations the high RSD is not unexpected.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from the wet solids sample by adding a know quantity of an organic standard. The TOC matrix spike recovery for the wet solids was somewhat low (i.e., 81%), but was within the acceptance criteria of 75% to 125% recovery.

Comparison of Furnace Results to Hot Persulfate Results

Supernatant RPL Number	Sample ID	HP TIC Results µgC/mL	Furn TIC Results(a) µgC/mL	HP TOC Results µgC/mL	Furn TOC Results µgC/mL	HP TC (b) Results µgC/mL	Furn TC Results µgC/mL
01-1844	AZ-101-SUP2 AR	9,810	5,540	560	1,780	10,400	7,040
01-1844 D	AZ-101-SUP2 AR Dup	9,820	6,080	500	1,160	10,300	6,960
01-1844 T	AZ-101-SUP2 AR Trip	9,920	5,880	470	1,600	10,400	7,190
	RSD	1%	5%	9%	21%	1%	2%
Wet Solids RPL Number	**Sample ID**	**HP TIC Results µgC/g**	**Furn TIC Results(a) µgC/g**	**HP TOC Results µgC/g**	**Furn TOC Results µgC/g**	**HP TC (b) Results µgC/g**	**Furn TC Results µgC/g**
01-1845	AZ-101-CS2 AR	8,780	1,200	960	9,300	9,770	10,500
01-1845 D	AZ-101-CS2 AR Dup	8,600	< 1,300	880	10,800	9,510	9,000
01-1845 T	AZ-101-CS2 AR Trip	7,750	< 1,000	630	9,600	8,410	9,500
	RSD	7%		21%	8%	8%	8%

HP = Hot Persulfate Method; Furn = Furnace Combustion Method; RSD = Relative Standard Deviation

a) TIC Furn is determined by difference (TC - TOC)
b) TC HP is determined by sum (TIC + TOC)

Wet Solids: The two method appear to produce similar results for TC for the wet centrifuged solids samples However, there are significant differences between the TIC and TOC results. It appears that the nearly all the carbon (both inorganic and organic compounds) is combusting at the 700 °C used for the furnace method. Some metal carbonates, such as iron, magnesium, and nickel, will typically fully oxidize at 700 °C, and may be the reason for the very high TOC results from the furnace method. It is unlikely that the hot persulfate acid wet oxidation is bias high; i.e., organic compounds typically present in the tank waste should not be oxidized. Conclusion: Best results are the hot persulfate results for the TIC and furnace results for TC. Based on this the best TOC results are those produced by the hot persulfate method for this sample.

Supernatant: For the supernatant sample, the trend is similar (i.e., inorganic carbon compounds combusting at the 700 °C furnace analysis), but not as pronounced. The analysis time for the furnace method TOC analysis is 10 minutes; extending the analysis time by 20 minute produced TOC results approximately twice as high as those reported. This effect is typically either from difficult to oxidize organic compounds or inorganic compounds that wholly or partially oxidize at 700 °C. The fact that the furnace TC results are significantly lower than the hot persulfate TC results is very unusual, and is difficult to evaluate. However, the hydroxide titration (2nd and 3rd inflection points equivalent to 0.88 and 0.70 molar OH, respectively) results suggest that the carbon from carbonate should be close to the hot persulfate TIC results. Based on the hydroxide supporting data, the furnace results for the supernatant should not be used.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- For both the TC and TOC, the analysis Method Detection Limit (MDL) is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's *t* values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.

Report Prepared by: ______________________ Date 6/4/02

Review/Approval by: ______________________ Date 6-5-02

Excel Archive File: ASR 6193 6265-01 6284some Furn.xls

F06-015

PNNL Radiochemical Processing Group: TOC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie/Urie/Geeting	Temp. 700 Degree C	Analyzer M&TE: WD13071 -- 701 Balance M&TE: 360-06-01-023
Project :			
Work Pkg:	CMC	Run time 10 Minutes	CAL STD - alpha-D-glucose: Aldrich, lot HY12603EY, CMS# 53219 40.00% Carbon <<[G]
Analyzed:	May 22, 2002		BS/MS -alpha-D-glucose: Sigma Lot 58H01281, CMS# 161713 40.00% Carbon <<[C]
ASR:	6193, 6265.01, 6284(some)		

		TOC (ug C)	
Blanks:	Calibration blank (start of batch)	28.5	
	Calibration blank (start of batch)	28.3	
	Calibration blank (end of batch)	10.2	

22.3	**<<< Blank Average (ug C)**
10.5	**<<< Blank Std Dev (ug C)**
3	***<<< # of Blanks analyzed***
47	***<<< Method Det. Limit (ug C)* [M]**

94.8 <<<< [L] Average TOC % Rec

		TOC			
		[A] Raw TOC (ug)	[B] Blk (ug)	[D] Std Wt (g)	TOC % Rec
Standards:	Calibration Standard (start of batch)	1371	22	0.0034	99.2
	Calibration Standard (start of batch)	1310	22	0.0032	100.6
	Calibration Standard (middle of batch)	930	22	0.0024	94.6
	Calibration Standard (end of batch)	498	22	0.0014	84.9
QC	Blank Spike/LCS	3295	22	0.0085	101.5

Formulas:

Standard TOC % Recovery = $((A-B)/((C/100)*D))*E^{-6}*100$

QC % Recovery = $(((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)$

Sample TOC (ug C/ml or ug C/g) = $(I-J)/(K*L/100)$

MS TOC % Recovery = $((((Q-R)/(L/100))-S*T)*100)/U$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).

For any TOC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

Note: Sample weights are on "as received" basis; i.e., wet weight

385.683 406.772502 9329.645

Sample Results		[I] Raw	[J]	[K] Sam		TOC	
RPL Number	Sample ID	TOC (ug C)	Blk (ug C)	Wt (g)		(ug C/g)	RPD (%)
ASR 6193							
01-1845	AZ-101 CS AR	408	22	0.0436	9330	9,330	
01-1845 D	AZ-101 CS AR Dup	270	22	0.0242	10794	10,794	15
01-1845 T	AZ-101 CS AR Trip	450	22	0.0472	9557	9,557	
01-1845 MS	AZ-101 CS AR Matrix Spike	1301	22	0.0265		see below	

Matrix Spike Results		[Q] Raw MS	[R] MS Bl	[S] Sam		[T] MS Sam	[V] Sampl	Spike	[U] Spike	MS
RPL Number	Client Sample ID	(ug C)	(ug C)	(ug C/ml)		Vol. (ml)	(ug C)	wt (g)	(ug C)	% Recovery
01-1845 MS	Total Organic Carbon Recovery	1301	22	9330		0.04	407	0.0029	1160	81.2

Reviewer/date: DL Baldwin 6-5-02

prepared by MJ Steele 6-4-02

F06-016

Note: Sample weights are on "as received" basis; i.e., wet weight

Sample Results		[I] Raw	[J]	[K] Sam	TOC	
RPL Number	Sample ID	TOC (ug C)	Blk (ug C)	Wt (g)	(ug C/g)	RPD (%)
ASR 6193						
01-1845	AZ-101 CS AR	408	22	0.044	9,330	
01-1845 D	AZ-101 CS AR Dup	270	22	0.024	10,794	15
01-1845 T	AZ-101 CS AR Trip	450	22	0.047	9,557	
01-1845 MS	AZ-101 CS AR Matrix Spike	1301	22	0.027	see below	

Matrix Spike Results		[Q] Raw MS	[R] MS Bl	[S] Sam	[T] MS Sam	[V] Sampl	Spike	[U] Spike	MS
RPL Number	Client Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol. (ml)	(ug C)	wt (g)	(ug C)	% Recovery
01-1845 MS	Total Organic Carbon Recovery	1301	22	9330	0.04	407	0.0029	1160	81.2

Reviewer/date: 6-5-02

Prepared by MJSteele 6-4-02

F06-017

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie/Urie/Geeting	Temp.	Balance M&TE: 360-06-01-023
Project :		1000 Degree C	
Work Pkg:	CMC	Run time	
Analyzed:	May 21, 2002	10 Minutes	TIC Cal Std: JT Baker CMS#161359 11.99% Carbon <<[G]
ASR:	6193, 6265.01, 6284some		LCS/BS/MS TIC Std: Mallinckrodt CMS#161732 11.99% Carbon <<[C]

		Raw TC (ug C)	
Blanks:	Calibration blank (start of batch)	20	
	Calibration blank (start of batch)	20	
	Calibration blank (end of batch)	13	

17.3	<<< Blank Average (ug C)
4.0	<<< Blank Std Dev (ug C)
3	<<< *# of Blanks analyzed*
18.0	<<< *Method Det. Limit (ug C)* [M]

		Total Carbon (TC)			
		[A] Raw TC (ug)	[B] Blk (ug)	[D] Std Vol (ml)	TIC % Rec
Standards:	Calibration Standard (start of batch)	2757	17	0.0230	99.3
	Calibration Standard (start of batch)	4288	17	0.0359	99.2
	Calibration Standard (end of batch)	2091	17	0.0164	105.5
QC	Blank Spike/LCS	1851	17	0.0156	96.7

101.3 <<< [L] Average TC % Rec

Formulas:

Standard TC % Recovery = ((A-B)/((G/100)*D))*E^{-6}*100

QC % Recovery = (((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)

Sample TC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

MS TC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).

For any TC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

F06-018

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie/Urie/Geeting	Temp.	Balance M&TE: 360-06-01-023
Project :		1000 Degree C	
Work Pkg:	CMC	Run time	
Analyzed:	May 21, 2002	10 Minutes	TIC Cal Std: JT Baker CMS#161359 11.99% Carbon <<[G]
ASR:	6193, 6265.01, 6284some		LCS/BS/MS TIC Std: Mallinckrodt CMS#161732 11.99% Carbon <<[C]

Sample Results		[I] Raw	[J]	[K] Sam	TC	TC
RPL Number	Sample ID	TC (ug C)	Blk (ug C)	Vol. (ml)	(ug C/ml)	RPD (%)
ASR 6193						
01-1844	AZ-101 SUP AR	1501	17	0.20	7,320	
01-1844 D	AZ-101 SUP AR Dup	1485	17	0.20	7,241	1
01-1844 T	AZ-101 SUP AR Trip	1533	17	0.20	7,478	
01-1844 MS	AZ-101 SUP AR Matrix Spike	2863	17	0.20	see below	

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol. (ml)	(ug C)	wt (g)	(ug C)	% Recovery
01-1844	Total Carbon Recovery (TC)	2863	17	7320	0.20	1464	0.0128	1535	87.6

Reviewer/date: DL Baldwin 6-5-02

prepared by MJ Steele 6-4-02

F06-019

Note: Sample weights are on "as received" basis; i.e., wet weight

Sample Results		[I] Raw	[J]	[K] Sam	TC	TC
RPL Number	Sample ID	TC (ug C)	Blk (ug C)	Wt (g)	(ug C/g)	RPD (%)
ASR 6193						
01-1845	AZ-101 CS AR	562	17	0.0513	10,478	
01-1845 D	AZ-101 CS AR Dup	337	17	0.0352	8,963	16
01-1845 T	AZ-101 CS AR Trip	219	17	0.0209	9,524	
01-1845 MS	AZ-101 CS AR Matrix Spike	1323	17	0.0421	see below	

Matrix Spike Results		[Q] Raw M	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	Sample ID	(ug C)	(ug C)	(ug C/ml)	Wt (g)	(ug C)	wt (g)	(ug C)	% Recovery
01-1845	Total Carbon Recovery (TC)	1323	17	10478	0.0421	441	0.0068	815	103.9

Reviewer/date: 6-5-02

Prepared by MJSteele 6-4-02

F06-020

F7

CYANIDE RESULTS - ASR 6193

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Radiological Processing Group (RPG)

Inorganic Analysis - Cyanide Data Report

Project / WP#:	42365 / K46059
ASR#:	6193
Client:	Mike Urie
Total Samples:	2

RPL #	Client ID
01-1844	AZ-101 SUP AR
01-1845	AZ-101 CS AR

Procedure:	PNL-ALO-287 Rev. 0, *Midi and Micro Distillation of Cyanide in Liquid and Solid Samples* PNL-ALO-298 Rev. 0, *Total Cyanide Determination by Spectrophotometry (Manual or Automated) or Argentometric Titration*	
M&TE Number:	WC36517	Lachet QuikChem Analyzer
	360-06-01-036	Mettler AT400 Balance
	360-06-01-031	Mettler AT400 Balance
Analyst:	LMP Thomas	
Analysis Date:	10/15/01	
Analysis Files:	Calibration - 01101502 Sample Trays - 01101502.RS	

For Calibration and Maintenance Records, see
Chemical Measurement Center 98620 file:

LMP Thomas 6/5/02
Prepared By

6-5-02
Reviewed By

Two samples, As-received AZ-101 Supernatant and Wet Centrifuged Solids, were submitted for cyanide analysis. The samples were aliquoted in the hot cell and analyzed by automated spectrophotometry. The Wet Centrifuged Solids were prepared in the hot cell and the Supernatant was prepared in a fume hood. The analysis of these samples included a duplicate, triplicate, matrix spike and matrix spike duplicate. Quality control check standards relative to the sample preparation and instrument performance were also prepared and analyzed.

1. Analysis

Results from the analysis of the As-received AZ-101 Supernatant are provided in the table below. The concentrations are reported in mg of cyanide per L of sample.

RPD success criteria: < 20%
RSD success criteria: <15%

RPL ID	Sample ID	Det Limit mg/L	Measured mg/L	RPD %
01-1844	AZ-101 SUP AR	0.057	1.53	
01-1844	AZ-101 SUP AR DUP	0.053	1.19	25
01-1844	AZ-101 SUP AR TRIP	0.054	3.40	92
	Average		2.04	
	RSD		58	
	Est MDL [1]		0.055	
	EQL [2]		0.59	
	Preparation DF (v/v)		59	
	Analysis DF (v/v)		1	

DF - dilution factor

(1) The estimated MDL is based on an MDL evaluated for liquid samples (ASR 6091) adjusted by the appropriate dilution factors.

(2) The EQL is based on the lowest calibration standard, 0.01 mg/L, multiplied by the total dilution factor.

Results from the analysis of the As-received AZ-101 Centrifuged Solids sample, are provided in the table below. The concentration is reported in mg of cyanide per kg of wet centrifuged solids.

RPD success criteria: < 20%
RSD success criteria: <15%

RPL ID	Sample ID	Det Limit mg/kg	Measured mg/kg	RPD %
01-1845	AZ-101 CS AR	0.042	0.891	
01-1845	AZ-101 CS AR DUP	0.039	< 0.039	183
01-1845	AZ-101 CS AR TRIP	0.039	0.692	37
	Average(1)		0.791	
	RSD		57	
	Est MDL (2)		0.040	
	EQL (3)		0.108	
	Preparation DF (mLs/g)		11	
	Analysis DF (v/v)		1	

DF - dilution factor

(1) The average is based on the initial and triplicate sample.

(2) The estimated MDL is based on an MDL evaluated for solid samples (ASR 6091) adjusted by the appropriate dilution factors.

(3) The EQL is based on the lowest calibration standard, 0.01 mg/L, multiplied by the total dilution factor.

2. Quality Control

Duplicate (DUP). The RPDs and RSDs for both supernatant and wet centrifuged solids do not meet the QC success criteria. When the releasing agent was added to the samples a vigorous reaction occurred. In other samples where this reaction was observed, poor yields were often the result and it was presumed that cyanide was being volatilized and lost before the sample tube could be capped by the distillation tube. For the analysis of the AZ-101 samples, the releasing agent was pipetted into small vials, which were then added to the sample tube. The small vials floated on the surface of the sample while the distillation tube was capped onto the sample tube. The distillation assembly was then inverted to mix the releasing agent into the sample. However, the reaction was so vigorous that the cyanide may have been released too rapidly for the trapping solution to capture all of the cyanide. In addition, when the distillation assembly was being inverted for mixing, there were some areas in the cross section of the distillation tube where the off-gases, for a brief time, did not contact the trapping solution.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD). The spike recoveries as well as the RPD between the MS and MSD in the supernatant meet the QC success criteria. However, for the wet centrifuged solids, the spike recoveries and RPD between the MS and MSD do not meet the QC success criteria. This is most likely due to the same cause as the QC failures from the analyses of the initial, duplicate and triplicate samples.

MS success criteria: 75% to 125% of expected value							
RPD success criteria: < 20%							
RPL ID	**Sample ID**	**Det Lim mg/L**	**Spike mg/L**	**Sample mg/L**	**Measured mg/L**	**Rec %**	**RPD %**
01-1844	AZ-101 SUP AR MS	0.053	9.49	2.04	10.3	87	
	AZ-101 SUP AR MSD	0.057	10.3	2.04	12.4	101	14
RPL ID	**Sample ID**	**Det Lim mg/kg**	**Spike mg/kg**	**Sample mg/kg**	**Measured mg/kg**	**Rec %**	**RPD %**
01-1845	AZ-101 CS AR MS	0.046	2.04	0.79	1.34	27	
	AZ-101 CS AR MSD	0.040	1.80	0.79	0.84	3.0	159

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PBs are based on the average sample size of the sample aliquots. The PBs meet the success criteria. The LCS solid-old is an expired solid standard, which has consistently produced satisfactory recovery. The LCS solid-new is an unexpired solid standard. However, the mesh size was larger. It is this characteristic that is thought to have caused the wide variability in recovery seen in previous analyses. The LCS solid-new used in this analysis is a portion of the unexpired solid standard, which has been ball-milled to a powder. However, previous analysis of the ball-milled standard has still yielded recoveries which vary widely. The LCS liquid and LCS solid-old meet the QC success criteria. The LCS solid-new is outside ±10% of the certified value and also outside the 80.4 to 322 mg/kg vendor performance acceptance limits.

Sample ID	**PB success criteria: < EQL**		**LCS success criteria: vendor certification or 80% - 120% of expected value**		
	Success Criteria mg/L	**Measured mg/L**	**Expected mg/L**	**Measured mg/L**	**Recovery %**
PB liquid	< 0.59	0.099			
LCS liquid			0.302	0.316	105
	Success Criteria mg/kg	**Measured mg/kg**	**Expected mg/kg**	**Measured mg/kg**	**Recovery %**
PB solid	< 0.11	< MDL			
LCS solid-old			177	161	91
LCS solid-new			201	75	37

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 0.25 M NaOH, which is the matrix of the distillates, and are not distilled. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: <EQL		
Sample ID	**Success Criteria mg/L [(1)]**	**Measured mg/L [(1)]**
ICB	< 0.010	< 0.010
CCB 1	< 0.010	< 0.010
CCB 2	< 0.010	< 0.010
CCB 3	< 0.010	< 0.010
(1) Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 85% to 115% recovery **Calibration Range: 0 - 0.30 mg/L**				
	ICV/CCV - low		**ICV/CCV - high**	
Sample ID	Measured mg/L (1)	Recovery %	Measured mg/L (1)	Recovery %
Expected Value	0.075	n.a.	0.226	n.a.
ICV	0.078	103	0.210	93
CCV 1	0.078	104	0.220	97
CCV 2	0.079	105	0.224	99
CCV 3	0.080	106	0.224	99
n.a. = not applicable (1) Units are based on per liter of sample at the instrument.				

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery **Lowest calibration std: 0.010 mg/L**		
Expected mg/L (1)	Measured mg/L (1)	Recovery %
0.0099	0.0102	104
(1) Units are based on per liter of sample at the instrument.		

3. Comments

a). The cyanide results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on detection limit studies using water (for the evaluation of the detection limit in liquid matrices) and sand (for the evaluation of the detection limit in solid matrices) and documented in WTP-RDQO-02, *MDL/EQL Evaluation of Cyanide for Sand and Water Matrices (Without EDTA)*. The estimated quantitation limit (EQL) is defined as the lowest calibration standard.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

F07-007

CYANIDE ANALYSIS REPORT

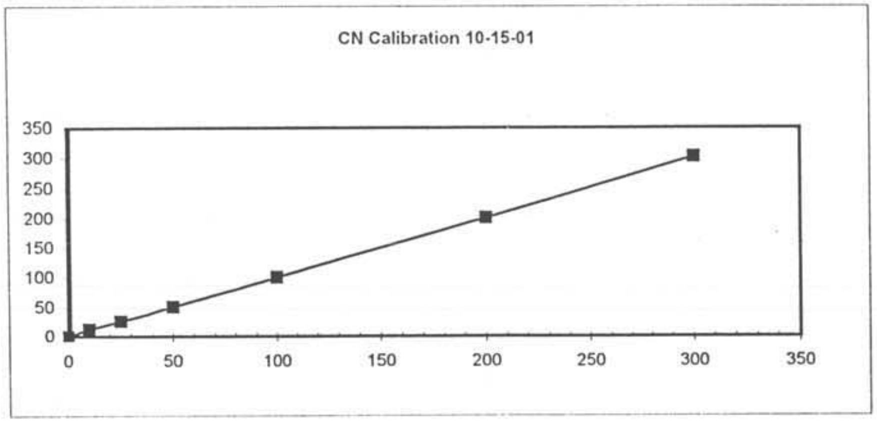
Client	Mike Urie		Sample Tray File:	01101502.RS
WP# / Project #:	K46059 / 42365		Calibration File:	01101502
ASR:	6193		Procedure:	PNL-ALO-287 & -289
Analyst:	LMP Thomas		M&TE:	Lachat QuickChem AE Autoanalyzer (WC36517)
Analysis date:	10/15/01			Mettler AT400 Balance (360-06-01-036, 360-06-01-031)
Report date:	6/5/02			S class Troemner weights (360-86-02-043)

Lab ID	Client ID	Sample Mass (g)	Solids (wt%)	Hot-Cell Dln Factor	Liquid Sample Quantity (g or mL)	Liquid Sample Density (g/mL)	Digest Volume (mL)	Add'l Dln Factor	Abs	Conc CN In digest (ppb)	DL		in Sample dry basis		Expect	Dup/ Rep (RPD)	Recovery (%)
Detection Limit for liquids (ASR 6091)										0.9215				ug/L			
Detection Limit for solids (ASR 6091)										3.678				ug/L			
ICV-low, 75.03ppb				1	1	1	1	1	0.0441	77.58	0.921		77.58	ug/L	75.03		103%
ICV-high, 225.94 ppb				1	1	1	1	1	0.1196	209.6	0.921		209.6	ug/L	225.9		93%
ICB, 0.25 M NaOH				1	1	1	1	1	0.0000	0.4418	0.921	<	0.921	ug/L			
Low-level std, 9.878 ppb				1	1	1	1	1	0.0056	10.24	0.921		10.24	ug/L	9.878		104%
PB-solid		0.5572	100	1			6	1	0.0005	1.316	0.040	<	0.040	ug/g			
LCS solid old		0.0504	100	1			6	10	0.0770	135.1	4.379		160.9	ug/g	177	-	91%
LCS solid new		0.0493	100	1			6	10	0.0349	61.49	4.476		74.83	ug/g	201		37%
01-1845	AZ-101-CS-AR	0.5308	100	1			6	1	0.0448	78.80	0.042		0.891	ug/g			
01-1845 DUP	AZ-101-CS-AR	0.5698	100	1			6	1	0.0012	2.541	0.039	<	0.039	ug/g		183%	
01-1845 TRIP	AZ-101-CS-AR	0.5710	100	1			6	1	0.0374	65.86	0.039		0.692	ug/g		37%	
01-1845 MS	AZ-101-CS-AR	0.4810	100	1			6	1	0.0610	107.1	0.046		1.336	ug/g	2.83		27%
01-1845 MSD	AZ-101-CS-AR	0.5506	100	1			6	1	0.0441	77.58	0.040		0.845	ug/g	2.58	159%	3%
CCV-1 low, 75.03ppb				1	1	1	1	1	0.0442	77.75	0.921		77.75	ug/L	75.03		104%
CCV-1 high, 225.94 ppb				1	1	1	1	1	0.1253	219.6	0.921		219.6	ug/L	225.9		
CCB-1, 0.25 M NaOH				1	1	1	1	1	0.0003	0.967	0.921		0.967	ug/L			
PB liquid	AZ-101-SUP-AR			1	0.1236	1.223	6	1	0.0007	1.666	54.71		98.92	ug/L			
LCS liquid				1	4.1208	1.007	6	1	0.1231	215.8	1.352		316.5	ug/L	301.9	105%	105%
01-1844	AZ-101-SUP-AR			1	0.1177	1.223	6	1	0.0138	24.58	57.47		1533	ug/L			
01-1844 DUP	AZ-101-SUP-AR			1	0.1274	1.223	6	1	0.0116	20.73	53.09		1194	ug/L		25%	
01-1844 TRIP	AZ-101-SUP-AR			1	0.1258	1.223	6	1	0.0331	58.34	53.77		3404	ug/L		92%	
01-1844 MS	AZ-101-SUP-AR			1	0.1279	1.223	6	1	0.1027	180.1	52.88		10335	ug/L	11539		87%
01-1844 MSD	AZ-101-SUP-AR			1	0.1195	1.223	6	1	0.1152	201.9	56.60		12404	ug/L	12339	14.2%	101%
CCV-2 low, 75.03ppb				1	1	1	1	1	0.0450	79.15	0.921		79.15	ug/L	75.03		105%
CCV-2 high, 225.94 ppb				1	1	1	1	1	0.1276	223.6	0.921		223.6	ug/L	225.9		99%
CCB-2, 0.25 M NaOH				1	1	1	1	1	0.0000	0.442	0.921	<	0.921	ug/L			
CCV-3 low, 75.03ppb				1	1	1	1	1	0.0453	79.68	0.921		79.68	ug/L	75.03		106%
CCV-3 high, 225.94 ppb				1	1	1	1	1	0.1278	224.0	0.921		224.0	ug/L	225.9		99%
CCB-3, 0.25 M NaOH				1	1	1	1	1	-0.0002	0.092	0.921	<	0.921	ug/L			

CYANIDE ANALYSIS REPORT

Client	Mike Urie
WP# / Project #:	K46059 / 42365
ASR:	6193
Analyst:	LMP Thomas
Analysis date:	10/15/01
Report date:	6/5/02

Sample Tray File:	01101502.RS
Calibration File:	01101502
Procedure:	PNL-ALO-287 & -289
M&TE:	Lachat QuickChem AE Autoanalyzer (WC36517) Mettler AT400 Balance (360-06-01-036, 360-06-01-031) S class Troemner weights (360-86-02-043)


CN Calibration 10-15-01

Calibration Standards: Aldrich (prep 10/08/01)

Calibration Data

target ppb	actual ppb	Abs	calculated ppb	recovery %
0	0.00	0.0000	0.44	NA
10	12.89	0.0074	13.39	104%
25	26.38	0.0151	26.85	102%
50	50.24	0.0285	50.29	100%
100	100.30	0.0568	99.79	99%
200	200.9	0.1128	197.75	98%
300	301.9	0.1736	304.10	101%

Correlation Coefficient	0.99990
SLOPE	0.000571704
INTERCEPT	-0.000252606

Definitions

01-xxxx = sample
01-xxxx DUP = duplicate
01-xxxx TRIP = triplicate
01-xxxx MS = spiked sample

PB = preparation blank
LCS = laboratory control standard

ICV = initial calibration verification
CCV = continuing calibration verification

ICB = initial calibration blank
CCB = continuing calibration blank

CYANIDE ANALYSIS REPORT

Client	Mike Urie
WP# / Project #:	K46059 / 42365
ASR:	6193
Analyst:	LMP Thomas
Analysis date:	10/15/01
Report date:	6/5/02

Sample Tray File:	01101502.RS
Calibration File:	01101502
Procedure:	PNL-ALO-287 & -289
M&TE:	Lachat QuickChem AE Autoanalyzer (WC36517) Mettler AT400 Balance (360-06-01-036, 360-06-01-031) S class Troemner weights (360-86-02-043)

AZ-101 SUP AR

	Back to the Sample							at the instrument					
Sample ID	Measured mg/L	Measured µg/L	PB µg/L	blk corr µg/L	Sa calc µg/L	Sp exp µg/L	Rec %	Meas µg/L	PB µg/L	blk corr µg/L	Sa calc µg/L	Sp exp µg/L	Rec %
AZ-101 SUP AR	1.533	1533	98.92	1434				24.58	1.666	22.91			
AZ-101 SUP AR DUP	1.194	1194		1096				20.73		19.07			
AZ-101 SUP AR TRIP	3.404	3404		3305				58.34		56.67			
Average	2.044	2044		1945									
Variance	1.42	1.4E+06		1.E+06									
Std Dev	1.190	1190		1190									
RSD	58.2%	58.2%		0.612									
MDL	0.0547	54.7						0.92					
Target MDL													
EQL	0.5937	593.7						10					
AZ-101 SUP AR MS	10.33	10335		10236	2044	9495	87%	180.1		178.4	35.61	165.5	87%
AZ-101 SUP AR MSD	12.40	12404		12305	2044	10295	101%	201.9		200.3	33.27	167.6	101%

PrepDF 59.37 v/v

AZ-101 CS AR

	Back to the Sample						at the instrument					
Sample ID	Measured µg/g	PB µg/g	blk corr µg/g	Sa calc µg/g	Sp exp µg/g	Rec %	Meas µg/L	PB µg/L	blk corr µg/L	Sa calc µg/L	Sp exp µg/L	Rec %
AZ-101 CS AR	0.891	0	0.891				78.80	0	78.80			
▨ AZ-101 CS AR DUP	0.039		0.039				2.541		2.541			
AZ-101 CS AR TRIP	0.692		0.692				65.86		65.86			
Average	0.791		0.791									
Variance	0.199		0.199									
Std Dev	0.446		0.446									
RSD	56.3%		56.3%									
MDL	0.040						3.68					
Target MDL												
EQL	0.108						10					
AZ-101 CS AR MS	1.336		1.336	0.791	2.043	26.7%	107.1		107.1	63.45	163.8	26.7%
AZ-101 CS AR MSD	0.845		0.845	0.791	1.796	3.0%	77.58		77.58	72.63	164.8	3.0%

PrepDF 10.77 mls/g

▨ not included in average

F8

MERCURY RESULTS - ASR 6193

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest Radiological Processing Group (RPG)

Inorganic Analysis - Mercury Data Report

Project / WP#:	42365 / K88408
ASR#:	6193
Client:	Mike Urie
Total Samples:	2

RPL #	Client ID
01-1844	AZ-101 SUP AR
01-1845	AZ-101 CS AR

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number:		
	WD30853	CETAC, Mercury Analyzer, Model M-6000A
	360-06-01-029	Mettler AT400 Balance
	360-06-01-048	Mettler AT400 Balance

Digestion Date: 10/10/01, 10/11/01
Analysis Date: 10/11/01

Analysis File: 01101101.DB

Analyst: LMP Thomas

For Calibration and Maintenance Records, see Chemical Measurement Center 98620 RIDS

LMP Thomas 6/5/02
Prepared By

MW Ur 6·5·02
Reviewed By

Two samples, As- Received AZ-101 Supernatant and Centrifuged Solids, were submitted for mercury analysis. The samples were aliquoted in the hot-cell, prepared by digestion, and analyzed by cold vapor atomic absorption spectroscopy (CVAA). The digestion was performed in the hot-cell for the centrifuged solids, and in a fume hood for the supernatant. Preparative and sample analysis quality control included a preparation blank, LCS, sample, duplicate, triplicate, matrix spike and matrix spike duplicate.

1. Analysis

Results from the analysis of the As-received AZ-101 Supernatant sample, are provided in the table below. The concentration is reported in µg of mercury per L of sample.

RPD success criteria: < 20%
RSD success criteria: <15%

RPL ID	Sample ID	Det Limit µg/L	Measured µg/L	RPD %
01-1844	AZ-101 SUP AR	0.14	67	
01-1844	AZ-101 SUP AR DUP	0.11	30	75
01-1844	AZ-101 SUP AR TRIP	0.14	69	3.1
Average(1)			68	
RSD			32	
Est MDL (2)			0.12	
EQL (3)			2.3	
Preparation DF (v/v)			46	
Analysis DF (v/v)			1	

DF - dilution factor

(1) The average is based on the initial and triplicate sample.

(2) The estimated MDL is based on an MDL evaluated for liquid samples (ASR 6145) adjusted by the appropriate dilution factors.

(3) The EQL is based on the lowest calibration standard, 0.05 µg/L, multiplied by the total dilution factor.

Results from the analysis of the As-received AZ-101 Centrifuged Solids sample, are provided in the table below. The concentration is reported in µg of mercury per g of wet centrifuged solids.

RPD success criteria: < 20%
RSD success criteria: <15%

RPL ID	Sample ID	Det Limit µg/g	Measured µg/g	RPD %
01-1845	AZ-101 CS AR	0.0070	2.4	
01-1845	AZ-101 CS AR DUP	0.0075	3.4	36
01-1845	AZ-101 CS AR TRIP	0.0092	3.6	41
Average			3.1	
RSD			21	
Est MDL (1)			0.0078	
EQL (2)			0.051	
Preparation DF (mLs/g)			102	
Analysis DF (v/v)			10	

DF - dilution factor

(1) The estimated MDL is based on an MDL evaluated for solid samples (ASR 6145) adjusted by the appropriate dilution factors.

(2) The EQL is based on the lowest calibration standard, 0.05 µg/L, multiplied by the total dilution factor.

2. Quality Control

Duplicate (DUP).

- AZ-101 Supernatant. The triplicate but not the duplicate meets the RPD success criteria. The RSD success criteria is not met. There was a vigorous reaction during the addition of the nitric acid, the first of the digestion reagents, which may have caused the QC failure of the duplicate, although the oxidizing strength of the digest was maintained.
- AZ-102 Wet Centrifuged Solids. The RPD between the duplicate and triplicate to the initial sample do not meet the RPD success criteria. The RSD success criteria is also not met. However, the RPD between the duplicate and triplicate sample does meet the success criteria. The oxidizing strength of the digestates may not have been maintained during the digestion (which is difficult to determine through the hot-cell windows). This may have caused what appears to be a low recovery of the initial sample. The homogeneity of solid samples may also be a factor in the QC failures.

Matrix Spike (MS).

- AZ-101 Supernatant. The matrix spikes recoveries and the RPD between the matrix spikes meet the QC success criteria.
- AZ-101 Wet Centrifuged Solids. The MS and MSD recoveries and RPD between the matrix spikes do not meet the QC success criteria, perhaps due to the same cause as the QC failures associated with the duplicate and triplicate analyses. If the average of the sample concentrations does not include the initial sample, the MS will just meet the QC criteria with a recovery of 124%, however the MSD still will not (167%). Because the matrix spikes did not meet the QC criteria, a post spike (PS) was performed on the initial sample. The recovery of the PS meets the QC success criteria.

MS success criteria: 75% to 125% of expected value

RPD success criteria: < 20%

RPL ID	Sample ID	Det Lim µg/L	Spike µg/L	Sample µg/L	Measured µg/L	Rec %	RPD %
01-1844	AZ-101 SUP AR MS	2.71	1970	68	2110	104	
	AZ-101 SUP AR MSD	2.71	1970	68	2130	105	1.0

RPL ID	Sample ID	Det Lim mg/kg	Spike mg/kg	Sample mg/kg	Measured mg/kg	Rec %	RPD %
01-1845	AZ-101 CS AR MS	0.042	5.9	3.1	11	131	
	AZ-101 CS AR MSD	0.035	5.0	3.1	12	175	29
	AZ-101 CS AR PS	0.014	4.6	2.4	6.7	93	

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PB and LCS analyses are presented in the table below. The results of the PBs are based on the average sample size. The PBs and LCSs meet the success criteria.

Sample ID	PB success criteria: < EQL		LCS success criteria: 80% - 120% of expected value		
	Success Criteria	Measured	Expected	Measured	Recovery
PB/LCS liquid	< 2.3 µg/L	0.64 µg/L	1601 µg/L	1410 µg/L	88%
PB/LCS solid	< 0.051 µg/g	0.02 µg/g	1.40 µg/g	1.42 µg/g	101%

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: < EQL		
Sample ID	**Criteria µg/L [1]**	**Measured µg/L [1]**
ICB	< 0.05	< 0.05
CCB 1	< 0.05	< 0.05
CCB 2	< 0.05	< 0.05
CCB-3	< 0.05	< 0.05
CCB 4	< 0.05	< 0.05
CCB-5	< 0.05	< 0.05
CCB-6	< 0.05	< 0.05
[1] Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 80% to 120% recovery Calibration Range: 0 - 5 µg/L		
Sample ID	**Measured µg/L[1]**	**Recovery %**
Expected	2.00	
ICV	1.94	97
CCV 1	2.00	100
CCV 2	1.94	97
CCV-3	2.00	100
CCV 4	1.92	96
CCV-5	1.99	100
CCV-6	1.93	97
[1] Units are based on per liter of sample at the instrument.		

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.05 µg/L		
Expected µg/L[1]	**Measured µg/L[1]**	**Recovery %**
0.050	0.049	98
[1] Units are based on per liter of sample at the instrument.		

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on detection limit studies using water (for the determination of the detection limit in liquid matrices) and sand (for the determination of the detection limit in solid matrices) and documented in WTP-RDQO-16, *MDL/EQL Evaluation of Mercury for Sand and Water Matrices*. The estimated quantitation limit (EQL) is defined as the lowest calibration standard.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

MERCURY ANALYSIS DATA CALCULATION REPORT

Client: Mike Urie
WP# / Project #: K88408 / 42365
ASR / RPL Log-in # ASR 6193 / 01-(01844-01845)

Data File: 01101101.DB
Date Digested: 10/10/01, 10/11/01
Date Analyzed: 10/11/01
M&TE: CETAC Technologies, Mercury Analyzer, Model M-6000A (WD30853)
Mettler AT400 Balance (360-06-01-048)

EMP2 6/5/02

Lab ID	Client ID	Solid Sample		Liquid Sample		Digest	Hot-Cell	Analytic	Absorbance							Hg Concentration						RPD	Rec
		Mass g	wet wt%	Quantity g or mL	Density g/mL	Volume mL	DF	DF	Read 1 µAbs	Read 2 µAbs	Read 3 µAbs	Read 4 µAbs	Ave µAbs	Std Dev	RSD %	in digest ppb	In Sample DL		dry basis		Expect	%	%
Detection Limit (liquids) for Mercury Analysis																0.0027				ug/L			
Detection Limit (solids) for Mercury Analysis																0.0077				ug/L			
ICV, 2 ppb				1	1	1	1	1	18414	18473	18500	18514	18475	20.1	0.11%	1.942	0.0027		1.942	ug/L	2.00		97%
ICB, 7% HCl				1	1	1	1	1	1	-7	-8	-13	-7	2.78	-41.9%	-0.0034	0.0027	<	0.0027	ug/L			
Low-level std, 0.05 ppb				1	1	1	1	1	490	493	490	492	491	1.53	0.31%	0.0490	0.0027		0.0490	ug/L	0.050		98%
CCV 1, 2 ppb				1	1	1	1	1	19013	19063	19094	19116	19071	24.0	0.13%	2.005	0.0027		2.005	ug/L	2.00		100%
CCB 1, 7% HCl				1	1	1	1	1	-4	-9	-21	-24	-14	6.78	-47.4%	-0.0042	0.0027	<	0.0027	ug/L			
PB - liquid				0.6531	1.223	25	1	1	158	158	157	149	155	4.09	2.64%	0.01364	0.1270		0.6385	ug/L			
LCS liquid				0.4944	1.007	25	1	10	26318	26347	26348	26339	26338	5.27	0.02%	2.769	1.382		1410	ug/L	1601		88.1%
01-1844	AZ-101 SUP AR			0.6055	1.223	25	1	1	12529	12538	12547	12584	12550	20.1	0.16%	1.318	0.1370		66.58	ug/L			
01-1844	AZ-101 SUP AR DUP			0.7735	1.223	25	1	1	7328	7306	7320	7362	7329	23.6	0.32%	0.7687	0.1073		30.39	ug/L		74.6%	
01-1844	AZ-101 SUP AR TRIP			0.6126	1.223	25	1	1	13041	13070	13095	13152	13089	35.0	0.27%	1.375	0.1355		68.64	ug/L		3.1%	
01-1844	AZ-101 SUP AR MS			0.6130	1.223	25	1	20	20157	20115	20076	20051	20100	27.8	0.14%	2.113	2.707		2108	ug/L	2038		104%
01-1844	AZ-101 SUP AR MSD			0.6134	1.223	25	1	20	20350	20313	20272	20231	20292	34.8	0.17%	2.133	2.705		2127	ug/L	2037	0.98%	105%
CCV 2, 2 ppb				1	1	1	1	1	18532	18511	18485	18459	18497	21.8	0.12%	1.944	0.0027		1.944	ug/L	2.00		97%
PB - solid		0.2458	100%			25	1	1	-786	-801	-805	-817	-802	7.28	-0.91%	-0.0871	0.00078	<	0.0008	ug/g			
LCS - solid		0.3004	100%			25	1	10	15487	15492	15486	15487	15488	2.47	0.02%	1.627	0.00638		1.354	ug/g	1.400		96.7%
01-1845	AZ-101 CS AR	0.2731	100%			25	1	10	26189	26110	26011	25908	26055	85.3	0.33%	2.739	0.00702		2.508	ug/g			
01-1845	AZ-101 CS AR DUP	0.2568	100%			25	1	10	34537	34476	34388	34300	34425	74.3	0.22%	3.620	0.00746		3.525	ug/g		33.7%	
01-1845	AZ-101 CS AR TRIP	0.2075	100%			25	1	10	30014	30022	30003	30003	30011	9.09	0.03%	3.156	0.00923		3.802	ug/g		41.0%	
01-1845	AZ-101 CS AR MS	0.1841	100%			25	1	40	18955	19021	19070	19124	19043	44.3	0.23%	2.001	0.04163		10.87	ug/g	9.222		127.8%
CCV 3, 2 ppb				1	1	1	1	1	18886	18968	19016	19063	18983	42.1	0.22%	1.995	0.0027		1.995	ug/L	2.00		100%
CCB 3, 7% HCl				1	1	1	1	1	12	5	0	-4	3	3.82	126%	-0.0024	0.0027	<	0.0027	ug/L			
PB - solid		0.2458	100%			25	1	1	220	221	215	207	216	5.58	2.59%	0.0200	0.00078		0.0020	ug/g			
LCS - solid		0.3004	100%			25	1	10	16163	16214	16245	16275	16224	27.1	0.17%	1.705	0.00638		1.419	ug/g	1.400		101%
01-1845	AZ-101 CS AR	0.2731	100%			25	1	10	24885	24862	24828	24793	24842	29.1	0.12%	2.612	0.00702		2.391	ug/g			
01-1845	AZ-101 CS AR DUP	0.2568	100%			25	1	10	33398	33456	33498	33552	33476	41.2	0.12%	3.521	0.00746		3.427	ug/g		35.6%	
01-1845	AZ-101 CS AR TRIP	0.2075	100%			25	1	10	28557	28611	28647	28658	28618	22.5	0.08%	3.009	0.00923		3.626	ug/g		41.0%	
CCV 4, 2 ppb				1	1	1	1	1	18149	18235	18289	18333	18252	43.5	0.24%	1.918	0.0027		1.918	ug/L	2.00		96%
CCB 4, 7% HCl				1	1	1	1	1	-40	-44	-53	-54	-48	4.74	-10.0%	-0.0077	0.0027	<	0.0027	ug/L			
01-1845	AZ-101 CS AR MS	0.1841	100%			25	1	40	19062	19119	19157	19196	19134	33.5	0.18%	2.011	0.04163		10.92	ug/g	9.092		130.8%
01-1845	AZ-101 CS AR MSD	0.2164	100%			25	1	40	24350	24444	24500	24565	24465	53.2	0.22%	2.572	0.03542		11.89	ug/g	8.147	28.8%	174.8%
CCV 5, 2 ppb				1	1	1	1	1	18834	18925	18980	19024	18941	44.3	0.23%	1.991	0.0027		1.991	ug/L	2.00		100%
CCB 5, 7% HCl				1	1	1	1	1	10	7	-4	-11	0	7.42	2015%	-0.0027	0.0027	<	0.0027	ug/L			
01-1844	AZ-101 SUP AR PS			0.6055	1.223	25	1	1.05	16467	16531	16543	16560	16525	15.3	0.09%	1.737	0.1443		92.33	ug/L	91.6		100%
01-1845	AZ-101 CS AR PS	0.2731	100%			25	1	20	34338	34520	34632	34722	34553	89.8	0.26%	3.634	0.0140		6.653	ug/g	6.968		93.1%
CCV 6 , 2 ppb				1	1	1	1	1	18254	18369	18447	18521	18398	66.5	0.36%	1.934	0.0027		1.934	ug/L	2.00		96.7%
CCB 6, 7% HCl				1	1	1	1	1	-36	-48	-56	-62	-50	6.40	-12.7%	-0.0080	0.0027	<	0.0027	ug/L			
01-1844	AZ-101 SUP AR PS			0.6055	1.223	25	1	1.05	16510	16509	16514	16555	16522	21.0	0.13%	1.736	0.1443		92.31	ug/L	91.6		100%
CCV 7 , 2 ppb				1	1	1	1	1	18176	18273	18343	18409	18300	59.1	0.32%	1.923	0.0027		1.923	ug/L	2.00		96.2%
CCB 7, 7% HCl				1	1	1	1	1	-26	-34	-39	-46	-37	5.21	-14.3%	-0.0065	0.0027	<	0.0027	ug/L			

▨ not reported

F08-006

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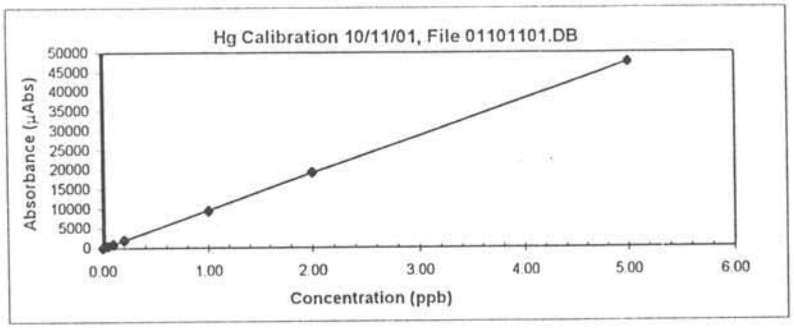
MERCURY ANALYSIS DATA CALCULATION REPORT

Client: Mike Urie
WP# / Project #: K88408 / 42365
ASR / RPL Log-in # ASR 6193 / 01-(01844-01845)

Data File: 01101101.DB
Date Digested: 10/10/01, 10/11/01
Date Analyzed: 10/11/01
M&TE: CETAC Technologies, Mercury Analyzer, Model M-6000A (WD30853)
Mettler AT400 Balance (360-06-01-048)



Calibration Data (Standards: Inorganic Ventures, prep 10/11/01

Std	Absorbance					Calc	Recovery	Std Dev	RSD
ppb	read 1	read 2	read 3	read 4	Ave	ppb	%		%
0.00	50.13	51.32	53.25	54.95	52.4	0.003	NA	2.12	4.1%
0.05	478.2	478.1	479.9	483.3	480	0.0478	95.6%	2.45	0.51%
0.10	947.3	946	955.5	954.6	951	0.097	97.4%	4.90	0.52%
0.20	1930	1934	1938	1936	1935	0.201	100.5%	3.63	0.19%
1.00	9384	9415	9431	9447	9419	0.99	99%	26.69	0.28%
2.00	19117	19177	19218	19243	19188	2.02	101%	55.1	0.29%
5.00	47311	47463	47556	47632	47490	5.00	100%	138.1	0.29%

Correlation Coefficient	0.99999
SLOPE	9501.37
INTERCEPT	25.625

AZ-101 SUP AR

Sample ID	Back to the Sample						
	Measured mg/L	Measured µg/L	PB µg/L	blk corr µg/L	Sa calc µg/L	Sp exp µg/L	Rec %
AZ-101 SUP AR	0.06658	66.58	0.6385	65.94			
AZ-101 SUP AR DUP	0.03039	30.39		29.75			
AZ-101 SUP AR TRIP	0.06864	68.64		68.00			
Average	0.06761	67.61					
Variance	2.1E-06	462.75					
Std Dev	1.46E-03	21.512					
RSD	2.16%	31.82%					
MDL	1.25E-04	0.1250					
Target MDL	9E-03	9.21					
EQL	2.30E-03	2.30					
AZ-101 SUP AR MS	2.11E+00	2108			67.61	1970	104%
AZ-101 SUP AR MSD	2.13E+00	2127			67.61	1969	105%

at the instrument					
Meas µg/L	PB µg/L	blk cor µg/L	Sa calc µg/L	Sp exp µg/L	Rec %
1.318	0.014	1.304			
0.769		0.755			
1.375		1.361			
0.00271					
0.20					
0.05					
2.11			0.068	1.975	104%
2.13			0.068	1.975	105%

Prep DF = 46.0685

AZ-101 CS AR

Sample ID	Back to the Sample					
	Measured µg/g	PB µg/g	blk corr µg/g	Sa calc µg/g	Sp exp µg/g	Rec %
AZ-101 CS AR	2.391					
AZ-101 CS AR DUP	3.427					
AZ-101 CS AR TRIP	3.626					
Average	3.148					
Variance	43.97%					
Std Dev	66.31%					
RSD	21.06%					
MDL	0.00780					
Target MDL	0.20342					
EQL	0.05085					
AZ-101 CS AR MS	10.92			3.148	5.944	131%
AZ-101 CS AR MSD	11.89			3.148	4.999	175%
AZ-101 CS AR PS	6.653			2.391	4.577	93.1%

at the instrument					
Meas µg/L	PB µg/L	blk cor µg/L	Sa calc µg/L	Sp exp µg/L	Rec %
2.612					
3.521					
3.009					
0.00766					
0.2					
0.05					
2.011			0.580	1.094	131%
2.572			0.681	1.082	175%
3.634			1.306	2.500	93.1%

Prep DF = 101.7

▨ not included in the average

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F08-007

F9

HYDROXIDE RESULTS - ASR 6193

Battelle
. . . Putting Technology To Work

Date: 11/06/01

Subject: Hydroxide Analyses for: 01-1844
ASR: 6193

Client: Mike Urie

Triplicate analyses of As Received **Supernatant AZ101 (AZ-101 SUP AR)** were analyzed for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto Titrator. A 0.1186 N NaOH -- ChemRec_55 - current hydroxide solution was used as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. The sample and both replicate results runs gave an average OH molarity of 0.67 with a standard deviation of <1%. This result is equivalent to 1.14 E+4 ug/ml, well below the required MRQ value of 7.5E+4 ug/ml. The hydroxide standard recovery was 93% and a sample spike recovered at 94%. No hydroxide was detected in the cell blanks or a reagent blank. The second and third inflection points frequently associated with carbonate and bicarbonate, were detected in the samples at an average of 0.88 molar, with a standard deviation of 3%, and at 0.70 molar, with a standard deviation of 4%, respectively, for the triplicate measurements. All of the results meet the QC acceptance criteria for spike recovery and RSD of triplicate measurements. The titration curves are included with the report.

Prepared By: ______ Date: 11-6-01

Reviewed By: LR Greenwood Date: 11-6-01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # **6193**

WP# **K88426**

Hydroxide and Alkalinity Determination
Procedure: **PNL-ALO-228** Equip # **WB76843**

Analyst: 11/6/01

Reviewer: Greenwood 11/6/01

			Concentration, moles		
RPG #	**Client ID**		**First Point**	**Second Point**	**Third Point**
01-1844	**AZ-101 SUP AR**		**0.67**	**0.86**	**0.68**
01-1844- dup	**AZ-101 SUP AR**	**Duplicate**	**0.67**	**0.87**	**0.71**
01-1844-Trip	**AZ-101 SUP AR**	**Triplicate**	**0.67**	**0.90**	**0.68**
		RPD	**0%**	**3%**	**4%**
		Average (3)	**0.67**	**0.88**	**0.70**
Hydroxide concentration in ug/ml			**1.14E+4**		
Reag. Blk.1			**0**		
Standard 1			**93%**		
MS 01-1844	**Matrix spike**		**94%**		

Note: Results are presented for the first, second, and third inflection points on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second and third points generally represent the carbonate and bicarbonate concentrations.

F09-004

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # **6193**
Client: Mike Urie
WP# K88426

File: R:\radchem\hydroxide\asr 6193
Analysis Date: **11/5/01**
Report Date: **11/6/01**

Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Equip # **WB76843** Lab Loc. 525

Analyst: 11/6/01
Reviewer: 11/6/01

Titrant	Molarity
HCl	**0.2040**
pH 7.0 reading =	6.997

Std. & Spike	Molarity
NaOH	**0.1186**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Diluted Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
01-1844	AZ-101 SUP AR		0.100	0.1231	1.231	3	11.888	0.345	10.744	0.070	0.70	
01-1844	AZ-101 SUP AR		0.200	0.2466	1.233	4	12.416	0.652	11.222	0.133	0.67	
01-1844- dup	AZ-101 SUP AR	Duplicate	0.200	0.2438	1.219	5	12.307	0.658	11.066	0.134	0.67	
01-1844-Trip	AZ-101 SUP AR	Triplicate	0.200	0.2471	1.236	6	12.432	0.659	11.147	0.134	0.67	1.07%
Reag. Blk.1			5.00			1	3.855				**OH % Recovery**	
Standard 1	0.1186 N NaOH		2.500	2.4819	0.993	2	12.475	1.357	10.511	0.2768	**93.4%**	
MS 01-1844	01-1844 + 2mL 0.1N NaOH		0.100	0.1228	1.228	7	12.651	1.433	10.962	0.2923	**94.2%**	MS

Buffer	JT Baker Lot #	CMS#	Expire Date
10	J38505	161304	Sep-02
4	V0510	161306	Jan-03
7	J34512	161305	Aug-02

Performance checks using Balance # 360--01-06-037

Pipet #	Vol.	Wt.	Pipet #	Vol.	Wt.
C301764	2.50	2.4814			
C301764	2.50	2.4986			
C301764	2.50	2.4885			
120737	0.200	0.1995			
120737	0.200	0.1982			
120737	0.200	0.1992			

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6193

WP# K88426

File: R:\radchem\hydroxide\asr

Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: 11/6/01

Reviewer: 11/6/01

Titrant	Molarity
HCl	0.2040
	6.997

RPG #		Sample Vol. (mL	2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
01-1844	0	0.100	0.776	7.526	0.088	0.879		1.087	4.838	0.063	0.63	
01-1844	0	0.200	1.500	8.381	0.173	0.865		2.170	4.970	0.137	0.68	
01-1844- dup	Duplica	0.200	1.507	8.382	0.173	0.866		2.204	4.903	0.142	0.71	
01-1844-Trip	Triplica	0.200	1.537	7.998	0.179	0.896	3.48%	2.205	4.901	0.136	0.68	0.3%
			2nd Equivalence-----			% Recovered		3rd Equivalence		% recovered		
Standard 1		2.500	1.46	7.845	0.02101	7.1%	sample					
MS 01-1844		0.100	1.862	8.17	0.08752	99%	sample	2.330	4.739			

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2041 M HCl is on following page.

F09-005

Chem Rec_72

Prep date: 6/21/01

Preparation and Standardization 0.02 and 0.005 M HCl

WP# K88426 for: RPL-CMC- OH analysis

Prepare 1- liter supply of 1M HCL and 0.2M HCl
Calculation: 1000 mL * 1.0 N HCl / 12M HCl = 83.33 mL of 12 N HCl diluted to 1liter with H20.
0.2 M HCl is a 1 : 5 dilution of 1M HCl
Used 83.5 mL reagent grade conc HCl (Barcode # 58914) and diluted to 1000 mL using nanopure (Type II ASTM grade) water. The 0.02M HCl was prepared by diluting 20 mL of 1M HCl to 1Liter
The 0.005 M HCl was prepared by diluting 5 mL of 1M HCl to 1Liter
The 0.02 M HCl will be titrated against standardized 0.1005M NaOH solution (Chem Rec_64), then used to standardized ~0.01M NaOH and then used to set
50 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalien endpoint using the recently standardized 0.1005 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be <0.3% @ 1s. Thus total error @ 1sigma is combined pipetting and titration error (i.e. Molarity error plus pipeting error ~0.3%)

NaOH Molarity veification --- from Chem Rec -64

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.40071	19.5	0.10062	
2	0.43252	21.1	0.10037	
3	0.41742	20.35	0.10044	
		Ave=	**0.10047**	0.00013
			certified value	0.13%

Hydrochloric Acid Molarity

Titration Id.	aliquot of acid	Vol. of **0.1005M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	50.00	10.10	0.0203	
2	50.00	10.04	0.0202	
3	50.00	10.15	0.0204	
		Ave Molarity =	**0.0203**	0.00011
				0.55%

Expires 6-21-2002

using Hydrochloric Acid set Molarity of more dilute NaOH

Titration Id.	aliquot of acid	Vol. of **0.01M NaOH** to neutralize	**Molarity of NaOH** in Sample	Molarity Error +/- @ 1 s
1	10.00	19.50	0.0104	
2	10.00	19.80	0.0102	
3	20.00	39.20	0.0103	
		Ave Molarity =	**0.0103**	0.00008
				0.78%

Hydrochloric Acid Molarity

Titration Id.	aliquot of acid	Vol. of **0.0103 M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	20.00	9.95	0.0051	
2	40.00	19.65	0.0051	
3	40.00	19.80	0.0051	
		Ave Molarity =	**0.0051**	0.00003
				0.64%

Analyst/Date rg Swoboda ---- 6-21-2001 11/6/01

F10

CHELATOR RESULTS - ASR 6193

Battelle, PNNL / RPG / Organic Analysis … Chelator Data Report (Revision 1)

Project / WP#:	42365 / W60426
ASR#:	6193
Client:	Mike Urie
Total Samples:	1 (in triplicate)

RPL #	Client ID
01-1844	AZ-101 SUP AR

Procedure:	TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products*	
M&TE Number:	Gas Chromatograph/Flame Ionization Detector Mettler PC4400 Balance Mettler AC100 Balance	WD14807 SN: 41100 SN: 821319
Analyst(s):	BR Valenzuela and AM Aman	
Analysis Date:	6/4,11/02	
Analysis Files:	Calibration – Sample Analysis -	060402CH 060402CH 061102CH (reruns)

For Calibration and Maintenance Records, see Calibration Data Packet 060402CH and Instrument Logbook

Blandina Valenzuela 7-15-02
Prepared By

J.A. Campbell 7/15/02
Reviewed By

Corrections Included in Revision 1

1. Correction of the calculated MDL for NIDA (global).
2. Clarification of the presence of IDA in the tank samples (page 3 of 8)
3. Addition of concentration for NIDA lowest calibration standard (page 4 of 8).
4. Correct transcription error for MSD Percent Recovery for EDTA (page 5 of 8).
5. Add found concentration for Succinic Acid found to Prep Blank (page 6 of 8).
6. Clarification of MS/MSD recovery results for EDTA and HEDTA (page 6 of 8).
7. Correction of HEDTA and NIDA results (page 6 of 8).

CHELATOR RESULTS

1. Sample Analysis

As-Received AZ-101 Supernatant

			Sample		Duplicate		Triplicate	
Analyte	CAS #	MDL µg/mL	01-1844 µg/mL	Data Flag	01-1844D µg/mL	Data Flag	01-1844T µg/mL	Data Flag
Chelators and Degradation Products								
EDTA	60-00-4	4.9	4.9	U	4.9	U	4.9	U
HEDTA	150-39-0	8.8	8.8	UX	8.8	UX	8.8	UX
ED3A		4.9	4.9	U	4.9	U	4.9	U
NTA	139-13-9	5.6	5.6	U	5.6	U	5.6	U
NIDA/IDA(b)	142-73-4	11.0	11.0	U	11.0	U	11.0	U
Citric acid(a)	77-92-9	5.8	5.8	U	5.8	U	5.8	U
Succinic acid	110-15-6	6.1	52	J	52	J	47	J
AA (surrogate)			103%(c)		101%(c)		98%(c)	

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids.Value represents percent recovery of the surrogate standard.

(b) IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.

(c) Value represents percent recovery of the surrogate standard.

Succinic Acid Results for AZ-101

RPL ID	Sample ID	MDL µg/mL	Measured µg/mL
01-1844	AZ-101 SUP AR	6.1	52
01-1844	AZ-101 SUP AR DUP	6.1	52
01-1844	AZ-101 SUP AR TRIP	6.1	47
Average			50
Standard Deviation			3
Percent RSD			6
RPD(1)			0
EQL(2)			61
Estimate MDL(3)			6.1
Preparation DF (v/v)			5.13

DF = Dilution Factor

(1) RPD is the difference between the 1st and 2nd replicate.

(2) The estimated EQL is based on the lowest calibration standard, 10 µg/L adjusted by the appropriate dilution factors.

(3) The MDL is based on the EQL divided by a factor of 10.

Narrative

Analysis was performed on the "As-received AZ-101 Supernatant" for chelators and chelator-degradation products. The chelators, ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), ethylenediaminetriacetic acid (ED3A), iminodiacetic acid (IDA), succinic acid, and nitrilotriacetic acid (NTA), have low volatility and high polarity precluding direct analysis by GC/FID. Derivatizing the chelators with a BF_3/methanol mixture results in a methyl ester product that is amenable to GC/FID separation and analysis. The derivatization process and analysis are still considered experimental.

Samples of the "As-received AZ-101 Supernatant," were submitted for chelator analysis. The 5-gram (nominal) sub-samples of the AZ-101 Supernatant were diluted with 5 mL of DI water and subjected to an IX procedure, TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* to reduce the sample dose. The diluted AZ-101 samples were removed from the hot cell and derivitization of the samples according to procedure TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products* was performed in a fume hood in 329. Adipic acid was added to 2-mL aliquots of each sub-sample (prior to derivatization step) as a derivatization monitor. The analysis of these samples included a duplicate and a triplicate. Only succinic acid was present in all three samples. Quality control check standards relative to the sample preparation and instrument performance were also prepared and analyzed.

The EQL was calculated using the lowest calibration standard (10 µg/mL; 20 µg/mL for NIDA) and multiplied by the preparation dilution factor recorded for the batch preparation blank.

Historically, the EQL was 10 times the value of the estimated MDL. Therefore, for the current analysis the MDL will be calculated by dividing the EQL by 10. The results above the MDL and below the EQL will be "J" flagged.

2. Quality Control Criteria

MS and MSD QC Results

Analyte	CAS #	MDL (µg/mL)	02-1832 Average Native Amt. (µg/mL)	Data Flag	Spiked Conc. (MS/MSD) (µg/mL)	Analyzed Conc. (µg/mL)	Matrix Spike (MS) % Rec	Analyzed Conc. (µg/mL)	Matrix Spike Duplicate(MSD) % Rec.
Acceptance Criteria							*75-125*		*75-125*
Chelators									
EDTA	60-00-4	4.9	570		710/630	1000	61	1100	85
HEDTA	150-39-0	8.8	8.8	UX	1400/1300	990	70	1300	103
ED3A(a)	(b)	4.9	310		(c)		(c)		(c)
NTA	139-13-9	5.6	250		890/790	940	78	890	81
NIDA	142-73-4	11.0	880		900(d)/800(d)	2000	121	1900	131
Citric Acid (e)	77-92-9	5.8	420		890/790	1200	92	1100	88
Succinic Acid	110-15-6	6.1	91		900/800	900	90	820	91
AA (surrogate)			99%(f)				99%(f)		106%(f)

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A not spiked into MS and MSD samples.
(d) Assumes spiked IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.
(e) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(f) Value represents percent recovery of the surrogate standard.

Process Blank and LCS Results

Analyte	CAS #	MDL (µg/mL)	Process Blank (µg/mL)	Data Flag	Spiked LCS concentration (µg/mL)	Analyzed LCS concentration (µg/mL)	Data Flag	Lab Control (LCS/BS) % Rec.
Acceptance Criteria								*80-120*
Chelators								
EDTA	60-00-4	4.9	4.9	U	480	500		103
HEDTA	150-39-0	8.8	8.8	UX	970	1700	X	170
ED3A(a)	(b)	4.9	4.9	U	(c)	(c)		(c)
NTA	139-13-9	5.6	5.6	U	600	670		111
NIDA	142-73-4	11.0	11.0	U	(c)	(c)		(c)
Citric Acid (d)	77-92-9	5.8	5.8	U	600	530		88
Succinic Acid	110-15-6	6.1	18.5	J	620	600		97
AA (surrogate)			73%(e)					62%(e)

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A and IDA/NIDA not spiked into LCS samples.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(e) Value represents percent recovery of the surrogate standard.

Narrative

The preparation blank results meet the success criteria of <EQL.

For the liquid batch chelator preparation, AP-104 Supernatant (RPL#: 02-1832) was used for the matrix spike and duplicate. Of the seven analytes, EDTA and HEDTA results for the MS failed the spiked recovery criterion; however, the MSD result passed. An additional dilution was performed on the MSD for EDTA only; the MSD sample response was above the calibration curve and was diluted into range using a 1:2 dilution. An assumption was made for the recovery results obtained for IDA in the MS and MSD. A known amount of IDA was spiked into the MS and MSD; it was assumed all of the IDA is converted to NIDA in the presence of nitrite found in the tank waste. The MSD percent recoveries on NIDA did not pass acceptance criterion; the opposite is true for the MS. The confirmation samples prepared at a later date (results reported below) did pass both acceptance criteria.

The LCS results passed the recovery acceptance criteria (80-120%) with the exception of HEDTA, which was quite a bit higher at 170% recovery.

Because of the failures observed with the MS/MSD and LCS data, the QC samples were reprepared from the diluted tank sample obtained from 325 Hot Cell. A different analyst performed the derivatization of the samples to confirm or refute original observed results. The reprepared data confirmed the trends observed in the original results; with the MS EDTA producing a low recovery result, and the LCS HEDTA percent recovery results were also confirmed as being higher than the acceptance criteria of 80-120%. One difference between the original and the confirmation results is the HEDTA recovery result for the MS and MSD. Originally, the recoveries for the MS/MSD were 70% and 103% respectively; the confirmation results produced slightly higher results with 135% (MS) and 155% (MSD). The reprepared sample results are shown in the tables below.

MS and MSD Confirmation Results (Reprepared Samples)

Analyte	CAS #	MDL (µg/mL)	02-1832 Native Amt. (µg/mL)	Data Flag	Spiked Conc. (MS/MSD) (µg/mL)	Analyzed Conc. (µg/mL)	Matrix Spike (MS) % Rec	Analyzed Conc. (µg/mL)	Matrix Spike Duplicate(MSD) % Rec.
Acceptance Criteria							*75-125*		*75-125*
Chelators									
EDTA	60-00-4	4.9	630		710/630	1100	65	1100	74
HEDTA	150-39-0	8.8	8.8	UX	1400/1300	1900	135	2000	155
ED3A[a]	(b)	4.9	310		(c)		(c)		(c)
NTA	139-13-9	5.6	240		890/790	950	80	930	89
NIDA	142-73-4	11.0	880		900[d]/800[d]	1900	110	1800	111
Citric Acid [e]	77-92-9	5.8	620		890/790	1300	82	1300	83
Succinic Acid	110-15-6	6.1	91		900/800	940	94	860	96
AA (surrogate)			107%[f]				107%[f]		110%[f]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A not spiked into MS and MSD samples.
(d) Assumes spiked IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.
(e) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(f) Value represents percent recovery of the surrogate standard.

LCS Confirmation Results (Reprepared Samples)

Analyte	CAS #	MDL (µg/mL)	Spiked LCS concentration (µg/mL)	Analyzed LCS concentration (µg/mL)	Data Flag	Lab Control (LCS/BS) % Rec.
Acceptance Criteria						*80-120*
Chelators						
EDTA	60-00-4	4.9	480	540		111
HEDTA	150-39-0	8.8	970	2200	X	221
ED3A(a)	(b)	4.9	(c)	(c)		(c)
NTA	139-13-9	5.6	600	720		118
NIDA	142-73-4	11.0	(c)	(c)		(c)
Citric Acid (d)	77-92-9	5.8	600	560		93
Succinic Acid	110-15-6	6.1	620	640		104
AA (surrogate)						118%(e)

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A and IDA/NIDA not spiked into LCS samples.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(e) Value represents percent recovery of the surrogate standard.

3. Recommendations

Additional work is required to provide a reliable, robust technique for the analysis of chelators in tank waste. A more vigorous technique for the analysis of chelators by direct injection is favorable; preliminary results from capillary electrophoresis appear to produce promising results.

F11

ORGANIC ACID RESULTS - ASR 6193

Battelle, PNNL / RPG / Organic Anions by Ion Chromatography

Project / WP#:	42365 / W60426
ASR#:	6193
Client:	Mike Urie
Total Samples:	1

RPL #	Client ID
01-1844	AZ-101 SUP AR

(Samples prepared in triplicate)

Procedure: **Adapted from test plan TP-RPP-WTP-024 MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste. (See narrative for adaptation descriptions).**

M&TE Number:		
	WD12888	Dionex IC Analyzer System
	P37596	Mettler AE50 Balance

Analyst: GM Mong

Analysis Date: 6/5/02

Analysis Files:
Data: gm0605E01 and gm0611E1
Spreadsheets: IC Std 14062-44a asr6193
IC Std 14062-44b asr6193

Gary Mong 6/28/02
Prepared By

Blandina Valenzuela 6-28-02
Reviewed By

Sample 01-01844 was sub-sampled in triplicate for organic anion analysis. The samples were prepared by dilution with 5 mL of DI water and eluted through a bed of Bio-Rad AG 50W-X8 cation exchange resin (50-100 mesh, sodium form) to relieve the samples of some of their fission product activity. Ion chromatography was done using two separate analytical columns which have different loading characteristics; a Dionex AS-11 separation was done to determine formate, oxalate, and citrate and a Dionex AS-15 separation was done for determination of glycolate and acetate. Dilutions were chosen for the ion-exchange eluant solution (IEX solution) which were demonstrated to not exceed the separation capacity for the columns. The dilutions for the IEX solutions were 400 µl/ 25 mL for the AS-15 separation and 200 µl/ 25 mL for the AS-11 separation. The analytical dilutions are sample dependent and are a principle effect on the overall method MDL estimation.

The results are corrected for sample density (1.22 g/mL). The IEX solution was found to have a density of 1.004 g/mL; results are not corrected for this density. All other dilutions are done on a v/v basis.

This work utilizes the QC acceptance criteria developed in test plan TP-RPP-WTP-024 MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste. The analyte list for this work differs substantially from that developed for the test plan; consequently, the analytical method has to be adapted to meet these new requirements.

One of the requested analytes (gluconate) cannot be reliably determined by the analysis developed here. Two critical analytical hurdles prevent direct analysis for gluconate: (1). Gluconate suffers from low sensitivity to conductivity detection. (2) Gluconate co-elutes with other anions native to tank waste materials in the analytical systems used. For the AS-11 column, gluconate and fluoride are both nearly non-retained and co-elute. For the AS-15 separation, there is great selectivity for the weakly retained analytes fluoride, glycolate, and acetate; however, gluconate was found to co-elute with glycolate. This point will be addressed in the comments below.

1. Analysis

Results for the triplicate analysis are detailed below. Results are in mg/L and are density corrected for the liquid sample; the IEX density was determined to be 1.004 by in laboratory measurement; and is ignored in the calculation. Duplicate injections were averaged for the data reported below. ✓

RPD success criteria: < 20% when > 10X MDL **RSD success criteria: <15% when > 10X MDL**					
Sample	Glycolate*	Acetate	Formate	Oxalate	Citrate
01-01844-1	170U <mdl	140U <mdl	180U <mdl	1100 mg/L	410U <mdl
01-01844-2	170U <mdl	140U <mdl	370 **J** mg/L	960 mg/L	410U <mdl
01-01844-3	170U <mdl	140U <mdl	180U <mdl	1000 mg/L	410U <mdl
MDL (est)	170 mg/L	140 mg/L	180 mg/L	210 mg/L	410 mg/L
EQL (est)	500 mg/L	410 mg/L	550 mg/L	640 mg/L	1200 mg/L
RSD	NA	NA	NA	6.3 % ✓	NA

* In the AS-15 separation glycolate and gluconate co-elute. Separate analysis found that the signal response for gluconate is approximatedly 38% that of glycolate. Thus, the result in this column could be interpreted as glycolate/gluconate; the instrument was calibrated versus glycolate.

Explanation of flags:
U = no peak observed, or determination was below the included MDL level.
J = determination is below the Low level standard, but above the MDL level. Value is estimated from the calibration curve

Acetate and Glycolate were determined by the AS-15 separation. Formate, Oxalate, and Citrate were determined by the AS-11 separation. Oxalate was also quantified using the AS-15 separation ; the values for each sample were determined to be 1070ug/mL, 1050 ug/mL, and 1100ug/mL respectively (these values are presented for comparison purposes for the two separation methods. Oxalate analysis afforded very similar values in each method.).

MDL levels are calculated from the response for the LLS using criteria developed in the DQO project.
In general, the LLS levels represent an area response about at the EQL level; the MDL level is approximately 1/3 of this value. Values reported are back calculated for the dilutions done to the sample.

2. Quality Control

Matrix Spike (MS) and Matrix Spike Duplicate (MSD). These samples cannot be successfully spiked in the matrix so that the included volume of spike is <10% of the sample. Several of the analytes exceed their respective solubilities in water if this exercise is undertaken. Therefore, a spike is added to the IEX solution delivered for analyses. The only QC available is therefore a **Post Spike**. The spike recoveries as well as the RPD between the MS and MSD meet the QC success criteria, with the exception of oxalate, which did not recover well in the MSD sample. Duplicated analyses are done for all samples; a similar result was observed in the repeat of the MSD sample.

Post Spike success criteria: 75% to 125% of expected value

RPD success criteria: < 20%

RPL ID	Analyte/Sample	MDL* mg/L	Spike mg/L	Sample mg/L	Measured mg/L	Rec %	RPD %
01-1844	Glycolate MS-post	6.7	130	0	120	95	
	Glycolate MSD-post		130	0	110	84	12

RPL ID	Analyte/Sample	MDL* mg/L	Spike mg/L	Sample mg/L	Measured mg/L	Rec %	RPD %
01-1844	Acetate MS-post	5.6	50	0	41	82	
	Acetate MSD-post		50	0	43	87	6

RPL ID	Analyte/Sample	MDL* mg/L	Spike mg/L	Sample mg/L	Measured mg/L	Rec %	RPD %
01-1844	Formate MS-post	7.4	35	6.9	41	97	
	Formate MSD-post		35	6.9	36	82	17

RPL ID	Analyte/Sample	MDL* mg/L	Spike mg/L	Sample mg/L	Measured mg/L	Rec %	RPD %
01-1844	Oxalate MS-post	8.6	43	33	75	97	
	Oxalate MSD-post		43	33	65	75	25 ✓

RPL ID	Analyte/Sample	MDL* mg/L	Spike mg/L	Sample mg/L	Measured mg/L	Rec %	RPD %
01-1844	Citrate MS-post	17	80	0	69	86	
	Citrate MSD-post		80	0	66	82	5

*MDL's are derived from the instrument MDL multiplied by the dilution factor applied to the post spike

Preparation Blank (PB) and Laboratory Control Standard (LCS). Two LCS samples were prepared. LCS-1 contained glycolate, acetate, formate, oxalate, citrate, and gluconate. LCS-2 contained glycolate, acetate, formate, oxalate, and citrate only. These samples were constructed to demonstrate the co-elution problems associated with gluconate; the LCS-1 sample contains 8.7 times as much gluconate as glycolate. In the AS-15 analysis gluconate and glycolate co-elute. There is no discernable retention time difference between fluoride and gluconate in the AS-11 analysis. (For 01-01844 samples, there is an apparent fluoride peak found in both analytical schemes.)

The LCS yield data below for glycolate indicates the co-elution of gluconate in LCS-1.

Sample ID	PB success criteria: < MDL		LCS success criteria: 80% - 120% of expected value		
	Glycolate (%) mg/L	Acetate (%) mg/L	Formate (%) mg/L	Oxalate (%) mg/L	Citrate(%) mg/L
PB	<170 U	<140 U	<180 U	<210 U	<410 U
LCS –1 found	**21100 (430%)**	4300 (97%)	4800(104%)	3600(103%)	9900(95%) ✓
LCS-1 expected	4900	4400	4600	3500	10000
LCS –2 found	8000 (109%)	4500 (102%)	5700(107%)	3400 (100%)	12000(107%)
LCS-2 expected	7300	4400	5300	3400	11000

Explanation of flags:
U = no peak observed, or determination was below the included MDL level.

LCS data is reported as concentrations in the hot cell. LCS-2 data is that obtained in the AS-15 analysis. LCS-1 data is assembled from AS-11 and AS-15 data as the analytical data above was reported. The LCS-1 data clearly demonstrates the overlap between gluconate and glycolate

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are water blanks meant to check the analytical system for carryover. The ICB/CCB standards meet the success criteria. (all analytes are below the MDL levels shown below). Since the analysis was done using two column separations, two sets of ICB and CCB data have to be examined.

ICB success criteria: < MDL at the instrument (values in µg/mL)					
	Glycolate	Acetate	Formate	Oxalate	Citrate
ICB/CCB MDL levels	0.11µg/mL	0.090µg/mL	0.060µg/mL	0.069µg/mL	0.13µg/mL

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV runs met the success criteria except for the CCV AS-11 for formate. This CCV is just outside the acceptable criteria; the peak's integration was rechecked. Since the analysis was done using two column separations, two sets of ICV and CCV data have to be examined.

CCVsuccess criteria: 90%-110% recovery values in µg/mL at the instrument **ICV success criteria: 90%-110% recovery values in µg/mL at the instrument**					
Sample	Glycolate	Acetate	Formate	Oxalate	Citrate
ICV-AS11	Not applied*	Not applied*	0.42(93%)	0.52(98%)	1.00(96%)
(expected)			0.45	0.53	1.10
CCV-AS11	Not applied*	Not applied*	**0.98 (112%)**	1.10(104%)	1.90(96%)
(expected)			0.88	1.10	2.00
ICV-AS15	1.50(104%)	1.20 (101%)	0.99(110%)	1.20(110%)	2.20(102%)
(expected)	1.50	1.20	0.90	1.10	2.20
CCV-AS15	1.50(101%)	1.20 (92%)	0.86 (97%)	1.20(108%)	2.10(102%)
(expected)	1.50	1.30	0.88	1.10	2.00

*The values for Glycolate and Acetate cannot by determined due to overlap of these peaks in this separation.

Low-Level Standard (LLS). *The LLS meets the success criteria.* . Since the analysis was done using two column separations, two sets of ICV and CCV data have to be examined. The LLS level approaches the EQL level. By examination of the integrated area of the LLS materials, it is thought the MDL level is approximately 1/3 of the LLS or EQL level. This criteria is applied to all data released by this laboratory.

LLS success criteria: 75% to 125% recovery					
Sample	Glycolate	Acetate	Formate	Oxalate	Citrate
LLS-AS11	Not applied*	Not applied*	0.18(102%)	0.21(96%)	0.40(99%)
(expected)			0.18	0.21	0.40
LLS-AS15	0.32(106%)	0.27(107%)	0.17(95%)	0.19(88%)	0.38(95%)
(expected)	0.30	0.25	0.18	0.21	0.40

*The values for Glycolate and Acetate cannot by determined due to overlap of these peaks in this separation

3. Comments

All analytical values have been rounded to two significant figures.

In general, the application of a secondary analytical column is beneficial to resolving the weakly retained analytes commonly requested for tank waste analysis. This work represents the first successful separation for glycolate/acetate. Unfortunately, the identity of glycolate in the AS-15 separation cannot be ascribed with certainty, since gluconate co-elutes with glycolate. Examination of the LCS data, along with independent verification, reveals that gluconate has a response of ca. 38% that of an equivalent weight of glycolate. Since there is an apparent separation of gluconate from glycolate on the AS-11 column, in principle a determination can be made from the retention differences between the two separations. Unfortunately, gluconate elutes with the very weakly retained analytes (such as fluoride) in the AS-11 routine; which diminishes the confidence that an early eluting peak is actually gluconate. Clearly, there is a need for method development to sensitively and specifically determine gluconate in the presence of the other inorganic/organic anions present in tank waste.

The AS-15 separation used is substantially that recommended for the determination of fluoride by ion chromatography recently developed in our laboratory (Goheen,et. al.).

Use of two column separations allows the comparison of formate and oxalate values between the two methods. Oxalate values for both of these separations agreed within 5%. The AZ-101 supernatants showed minimal evidence of formate; no comparison was possible between the two methods for formate. The AS-15 analysis is not useful for analysis of citrate in tank waste material; citrate elutes very close to the nitrate peak.

These samples contain an apparent fluoride and chromate component; though positive identity would require additional analysis.

F12

RADIOCHEMICAL RESULTS
ASR 6193
(including U by KPA and Ammonia)

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Project No. 42365

Internal Distribution
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Date September 9, 2002

To M. W. Urie

From L. R. Greenwood

Subject Radiochemical Analyses for AZ-101 Supernatant and Centrifuged Solids AZ101 – ASR 6193

Samples of the supernatant and centrifuged solids from tank AZ-101 were analyzed for gamma emitters, ^{90}Sr, total alpha, Pu, Am/Cm, ^{3}H, ^{14}C, ^{99}Tc as pertechnetate, and U according to ASR 6193. Most of the analyses were performed on aliquots of acid digestions of the supernate or fusions of the centrifuged solids samples prepared in the hot cells according to procedures PNL-ALO-128 and PNL-ALO-115, respectively. Uranium in the supernate was measured on a direct sample of the liquid prepared by acid digestion in the laboratory. Tritium was measured on a direct sub-sample of the supernate and on a water leach of the solids according to procedure PNL-ALO-103. ^{99}Tc as pertechnetate was measured on a direct sub-sample of the supernate and ^{14}C was measured on a direct sub-sample of the centrifuged solids. The attached reports list measured analyte activities in units of mCi/L for the supernatant using a measured density of 1.223 g/ml for the hot cell preparations. The solids are reported in mCi/kg. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. The samples were counted for up to 14 hours to give extended time detection limits, as requested. Triplicate results were in good agreement taking into account the relative uncertainties in the measurements. Negligible levels of ^{137}Cs were detected in the process blanks. Due to the very high level of ^{137}Cs in the supernate, we were unable to meet the requested MRQ values for any of the other isotopes and the detection limits are listed on the report. For the centrifuged solids, we were able to detect all of the requested isotopes except for ^{126}Sn, ^{134}Cs, and ^{152}Eu. The detection limits for ^{134}Cs were below the requested MRQ value; however, we were not able to meet the requested MRQ values for ^{126}Sn and ^{152}Eu. ^{125}Sb was seen in one of the triplicate samples; however, it was not seen in the other two samples and the detection limits were at or just below the requested MRQ value.

Total Alpha

The total alpha activity was determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. Alpha activity could not be detected in the supernatant by this method. The detection limits were higher than the requested MRQ value; however, summing the individual alpha emitters provides the best estimate of the total alpha activity, using the results described below. The total alpha activities determined by this method for the solids were significantly higher than the sum of the alpha emitters for reasons that are not understood. Negligible alpha activity was seen in the process and lab blanks and blank spike and matrix spike recoveries were acceptable. The total alpha activities for the centrifuged solids were much higher than the requested MRQ value.

Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and the samples were counted by alpha spectrometry according to PNL-ALO-422. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. Both the plutonium and americium radiochemical yields were acceptable, averaging about 95% for Pu and about 80% for Am/Cm. The LCS and matrix spike recoveries were acceptable at 86-112%. Triplicate results are in good agreement, taking into account the measurement uncertainties. The process blank for the supernatant showed significant contamination with ^{238}Pu, Am, and Cm at levels up to 50% of the activities in the samples. However, these alpha activities are well below the requested MRQ values and most of the total alpha activity is due to ^{239}Pu+^{240}Pu, which does not have an issue with contamination of the hot cell blank. The process blanks for the solids as well as the lab blanks did not have any significant alpha contamination. The sum of the alpha emitters given in the last column of the report is the best indicator of the total alpha content for these samples. All of the requested alpha emitters were detected in the centrifuged solids except for ^{242}Cm in one of the triplicate samples. However, the detection limit was well below the requested MRQ value in this case.

The ^{241}Pu activity in the centrifuged solids samples was determined by performing a liquid scintillation beta count on the precipitation mount that was prepared for the alpha energy analyses, as described above. A standard was prepared by the same procedure to determine the counting efficiency, which is somewhat lower than seen with a direct spike due to the presence of the filter. The LCS recovery was 113% and a matrix spike prepared with another sample in the same batch gave a recovery of 114%. The ^{241}Pu activity in the two hot cell blanks and the lab blank were negligible with respect to the activity in the samples. The triplicate results were in excellent agreement with an RSD of 3%. The sample activities were well above the requested MRQ value.

Since ^{242}Pu was used as a tracer, we could not report it directly from the first alpha measurements. A second Pu separation was performed without using any tracer. The ratio of the counts in the ^{242}Pu peak to that in the $^{239/240}$Pu peak times the $^{239/240}$Pu activity measured in the run using the tracer was then used to determine the absolute ^{242}Pu activity. No peak was observed for 242Pu in the triplicate samples or the two process blanks and the MDA values were calculated from the background on the tail of the $^{239/240}$Pu alpha peak. A ^{242}Pu matrix spike gave a recovery of 98% by this method. No activity was seen in the lab blank.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). The supernates and solids were analyzed in two batches. Negligible levels of ^{90}Sr were found in the process and laboratory preparation blanks for the solids or the supernates. The LCS and matrix spike recoveries were 107-111% for the solids. The matrix spike and LCS recoveries were 95% and 94%, respectively, for the supernates. No ^{137}Cs was detected in the separated Sr fractions. The ^{90}Sr activities were well above the requested MRQ value for the centrifuged solids. However, the activities are below the MRQ value for the supernates. RSD values are acceptable for both samples.

Uranium

Uranium was measured by kinetic phosphorescence analysis (KPA) according to procedure PNL-ALO-4014. The AZ101 Sup AR sample was digested in nitric acid in a hot cell and the AZ101 CS AR sample was fused with KOH in a hot cell. The hot cell preparations were used for uranium analysis by KPA. The hot cell preparation for the centrifuged solids worked well for uranium analysis, but the hot cell prep for the supernate was unusable for uranium analysis, as explained below. Uranium was finally measured in the supernate by starting with raw sample.

Before analysis in the KPA, the hot cell preparations were evaporated dry several times with nitric acid to eliminate organics and halides, then reconstituted in 0.5M nitric acid. The reconstituted solutions, free of organics and halides, were used for KPA uranium measurement.

The CS sample (the solids, prepared for analysis by KOH fusion) ran well by KPA. The triplicate analyses agreed well. One hot cell blank was 1% of the sample concentration, and the other hot cell blank was 0.1% of the sample concentration. The LCS and matrix spike gave excellent analyte recoveries.

The SUP sample preparation from the hot cell (an acid digestion) ran poorly on the KPA and gave unusable data. (The linearity was poor, the lifetimes were short, and the matrix spike failed.) Instead, we had to re-prepare this sample from raw material in the lab and separate the uranium by anion exchange to obtain a solution suitable for KPA uranium measurement. After the anion exchange uranium separation, the samples ran very well by KPA. The RSD between triplicate analyses was 2%. A matrix spike and four uranium standards accompanying the samples through anion exchange gave results between 98% and 103%. The anion exchange blank had negligible uranium relative to the samples.

The anion exchange separation consisted of this:

1. Measure the samples into beakers. Prepare duplicates, spikes, standards, and a blank.
2. Heat the samples with nitric acid to destroy organics, then evaporate the samples dry.

3. Prepare an anion exchange column for each sample, with 1 mL of strong base anion exchanger in concentrated hydrochloric acid. We used AG MP-1, 50-100 mesh, chloride form.
4. Dissolve each sample in concentrated hydrochloric acid and pass it through its column. Discard the effluent. Wash each column with clean hydrochloric acid and discard the effluent.
5. Put clean uranium collection beakers under the columns. Strip the uranium off the columns with 0.5M nitric acid.
6. Evaporate the samples dry and eliminate the chloride ion, then re-constitute them in a known volume of 0.5M nitric acid.
7. Measure the uranium concentrations by KPA as usual.

This anion exchange separation is an abbreviated form of the uranium separation in procedure RPG-CMC-4017. It is very fast, gives quantitative recovery of the uranium, and eliminates all the interferences in KPA uranium measurement.

Tritium

Tritium was distilled from the samples according to procedure PNL-ALO-418 and measured by liquid scintillation counting according to procedure PNL-ALO-474. These samples have very high beta activity and a low concentration of tritium, a combination that has caused trouble in previous tritium analyses using procedure PNL-ALO-418. To avoid such trouble, two procedure modifications were made to eliminate the other beta emitters that would interfere in tritium measurement:

(1) After distillation, a cation exchange column was used to remove any remaining beta emitters, and then

(2) The sample was distilled a second time.

These modifications resulted in very clean beta energy spectra with no trace of any other beta emitters. The hot cell process blanks did not show any significant tritium activities. Both the supernate and centrifuged solids showed excellent reproducibility with RSD values of 1% in both cases. The hot cell matrix spike for the supernate was too small compared to the sample activity so that the result had a very high uncertainty. The hot cell matrix spike for the solids recovered at 90%. No tritium was detected in the laboratory blank. The hot cell reagent spikes recovered at 92% and 91% and the laboratory reagent spike recovered at 89%. Tritium was detected just below the requested MRQ value for the supernatant and well below the MRQ value for the centrifuged solids.

Pertechnetate ^{99}Tc

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated Tc fractions were then counted using a liquid scintillation detector according to procedure RPG-CMC-474. The beta energy spectra did not show any measurable contamination from other beta emitters. The triplicate analyses are in excellent agreement with a RSD of 1%. The level of ^{99}Tc in the lab blank was negligible. The LCS blank spike recovery of a ^{99}Tc standard was 95% and a matrix spike gave a standard recovery of 94%. The measured sample activities were well above the requested MRQ value of 1.50E-3 mCi/L, while the MDA levels were well below the MRQ value.

Carbon-14

Direct samples of the supernate and centrifuged solids from tank AZ-101 were analyzed in triplicate for ^{14}C according to procedure PNL-ALO-482. Following sample combustion, the collected $^{14}CO_2$ was determined by liquid scintillation counting according to procedure PNL-ALO-474. The attached report lists the measured ^{14}C activities in units of mCi/L or kg. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. The ^{14}C spike recovery through the combustion and collection procedure (LCS) averaged 101% for the supernate and 94% for three standards. A matrix spike showed excellent recovery at 112% for the supernate and 100% for the solids. The triplicate analyses showed excellent agreement for the supernate with and RSD value of 4%. However, triplicate results for the solids showed poor agreement with an RSD value of 53%. For both the supernate and the solids, there was no ^{14}C in the lab blanks. However, for the solids, the level of ^{14}C seen in a process blank prepared during the sample aliquoting in the hot cells was about the same as one of the triplicate samples and about 25-30% of the other two sample duplicates suggesting that these samples have significant hot cell contamination. Periodic rinses analyzed with both batches of samples did not show any significant ^{14}C retention by the furnace and gas collection system. For the supernate, the activities in the samples were much higher than the requested MRQ value of 7.2E-4 mCi/L. For the solids, the triplicate ^{14}C measurements were all below the requested MRQ value of 1.83E-3 mCi/kg.

Ammonia

Ammonia was measured in the supernatant sample by ion selective electrode, using procedure RPG-CMC-226. In a hot cell, the raw sample was measured into vials and preserved with dilute sulfuric acid. The sample was prepared in triplicate, with a hot cell blank, a matrix spike, and an LCS. The preserved samples were stirred to allow CO_2 to evaporate, then transferred to clean vials and removed from cell. (The supernatant sample is high in carbonate and evolves CO_2 upon acidification.) In the laboratory, ammonia was measured in the preserved samples by ion selective electrode.

The supernatant sample was diluted as it was preserved, to reduce dose to the analyst. The samples were diluted again in the laboratory at analysis time. The total dilution factor is about 51. Ammonia is detectable in the supernatant sample, but at a low concentration, below the linear range of the ammonia electrode. The results are well below the MRQ. Because the results are so low, they have high analytical uncertainty. The observed relative standard deviation of the triplicate analyses is 28%. The matrix spike recovery was 85%, and the LSC recovery was 95%. The hot cell blank had no detectable ammonia.

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File: 01-1844.xls

Rev. 2
09/09/02

Client : Urie
ASR: 6193

Cognizant Scientist: C Soderquist Date : 9-9-02

Concur : Date : 9-9-02

GEA Procedure/Reference Date: PNL-ALO-450/ Oct. 9, 2001
Sr-90 Procedure/Reference Date: PNL-ALO-476,408/ Oct. 15, 2001 for the solids; Sept. 4, 2002 for the supernates

Measured Activities (mCi/L) with 1-sigma error

Density = 1.223 g/ml

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Sn-126 Error %	Pa-231 Error %
01-1844B Process Blank MDA	<1.E-3	<1.E-4	<4.E-4	<1.E-4	2.25E-3 4% <1.E-4	<5.E-4	<4.E-4	<4.E-4	<4.E-4	<1.E-4	<6.E-3
01-1844 AZ101 SUP AR MDA	4.97E-2 5% <1.E-3	<4.E-2	<4.E+0	<2.E-1	1.66E+3 2% <7.E-1	<2.E-1	<2.E-1	<2.E+0	<2.E+0	<2.E+0	<5.E+1
01-1844 DUP AZ101 SUP AR MDA	4.76E-2 5% <1.E-3	<5.E-2	<4.E+0	<2.E-1	1.64E+3 2% <7.E-1	<2.E-1	<2.E-1	<2.E+0	<2.E+0	<2.E+0	<5.E+1
01-1844 TRIP AZ101 SUP AR MDA	5.30E-2 5% <1.E-3	<5.E-2	<4.E+0	<2.E-1	1.63E+3 2% <7.E-1	<2.E-1	<1.E-1	<2.E+0	<2.E+0	<2.E+0	<5.E+1
RSD	5%				1%						

Measured Activities (mCi/kg) with 1-sigma error

File: 01-1844.xls

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Sn-126 Error %	Pa-231 Error %
01-1845B1	6.80E-2				1.10E-1						
Process Blank	4%				4%						
MDA	<8.E-3	<2.E-3	<7.E-3	<3.E-3	<3.E-3	<8.E-3	<5.E-3	<8.E-3	<1.E-2	<3.E-3	<1.E-1
01-1845B2	8.75E-2				2.39E-1						
Process Blank	4%				4%						
MDA	<1.E-2	<2.E-3	<8.E-3	<2.E-3	<3.E-3	<9.E-3	<6.E-3	<8.E-3	<2.E-2	<3.E-3	<2.E-1
01-1845	2.12E+4	2.06E+0			7.51E+2		2.27E+1	3.27E+1	5.93E+1		
AZ101 CS AR	3%	8%			3%		3%	8%	11%		
MDA	<4.E+2	<3.E-1	<6.E0	<7.E-1	<2.E+0	<2.E+0	<1.E+0	<8.E+0	<2.E+1	<2.E+0	<8.E+1
01-1845DUP	2.03E+4	1.97E+0	8.07E+0		7.64E+2		2.45E+1	3.38E+1	4.27E+1		
AZ101 CS AR	3%	7%	16%		4%		3%	8%	9%		
MDA	<3.E+2	<2.E-1	<4.E+0	<6.E-1	<2.E+0	<8.E-1	<9.E-1	<7.E+0	<9.E+0	<2.E+0	<7.E+1
01-1845 TRIP	1.91E+4	2.06E+0			7.42E+2		2.31E+1	2.52E+1	3.26E+1		
AZ101 CS AR	3%	7%			4%		3%	9%	12%		
MDA	<4.E+2	<3.E-1	<5.E0	<6.E-1	<2.E+0	<1.E+0	<8.E-1	<6.E+0	<9.E+0	<3.E+0	<7.E+1
RSD	5%	3%			1%		4%	15%	30%		
Reagent Spike	111%										
Reagent Spike 1844	94%										
Matrix Spike1844	95%										
Matrix Spike1845	107%										
Lab Blank 1844	<1.E-3										
Lab Blank 1845	<7.E-3										

Note: The process blank 01-1844B was analyzed with the original Sr-90 batch on Oct. 15, 2001.

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File: 01-1844.xls

06/27/02

Client : Urie
ASR: 6193
Cognizant Scientist: LRGreenwood Date : 6/27/02
Concur : T Trang-le Date : 6/27/02

Reference Date: Oct. 8,2001 for Alpha
Reference Date: Oct. 17,2001 for Pu
Reference Date: Oct. 18,2001 for Am

Procedure: PNL-ALO-4001 for Alpha
Procedure: PNL-ALO-417/422 for Am/Pu
Procedure: PNL-ALO-417/422 for Am/Pu

Measured Activities (uCi/ml) with 1-sigma error

Density = 1.223 g/ml ALO ID Client ID	Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Alpha Sum Error %
01-1844B	1.43E-4	9.77E-6	2.89E-5		4.07E-5	1.83E-5		9.77E-5
Process Blank	15%	8%	5%		4%	6%		3%
MDA	<6.E-5	<7.E-7	<6.E-7	<6.E-7	<9.E-7	<7.E-7	<5.E-7	
01-1844		1.76E-3	3.77E-4		1.02E-4	3.46E-5		2.27E-3
AZ101 SUP AR		3%	6%		11%	20%		3%
MDA	<5.E-3	<2.E-5	<2.E-5	<2.E-5	<1.E-5	<1.E-5	<9.E-6	
01-1844 DUP		1.82E-3	3.40E-4		1.43E-4	4.89E-5		2.35E-3
AZ101 SUP AR		3%	6%		10%	17%		3%
MDA	<6.E-3	<7.E-6	<1.E-5	<6.E-6	<1.E-5	<1.E-5	<9.E-6	
01-1844 TRIP		1.83E-3	3.14E-4		6.95E-5			2.22E-3
AZ101 SUP AR		4%	8%		16%			4%
MDA	<5.E-3	<2.E-5	<2.E-5	<1.E-5	<2.E-5	<2.E-5	<1.E-5	
RSD		2%	9%		35%	24%		2%

Measured Activities (uCi/g) with 1-sigma error

	Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Pu-242 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Alpha Sum Error %
01-1845B1	1.41E-3	2.62E-4	7.50E-4			6.41E-4	4.44E-4		2.10E-3
Process Blank	20%	7%	4%			4%	5%		2%
MDA	<8.E-4	<8.E-6	<1.E-5	<8.E-6	<2.E-5	<1.E-5	<8.E-6	<7.E-6	
01-1845B2	6.22E-3	6.00E-4	2.20E-3			1.85E-3	2.58E-3		7.23E-3
Process Blank	9%	5%	3%			3%	3%		2%
MDA	<9.E-4	<9.E-6	<1.E-5	<1.E-5	<2.E-5	<2.E-5	<2.E-5	<1.E-5	
01-1845	5.34E+1	2.48E+0	3.27E-1			3.92E+1	8.38E-2		4.21E+1
AZ101 CS AR	11%	4%	11%			2%	25%		2%
MDA	<1.E+1	<3.E-2	<3.E-2	<4.E-2	<1.E-2	<4.E-2	<4.E-2	<4.E-2	
01-1845DUP	5.35E+1	2.41E+0	2.99E-1			3.56E+1	7.76E-2	3.70E-2	3.84E+1
AZ101 CS AR	8%	4%	10%			2%	23%	33%	2%
MDA	<7.E+0	<3.E-2	<3.E-2	<3.E-2	<6.E-3	<4.E-2	<3.E-2	<3.E-2	
01-1845 TRIP	5.49E+1	2.34E+0	2.93E-1			3.78E+1	1.23E-1	4.11E-2	4.06E+1
AZ101 CS AR	9%	4%	10%			2%	19%	33%	2%
MDA	<1.E+1	<3.E-2	<4.E-2	<3.E-2	<1.E-2	<5.E-2	<3.E-2	<3.E-2	
RSD	2%	3%	6%			5%	26%	7%	
Reagent Spike	104%	102%				91%			
Matrix Spike1844	103%	112%				86%			
Matrix Spike1845	108%	103%			98%	89%			
Lab Blank 1844	<4.E-5	<6.E-7	<1.E-6	<5.E-7		<2.E-6	<6.E-7	<5.E-7	
Lab Blank 1845	<8.E-4	<2.E-5	<2.E-5	<9.E-6	<2.E-5	<3.E-5	<2.E-5	<1.E-5	

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Client : Urie
ASR: 6193

7/1/2002

Cognizant Scientist: C Soderqu Date : 7-1-02

Concur : LRGreenwood Date : 7-1-02

Pu Procedure PNL-ALO-417; Reference Date: 6/20/02

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Pu-241	Error%	MDA
01-1845 B1 Hot cell blank 1	1.40E-2	7%	1E-3
01-1845 B2 Hot cell blank 2	3.77E-2	7%	1E-3
01-1845 AZ101 CS AR	1.20E+1	7%	3E-3
01-1845DUP AZ101 CS AR	1.17E+1	7%	2E-3
01-1845 TRIP AZ101 CS AR	1.12E+1	7%	2E-3
RSD	3%		
Lab Blank 1845	1.56E-3	21%	1E-3
Lab reagent spike	113%		
Matrix Spike 2246	114%		

Page 1 of 1

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 01-1844
02/14/02

Client: Urie
ASR 6193

Cognizant Scientist: C Soderquist 6-12-02
Concur: LRGreenwood 6/12/02

Procedure RPG-CMC-4014 Rev 1, Uranium Analysis by Kinetic Phosphorescence
Chem Chek Instruments model KPA-11R uranium analyzer
Analysis dates are 1/8-18/2002
Sample AZ101 Sup AR was processed by anion exchange to separate uranium before KPA uranium measurement (see narrative).

Uranium Concentration Measured by Kinetic Phosphorescence following Anion Exchange Uranium Separation

Sample	Lab ID	**µg U/ml ± 1s**		Detection Limit
AZ101 SUP AR	01-1844	**5.04E-1**	**± 4%**	4E-4
AZ101 SUP AR Dup	01-1844	**5.18E-1**	**± 4%**	4E-4
AZ101 SUP AR Trip	01-1844	**5.24E-1**	**± 4%**	4E-4
RSD		**2%**		
Matrix spike 01-1844		98%		
Reagent spike 1844-1		103%		
Reagent spike 1844-2		103%		
Reagent spike 1844-3		102%		
Reagent spike 1844-4		100%		
Blank 01-1844		1.6E-03	± 4%	

Uranium Concentration Directly Measured by Kinetic Phosphorescence

		µg U/ml ± 1s		Detection Limit
AZ101 CS AR	01-1845	**3.13E+3**	**± 4%**	2E-1
AZ101 CS AR lab replicate	01-1845 Rep	**3.13E+3**	**± 4%**	2E-1
AZ101 CS AR Dup	01-1845	**3.40E+3**	**± 4%**	2E-1
AZ101 CS AR Trip	01-1845	**2.97E+3**	**± 4%**	2E-1
RSD		**6%**		
Matrix spike 01-1613		95%		
LCS 1845		100%		
Hot cell blank 1845-1		3.79E-1	± 2%	
Hot cell blank 1845-2		2.26E+0	± 2%	
Lab blank 1845		4.06E-03	± 2%	

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Radiochemical Science & Engineering -325 Building

File: 01-1844.xls

Client : Urie
ASR: 6193

03/04/02

Cognizant Scientist: L.R. Greenwood Date : 3/04/02

Concur : C. Soderquist Date : 3-5-02

Tritium Procedures 418/474; Reference Date 2/01/02

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	H-3	Error%	MDA
01-1844B Cell Blank	2.79E-5	23%	2.0E-5
01-1844 AZ101 SUP AR	1.82E-2	4%	1.9E-5
01-1844 DUP AZ101 SUP AR	1.80E-2	4%	1.9E-5
01-1844 TRIP AZ101 SUP AR	1.81E-2	4%	1.9E-5
RSD	1%		
	Measured Activities (uCi/g) with 1-sigma error		
01-1845B Process Blank			1.9E-5
01-1845 AZ101 CS AR	7.19E-3	5%	4.2E-4
01-1845DUP AZ101 CS AR	7.05E-3	5%	4.1E-4
01-1845 TRIP AZ101 CS AR	7.00E-3	5%	4.2E-4
RSD	1%		
Lab Blank			3.6E-6
Lab reagent spike	89%		
Cell reagent Spike 1844	92%		
Cell reagent Spike 1845	91%		
Matrix Spike1844	57%	Spike << sample; high uncertainty	
Matrix Spike1845	90%		

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Radiochemical Science & Engineering -325 Building

File: 01-1844.xls

08/13/02

Client : Urie
ASR: 6193

Cognizant Scientist: C-Soderqu Date : 8-13-02

Concur : LRGreenwood Date : 8/13/02

Procedures PNL-ALO-432/474
Reference Date: August 5, 2002

Reanalysis of previously reported sample

Measured Activities (mCi/L) with 1-sigma error

RPL ID Client ID	Pertechnetate* Tc-99	Error%	MDA
01-1844 AZ101 SUP AR	3.80E-1	2%	6.E-4
01-1844 DUP AZ101 SUP AR	3.74E-1	2%	6.E-4
01-1844 TRIP AZ101 SUP AR	3.76E-1	2%	6.E-4
RSD	1%		
Lab Blank	6.75E-5	4%	6.E-4
Lab reagent spike	95%		
Matrix Spike 01-1844	94%		

*Note: The Tc separation procedure did not include any oxidation steps so that only the fraction of Tc in the pertechnetate form would be separated and counted.

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Radiochemical Science & Engineering -325 Building

File: 01-1844.xls

Client : Urie
ASR: 6193

06/25/02

Cognizant Scientist: LRGreenwood Date : 6/25/02

Concur : T Trang-Le Date : 6/26/02

C-14 Procedure PNL-ALO-482; Reference Date: 6/19/02

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	C-14	Error%	MDA
01-1844 AZ101 SUP AR	1.98E-3	4%	4E-5
01-1844 DUP AZ101 SUP AR	1.95E-3	4%	4E-5
01-1844 TRIP AZ101 SUP AR	1.85E-3	4%	4E-5
RSD	4%		
Lab Blank			2E-5
Lab reagent spike	101%	Average of four LCS spikes	
Matrix Spike1844	112%		

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Radiochemical Science & Engineering -325 Building

File: 01-1844.xls

Client : Urie
ASR: 6193

03/15/02

Cognizant Scientist: LRGreenwood Date : 3/15/02

Concur : MJ Steele Date : 3/19/02

C-14 Procedure PNL-ALO-482; Reference Date: 3/5/02

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	C-14	Error%	MDA
01-1845B Process Blank	4.21E-4	15%	2E-4
01-1845 AZ101 CS AR	4.62E-4	12%	2E-4
01-1845DUP AZ101 CS AR	1.56E-3	6%	2E-4
01-1845 TRIP AZ101 CS AR	1.11E-3	7%	2E-4
RSD	53%		
Lab Blank			2E-4
Lab reagent spike	94%	Average of two LCS spikes	
Matrix Spike1845	100%		

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Radiochemical Science & Engineering -325 Building

6/14/2002
File: 01-1844.xls

Client : Urie
ASR: 6193

Cognizant Scientist: C Soderquist Date : 6-24-02

Concur : LR Greenwood Date : 6-24-02

Procedure RPG-CMC-226, Rev 0, Ammonia Analysis by Ion Selective Electrode

Measured Concentration with 1-sigma error

RPL ID Client ID	Units	NH_3 Concentration	1s %	Sample Dilution	Sample MDL	Sample EQL	MRQ
01-1844 AZ101 SUP AR	µg/mL	3.02E+0	± 27%	51	1.5	13	140
01-1844 DUP AZ101 SUP AR	µg/mL	2.26E+0	± 28%	51	1.5	13	140
01-1844 TRIP AZ101 SUP AR	µg/mL	1.74E+0	± 28%	51	1.5	13	140
RSD		28%					
Hot cell blank 1844	µg/mL	1.18E+0	± 543%	51	1.5	13	140
Matrix spike 01-1844		85%					
LCS 1844		95%					

EQL at instrument is 0.26 µg/mL

MDL at instrument is approximately 0.03 µg/mL

Battelle
. . . Putting Technology To Work

Date: 11/13/01

		Client ID	Sample RPG ID	ASR	6193
Subject:	**Se-79** Analysis Report on:	AZ-101 SUP AR	**01-1844**	WP#	K88422
Project:	42365				

To: Mike Urie

Se-79 was measured in triplicate on As Received Supernatant AZ101 (AZ-101 SUP AR) according to procedure PNL-ALO-440 and the results are listed below. Since Se-79 is not available as a radioactive standard, Se carrier was used in the analysis for establishing the yield and C-14 was used to establish the instrument efficiency since it has a very similar beta max energy (156 Kev vs 149 Kev for Se-79). Direct 2 mL aliquots of the diluted acid digest material provided to the lab were analyzed. The gravimetric recoveries for the reagent blank, lab blank, and triplicate samples, are listed below. The Se-79 activities were measured by liquid scintillation counting according to procedure PNL-ALO-474. Peaks were observed in the Se-79 R.O.I. beta energy spectral plots provided and no other higher energy beta contaminants were observed. A second count was performed to determing reproducablity and this data showed excellent agreement with the first count data. The reagent blank doesn't show any peak in the beta energyt spectrum so the counts above background may not be due to Se-79. Since Se-79 is not available as a standard, it was not possible to provide a LCS or matrix spike. The uncertainties look very low, especially with the high RSD. Are we adding in uncertainties for the grav. yield and standard. The recovery for duplicate sample 1844 was low, however the final result did meet the MRQ value and the result was in good agreement with the other two Se-79 results. The MRQ value for Se-79 was 9.0E-05 mCi/L or 9.0 E-05 uCi/mL as provided by the client.

I.D.	Client ID	Se Recovery	Se-79 Result uCi/mL	Se-79 1s TPU uCi/mL	Se-79 MDC uCi/mL	TPU,%
Reag Blk		0.68	2.84E-5	7.19E-6	2.09E-5	25%
01-1844	AZ-101 SUP AR	0.74	9.76E-4	3.31E-5	1.54E-5	3%
01-1844	AZ-101 SUP AR	0.24	8.01E-4	3.84E-5	4.49E-5	5%
01-1844	AZ-101 SUP AR	0.80	1.13E-3	3.66E-5	1.35E-5	3%
		Ave =	9.69E-4			
		Std. Dev.=	1.65E-4			

Prepared by: Date: 11-13-01

Reviewed by: L.R. Greenwood Date: 11-13-01

Se-79 Analysis --- via RPG-CMC-440 ASR # 6193

This procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences elimination by the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. The Se is finally dissolved and counted by liquid scintillation using C-14 (prep'd in the same matrix configuration) for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV.

Se-79 Procedure Flow Diagram

Add 20 mg elemental Se (2 mL of 10mgSe/mL to each aliquot for analysis

↓

.Prep plastic disposable columns: 1mL AG--50W: X 8 100-200 mesh H+ + 1ml AG-1, X-4 100-200 Cl-

↓

Pass sample through column and collect in 35 mL vial. Wash with 4-1ml portions of 0.5 M HNO3

↓

Add 10ml hydroxlamaine hydrochloride to sample in a glass vial. Cap and place the vial in a heating block and heat @ 90C for ~30 min (I.e. just until black ppt forms).

↓

.Dissove ppt in a few drops of conc HNO3. Add 5 mL of water and repeat the NH2OH.HCl addition and precipitation. Then dissolve in ~ 0.5 M HNO3 for transfer to distillation flask.

Set up still by placing Nitrogen purge ~100ml boiling flask in heating mantle connected to a variable power supply and supported on a ring stand.

↓

Transfer the solution to the boiling flask rinsing the 35mL vial with 2-5mL portions of concentrated HBr.

↓

Place a rinsed (clean) 35ml vial on the exit port of the boiling flask. Add 5ml of 25% hydroxlamaine hydrochloride solution to this vial. Cool this vial in an ice bath.

↓

Connect nitrogen supply to top of flask and set flow to about 5 bubbles/sec. Then turn on power supply and adjust to 110 V amd distill for at least 35 min.

↓

Add 5 ml additional hydroxlamaine hydrochloride solution to the collection vial. Cap and place the vial in a heating block and heat for a couple hours. Black ppt. forms

Prep. and tare- weigh filter disks Centrifuge, decant most of solution and transfer black ppt. using ethanol to the top of filter mounted on vaccum system.

↓

Dry ppt. and obtain final wt. for Se grav. recovery. 20 mg =100% .

↓

Transfer filter + ppt. To glass LCS vial, add 2-3 drops of conc HNO3, allow to dissolve completely, then take to dryness. Add 2 mL 0.1 M HCL and 10 ml of LSC cocktail, place on labeled caps.

↓

Prepare C-14 reference stds. similarly and count all on Packard LSC 2550 for 2 X 30 min. (Prog. # 6).

Analysis raw data:

Sample ID.	Aliqout Vol. (mL)	Diluted to	Tare Wt (mg) Filter + holder	Final Wt. (mg)	Net Se Wt.	Recovery %		
Reag Blk	1.00		746.5	762.8	16.3	81%		
01-1844-B	2.00	Process Blk	748.3	761.8	13.5	68%		
01-1844	2.00		758.4	773.2	14.8	74%		
01-1844 -dup	2.00		774.2	779.1	4.9	24%		
01-1844 -trip	2.00		748.1	764.1	16	80%		

11/13/01

Entered by Date

11/13/01

Reviewed by Date

Pipet verify check	0.1 mL	1.0 mL
Pipet #	78868	33
		1.0021
		0.9948
		0.9979
Ave		0.9983
Std Dev.		0.0037

Se-79 Analysis

WP# **K88422**
ASR # **6193**

Supernatant AZ101 (AZ-101 SUP AR)

Data File: r:\radchem\se79\se-**6193**.xls

Analysis for Se-79 was performed using PNL-ALO-440. This procedure involves an anion/cation exchange to remove of most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences completely eliminated in the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. No vendor supplied Se-79 source material is available, therefore C-14 was chosen for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV.
W-115-1, a secondary dilution of NIST C-14 SRM 4222, was used for the efficiency calibration of the liquid scintillation counter. These calibration standards were prepared in the same geometry as the prepared samples and at the same time the batch was prepared to monitor efficiency of the cocktail over time. Volume of W-115-1 used was 0.1ml.

Process Data: Start Date: 9/4/01
Se Carrier: Selenium Std. @ 10,000 ppm
CMS # 126666 Inor. Ventures Std
Vol. added: Expires: 1-Mar-01
2 mL = 20.0 mg

Performance checks
Balance # 111311964
Pipet # 78868
Lab Loc. 525

Sample ID	Leached Sample Vol (g)	Total dil. volume (g)	dil factor	Diln. Aliquot (mL)	Sample Analyzed (g)	Tare Wt. of filter & holder (mg)	Gross Wt. of filter plu Se ppt.(mg)	Net Wt. of Se (mg)	Wt. of Se added (mg)	Grav. Recovery %
Reag Blk	1.000				1.00	746.5	762.8	16.3	20.00	81.5
01-1844-B	1.000	26.32	26.320	2.00	0.07599	748.3	761.8	13.5	20.00	67.5
01-1844	1.249	26.60	21.295	2.00	0.09392	758.4	773.2	14.8	20.00	74.0
01-1844 -dup	1.248	25.58	20.497	2.00	0.09758	774.2	779.1	4.9	20.00	24.5
01-1844 -trip	1.245	25.10	20.159	2.00	0.09921	748.1	764.1	16.0	20.00	80.0

11/13/01
Entered by Date

LRGreenwood 11/13/01
Reviewed by Date

SELENIUM-79 CALCULATIONS FOR LIQUID SAMPLES

Procedure PNL-ALO-440

Data file name:	r:\radchem\se79\se-6193.xls	Entered by: rgs	
Date Counted	11/08/01	Date Calc'd	11/13/01
Sample Counting Time,min.	100.00	LSC progam # 6	

C-14 is used for efficiency since Beta Emax is very similar to Se-79.

T1/2 = 5715. yr

C-14 Std No.	W-115-1	Activity=	266700	dpm/ml
Ref. date:	12/01/90	error=	4000	dpm/ml

I.D.	aliquot ml	error ml	Se-79 R.O.I. cpm		1S%
Reag. Blank #1	1.000	0.0000	13.31		2.74
Blank 2	1.000	0.0000			
		Avg =	13.31		cpm
					cpm

I.D.	aliquot ml	error ml	Se-79 R.O.I. cpm		1S%	efficiency	efficiency error
100ul Std - C-14 Spk 1	0.100	0.0003	19231.3		0.07	0.722	0.0111
100ul Std -C-14 Spk 3	0.100	0.0003	19331.0		0.07	0.726	0.0111
					Avg efficiency =	0.724	
					1s error =	0.0078	
					%error	1.08	

SAMPLES

Requested activity units: uCi

Sample quantity units: mL

I.D.	Sample Vol. / mass	Sample units g or mL	Se Recovery	aliquot frac. anal.		Se -79 R.O.I. cpm	ct. error 1 sigma %	Se-79 Result uCi/mL	Se-79 1s TPU uCi/mL	Se-79 MDC uCi/mL	TPU,%
Reag BlK	1.000	mL	0.815	1.00		13.27	1289	-3.05E-08	3.93E-07	1.32E-06	1287%
01-1844-B	2.000	mL	0.675	0.07599		15.65	23	2.84E-05	7.19E-06	2.09E-05	25%
01-1844	2.000	mL	0.740	0.09392		122.30	1.1	9.76E-04	3.31E-05	1.54E-05	3%
01-1844 -dup	2.000	mL	0.245	0.09758		44.07	2.5	8.01E-04	3.84E-05	4.49E-05	5%
01-1844 -trip	2.000	mL	0.800	0.09921		157.41	0.9	1.13E-03	3.66E-05	1.35E-05	3%
							Ave	9.69E-04			
							Std dev.	1.65E-04			
							% 1s	17%			

11/13/01

Entered by Date

LRGreenwood 11-13-01

Reviewed by Date

F13

ICP-MS RESULTS – ASR 6193, 6193.01 AND 619.06

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W57932
ASR#:	6193
Client:	Mike Urie
Total Samples:	2

Analysis: Multi-Elemental

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 3 Jul 02

Analysis Files:
Experiment – Several
Procedure – Several
Element Menu – Several

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Reviewed By 3Jul02
Date

Concur 3 Jul 02
Date

F13-002

Samples Submitted for Analysis:

RPL #	Client I.D.
01-1844	AZ 101 SUP AR
01-1845	AZ 101 CS AR

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Quality Control

PNL-ALO-114 Narrative

I-127

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The Matrix Spike (MS) and the Blank Spike (BS) were not recovered due to low spiking levels and high preparation blank I-127 analyte concentrations. The Post Blank Spike (PBS) was inadvertently not spiked with I-127. However the Post Matrix Spike (PMS) was spiked, analyzed and met the success criteria +/- 25 % recovery (90%). The ending CCV was recovered 4% low (86%).

I-129

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. I-129 wash not spiked in the MS or the BS. The PBS was inadvertently not spiked with this analyte. However the PMS was spiked, analyzed and met the success criteria +/- 25 % recovery (89%). The ending CCV was recovered 2% low (88%).

PNL-ALO-115 Narrative

V

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. All batch QC's and instrument QC's met their success criteria for V.

Co

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. Co recovered high (144%) in the LCS/SRM due to high levels present in the preparation blank sample. Co is not a certified element in the LCS/SRM-2710; values are given on for information purposes only. All other batch QC's and instrument QC's met their success criteria.

As

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The MS recovered low (23%) recovery, however a PMS was analyzed and met the success criteria of +/- 25% (103%). All other batch QC's and instrument QC's met their success criteria.

Se

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement (<10XMDL). All batch QC's and instrument QC's met their success criteria for Se. Se is not a listed element in the LCS/SRM.

Rb

Both the BS and the MS were under spiked and NR (No Recovered) for Rb due to a high Prep Blank (PB) for Rb. However a post blank spike and a post matrix spike were analyzed and met the success criteria of +/- 20% for the post blank spike (111%) and +/- 25% for the post matrix spike (102%). All other batch QC's and instrument QC's met their success criteria.

Y

The MS high recovery (129%) for Y in the BS is surprising, the count rate was more then adequate to recovery this batch QC. All other batch QC's and instrument QC's met their success criteria.

Mo

The low recovery value for Mo in the MS is surprising, the count rate was twice the value of the sample matrix and with more then enough response to recovery this batch QC. All other batch QC's and instrument QC's met their success criteria. Mo is not a certified element in the LCS/SRM-2710 and recovered high (178%) due to high levels of Mo in the preparation blank, values are given on for information purposes only.

Tc

The Blank Spike and Matrix Spike were Not Spiked (NS) for Tc. All other batch QC's and instrument QC's met their success criteria for Tc. Tc is not a listed element in the LCS/SRM.

Sb

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. All three Batch QC's BS, MS and LCS/SRM recovered low for Sb for this analysis. This failure appears to be more related to the sample preparation procedure Method PNL-ALO-115 for this analyte. All other batch QC's and instrument QC's met their success criteria.

Te

Both Batch QC samples the BS and MS for Sb recovered low for this analysis. This failure appears to be more related to the sample preparation procedure Method PNL-ALO-115 for this analyte. Te is not a listed element in the LCS/SRM. All other batch QC's and instrument QC's met their success criteria. Te is not a listed element in the LCS/SRM.

Cs

The low recovery value for Cs (53%) in the MS is surprising, in that the count rate was twice the value of the sample matrix and with more then enough response to recovery this batch QC. All other batch QC's and instrument QC's met their success criteria. Cs is not a certified element in the LCS/SRM-2710 and values are given on for information purposes only.

Ce

The MS was recovered low (11%) due to under spiking. All other batch QC's and instrument QC's met their success criteria. Cs-isotopic analysis was performed using an older instrument, which determined only detection of Cs-133 and <MDL results for Cs-135 and Cs-137 in this sample. Cs is not a certified element in the LCS/SRM.

Pr

The high recovery value for Pr in the BS (135%) and MS (163%) is surprising in that the count rate was more then enough to recovery these batch QC samples. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (105%) and +/- 25% recovery for the matrix spike recovery (99%) recovery. Pr is not a listed element in the LCS/SRM. All instruments QC solutions have met their success criteria.

AMU-151

The response for 151amu was determined using the response of Eu -151. The BS and MS was not spike with either Sm-151 or Eu, therefore no recovery for the BS and MS batch QC solutions was expected. All batch QC's and instrument QC's met their success criteria. Eu is not a certified element in the LCS/SRM.

Ta

Both the BS and the MS were recovered low for Ta. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (101%) and +/- 25% recovery for the matrix spike recovery (97%) recovery. This failure appears to be more related to the sample preparation procedure Method PNL-ALO-115 for this analyte. All instruments QC solutions have met their success criteria. Ta is not a listed element in the LCS/SRM.

W

The low recovery value for W (54%) in the MS is surprising, the count rate was at least 3 times the value of the sample matrix and with more then enough response to recovery this batch QC. A matrix spike solution was prepared and analyzed and met the success criteria of +/- 25% recovery for the post matrix spike solution (101%). All other batch QC's and instrument QC's met their success criteria. W is not a certified element in the LCS/SRM-2710.

Tl

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. All batch QC's and instrument QC's met their success criteria.

Th

Both the BS and MS were recovered low due to under spiking the samples, the low recovery for Th in the BS is surprising, the count rate was more then enough to recovery this batch QC sample. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (107%) and +/- 25% recovery for the matrix spike recovery (110%) recovery. All instruments QC solutions have met their success criteria.Th is not a certified element in the LCS/SRM.

NP-237

The Blank Spike and Matrix Spike were not spiked for Np. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (106%) and +/- 25% recovery for the matrix spike recovery (111%) recovery. All instruments QC solutions have met their success criteria. Np is not a listed element in the LCS/SRM.

Pu-239

The Blank Spike and Matrix Spike were not spiked for Pu-239. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (106%) and +/- 25% recovery for the matrix spike recovery (111%) recovery. All instruments QC solutions have met their success criteria. Pu-239 is not a listed element in the LCS/SRM.

Pu-240

The Blank Spike and Matrix Spike were not spiked for Pu-239. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (110%) and +/- 25% recovery for the matrix spike recovery (118%) recovery. All instruments QC solutions have met their success criteria. Pu-240 is not a listed element in the LCS/SRM.

U

The MS was not recovered due to being under spiked, however a post matrix spike was analyzed and met the success criteria of +/- 25 % recovery (106%). The BS was recovered high (124%) due to low analyte response and elevated preparation blank levels, however a post blank spike was analyzed and met the success criteria of +/-20% recovery (102%). All other batch QC's and instrument QC's met their success criteria. The analysis of U-233, U-234, U-235 and U-236 were preformed using the U-238 standard calibration, and QC responses.

PNL-ALO-116 Narrative

Pt

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. All batch QC's and instrument QC's met their success criteria for Pt. There is no assigned value for Pt in the LCS/SRM-2710.

Pd

The serial dilution met the success criteria of +/- 10%. The BS met the success criteria of +/- 20% recovery. The MS was not recovered due to under spiking of the analyte, however a post matrix spike was analyzed and meet the success criteria of +/- 25% recovery (104%). All other instrument QC's also met their success criteria. Pd is not a listed element in the LCS/SRM.

Rh

The serial dilution met the success criteria of +/- 10%. The MS was not recovered due to under spiking of the analyte, however a post matrix spike was analyzed and meet the success criteria of +/- 25% recovery (95%). The BS met the success criteria of +/- 20% recovery. Rh is not a listed element in the LCS/SRM.

Ru

The serial dilution met the success criteria of +/- 10%. The MS was not recovered due to under spiking of the analyte, however a post matrix spike was analyzed and meet the success criteria of +/- 25% recovery (106%). The BS met the success criteria of +/- 20% recovery. Rh is not a listed element in the LCS/SRM.

Rb

All batch QC's and instrument QC's met their success criteria.

Cs

All batch QC's and instrument QC's met their success criteria. The analysis of Cs-isotopics was preformed using High Pressure Ion Chromatography on-line to the ICP/MS.

Tc

The Blank Spike and Matrix Spike were not spiked for Tc-99. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (105%) and +/- 25% recovery for the matrix spike recovery (107%) recovery. All instruments QC solutions met their success criteria.

I-127

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The MS were under spiked and not recovered for I-127 however a post matrix spike was analyzed and met the success criteria of +/- 25% for the post matrix spike (99%). All instruments QC solutions have met their success criteria.

I-129

The Blank Spike and Matrix Spike were not spiked for I-129. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (96%) and +/- 25% recovery for the matrix spike recovery (84%) recovery. All instruments QC solutions met their success criteria.

NP-237

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The Blank Spike and Matrix Spike were not spiked for Np-237. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (101%) and +/- 25% recovery for the matrix spike recovery (101%) recovery. All instruments QC solutions have met their success criteria.

Pu-239

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The Blank Spike and Matrix Spike were not spiked for Pu-239. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (102%) and +/- 25% recovery for the matrix spike recovery (99%) recovery. All instruments QC solutions have met their success criteria.

Pu-240

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The Blank Spike and Matrix Spike were not spiked for Pu-240. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (102%) and +/- 25% recovery for the matrix spike recovery (99%) recovery. All instruments QC solutions have met their success criteria.

AMU-241

The serial dilution sample was not high enough in concentration after the dilution (5x) to meet this requirement. The Blank Spike and Matrix Spike were not spiked for amu-241. A post blank spike and matrix spike solution was prepared and analyzed and met the success criteria of +/- 20% recovery for the post blank spike (92%) and +/- 25% recovery for the matrix spike recovery (91%) recovery. All instruments QC solutions have met their success criteria.

U

All batch QC's and instrument QC's met their success criteria. The analysis of U-233, U-234, U-235 and U-236 were preformed using the U-238 standard calibration, and QC responses.

				01-1845-B1-Ni					01-1845-B2-Ni				
				MDL	Proc. Blank-1	Uncert.	Detection	DF	MDL	Proc. Blank-2	Uncert.	Detection	DF
1-Jul-02	ICP-MS	Fusion-115	129I	4.95E-03	3.57E-01	3.13E-02		12955	4.95E-03	2.52E-01	1.47E-02		12955
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
27-Jun-02	ICP-MS	Fusion-115	51V	2.66E-02	6.97E+00	6.23E-02		91973	2.66E-02	6.17E+00	4.28E-02		91973
27-Jun-02	ICP-MS	Fusion-115	59Co	6.01E-03	1.14E+01	1.20E-01		91973	6.01E-03	9.76E+00	1.09E-01		91973
27-Jun-02	ICP-MS	Fusion-115	75As	1.17E-01			<	91973	1.17E-01			<	91973
27-Jun-02	ICP-MS	Fusion-115	82Se	1.17E+00			<	91973	1.17E+00	2.27E+00	2.04E+00		91973
27-Jun-02	ICP-MS	Fusion-115	85Rb	2.36E-02	2.87E+02	1.86E+00		91973	2.36E-02	2.71E+02	1.99E+00		91973
27-Jun-02	ICP-MS	Fusion-115	89Y	8.56E-03	3.88E-01	2.44E-03		91973	8.56E-03	2.25E-02	1.60E-03		91973
27-Jun-02	ICP-MS	Fusion-115	100Mo	2.80E-02	9.48E+00	1.30E-01		91973	2.80E-02	4.00E+00	4.02E-02		91973
27-Jun-02	ICP-MS	Fusion-115	99Tc	1.60E-03	2.13E-01	8.11E-03		91973	1.60E-03	1.78E-01	6.75E-03		91973
27-Jun-02	ICP-MS	Fusion-115	121Sb	5.57E-03	1.77E-01	5.59E-03		91973	5.57E-03	1.56E-01	9.63E-03		91973
27-Jun-02	ICP-MS	Fusion-115	130Te	2.39E-01	7.18E-01	4.78E-01		91973	2.39E-01	4.06E-01	3.53E-01		91973
27-Jun-02	ICP-MS	Fusion-115	133Cs	3.02E-03			<	91973	3.02E-03			<	91973
27-Jun-02	ICP-MS	Fusion-115	140Ce	3.66E-02	4.07E+00	6.09E-02		91973	3.66E-02	5.12E+00	6.28E-02		91973
27-Jun-02	ICP-MS	Fusion-115	141Pr	4.14E-03	4.02E-01	3.42E-03		91973	4.14E-03	2.61E-02	1.34E-03		91973
27-Jun-02	ICP-MS	Fusion-115	151AMU	3.82E-04	2.45E-02	1.78E-03		91973	3.82E-04	8.66E-03	5.19E-04		91973
27-Jun-02	ICP-MS	Fusion-115	181Ta	6.75E-04	3.94E-01	1.49E-02		91973	6.75E-04	4.65E-01	4.86E-03		91973
27-Jun-02	ICP-MS	Fusion-115	182W	1.33E-02	1.53E+00	6.02E-02		91973	1.33E-02	1.12E+00	2.73E-02		91973
27-Jun-02	ICP-MS	Fusion-115	205Tl	2.05E-03	5.28E-02	2.87E-03		91973	2.05E-03	1.62E-02	6.03E-04		91973
26-Jun-02	ICP-MS	Fusion-115	240Pu	2.52E-04	2.42E-02	2.24E-03		91973	2.52E-04	7.08E-03	1.76E-04		91973
26-Jun-02	ICP-MS	Fusion-115	239Pu	6.84E-03	3.91E-01	1.16E-02		91973	6.84E-03	1.16E-01	9.86E-03		91973
26-Jun-02	ICP-MS	Fusion-115	232Th	4.85E-03	1.36E-01	1.07E-02		91973	4.85E-03	9.21E-02	7.25E-03		91973
26-Jun-02	ICP-MS	Fusion-115	237Np	8.61E-04	1.43E-02	7.96E-03		91973	8.61E-04	1.04E-02	4.14E-03		91973
30-Jun-02	ICP-MS	Fusion-115	233U	9.00E-04	6.10E-03	7.24E-04		909938	9.00E-04	1.90E-03	2.43E-03		909938
30-Jun-02	ICP-MS	Fusion-115	234U	1.14E-03	3.99E-03	4.07E-04		909938	1.14E-03	2.51E-03	2.61E-03		909938
30-Jun-02	ICP-MS	Fusion-115	235U	2.36E-03	2.43E-02	4.88E-03		909938	2.36E-03	1.85E-02	2.87E-03		909938
30-Jun-02	ICP-MS	Fusion-115	236U	1.49E-03	3.70E-03	2.40E-03		909938	1.49E-03	2.72E-03	2.43E-03		909938
30-Jun-02	ICP-MS	Fusion-115	238U	1.03E-01	1.11E+00	5.12E-02		909938	1.03E-01	1.75E+00	1.79E-01		909938

				01-1845-B1-Zr					01-1845-B2-Zr				
				MDL	Proc. Blank-1-Zr	Uncert.	Detection	DF	MDL	Proc. Blank-2-Zr	Uncert.	Detection	DF
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
25Jun02a	ICP-MS	Fusion-116	101Ru	3.16E-01			<	123763	3.16E-01			<	123763
25Jun02a	ICP-MS	Fusion-116	103Rh	1.15E-03	4.43E-02	4.33E-03		123763	1.15E-03	3.50E-02	2.34E-03		123763
25Jun02a	ICP-MS	Fusion-116	105Pd	6.09E-03	5.50E-01	4.29E-02		123763	6.09E-03	7.60E-01	7.04E-03		123763
25Jun02a	ICP-MS	Fusion-116	196Pt	1.51E-02	2.69E-01	1.42E-02		123763	1.51E-02	5.94E-01	2.06E-02		123763

				01-1845-B1-Zr					01-1845-B2-Zr				
				MDL	Proc. Blank-1-Zr-114	Uncert.	Detection	DF	MDL	Proc. Blank-2-Zr-114	Uncert.	Detection	DF
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
1-Jul-02	ICP-MS	Fusion-114	129I	4.95E-03	3.57E-01	3.13E-02		12955	4.95E-03	2.52E-01	1.47E-02		12955
1-Jul-02	ICP-MS	Fusion-114	127I	2.89E-03	2.61E-01	1.83E-02		12955	2.89E-03	1.98E-01	8.70E-03		12955

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				AZ-101 CS AR					AZ-101 CS AR-Dup.					AZ-101 CS AR-Trip.				
				MDL	01-1845-Ni	Uncert.	Detection	DF	MDL	01-1845-D-Ni	Uncert.	Detection	DF	MDL	01-1845-T-Ni	Uncert.	Detection	DF
1-Jul-02	ICP-MS	Fusion-115	129I	2.60E-01	8.74E+00	3.48E-01		679551	2.47E-01	6.61E+00	7.43E-01		644842	2.52E-01	5.55E+00	2.61E-01		660046
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
27-Jun-02	ICP-MS	Fusion-115	51V	3.23E-02	2.40E+01	3.85E-01		111832	2.27E-02	2.39E+01	1.53E-01		78643	2.55E-02	2.22E+01	1.12E-01		88443
27-Jun-02	ICP-MS	Fusion-115	59Co	7.31E-03	4.67E+01	2.07E-01		111832	5.14E-03	3.39E+01	1.47E-01		78643	5.78E-03	3.44E+01	1.47E-01		88443
27-Jun-02	ICP-MS	Fusion-115	75As	1.42E-01	4.93E+01	2.33E-01		111832	1.00E-01	4.57E+01	1.61E-01		78643	1.13E-01	4.34E+01	3.26E-01		88443
27-Jun-02	ICP-MS	Fusion-115	82Se	1.42E+00			<	111832	9.98E-01			<	78643	1.12E+00			<	88443
27-Jun-02	ICP-MS	Fusion-115	85Rb	2.87E-02	3.23E+02	1.65E+00		111832	2.02E-02	2.17E+02	4.10E-01		78643	2.27E-02	2.47E+02	2.44E+00		88443
27-Jun-02	ICP-MS	Fusion-115	89Y	1.04E-02	1.18E+02	1.83E-01		111832	7.32E-03	1.15E+02	3.81E-01		78643	8.23E-03	1.17E+02	4.78E-01		88443
27-Jun-02	ICP-MS	Fusion-115	100Mo	3.40E-02	1.47E+02	1.41E+00		111832	2.39E-02	1.46E+02	4.45E-01		78643	2.69E-02	1.45E+02	7.28E-02		88443
27-Jun-02	ICP-MS	Fusion-115	99Tc	1.94E-03	8.99E+00	2.15E-02		111832	1.37E-03	8.41E+00	4.82E-02		78643	1.54E-03	8.68E+00	4.30E-02		88443
27-Jun-02	ICP-MS	Fusion-115	121Sb	6.78E-03	3.83E+00	1.04E-02		111832	4.77E-03	3.20E+00	5.47E-02		78643	5.36E-03	3.24E+00	2.13E-02		88443
27-Jun-02	ICP-MS	Fusion-115	130Te	2.91E-01	2.06E+02	1.72E+00		111832	2.05E-01	1.93E+02	1.11E+00		78643	2.30E-01	1.89E+02	7.40E-01		88443
27-Jun-02	ICP-MS	Fusion-115	133Cs	3.67E-03	1.68E+01	8.75E-02		111832	2.58E-03	1.67E+01	1.02E-01		78643	2.90E-03	1.63E+01	1.41E-01		88443
27-Jun-02	ICP-MS	Fusion-115	140Ce	4.45E-02	3.01E+02	3.95E-01		111832	3.13E-02	2.89E+02	1.49E+00		78643	3.52E-02	3.01E+02	2.49E+00		88443
27-Jun-02	ICP-MS	Fusion-115	141Pr	5.03E-03	2.73E+02	1.24E+00		111832	3.54E-03	2.59E+02	1.79E+00		78643	3.98E-03	2.59E+02	6.57E-01		88443
27-Jun-02	ICP-MS	Fusion-115	151AMU	4.65E-04	2.14E+01	6.35E-02		111832	3.27E-04	2.05E+01	1.43E-01		78643	3.68E-04	2.04E+01	7.60E-02		88443
27-Jun-02	ICP-MS	Fusion-115	181Ta	8.21E-04	9.20E+00	3.45E-01		111832	5.77E-04	4.52E-01	3.74E-02		78643	6.49E-04	1.31E+00	8.43E-02		88443
27-Jun-02	ICP-MS	Fusion-115	182W	1.61E-02	3.83E+01	3.19E-01		111832	1.14E-02	3.22E+01	1.53E-01		78643	1.28E-02	2.94E+01	2.07E-01		88443
27-Jun-02	ICP-MS	Fusion-115	205Tl	2.49E-03	1.27E-01	8.82E-04		111832	1.75E-03	1.16E-01	5.92E-03		78643	1.97E-03	2.23E-01	4.36E-03		88443
26-Jun-02	ICP-MS	Fusion-115	240Pu	1.89E-04	1.47E+00	1.59E-02		69029	1.33E-04	1.28E+00	1.59E-02		48543	1.50E-04	1.24E+00	1.36E-02		54592
26-Jun-02	ICP-MS	Fusion-115	239Pu	5.13E-03	2.07E+01	2.55E-02		69029	3.61E-03	1.76E+01	3.97E-02		48543	4.06E-03	1.69E+01	5.19E-02		54592
26-Jun-02	ICP-MS	Fusion-115	232Th	3.64E-03	1.16E+02	5.00E-01		69029	2.56E-03	1.06E+02	7.70E-01		48543	2.88E-03	1.04E+02	4.12E-01		54592
26-Jun-02	ICP-MS	Fusion-115	237Np	6.46E-04	3.00E+01	4.89E-02		69029	4.55E-04	2.66E+01	4.91E-02		48543	5.11E-04	2.53E+01	9.32E-02		54592
30-Jun-02	ICP-MS	Fusion-115	233U	1.11E-03	1.73E-01	1.03E-02		1118321	7.78E-04	1.65E-01	1.12E-02		786426	8.74E-04	1.59E-01	2.50E-02		884433
30-Jun-02	ICP-MS	Fusion-115	234U	1.40E-03	2.98E-01	1.24E-02		1118321	9.82E-04	2.77E-01	3.93E-03		786426	1.10E-03	2.87E-01	1.05E-02		884433
30-Jun-02	ICP-MS	Fusion-115	235U	2.90E-03	3.45E+01	3.80E-01		1118321	2.04E-03	3.23E+01	1.27E-01		786426	2.29E-03	3.31E+01	3.03E-01		884433
30-Jun-02	ICP-MS	Fusion-115	236U	1.83E-03	2.64E+00	4.48E-02		'1118321'	1.29E-03	2.40E+00	3.74E-02		786426	1.45E-03	2.37E+00	3.02E-02		884433
30-Jun-02	ICP-MS	Fusion-115	238U	1.26E-01	3.77E+03	2.37E+01		1118321	8.89E-02	3.53E+03	3.14E+01		786426	1.00E-01	3.64E+03	1.45E+01		884433

				AZ-101 CS AR					AZ-101 CS AR-Dup.					AZ-101 CS AR-Trip.				
				MDL	01-1845-Zr	Uncert.	Detection	DF	MDL	01-1845-D-Zr	Uncert.	Detection	DF	MDL	01-1845-T-Zr	Uncert.	Detection	DF
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
25Jun02a	ICP-MS	Fusion-116	101Ru	1.61E-01	1.18E+03	7.16E+00		63001	1.54E-01	1.19E+03	9.71E-01		60515	1.64E-01	1.07E+03	1.90E+01		64388
25Jun02a	ICP-MS	Fusion-116	103Rh	5.85E-04	9.49E+01	6.21E-01		63001	5.62E-04	9.47E+01	4.88E-01		60515	5.98E-04	8.28E+01	1.96E-01		64388
25Jun02a	ICP-MS	Fusion-116	105Pd	3.10E-03	1.10E+02	4.52E-01		63001	2.98E-03	1.42E+02	9.78E-01		60515	3.17E-03	4.99E+01	3.53E-01		64388
25Jun02a	ICP-MS	Fusion-116	196Pt	7.70E-03	5.58E-01	2.89E-03		63001	7.40E-03	5.97E-01	4.52E-02		60515	7.87E-03	9.40E-01	1.14E-02		64388

				AZ-101 CS AR					AZ-101 CS AR-Dup.					AZ-101 CS AR-Trip.				
				MDL	01-1845-Zr-114	Uncert.	Detection	DF	MDL	01-1845-D-Zr-114	Uncert.	Detection	DF	MDL	01-1845-T-Zr-114	Uncert.	Detection	DF
Experiment	Method	Prep	Analyte	(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)			(mg/Kg)	(mg/Kg)	(mg/Kg)		
1-Jul-02	ICP-MS	Fusion-114	129I	2.60E-01	8.74E+00	3.48E-01		679551	2.47E-01	6.61E+00	7.43E-01		644842	2.52E-01	5.55E+00	2.61E-01		660046
1-Jul-02	ICP-MS	Fusion-114	127I	1.52E-01	1.10E+01	2.82E-01		679551	1.44E-01	1.08E+01	3.61E-01		644842	1.47E-01	1.01E+01	2.21E-01		660046

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					MRQ	avg. MDL	MRQ > 3xMDL?	Average (mg/Kg)	Average D.F.	RSD
1-Jul-02	ICP-MS	Fusion-115	129I	129I	1.50E+00	2.53E-01	yes	6.97E+00	661480	23
Experiment	Method	Prep	Analyte	Analyte	(mg/Kg)	(mg/Kg)	(Y / N)	(mg/Kg)		%
27-Jun-02	ICP-MS	Fusion-115	51V	51V	6.00E+00	2.68E-02	yes	2.34E+01	92973	4
27-Jun-02	ICP-MS	Fusion-115	59Co	59Co	3.00E+00	6.08E-03	yes	3.83E+01	92973	19
27-Jun-02	ICP-MS	Fusion-115	75As	75As	3.00E+00	1.18E-01	yes	4.61E+01	92973	6
27-Jun-02	ICP-MS	Fusion-115	82Se	82Se	3.00E+02	1.18E+00	yes	ND	92973	
27-Jun-02	ICP-MS	Fusion-115	85Rb	85Rb	6.00E+00	2.38E-02	yes	2.62E+02	92973	21
27-Jun-02	ICP-MS	Fusion-115	89Y	89Y	6.00E+00	8.65E-03	yes	1.17E+02	92973	1
27-Jun-02	ICP-MS	Fusion-115	100Mo	100Mo	3.00E+01	2.83E-02	yes	1.46E+02	92973	1
27-Jun-02	ICP-MS	Fusion-115	99Tc	99Tc	6.00E+02	1.61E-03	yes	8.69E+00	92973	3
27-Jun-02	ICP-MS	Fusion-115	121Sb	121Sb	1.20E+01	5.64E-03	yes	3.42E+00	92973	10
27-Jun-02	ICP-MS	Fusion-115	130Te	130Te	6.00E+00	2.42E-01	yes	1.96E+02	92973	4
27-Jun-02	ICP-MS	Fusion-115	133Cs	133Cs	7.00E-04	3.05E-03	no	1.66E+01	92973	2
27-Jun-02	ICP-MS	Fusion-115	140Ce	140Ce	6.00E+00	3.70E-02	yes	2.97E+02	92973	2
27-Jun-02	ICP-MS	Fusion-115	141Pr	141Pr	6.00E+00	4.18E-03	yes	2.64E+02	92973	3
27-Jun-02	ICP-MS	Fusion-115	151AMU	151AMU	6.00E+00	3.87E-04	yes	2.08E+01	92973	2
27-Jun-02	ICP-MS	Fusion-115	181Ta	181Ta	6.00E+00	6.82E-04	yes	3.65E+00	92973	132
27-Jun-02	ICP-MS	Fusion-115	182W	182W	6.00E+00	1.34E-02	yes	3.33E+01	92973	14
27-Jun-02	ICP-MS	Fusion-115	205Tl	205Tl	6.00E+02	2.07E-03	yes	1.55E-01	92973	38
26-Jun-02	ICP-MS	Fusion-115	240Pu	240Pu	6.00E+00	1.57E-04	yes	1.33E+00	57388	9
26-Jun-02	ICP-MS	Fusion-115	239Pu	239Pu	6.00E+00	4.27E-03	yes	1.84E+01	57388	11
26-Jun-02	ICP-MS	Fusion-115	232Th	232Th	6.00E+02	3.03E-03	yes	1.09E+02	57388	6
26-Jun-02	ICP-MS	Fusion-115	237Np	237Np	6.00E+02	5.37E-04	yes	2.73E+01	57388	9
30-Jun-02	ICP-MS	Fusion-115	233U	233U	1.50E+00	9.19E-04	yes	1.66E-01	929727	4
30-Jun-02	ICP-MS	Fusion-115	234U	234U	1.50E+00	1.16E-03	yes	2.87E-01	929727	4
30-Jun-02	ICP-MS	Fusion-115	235U	235U	1.50E+00	2.41E-03	yes	3.33E+01	929727	3
30-Jun-02	ICP-MS	Fusion-115	236U	236U	1.50E+00	1.52E-03	yes	2.47E+00	929727	6
30-Jun-02	ICP-MS	Fusion-115	238U	238U	1.50E+00	1.05E-01	yes	3.64E+03	929727	3

					MRQ	avg. MDL	MRQ > 3xMDL?	Average (mg/Kg)	Average D.F.	RSD
Experiment	Method	Prep	Analyte	Analyte	(mg/Kg)	(mg/Kg)	(Y / N)	(mg/Kg)		%
25Jun02a	ICP-MS	Fusion-116	101Ru	101Ru	3.00E+02	1.60E-01	yes	1.15E+03	62635	6
25Jun02a	ICP-MS	Fusion-116	103Rh	103Rh	3.00E+02	5.82E-04	yes	9.08E+01	62635	8
25Jun02a	ICP-MS	Fusion-116	105Pd	105Pd	3.00E+02	3.08E-03	yes	1.01E+02	62635	47
25Jun02a	ICP-MS	Fusion-116	196Pt	196Pt	3.00E+02	7.65E-03	yes	6.98E-01	62635	30

					MRQ	avg. MDL	MRQ > 3xMDL?	Average (mg/Kg)	Average D.F.	RSD
Experiment	Method	Prep	Analyte	Analyte	(mg/Kg)	(mg/Kg)	(Y / N)	(mg/Kg)		%
1-Jul-02	ICP-MS	Fusion-114	129I	129I	1.50E+00	2.53E-01	?	6.97E+00	661480	23
1-Jul-02	ICP-MS	Fusion-114	127I	127I	1.50E+00	1.48E-01	yes	1.06E+01	661480	4

					80% - 120%	80% - 120%	75%-125%	75%-125%	80% - 120%	< +/-10%
					Blank Spike (LCS)	Post Blank Spike (LCS)	Matrix Spike (MS)	Post Spike A	SRM2710 (LCS)	Serial Dilution
1-Jul-02	ICP-MS	Fusion-115	129I	129I	-16	1	-288	89	0	603
Experiment	Method	Prep	Analyte	Analyte	%Rec	%Rec	%Rec	%Rec	%Rec	%Rec
27-Jun-02	ICP-MS	Fusion-115	51V	51V	115	114	75	101	110	11
27-Jun-02	ICP-MS	Fusion-115	59Co	59Co	98	109	75	101	144	11
27-Jun-02	ICP-MS	Fusion-115	75As	75As	96	107	23	103	94	45
27-Jun-02	ICP-MS	Fusion-115	82Se	82Se	106	96	77	96		<10 X MDL
27-Jun-02	ICP-MS	Fusion-115	85Rb	85Rb	NR	111	NR	102	107	9
27-Jun-02	ICP-MS	Fusion-115	89Y	89Y	129	107	106	101	96	2
27-Jun-02	ICP-MS	Fusion-115	100Mo	100Mo	106	109	53	101	178	2
27-Jun-02	ICP-MS	Fusion-115	99Tc	99Tc	NS	105	NS	100		0
27-Jun-02	ICP-MS	Fusion-115	121Sb	121Sb	69	102	68	103	39	29
27-Jun-02	ICP-MS	Fusion-115	130Te	130Te	73	93	45	90		8
27-Jun-02	ICP-MS	Fusion-115	133Cs	133Cs	103	104	53	106	105	1
27-Jun-02	ICP-MS	Fusion-115	140Ce	140Ce	88	104	11	102	101	2
27-Jun-02	ICP-MS	Fusion-115	141Pr	141Pr	135	105	163	99		1
27-Jun-02	ICP-MS	Fusion-115	151AMU	151AMU	NS	105	NS	105	124	1
27-Jun-02	ICP-MS	Fusion-115	181Ta	181Ta	NR	101	NR	97		39
27-Jun-02	ICP-MS	Fusion-115	182W	182W	106	102	54	101	103	1
27-Jun-02	ICP-MS	Fusion-115	205Tl	205Tl	82	96	80	95	111	50
26-Jun-02	ICP-MS	Fusion-115	240Pu	240Pu		110		118		8
26-Jun-02	ICP-MS	Fusion-115	239Pu	239Pu		106		114		5
26-Jun-02	ICP-MS	Fusion-115	232Th	232Th	50	107	NR	110	111	5
26-Jun-02	ICP-MS	Fusion-115	237Np	237Np	NS	106	NS	111		5
30-Jun-02	ICP-MS	Fusion-115	233U	233U						13
30-Jun-02	ICP-MS	Fusion-115	234U	234U						4
30-Jun-02	ICP-MS	Fusion-115	235U	235U						8
30-Jun-02	ICP-MS	Fusion-115	236U	236U						9
30-Jun-02	ICP-MS	Fusion-115	238U	238U	124	102	NR	106	109	9

NR = not recovered NS = not spiked

					80% - 120%	80% - 120%	75%-125%	75%-125%	80% - 120%	< +/-10%
					Blank Spike (LCS)	Post Blank Spike (LCS)	Matrix Spike (MS)	Post Spike A	SRM2710 (LCS)	Serial Dilution
Experiment	Method	Prep	Analyte	Analyte	%Rec	%Rec	%Rec	%Rec		%Rec
25Jun02a	ICP-MS	Fusion-116	101Ru	101Ru	88	100	NR	106		2
25Jun02a	ICP-MS	Fusion-116	103Rh	103Rh	94	95	66	95		3
25Jun02a	ICP-MS	Fusion-116	105Pd	105Pd	84	93	NR	104		3
25Jun02a	ICP-MS	Fusion-116	196Pt	196Pt	92	92	97	94		24

					80% - 120%	80% - 120%	75%-125%	75%-125%	80% - 120%	< +/-10%
					Blank Spike (LCS)	Post Blank Spike (LCS)	Matrix Spike (MS)	Post Spike A	SRM2710 (LCS)	Serial Dilution
Experiment	Method	Prep	Analyte	Analyte	%Rec	%Rec	%Rec	%Rec		%Rec
1-Jul-02	ICP-MS	Fusion-114	129I	129I	NS	1	NS	89	0	NR
1-Jul-02	ICP-MS	Fusion-114	127I	127I	NR	0	NR	90	0	NR

				Proc. Blank-1				AZ-101 SUP AR				AZ-101 SUP AR-Dup.				AZ-101 SUP AR-Trip.		
			MDL	01-1844-B	Uncert.		MDL	01-1844	Uncert.		MDL	01-1844-Dup	Uncert.		MDL	01-1844-Trip	Uncert.	
Method	Prep	Analyte	uCi/mL	uCi/mL		DF	uCi/mL	uCi/mL		DF	uCi/mL	uCi/mL		DF	uCi/mL	uCi/mL		DF
ICP-MS	ALO-128	Pu-239	3.79E-06	9.24E-06	7.95E-06		5.58E-04	2.49E-03	4.37E-05		5.61E-04	2.00E-03	3.36E-04		5.44E-04	1.50E-03	7.19E-04	
ICP-MS	ALO-128	Pu-240	1.11E-05			<	1.64E-03			<	1.65E-03			<	1.60E-03			<
ICP-MS	ALO-128	Np-237	5.03E-08			<	7.41E-06	3.87E-05	4.23E-06		7.45E-06	3.88E-05	1.98E-06		7.22E-06	3.55E-05	4.39E-06	
ICP-MS	ALO-128	U-233	5.79E-08			<	8.73E-06			<	9.04E-06			<	8.86E-06			<
ICP-MS	ALO-128	U-234	3.84E-08			<	5.80E-06			<	6.01E-06			<	5.88E-06			<
ICP-MS	ALO-128	U-235	1.81E-11	1.26E-10	7.93E-06		2.73E-09	4.66E-08	7.34E-09		2.83E-09	4.75E-08	6.27E-09		2.77E-09	4.78E-08	2.69E-09	
ICP-MS	ALO-128	U-236	3.94E-10			<	5.94E-08	9.15E-08	2.53E-08		6.16E-08	9.69E-08	4.59E-08		6.03E-08	9.11E-08	2.42E-08	
ICP-MS	ALO-128	U-238	1.04E-11	2.44E-09	3.25E-06		1.57E-09	9.22E-07	1.30E-09		1.62E-09	9.56E-07	9.78E-10		1.59E-09	9.36E-07	3.02E-10	
ICP-MS	ALO-128	I-129	8.97E-08			<	1.31E-05			<	1.28E-05			<	1.30E-05			<
ICP-MS	ALO-128	Tc-99	1.21E-06	2.05E-05	2.59E-06		1.73E-04	3.32E-01	2.70E-03		1.76E-04	3.31E-01	4.32E-03		1.87E-04	3.18E-01	2.75E-03	

				Proc. Blank-1				AZ-101 SUP AR				AZ-101 SUP AR-Dup.				AZ-101 SUP AR-Trip.		
			MDL	01-1844-B	Uncert.		MDL	01-1844	Uncert.		MDL	01-1844-Dup	Uncert.		MDL	01-1844-Trip	Uncert.	
Method	Prep	Analyte	ug/mL	ug/mL		DF	ug/mL	ug/mL		DF	ug/mL	ug/mL		DF	ug/mL	ug/mL		DF
ICP-MS	ALO-128	U	2.98E-05	7.30E-03	1.57E-04		4.50E-03	2.77E+00	3.76E-02		4.66E-03	2.87E+00	4.78E-02		4.57E-03	2.81E+00	5.50E-02	
ICP-MS	ALO-128	I-127	7.95E-04	1.11E-03	2.33E-04		1.16E-01	3.71E-01	1.36E-01		1.14E-01	1.87E-01	3.42E-02		1.15E-01	1.27E-01	7.87E-03	
ICP-MS	ALO-128	AMU-241	7.20E-06			<	1.06E-03			<	1.08E-03			<	1.08E-03	1.23E-03	1.25E-03	
ICP-MS	ALO-128	Rb	1.70E-04	2.16E-03	3.55E-04		2.42E-02	9.53E+00	9.63E-02		2.47E-02	9.54E+00	2.08E-01		2.62E-02	8.53E+00	8.34E-02	
ICP-MS	ALO-128	Cs	2.08E-04	4.38E-04	1.25E-04		8.94E-03	2.62E+01	2.53E-01		9.17E-03	2.68E+01	1.86E-01		8.77E-03	2.63E+01	1.57E-01	
ICP-MS	ALO-128	Cs-133	8.49E-05			<	1.61E-02	2.68E+01	8.94E+00		1.55E-02	2.75E+01	9.17E+00		1.55E-02	2.63E+01	8.77E+00	
ICP-MS	ALO-128	Cs-135	7.50E-05				1.31E-02	8.82E+00	2.94E+00		1.35E-02	9.06E+00	3.02E+00		1.37E-02	8.61E+00	2.87E+00	
ICP-MS	ALO-128	Cs-137	7.05E-05				1.33E-02	1.61E+01	5.38E+00		1.49E-02	1.64E+01	5.46E+00		1.35E-02	1.58E+01	5.25E+00	

3Jul02

										80% - 120%	80% - 120%	75%-125%	75%-125%	< +/-10%
				MRQ	MDL	MRQ > 3xMDL?	Average		RSD	Blank Spike (LCS)	Post Blank Spike (LCS)	Matrix Spike (MS)	Post Spike A	Serial Dilution
Method	Prep	Analyte	Analyte	uCi/mL	uCi/mL		uCi/mL	DF	%	%Rec	%Rec	%Rec	%Rec	%Diff
ICP-MS	ALO-128	Pu-239	Pu-239							NS	101.6	NS	99.1	8.8
ICP-MS	ALO-128	Pu-240	Pu-240							NS	100.6	NS	99.3	10.9
ICP-MS	ALO-128	Np-237	Np-237							NS	101.1	NS	100.5	2.2
ICP-MS	ALO-128	U-233	U-233											<10 X MDL
ICP-MS	ALO-128	U-234	U-234											<10 X MDL
ICP-MS	ALO-128	U-235	U-235											7.2
ICP-MS	ALO-128	U-236	U-236											<10 X MDL
ICP-MS	ALO-128	U-238	U-238											0.9
ICP-MS	ALO-128	I-129	I-129							NS	95.6	NS	83.7	<10 X MDL
ICP-MS	ALO-128	Tc-99	Tc-99							NS	105.1	NS	106.5	1.5

NR= not recovered, NS=not spiked

										80% - 120%	80% - 120%	75%-125%	75%-125%	< +/-10%
				MRQ	MDL	MRQ > 3xMDL?	Average		RSD	Blank Spike (LCS)	Post Blank Spike (LCS)	Matrix Spike (MS)	Post Spike A	Serial Dilution
Method	Prep	Analyte	Analyte	ug/mL	ug/mL		ug/mL	DF	%	%Rec	%Rec	%Rec	%Rec	%Diff
ICP-MS	ALO-128	U	U							104.8	101.1	106.4	100.8	0.8
ICP-MS	ALO-128	I-127	I-127							91.2	93.2	20.9	99.0	<10 X MDL
ICP-MS	ALO-128	AMU-241	AMU-241							NS	91.8	NS	91.3	<10 X MDL
ICP-MS	ALO-128	Rb	Rb							103.0	100.8	128.5	95.3	5.3
ICP-MS	ALO-128	Cs	Cs							106.7	103.4	97.9	111.5	0.8
ICP-MS	ALO-128	Cs-133	Cs-133											
ICP-MS	ALO-128	Cs-135	Cs-135											
ICP-MS	ALO-128	Cs-137	Cs-137											

NR= not recovered, NS=not spiked

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F13-016

Battelle, PNNL
Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Project / WP#:	42365/W65128
ASR#:	6193.06
Client:	Mike Urie
Total Samples:	2

Analysis: Multi-Element (^{127}I, ^{129}I)

Procedure: 329-OP-SCO1 Rev. 0, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	R102924	PQ-Excell, Thermal Elemental
	512-06-01-031	Sartorius R200D Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 22 May 2003

Analysis Files: 21may03 (Iodine)

For Maintenance Records, see the ICPMS Service Center 98038 RIDS.
For standards Certificate of Analysis and balance certificate of calibration see the ICPMS Service Center 98038 RIDS or the Chemical Measurements Center 98620 RIDS.

Analyst(s): Orville Thomas Farmer III

Orville Thomas Farmer III
Prepared By

06Oct03
Date

Reviewed By

30Oct03
Date

Sample Solutions Submitted for Analysis:

RPL #	Client I.D.
01-01844-r2-b1-wd	Process BLK-1
01-01844-r2-bs-wd	ICP/MS Blank Spike
01-01844-r2-wd	AZ-101 SUP AR-R2
01-01844-r2-dup-wd	AZ-101 SUP AR-R2-Dup
01-01844-r2-trip-wd	AZ-101 SUP AR-R2-Trip
01-01844-r2-ms-wd	ICP/MS Matrix Spike
01-01845-r2-b1-zr (114)	Process BLK-1
01-01845-r2-b2-zr (114)	Process BLK-2
01-01845-r2-bs-zr (114)	ICP/MS Blank Spike
01-01845-r2-zr (114)	AZ-101 CS AR-R2
01-01845-r2-dup-zr (114)	AZ-101 CS AR-R2-Dup
01-01845-r2-trip-zr (114)	AZ-101 CS AR-R2-Trip
01-01845-r2-ms-zr (114)	ICP/MS Matrix Spike

Two (2) samples, AZ-101 centrifuged solids and supernatant, with Batch QC samples were prepared for analysis using PNL-ALO-114 (for the centrifuged solids samples) and water dilution (for the supernatant samples) and submitted for analysis per 329-OP-SC01 on a radioactive-material-contained ICP/MS for all requested analyte(s).

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis. Results are reported in mg/L for sample 01-01844-R2 and mg/kg for sample 01-01845-R2.

The 127I isotope concentrations were determined using a 129I calibration standard. Batch and instrument QC checks were performed only on 129I. It is assumed that a QC success for 129I represents the 127I analyses.

The analytical results in the tables below show the isotope determined for each elemental/isotope analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 3 instrument standard blank solutions, which were evaluated, at the beginning of the analytical run. The IDL was calculated by multiplying the observed standard deviation of the 3 standard blanks solutions by 6.57. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and the sample total dilution factor. Other instrument QCs are those labeled ICS (Interference Correction Standard), ICSD (Interference Correction Standard Duplicate), ICSDS (ICSD Spike) or often called a Post Matrix Spike, and a DT (Dilution Test) or serial dilution. The ICSDS can be used if the Matrix Spike fails. The application of ICS and ICSD is to evaluate instrument RPD as apposed to method RPD (client sample and duplicate). The DT is used as an instrument check to evaluate interferences. This test is only valid if the sample concentration used to dilute is at least 50 times the MDL.

2. Final Results

2.1 Water Dilution Results

Sample ID (^{129}I)		Sample (mg/L)	+/- 1 σ (mg/L)	MDL (mg/L)	Total Diln Factor	%Rec %RSD
Process BLK-1	<	2.46E-02		2.46E-03	1034	
ICP/MS Blank Spike	<	8.91E-01	7.15E-02	1.23E-01	51719	90
AZ-101 SUP AR-R2	<	2.46E-02		2.46E-03	1034	
AZ-101 SUP AR-R2-Dup	<	2.46E-02		2.46E-03	1034	
AZ-101 SUP AR-R2-Trip	<	2.46E-02		2.46E-03	1034	
ICP/MS Matrix Spike		1.94E+00	8.9E-02	9.80E-02	41213	97

2.2 PNL-ALO-114 Results

Sample ID (^{129}I)		Sample (mg/kg)	+/- 1 σ (mg/kg)	MDL (mg/kg)	Total Diln Factor	%Rec %RSD
Process BLK-1 (1)	<	9.55E-04		9.55E-05	50	
Process BLK-2 (1)	<	9.55E-04		9.55E-05	50	
ICP/MS Blank Spike (1)		2.02E-02	7.6E-04	4.77E-04	250	82
AZ-101 CS AR-R2	<	3.69E-01		3.69E-02	19312	
AZ-101 CS AR-R2-Dup	<	4.45E-01		4.45E-02	23288	
AZ-101 CS AR-R2-Trip	<	5.11E-01		5.11E-02	26738	
ICP/MS Matrix Spike		1.09E+01	4.2E-01	2.55E-01	133333	83

(1) Concentration in the solution analyzed by the ICP/MS are in mg/L.

Narrative:

All batch and instrument QC solutions except the post matrix spike met their respective success criteria. The blank spike in the water dilution did recover at 90% but was detected below 10 times the MDL and is reported for information only. The Post Matrix spike on the water dilution preparation failed having a Recovery of 68%.

2.3. Water Dilution Results

Sample ID (^{127}I)	Sample (mg/L)	+/- 1 σ (mg/L)	MDL (mg/L)	Total Diln Factor	%Rec %RSD
Process BLK-1	< 7.42E-02		7.4E-03	1034	
ICP/MS Blank Spike	< 3.71E+00		3.7E-01	51719	
AZ-101 SUP AR-R2	< 7.42E-02		7.4E-03	1034	
AZ-101 SUP AR-R2-Dup	< 7.42E-02		7.4E-03	1034	
AZ-101 SUP AR-R2-Trip	< 7.42E-02		7.4E-03	1034	
ICP/MS Matrix Spike	< 2.96E+00		3.0E-01	41213	

2.4. PNL-ALO-114 Results

Sample ID (^{127}I)	Sample (mg/kg)	+/- 1 σ (mg/kg)	MDL (mg/kg)	Total Diln Factor	%Rec %RSD
Process BLK-1	< 7.97E-04		8.0E-05	50	
Process BLK-2	< 7.97E-04		8.0E-05	50	
ICP/MS Blank Spike	< 3.99E-03		4.0E-04	250	
AZ-101 CS AR-R2	2.78E+00	6.8E-03	3.1E-02	19312	
AZ-101 CS AR-R2-Dup	2.13E+00	8.8E-03	3.7E-02	23288	
AZ-101 CS AR-R2-Trip	2.44E+00	1.1E-02	4.3E-02	26738	**13**
ICP/MS Matrix Spike	3.69E+00	6.3E-02	2.1E-01	133333	

Narrative:

All batch and instrument QC solutions for 127I were evaluated using the 129I analysis results. The matrix spike result for 127I in the ALO-114 preparation is due to 127I impurity in the I-129 standard material used for batch QC spiking.

CHEMICAL ANALYSIS AND PHYSICAL PROPERTY TESTING 241-AZ-101 TANK WASTE

APPENDIX G

ICP-AES COMPARISON RESULTS FOR WCS

2004

Battelle - Pacific Northwest Division

ICP-AES COMPARISON RESULTS FOR WCS

Digestion Method Comparison using AZ-101 WCS (RPL #01-1845)
ASR 6193 and ASR 6193.01

The analytical service request (ASR) 6193 requested ICP-AES and ICP-MS analysis of the AZ-101 wet centrifuged solids (WCS) using two digestion methods; PNL-ALO-129 and PNL-ALO-115. PNL-ALO-129 is an HNO_3-HCl leaching procedure similar to SW-846 Method 3050. PNL-ALO-115 is a KOH-KNO_3 fusion procedure similar to the KOH option in ASTM C1317-95. The intent of the dual digestion was to obtain better detection limits for those analytes susceptible to the acid digesting processing. The fusion processing was identified as the prep method of choice for total dissolution, although the method required significant dilution for the ICP-AES analysis.

Besides these dissolution procedures, the AZ-101 WCS were also subjected digestion procedure PNL-ALO-116. PNL-ALO-116 is a Na_2O_2-NaOH procedure that is similar to the Na_2O_2 option in ASTM C1317-95. This dissolution was identified for use for the ICP-MS analysis of platinum group metals, since it is considered more rigorous. This procedure is not typically used for the ICP-AES analyses since Na and Zr are typically major components of the wastes and cannot be determined by this fusion method.

Following the HNO3-HCl leach, a significant quantity of the WCS sample remained undissolved. A decision was made to process the residual solids using a modification (mod 2) of the PNL-ALO-129 procedure, which uses HF and evaporation to dryness. This modification was used for the Regulatory DQO work for Project 42365. (However, mod 2 is not applicable to silicon or other elements that may volatilize as fluorides at the digestion processing temperature, and is only applicable to rare earths if a releasing agent such a B or Al is used to facilitate redissolution of rare earth fluoride precipitates.) Besides processing the residual solids, the AZ-101 WCS were also subjected to the PNL-ALO-129 mod 2. Since the Na2O2-NaOH dissolutions were also available, the decision was made to analyze:

1. WCS prepared by PNL-ALO-129 mod 1 (*leach procedure*)
2. Residual solids from 1) prepared by PNL-ALO-129 mod 2 (*total digest procedure*)
3. WCS prepared by PNL-ALO-129 mod 2 (*total digest procedure*)
4. WCS prepared by PNL-ALO-115 (*total digest procedure*)
5. WCS prepared by PNL-ALO-116 (*total digest procedure*)

Although analyses were conducted by both ICP-AES and ICP-MS, only the ICP-AES is included in Table A. For the analytes being measured by ICP-MS, the residuals contained very low and insignificant concentrations. The ICP-MS analysis of the WCS by mod 2 demonstrated very large variation and is considered unusable for comparison. Table A includes all the analyses for all the dissolutions listed above. For (1) and (2) above, the results are summed to provide a 'total' metals concentration and is compared against the results from (3), (4) and (5). Most results were very comparable. However, a number of analytes (primarily Al, Fe, and Zr) demonstrated significant differences between the methods.

Based on this single set of data and without considering the cost impact (e.g., Zr crucibles versus Ni crucibles), the following is suggested:

a) Using PNL-ALO-129 mod 2 on tank solids
b) Discontinuing routine use of PNL-ALO-129 mod 1 on tank solids (exception being Salt Cake)
c) Use of PNL-ALO-116 on tank solids
d) Discontinuing routine use of PNL-ALO-115 on tank solids

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Reviewed By: **Date:** 7-3-02

Table 1: AZ-101 WCS -- Comparison of Dissolution Procedures

Sample A: WCS; RPL# 01-1845
Sample B: Residual solids remaining after Preparation 1; RPL# 01-2273
Preparation 1: PNL-ALO-129 Mod 1; HNO3-HCl digestion
Preparation 2: PNL-ALO-129 Mod 2; HNO3-HCL-HF digestion
Preparation 3: PNL-ALO-115; KOH fusion
Preparation 4: PNL-ALO-116; Na2O2 fusion

Prep >	1 Acid-129M1		2 Acid-129M2		1 & 2 Acid 129M1/M2	(a)	2 Acid-129M2		3 Fusion-115		4 Fusion-116	
Sample	A: WCS		B: Residuals		A+B: WCS+ Residuals	B >5% of A	A: WCS		A: WCS		A: WCS	
Analyte	Average µg/g	DF	Average µg/g	DF	Average µg/g		Average µg/g	DF	Average µg/g	DF	Average µg/g	DF
Ag	100		46	J	145	Yes	55	J	257	JX	303	J
Al	104,400		32,704		137,104	Yes	136,333		130,093		145,183	
Ba	468		30	J	498	Yes	489		425	B	481	B
Be	9	J	4	U	9	No	9	J	23	U	24	U
Ca	2,307		196	J	2,502	Yes	2,400		2,333	J	2,650	JB
Cd	4,473		176		4,650	No	4,673		4,380		4,763	
Ce	417	J	83	U	417	No	377	J	525	J	1,117	J
Cr	473		118		591	Yes	800		610		612	
Cu	220		17	J	237	Yes	238		57	U	61	U
Fe	65,433		2,380		67,813	No	66,767		61,492		68,665	
K	1,667	J	833	U	1,667	No	1,367	J	na		4,883	
La	1,713		58	J	1,771	No	1,793		1,580		1,767	
Li	69	J	13	U	69	No	70	J	74	J	78	J
Mg	397		62	J	459	Yes	440	J	527	J	493	J
Mn	1,363		74	J	1,437	Yes	1,480		1,483		1,440	
Mo	29	J	21	U	29	No	64	J	113	U	122	U
Na	63,267		2,035		65,301	No	70,100		69,100		na	
Nd	1,210		58	J	1,268	No	1,263		1,233	J	1,333	J
Ni	2,480		88	J	2,568	No	2,767		na		2,300	
P	57	J	1,491		1,548	Yes	1,787		347	J	1,967	J
Pb	480		42	U	480	No	490	J	513	J	650	J
Pd	510	J	313	U	510	No	493	J	1,696	U	1,831	U
Si	730	JBX	208	U	730	No	970	JX	3,800	J	3,433	J
Sn	840	J	625	U	840	No	1,000	J	3,393	U	3,662	U
Sr	356		12	J	368	No	356		321	J	333	B
Ti	44	J	11	J	55	Yes	64	J	68	J	65	J
U	3,467	J	833	U	3,467	No	3,567	J	5,300	J	4,883	U
Y	103	J	21	U	103	No	113	J	100	J	120	J
Zn	68	J	21	U	68	No	79	J	127	J	125	J
Zr	8,637		9,595		18,232	Yes	19,467		13,367		na	

Shaded: Results <MDL

Note: U flagged results reportable to 1 significant digit; J flagged results reportable to 2 significant digits; unflagged data reportable to 3 significant digits.

(a) B > 5% of A = Is residual concentration greater than 5% of the sample concentration

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