PNWD-3172
WTP-RPT-021

Chemical and Physical Properties Testing of 241-AN-102 Tank Waste Blended with 241-C-104 Wash/Leachate Solutions

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August 2002

Prepared for Bechtel National, Inc.
under Contract No. 24590-101-TSA-W0000-0004

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Battelle, Pacific Northwest Division
Richland, Washington, 99352

Completeness of Testing

This report describes the results of work and testing specified by test specification TSP-W375-00-00007 and test plan TP-41500-005. 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:

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

Approved for RPP-WTP Project Use:

G. Todd Wright, Manager Research and Technology	Date

Summary

Battelle – Pacific Northwest Division (PNWD) is conducting integrated, process verification, and waste-form qualification tests on Hanford waste from underground storage Tank 241-AN-102 (AN-102) [1] combined with wash and leachate solutions from Tank 241-C-104 (C-104) sludge in support of the River Protection Project-Waste Treatment Plant (RPP-WTP). Testing includes sample compositing, homogenization, and as-received characterization of the AN-102 material, combination with C-104 permeate solutions to produce an AN-102/C-104 blended feed, blended feed characterization, Sr/TRU (transuranic) precipitation, and Cs and Tc removal using ion exchange (IX). This report summarizes the blending of the AN-102 and C-104 materials and the characterization of the resulting AN-102/C-104 blended feed. This blended feed is measured for selected chemical, radiochemical, and organic analytes, and the analyte concentrations compared to the chemical and radiochemical WTP envelope Specification 7.

To obtain the C-104 filtrate, leachate, and wash solutions, the High Level Waste sludge from tank C-104 was retrieved, homogenized, and settled. The feed was filtered using a cross-flow filtration system (Brooks et al. 2000). Solids remaining after filtration were washed and leached with 3 M sodium hydroxide. The initial C-104 filtrates, plus wash and leach solutions produced were characterized and then used as the C-104 blending material in this task.

High Level Waste slurry from tank AN-102 was retrieved using grab samples, in bottles, during the August 2000 sampling event. The AN-102 as-received slurry was homogenized and characterized (Urie 2002). Supernatant and solids from homogenized jars of AN-102 were blended to make waste material consisting of 2 weight-percent undissolved solids (UDS), to be used as feed material in this task. Measurements of selected physical properties (i.e., heat capacity, viscosity, and particle size) for the 2 weight-percent solids AN-102 waste are reported.

Filtrate, leachate, and wash solutions from tank C-104 processing were blended with the 2 percent solids AN-102 waste to produce the AN-102/C-104 blended feed used for characterization and all additional process testing. The blending of the AN-102 and C-104 materials was conducted per test plan (TP) TP-41500-005 (Appendix A) in accordance with the requirements set forth in test specification (TS) TSP-W375-00-00007 (Appendix A), which is further defined in Test Scoping Statement B-1a (work breakdown structure 1.01.2.02). No visible gel formation or net solids increase occurred during blending process.

The AN-102/C-104 blended feed was homogenized, sub-sampled, separated into solids and liquids fractions, and characterized for inorganic, radiochemical, and selected organic analytes. The analytical characterization of the homogenized AN-102/C-104 blended feed included:

- inductively coupled plasma atomic emission spectrometry (ICP-AES)
- radiochemical analyses, including $^{99}Tc^{+7}$

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[1] High Level Waste slurry from tanks AN-102 and C-104 were received by the PNWD from Hanford's 222-S laboratory in November 2000 and March 1999, respectively.

- inductively coupled plasma mass spectrometry (ICP-MS)
- kinetic phosphorescence (KPA) analysis for total uranium
- ion chromatography (IC, inorganic and organic anions)
- total inorganic carbon (TIC) and total organic carbon (TOC)
- selected organic analytes
- titration for hydroxide
- density and weight percent total dissolved solid (TDS) of supernatant
- weight percent solids of centrifuged solids

Table S.1 presents the summary of the physical properties measurements performed on the 2-wt% UDS AN-102 slurry (blend feed) prior to the addition of the C-104 blending solutions.

Table S.1 Physical Properties -- 2-Wt% UDS AN-102 Blend Feed

	Average	Std Dev
TDS – Supernatant (wt%)	51.5	2.3
Wt% Solids – Wet Centrifuged Solids (wt%)	52.9	2.6
Wt% Undissolved Solids	2.4	0.4
Heat Capacity (J/g-K)	2.78	0.04 [(a)]
Viscosity (cP)		
Average @ 25°C and 33 $^1/_s$	30.5	5.6
Average @ 25°C and 300 $^1/_s$	21.8	0.4
Average @ 25°C and 990 $^1/_s$	15.5	0.1
Average @ 35°C and 33 $^1/_s$	24.9	11.9
Average @ 35°C and 300 $^1/_s$	14.2	1.8
Average @ 35°C and 990 $^1/_s$	10.5	0.4
Average @ 50°C and 33 $^1/_s$	18.7	1.3
Average @ 50°C and 300 $^1/_s$	12.5	0.0
Average @ 50°C and 990 $^1/_s$	9.9	0.0
Particle Size (following 90s sonication)		
Volume Distribution – 100% Peak 1 (μm)	4.1	8.0 [(b)]
Number Distribution – 100% Peak 1 (μm)	0.89	0.84 [(b)]

(a) Standard deviation about the mean for 16 measurements.
(b) Peak/mode width

Tables S.2 and S.3 present chemical and radioisotope results for the AN-102/C-104 blended feed supernatant and wet centrifuged solids (WCS). The WCS phase results column presents the analyte concentrations in the wet solids, including the contribution from the supernatant entrained in the solids. The last two UDS results columns present the analyte concentrations of the undissolved solids, after subtraction of the entrained supernatant contribution. The calculated UDS results are reported on both a wet-weight basis (e.g., micrograms per gram of wet centrifuged solids) and a dry-weight basis (e.g., microcuries per gram of dried centrifuged solids). Because the solids were not washed, the UDS contain components that may be removed during pretreatment.

Table S.2. AN-102/C-104 Blended Feed – Analyte Summary

		Supernatant Phase		WCS Phase		UDS	
Analyte	Measure Method	Average µg/mL	Data Flag (a)	Average µg/g	Data Flag (a)	Average µg/g (wet)	Average µg/g (dry)
Ag	ICP-AES	*1E+00*	*UX*	2E+00	UX	<2E+00	<9E+00
Al	ICP-AES	5.34E+03		5.56E+04		5.2E+04	2.8E+05
As	ICP-AES	*1E+01*	*U*	*1.70E+01*	*J*	1.7E+01	9.3E+01
As	ICP-MS	n/m		6.88E+00	JB	6.9E+00	3.8E+01
B	ICP-AES	*5.00E+01*	*B*	*5.70E+01*	*B*	2.2E+01	1.2E+02
B	ICP-MS	n/m		1.74E+01	JBX	1.7E+01	9.5E+01
Ba	ICP-AES	*5E-01*	*U*	7.90E+01		7.9E+01	4.3E+02
Be	ICP-AES	*5E-01*	*U*	*7.05E+00*		7.1E+00	3.8E+01
Be	ICP-MS	n/m		1.43E+01		1.4E+01	7.8E+01
Bi	ICP-AES	*5E+00*	*U*	1.15E+01	J	1.2E+01	6.3E+01
Ca	ICP-AES	1.53E+02	X	4.23E+02		3.2E+02	1.7E+03
Cd	ICP-AES	1.67E+01		2.00E+01		8.3E+00	4.5E+01
Ce	ICP-AES	*1E+01*	*U*	*7.10E+01*	*J*	7.1E+01	3.9E+02
Ce	ICP-MS	n/m		7.02E+01		7.0E+01	3.8E+02
Citrate	Organic/IC	4.70E+02	J	n/m		(b)	(b)
Co	ICP-AES	*3E+00*	*U*	*3E+00*	*U*	<3E+00	<2E+01
Co	ICP-MS	n/m		1.38E+00		1.4E+00	7.5E+00
Cr	ICP-AES	6.91E+01		4.57E+03		4.5E+03	2.5E+04
^{133}Cs	ICP-MS	2.63E+00		n/m		(b)	(b)
Cs Total (g)	ICP-MS and GEA	4.35E+00		4.24E+00		1.2E+00	6.4E+00
Cu	ICP-AES	*6.75E+00*	*J*	1.20E+01	J	7.2E+00	4.0E+01
D2EHP	Deriv/GC/FID	5E-01	U	n/m		(b)	(b)
Dy	ICP-AES	*3E+00*	*U*	*3E+00*	*U*	<3E+00	<2E+01
ED3A	Deriv/GC/FID	6.50E+02	JX	n/m		(b)	(b)
EDTA	Deriv/GC/FID	6.20E+02	JX	n/m		(b)	(b)
Eu	ICP-AES	*5E+00*	*U*	*6E+00*	*U*	<6E+00	<3E+01
F (d)	IC	3.70E+03		n/m		(c)	(c)
Fe	ICP-AES	6.55E+00	J	3.45E+03		3.4E+03	1.9E+04
Formate	Organic/IC	2.80E+03		n/m		(b)	(b)
Gluconate	Organic/IC	4.05E+04		n/m		(b)	(b)
Glycolate (h)	Organic/IC	3.30E+03		n/m		(b)	(b)
HEDTA	Deriv/GC/FID	1E+02	UX	n/m		(b)	(b)
IDA	Deriv/GC/FID	1.40E+03	X	n/m		(b)	(b)
K	ICP-AES	5.45E+02	JX	*5.65E+02*	*JX*	1.8E+02	9.9E+02
La	ICP-AES	3.25E+00	J	1.06E+02		1.0E+02	5.6E+02
Li	ICP-AES	*1.60E+00*	*J*	*1.10E+01*	*J*	9.9E+00	5.4E+01
Li	ICP-MS	n/m		2.38E+01	X	2.4E+01	1.3E+02
Mg	ICP-AES	5E+00	U	5.40E+01	J	5.4E+01	2.9E+02
Mn	ICP-AES	*3E+00*	*U*	7.65E+02		7.6E+02	4.2E+03
Mo	ICP-AES	*1.55E+01*	*J*	*1.55E+01*	*J*	4.6E+00	2.5E+01
Mo	ICP-MS	n/m		1.27E+01	J	1.3E+01	6.9E+01
Na	ICP-AES	7.32E+04		5.90E+04		7.4E+03	4.1E+04

Table S.2. (Cont'd)

		Supernatant Phase		WCS Phase		UDS	
Analyte	Measure Method	Average µg/mL	Data Flag (a)	Average µg/g	Data Flag (a)	Average µg/g (wet)	Average µg/g (dry)
Nd	ICP-AES	*6.45E+00*	*J*	1.80E+02		1.8E+02	9.6E+02
Ni	ICP-AES	1.22E+02		1.31E+02	X	4.5E+01	2.5E+02
NO_2	IC	2.59E+04		n/m		(c)	(c)
NO_3	IC	6.10E+04		n/m		(c)	(c)
NTA	Deriv/GC/FID	1.70E+02	JX	n/m		(b)	(b)
OH	Titration	4.60E+03		n/m		(b)	(b)
Oxalate	Organic/IC	1.80E+03		n/m		(b)	(b)
Oxalate (i)	IC	*2.29E+03*		n/m		(c)	(c)
P	ICP-AES	6.01E+02		1.06E+03		6.4E+02	3.5E+03
Pb	ICP-AES	4.90E+01	J	4.06E+02	B	3.7E+02	2.0E+03
Pd	ICP-AES	*4E+01*	*U*	5E+01	U	<5E+01	<3E+02
PO_4	IC	2E+02	U	n/m		(c)	(c)
Pr	ICP-MS	n/m		4.69E+01		4.7E+01	2.6E+02
Pt	ICP-MS	n/m		3E-02	U	<3E-02	<2E-01
Rb	ICP-MS	2.28E+00		1.18E+00	J	<1E+00 (j)	<5E+00 (j)
Rh	ICP-AES	*2E+01*	*U*	2E+01	U	<2E+01	<1E+02
Ru	ICP-AES	*6E+01*	*U*	7E+01	U	<7E+01	<4E+02
Sb	ICP-AES	*3E+01*	*U*	*3E+01*	*U*	<3E+01	<2E+02
Sb	ICP-MS	n/m		1.45E-01	J	1.5E-01	7.9E-01
Se	ICP-AES	*1E+01*	*U*	*2E+01*	*U*	<2E+01	<9E+01
Se	ICP-MS	n/m		1E+01	U	<1E+01	<5E+01
Si	ICP-AES	*2.80E+02*	*BX*	3.63E+03		3.4E+03	1.9E+04
SO_4	IC	4.43E+03		n/m		(c)	(c)
Sn	ICP-AES	*8E+01*	*U*	*9E+01*	*U*	<9E+01	<5E+02
Sr	ICP-AES	*8E-01*	*U*	2.40E+01		2.4E+01	1.3E+02
Succinic acid	Deriv/GC/FID	3E+01	UX	n/m		(b)	(b)
Ta	ICP-MS	n/m		2.27E-01	JBX	2.3E-01	1.2E+00
Te	ICP-AES	*8E+01*	*U*	*9E+01*	*U*	<9E+01	<5E+02
Te	ICP-MS	n/m		4.23E+00		4.2E+00	2.3E+01
Th	ICP-AES	*5E+01*	*U*	*1.60E+02*	*J*	1.6E+02	8.7E+02
Th	ICP-MS	n/m		6.29E+01		6.3E+01	3.4E+02
Ti	ICP-AES	*1E+00*	*U*	3.25E+00	J	3.3E+00	1.8E+01
TIC-F (e)	TIC Furnace	3.00E+00	J	n/m		(c)	(c)
TIC-P (e)	TIC Hot Pers.	6.00E+03		n/m		(c)	(c)
Tl	ICP-AES	*3E+01*	*U*	*3E+01*	*U*	<3E+01	<2E+02
Tl	ICP-MS	n/m		3.92E-02	J	3.9E-02	2.1E-01
TOC-F (e)	TOC Furnace	1.58E+04		n/m		(c)	(c)
TOC-P (e)	TOC Hot Pers.	9.50E+03		n/m		(c)	(c)
U	ICP-AES	1E+02	U	*3.20E+02*	*J*	3.2E+02	1.7E+03
U	KPA/ICP-MS (f)	1.50E+01		2.79E+02	X	2.7E+02	1.5E+03
V	ICP-AES	*3E+00*	*U*	*3E+00*	*U*	<3E+00	<2E+01
V	ICP-MS	n/m		5.38E+00	B	5.4E+00	2.9E+01
W	ICP-AES	*1E+02*	*U*	*1E+02*	*U*	<1E+02	<7E+02

Table S.2. (Cont'd)

		Supernatant Phase		WCS Phase		UDS	
Analyte	Measure Method	Average µg/mL	Data Flag [(a)]	Average µg/g	Data Flag [(a)]	Average µg/g (wet)	Average µg/g (dry)
W	ICP-MS	n/m		4.23E+01	X	4.2E+01	2.3E+02
Y	ICP-AES	*3E+00*	*U*	*2.60E+01*	*J*	2.6E+01	1.4E+02
Y	ICP-MS	n/m		2.58E+01		2.6E+01	1.4E+02
Zn	ICP-AES	*3.30E+00*	*J*	7.90E+01		7.7E+01	4.2E+02
Zr	ICP-AES	*3E+00*	*U*	7.80E+01	X	7.8E+01	4.3E+02

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

Results in italics represent analytes measured that were not listed in the TS (i.e., opportunistic analytes)

(a) Data flags: U = undetected above detection limit; J = estimated value; B = analyte in blank measured above acceptance criteria; X = QC deficiency
(b) Not calculated. Analysis of WCS for analyte not specified in TS.
(c) Not calculated. TS required analysis of analyte in WCS; however insufficient WCS available for analysis. BNI prioritized analyses to be performed on limited WCS material.
(d) 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), possibly formate or acetate.
(e) For TOC and TIC: P=by hot persulfate method; F=by furnace method/TIC by difference (TC – TOC).
(f) Uranium measured in the supernatant and centrifuged solids by KPA and ICP-MS, respectively.
(g) The total Cs concentration is estimated in the solids based on the assumption the Cs isotopic distribution in the solids is equivalent to the isotopic distribution in the supernatant. Concentration is thus based on the relative contribution of ^{137}Cs in the solids.
(h) Glycolate is not resolved from acetate by the IC method performed.
(i) Oxalate determined by inorganic IC method.
(j) The calculated UDS results <0; the UDS results set to <MDL of the WCS.

Table S.3. AN-102/C-104 Blended Feed – Radioisotope Summary

		Supernatant		WCS		Calculated UDS	
Analyte	**Measure Method**	**Average µCi/mL**	**Data Flag (a)**	**Average µCi/g**	**Data Flag (a)**	**µCi/g WCS**	**µCi/g DS**
^{60}Co	Rad	2.54E-02		2.09E-02		3.01E-03	1.64E-02
^{90}Sr	Rad	1.38E+01		4.17E+02		4.07E+02	2.22E+03
^{99}Tc	ICP-MS	4.24E-02		3.21E-02		2.23E-03	1.22E-02
^{99}Tc (pert.)	Rad	2.19E-02	X	n/m		(b)	(b)
^{125}Sb	Rad	n/m		2E-02	U	<2E-02	<1E-01
^{126}SnSb	Rad	n/m		1E-02	U	<1E-02	<5E-02
^{134}Cs	Rad	n/m		2E-03	U	<2E-03	<1E-02
^{137}Cs	Rad	1.02E+02		8.44E+01		1.26E+01	6.85E+01
^{152}Eu	Rad	n/m		2.17E-02		2.17E-02	1.18E-01
^{154}Eu	Rad	5.82E-02		1.22E+00		1.17E+00	6.41E+00
^{155}Eu	Rad	3.77E-02		7.22E-01		6.95E-01	3.79E+00
^{233}U	ICP-MS	n/m		1.12E-03		1.12E-03	6.11E-03
^{234}U	ICP-MS	n/m		1.27E-04	J	1.27E-04	6.90E-04
^{235}U	ICP-MS	n/m		4.40E-06		4.40E-06	2.40E-05
^{236}U	ICP-MS	n/m		6.32E-06		6.32E-06	3.45E-05
^{237}Np	ICP-MS	5.21E-05		6.01E-04		5.64E-04	3.08E-03
^{238}Pu	Rad	4.89E-04	BJ	2.63E-02		2.59E-02	1.41E-01
^{238}U	ICP-MS	n/m		9.29E-05		9.29E-05	5.07E-04
$^{239/240}$Pu	Rad	1.49E-03		1.12E-01		1.11E-01	6.05E-01
^{239}Pu	ICP-MS	1.72E-03	J	*1.39E-01*		1.37E-01	7.49E-01
^{240}Pu	ICP-MS	6.52E-04	J	*3.77E-02*	*J*	3.72E-02	2.03E-01
^{241}Am (GEA)	Rad	*3.70E-02*	*J*	1.16E+00		1.13E+00	6.16E+00
^{241}Am	Rad	3.75E-02		1.08E+00		1.05E+00	5.75E+00
^{241}Am/^{241}Pu (c)	ICP-MS	3.84E-02		n/m		(b)	(b)
^{242}Cm	Rad	1.55E-04	J	3.00E-03	J	2.89E-03	1.58E-02
$^{243/244}$Cm	Rad	1.92E-03	B	5.56E-02		5.43E-02	2.96E-01
Alpha	Rad	4.62E-02		1.34E+00		1.31E+00	7.14E+00
Sum of Alpha	Rad	4.15E-02		1.28E+00		1.25E+00	6.80E+00

WCS = wet centrifuged solids; UDS = undissolved solids; n/m = not measured

Result in italics represent analytes measured that were not listed in the TS; opportunistic analytes.

(a) J data flag indicates results that are >10% error and typically within 10 times the minimum detectable activity (MDA) for Rad and >MDL but <EQL for ICP-MS. Other data flags: B = analyte measured in blank above the EQL; U = undetected; X = QC deficiency.

(b) Not calculated due to the analyte not being measured for the WCS sample.

(c) ^{241}Am/^{241}Pu calculated by applying the specific activity of ^{241}Am to the mass-241 response; ^{241}Am used for calibration of the mass.

Reference dates extend from March 2001 to May 2001.

Table S.4 presents the summary of the AN-102/C-104 blended feed compared to Specification 7 Envelope C criteria. The table presents the results of the Specification 7 analytes of interest as a percentage of the Envelope C limit. For those analytes that were measured but not detected above the MDL, the results are presented as a 'less than' percent based on the MDL of the measured analyte. As shown in the table, none of the Specification 7 analytes exceed Envelope C limit. However, a few exceed 50% of the limit.

Table S.4. AN-102/C-104 Blended Feed --Specification 7 Envelope C Summary

Analytes Measured above MDL		**Analytes not Detected above MDL**	
Results % of Limit	**Analyte**	**Results % of Limit based on MDL**	**Analyte**
Table TS 7.1 Analytes			
82	TOC-F [(b)]	< 11	U
73	SO_4	< 5 or less[(a)]	PO_4, Ba
67	F		
59	TOC-P [(b)]		
52	TIC-P [(b)]		
46	NO_2		
39	NO_3		
28	Cl		
25	Al		
22	Ni		
16	PO_4 [(d)]		
11	Pb		
9	La		
6	Cr		
5 or less[(a)]	Ca, TIC-F[(b)], K, Cd, Fe		
Table TS7.2 Analytes			
80	^{60}Co		
27	^{137}Cs		
20	^{90}Sr		
16	^{154}Eu		
16	TRU [(c)]		
7	^{99}Tc		

(a) Analytes are presented from 5% in descending order.

(b) For TOC and TIC: P=by hot persulfate method; F=by furnace method/TIC by difference (TIC = TC-TOC).

(c) TRU = alpha emitting radionuclides with atomic number greater than 92 and half-life greater than ten years; Alpha summation of ^{238}Pu, $^{239+240}$Pu, ^{241}Am, and $^{243+244}$Cm.

(d) Phosphate based on ICP-AES average total P result of 601 µg/mL.

Terms and Abbreviations

AEA	Alpha Energy Analysis
AMU	atomic mass unit
ASR	Analytical Service Request
BNI	Bechtel National Inc.
BS	blank spike
D2EHP	bis-(2-ethylhexyl) phosphate
DI	deionized
DPP	diphenylphosphate
DS	dry solids
EDTA	ethylenediaminetetraacetic acid
ED3A	ethylenediaminetriacetic acid
EQL	estimated quantitation limit
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
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
IX	ion exchange
KPA	kinetic phosphorescence
LAW	low-activity waste
LCS	laboratory control standard
MDA	minimum detectable activity
MDL	method detection limit
MRQ	minimum reportable quantity
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
NIST	National Institute of Standards and Technology
N/M	not measured
N/R	not recovered
NTA	nitrilotriacetic acid
%D	percent difference
PB	process blank

PNWD	Battelle - Pacific Northwest Division
QA	quality assurance
QC	quality control
RPD	relative percent difference
RPL	Radiochemical Processing Laboratory
RPP	River Protection Project
RSD	relative standard deviation
SAL	Shielded Analytical Laboratory
SD	standard deviation
SRM	Standard Reference Material
TC	total carbon
TDS	total dissolved solids
TIC	total inorganic carbon
TOC	total organic carbon
TP	test plan
TRU	transuranic
TS	test specification
UDS	undissolved solids
UPA	MicroTrac™ Ultrafine Particle Analyzer
WCS	wet centrifuged solids
WTP	Waste Treatment Plant
X100	MicroTrac™ X-100 Particle Analyzer

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 gravities). 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 undissolved solids 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.

Units

°C	degree Centigrade
cP	centipoise
Bq	Becquerel
g	gram
G	gravitational force
µCi	microcurie
µg	microgram
µm	micrometer (micron)
mL	milliliter
M	molarity
N	normality
nm	nanometer
Pa	Pascal
rpm	revolutions per minute
s or sec	second
vol%	volume percent
W	watt
wt%	weight percent

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1.0 Introduction

The RPP-WTP has identified a reference process flowsheet [2] for treatment of LAW solutions and HLW sludge. To verify that Tank 241-C-104 (C-104) LAW solutions and Tank 241-AN-102 (AN-102) HLW sludge[3] can be treated using the reference process flowsheet, the PNWD is conducting integrated, process verification, and waste-form qualification tests for Bechtel National, Inc. (BNI) in support of the RPP-WTP. Testing includes sample compositing, homogenization, and as-received characterization of the AN-102 material, combination of the AN-102 waste material with tank C-104 permeate solutions to produce an AN-102/C-104 blended feed, blended feed characterization, Sr/TRU precipitation, Cs and Tc removal using IX, and vitrification of the resulting LAW and HLW streams. This report summarizes the blending of the AN-102 and C-104 materials and the physical and chemical characterization measurement performed on the resulting AN-102/C-104 blended feed.

This blended feed was measured for selected chemical, radiochemical, and organic analytes and the analyte concentrations compared to the chemical and radiochemical WTP envelope Specifications. All work was conducted per TP-41500-005 (Appendix A) in accordance with the requirements set forth in test specification (TS) TSP-W375-00-00007 (Appendix A). The primary objectives of this work were to:

- prepare a 2-wt% UDS AN-102 composite slurry from the as-received AN-102 sub-samples and measure for selected physical properties
- perform compatibility testing of the 2-wt% UDS AN-102 composite slurry with selected C-104 filtrates, leachates, and washes
- blend the 2-wt% UDS AN-102 composite slurry with the C-104 filtrates, leachates, and washes in proportions defined per the TS.
- ensure that blended feed material used for analysis and process testing is homogenized
- separate homogenized blended feed into the liquids fraction and solids fraction
- analyze the liquids and solids fractions for the inorganic, radiochemical, and selected organic constituents required for the process definition, while conforming to all appropriate laboratory quality control (QC) protocols and QC acceptance criteria.
- compare the results of the analyses on the supernatant to the WTP Specification 7. (This is an additional objective transmitted to the PNWD by BNI via e-mail and is not required by the TS).

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[2] Section C.7: Waste Treatment Plant Request for Proposals Solicitation No. DE-RP27-00RV14136, U.S. Department of Energy Office of River Protection, Richland, Washington, August 31, 2000.

[3] High Level Waste slurry from tanks AN-102 and C-104 were received by the PNWD from Hanford's 222-S laboratory in November 2000 and March 1999, respectively.

- calculate the analyte concentration of the UDS in the WCS. For the purposes of comparison of the as-received AN-102 material to the WTP Specification 8, the TS identifies “insoluble solids” as the total dried solids remaining after phase separating centrifuged solids minus the contribution to the dried solids from the interstitial liquid. For this report, “insoluble solids” are assumed to be the UDS, although some of the solids may be the result of salt precipitates from the saturated supernatant. (This is an additional objective transmitted to the PNWD to BNI via e-mail and is not required by the TS).

Selected samplings taken August 2000 from tank AN-102 were composited, homogenized, and sub-sampled in the High Level Radiation Facility (HLRF) hot cells located in the Radiochemical Processing Laboratory (RPL) as detailed in the Test Plan (TP-45100-0005; Appendix A). These as-received composite sub-samples were analyzed and the results reported in WTP-RPT-020 (Urie 2002). The as-received composite sub-samples were used to prepare an AN-102 composite slurry consisting of 2-wt% UDS per the TP. Selected physical properties measurements were performed on the 2-wt% UDS AN-102 composite and the results are included in this report.

Following blending compatibility testing which verified the compatibility of the AN-102 and C-104 blending materials (i.e., no visible gel formation or net solids increase occurred during blend testing), the 2-wt% UDS AN-102 composite slurry was blended with selected C-104 filtrate, wash and leachate solutions. The C-104 solutions resulted from small scale radioactive cross flow filtration, water washing, and caustic leaching tests (Brooks et al. 2000). The C-104 solutions were blended with the 2-wt% AN-102 composite slurry to produce a blended feed waste material as described in the TP. Sub-samples were then removed from this blended feed and the liquid (supernatant) and solid fractions were phase separated in the Shielded Analytical Laboratory (SAL) by centrifuging. The supernatant and centrifuged solids (dried) fractions were processed (as applicable) for analysis, then transferred to various laboratories for specific chemical, radioisotope, and organic analyses as defined in the analytical service request (ASR) 6025.00/01 and addendums to the ASR (Appendix B). The supernatant and centrifuged solids samples were given internal tracking numbers of 00-441 and 00-442, respectively. All analyses were run in duplicate unless noted otherwise.

Due to very low solids content of the AN-102/C-104 blended feed, all of the analyses listed in the TS could not be performed. The BNI contact prioritized the requested analytes and the following analyses were not performed on the centrifuged solids:

- ICP-MS for platinum, palladium, rhodium and ruthenium on fusion processed samples.
- IC for the inorganic ions fluoride, chloride, nitrite, nitrate, phosphate, and sulfate.
- TOC and TIC by hot persulfate and furnace methods.
- Uranium by kinetic phosphorescence analysis (KPA)

Also, due to the very limited quantity of solids available for characterization, the entire quantity of WCS collected were dried at 105°C to obtain a single wt% total solids result and the dried material was used for preparing samples for subsequent analysis. The analytical results for the solids fraction are reported on a wet weight basis by adjusting the analytical results for the wt% solids results.

The PNWD Quality Assurance (QA) Program's plan "Conducting Analytical Work in Support of Regulatory Programs" was used in support of all analytical operations and is compliant with the Hanford Analytical Service Quality Assurance Requirements Documents (HASQARD) DOE/RL-96-68. The inorganic, radioisotopic, and organic analytes tested were identified in the TS. The quality requirements for both the supernatant and solids fractions were included in the TS and transmitted to the laboratory staff via ASR 6025.01.

Data and results limitations are described. Physical measurements performed on the diluted AN-102 waste material, and the initial compatibility testing, blending parameters, and chemical, radioisotope, and organic analytical results on the AN-102/C-104 blended feed are reported. The QC sample results, method detection limits (MDL), and other QC indicators are described in Section 6.0, Procedures, Quality Control, and Data Evaluation.

2.0 Initial Sample Processing

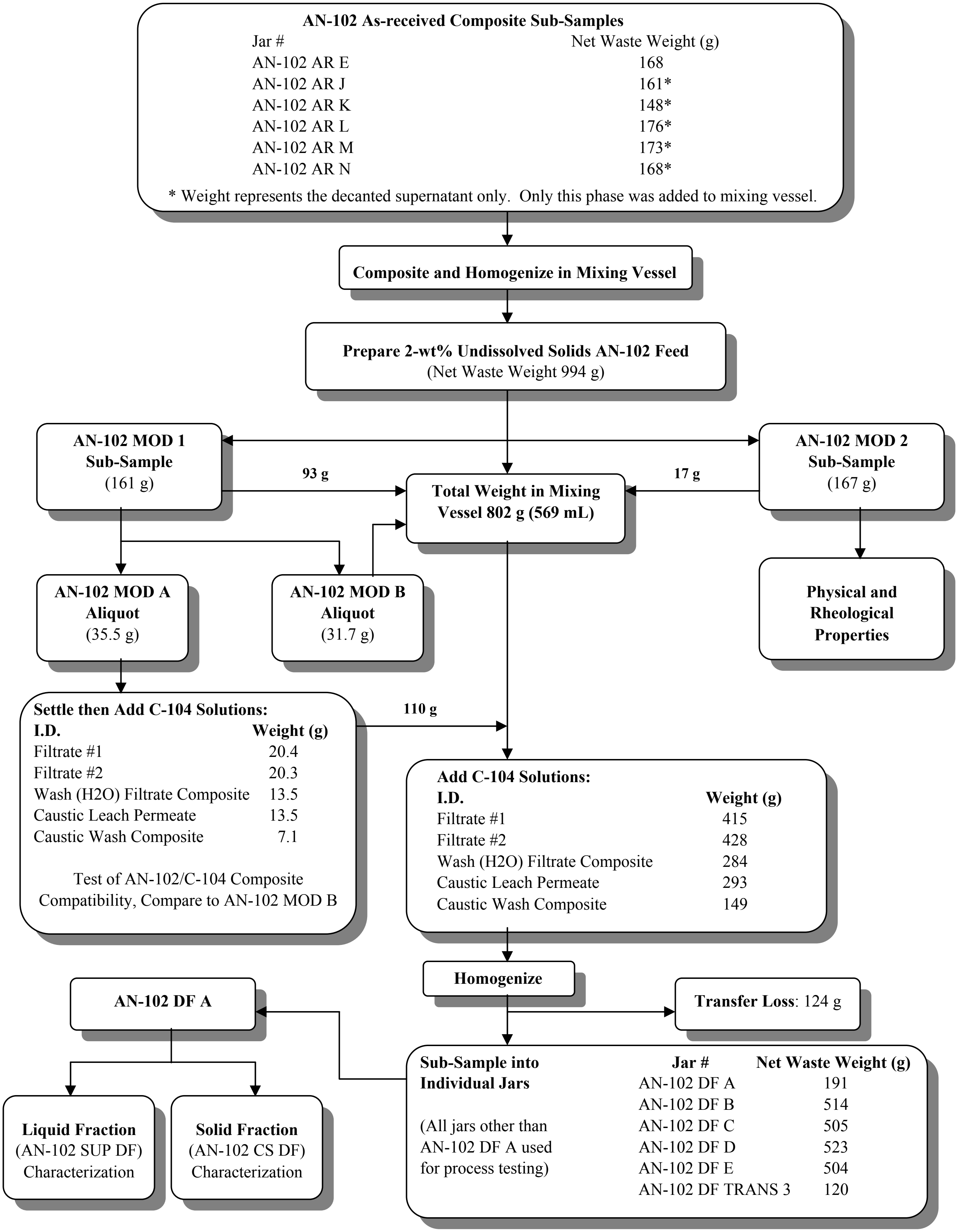
2.1 Overall Process Description

Preparation of the AN-102/C-104 blended feed is illustrated in Figure 2.1. The figure includes the AN-102 as-received composite jar descriptions, the dilution of the AN-102 as-received composite to a nominal 2-wt% insoluble solids, identification of the C-104 samples used for blending, the blending compatibility testing information, and the description of the final AN-102/C-104 blended feed sub-samples collected. All sample processing was conducted per to TP TP-41500-005 (Appendix A) per the requirements set forth in TS TSP-W375-00-00007 (Appendix A).

The AN-102 as-received waste was composited, homogenized, and sub-sampled as reported in Urie, et al. (2002). Using the as-received composite sub-samples as a starting basis, an AN-102 composite slurry consisting of from 2 to 5 wt% (hereafter referred to as 2-wt%) UDS was prepared by diluting one of the AN-102 as-received sub-samples with the supernatant from five additional AN-102 as-received sub-samples. Selected physical measurements (i.e., shear stress versus shear rate, heat capacity, and particle size) were performed on the homogenized 2-wt% AN-102 composite slurry and the results compared to the TS expected ranges.

Blending compatibility testing was conducted on the homogenized 2-wt% AN-102 composite slurry and selected filtrate, wash, and leachate solutions derived from waste tank C-104 to ensure that mixing of these waste material would not create gels or excessive solids. The TS provided specific instructions on the volume ratios of C-104 solutions and the 2-wt% AN-102 slurry to be blended. The C-104 additive solutions were retrieved from prior processing of the C-104 waste material and the pedigree of the C-104 filtrate, wash, caustic-wash, and caustic-leach solutions is described by Brooks, et al. (2000). A flow sheet from the Brooks report detailing the C-104 additive solutions is provided in Appendix A.

Following the successful compatibility testing, the approximately 2 kg of AN-102/C-104 blended feed was prepared. The blended feed was homogenized and sub-sampled for physical testing, chemical and radioisotope characterization, and further process testing. For chemical and radioisotope characterization, the AN-102/C-104 blended feed sample was phase separated (by centrifuging) so that the characterization could be performed on the supernatant and solid phases individually according to the TS.



AN-102 As-received Composite Sub-Samples

Jar #	Net Waste Weight (g)
AN-102 AR E	168
AN-102 AR J	161*
AN-102 AR K	148*
AN-102 AR L	176*
AN-102 AR M	173*
AN-102 AR N	168*

* Weight represents the decanted supernatant only. Only this phase was added to mixing vessel.

Settle then Add C-104 Solutions:

I.D.	**Weight (g)**
Filtrate #1	20.4
Filtrate #2	20.3
Wash (H2O) Filtrate Composite	13.5
Caustic Leach Permeate	13.5
Caustic Wash Composite	7.1

Test of AN-102/C-104 Composite Compatibility, Compare to AN-102 MOD B

Add C-104 Solutions:

I.D.	**Weight (g)**
Filtrate #1	415
Filtrate #2	428
Wash (H2O) Filtrate Composite	284
Caustic Leach Permeate	293
Caustic Wash Composite	149

Sub-Sample into Individual Jars

(All jars other than AN-102 DF A used for process testing)

Jar #	**Net Waste Weight (g)**
AN-102 DF A	191
AN-102 DF B	514
AN-102 DF C	505
AN-102 DF D	523
AN-102 DF E	504
AN-102 DF TRANS 3	120

Figure 2.1. Flow Diagram for Preparation of AN-102/C-104 Blended Feed

2.2 AN-102 Sample Dilution with AN-102 Supernatant

The TS required the preparation of an AN-102 composite feed material containing 2-wt% UDS. The UDS content of the AN-102 composite slurry was estimated using the data produced from the AN-102 as-received composite bottle AN-102-AR H and reported in Urie, et al. (2002). Table 2.1 replicates Table 4.2 from that report.

Table 2.1. AN-102 Homogenized Slurry Composite Sample Percent Solids and Densities

	Sub-Sample ID						
	AN-102 AR-H A	AN-102 AR-H B	AN-102 AR-H C	AN-102 AR-H D	Average	Std Dev (1-σ)	RSD
Density (g/mL)							
Slurry	1.494	1.500	1.457	1.491	1.485	0.02	1%
Supernatant	1.405	1.434	1.435	1.423	1.424	0.02	1%
Settled Solids	1.48	1.49	n/m	n/m	1.49	n/a	n/a
Wet Centrifuged Solids	1.550	1.571	1.518	1.619	1.564	0.04	3%
Weight Percent Solids (%)							
Centrifuged Slurry	50.0	51.8	47.1	47.4	49.1	2	4%
Volume Percent Solids (%)							
Settled Slurry	71.3	70.2	n/m	n/m	70.8	n/a	n/a
Centrifuged Slurry	47.3	48.6	45.3	43.6	46.2	2	5%
Weight Percent Dissolved Solids in Supernatant and Weight Percent Total Solids in Centrifuged Solids (%)							
Solids Wt % in Supernatant After Drying at 105°C	50.6	50.2	50.3	50.3	50.4	0.2	0.4%
Solids Wt% in Solids After Drying at 105°C	63.7	64.5	60.3	63.8	63.1	2	3%

RSD = relative standard deviation; n/m = not measured; n/a = not applicable

Equation 2.1 was used to estimate the wt% UDS in the AN-102 as-received composite slurry.

$$UDS = \left[1 - \frac{(1-W)}{(1-T)}\right] * Z * 100 \qquad \text{(Equation 2.1)}$$

Where, UDS = wt% undissolved solids (%)
W = mass fraction of solids in centrifuged solids,
(i.e., Solids Wt% in Solids after Drying at 105°C / 100)
T = mass fraction of solids in supernatant,
(i.e., Solids Wt% in Supernatant after Drying at 105°C / 100)
Z = mass fraction of centrifuged solids in composite slurry,
(i.e., Wt% Solids in Centrifuged Slurry / 100)

Based on Equation 2.1, the UDS for the A, B, C, and D aliquots is 13.3%, 14.9%, 9.6%, and 12.9%, respectively, with the average being 12.6% or 13.7% if one discounts the 9.6% result. Since the 9.6% is significantly lower than the other values, 13.7% was used as the estimated UDS content of the AN-102 as-received composite slurry. Although no statistical criterion was used to discard the 9.6% result, the use of the 13.7% UDS value to prepare the 2-wt% UDS AN-102 composite slurry appears to be supported by the 'calculated UDS' in Table 2.3 and the 'measured UDS' in Table 2.4.

Using six bottles of the AN-102 as-received composite slurry sub-sampled as part of the initial AN-102 compositing, homogenization, and sub-sampling effort (Urie et al. 2002) and the estimated UDS content for the AN-102 as-received composite slurry, a 2-wt% UDS AN-102 composite slurry was prepared. One AN-102 as-received sub-sample (AN-102 AR-E) was combined with the supernatant of five additional AN-102 as-received sub-samples (AN-102 AR-J through -N) to create the slurry. Prior to preparing the 2-wt% composite feed, the solids in each bottle were allowed to settle for a minimum of three days. After the solids were settled, the volume percent (vol%) of each sub-sample was determined. The average vol% settled solids was 69% with a standard deviation (SD) of 1.5%, which met the homogeneity acceptance criterion of <5% for the SD (Table 2.2).

Table 2.2. AN-102 As-received Composite Samples Used For Preparing 2-Wt% Feed

Sub-Sample ID	Sample Mass (g)	Sample Volume (mL)	Vol% Visual Settled Solids
AN-102 AR-E	168	116	71.1
AN-102 AR-J	585	393	67.7
AN-102 AR-K	590	396	70.1
AN-102 AR-L	597	402	68.4
AN-102 AR-M	605	408	68.1
AN-102 AR-N	605	405	67.2
Sum	3,150 g	2,120 mL	n/a
Average	n/a	n/a	68.8%
SD	n/a	n/a	1.5%

After determining the sub-samples were homogenous, the supernatant from the five sub-samples AN-102 AR-J through -N were decanted into the mixing vessel and combined with the entire sub-sample AN-102 AR-E (Table 2.3). Sample AN-102 AR-E contained approximately 168 g of as-received composite. Assuming that the as-received composite contains approximately 13.7% UDS, AN-102 AR-E contained about 23 g of UDS. Based on the quantity of supernatant added to AN-102 AR-E, the estimated final UDS content of the 2-wt% feed is approximately 2.3% [i.e., (23 g / (23 g + 973 g)) * 100].

Table 2.3. Preparation of 2-Wt% Undissolved Solids AN-102 Composite Feed

Sub-Sample ID	Wet Centrifuged Solids (g)	Liquid (g)
AN-102 AR-E	23	144
AN-102 AR-J		161
AN-102 AR-K		148
AN-102 AR-L		176
AN-102 AR-M		173
AN-102 AR-N		168
Total	23	973
Wt% UDS	2.3%	

The 2-wt% UDS feed material was stirred for approximately 60 minutes. With the impeller running, a 100-mL portion was removed through the ¾-inch valve located at the bottom of the vessel to flush the valve and connecting tubing. The flush material was poured back into the mixing vessel, and then two approximately 100-mL aliquots of the homogenized 2-wt% AN-102 composite feed material were collected (AN-102 MOD 1 and AN-102 MOD 2). The material in AN-102 MOD 1 was used for the solubility/compatibility testing and AN-102 MOD 2 was retained for analysis of solids content, rheology testing, and other physical measurement.

To confirm the solids content of the 2-wt% AN-102 composite slurry, three sub-samples (A, B, and C) of approximately 14 g were sub-sampled from AN-102 MOD 2 and analyzed for solids content using the same protocol as used for determining the solids content of the AN-102 as-received composite. Results of the testing are presented in Table 2.4.

Table 2.4. Solids Analysis of 2-Wt% Slurry Feed Taken After Homogenization

	Sub-Sample ID					
	AN-102 MOD2 A	**AN-102 MOD2 B**	**AN-102 MOD2 C**	**Average**	**RSD**	**RPD**
Density (g/mL)						
Slurry	1.403	1.431	1.400	1.411	1%	
Wet Centrifuged Solids	1.45	1.53	n/m	1.49		5%
Weight Percent Solids (%)						
Centrifuged Slurry	14.3	13.9	n/m	14.1		7%
Volume Percent Solids (%)						
Settled Slurry	21.1	22.8	n/m	22.0		6%
Centrifuged Slurry	13.8	13.0	n/m	13.4		5%
Weight Percent Dissolved Solids in Supernatant and Weight Percent Total Solids in Centrifuged Solids (%)						
Solids Wt % in Supernatant After Drying at 105°C	53.4	52.4	48.8	51.5	5%	
Solids Wt% in Solids After Drying at 105°C	54.6	54.3	49.9	52.9	5%	
Weight Percent Undissolved Solids (%)						
Wt% UDS per Equation 2.1	2.5	2.9	2.0	2.4	17%	

RSD = relative standard deviation; RPD = relative percent difference; n/m = not measured

Using the Equation 2.1, the wt% UDS for the 2-wt% AN-102 composite slurry material for A, B, and C is 2.5%, 2.9%, and 2.0%, respectively, with the average being 2.4% with an RSD of 17%. The average of 2.4% UDS is very close to the estimated 2.3% based on the blending detailed in Table 2.3.

2.3 Physical Measurement of 2-Wt% UDS AN-102 Composite Slurry

2.3.1 Rheology

Rheology testing of the 2-wt% UDS AN-102 slurry (AN-102 Mod 2) was performed with the Haake® M5 head in the HLRF. The rheology testing produced a standard set of shear stress vs. shear rate curves.

2.3.1.1 Background

Viscosity is the internal resistance to flow of a fluid against external forces. Viscosity is mathematically defined as the shear stress divided by the shear rate. For a Newtonian fluid this ratio is constant. For non-Newtonian fluids this ratio can change based on flow conditions and shear history. High-molecular-weight liquids, slurries, and suspensions are often non-Newtonian, in that the viscosity is a function of shear rate. The rheological data most often requested and provided is 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 a rheogram viscosity data, yield stress data and flow curve information are obtained. Viscosity is usually reported in centipoises (cP). One cP is equal to a millipascal second. There are several types of 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:

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

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

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

4) Herschel-Bulkley Equation
(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, $^1/_s$)
η = Viscosity (Pascal-seconds, Pa-s; reported in cP)
η_p = coefficient related to flow resistance, similar to 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

Classic examples of Newtonian fluids are water and honey. The viscosity is a constant over all shear conditions. A Bingham plastic is a fluid that contains a yield point but 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 pseudo-plastic, or power law fluid, has a viscosity that varies with stress in a non-linear fashion. A yield pseudo-plastic is a power law fluid with a yield point.

2.3.1.2 Equipment Capabilities and Sensor Selection

The Haake® 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 design specification of the Haake® M5 head gives it a maximum deflection of 1% of full torque with a sensitivity range of up to 0.001^{o} 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 Haake® NV sensor geometry determines the optimum viscosity and shear rate ranges available for any given measurement. For the 2-wt% UDS AN-102 slurry samples, the Haake® NV sensor system was utilized. This sensor system has the most available surface area and therefore yields the highest sensitivity, which is important for low viscosity fluids. The small gap size of the NV sensor limits use to material with low solids concentrations. This measurement head and sensor combination has an optimum effective viscosity range of 1 cP to 10,000 cP over a shear rate range of 0 $^{1}/_{s}$ to 2700 $^{1}/_{s}$. A 100 cP standard oil is used to validate the calibration of the machine.

2.3.1.3 Sample Testing Parameters

For the 2-wt% UDS AN-102 slurry samples a standard 0 to 1000 $^{1}/_{s}$ ramp test over 6 minutes was completed for each sample. Several samples were ramped over a period of 20 minutes with no discernable difference in observed behavior; therefore, 6 minutes was chosen as the ramp time for the remaining analyses. Each sample was tested at least twice. The pertinent resulting rheograms and viscosity curves are included in Appendix B.

The samples were loaded and then ramped up from 0 to 1000 $^{1}/_{s}$ in 3 minutes and then 1000 to 0 $^{1}/_{s}$ in 3 minutes. There was a repeat of this ramp cycle at least once for each sample. Thus each sample was tested through a minimum of two complete ramp cycles from 0 to 1000 $^{1}/_{s}$ over a total time of 6 minutes. If the second run data was a close overlay of the first run data, then the testing for that sample was considered complete. If there was a noticeable variation in the data, then the sample was ramped though this cycle again until two consecutive similar data sets were obtained. This repetition is 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.

The purpose of this set of testing parameters was to identify the rheological behavior and shear sensitivity of the materials. 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 with the used sample shows the effects of a shear history with a short time of recovery for the sample.

The 2-wt% UDS AN-102 slurry samples were tested at three separate temperatures, 25°C, 35°C and 50°C to evaluate the temperature effects on viscosity and flow curves at possible expected process conditions.

2.3.1.4 Sample Rheology Results

Each run of the 2-wt% UDS AN-102 slurry samples showed a pseudoplastic nature with little or no yield point. 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. Table 2.5 presents viscosity data calculated at specific shear rates.

As shown by Figures 2.2 and 2.3, there was a drop in the fluid resistance (i.e., viscosity) with increasing temperatures. The apparent viscosity decreased approximately 50% when the temperature was increased from 25°C to 35°C. The continued rise in temperature up to 50°C resulted in a further decrease of the viscosity but not such a significant one. The change in temperature did not change the flow profile to any relevant extent.

Table 2.5. Viscosity at Specific Shear Rates; 2-Wt% UDS AN-102

	Yield (a)	**Viscosity (cP) (b)**					
Material	**(Pa)**	**@33 $^1/_s$**	**@150 $^1/_s$**	**@300 $^1/_s$**	**@500$^1/_s$**	**@750 $^1/_s$**	**@990 $^1/_s$**
AN-102 25°C Run 1	<2	34.4	22.6	22.0	19.7	17.2	15.4
AN-102 25°C Run 2	<2	26.5	22.3	21.5	18.5	16.9	15.5
AN-102 35°C Run 1	<2	33.3	15.6	15.4	12.7	11.8	10.7
AN-102 35°C Run 2	<2	16.5	13.2	12.9	11.3	11.0	10.2
AN-102 50°C Run 1	<2	19.6	15.7	12.5	11.6	10.7	9.9
AN-102 50°C Run 2	<2	17.8	13.0	12.5	11.1	10.3	9.9

(a) Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits, the numbers are not exact, and when there is a disparity between multiple runs the highest yield is reported to be conservative. A <2 results indicates the material may have a small yield but it is insignificant.

(b) Viscosity data is in cP and is a specific number selected at a specific shear rate for each run.

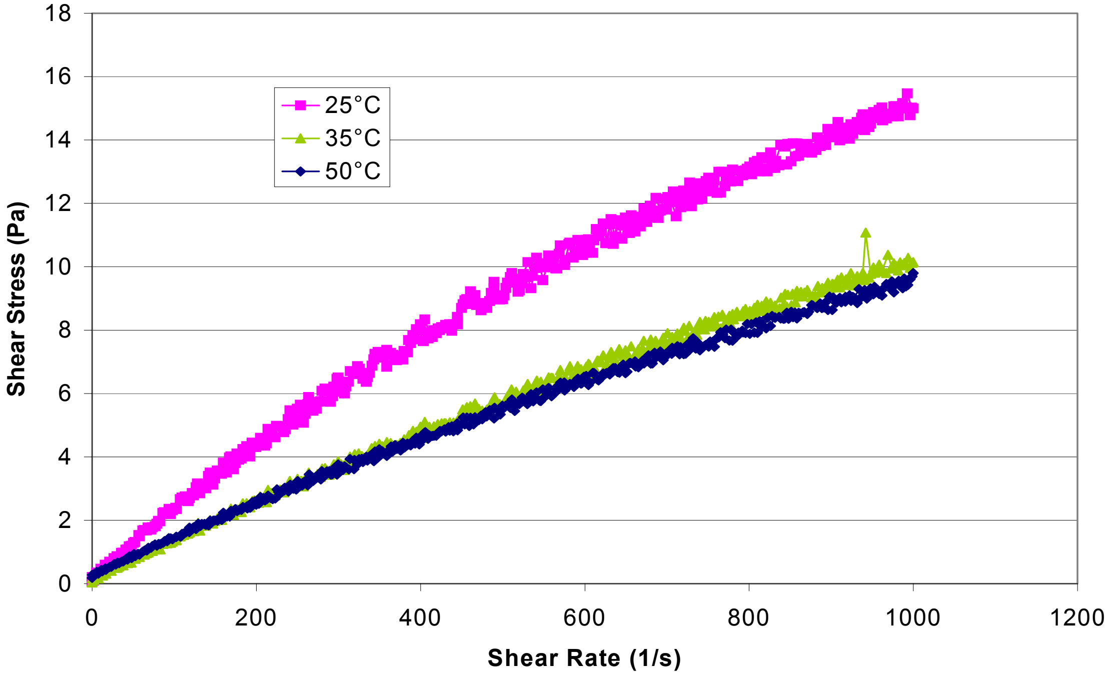


Figure 2.2. Shear Stress Versus Shear Rate for 2-Wt% UDS AN-102 Slurry

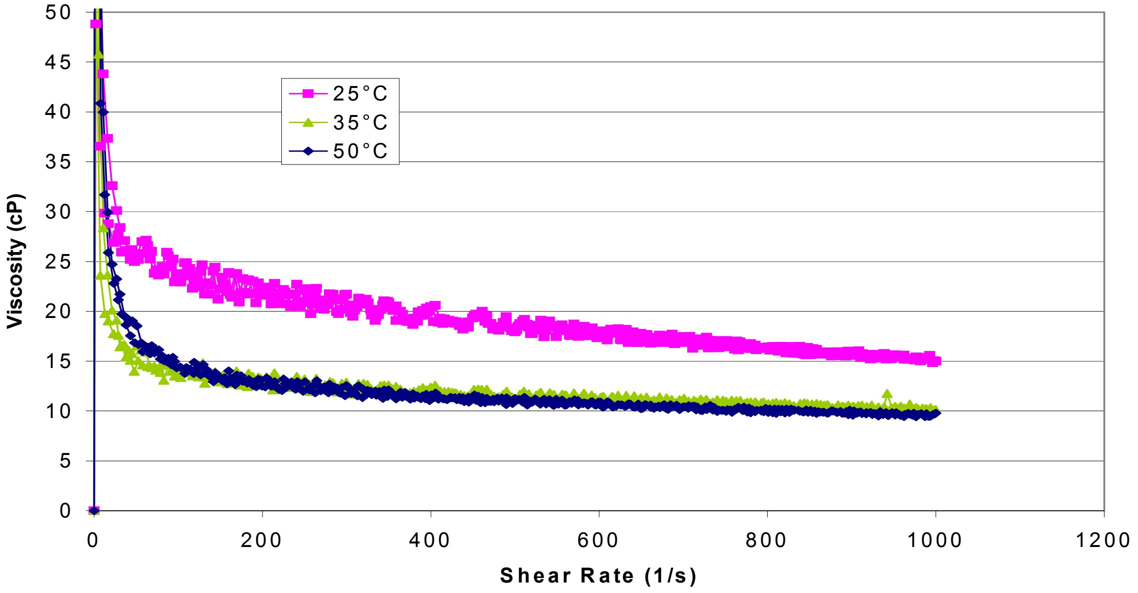


Figure 2.3. Viscosity Versus Shear Rate for 2-Wt% UDS AN-102 Slurry

The Haake® M5 system with NV sensor 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 itself rather than actual fluid resistance. It is likely that this contributed to the small "yield stress" seen 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. For example, at 33 $^{1}/_{s}$ shear rate the 35°C samples had vastly different "single point" values (i.e., 33 cP and 16 cP). However the overall curve data is almost identical. It is better to use the curve equations to predict the viscosities at any given shear rate than to use singular data points.

2.3.1.5 Rheology Curve Fits

Based on the data in Table 2.6, the best curve fits for the material were found to be Herschel-Bulkley or Ostwald fits with an 'n' factor of > 0.8 and often above 0.9 for most of the fits. The 'n' factor is a measure of the degree of pseudo-plasticity or deviation from Newtonian the fluid exhibits. For Newtonian fluids the 'n' factor equals unity. The 'n' factor of greater than 0.8 for the 2-wt% UDS AN-102 slurry composite indicates the pseudo-plastic nature of this material is present, but is slight. The Herschel-Bulkley model does have a yield factor, but in this case it is most likely an artifact of the Haake® system due to the need to overcome inertial mechanical forces and can be neglected. Given the lack of the observed yield stress in the Herschel-Bulkley model, an Ostwald model is the most accurate overall model for this material. The Newtonian model was not evaluated since all samples demonstrated some degree of pseudo-plasticity.

Table 2.6. Model Fit Parameters

Sample [a]	**Ostwald**			**Herschel-Bulkley**				**Bingham**		
	η_p	**n**	R^2	τ_o	η_p	**n**	R^2	τ_o	η_p	R^2
25°C Run 1	0.05943	0.8135	1.00	-0.1162	0.07828	0.7713	1.00	1.592	0.01478	0.98
25°C Run 2	0.05726	0.8166	1.00	0.0840	0.04164	0.8666	0.99	1.486	0.01473	0.98
35°C Run 1	0.08108	0.7081	0.97	0.2102	0.03547	0.8358	0.91	1.390	0.009876	0.98
35°C Run 2	0.03030	0.8443	0.99	0.05816	0.02214	0.8931	1.00	0.6015	0.01006	0.99
50°C Run 1	0.1207	0.6236	0.88	0.8383	0.0121	0.9631	0.76	1.220	0.00891	0.94
50°C Run 2	0.05002	0.7581	0.98	0.2362	0.0158	0.9362	1.00	0.7177	0.009415	0.99

[a] Results for first and second run at each temperature presented. Data for additional runs is included in Appendix B.

2.3.2 Heat Capacity

2.3.2.1 Background

A differential scanning calorimeter (DSC) was used to measure the heat capacity. The DSC measures enthalpy (heat) changes either 1) as the temperature is increased at a known and constant rate or 2) at constant temperature by measuring differences between heat requirements between a sample and a reference located in a second sample holder. The heat capacity was measured on 2-wt% UDS AN-102 slurry (sample AN-102 Mod2-1) with the nominal sample size being 10 mg.

2.3.2.2 Sample Testing Parameters and Model

For the heat capacity measurement, a three-step approach is generally used. First the empty sample pan is heated at the starting temperature, held for 10 minutes, then heated over the temperature range of interest at a controlled rate, and then held at the final temperature for 10 minutes. Second, the sample pan is filled with roughly the same amount of reference material as will be used for the sample and the same temperature program repeated. The reference material is removed and replaced with the sample and the same temperature program repeated. The heat capacity of the sample is calculated based on differences between the reference and the baseline and differences between the sample and the baseline taking into account differences in reference and sample masses. Typically, the reference material is similar in state and mass to the samples to be analyzed; e.g., typically water is used for aqueous samples and sapphire is used for solids samples.

The temperature program used was to 1) heat to 30°C, 2) hold at 30°C for 10 minutes, 3) heat to 120°C at 5°C/min, and 4) hold at 120°C for 10 min. The analysis of aqueous slurry samples complicates heat capacity measurements because of water's volatility between room temperature and 100°C and above. To accommodate water's volatility and eliminate enthalpy changes due to evaporation, the water reference and aqueous slurry samples were contained in sealed gold sample pans. The use of sealed sample pans prevented the reuse of the same sample pan for baseline, water reference standard, and sample.

The 2-wt% UDS AN-102 slurry sample material was principally an aqueous sample with the possibility of some solids, which presented a particular challenge because of the temperature range of interest (room temperature to above 100°C) and the volatility of water in that range. To prevent water evaporation and eliminate associated heat losses, which would mask measurement of the heat capacity, a 10 mg deionized (DI) water reference and 10 mg 2-wt% UDS AN-102 slurry sample were placed in hermetically sealed gold pans, with a sealed volume of approximately 15 μL. The DI water reference was encapsulated in its own gold pan; the DI water reference was reused for each analysis and weighed between analyses to ensure no water loss. A linear least squares regression of the heat capacity of water from 300 K (30°C) to 473 K (100°C) (Weast 1984) was used to model the DI water reference standard's heat capacity at constant pressure (C_p); e.g. $C_p = 0.000562*T+3.9998$ J/(g K). This model was used to extrapolate above the boiling point of water based on the assumption that the bulk of the water remained as liquid until the sealed pan ruptures; a rupture would be observed as a major endotherm by the DSC. The model was used to calculate the reference C_p at each data point's temperature; data points were taken by the instrument every 0.4°C. Each 2-wt% UDS AN-102 slurry sample analyzed was encapsulated in the same pan used for its baseline (i.e., empty pan) determination, and it was assumed that the behavior for these pans is equivalent to the behavior of the pan used for the DI water reference.

2.3.2.3 Sample Heat Capacity Results

The temperature program used for the triplicate analysis of the AP-101 diluted feed was to 1) heat to 30°C, 2) hold at 30°C for 10 minutes, 3) heat to 120°C at 5°C/min, and 4) hold at 120°C for 10 min. High purity water, which is recommended by the equipment manufacturer as the reference for aqueous samples, was used as a reference for the 2-wt% UDS AN-102 slurry sample analysis. The results are presented in Table 2.7 and Figure 2.4 and provide the average measured heat capacity of the 2-wt% UDS AN-102 slurry sample, 2.78 J/(g K). Figure 2.4 also provides a linear regression of the average heat capacity as a function of temperature. The linear least squares regression of data from the three analyses yields Equation 2.2 with a 95% confidence interval of ±0.12 J/(g K).

$$C_p = (-0.0018 * T) + 3.42 \qquad \text{(Equation 2.2)}$$

Where, C_p = heat capacity at constant pressure (J/(g K)
T = temperature in degrees Kelvin.

As shown in Table 2.7, the heat capacity of the 2-wt% UDS AN-102 is lower than the heat capacity for water at the same temperature range. This is likely due to the dissolved solids in the sample, which typically have much lower heat capacities than water. For example, sodium nitrate at 300 K has a C_p of 1.10 J/(g K) compared to liquid water's C_p of 4.18 J/(g K). The slight negative slope is unexpected. Rough calculations indicate that changes in the distribution of gaseous and liquid water (i.e., evaporation into the 5 μL container headspace) is not responsible for the negative slope; gaseous water at 300 K has a C_p of 1.88 J/(g K) or about half that of liquid water.

Table 2.7. Measured Heat Capacity of 2-Wt% UDS AN-102 Slurry

T, K	**Analysis 1 Cp J/(g K)**	**Analysis 2 Cp J/(g K)**	**Analysis 3 Cp J/(g K)**	**Average Cp J/(g K)**	**Water Cp J/(g K)**
310	2.86	2.89	2.75	2.83	4.17
315	2.87	2.89	2.75	2.83	4.18
320	2.84	2.87	2.74	2.82	4.18
325	2.84	2.89	2.72	2.82	4.18
330	2.83	2.86	2.73	2.8	4.19
335	2.81	2.83	2.8	2.82	4.19
340	2.79	2.84	2.75	2.79	4.19
345	2.82	2.83	2.75	2.8	4.19
350	2.78	2.82	2.67	2.76	4.2
355	2.76	2.82	2.63	2.74	4.2
360	2.73	2.82	2.66	2.74	4.2
365	2.74	2.81	2.61	2.72	4.2
370	2.73	2.72	2.67	2.71	4.21
375	2.73	2.75	2.67	2.72	4.21
380	2.77	2.74	2.69	2.74	4.21
382	2.83	2.83	2.68	2.78	4.22
			Average Heat Capacity	2.78	

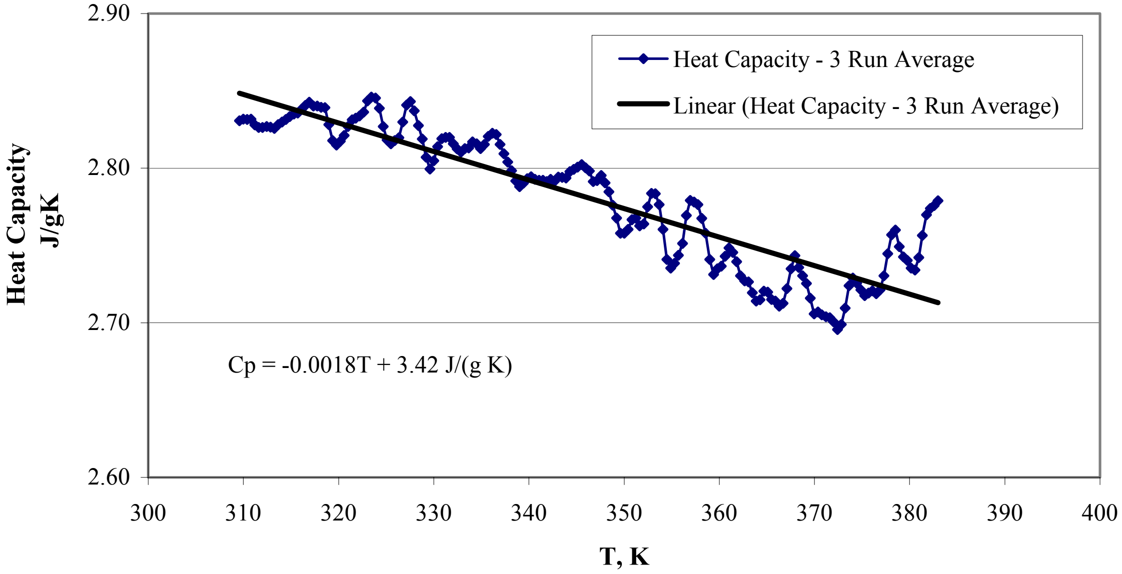


Figure 2.4. Average Heat Capacity of 2-Wt% UDS AN-102 Slurry

2.3.3 Particle Size

2.3.3.1 Background

The particle size distribution measurement of the 2-wt% UDS AN-102 slurry was performed on the sample labeled AN-102 MOD2. A Microtrac™ X-100 (X100) Particle Analyzer and a Microtrac™ Ultrafine Particle Analyzer (UPA) were both used to measure particle size distribution. The Microtrac™ X-100 Particle Analyzer determines particle diameter by measurement of 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 is 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 700 μm. The Microtrac™ UPA measures particle diameter by Doppler shifted scattered light. This method is limited to particles with diameters between 3 nm and 6.5 μm. The particle size distribution was measured on small aliquots of 2-wt% UDS AN-102 slurry (sample AN-102 MOD2) suspended in a simulant representative of AN-102 supernatant.

2.3.3.2 Sample Testing Parameters and Suspension Simulant Medium

The particle size distribution of the 2-wt% UDS AN-102 sample was measured in the X100 at an initial flow rate of 40 mL/s. The flow rate was increased to 60 mL/s and the particle size distribution was measured. Then the sample was sonicated with 40 W ultrasonic waves for 90 seconds at a flow rate of 60 mL/s and the particle size distribution was measured. Finally, the sample was sonicated a second time with 40 W ultrasonic waves for 90 second at a flow rate of 60 mL/sec and the particle size distribution was measured. Sonication was performed to separate loose agglomerates and to emulate the effects of pulse-jet or mixer pump mixing during normal feed processing. In the X100 instrument, the particle size analyses were performed in triplicate on each sample under all flow/sonication conditions. The average of these triplicate measurements are reported. Samples for particle size analysis by the UPA were sub-sampled both prior and after sonication; however, only a sample and duplicate measurement were performed on the sub-samples.

The suspending medium for these analyses was a simulant supernatant based on the ICP and IC data obtained for the AN-102 supernatant liquid. The composition of the simulant supernatant liquid is reported in Table 2.8.

Table 2.8. Simulant Supernatant Composition

Component	Concentration (M)	Component	Concentration (M)
$NaNO_3$	1.51	NaCl	0.107
NaOH	4.39	$NaNO_2$	2.31
$Al(NO_3)_3 \bullet 9H_2O$	1.03	$NaCO_3$	1.00
Na2SO4	0.135	$Na_2C_2O_4$	0.00637
$Na_2HPO_4 \bullet 7H_2O$	0.0453	NaF	0.0120

The performance of each instrument was checked against a range of NIST-traceable standards from Duke Scientific Corporation. These standards are polymer microspheres dispersed in a 0.001M potassium chloride solution. These standards were run prior to analysis of the sample. To show that the instrument worked properly throughout the experiment, the standards were run again after the analysis of the sample. Results from these standard tests are presented in Table 2.9. The percentile data shown in the table represent the given percent of the volume (or weight 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. The 20 μm calibration check standard for the X100 (before and after) and the 895 μm for the UPA (before) do not meet the PNWD QA Plan requirement of "±10% of expected values"; however, the X100 demonstrates a consistent high bias for the low 20 μm standard. The reason for the UPA high 895 μm standard is unknown, but is bias high due to large volume particles.

Table 2.9. Calibration Check Standards

	X100 Instrument				**UPA Instrument**			
	Before Analysis		**After Analysis**		**Before Analysis**		**After Analysis**	
	Size (μm)	**Size (μm)**	**Size (μm)**	**Size (μm)**	**Size (nm)**	**Size (nm)**	**Size (nm)**	**Size (nm)**
Standard Mean Size	20	301	20	301	96	895	96	895
Measured Mean Size (a)	24.5	329	25.6	300	101	1178	101	907
10 vol% < Size	17.7	237	17.3	250	87	745	83	712
50 vol% < Size	23.5	301	23.5	282	100	885	100	901
90 vol% < Size	32.6	476	36.6	333	117	1085	120	1110

(a) Mean Particle Size Calculated on a Volume Basis

2.3.3.3 Sample Particle Size Results

Table 2.10 and Table 2.11 contain a summary of the particle size analysis from the X100 instrument on a volumetric and numeric basis, respectively. This data presented in these tables 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 data presented in Table 2.10 indicate a large volume of particles in the 0.5 to 15.0 micron range. The peak value is at about 4.0 microns. At the low shear rates (i.e. 40 mL/s recirculation flow rate), larger particles in the range of 15 to 300 microns are present. At this shear rate these particles represent about 26% of the total volume. As the shear rate increases (due to increasing the flow rate from 40 mL/s to 60 mL/s), the volume of large particles is reduced significantly to about 6% of the total volume in the 15 to 40 micron size range. Another reduction in large particles occurs due to the large shearing forces applied through sonication.

Table 2.11 presents the same data as in Table 2.10 but on a particle number basis. Obviously, a large difference in particle sizes reported between the volume basis and the number basis distributions exist. These differences can be explained by recognizing that as particle size increases the volume contribution of a particle increases cubically. This weights the volume distribution more heavily towards larger particles (i.e., a few larger particles will greatly effect the volume basis but will have no significant effect on the number basis). Hence, Table 2.11 suggests that a large number of particles at approximately 0.9 microns exist. The bulk of these particles do not change due to changes in the shear force applied. However, small changes in the number of larger particles are greatly amplified on a volume basis (see Table 2.10).

Table 2.10. Particle Size Distribution (Volume) of 2-Wt% UDS AN-102 Slurry

Sample Conditions	Peak/Mode Number	Peak/ Mode Particle Size (μm)	Peak/Mode Width (μm)	Approximate Volume Percent of Particles in Peak/Mode
X100 at 40 mL/s	1	231	78	5%
	2	68	95	21%
	3	3.9	6.4	74%
X100 at 60 mL/s	1	30	10	6%
	2	4.0	6.7	94%
X100 at 60 mL/s with 90 second sonication at 40 W (#1)	1	4.1	8.0	100%
X100 at 60 mL/s with 90 second sonication at 40 W (#2)	1	4.1	7.9	100%

Table 2.11. Particle Size Distribution (Number) of 2-Wt% UDS AN-102 Slurry

Sample Conditions	Peak/Mode Number	Peak/ Mode Particle Size (μm)	Peak/Mode Width (μm)	Approximate Number Percent of Particles in Peak/Mode
X100 at 40 mL/s	1	0.85	0.84	100%
X100 at 60 mL/s	1	0.88	0.85	100%
X100 at 60 mL/s with 90 second sonication at 40 W (#1)	1	0.89	0.84	100%
X100 at 60 mL/s with 90 second sonication at 40 W (#2)	1	0.89	0.84	100%

The particle size distributions on a volume basis are presented graphically in Figure 2.5 through Figure 2.7. In these figures, the left y-axis represents the volume percent of particles in a small, discrete range. The right y-axis represents the sum of these data from small particles to large particles; often referred to as the cumulative volume percent. These data represent the volume percent of particles smaller than a given particle size. The volume distribution data indicate that most of the particles are in the 0.5 to 15 micron particle size. As the shear rate in the instrument increased (due to increasing the flow rate from 40 mL/s to 60 mL/s) some of the larger particles (i.e. particles greater than 40 micron) appear to break apart, resulting in a larger number of smaller particles. When sonication is applied (i.e. 40 W for 90 seconds) at the same flow rate (60 mL/s), the particles in the 20 to 40 micron size appeared to further break apart. A second sonication resulted in no significant change.

The particle size distributions on a number basis are presented graphically in Figure 2.8 through Figure 2.10. As discussed above, only small changes are observed at each shearing condition.

Lastly, the sub-samples taken form the X100 recirculation system before and after sonication were analyzed in the UPA instrument. Unfortunately, the UPA instrument indicated a low solids loading from both of these samples and the data from this instrument is considered unreliable. However, the UPA consistently indicated a large volume/number of particles in the 5 to 20 nm range.

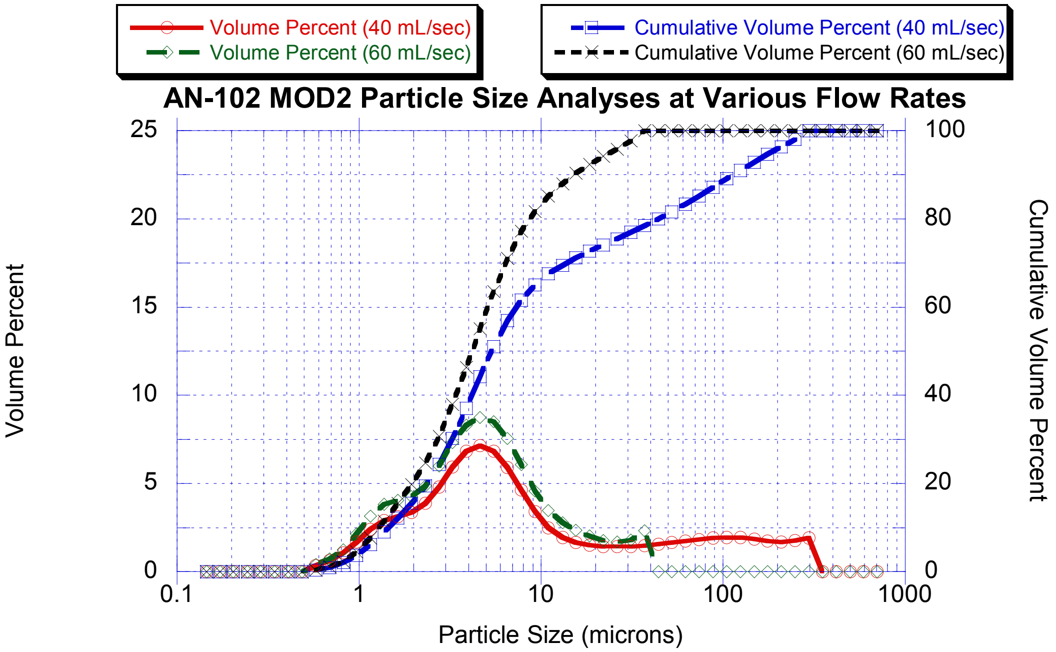


Figure 2.5. Particle Size Analyses at Various Flow Rates (Volume Basis) of 2-Wt% UDS AN-102 Slurry

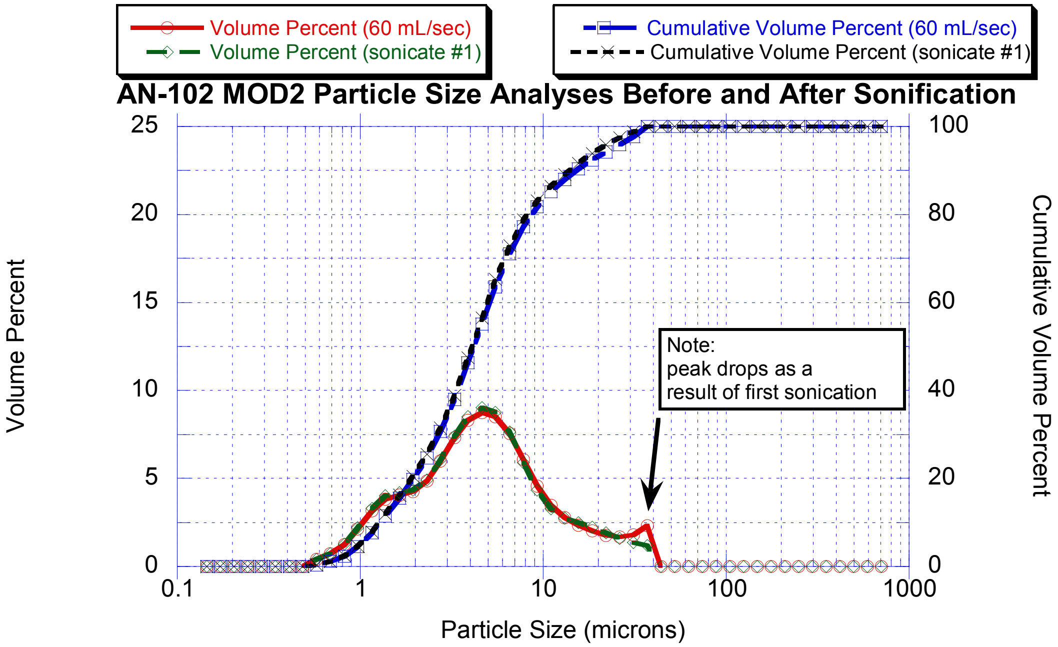


Figure 2.6. Particle Size Analyses Before and After Sonication (Volume Basis) of 2-Wt% UDS AN-102 Slurry

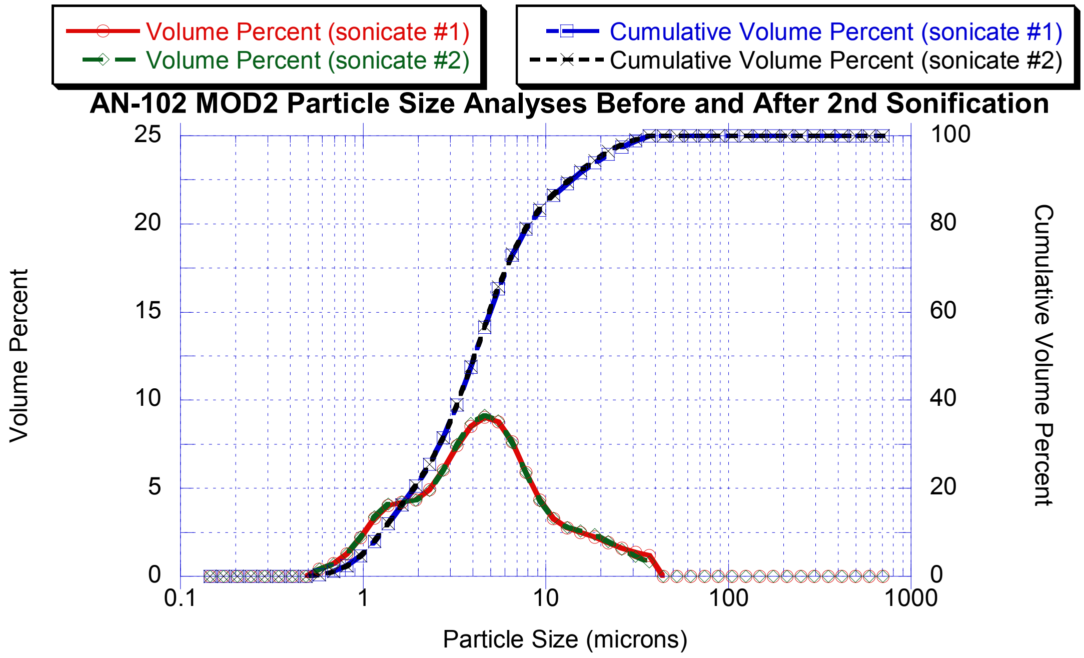


Figure 2.7. Particle Size Analyses Before and After 2nd Sonication (Volume Basis) of 2-Wt% UDS AN-102 Slurry

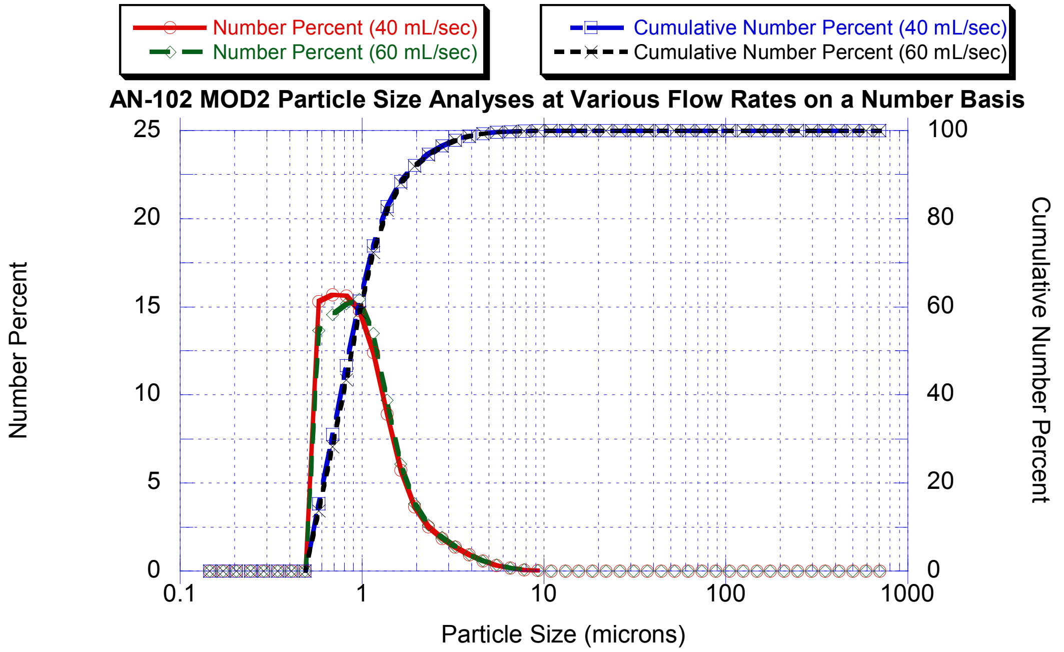


Figure 2.8. Particle Size Analyses at Various Flow Rates (Number Basis) of 2-Wt% UDS AN-102 Slurry

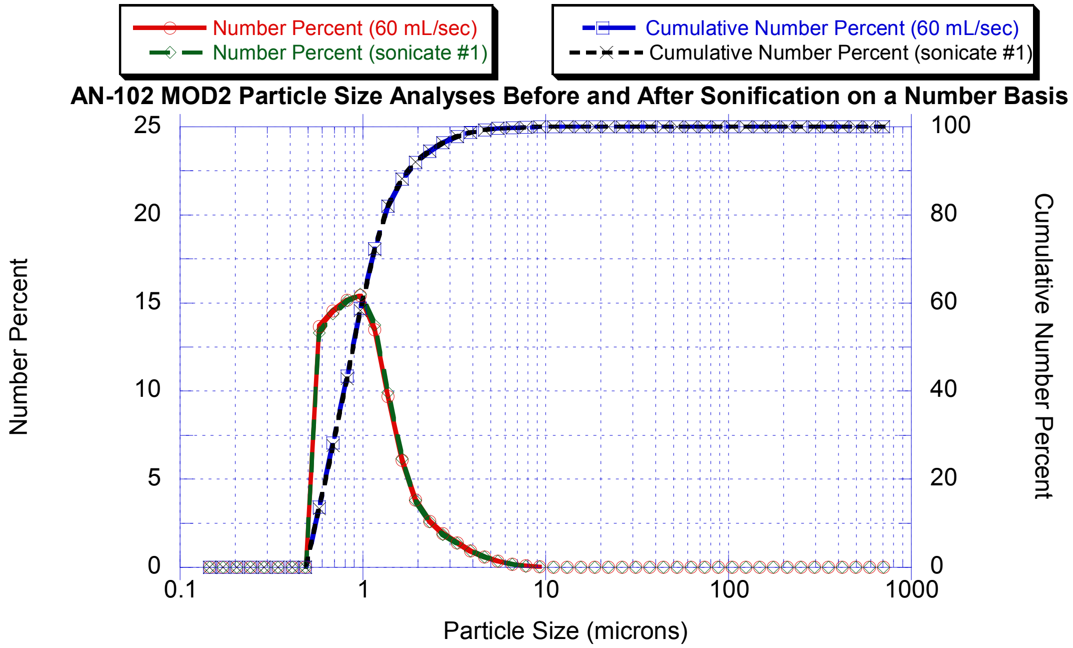


Figure 2.9. Particle Size Analyses Before and After Sonication (Number Basis) of 2-Wt% UDS AN-102 Slurry

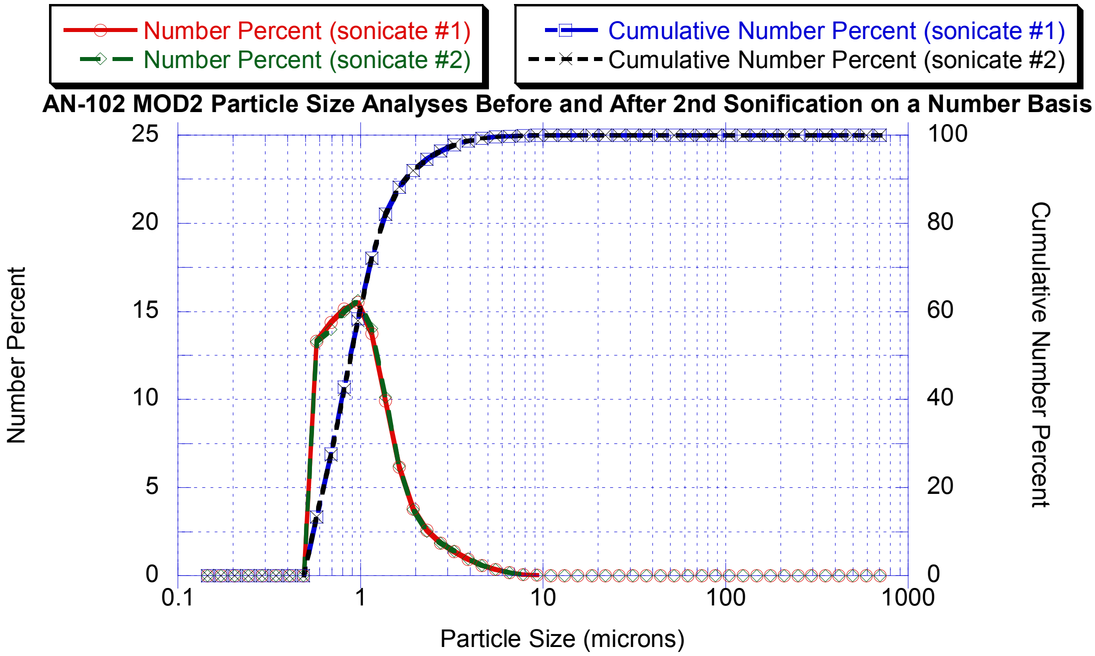


Figure 2.10. Particle Size Analyses Before and After 2nd Sonication (Number Basis) of 2-Wt% UDS AN-102 Slurry

2.4 C-104 Filtrate, Leachate, and Wash Solutions

The C-104 solution used for blending with the 2-wt% UDS AN-102 composite slurry were generated as a result of filtration, water-washing, caustic-washing, and caustic leaching of tank C-104 sludge (Brooks et al. 2000). The liquids were produced during tests performed on dates 8/24/99 through 8/28/99 in the HLRF hot cells located in the RPL. A flow sheet of the processes used to produce the liquids is provided as Appendix A (C-104 Cross-flow Filtration Test Experiment Steps).

As shown in Appendix A, the cross-flow ultra-filtration (CUF) unit was first prepared and tested with DIwater (top block). The composite and homogenized C-104 samples RIN, RIN2 and Comp C were admitted into the CUF unit. Further, Comp D was added and the C-104 slurry comprised of the four composites was dewatered using the CUF unit. Part of the filtrate resulting from the dewatering step was collected, divided, labeled 'C-104 Filtrate #1' and 'C-104 Filtrate #2'.

The C-104 sludge remaining in the CUF unit was further washed with water and three C-104 water wash permeates were produced. The permeates were combined into a composite and labeled 'C-104 Wash (H_2O) Filtrate Composite'. The C-104 sludge remaining in the CUF unit was removed and centrifuged. The sludge was then washed in 3.0 M NaOH for 8 hours at 85°C and the resulting caustic leach permeate were combined into a composite and labeled 'C-104 Caustic Leach Permeate'. The leached C-104 sludge was lastly washed with a dilute 0.01 M NaOH and the resulting caustic wash permeate was labeled 'C-104 Caustic Wash Composite'.

Per the TS, the solutions generated from the C-104 sludge washing are to be blended in a ratio of 1 : 0.73 : 0.73 : 0.49 : 0.47 : 0.25 (2wt% AN-102 : Filtrate #1 : Filtrate #2 : Wash (H_2O) Filtrate : Caustic Leach : Caustic Wash).

2.5 Small-Scale Solubility/Compatibility Testing

The purpose of the small-scale solubility/compatibility testing is to confirm that the C-104 solutions and 2-wt% AN-102 composite slurry, when mixed together, do not result in gel formation, solidification, or a significant increase in solids content. This small-scale mixing test was performed by adding the appropriate volumes (as defined by the TS) of the C-104 solutions to an approximate 25-mL aliquot of the 2-wt% AN-102 composite slurry.

For the solubility/compatibility testing, two aliquots of the 2-wt% AN-102 composite slurry were extracted from jar AN-102 MOD1. Each aliquot was transferred to a separate 100-mL volume graduated centrifuge cone (labeled MOD A and MOD B, See Figure 2.1). These aliquots were allowed to settle for 24 hours after which time the level of settled solids and total slurry were recorded. Sample MOD A contained 26.5 ml of total material of which 6.3 mL were settled solids. Sample MOD B contained 24.2 mL of total material of which 5.9 mL were settled solids. The vol% settled solids of these samples after 24 hours (23.8 and 24.4 vol%) appear only slightly higher than the vol% settled solids in Table 2.4 for similar aliquots settled for 72 hours (average 22 vol%).

Following the 24 hour settling, predetermined volumes of the C-104 solutions were added to sample MOD A as detailed in Table 2.12. Sample MOD B was retained for reference. Following the additions, sample MOD A was thoroughly mixed by inverting the cone a minimum of 10 times and then allowed to sit undisturbed for 3 days. After 3 days, sample MOD A contained approximately 97 mL of total material of which about 1.8 mL was settled solids (1.9 vol%). There was no indication of gel formation or precipitation of additional solids.

Table 2.12. Small-Scale Testing: C-104 and AN-102 2-Wt% Feed

Solution	TS Target (mL)	Mass Added (g)	Density (g/mL)	Volume of Addition (mL)
AN-102 2wt% Feed (MOD A)	27.1	35.5	1.410	25.2
C-104 Filtrate #1	19.7	20.4	1.021	20.0
C-104 Filtrate #2	19.7	20.3	1.060	19.2
C-104 Wash (H_2O) Filtrate Composite	13.3	13.5	1.013	13.3
C-104 Caustic Leach Permeate	12.6	13.5	1.095	12.3
C-104 Caustic Wash Composite	6.8	7.1	1.028	6.9
Total	99.2	110.4		96.9

2.6 Addition of C-104 Solutions to AN-102 Samples, Compositing and Sub-Sampling

Since no solids or gel formation was observed in the MOD A small-scale test, C-104 solutions were added to the remaining 2-wt% AN-102 slurry composite resident in the mixing vessel. Prior to the additions, the material in AN-102 MOD 1 and MOD B used in the small-scale test was returned to the mixing vessel. This left a total of 569 mL of the 2-wt% AN-102 feed in the mixing vessel (This does not include the material in MOD A. The material in MOD A was returned to the mixing vessel after the addition of the C-104 solutions). The impeller in the mixing vessel was turned on, and while stirring, the C-104 solutions were added. Table 2.13 details the volumes of the additions.

Table 2.13. AN-102/C-104 Blended Feed Preparation

Solution	TS Target (mL)	Mass Added (g)	Density (g/mL)	Volume of Addition to Mixing Vessel (mL)	Total After MOD A Addition [(a)] (mL)
AN-102 2wt% Feed	569	802 [(c)]	1.410	569 [(b)]	596
C-104 Filtrate #1	414	415	1.021	407	427
C-104 Filtrate #2	414	428	1.060	404	423
C-104 Wash (H_2O) Filtrate Composite	279	284	1.013	281	294
C-104 Caustic Leach Permeate	267	293	1.095	267	279
C-104 Caustic Wash Composite	144	149	1.028	145	152
Total		2371			2171 (2481 g)

(a) See Table 2.6 for MOD A volumes.
(b) Material in mixing vessel after return of material in AN-102 MOD 1 and MOD B.
(c) Based on density of 1.41 g/mL.

The material in the mixing vessel was stirred for one hour. With the impeller still running, a 100-mL portion was removed through the ¾-inch valve located on the bottom of the vessel to flush the valve and connecting tube. This flush material was poured back into the mixing vessel. Then the entire contents of the mixing vessel were collected into six clean volume-graduated glass jars. Table 2.14 lists the samples collected. The jar labeled AN-102 DF A was allocated for characterization, while the remainder of the material was allocated for process testing. The difference between the mass blended and the mass transferred to the collection jars represents an approximate 5% sample loss, which is most likely due to evaporation while blending.

Table 2.14. Sub-Samples of the AN-102/C-104 Blended Feed

Jar Identification	Mass (g)		Jar Identification	Mass (g)
AN-102 DF A	191		AN-102 DF D	523
AN-102 DF B	514		AN-102 DF E	504
AN-102 DF C	505		AN-102 DF TRANS 3	120
Total Mass = 2357 g				

3.0 Analytical Sample Preparation (Phase Separation)

Following the blending, homogenization, and sub-sampling of the AN-102/C-104 material reported in Section 2, the bottle identified for characterization was transferred from the HLRF to the SAL. Phase separation and supernatant and solids analysis were conducted according to instructions delineated in ASR 6025.01. The phase separation was performed by centrifuging bottle "AN-102-DF A" at 1000 G for 1 hour (with secondary containment in case of breakage). The supernatant was then decanted into a bottle labeled "AN-102 SUP DF" and the WCS transferred to a bottle labeled "AN-102 CS DF". The decanting and transfer operations were performed as rapidly as possible with remote manipulators to minimize the time that the supernatant and WCS were exposed to the SAL environment (i.e., to reduce potential of cross contamination and weight change due to evaporation/drying). Table 3.1 identifies the masses of supernatant and WCS collected for analytical characterization. The majority of the mass lost during phase separation is most likely from the centrifuged solids, i.e., not all the solids could be removed from the "AN-102-DF A" bottle.

Based on the blending ratio of the 2-wt% AN-102 slurry composite and the C-104 solutions detailed in Table 2.7, and barring any solubilization of the UDS by the C-104 liquids, the blended UDS should be approximately 0.8 wt% [i.e., ((802 g * 0.024) / 2371 g) * 100]. Assuming 0.8 wt% UDS and using the wt% solids and total dissolved solids (TDS) results from Table 3.2, the wt% WCS should be about 1.7%. The wt% WCS of the material contained in bottle "AN-102-DF A" is about 1.4% [i.e., (2.7 g / 191 g) * 100], which is considered good agreement for such low solids content.

Table 3.1. Supernatant and Wet Centrifuged Solids Quantities After Phase Separation

Bottles Sub-sampled for Characterization		**Sample Mass (g)**	**Mass Lost during Phase Separation (g)**	**Percent Lost during Phase Separation (%)**
RPL Number	**Bottle ID**			
None [(a)]	AN-102-DF A	191.4	2.1	1

Supernatant and Wet Centrifuged Solids Phase Separated Samples		**Bottle Tare Mass (g)**	**Bottle + Sample Mass (g)**	**Sample Mass (g)**	**Total Mass (g)**
RPL Number	**Bottle ID**				
01-441	AN-102 SUP DF	217.7	404.3	186.6	189.3
01-442	AN-102 CS DF	133.6	136.3	2.7	

(a) Bottles were not given individual RPL numbers since no analytical testing on the contents of the individual bottle was performed.

Following phase separation, the density and TDS of the supernatant, and the wt% solids of the WCS were measured. These limited physical-property measurement on the phase-separated supernatant and WCS were performed in duplicate, where quantities permitted; the results are presented in Table 3.2.

Table 3.2. Density and Percent Solids on Analytical Characterization Samples

RPL Number	Sample ID	Analysis	Sample	Duplicate	Average	RPD
01-0441	AN-102 SUP DF	Density (g/mL)	1.159	1.160	1.160	0%
		TDS (%) [(a)]	20.5	20.5	20.5	0%
01-0442	AN-102 CS DF	Wt% Total Solids (%) [(a)]	35.1	[(b)]	35.1	[(b)]

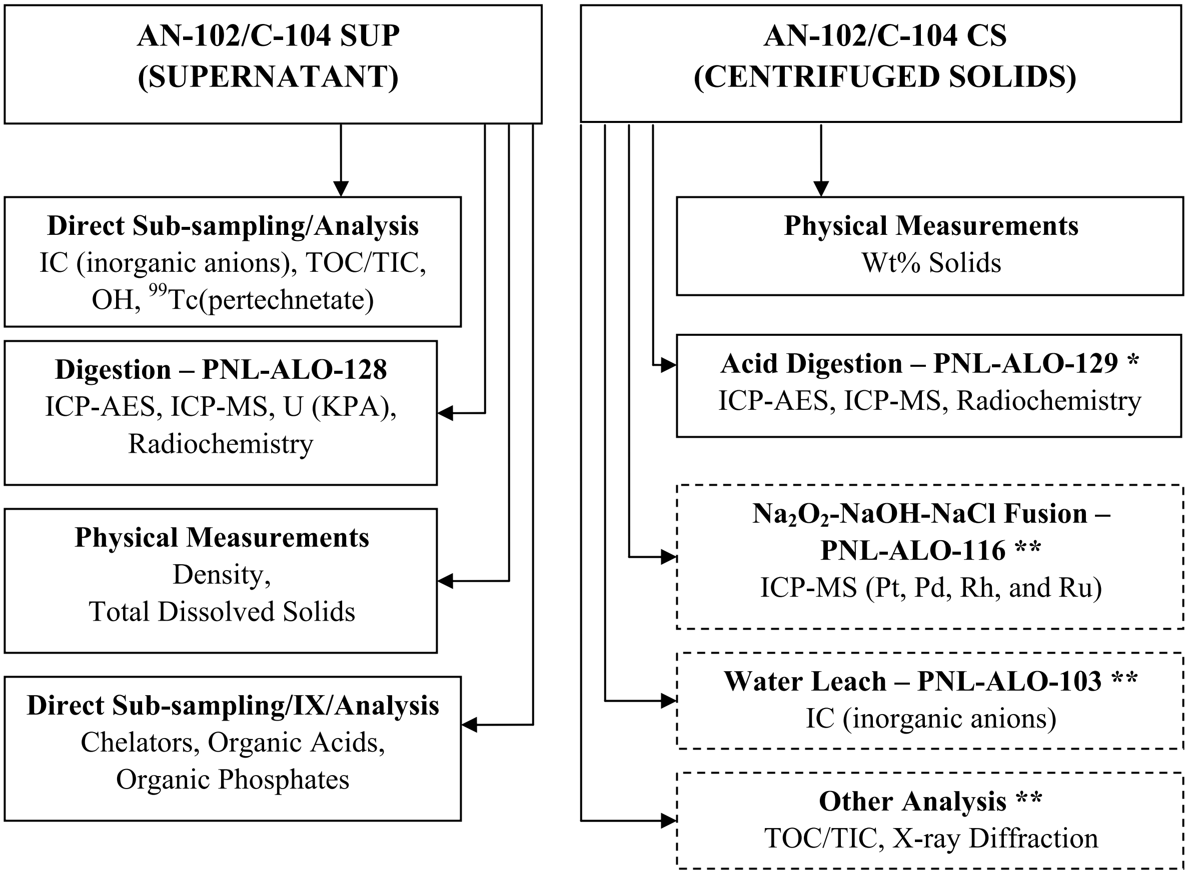
(a) After Drying at 105°C to constant weight

(b) Insufficient centrifuged solids available to perform analysis in duplicate.

Based on the physical-measurement results, the supernatant and WCS phases separated in the SAL (i.e., AN-102 SUP DF and AN-102 CS DF) are representative of the supernatant and solids material sub-sampled for process testing and should provide excellent baseline characterization results for process testing and good results for comparison of the waste phases to Specification 7 (Envelope C for the supernatant) and for providing the UDS composition for a future comparison to Specification 8.

4.0 Analytical Sample Processing

Following the phase separation of the AN-102/C-104 blended feed composite into supernatant and WCS phases, each phase was analyzed for the target analytes defined in the TS. The analytical processing of the supernatant and centrifuged solids and distribution of the unprocessed and processed sample aliquots are detailed in Figure 4.1. An ASR (6025.01) provided instructions to the laboratory to successfully complete the analytical and QC requirements defined in the TS.



* Acid digestion performed on dried solids

** Sample preparation/analysis not performed due to insufficient sample.

Figure 4.1. Flow Diagram for Analytical Processing of Supernatant and Centrifuged Solids

4.1 Supernatant

4.1.1 Direct Sub-sampling/Analysis

The AN-102/C-104 blended feed supernatant was sub-sampled in the SAL hot cells and then delivered to the RPL analytical workstations for various measurements including inorganic anion, hydroxide, total organic and inorganic carbon (TOC/TIC), and ^{99}Tc (pertechnetate). 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 providing any additional processing to the sub-samples that might be required (e.g., dilutions for inorganic ion analysis).

4.1.2 Direct Sub-sampling/IX/Analysis

The AN-102/C-104 blended feed supernatant was sub-sampled and subjected to IX procedures (Test Plan TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* for chelators and TI-RPP-WTP-059, *Organic Acids Sample Preparation of Tank AN-102 Supernatant* for organic acids) in the SAL to reduce the dose levels. The resulting effluents from the IX procedure were delivered to the 329 Facility analytical workstations for measurements of organic acids and chelators. Additional sample was processed through the IX 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 of the chelators).

4.1.3 Acid Digestion

The AN-102/C-104 blended feed supernatant was acid digested according to procedure PNL-ALO-128, *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*, in the SAL hot cells. Aliquots of the digested sub-samples were delivered to the 329 Facility for ICP-MS analysis and to various RPL analytical workstations for ICP-AES, total U by KPA, and the following radiochemical analyses: total alpha, gamma emitters by gamma energy analysis (GEA), $^{239+240}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm, and ^{90}Sr.

The SAL processed 1-mL aliquots of the supernatant in duplicate. 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 a sample and duplicate, the SAL processed duplicate digestion process blanks (PB), two blank spikes (BS), one for ICP-AES and one for ICP-MS, and two MSs (one for ICP-AES and one for ICP-MS). Aliquots of the BS, MS, and the PBs were sent with aliquots of the duplicate samples for ICP-AES or ICP-MS analyses. For radiochemical analyses, only the two process blanks were sent with aliquots of the duplicate samples for analysis. Post digestion BS and MS samples were prepared at the time of radiochemical separation except for GEA, which did not require any additional sample preparation.

4.1.4 Solvent Extraction for Organic Phosphates

The AN-102/C-104 blended feed supernatant was sampled and extracted in the SAL for analysis of bis-(2-ethylhexyl)phosphoric acid (D2EHP) according to the TP-RPP-WTP-047, *Identification and Quantification of D2EHP in Tank Waste*. Sub-samples consisted of duplicate aliquot samples of the supernatant (surrogate spike only) and duplicate MS samples (surrogate and D2EHP spike) adjusted to pH <2. A PB consisting of distilled DI water (surrogate spike only) and a BS consisting of D2EHP spiked distilled DI water 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 where the methylene chloride extracts were each concentrated to a volume <1 mL, derivatized with diazomethane/ether solution, and again concentrated to a volume of 1 mL for analysis. However, the BS sample was inadvertently spilled during the concentration procedure, so an additional BS was synthesized outside the hot cell.

4.2 Centrifuged Solids (Dried)

Due to an insufficient amount of the WCS sample, all of the analyses listed in the TS could not be performed. The BNI contact prioritized the listed analyses and only those analytes that could be measured from an acid digestion preparation of the available solid were analyzed.

A total wt% solids was performed once on the entire quantity of WCS obtained. Following the wt% solids determination, the resulting dried centrifuged solids from the AN-102/C-104 blended feed were acid digested in the SAL according to procedure PNL-ALO-129, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*. There were no visible residual solids remaining following the acid digestion. Aliquots of the digested samples were delivered to the 329 Facility for ICP-MS analysis and to various RPL analytical workstations for ICP-AES and the following radiochemical analyses: total alpha, GEA, $^{239+240}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm, and ^{90}Sr.

The SAL processed ~0.3-g aliquots of the dry centrifuged solids in duplicate according to PNL-ALO-129. The acid extracted solutions were brought to a nominal 25-mL volume and absolute volumes determined based on final solution weights and densities. The results were reported as wet sample (µg/g-wet) based upon 35.1% wt% solids measured on the WCS material. Along with a sample and duplicate, the SAL processed duplicate digestion PBs, two BS and two MS samples, one for ICP-AES and one for ICP-MS, were prepared from the AN-102 as-received sample 01-430 which was processed with the AN-102/C-104 centrifuged solids. A single MS from AN-102/C-104 blended feed centrifuged solids (dried) sample 01-442 was also processed for ICP-AES analysis by the SAL. Centrifuged solids samples from the AN-102 as-received composite and the AN-102/C-104 blended feed composite were prepared in the same batch by the SAL. Therefore, the MS from AN-102 as-received sample 01-430 served as the batch QC MS for the ICP-MS. Aliquots of the BSs, MSs, and the PBs were sent with aliquots of the duplicate samples for ICP-AES and ICP-MS analyses. For radiochemical analyses, only the duplicate PBs were sent with the duplicate samples for analysis. Post digestion BS and MS samples were prepared at the time of radiochemical separation except for GEA, which did not require any additional sample preparation.

5.0 Analytical Results

5.1 Introduction

Tables 5.1 through 5.9 provide inorganic, radioisotopic, and organic analytical results for the AN-102/C-104 blended feed supernatant and WCS samples. Results are reported in µg/mL, µg/g, µCi/mL, or µCi/g, as appropriate. For many analyses the nominal propagated uncertainties are also provided (as 1-σ, unless otherwise noted). However, for most analyses, no uncertainties are included in the tables. For these analyses, the estimated uncertainty is 10 to 15% for results above the estimated quantitation limit (EQL). Besides the duplicate sample results, the results obtained on the PBs are also reported, as appropriate.

The analytical results (Table 5.1 through Table 5.9) and the QC results (Table 6.2 through Table 6.10) include a Data Flag column (i.e., a "Data Qualifier Code") and the analyte concentrations or averages are flagged, as appropriate. The data qualifier codes utilized were 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 processing and analysis dilutions. In these cases, results less than 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 estimated quantitation limit (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 laboratory processing blank above the QA plan acceptance criteria (i.e., the blank is greater than the EQL or the blank exceeds 5% of sample concentration).
X	A QC deficiency was associated with the reported result. For this report the X flag is used for the following: a) batch laboratory control sample (LCS) or blank spike (BS) fails or was not analyzed, b) both the MS and the post spike fail, 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 AN-102 tank waste matrix per SW-846 [4] protocol. For most inorganic and organic methods, the 'estimated' MDLs are based on an instrument detection limit (IDL) estimated from using reagents and/or low concentration high-purity standards as samples and evaluating instrument response near background levels. The estimated quantitation limit (EQL) is typically set

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[4] U.S. Environmental Protection Agency (EPA). 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition including Updates I, II, IIA, IIB, III, and IIIA, Office of Solid Waste and Emergency Response. Washington. D.C.

at 10 times the estimated MDL adjusted for dilution factors resulting from digestion or leaching processing. For a few methods (e.g., IC), 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, the minimum detectable activity (MDA) is calculated per the QA Plan and is based on the background counting statistics.

Specific QC and QA discussions are given in Section 6.0.

5.2 Analyte List Modifications

The supernatant and ‘insoluble solids’ analyte list were defined by the TS. A few modifications to the analyte list or procedures defined by the TS had to be incorporated, and are detailed below:

- The laboratory was directed to determine pertechnetate ($^{99}TcO_4^-$) using separations and beta counting techniques, as opposed to total ^{99}Tc. The procedure was modified slightly to exclude the sample oxidation step so that the non-pertechnetate fraction was not oxidized. Also, instead of measuring the ^{99}Tc by liquid scintillation, sample preparations were counted with gas-flow proportional counters.
- Analyte concentrations in addition to those required by the TS are provided. These additional analytes were measured as part of the method and are provided for additional information only.
- Sulfur by ICP-AES could not be performed, since the ICP-AES used for this work does not have a sulfur channel, and sulfur could not be obtained by any other method.
- The organic acids were measured by IC, organic phosphates by GC/FID following derivatization, and chelators by GC/FID following derivatization, not by high performance liquid chromatography – mass spectrometry as defined by the TS.
- Succinic acid and ethylenediaminetriacetic acid (ED3A) were added to the supernatant analyte list in response to a request by BNI.
- The total Cs concentration in the solids was estimated based on the assumption the Cs isotopic ratio in the solids is equivalent to the isotopic ratio in the supernatant. The estimated total Cs in the supernatant was calculated relative to the ^{133}Cs from the ICP-MS analysis. The estimated total Cs in the WCS was calculated relative to the ^{137}Cs measured by GEA.

5.3 Data Limitations

- The reported fluoride results may represent the summation of fluoride, acetate, and formate concentrations, as these were not resolvable on the inorganic anion analysis IC system. Based on the formate and glycolate/acetate results reported from the organic anion IC analysis, the fluoride results are most likely biased high.
- Concentrations of numerous elements are reported by ICP-MS (Table 8.7). 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. No chemical separations were performed on the AN-102 as-received sample.

- The reported glycolate results represent the summation of glycolate and acetate, as these are not resolvable on the organic IC system.
- The pertechnetate QC (process duplicate and MS) failed so this analysis does not meet the QC acceptance criteria and the results have been flagged. However, the ratio of pertechnetate to total Tc is about 50%, which is consistent with analysis from another Envelope C waste, AN-107 (Blanchard et al. 2000).
- The IC phosphate results for the supernatant are reported as less than the EQL (i.e., 200 µg/mL based on the dilution required at the IC). This result is inconsistent with the ICP-AES phosphorous results of approximately 600 µg/mL; i.e., the ICP-AES result on a phosphate basis is approximately 1800 µg/mL. This suggests the possibility of fine particulate in the supernatant (i.e., acid digestion of supernatant increases the soluble phosphate concentration versus direct IC analysis of the supernatant).

5.4 General Observations

- The total ^{99}Tc measured by ICP-MS is about two times the ^{99}Tc+7 (pertechnetate) analysis measured by separations and beta counting. This suggests that about half of the ^{99}Tc is in the pertechnetate state. However, the pertechnetate analysis exhibited extremely unrealistic MS recoveries and the pertechnetate results have been flagged as having a severe QC deficiency.
- The comparison of the ^{241}Am activities determined by ICP-MS with those found by radiochemical methods (GEA and alpha energy analysis [AEA]) is good, indicating that the ICP-MS AMU 241 is predominately ^{241}Am.
- Three analytes (Ce, Mo, and Y) were analyzed by both ICP-AES and ICP-MS on the centrifuged solids. The agreement between the results is excellent. The average results from ICP-AES are 71, 16, and 26 µg/g for Ce, Mo, and Y, respectively. For ICP-MS, the average results were 70, 13, and 26 µg/g for Ce, Mo, and Y, respectively.
- The supernatant $^{239+240}$Pu results from AEA compare reasonably well with the sum of the ^{239}Pu and ^{240}Pu from ICP-MS (i.e., less than a factor of 2). The supernatant average being 1.5E-03 µg/mL from AEA versus the sum of 2.3E-03 µg/mL from ICP-MS. However, comparable results were not obtained on the centrifuged solids, where the averages are 1.1E-01 µg/g for AEA versus 2.4E+00 µg/g for ICP-MS (a factor of 20 difference).
- The total alpha measurement agreed well with the sum of alpha emitting radioisotopes measured (^{238}Pu, $^{239/240}$Pu, ^{241}Am, $^{243/244}$Cm, and ^{242}Cm) for both the supernatant and centrifuged solids samples.
- The comparison of the supernatant ^{137}Cs by GEA with the ^{137}Cs calculated from the ICP-MS ^{133}Cs and Cs isotope mass ratios is reasonable, averaging 1.17 µg/mL and 1.01 µg/mL, respectively.
- The total TOC based on summation of measured organic acids and chelators is approximately 19,000 µg C/mL for the supernatant. The TOC from the furnace method is reported as about

16,000 µg C/mL and compares well with the summed organics. The TOC from the persulfate method (i.e., approximately 10,000 µg C/mL) is approximately half that of the summed organic TOC. The persulfate method measures a significant TIC concentration, which is expected; whereas the furnace method measures essentially no TIC. Also, the summed organic TOC is driven primarily by the gluconate results (i.e., 15,000 µg C/mL), which is difficult to analyze due to interference from fluoride and its proximity to the void volume retention time.

5.5 Analytical Results Tables

The analytical results for the AN-102/C-104 blended feed supernatant are presented in Table 5.1 through Table 5.5 and the AN-102/C-104 blended feed WCS results are presented in Table 5.6 through Table 5.9. The PBs and BSs for the supernatant (labeled 01-429-PB and 01-430-BS) and WCS (labeled 01-430-PB and 01-430-BS) were batched with the AN-102/C-104 blended feed samples during sample preparation and analysis. Comparison of the supernatant analyte concentrations to Specification 7 is presented in Section 5.7.

Table 5.1. AN-102/C-104 Blended Feed Supernatant, ICP-AES Results

	Process Blank 1			Process Blank 2			Sample			Duplicate		
	MDL	01-429-PB1	(b) Data	MDL	01-429-PB2	(b) Data	MDL	01-441	(b) Data	MDL	01-441D	(b) Data
Analyte	µg/mL		Flag	µg/mL		Flag	µg/mL		Flag	µg/mL		Flag
ICP-AES Test Specification Analytes (a)												
Al	1.6	5.0	J	1.6	4.9	J	3.0	5,390		3.0	5,280	
Ba	0.3	0.3	U	0.3	0.3	U	0.5	0.5	U	0.5	0.5	U
Ca	6.6	6.6	U	6.6	6.6	U	13	169	X	13	137	X
Cd	0.4	0.4	U	0.4	0.4	U	0.8	16.9		0.7	16.4	
Cr	0.5	0.5	U	0.5	0.5	U	1.0	69.9		1.0	68.2	
Fe	0.7	1.6	J	0.7	0.7	U	1.3	6.7	JB	1.2	6.4	JB
K	52	52	U	53	53	U	100	540	JX	100	550	JX
La	1.3	1.3	U	1.3	1.3	U	2.5	3.2	J	2.5	3.3	J
Mg	2.6	2.6	U	2.6	2.6	U	5.0	5.0	U	5.0	5.0	U
Na	3.9	55		3.9	58		38	74,100		37	72,300	
Ni	0.8	1.0	J	0.8	0.9	J	1.5	123		1.5	120	
P	2.6	2.6	U	2.6	2.6	U	5.0	607		5.0	594	
Pb	2.6	2.6	U	2.6	2.6	U	5.0	49	J	5.0	49	J
U	52	52	U	53	53	U	101	101	U	100	100	U
Other Analytes Measured												
Ag	0.7	0.7	U	0.7	0.7	U	1.3	1.3	UX	1.2	1.2	UX
As	6.6	6.6	U	6.6	6.6	U	13	13	U	13	13	U
B	1.3	43		1.3	44		2.5	52	B	2.5	48	B
Be	0.3	0.3	U	0.3	0.3	U	0.5	0.5	U	0.5	0.5	U
Bi	2.6	2.6	U	2.6	2.6	U	5.0	5.0	U	5.0	5.0	U
Ce	5.2	5.2	U	5.3	5.3	U	10	10	U	10	10	U
Co	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U
Cu	0.7	0.7	U	0.7	0.7	U	1.3	6.8	J	1.2	6.7	J
Dy	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U

Table 5.1. (Cont'd)

	Process Blank 1			Process Blank 2			Sample			Duplicate		
	MDL	01-429-PB1	(b) Data	MDL	01-429-PB2	(b) Data	MDL	01-441	(b) Data	MDL	01-441D	(b) Data
Analyte	µg/mL		Flag	µg/mL		Flag	µg/mL		Flag	µg/mL		Flag
Eu	2.6	2.6	U	2.6	2.6	U	5.0	5.0	U	5.0	5.0	U
Li	0.8	0.8	U	0.8	0.8	U	1.5	1.5	U	1.5	1.6	J
Mn	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U
Mo	1.3	1.3	U	1.3	1.3	U	2.5	16	J	2.5	15	J
Nd	2.6	2.6	U	2.6	2.6	U	5.0	6.2	J	5.0	6.7	J
Pd	20	20	U	20	20	U	38	38	U	37	37	U
Rh	7.9	7.9	U	7.9	7.9	U	15	15	U	15	15	U
Ru	29	29	U	29	29	U	55	55	U	55	55	U
Sb	13	13	U	13	13	U	25	25	U	25	25	U
Se	6.6	6.6	U	6.6	6.6	U	13	13	U	13	13	U
Si	13	151		13	139		25	290	BX	25	270	BX
Sn	39	39	U	39	39	U	76	76	U	75	75	U
Sr	0.4	0.4	U	0.4	0.4	U	0.8	0.8	U	0.7	0.7	U
Te	39	39	U	39	39	U	76	76	U	75	75	U
Th	26	26	U	26	26	U	50	50	U	50	50	U
Ti	0.7	0.7	U	0.7	0.7	U	1.3	1.3	U	1.2	1.2	U
Tl	13	13	U	13	13	U	25	25	U	25	25	U
V	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U
W	52	52	U	53	53	U	101	101	U	100	100	U
Y	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U
Zn	1.3	1.3	U	1.3	1.3	U	2.5	3.3	J	2.5	2.5	U
Zr	1.3	1.3	U	1.3	1.3	U	2.5	2.5	U	2.5	2.5	U

(a) All ICP-AES analytes reported except sulfur. (See Section 5.2)

(b) 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 (See Section 5.1).

Table 5.2. AN-102/C-104 Blended Feed Supernatant, ICP-MS Results

Analyte	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-429-PB1	± 1SD	(d) Data Flag	MDL	01-429-PB2	± 1SD	(d) Data Flag	MDL	01-441	± 1SD	(d) Data Flag	MDL	01-441D	± 1SD	(d) Data Flag
	µg/mL				µg/mL				µg/mL				µg/mL			
U(KPA)(a)	6E-03	6.08E-02	2% (a)		6E-03	3.23E-02	2% (a)		5E-03	1.44E+01	4% (a)		5E-03	1.55E+01	4% (a)	
^{133}Cs	2E-01	2E-01		U	2E-01	2E-01		U	6E-02	2.59E+00	1.36E-02		6E-02	2.65E+00	5.04E-02	
^{135}Cs (b)		n/a				n/a				7.13E-01	1.00E-02			7.25E-01	1.50E-02	
^{137}Cs (b)		n/a				n/a				9.92E-01	8.00E-03			1.02E+00	2.20E-02	
Rb	1E-01	1E-01		U	1E-01	1E-01		U	8E-02	2.28E+00	1.26E-02		8E-02	2.27E+00	4.85E-03	
AMU-241 (c)	2E-03	2E-03		U	2E-03	2E-03		U	1E-03	1.15E-02	1.40E-03	J	1E-03	1.22E-02	8.35E-04	J
	µCi/mL			Flag	µCi/mL			Flag	µCi/Ml			Flag	µCi/mL			Flag
^{99}Tc	6E-03	6E-03		U	7E-03	7E-03		U	2E-03	4.20E-02	3.35E-04		2E-03	4.28E-02	4.84E-04	
^{237}Np	1E-06	1E-06		U	1E-06	1E-06		U	2E-06	5.20E-05	2.26E-06		2E-06	5.22E-05	9.14E-07	
^{239}Pu	2E-04	2E-04		U	2E-04	2E-04		U	3E-04	1.79E-03	4.91E-05	J	3E-04	1.65E-03	4.35E-05	J
^{240}Pu	3E-04	3E-04		U	3E-04	3E-04		U	6E-04	6.80E-04	4.86E-05	J	6E-04	6.23E-04	1.13E-04	J
AMU-241 (c)	6E-03	6E-03		U	6E-03	6E-03		U	4E-03	3.73E-02	4.52E-03		4E-03	3.95E-02	2.70E-03	

(a) Uranium results by KPA; standard deviation reported in percent.

(b) The ^{135}Cs and ^{137}Cs concentrations are calculated from the ICP-MS ^{133}Cs concentration and the Cs atomic mass ratios determined by ICP-MS. The GEA ^{137}Cs results for both the sample and duplicate are 102 µCi/mL (i.e., 1.17E+00 µg/mL) – See Table 5.3.

(c) AMU-241 is either ^{241}Am or ^{241}Pu or a combination thereof. The µg/mL results are calculated based on the calibration of the AMU-241 as ^{241}Am and the µCi/mL results are calculated using the specific activity of ^{241}Am (3.23 Ci/g).

(d) Data flags: U = not detected above reported MDL; J = estimated value (See Section 5.1).

Decay correction reference date is nominally April 2001.

Table 5.3. AN-102/C-104 Blended Feed Supernatant, Radioisotope Results

	Process Blank 1				**Process Blank 2**				**Sample**				**Duplicate**			
Analyte	**MDA µCi/mL**	**01-429-PB1**	**Err(a) %**	**Data Flag(c)**	**MDA µCi/mL**	**01-429-PB2**	**Err(a) %**	**Data Flag(c)**	**MDA µCi/mL**	**01-441**	**Err(a) %**	**Data Flag(c)**	**MDA µCi/mL**	**01-441D**	**Err(a) %**	**Data Flag(c)**
Alpha	6E-05	1.10E-03	5		6E-05	3.60E-04	10		4E-03	4.60E-02	7		6E-03	4.64E-02	8	
^{238}Pu	2E-06	3.20E-04	3		6E-07	9.49E-05	3		1E-04	6.75E-04	14	BJ	2E-04	3.03E-04	26	BJ
$^{239/240}$Pu	2E-06	4.07E-05	6		5E-07	1.67E-05	6		9E-05	1.40E-03	9		9E-05	1.58E-03	8	
^{241}Am	4E-06	1.06E-04	6		1E-06	4.45E-05	4		2E-04	3.83E-02	2		2E-04	3.66E-02	2	
^{241}Am (GEA)	6E-04	6E-04		U	6E-04	6E-04		U	2E-02	4.17E-02	14	J	2E-02	3.22E-02	16	J
$^{243/244}$Cm	4E-06	5.71E-04	3		1E-06	1.56E-04	3		7E-05	2.26E-03	6	B	7E-05	1.57E-03	8	B
^{242}Cm	2E-06	2E-06		U	5E-07	5E-07		U	7E-05	1.79E-04	23	J	6E-05	1.30E-04	28	J
Sum of Alpha		1.04E-03	4			3.12E-04	4			4.28E-02	3			4.02E-02	3	
^{90}Sr	4E-04	1.47E-02	3		4E-04	4.47E-03	5		4E-01	1.41E+01	3		4E-01	1.34E+01	3	
^{99}Tc (b)	2E-06	2E-06		U		n/a			4E-06	2.20E-02	4	X	4E-06	2.17E-02	4	X
^{137}Cs	2E-04	6.42E-03	3		3E-04	2.17E-03	7		7E-03	1.02E+02	2		7E-03	1.02E+02	2	
^{60}Co	3E-04	3E-04		U	3E-04	3E-04		U	4E-04	2.55E-02	2		3E-04	2.53E-02	2	
^{154}Eu	6E-04	6E-04		U	6E-04	6E-04		U	2E-03	5.83E-02	2		2E-03	5.80E-02	2	
^{155}Eu	6E-04	6E-04		U	6E-04	6E-04		U	2E-02	4.00E-02	10		2E-02	3.53E-02	10	

n/a = not applicable

(a) The % error represents the uncertainty at 1-s.

(b) Duplicate not sub-sampled from SAL, laboratory replicate sample analyzed for 99Tc; only one PB prepared and analyzed

(c) 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 (See Section 5.1).

Decay correction reference date is nominally May 2001

Table 5.4. AN-102/C-104 Blended Feed Supernatant, Other Analyte Results

	Process Blank			**Sample**			**Duplicate**		
	EQL/ MDL (a)	**01-441-PB**	**(g) Data Flag**	**EQL/ MDL (a)**	**01-441**	**(g) Data Flag**	**EQL/ MDL (a)**	**01-441D**	**(g) Data Flag**
Analyte	**µg/mL**			**µg/mL**			**µg/mL**		
Test Specification Analytes									
F (b)	0.25	0.25	U	200	3,700	U	200	3,700	U
Cl	0.25	0.25	U	200	1,170		200	1,130	
NO_2	0.5	0.5	U	500	25,900		500	25,800	
NO_3	0.5	0.5	U	2,000	61,400		2,000	60,600	
PO_4	0.5	0.5	U	200	200		200	200	
SO_4	0.5	0.5	U	400	4,470		400	4,390	
OH	170	170 (d)	U	170	4,230	J	170	5,030	J
TOC-F (c)		n/a		140	15,800		60	15,700	
TIC-F (c)		n/a		140	200		60	400	
TOC-P (c)		n/a		87	9,300		87	9,700	
TIC-P (c)		n/a		33	6,000		33	6,000	
Gluconate	1	1	U	500	41,000		500	40,000	
Glycolate (e)	0.1	0.1	U	50	3,200		50	3,400	
Formate	0.1	0.1	U	50	2,800		50	2,800	
Oxalate	0.2	0.2	U	100	1,800		100	1,800	
Citrate	0.2	0.2	U	100	1,200		100	1,200	
Other Analytes Measured									
Br	0.25	0.25	U	250	250	U	250	250	U
Oxalate (f)	0.5	0.5	U	400	2,300		400	2,270	

(a) F, Cl, NO_2, NO_3, PO_4, SO_4, and Br report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

(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), possibly formate or acetate.

(c) For TOC and TIC: P=by hot persulfate method; F=by furnace method/TIC by difference (TIC = TC-TOC); System blanks are subtracted from all sample results per procedure and are not applicable (i.e., n/a).

(d) For OH blank, no inflection point was detected.

(e) Glycolate is not resolved from acetate by the IC method performed.

(f) Oxalate results from inorganic IC analysis; for information only and comparison with organic IC results.

(g) Data flags: U = not detected above reported MDL; J = estimated value (See Section 5.1).

Table 5.5. AN-102/C-104 Blended Feed Supernatant, Additional Organic Analyte Results

			Process Blank		Sample		Duplicate	
Analyte	CAS #	MDL µg/mL	01-429-PB µg/mL	Data Flag[(d)]	01-441 µg/mL	Data Flag[(d)]	01-441D µg/mL	Data Flag[(d)]
Organic Phosphate Analytes								
D2EHP	298-07-7	0.5	0.5	U	0.5	U	0.5	U
DPP (surrogate)	838-85-7		3% (a)		83% (a)		87% (a)	
Chelators and Degradation Products								
EDTA	60-00-4	120	120	U	770	JX	460	JX
HEDTA(c)	150-39-0	120	120	U	120	UX	120	UX
ED3A(c)		120	120	U	760	JX	540	JX
NTA	139-13-9	100	100	U	200	JX	130	JX
IDA (as NIDA)(c)	142-73-4	120	120	U	1,600	X	1,100	X
Citric acid(b)	77-92-9	130	130	U	570	J	290	J
Succinic acid	110-15-6	34	34	U	34	UX	34	UX
AA (surrogate)			86% (a)		98% (a)		85% (a)	

D2EHP = bis-(2-ethylhexyl)phosphate; EDTA= ethylenediaminetetraacetic acid;
DDP = diphenylphosphate; 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) Value represents percent recovery of the surrogate standard.
(b) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids.
(c) Concentration results based on EDTA calibration.
(d) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 5.1).

Table 5.6. AN-102/C-104 Blended Feed Wet Centrifuged Solids, ICP-AES Results

	Process Blank 1			Process Blank 2			Sample (a)			Duplicate (a)		
	MDL	01-430-PB1	(c) Data	MDL	01-430-PB2	(c) Data	MDL	01-442	(c) Data	MDL	01-442D	(c) Data
Analyte	µg/g		Flag	µg/g		Flag	µg/g		Flag	µg/g		Flag
ICP-AES Test Specification Analytes (b)												
Ag	1.0	1.0	U	0.9	0.9	U	1.6	1.6	UX	1.6	1.6	UX
Al	2.4	2.4	U	2.3	2.6	J	3.8	58,100		19	53,100	
Ba	0.4	0.4	U	0.4	0.4	U	0.6	79		0.6	79	
Bi	4.0	4.0	U	3.8	3.8	U	6.4	12	J	6.2	11	J
Ca	10	10	U	9.5	9.5	U	16	441		16	405	
Cd	0.6	0.6	U	0.6	0.6	U	1.0	20		0.9	20	
Cr	0.8	0.8	U	0.8	0.8	U	1.3	4,550		1.2	4,580	
Cu	1.0	1.0	U	0.9	0.9	U	1.6	11	J	1.6	13	J
Fe	1.0	4.9	J	0.9	1.2	J	1.6	3,260		1.6	3,640	
La	2.0	2.0	U	1.9	1.9	U	3.2	106		3.1	105	
Mg	4.0	4.0	U	3.8	3.8	U	6.4	55	J	6.2	53	J
Mn	2.0	2.0	U	1.9	1.9	U	3.2	764		3.1	765	
Na	6.0	73		5.7	87		9.5	58,700		9.4	59,300	
Nd	4.0	4.0	U	3.8	3.8	U	6.4	181		6.2	179	
Ni	1.2	1.2	U	1.1	1.1	U	1.9	105	X	1.9	157	X
P	4.0	4.0	U	3.8	3.8	U	6.4	1,060		6.2	1,060	
Pb	4.0	4.0	U	3.8	40.3		6.4	412	B	6.2	400	B
Pd	30	30	U	28	28	U	48	48	U	47	47	U
Rh	12	12	U	11	11	U	19	19	U	19	19	U
Ru	44	44	U	42	42	U	70	70	U	69	69	U
Si	20	120	J	19	130	J	32	3,630		31	3,630	
Sr	0.6	0.6	U	0.6	0.6	U	1.0	24		0.9	24	
Ti	1.0	1.0	U	0.9	0.9	U	1.6	3.2	J	1.6	3.3	J
Zn	2.0	2.0	U	1.9	1.9	U	3.2	80		3.1	78	
Zr	2.0	2.0	U	1.9	1.9	U	3.2	58	X	3.1	98	X
ICP-MS Test Specification Analytes Measured by ICP-AES												
As	10	10	U	9.5	9.5	U	16	17	J	16	16	U
B	2.0	50		1.9	60		3.2	60	B	3.1	54	B
Be	0.4	0.4	U	0.4	0.4	U	0.6	7.1		0.6	7.0	
Ce	8.1	8.1	U	7.6	7.6	U	13	72	J	12	70	J
Co	2.0	2.0	U	1.9	1.9	U	3.2	3.2	U	3.1	3.1	U
K	81	81	U	76	76	U	130	580	JX	130	550	JX
Li	1.2	1.2	U	1.1	1.1	U	1.9	11	J	1.9	11	J
Mo	2.0	2.0	U	1.9	1.9	U	3.2	15	J	3.1	16	J
Sb	20	20	U	19	19	U	32	32	U	31	31	U
Se	10	10	U	9.5	9.5	U	16	16	U	16	16	U
Te	60	60	U	57	57	U	95	95	U	94	94	U
Th	40	40	U	38	38	U	64	160	J	62	160	J
Tl	20	20	U	19	19	U	32	32	U	31	31	U

Table 5.6. (Cont'd)

Analyte	Process Blank 1			Process Blank 2			Sample [(a)]			Duplicate [(a)]		
	MDL	01-430-PB1	[(c)] Data Flag	MDL	01-430-PB2	[(c)] Data Flag	MDL	01-442	[(c)] Data Flag	MDL	01-442D	[(c)] Data Flag
	µg/g			µg/g			µg/g			µg/g		
U	81	81	U	76	76	U	130	330	J	130	310	J
V	2.0	2.0	U	1.9	1.9	U	3.2	3.2	U	3.1	3.1	U
W	81	81	U	76	76	U	130	130	U	130	130	U
Y	2.0	2.0	U	1.9	1.9	U	3.2	26	J	3.1	26	J
Other Analytes Measured												
Dy	2.0	2.0	U	1.9	1.9	U	3.2	3.2	U	3.1	3.1	U
Eu	4.0	4.0	U	3.8	3.8	U	6.4	6.4	U	6.2	6.2	U
Sn	60	60	U	57	57	U	95	95	U	94	94	U

(a) Concentration based on the mass of wet centrifuged solids; results contain a contribution from the interstitial supernatant.

(b) All ICP-AES analytes reported except sulfur. (See Section 5.2)

(c) 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 (See Section 5.1).

Table 5.7. AN-102/C-104 Blended Feed Wet Centrifuged Solids, ICP-MS Metals Results

Analyte	Process Blank 1				Process Blank 2				Sample [(a)]				Duplicate [(a)]			
	MDL	01-430-PB1	± 1SD	Data	MDL	01-430-PB2	± 1SD	Data	MDL	01-442	± 1SD	Data	MDL	01-442D	± 1SD	Data
	µg/g			Flag[(c)]	µg/g			Flag[(c)]	µg/g			Flag[(c)]	µg/g			Flag[(c)]
As	9E-01	3.52E+00	3.46E-01	J	1E+00	2.29E+00	1.10E-01	J	2E+00	6.43E+00	4.16E-01	JB	2E+00	7.32E+00	1.29E+00	JB
B	4E-01	2.30E+01	3.14E-01		4E-01	1.86E+01	7.11E-01		2E+00	1.72E+01	8.87E-02	JBX	2E+00	1.76E+01	2.68E-01	JBX
Be	1E-01	1E-01		U	1E-01	1E-01		U	3E-01	1.47E+01	4.03E-01		3E-01	1.39E+01	7.87E-01	
Ce	4E-01	4E-01		U	4E-01	4E-01		U	2E-01	7.14E+01	1.03E+00		2E-01	6.90E+01	8.26E-01	
Co	4E-01	4E-01		U	4E-01	4E-01		U	7E-02	8.61E-01	2.22E-02		8E-02	1.89E+00	1.10E-01	
Total Cs [(b)]		3.7E-04	3.9E-05			1.2E-03	6.7E-05			4.27E+00	1.60E-01			4.21E+00	1.57E-01	
^{127}I	1E-01	1E-01		U	1E-01	1E-01		U	2E-01	8.37E-01	1.45E-01	J	2E-01	6.12E-01	3.62E-02	J
Li	4E-01	4E-01		U	4E-01	4E-01		U	2E-01	2.41E+01	1.81E-01	X	3E-01	2.35E+01	5.30E-01	X
Mo	1E+00	1E+00		U	1E+00	1E+00		U	2E+00	1.19E+01	4.35E-01	J	3E+00	1.35E+01	1.12E+00	J
Pr	1E-01	1E-01		U	1E-01	1E-01		U	8E-02	4.71E+01	2.11E-01		7E-02	4.67E+01	1.05E+00	
Pt	5E-02	5E-02		U	6E-02	6E-02		U	3E-02	3E-02		U	3E-02	3E-02		U
Rb	3E-01	3E-01		U	3E-01	3E-01		U	1E-01	1.11E+00	1.65E-01	J	2E-01	1.24E+00	5.59E-02	J
Sb	5E-02	5E-02		U	5E-02	5E-02		U	3E-02	1.34E-01	2.39E-02	J	3E-02	1.56E-01	2.86E-02	J
Se	1E+01	1E+01		U	1E+01	1E+01		U	1E+01	1E+01		U	1E+01	1E+01		U
Ta	3E-02	1.99E-01	9.14E-02	J	3E-02	2.00E-01	5.72E-02	J	2E-02	2.65E-01	2.28E-02	BX	2E-02	1.89E-01	1.70E-02	JBX
Te	7E-01	7E-01		U	7E-01	7E-01		U	4E-01	4.38E+00	4.31E-01		4E-01	4.07E+00	2.28E-01	
Tl	3E-02	3E-02		U	3E-02	3E-02		U	2E-02	2.03E-02	5.88E-03	J	2E-02	5.81E-02	5.08E-03	J
Th	7E-01	7E-01		U	7E-01	7E-01		U	1E+00	6.40E+01	1.21E+00		1E+00	6.18E+01	7.10E-01	
U	5E-01	5E-01		U	5E-01	5E-01		U	1E+00	2.80E+02	2.93E+00	X	1E+00	2.77E+02	2.34E+00	X
V	3E-02	5.14E+00	9.18E-02		3E-02	4.01E+00	3.25E-01		2E-01	5.10E+00	1.39E-01	B	2E-01	5.65E+00	2.69E-01	B
W	6E-01	6E-01		U	6E-01	6E-01		U	3E-01	4.26E+01	1.86E+00	X	3E-01	4.20E+01	2.48E+00	X
Y	1E-02	1E-02		U	1E-02	3.78E-02	9.44E-03	J	2E-01	2.61E+01	3.41E-01		2E-01	2.55E+01	9.67E-01	

(a) Concentration based on the mass of wet centrifuged solids; results contain a contribution from the interstitial supernatant.

(b) Total Cs is estimated based on the assumption the Cs isotopic distribution in the solids is equal to the Cs isotopic distribution in the liquid. The total Cs is determined relative to the ^{137}Cs measured by GEA in the solids and solids preparation blank.

(c) 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 (See Section 5.1).

Table 5.8. AN-102/C-104 Blended Feed Wet Centrifuged Solids, ICP-MS Radioisotope Results

	Process Blank 1 [(b)]				Process Blank 2 [(b)]				Sample [(a,b)]				Duplicate [(a,b)]			
	MDL	01-430-PB1	± 1SD	Data	MDL	01-430-PB2	± 1SD	Data	MDL	01-442	± 1SD	Data	MDL	01-442D	± 1SD	Data
Analyte	µg/g			Flag[(c)]	µg/g			Flag[(c)]	µg/g			Flag[(c)]	µg/g			Flag[(c)]
^{99}Tc	2E-01	2E-01		U	2E-01	2E-01		U	1.00E-01	1.99E+00	1.39E-01		2.00E-01	1.78E+00	1.49E-01	
^{129}I	1E-01	1E-01		U	1E-01	1E-01		U	1.00E-01	3.45E-01	5.74E-02	J	1.00E-01	3.11E-01	9.56E-03	J
^{233}U	8E-04	8E-04		U	6E-04	6E-04		U	8.00E-04	1.12E-01	6.03E-03		8.00E-04	1.18E-01	7.02E-03	
^{234}U	5E-03	5E-03		U	1E-03	1E-03		U	5.00E-03	2.14E-02	3.31E-03	J	5.00E-03	1.93E-02	8.26E-04	J
^{235}U	5E-03	5E-03		U	7E-03	7E-03		U	5.00E-03	2.07E+00	3.99E-02		5.00E-03	2.00E+00	7.18E-02	
^{236}U	3E-03	3E-03		U	2E-03	2E-03		U	3.00E-03	1.02E-01	2.46E-03		3.00E-03	9.31E-02	3.78E-03	
^{238}U	5E-01	5E-01		U	5E-01	5E-01		U	5.00E-01	2.77E+02	1.95E+00		5.00E-01	2.75E+02	4.28E+00	
^{237}Np	3E-03	3E-03		U	3E-03	3E-03		U	7.00E-02	9.27E-01	4.53E-02		6.00E-02	8.40E-01	4.08E-02	
^{239}Pu	4E-03	4E-03		U	3E-03	3E-03		U	1.00E-01	2.31E+00	4.65E-02		1.00E-01	2.16E+00	6.44E-02	
^{240}Pu	2E-03	2E-03		U	2E-03	2E-03		U	2.00E-02	1.71E-01	1.77E-02	J	2.00E-02	1.61E-01	1.60E-03	J
	µCi/g			Flag	µCi/g			Flag	µCi/g			Flag	µCi/g			Flag
^{99}Tc	3E-03	3E-03		U	3E-03	3E-03		U	2.00E-03	3.39E-02	2.37E-03		3.00E-03	3.03E-02	2.54E-03	
^{129}I	2E-05	2E-05		U	2E-05	2E-05		U	2.00E-05	6.10E-05	1.02E-05	J	2.00E-05	5.50E-05	1.69E-06	J
^{233}U	8E-06	8E-06		U	6E-06	6E-06		U	8.00E-06	1.09E-03	5.88E-05		8.00E-06	1.15E-03	6.85E-05	
^{234}U	3E-05	3E-05		U	7E-06	7E-06		U	3.00E-05	1.33E-04	2.06E-05	J	3.00E-05	1.20E-04	5.14E-06	J
^{235}U	1E-08	1E-08		U	2E-08	2E-08		U	1.00E-08	4.47E-06	8.62E-08		1.00E-08	4.33E-06	1.55E-07	
^{236}U	2E-07	2E-07		U	1E-07	1E-07		U	2.00E-07	6.62E-06	1.59E-07		2.00E-07	6.02E-06	2.45E-07	
^{238}U	2E-07	2E-07		U	2E-07	2E-07		U	2.00E-07	9.34E-05	6.57E-07		2.00E-07	9.24E-05	1.44E-06	
^{237}Np	2E-06	2E-06		U	2E-06	2E-06		U	4.00E-05	6.30E-04	3.08E-05		4.00E-05	5.71E-04	2.77E-05	
^{239}Pu	2E-04	2E-04		U	2E-04	2E-04		U	7.00E-03	1.43E-01	2.89E-03		7.00E-03	1.34E-01	4.00E-03	
^{240}Pu	5E-04	5E-04		U	5E-04	5E-04		U	5.00E-03	3.88E-02	4.01E-03	J	4.00E-03	3.66E-02	3.64E-04	J

(a) Concentrations are based on the mass of wet centrifuged solids; results contain a contribution from the interstitial supernatant.

(b) Results are presented in both µg/g and µCi/g for easy comparison to the TS minimum reportable quantities (MRQ) and direct comparison with radiochemistry data.

(c) Data flags: U = not detected above reported MDL; J = estimated value (See Section 5.1).

Decay correction reference date is nominally April 2001.

Table 5.9. AN-102/C-104 Blended Feed Wet Centrifuged Solids, Radioisotope Results

	Process Blank 1				Process Blank 2				Sample $^{(a)}$				Duplicate $^{(a)}$			
	MDA	01-430-PB1	Err %	Data Flag$^{(b)}$	MDA	01-430-PB2	Err %	Data Flag$^{(b)}$	MDA	01-442	Err %	Data Flag$^{(b)}$	MDA	01-442D	Err %	Data Flag$^{(b)}$
Analyte	µCi/mL				µCi/mL				µCi/mL				µCi/mL			
Alpha	2E-04	3.92E-03	4		2E-04	3.11E-03	4		6E-03	1.35E+00	2		7E-03	1.33E+00	2	
^{238}Pu	2E-06	1.27E-03	2		2E-06	8.59E-04	2		7E-04	2.61E-02	6		6E-04	2.64E-02	6	
$^{239/240}$Pu	2E-06	1.52E-04	3		2E-06	9.49E-05	4		8E-04	1.16E-01	3		5E-04	1.08E-01	3	
^{241}Am	5E-06	3.33E-04	5		6E-06	2.15E-04	6		5E-04	1.08E+00	2		1E-03	1.08E+00	2	
^{241}Am (GEA)	1E-03	1E-03		U	1E-03	1E-03		U	2E-02	1.16E+00	4		2E-02	1.15E+00	4	
$^{243/244}$Cm	5E-06	1.97E-03	3		5E-06	1.61E-03	3		4E-04	6.80E-02	3		9E-04	4.32E-02	5	
^{242}Cm	4E-06	4E-06		U	4E-06	4E-06		U	4E-04	3.52E-03	14	J	7E-04	2.48E-03	22	J
Sum of Alpha		3.73E-03	4			2.78E-03	4			1.29E+00	3			1.26E+00	3	
^{90}Sr	2E-03	7.84E-02	3		2E-03	4.59E-02	3		7E+00	4.31E+02	3		7E+00	4.02E+02	3	
^{134}Cs	5E-04	5E-04		U	4E-04	9.60E-04	13	J	2E-03	2E-03		UB	2E-03	2E-03		UB
^{137}Cs	6E-04	7.32E-03	6		5E-04	2.48E-02	3		6E-03	8.49E+01	2		6E-03	8.39E+01	2	
^{60}Co	4E-03	4E-03		U	4E-03	4E-03		U	6E-04	2.07E-02	2		7E-04	2.11E-02	2	
^{152}Eu	2E-03	2E-03		U	2E-03	2E-03		U	3E-03	2.15E-02	5		3E-03	2.18E-02	5	
^{154}Eu	2E-03	2E-03		U	2E-03	2E-03		U	3E-03	1.22E+00	2		3E-03	1.21E+00	2	
^{155}Eu	2E-03	2E-03		U	2E-03	2E-03		U	2E-02	7.27E-01	3		2E-02	7.16E-01	3	
^{125}Sb	1E-03	1E-03		U	1E-03	1E-03		U	2E-02	2E-02		U	2E-02	2E-02		U
^{126}SnSb	5E-04	5E-04		U	5E-04	5E-04		U	1E-02	1E-02		U	1E-02	1E-02		U

(a) Concentrations are based on the mass of wet centrifuged solids; results contain a contribution from the interstitial supernatant.

(b) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 5.1).

Decay correction reference date is nominally May 2001.

5.6 Undissolved Solids Results

Table 5.10 and Table 5.11 present the calculated UDS results derived from subtracting the contribution of the interstitial liquid from the WCS results. When the concentration of the analyte in the WCS is above the MDL, the density and wt% solids data from Table 3.2 have been used to calculate the concentration of each analyte in the UDS per Equation 5.1.

$$X = C - \left(\left(\frac{S}{D} \right) * \left(\frac{1-W}{1-T} \right) \right) \qquad \text{(Equation 5.1)}$$

Where: X = UDS analyte concentration (μg/g or μCi/g) on a per g of WCS (i.e., UDS concentration on a wet-weight basis)
C = average measured concentration of analyte in WCS (μg/g or μCi/g)
S = average measured concentration of analyte in supernatant (μg/mL or μCi/L)
D = density of supernatant (1.406 g/mL)
W = fractional solids weight in WCS after drying at 105°C (0.351)
T = fractional solids weight in supernatant after drying at 105°C (0.205).

Also reported in the Tables is the calculated analyte concentration on a dry-weight basis. This is calculated by subtracting the supernatant from the solids fraction according to Equations 5.2 and 5.3.

$$Y = \frac{X}{F} \qquad \text{(Equation 5.2)}$$

Where: Y = UDS analyte concentration (μg/g or μCi/g) on a per gram of UDS (i.e., UDS concentration on a dry-weight basis)
X = UDS concentration (μg/g or μCi/g) on a per g of WCS (defined above)
F = UDS weight fraction (g undissolved solids per g WCS) and F is calculated according to Equation 5.3.

$$F = 1 - \left[\frac{(1-W)}{(1-T)} \right] \qquad \text{(Equation 5.3)}$$

The following apply to both UDS calculations:

a) If the analyte is not measured on the supernatant, the supernatant is assumed to make no contribution to the WCS and 'S' is set to zero (0). (i.e., 'X' = 'C')
b) If the analyte is measured on the supernatant, but not detected above the MDL, 'S' is set to zero (0). (i.e., 'X' = 'C')
c) If the analyte is measured on the WCS but is not detected, the UDS concentration is set to <MDL of the WCS even if the analytes is detected in the supernatant. (i.e., 'X' = < 'C')
d) If the calculated UDS analyte concentration is less than or equal to zero, the analyte is assumed to come only from the supernatant within uncertainty of the measurement method. The UDS concentration is then set to <MDL of the WCS. (i.e., 'X' = < 'C')
e) If only one of the duplicate sample results is above the MDL, this value is used in place of the "average" concentration (i.e., 'C' or 'S') in the calculation.

The results presented in Table 5.10 and Table 5.11 represent only those analytes analyzed in the WCS. Due to the lack of WCS available for characterization some of the analytes defined by the TS could not be analyzed and therefore their contribution to the UDS is unknown.

Table 5.10. AN-102/C-104 Blended Feed Undissolved Solids Concentration, µg/g on a Wet Centrifuged Solids and Dry Solids Basis

	WCS				**Supernatant (a)**			**UDS (b)**	
Analyte	**Measure Method**	**MDL µg/g**	**Average µg/g**	**Data Flag(d)**	**Measure Method**	**Average µg/mL**	**Data Flag(d)**	**Average µg/g (wet)**	**Average µg/g (dry)**
Ag	ICP-AES	2E+00	2E+00	UX	ICP-AES		UX	<2E+00	<9E+00
Al	ICP-AES	4E+00	5.56E+04		ICP-AES	5.34E+03		5.2E+04	2.8E+05
As	ICP-AES	2E+01	1.70E+01	J	ICP-AES	0	U	1.7E+01	9.3E+01
As	ICP-MS	2E+00	6.88E+00	JB	N/M			6.9E+00	3.8E+01
B	ICP-AES	3E+00	5.70E+01	B	ICP-AES	5.00E+01	B	2.2E+01	1.2E+02
B	ICP-MS	2E+00	1.74E+01	JBX	N/M			1.7E+01	9.5E+01
Ba	ICP-AES	6E-01	7.90E+01		ICP-AES	0	U	7.9E+01	4.3E+02
Be	ICP-AES	6E-01	7.05E+00		ICP-AES	0	U	7.1E+00	3.8E+01
Be	ICP-MS	3E-01	1.43E+01		N/M			1.4E+01	7.8E+01
Bi	ICP-AES	6E+00	1.15E+01	J	ICP-AES	0	U	1.2E+01	6.3E+01
Ca	ICP-AES	2E+01	4.23E+02		ICP-AES	1.53E+02	X	3.2E+02	1.7E+03
Cd	ICP-AES	1E+00	2.00E+01		ICP-AES	1.67E+01		8.3E+00	4.5E+01
Ce	ICP-AES	1E+01	7.10E+01	J	ICP-AES	0	U	7.1E+01	3.9E+02
Ce	ICP-MS	2E-01	7.02E+01		N/M			7.0E+01	3.8E+02
Co	ICP-AES	3E+00	3E+00	U	ICP-AES	0	U	<3E+00	<2E+01
Co	ICP-MS	7E-02	1.38E+00		N/M			1.4E+00	7.5E+00
Cr	ICP-AES	1E+00	4.57E+03		ICP-AES	6.91E+01		4.5E+03	2.5E+04
Cu	ICP-AES	2E+00	1.20E+01	J	ICP-AES	6.75E+00	J	7.2E+00	4.0E+01
Dy	ICP-AES	3E+00	3E+00	U	ICP-AES	0	U	<3E+00	<2E+01
Eu	ICP-AES	6E+00	6E+00	U	ICP-AES	0	U	<6E+00	<3E+01
Fe	ICP-AES	2E+00	3.45E+03		ICP-AES	6.55E+00	JB	3.4E+03	1.9E+04
K	ICP-AES	1E+02	5.65E+02	JX	ICP-AES	5.45E+02	JX	1.8E+02	9.9E+02
La	ICP-AES	3E+00	1.06E+02		ICP-AES	3.25E+00	J	1.0E+02	5.6E+02
Li	ICP-AES	2E+00	1.10E+01	J	ICP-AES	1.60E+00	J	9.9E+00	5.4E+01
Li	ICP-MS	2E-01	2.38E+01	X	N/M			2.4E+01	1.3E+02
Mg	ICP-AES	6E+00	5.40E+01	J	ICP-AES	0	U	5.4E+01	2.9E+02
Mn	ICP-AES	3E+00	7.65E+02		ICP-AES	0	U	7.6E+02	4.2E+03
Mo	ICP-AES	3E+00	1.55E+01	J	ICP-AES	1.55E+01	J	4.6E+00	2.5E+01
Mo	ICP-MS	2E+00	1.27E+01	J	N/M			1.3E+01	6.9E+01
Na	ICP-AES	1E+01	5.90E+04		ICP-AES	7.32E+04		7.4E+03	4.1E+04
Nd	ICP-AES	6E+00	1.80E+02		ICP-AES	6.45E+00	J	1.8E+02	9.6E+02
Ni	ICP-AES	2E+00	1.31E+02	X	ICP-AES	1.22E+02		4.5E+01	2.5E+02
P	ICP-AES	6E+00	1.06E+03		ICP-AES	6.01E+02		6.4E+02	3.5E+03
Pb	ICP-AES	6E+00	4.06E+02	B	ICP-AES	4.90E+01	J	3.7E+02	2.0E+03
Pd	ICP-AES	5E+01	5E+01	U	ICP-AES	0	U	<5E+01	<3E+02

Table 5.11. (Cont'd)

	WCS				Supernatant (a)			UDS (b)	
Analyte	Measure Method	MDL µg/g	Average µg/g	Data Flag(d)	Measure Method	Average µg/mL	Data Flag(d)	Average µg/g (wet)	Average µg/g (dry)
Pr	ICP-MS	8E-02	4.69E+01		N/M			4.7E+01	2.6E+02
Pt	ICP-MS	3E-02	3E-02	U	N/M			<3E-02	<2E-01
Rb	ICP-MS	1E-01	1.18E+00	J	ICP-MS	2.28E+00		<1E+00 (C)	<5E+00 (C)
Rh	ICP-AES	2E+01	2E+01	U	ICP-AES	0	U	<2E+01	<1E+02
Ru	ICP-AES	7E+01	7E+01	U	ICP-AES	0	U	<7E+01	<4E+02
Sb	ICP-AES	3E+01	3E+01	U	ICP-AES	0	U	<3E+01	<2E+02
Sb	ICP-MS	3E-02	1.45E-01	J	N/M			1.5E-01	7.9E-01
Se	ICP-AES	2E+01	2E+01	U	ICP-AES	0	U	<2E+01	<9E+01
Se	ICP-MS	1E+01	1E+01	U	N/M			<1E+01	<5E+01
Si	ICP-AES	3E+01	3.63E+03		ICP-AES	2.80E+02	BX	3.4E+03	1.9E+04
Sn	ICP-AES	1E+02	9E+01	U	ICP-AES	0	U	<9E+01	<5E+02
Sr	ICP-AES	1E+00	2.40E+01		ICP-AES	0	U	2.4E+01	1.3E+02
Ta	ICP-MS	2E-02	2.27E-01	JBX	N/M			2.3E-01	1.2E+00
Te	ICP-AES	1E+02	9E+01	U	ICP-AES	0	U	<9E+01	<5E+02
Te	ICP-MS	4E-01	4.23E+00		N/M			4.2E+00	2.3E+01
Th	ICP-AES	6E+01	1.60E+02	J	ICP-AES	0	U	1.6E+02	8.7E+02
Th	ICP-MS	1E+00	6.29E+01		N/M			6.3E+01	3.4E+02
Ti	ICP-AES	2E+00	3.25E+00	J	ICP-AES	0	U	3.3E+00	1.8E+01
Tl	ICP-AES	3E+01	3E+01	U	ICP-AES	0	U	<3E+01	<2E+02
Tl	ICP-MS	2E-02	3.92E-02	J	N/M			3.9E-02	2.1E-01
U	ICP-AES	1E+02	3.20E+02	J	ICP-AES	0	U	3.2E+02	1.7E+03
U	ICP-MS	1E+00	2.79E+02	X	ICP-MS	1.50E+01		2.7E+02	1.5E+03
V	ICP-AES	3E+00	3E+00	U	ICP-AES	0	U	<3E+00	<2E+01
V	ICP-MS	2E-01	5.38E+00	B	N/M			5.4E+00	2.9E+01
W	ICP-AES	1E+02	1E+02	U	ICP-AES	0	U	<1E+02	<7E+02
W	ICP-MS	3E-01	4.23E+01	X	N/M			4.2E+01	2.3E+02
Y	ICP-AES	3E+00	2.60E+01	J	ICP-AES	0	U	2.6E+01	1.4E+02
Y	ICP-MS	2E-01	2.58E+01		N/M			2.6E+01	1.4E+02
Zn	ICP-AES	3E+00	7.90E+01		ICP-AES	3.30E+00	J	7.7E+01	4.2E+02
Zr	ICP-AES	3E+00	7.80E+01	X	ICP-AES	0	U	7.8E+01	4.3E+02

WCS = wet centrifuged solids; UDS = undissolved solids; n/m = 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, the supernatant is assumed to have no contribution to the WCS results; the average field is left blank and the supernatant concentration is set to 0 (zero) when calculating the UDS concentration.

(b) If analyte is measured in the WCS, but is not above the MDL, the UDS results is set to < MDL of the WCS.

(c) If the calculated UDS results is <0 or =0, the UDS results is set to <MDL of the WCS.

(d) 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 (See Section 5.1).

Table 5.11. AN-102/C-104 Blended Feed Undissolved Solids Concentration, µCi/g on a Wet Centrifuged Solids and Dry Solids Basis

	WCS				**Supernatant** $^{(a)}$			**UDS** $^{(b)}$	
Analyte	**Measure Method**	**MDL/MDA µCi/g**	**Average µCi/g**	**Data Flag**$^{(c)}$	**Measure Method**	**Average µCi/mL**	**Data Flag**$^{(c)}$	**Average µCi/g (wet)**	**Average µCi/g (dry)**
^{60}Co	Rad	6E-04	2.09E-02		Rad	2.54E-02		3.0E-03	1.6E-02
^{90}Sr	Rad	7E+00	4.17E+02		Rad	1.38E+01		4.1E+02	2.2E+03
^{99}Tc	ICP-MS	2E-03	3.21E-02		ICP-MS	4.24E-02		2.2E-03	1.2E-02
^{125}Sb	Rad	2E-02	2E-02	U	n/m			<2E-02	<1E-01
^{126}SnSb	Rad	1E-02	1E-02	U	n/m			<1E-02	<5E-02
^{129}I	ICP-MS	2E-05	5.80E-05	J	n/m			5.8E-05	3.2E-04
^{134}Cs	Rad	2E-03	2E-03	U	n/m			<2E-03	<1E-02
^{137}Cs	Rad	6E-03	8.44E+01		Rad	1.02E+02		1.3E+01	6.8E+01
^{152}Eu	Rad	3E-03	2.17E-02		n/m			2.2E-02	1.2E-01
^{154}Eu	Rad	3E-03	1.22E+00		Rad	5.82E-02		1.2E+00	6.4E+00
^{155}Eu	Rad	2E-02	7.22E-01		Rad	3.77E-02		6.9E-01	3.8E+00
^{233}U	ICP-MS	8E-06	1.12E-03		n/m			1.1E-03	6.1E-03
^{234}U	ICP-MS	3E-05	1.27E-04	J	n/m			1.3E-04	6.9E-04
^{235}U	ICP-MS	1E-08	4.40E-06		n/m			4.4E-06	2.4E-05
^{236}U	ICP-MS	2E-07	6.32E-06		n/m			6.3E-06	3.4E-05
^{237}Np	ICP-MS	4E-05	6.01E-04		ICP-MS	5.21E-05		5.6E-04	3.1E-03
^{238}Pu	Rad	7E-04	2.63E-02		Rad	4.89E-04	BJ	2.6E-02	1.4E-01
^{238}U	ICP-MS	2E-07	9.29E-05		n/m			9.3E-05	5.1E-04
$^{239/240}$Pu	Rad	8E-04	1.12E-01		Rad	1.49E-03		1.1E-01	6.1E-01
^{239}Pu	ICP-MS	7E-03	1.39E-01		ICP-MS	1.72E-03	J	1.4E-01	7.5E-01
^{240}Pu	ICP-MS	5E-03	3.77E-02	J	ICP-MS	6.52E-04	J	3.7E-02	2.0E-01
^{241}Am	Rad	5E-04	1.08E+00		Rad	3.75E-02		1.1E+00	5.7E+00
^{241}Am (GEA)	Rad	2E-02	1.16E+00		Rad	3.70E-02	J	1.1E+00	6.2E+00
^{242}Cm	Rad	4E-04	3.00E-03	J	Rad	1.55E-04	J	2.9E-03	1.6E-02
$^{243/244}$Cm	Rad	4E-04	5.56E-02		Rad	1.92E-03	B	5.4E-02	3.0E-01
Alpha	Rad	6E-03	1.34E+00		Rad	4.62E-02		1.3E+00	7.1E+00
Sum of Alpha	Rad		1.28E+00		Rad	4.15E-02		1.2E+00	6.8E+00

WCS = wet centrifuged solids; UDS = undissolved solids; n/m = 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, the supernatant is assumed to have no contribution to the WCS results; the average field is left blank and the supernatant concentration is set to 0 (zero) when calculating the UDS concentration.

(b) If analyte is measured in the WCS, but is not above the MDL, the UDS results is set to < MDL of the WCS.

(c) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 5.1).

5.7 Comparison of Supernatant Results to Specification 7

Specification 7 for Envelope C defines limits for several analytes relative to sodium concentration (moles analyte per mole Na or Bq analyte per mole Na). Table 5.12 and Table 5.13 present the ratio limits and the as-measured ratios. In all cases the mole or Bq analyte to moles Na ratio did not exceed the limits defined in Specification 7 for Envelope C.

Table 5.12. AN-102/C-104 Blended Feed Supernatant - Measured Mole Analyte per Mole Na Ratio Results

Analyte	MRQ µg/mL	MDL/EQL (a) µg/mL	01-441 Average µg/mL	Data Flag(d)	Measured Mole Analyte per Mole Na Ratio	Specification 7 Envelope C Limits Mole Analyte per Mole Na Ratio	% of Limit	Meets Spec 7?
Al	75	3.0	5,340		6.2E-02	2.5E-01	25	Yes
Ba	2.3	0.5	0.5	U	<1.1E-06	1.0E-04	< 1	Yes
Ca	150	13	153	X	1.2E-03	4.0E-02	3	Yes
Cd	7.5	0.8	17		4.7E-05	4.0E-03	1	Yes
Cl	300	200	1,150		1.02E-02	3.7E-02	28	Yes
Cr	15	1.0	69		4.2E-04	6.9E-03	6	Yes
F	150	200	3,700		6.12E-02	9.1E-02	67	Yes
Fe	150	1.3	6.6	JB	3.7E-05	1.0E-02	0	Yes
K	75	100	545	JX	4.4E-03	1.8E-01	2	Yes
La	35	2.5	3.3	J	7.3E-06	8.3E-05	9	Yes
Na	75	38	73,200			N/A		
Ni	30	1.5	122		6.5E-04	3.0E-03	22	Yes
NO_2	3,000	500	25,900		1.76E-01	3.8E-01	46	Yes
NO_3	3,000	2,000	61,000		3.09E-01	8.0E-01	39	Yes
Pb	300	5.0	49	J	7.4E-05	6.8E-04	11	Yes
PO_4 as P (b)	600	5.0	601		6.1E-03	3.8E-02	16	Yes
PO_4	2,500	200	200	U	<6.6E-04	3.8E-02	< 2	Yes
SO_4	2,300	400	4,430		1.45E-02	2.0E-02	73	Yes
TIC-P (c)	150	33	6,000		1.57E-01	3.0E-01	52	Yes
TOC-P (c)	1,500	87	9,500		2.48E-01	5.0E-01	50	Yes
TIC-F (c)	150	100	300	J	7.84E-03	3.0E-01	3	Yes
TOC-F (c)	1,500	100	15,800		4.12E-01	5.0E-01	82	Yes
U (ICP)	600	100	100	U	<1.3E-04	1.2E-03	< 11	Yes

(a) F, Cl, NO_2, NO_3, PO_4, and SO_4 report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

(b) Phosphate based on ICP-AES average total P result of 385 µg/mL.

(c) For TOC and TIC: P=by hot persulfate method; F=by furnace method:TIC by difference (TIC =TC–TOC).

(d) 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 (See Section 5.1).

Table 5.13. AN-102/C-104 Blended Feed Supernatant - Measured Bq Analyte per Mole Na Ratio Results

Radioisotopes	MRQ µCi/mL	MDA/ MDL (a) µCi/mL	01-441 Average µCi/mL	Data Flag	Measured Bq Analyte per Mole Na Ratio	Specification 7 Envelope C Limits Bq Analyte per Mole Na Ratio	% of Limit	Meets Spec 7?
TRU(b)	2.3E-01 (C)	n/a	4.13E-02		4.80E+05	3.0E+06	16	Yes
^{137}Cs	9.0E+00	7.0E-03	1.02E+02		1.18E+09	4.3E+09	27	Yes
^{90}Sr	1.5E-01	4.0E-01	1.38E+01		1.60E+08	8.0E+08	20	Yes
^{99}Tc	1.5E-03	1.7E-03	4.24E-02		4.92E+05	7.1E+06	7	Yes
^{60}Co	1.0E-02	3.5E-04	2.54E-02		2.95E+05	3.7E+05	80	Yes
^{154}Eu	2.0E-03	2.0E-03	5.81E-02		6.75E+05	4.3E+06	16	Yes

(a) Values represent MDA for all analytes except ^{99}Tc.

(b) TRU = alpha emitting radionuclides with atomic number greater than 92 and half-life greater than ten years; alpha summation of ^{238}Pu, $^{239+240}$Pu, ^{241}Am, and $^{243+244}$Cm.

(c) MRQ for total alpha used as TRU MRQ.

Decay correction reference date is nominally April 2001 to May 2001.

6.0 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 Program's plan *Conducting Analytical Work in Support of Regulatory Programs*, which is in compliance with HASQARD. Raw data including bench sheets, instrument printouts, data reduction, and calibration files are maintained or cross-referenced in Project 42365 files.

The QC and sample results evaluations provided in the following sections are limited to the analytes of interest defined by the TS. Analytes other than those specified by the TS are included in some tables and are provided for additional information. Some of these other analytes were measured per the requirements stated in the governing QA Plan; however, the data has not been fully evaluated against the acceptance criteria.

The sample average, MRQ, data flags, QC parameters and QC acceptance criteria are summarized in Table 6.1 through Table 6.10. In some cases, one sample value was reported as less than the MDL/MDA (i.e., U flagged) and the duplicate reported with a value (i.e., either J flagged or a value measured above the EQL). The reported average is conservatively estimated as the one reported value above the MDL/MDA.

6.1 Inductively Coupled Plasma-Atomic Emission Spectroscopy

Tables 5.1, 5.6, 6.2 and 6.7

The PNL-ALO-128 and -129 acid digested samples required 5-fold dilutions in order to quantify all analytes of interest 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 increased to 100%.

Quality control for the ICP-AES analysis consisted of sample duplicates, PBs, MSs, LCS (or BS), post spikes, serial dilution, calibration verification check standards, interference check standards, and linear range check standards. Matrix spike recovery, LCS (or BS) recovery and precision (based on duplicate analyses) QC acceptance criteria are defined by the TS. These QC criteria were evaluated in detail and are summarized below.

Duplicates analyzed for the analytes of interest with concentrations greater than the EQL for supernatant and centrifuged solids demonstrated Relative Percent Differences (RPD) within the acceptance criteria of <15%, except for calcium (21%) in the supernatant samples and nickel (40%) and zirconium (51%) in the centrifuged solids sample. Sodium values from the supernatant and centrifuged solids samples were recovered within the acceptance criteria of <3.5% RPD.

All PB analytes of interest were within acceptance of ≤EQL or ≤5% of sample concentration in the prepared samples except for lead in the centrifuged solids samples.

Serial dilution was required for aluminum and sodium for the supernatant analysis and aluminum, chromium, and sodium in the centrifuged solids analysis. Except for sodium in the supernatant analysis, the serial dilution results were within the acceptance criteria for percent difference (%D) of <±10%. The sodium in the supernatant was so high that sufficient successive serial dilutions were not obtained during the analysis of the sample to calculate the %D. Since the supernatant sodium concentration is too high for matrix spiking and the data for serial dilution was not obtained, the sodium results have been flagged with and "X", indicating a QC deficiency. However, the other analytical QC for sodium (i.e., LCS/BS and calibration checks) indicates that the reported sodium results are most likely accurate.

The AN-102/C-104 blended feed and the AN-102 as-received materials were acid digested in the same batch. A MS was prepared from the AN-102 as-received supernatant and the AN-102/C-104 blend and AN-102 as-received centrifuged solids. The MS results reported in Table 6.2 are for the AN-102 as-received MS. All matrix-spiked analytes of interest (spiked at greater than 20% of the sample concentration) were recovered within acceptance criteria of 75% to 125% except potassium (65%) in supernatant sample and silver (35%), copper (129%), magnesium (131%), nickel (163%), palladium (134%), and zirconium (54%) in the dry centrifuged solid. Low silver recovery is most likely due to the small amount of hydrochloric acid used during sample processing (or from chloride present in the sample) resulting in some silver chloride precipitation. Silver was not detected in the samples. Low or over recovery of the other analytes may be due to non-homogeneity of the dried centrifuged solids. All analytes of interest were post spiked and recovered within acceptance criteria of 75% to 125%.

For the LCS/BS, all analytes of interest were recovered within acceptance criteria of 80% to 120% except potassium in liquid LCS and silver in the solids LCS. Low recovery of silver in the BS (22%) for the solid sample preparation is most likely due to the small amount of hydrochloric acid used during sample preparation resulting in some silver chloride precipitation. Potassium was only slightly low (72%) for the liquid sample preparations.

6.2 Inductively Coupled Plasma-Mass Spectrometry

Tables 5.2, 5.7, 5.8, 6.3 and 6.8

The PNL-ALO-128 and -129 acid digested samples of the supernatant and dried centrifuged solids, respectively, were submitted for ICP-MS analysis according to procedure PNL-ALO-280 Rev. 1, *Inductively Coupled Plasma-Mass Spectrometer Analysis*. Except for the MS and LCS, the acid digested samples were from the same processed solutions as were delivered for ICP-AES analysis.

Quality control for the ICP-MS analysis consisted of sample duplicates, PBs, MS, LCS or BS, post spikes, and calibration verification check standards and blanks. Matrix spike recoveries, LCS recovery, and precision (based on duplicate analyses) QC criteria are defined by the TS. These QC criteria were evaluated in detail and are summarized below.

Many of the QC failures noted in the Tables (i.e., LCS, MS and PS recoveries) are attributed to lithium, beryllium, boron, and/or selenium analyses and in many cases have wide variations in the results. Lithium, beryllium, and boron are at the end of the detector range and the instrument response tends to have a wide variation in this region. Selenium may have a wide variation in the results due to the extensive chain of isobaric interference corrections. Also, selenium is difficult to ionize; therefore, its sensitivity is very poor.

Duplicates analyzed for the analytes of interest with concentrations greater than the EQL for supernatant and centrifuged solids fractions demonstrated RPDs within the acceptance criteria of <15%, except for cobalt (75%) of the centrifuged solids sample.

All PBs for the analytes of interest were within acceptance of ≤EQL or ≤5% of sample concentration in the prepared samples except for boron, tantalum, and vanadium in the centrifuged solids samples.

The AN-102/C-104 blended feed and the AN-102 as-received materials were acid digested in the same batch. A MS was prepared from the AN-102 as-received supernatant and the AN-102 as-received centrifuged solids. The element MS results reported in Table 6.3 and Table 6.8 are for the AN-102 as-received MS. The matrix-spiked analytes of interest were recovered within acceptance criteria except boron (35%), lithium (241%), and tungsten (66%) in the centrifuged solids sample. Nearly all analytes of interest were post spiked and recovered within acceptance criteria of 75% to 125%, except cerium (135%), lithium (139%), selenium (135%), and ^{240}Pu (69%).

All LCS/BS analytes of interest were recovered within acceptance criteria except for boron (52%), lithium (122%), tantalum (76%), and tungsten (69%) in the centrifuged solids sample.

Cesium isotopic atomic abundance for ^{133}Cs, ^{135}Cs, and ^{137}Cs were determined on the AN-102/C-104 blended feed supernatant in duplicate. The ^{133}Cs atomic abundance (0.603 and 0.603), ^{135}Cs atomic abundance (0.166 and 0.165), and ^{137}Cs atomic abundance (0.231 and 0.232) are assumed to be the same in the supernatant and in the centrifuged solids. These atomic abundances are used for determining the total Cs in the supernatant (by ICP-MS) and in the solids (using the ^{137}Cs radiochemical results).

6.3 U Analysis by KPA

Tables 5.2 and 6.3

Acid digested (PNL-ALO-128) sample solutions of the AN-102/C-104 blended feed supernatant were evaporated dry with nitric acid, then re-dissolved in dilute nitric acid for uranium analysis. Total uranium was measured according to procedure RPG-CMC-4014, *Uranium by Kinetic Phosphorescence Analysis*. No uranium separation was performed. The uranium content was well below the MRQ value of 780 µg/mL. The repeatability of duplicate samples was excellent with a RPD value of 7%. Uranium was detected in the SAL hot cell blanks, but at a concentration less than 1% of the sample uranium concentration. The LCS (i.e., a mid-range standard) recovered at 100% and within the 80% to 120% acceptance criteria. No MS sample was analyzed.

6.4 Radiochemical Analyses

Tables 5.3, 5.9, 6.4 and 6.10

The supernatant and centrifuged solids samples were acid digested according to procedures PNL-ALO-128 and PNL-ALO-129, respectively. The supernatant and centrifuged solid digest was analyzed for gamma emitters, ^{90}Sr, total alpha, $^{239+240}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and $^{243+244}$Cm. An aliquot of the supernatant was provided directly from the SAL hot cells (i.e., not subjected the acid digestion procedure) for subsequent analysis of the pertechnetate form of ^{99}Tc.

6.4.1 Gamma Spectrometry

Digested sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Photon Spectrometry*. Laboratory blanks and spikes were not prepared, nor required, for this analysis since the measurement is a direct reading of the gamma energy and is not subject to matrix interferences. Initially, the samples were diluted prior to GEA. However, in order to meet the requested MRQs, direct aliquots of the SAL hot cell preparations were counted for periods of 4 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. Most of the samples also showed the presence of ^{60}Co, ^{154}Eu, ^{155}Eu, and ^{241}Am. The MRQ values for extended counting time GEA were met in all cases. All of the SAL hot cell blanks showed the presence of ^{137}Cs, but the activities in the blanks were negligible with respect to the samples. For those radioisotopes determined at concentrations greater than 10 times the MDA, the sample duplicates showed excellent repeatability with RPD values <15%.

6.4.2 Total Alpha

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001, *Source Requirements for Gross Alpha and Gross Beta Analysis*. The samples were then counted on Ludlum detectors according to RPG-CMC-408, *Low Background Alpha and Beta Counting - Proportional*. The sums of the individual alpha emitters, as discussed below, are in very good agreement with the total alpha data indicating minimal losses due to alpha self-absorption. All of the SAL hot cell preparation batches showed alpha contamination at approximately 2% of the sample concentration. Although this effect is not so evident in the total alpha data, the data for individual alpha emitters is compromised in some cases. The LCS and MS recoveries with ^{239}Pu were 100% and 103%, respectively. No alpha contamination was detected in the laboratory workstation blank. The duplicate sample RPD values are < 3%.

6.4.3 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 RPG-CMC-422, *Solution Analysis: Alpha Spectrometry*. 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 excellent, averaging about 95%. Neither Pu, Am, nor Cm were detected in the laboratory workstation blank. However, all of the SAL hot cell PBs indicated significant contamination for ^{238}Pu and $^{243/244}$Cm. For the first hot cell batch with samples of the supernatant, the PB contamination levels were ~100% and ~40% of the sample activities for ^{238}Pu and $^{243/244}$Cm, respectively. For the second SAL hot cell batch with samples of the centrifuged solids, the PB contamination levels were 12% of the sample activities for $^{243/244}$Cm. Other alpha emitting isotope contamination levels were not significant in either PB.

The LCS recoveries for ^{241}Am and $^{239/240}$Pu were 96% and 107%, respectively. The MS recoveries for ^{241}Am and $^{239/240}$Pu were 97% and 105%, respectively. For those radioisotopes determined at concentrations greater than 10 times the MDA, the RPD values were within the acceptance criteria of <15% except for $^{243/244}$Cm (36% supernatant and 45% centrifuged solids).

6.4.4 Strontium-90

The Sr separation was performed according to PNL-ALO-476, *Strontium Determination using Sr-SPEC*, and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta counted according to RPG-CMC-408, *Low Background Alpha and Beta Counting – Proportional* (for ^{90}Sr determination). Following the beta counting, the samples were gamma counted according to PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Photon Spectrometry* (for ^{137}Cs impurity assessment). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. However, this correction was negligible with respect to the activity in the samples. No ^{90}Sr was found in the laboratory workstation blank. The SAL hot cell PBs showed ^{90}Sr activities, although the levels were not significant. RPD values of the duplicates were ≤7%, and the LCS and MS recoveries were 88% and 91%, respectively. Per the TS, the MS was not required and the acceptance criterion was not defined.

6.4.5 Technetium-99 (as Pertechnetate)

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. A small aliquot from the blended feed 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 the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure was otherwise 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 RPG-CMC-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 LCS (or BS) recovery of a ^{99}Tc standard was 92%. However, the MS gave a recovery of 248%, which was outside of the 70% to 130% acceptance criteria. The batch MS was prepared from the AN-102 as-received sample that was processed and analyzed in the same batch as the AN-102/C-104 blended feed. The quantity of pertechnetate added to the AN-102 as-received sample contributed only about 12% to the measure pertechnetate. The MS preparation benchsheets provide no indication that an error was made in either the preparation of the MS solution or the addition of the MS to the sample. Since the poor MS recovery is most likely due to insufficient MS addition and the measured ^{99}Tc (as pertechnetate) is about 50% of the total ^{99}Tc (which is in a typical range for tank waste material), the reported results are considered good. However, due to the MS failure, the results have been flagged as having a QC deficiency.

The ^{99}Tc activities in the SAL hot cell processing blank and a laboratory reagent blank were negligible or non-detectable and well below the requested MRQ value of 0.0015 µCi/mL. No duplicate was sub-sampled in the SAL; however, a replicate was prepared at the laboratory workstation and the sample replicates demonstrated good agreement with an RPD of 1%.

6.5 Inorganic Anions

Tables 5.4 and 6.5

Inorganic anion analysis was conducted according to method PNL-ALO-212, *Determination of Inorganic Anions by Ion Chromatography*. The method was used to evaluate the anions of interest on unprocessed sub-samples of supernatant. No IC analyses were performed on the AN-102/C-104 centrifuged solids sample due to insufficient quantity of solids available for water leaching.

The supernatant samples did not require preparation, but were diluted 800-fold to 10,000-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample at dilutions less than 800-fold. Supernatant samples of the AN-102 as-received composite and the AN-102/C-104 blended feed composite were processed in a single batch and thus have the same batch and analysis QC.

Quality control for the anions analysis consisted of sample duplicates, PBs, MSs, BSs, and calibration verification check standards and blanks. The MS recovery, BS recovery, and precision (based on duplicate analyses) QC acceptance criteria are defined by the TS. These QC criteria were evaluated in detail and are summarized below.

A MS was prepared from a sample of the AN-102 as-received supernatant (sample 01-429) and analyzed with the AN-102/C-104 blended feed supernatant samples. Fluoride and nitrate spikes of the initial MS produced very high recoveries (>130%). The supernatant MS was re-prepared and reanalyzed; meeting the acceptance criteria of 75% to 125% recovery. The duplicate RPD for the supernatant sample met the acceptance criterion of <15%.

A LCS (or BS) and PB (i.e., water used in SAL for dilutions and handled like the sample) were prepared in the SAL and analyzed at the same time as the samples and MS. The LCS demonstrated recoveries within the acceptance criteria of 80% to 120%, and no anions were detected in the SAL PB.

6.6 TOC/TIC by Hot Persulfate and Furnace

Tables 5.4 and 6.5

The AN-102/C-104 blended feed supernatant was analyzed for total 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*. No TIC/TOC analyses were performed on the AN-102/C-104 centrifuged solids sample due to insufficient quantity of solids available for analysis.

6.6.1 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 were corrected for average percent recovery of system calibration standards and were also corrected for contribution from the system blanks, as per procedure PNL-ALO-381 calculations. The QC for the method involves sample duplicates, LCS (or BS), and a MS.

For the supernatant analysis, five calibration blanks were run at the beginning, middle, and end of the analysis run averaging 21 µgC TIC and 74 µgC TOC. These calibration/system blanks are considered acceptable for the concentration of TIC and TOC measured in the samples. However, the standard deviation for the TIC blanks was outside the historical pooled standard deviation used to establish the MDL; this indicates that there is significantly more variability in the blank than normal.

All QC sample results for hot persulfate TIC/TOC analysis method meet the acceptance criteria defined by the TS. The RPD for duplicates is <15%. The LCS (or BS) recoveries ranged form 100% to 104%, well within the acceptance criteria of 80% to 120%. The performance of the MS was similar with recoveries ranging from 98% to 99%, well within the acceptance criteria of 75% to 125%.

6.6.2 Furnace Oxidation Method (PNL-ALO-380)

The furnace method that determines the TOC is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 750°C for 30 minutes. The total carbon (TC) is determined on another aliquot of the sample by combusting at 1000°C for 30 minutes, and the TIC is obtained by difference between the TC and TOC. All sample results were corrected for average percent recovery of system calibration standards and were also corrected for contribution from the system blanks, as per procedure PNL-ALO-380 calculations. The QC for the method involves sample duplicates, LCS (or BS), and a MS.

For the supernatant analysis, calibration blanks were analyzed at the beginning, middle, and end of the analysis runs (two days). The TOC determination produced average blanks of 5 µgC and the TC determination produced average blanks of 4 µgC, both well within the procedures acceptance range for the calibration blank.

All QC sample results for furnace TC and TOC analysis method meet the acceptance criteria defined by the TS. For duplicate results The RPD for duplicates is <15%. The LCS (or BS) recoveries ranged form 97 % to 99%, well within the acceptance criteria of 80% to 120%. The performance of the MS was similar with recoveries ranging from 85% to 95%, well within the acceptance criteria of 75% to 125%.

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

Table 5.4 presents the TOC and TIC results obtained from the hot persulfate method and the furnace oxidation method for the AN-102/C-104 blended feed supernatant. 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.

The average TC result from the hot persulfate method is 15,400 µg C/mL and the average TC result from the furnace method is 16,100 µg C/mL; essentially the same results. However, there are significant differences between the TIC and TOC results reported by each method. The reason for the discrepancy between the persulfate method and furnace method is unknown, but it appears that the TIC, perhaps in the form of easily oxidized metal carbonate, is being combusted at 750°C with the furnace method and thus measuring the same carbon concentration as that measured at 1000°C (i.e., TOC + TIC). Evaluation of all the data suggests that the hot persulfate results provide the best estimate of the TIC results and the furnace method provides the best estimate of the TC results. Also, it is unlikely that there is no TIC in the samples based on the OH titration (Section 6.7), which produced inflection points typical of carbonate. Although these inflection points are not unique to carbonate in complex tank matrices (i.e., aluminate contributes significantly to the first inflection point) and have not been verified in the OH titration, the estimated carbon (as carbonate) from the OH titration equate to about 4,600 µg C/mL (or approximately equivalent to the hot persulfate results).

6.7 Hydroxide Titration

Tables 5.4 and 6.5

The AN-102/C-104 blended feed supernatant was analyzed in duplicate 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 NaOH solution was prepared for use as a standard and spiking solution. The titrant was 0.2040 M HCl. Duplicate results gave an average OH molarity of 0.28, which equaled 4,630 µg/mL, with a 17% RPD for the duplicate measurements. The RPD value was greater than the QC acceptance criteria; however, the hydroxide level was very low in the samples, and is at only 6% of the MRQ. The standard recovery averaged 98% and a MS recovered at 97%, thus satisfying the QC acceptance criteria. No hydroxide was detected in the SAL hot cell blank.

For information only – The second and third inflection points were detected in the samples at an average of 0.76 molar with a 15% RPD and 0.56 molar with a 7% 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.

6.8 Organic Acids

Tables 5.4 and 6.5

Duplicate samples of the AN-102/C-104 blended feed supernatant were sub-sampled in the SAL and subjected to an IX procedure to reduce the sample dose, such that the resulting samples could be analyzed in the 329 Facility organic IC workstation. Following the IX dose reduction in the SAL which diluted the samples about 5-fold, the samples were further diluted 500-fold at the IC workstation, then analyzed for the organic acids gluconate, glycolate, formate, oxalate, and citrate by IC procedure TP-RPP-WTP-046*, Method for the Analysis and Quantification of Organic Acids in Simulated and Actual Tank Waste by Ion Chromatography*. A PB, BS, MS, and MSD were also prepared for analysis. Neither the TS nor the QA Plan provides QC parameters for the organic acids; therefore, the results were compared to QC acceptance criteria for inorganic anions by IC. For those organic acids above the EQL, all demonstrated an RPD within acceptance criteria of <15% RPD. However, gluconate eluted near the void volume and may not constitute a firm identity of the material. Glycolate and acetate co-elute and firm identification is not possible; the IC system was calibrated using glycolate. No organic acids were detected in the SAL hot cell blank above the MDL. The BS recoveries of the organic acids met acceptance criteria of 80% to 120% except for oxalate (78%); the reason for the slightly low oxalate recovery is not known. The oxalate results are about 20% lower than those reported by the inorganic IC (which are reported for information only). The MS and MSD recoveries met acceptance criteria of 75% to 125% for all organic acids measured.

6.9 Chelator and Degradation Product Analysis

Tables 5.5 and 6.6

The analysis of AN-102/C-104 blended feed for chelators and chelator-degradation products was performed on a best-effort basis. The chelators, ethylenediaminetetraacetic acid (EDTA),), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), 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.

Duplicate 5-mL (nominal) sub-samples of the AN-102/C-104 blended feed 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 resulting samples were then transferred to the 329 Facility organic analysis workstation for derivatization and 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 sub-sample (following the dose reduction step) as a derivatization monitor. A reagent blank was spiked with citric acid, EDTA, NTA and HEDTA. The MS and MSD for the analytical batch were prepared by spiking aliquots with citric acid and EDTA. The citric acid mimics the behavior of succinic acid and EDTA mimics the behavior of HEDTA, ED3A, NTA, and IDA.

Neither the TS nor the QA Plan provides QC parameters for the QC acceptance criteria for the chelator and degradation product analysis. For the QC evaluation of the results, the EQL is set at 10 times the estimated MDL. Only IDA was detected above the EQL, and the 37% RPD for IDA far exceeded the typical 20% standard acceptance criterion. The LCS recovery for citric acid was within the typical 80% to 120% recovery acceptance criterion. The NTA and EDTA LCS recoveries of 71% and 78%, respectively, were slightly below the typical lower threshold of 80% recovery. The HEDTA LCS was low at 38% recovery. The MS and MSD results for citric acid were 47% and 50% and for EDTA were 66% and 58%, showing fairly good precision, but a low bias. Confirmation of the identification of the analytes was performed using GC/MS.

The organic carbon concentration (18,500 μg C/mL) determined by summing the individual organic analytes on a μg C/mL basis, is about the same as the TOC determined by the furnace oxidation method (15,800 μg C/mL) and about twice the TOC as determined by the hot-persulfate method (9,500 μg C/mL). However, this may be misleading since majority of the TOC (15,000 μgC/mL) from the organic analysis is attributed to gluconate, which may or may not actually be present (See Organic Acids Section 6.8).

The citric acid concentration from this derivatization procedure is only 25% to 50% of that determined from organic acids by IC. It is probable that the reported chelator and degradation product concentrations are underestimated as evidenced by the citrate result comparison between the IC method (1,200 μg/mL) and the derivatization method (average of 430 μg/mL).

The derivatization process and analysis are still considered experimental. Additional work is required to provide a reliable, robust technique for the analysis of chelators in tank waste. A direct analysis method, with no derivatization, potentially would be a more robust and reliable technique. Preliminary results with capillary electrophoresis shows promise as a direct analysis technique for chelators and degradation products; however, additional development is required for this techniques as well.

6.10 Organic Phosphates Analysis

Tables 5.5 and 6.6

Following extraction of the AN-102/C-104 blended feed supernatant in the SAL for the organic phosphate analysis; the resulting extracts (both methylene chloride and butanol) were transferred to the 329 Facility for analysis. The methylene chloride extracts were concentrated, derivatized using a diazomethane/ether solution, and analyzed using gas chromatography with a flame ionization detector (GC/FID). 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 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 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.

Neither the TS nor the QA Plan provides QC parameters for the organic phosphate analysis; therefore, the results were compared to QC acceptance criteria for inorganic anions by IC. The surrogate recoveries in the sample and duplicate were 83% and 87%, respectively. D2EHP was not detected in either the sample or the process blank. The LCS recovered at 42% for D2EHP, significantly below the anion acceptance criteria of 80% to 120%. The reason for the very low recoveries of the DPP in the process blank and LCS and the low recovery of D2EHP in the LCS cannot be confirmed. However, additional testing was conducted to evaluate these poor recoveries. This testing suggest that 1) the low recoveries may be due to ionic strength variations, 2) pH of extraction, and/or 2) stability of the derivative.

Besides the routine QC samples (e.g., MS, MSD, LCS, and process blank), three replicates of a standard containing D2EHP and DPP were derivatized and analyzed like the samples. The concentrations and recoveries are included in Table 6.1 for information.

Table 6.1. Recoveries for D2EHP and DPP Standard

Standard @ 58 μg/mL DPP and 65 μg/ml				
Run	**DPP (μg/mL)**	**DPP (%Rec)**	**D2EHP (μg/mL)**	**D2EHP (%Rec)**
1	36	62	47	73
2	57	98	53	82
3	70	121	54	84

Table 6.2. AN-102/C-104 Blended Feed Supernatant – ICP-AES QC Results

Analyte	MRQ µg/mL	MDL µg/mL	01-441 Average µg/mL	(b) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS-A) % Rec.	Post Matrix Spike (PS-B) % Rec.	Serial Dilution % Diff.
Acceptance Criteria							*80% - 120%*	*75% - 125%*	*75% - 125%*	*75% - 125%*	*< ±10%*
Test Specification Analytes											
Al	75	3.0	5,340		2	<15	92	n/r	n/r		-3.9
Ba	2.3	0.5	0.5	U	(a)	<15	90	87	97		
Ca	150	13	153	X	$\boxed{21}$	<15	97	n/r	103		
Cd	7.5	0.8	17		3	<15	95	94	105		
Cr	15	1.0	69		2	<15	96	80	110		
Fe	150	1.3	6.6	J	(a)	<15	99	95	107		
K	75	100	545	JX	(a)	<15	$\boxed{72}$	$\boxed{65}$	91		
La	35	2.5	3.3	J	(a)	<15	91	88		97	
Mg	300	5.0	5.0	U	(a)	<15	99	99	110		
Na	75	38	73,200		2	<3.5	89	n/r	n/r		$\boxed{\text{n/m}}$
Ni	30	1.5	122		2	<15	98	n/r	119		
P	600	5.0	601		2	<15	94	n/r	107		
Pb	300	5.0	49	J	(a)	<15	102	93	110		
U	600	100	100	U	(a)	<15	89	86		101	
Other Analytes											
Ag		1.3	1.3	UX	(a)		23	23	98		
As		13	13	U	(a)				112		
B		2.5	50	B	7				102		
Be		0.5	0.5	U	(a)				100		
Bi		5.0	5.0	U	(a)		91	91	99		
Ce		10	10	U	(a)					104	
Co		2.5	2.5	U	(a)				110		
Cu		1.3	6.8	J	(a)		94	93	99		
Dy		2.5	2.5	U	(a)					99	
Eu		5.0	5.0	U	(a)					107	
Li		1.5	1.6	J	(a)				89		
Mn		2.5	2.5	U	(a)		97	93	106		
Mo		2.5	16	J	6				105		
Nd		5.0	6.5	J	8		90	86		96	
Pd		38	38	U	(a)		89	100		82	
Rh		15	15	U	(a)		88	94		95	
Ru		55	55	U	(a)		93	113		111	
Sb		25	25	U	(a)				104		
Se		13	13	U	(a)				104		
Si		25	280	BX	7		108	59	154		
Sn		75	75	U	(a)					87	
Sr		0.8	0.8	U	(a)		90	89	98		
Te		75	75	U	(a)					104	

Table 6.2. (Cont'd)

Analyte	MRQ µg/mL	MDL µg/mL	01-441 Average µg/mL	(b) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS-A) % Rec.	Post Matrix Spike (PS-B) % Rec.	Serial Dilution % Diff.
Acceptance Criteria							*80% - 120%*	*75% - 125%*	*75% - 125%*	*75% - 125%*	*< ±10%*
Th		50	50	U	(a)					103	
Ti		1.3	1.3	U	(a)		91	87	97		
Tl		25	25	U	(a)				99		
V		2.5	2.5	U	(a)				101		
W		100	100	U	(a)						
Y		2.5	2.5	U	(a)				104		
Zn		2.5	3.3	J	(a)		97	110	109		
Zr		2.5	2.5	U	(a)		93	38	102		

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

n/r = not recovered (spike concentration <20% of sample concentration); n/m = not measured.

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL.

(b) 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 (See Section 5.1).

Table 6.3. AN-102/C-104 Blended Feed Supernatant - ICP-MS QC Results

Analyte	MRQ µg/mL	MDL µg/mL	01-441 Average µg/mL	(g) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS) % Rec.
Acceptance Criteria							*80% - 120%*	*70% - 130%*	*70% - 130%*
U(KPA) (a)	7.8E+02	5E-03	1.5E+01		7		100 (b)	(b)	(b)
Cs	1.5E+00	6E-02	2.6E+00		2		94	102	94
Rb	1.0E+00	8E-02	2.3E+00		0	<15	103	117	109
^{241}as Am	--	1E-03	1.2E-02	J	(c)		92 (d)	(f)	96
	µCi/mL	µCi/mL	µCi/mL						
^{241}as Am	5.1E-02	4E-03	3.8E-02		6	<15	92 (d)	(f)	96
^{241}as Pu	5.1E-02	1E-01	1.2E+00		6	<15	92 (d)	(f)	96
^{99}Tc	1.5E-03	2E-03	4.2E-02		2	<15	83 (d)	(f)	94
^{237}Np	2.7E-02	2E-06	5.2E-05		0	<15	95 (d, e)	(f)	92 (e)
^{239}Pu	3.0E-02	3E-04	1.7E-03	J	(c)		92 (d)	(f)	124
^{240}Pu	1.0E-02	6E-04	6.5E-04	J	(c)		77 (d)	(f)	101

Blank areas indicate QC not required for specified analyte.

(a) Uranium result by KPA.

(b) No acceptance criteria for BS, MS, or PS samples.

(c) RPD only calculated when both the sample and duplicate results are greater than the EQL.

(d) The post-spiked blank sample is used as the LCS.

(e) A BS acceptance criterion is 90% - 110%, MS and PS acceptance criteria are 75% - 125%.

(f) Post spiking of radioisotope performed following digestion.

(g) Data flag: J = estimated value (See Section 5.1).

Table 6.4. AN-102/C-104 Blended Feed Supernatant - Radioisotope QC Results

Analyte	MRQ µCi/mL	MDA µCi/mL	01-441 Average µCi/mL	(a) Err %	(e) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.
Acceptance Criteria								*(c)*	*(c)*
Alpha	2.3E-01	5E-03	4.62E-02	8		1	<15	100	103
^{238}Pu	1.0E-02	2E-04	4.89E-04	20	BJ	(b)	<15		
$^{239/240}$Pu	3.0E-02	9E-05	1.49E-03	9		12	<15	107	105
^{241}Am	3.0E-02	2E-04	3.75E-02	2		5	<15	96	97
^{241}Am by GEA			3.70E-02	15	J	(b)			
$^{243/244}$Cm	1.5E-02	7E-05	1.92E-03	7	B	**36**	<15		
^{242}Cm	1.5E-01	7E-05	1.55E-04	26	J	(b)	<15		
Sum of Alpha			4.15E-02	3		6			
^{90}Sr	1.5E-01	4E-01	1.38E+01	3		5	<15	91	88
^{99}Tc (d)	1.5E-03	4E-06	2.19E-02	4	X	1	<15	92	**248**
^{137}Cs	9.0E+00	7E-03	1.02E+02	2		0	<15		
^{60}Co	1.0E-02	4E-04	2.54E-02	2		1	<15		
^{154}Eu	2.0E-03	2E-03	5.81E-02	2		1	<15		
^{155}Eu	9.0E-02	2E-02	3.77E-02	10		12	<15		

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

(a) The ‘Err %’ represents the uncertainty at 1-σ.

(b) RPD only calculated when both the sample and duplicate results are greater than 10 times the MDA.

(c) QC acceptance criteria defined in TS for total alpha (LCS 70% - 130%, MS 70% - 130%), ^{90}Sr (LCS 75% - 125%), and ^{99}Tc (LCS 80% - 120%, MS 70% - 130%).

(d) Measured as pertechnetate. Lab replicate analyzed for ^{99}Tc RPD; duplicate not sub-sampled from SAL. MS prepared from AN-102 as-received sample processed and analyzed in same batch.

(e) Data flags: J = estimated value; B = analyte in blank above the blank acceptance criteria; X = quality control (QC) deficiency (See Section 5.1).

Table 6.5. AN-102/C-104 Blended Feed Supernatant - Other Analysis QC Results

Analyte	MRQ µg/mL	(f) EQL/ MDL µg/mL	01-441 Average µg/mL	(g) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Matrix Spike Duplicate (MSD) % Rec.
Acceptance Criteria							*80% - 120%*	*75% - 125%*	*75% - 125%*
Test Specification Analytes									
F (b)	150	200	3,700		0	<15	107	96	
Cl	300	200	1,150		3	<15	105	94	
NO_2	3,000	500	25,900		1	<15	105	104	
NO_3	3,000	2,000	61,000		1	<15	101	115	
PO_4	2,500	200	200	U	(a)	<15	104	95	
SO_4	2,300	400	4,430		1	<15	102	93	
OH	75,000	170	4,600		**17**	<15	98	97	
TOC-F (c)	1,500	100	15,800		1	<15	97	85	
TIC-F (c)	150	100	300	J	(a)	<15	99 (d)	95 (d)	
TOC-P (c)	1,500	87	9,500		4	<15	104	98	
TIC-P (c)	150	33	6,000		0	<15	100	99	
Gluconate (e)	1,500	500	40,500		2		106	121	107
Glycolate (e)	1,500	50	3,300		6		100	105	105
Formate (e)	1,500	50	2,800		0		108	123	109
Oxalate (e)	1,500	100	1,800		0		78	89	86
Citrate (e)	1,500	100	1,200		0		89	112	116
Other Analytes Measured									
Br		250	250	U	(a)	<15	106	96	96
C_2O_4		400	2,290		1	<15	107	102	

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL.

(b) The 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), possibly formate or acetate.

(c) For TOC and TIC: P=by hot persulfate method; F=by furnace method/TIC by difference (TC-TOC).

(d) TIC from furnace is measure by difference from TC and TOC determinations. The LCS and MS recovery represents the recovery for the TC analysis.

(e) No QC acceptance criteria defined in TS.

(f) F, Cl, NO_2, NO_3, PO_4, SO_4, Br, and C_2O_4 IC analysis report only results above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

(g) Data flags: U = not detected above reported MDL; J = estimated value (See Section 5.1).

Table 6.6. AN-102/C-104 Blended Feed Supernatant - Other Organic Analyses QC Results

Analyte	**CAS #**	**MRQ µg/mL**	**MDL µg/mL**	**01-441 Average µg/mL**	**(g) Data Flag**	**RPD %**	**Target RPD %**	**Lab Control (LCS/BS) % Rec.**	**Matrix Spike (MS) % Rec.**	**Matrix Spike Duplicate (MSD) % Rec.**
Acceptance Criteria								*n/a*	*n/a*	*n/a*
Organic Phosphate Analytes										
D2EHP	298-07-7	1,500	0.5	0.5	U	(a)		42	84	107
DPP (surrogate)	838-85-7			85% (b)		5		5	73	93
Chelators										
EDTA (c)	60-00-4	1,500	120	620	JX	(a)		78	66	58
HEDTA(f)	150-39-0	1,500	120	120	UX	(a)		38	n/m	
ED3A (d,f)		1,500	120	650	JX	(a)		n/m	n/m	
NTA	139-13-9	1,500	100	170	JX	(a)		71	n/m	
IDA (as NIDA)(f)	142-73-4	1,500	120	1,400	X	37		n/m	n/m	
Citric Acid (e)	77-92-9		130	430	J	(a)		83	47	50
Succinic Acid	110-15-6	1,500	34	34	UX	(a)		69	n/m	
AA (surrogate)				92% (b)				106% (b)	107% (b)	103% (b)

D2EHP = bis-(2-ethylhexyl)phosphate; EDTA= ethylenediaminetetraacetic acid;
DDP = diphenylphosphate; 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)
n/m = not measured

Blank areas indicate QC not required for specified analyte.

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL (10x MDL).

(b) Value represents percent recovery of the surrogate standard.

(c) EDTA BS and MS mimic the behavior of HEDTA, NTA, and IDA.

(d) The CAS number is not available for ED3A.

(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) Concentration results based on EDTA calibration.

(g) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 5.1).

Table 6.7. AN-102/C-104 Blended Feed Wet Centrifuged Solids – ICP-AES QC Results

Analyte	MRQ µg/g	MDL µg/g	01-442 Average µg/g	[c] Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS-A) % Rec.	Post Matrix Spike (PS-B) % Rec.	Serial Dilution % Diff.
Acceptance Criteria							*80% - 120%*	*75% - 125%*	*75% - 125%*	*75% - 125%*	*< ±10%*
ICP-AES Test Specification Analytes											
Ag	900	1.6	1.6	UX	[a]	<15	$\boxed{22}$	$\boxed{35}$	97		
Al	330	11	55,600		9	<15	92	n/r	n/r		-7.8
Ba	600	0.6	79		0	<15	90	87	99		
Bi	6,000	6.3	12	J	[a]	<15	90	118	99		
Ca	180	16	423		9	<15	93	81	102		
Cd	11	0.9	20		2	<15	92	121	101		
Cr	120	1.3	4,570		1	<15	94	n/r	n/r		-3.8
Cu	18	1.6	12	J	[a]	<15	92	$\boxed{129}$	100		
Fe	140	1.6	3,450		11	<15	98	n/r	107		
La	60	3.2	106		1	<15	90	106		95	
Mg	540	6.3	54	J	[a]	<15	96	$\boxed{131}$	108		
Mn	300	3.2	765		0	<15	95	n/r	111		
Na	150	9.5	59,000		1	<3.5	94	n/r	n/r		-8.8
Nd	600	6.3	180		1	<15	90	107		94	
Ni	160	1.9	131	X	$\boxed{40}$	<15	96	$\boxed{163}$	108		
P	600	6.3	1,060		0	<15	90	87	97		
Pb	600	6.3	406	B	3	<15	90	117	104		
Pd	300	47	47	U	[a]		86 [b]	134 [b]			
Rh	300	19	19	U	[a]		88 [b]	118 [b]		86 [b]	
Ru	300	69	69	U	[a]		89 [b]	124 [b]			
Si	3,000	32	3,630		0	<15	104	82	119		
Sr	300	0.9	24		0	<15	91	114	101		
Ti	150	1.6	3.3	J	[a]	<15	89	116	98		
Zr	600	3.2	78	X	$\boxed{51}$	<15	92	$\boxed{54}$	103		
Zn	6	3.2	79		2	<15	95	116	105		
ICP-MS Test Specification Analytes Measured by ICP-AES											
As	3	16	16	J	[a]				106		
B	3	3.2	57	B	10				102		
Be	3	0.6	7.1		1				101		
Ce	6	13	71	J	[a]					97	
Co	3	3.2	3.2	U	[a]				106		
K	1,500	130	565	JX	[a]		75	94	98		
Li	30	1.9	11	J	[a]				98		
Mo	30	3.2	16	J	[a]				102		
Sb	12	32	32	U	[a]				99		
Se	300	16	16	U	[a]				100		
Te	6	95	95	U	[a]					97	
Th	600	63	160	J	[a]					105	

Table 6.7. (Cont'd)

Analyte	MRQ µg/g	MDL µg/g	01-442 Average µg/g	[(c)] Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS-A) % Rec.	Post Matrix Spike (PS-B) % Rec.	Serial Dilution % Diff.
Tl	600	32	32	U	(a)				97		
U	600	130	320	J	(a)	<15	89	116		94	
V	6	3.2	3.2	U	(a)				99		
W	6	130	130	U	(a)						
Y	6	3.2	26	J	(a)				102		
Other Analytes Measured											
Dy		3.2	3.2	U	(a)					100	
Eu		6.3	6.3	U	(a)					107	
Sn		95	95	U	(a)					71	

Blank areas indicate QC not required for specified analyte.
Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.
n/r = not recovered (spike concentration <20% of sample concentration)

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL.

(b) No QC acceptance criteria for BS, MS, or PS in TS.

(c) 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 (See Section 5.1).

Table 6.8. AN-102/C-104 Blended Feed Wet Centrifuged Solids - ICP-MS Metals QC Results

Analyte	MRQ µg/g	MDL µg/g	01-442 Average µg/g	(d) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS) % Rec.
Acceptance Criteria							*80% - 120%*	*70% - 130%*	*70% - 130%*
As	3.0E+00	2E+00	6.9E+00	JB	(a)	<15	94	102	91
B	3.0E+00	2E+00	1.7E+01	JBX	(a)	<15	**52**	**35**	111
Be	3.0E+00	3E-01	1.4E+01		5	<15	117	99	126
Ce	6.0E+00	2E-01	7.0E+01		3	<15	85	100	**135**
Co	3.0E+00	7E-02	1.4E+00	X	**75**	<15	109	126	89
^{127}I	1.5E+00	2E-01	7.2E-01	J	(a)		106 (b, c)	(b)	116 (b)
Li	3.0E+01	3E-01	2.4E+01	X	2	<15	**122**	**241**	**139**
Mo	3.0E+01	3E+00	1.3E+01	J	(a)	<15	101	108	75
Pr	6.0E+00	7E-02	4.7E+01		1	<15	92	97	101
Pt	3.0E+00	3E-02	3E-02	U	(a)		100 (b, c)	(b)	102 (b)
Rb	6.0E+00	1E-01	1.2E+00	J	(a)	<15	89	122	90
Sb	1.2E+01	3E-02	1.4E-01	J	(a)	<15	99	103	125
Se	3.0E+02	1E+01	1E+01	U	(a)	<15	84		**135**
Ta	6.0E+00	2E-02	2.3E-01	BX	(a)	<15	**76**	70	77
Te	6.0E+00	4E-01	4.2E+00		7	<15	101	100	107
Tl	6.0E+02	2E-02	3.9E-02	J	(a)	<15	92	97	102
Th	6.0E+02	1E+00	6.3E+01		4	<15	81	102	88
U	6.0E+02	1E+00	2.8E+02	X	1		71 (b)	119 (b)	96 (b)
V	6.0E+00	2E-01	5.4E+00	B	10	<15	104	126	87
W	6.0E+00	3E-01	4.2E+01	X	1	<15	**69**	**66**	111
Y	6.0E+00	2E-01	2.6E+01		2	<15	95	95	83

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL

(b) No acceptance criteria for BS, MS, or PS samples.

(c) The post-spiked blank sample used as the LCS.

(d) 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 (See Section 5.1).

Table 6.9. AN-102/C-104 Blended Feed Wet Centrifuged Solids - ICP-MS Radioisotope QC Results

Analyte	MRQ µg/g	MDL µg/g	01-442 Average µg/g	(f) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Post Matrix Spike (PS) % Rec.
Acceptance Criteria							*80% - 120%*	*70% - 130%*	*70% - 130%*
^{99}Tc	6.0E+00	2E-01	1.9E+00		11	<15	103 (b)		100
^{129}I	3.0E+01	1E-01	3.3E-01	J	(a)	<15	76 (b, c)	(c)	92 (c)
^{233}U	6.0E+00	8E-04	1.1E-01		5	<15	(d, e)	(d, e)	(d, e)
^{234}U	6.0E+00	5E-03	2.0E-02	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{235}U	6.0E+00	5E-03	2.0E+00		3	<15	(d, e)	(d, e)	(d, e)
^{236}U	6.0E+00	3E-03	9.8E-02		9	<15	(d, e)	(d, e)	(d, e)
^{238}U	6.0E+00	5E-01	2.8E+02		1	<15	(d)	(d)	(d)
^{237}Np	1.8E+00	6E-02	8.8E-01		10	<15	100 (b, e)	(e)	96 (e)
^{239}Pu	6.0E+00	1E-01	2.2E+00		7	<15	99 (b)		126
^{240}Pu	6.0E+00	2E-02	1.7E-01	J	(a)	<15	101 (b)		**69**
	µCi/g	**µCi/g**	**µCi/g**						
^{99}Tc		3E-03	3.2E-02		11	<15	103 (b)		100
^{129}I		2E-05	5.8E-05	J	(a)	<15	76 (b, c)	(c)	92 (c)
^{233}U		8E-06	1.1E-03		5	<15	(d, e)	(d, e)	(d, e)
^{234}U		3E-05	1.3E-04	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{235}U		1E-08	4.4E-06		3	<15	(d, e)	(d, e)	(d, e)
^{236}U		2E-07	6.3E-06		9	<15	(d, e)	(d, e)	(d, e)
^{238}U		2E-07	9.3E-05		1	<15	(d)	(d)	(d)
^{237}Np		4E-05	6.0E-04		10	<15	100 (b, e)	(e)	96 (e)
^{239}Pu		7E-03	1.4E-01		7	<15	99 (b)		126
^{240}Pu		5E-03	3.8E-02	J	(a)	<15	101 (b)		**69**

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

(a) RPD only calculated when both the sample and duplicate results are greater than the EQL.

(b) The post-spiked blank sample used as the LCS.

(c) No acceptance criteria for BS, MS, or PS samples.

(d) Individual isotopic QC samples for uranium not prepared; refer to uranium results in Table 6.8.

(e) A BS acceptance criterion is 90% - 110%, MS and PS acceptance criteria are 75% - 125%.

(f) Data flag: J = estimated value (See Section 5.1).

Table 6.10. AN-102/C-104 Blended Feed Wet Centrifuged Solids - Radioisotope QC Results

Analyte	MRQ µCi/g	MDA µCi/g	01-442 Average µCi/g	Err %	Data Flag [(a)]	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.
Acceptance Criteria								*(b)*	*(b)*
Alpha	1.0E-03	7E-03	1.34E+00	2		2	<15	100	103
^{238}Pu	6.0E-02	7E-04	2.63E-02	6		1	<15		
$^{239/240}$Pu	6.0E+00	7E-04	1.12E-01	3		7	<15	107	105
^{241}Am	1.8E-02	8E-04	1.08E+00	2		0	<15	96	97
^{241}Am by GEA	6.0E+00	2E-02	1.16E+00	4		0			
$^{243/244}$Cm	1.2E-02	7E-04	5.56E-02	4		$\boxed{\mathbf{45}}$	<15		
^{242}Cm	1.2E-02	6E-04	3.00E-03	18	J	(c)	<15		
Sum of Alpha			1.28E+00	3		3			
^{90}Sr	7.0E+01	7E+00	4.17E+02	3		7	<15	91	88
^{134}Cs	9.0E-01	2E-03	2E-03		U	(c)			
^{137}Cs	6.0E-02	6E-03	8.44E+01	2		1	<15		
^{60}Co	1.2E-02	7E-04	2.09E-02	2	B	2	<15		
^{152}Eu	6.0E-02	3E-03	2.17E-02	5	B	2	<15		
^{154}Eu	6.0E-02	3E-03	1.22E+00	2		1	<15		
^{155}Eu	6.0E-02	2E-02	7.21E-01	3		1	<15		
^{125}Sb	6.0E+00	2E-02	2E-02		U	(c)			
^{126}SnSb	6.0E-02	1E-02	1E-02		U	(c)	<15		

Blank areas indicate QC not required for specified analyte.

Bolded and outlined results indicate non-compliances with BNI acceptance criteria; see report for discussion.

(a) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 5.1)..

(b) QC acceptance criteria defined in TS for total alpha (LCS 70% - 130%, MS 70% - 130%) and ^{90}Sr (LCS 75% - 125%) only.

(c) RPD only calculated when both the sample and duplicate results are greater than the EQL.

7.0 References

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Urie, M.W, S.K. Fiskum, J.A. Campbell, O.T. Farmer, L.R. Greenwood, E.W. Hoppe, G.M. Mong, C.Z. Soderquist, R.G. Swoboda, M.P. Thomas, J.J. Wagner. 2002. *Chemical Analysis and Physical Property Testing of 241-AN-102 Tank Waste – Supernatant and Centrifuged Solids*, PNWD-3173, Rev. 0, Battelle Pacific Northwest Division, Richland Washington.

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Appendix A

- Test Specification TSP-W375-00-00007, *Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate*
- Test Plan TP-41500-005, AN-102 Sample Compositing for Process Testing
- Test Instruction TI-41500-015, AN-102 Integrated Test: Solids Analysis
- Figure 2.3 'C-104 Cross-flow Filtration Test Experiment Steps' (Brooks et al. 2000)

River Protection Project
Waste Treatment Plant

RPP-WTP

Tank 241-AN-102 Sample
Composite, Homogeneity, Analysis, and Mixing with HLW Permeate

Test Specification

Revision 0

November 1, 2000

Michael E. Johnson
Pretreatment Technical Manager
CH2MHill Hanford Group

Approval	Date
Michael Johnson Michael E. Johnson / Technical Manager	11/1/00
Stewart Mackay / Engineering Manager	11/1/00

Revision Summary

Revision Number	Section	Purpose
Draft issued for comment 10/16/00	Background	Organized background discussion to group information on 1995 analysis of AN-102 solids and 1999 analysis of supernate fractions. Created subsection to group discussion on work scope planned with current AN-102 sample. Included requirement to conduct PCB analysis of AN-102 sample. PNNL personnel require this information to determine waste handling and disposal protocols at the PNNL laboratory. Added discussion on basis for mixing AN-102 with C-104 wash / leachate.
Draft issued for comment 10/16/00	Section 1.5, Test Conditions	*General*: Clarified instructions in section 1.5, Test Conditions, to denote that contents of AN102 sample bottles will be combined to prepare a composite sample and included clear reference to the composite sample or the composite slurry. *Step 3*: Included the use of a 1/8-inch screen to remove large particles from AN-102 sample before adding sample bottle contents to compositing vessel. The solids collected on the screen are to be crushed and added to the compositing vessel. Replaced requirement to continuously operate agitator in compositing vessel to state that agitator should be operated at least one hour in advance of obtaining sub-samples. *Step 4*: Increased sub-sample size from 25-ml to 100-ml. *Step 6*: Correct error in text that stated that diluted feed sample was to be analyzed. *Step 7*: Changed solids content of AN-102 composite slurry from 5 to 10vol% solids to 2 to 5wt% undissolved solids. Included analysis of AN-102 composite slurry for wt % centrifuged solids, heat capacity, viscosity versus shear rate, thermal conductivity, and particle size analysis. *Formerly Step 9, now step 14*: Decided to use sub-sample of combined AN-102 composite slurry and HLW permeate for Sr/TRU reaction mechanism tests instead of sub-sample of AN-02 composite slurry. *Step 9*: Required duplicate (instead of single) solids measurements.
Draft issued for comment 10/16/00	Tables 1 and 2	Deleted analyses for wt % centrifuged solids, heat capacity, viscosity versus shear rate, thermal conductivity, and particle size analysis for both liquid and solid fractions of AN-102 composite sample. Added x-ray diffraction analysis of the solids to aid in determining types of minerals present.
Draft issued for comment 10/16/00	Section 1.4 Quality Assurance	Revised quality assurance requirements section based on input from WTP QA group.
0	N/A	First. issue of document.

Purpose

This specification provides instructions to Pacific Northwest National Laboratory (PNNL) personnel for:

- Preparing a composite sample from multiple tank 241-AN-102 waste samples,
- Verifying the homogeneity of the composite sample,
- Analyzing the composite,
- Separating AN-102 sub-samples for other process verification tests,
- Mixing the AN-102 sample with C-104 wash / leachate solutions to simulate operations in the WTP,
- Analyzing the mixture of AN-102 sample and C-104 wash / leachate solutions,
- 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

Background

Previous Tank 241-AN-102 Characterization Information

In 1998, Hanford site personnel obtained ~16-liters of waste from tank 241-AN-102. The prefix "241" is common to all Hanford Site tanks and will not be used further. The 16-liters of AN-102 waste were obtained from five equally spaced elevations within the liquid layer in the double-shell tank. This sample was transfer from the 222-S laboratory to the Savannah River Technology Center (SRTC) in 1999 for characterization[1], process verification and waste form qualification testing. SRTC personnel analyzed the AN-102 sample and determine that the sodium concentration was ~10.2M and that waste contained in tank AN-102 meets the criteria of LAW Envelope C feed, as defined in specification 7 of request for proposal (RFP) solicitation number DE-RP27-00RV14136[2]. In 1990, the Westinghouse Hanford Company (WHC) also analyzed an AN-102 sludge core-sample and the liquid separated from the sludge core-sample[3]. The solids recovered from the 1990 core-sample were comprised primarily of Al, Ca, Cr, Fe, Mn, Na, Ni, Si, U, Zr, Cl, NO_2, NO_3, SO_4, and PO_4. PNNL personnel should review the SRTC and WHC characterization reports for the AN-102 supernate and solid samples to familiarize themselves with expected composition of analytes.

Fiscal Year 2001 Work Scope with AN-102 Sample

In support of this test specification, Tank Farms personnel sampled the sludge layer in tank AN-102 to obtain ~1,000-grams of wet solids plus supernate incidental to obtaining the wet solids. 222-S laboratory personnel will ship to PNNL in October / November 2000 the ~1,000-gm sample of AN-102 wet solids and associated supernate in several sample bottles. PNNL personnel will combine the tank AN-102 sample bottles and characterize the composite AN-102 sample to verify compliance with the LAW feed specification. The composite sample will also be analyzed to determine the concentration of polychlorinated biphenyls (PCB), this information is needed to establish waste handling and disposal protocols for the PNNL laboratory.

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[1] Chemical Characterization of an Envelope C Sample from Hanford Tank 241-AN-102, BNF-003-98-0250, June 2000, Westinghouse Savannah River Company.

[2] Waste Treatment and Immobilization Plant Request for Proposal solicitation DE-RP27-00RV14136, final draft issued August 31, 2000, U. S. Department of Energy, Office of River Protection, Richland, Washington.

[3] Tank Characterization Report for Double-Shell Tank 241-AN-102, WHC-SD-WM-ER-545, revision 1, August 1996, Westinghouse Hanford Company, Richland, Washington.

After characterizing the as-received AN-102 composite sample, PNNL personnel will separate some of the AN-102 solids for gas generation and energetics studies. PNNL personnel will mix the remaining AN-102 composite sample with C-104 leachate and wash solution (i.e., HLW permeate) to mimic the mixing of these waste solutions that will occur in the planned waste treatment plant (WTP). PNNL personnel conducted 3M sodium hydroxide leaching and 0.01M sodium hydroxide washing tests with a sample of tank C-104 high-level waste (HLW) sludge[4] in fiscal year 2000. The caustic leachate and wash solutions were separately analyzed and a portion of each archived at PNNL for use in this fiscal year 2001 test.

Mixing of the AN-102 waste with the C-104 wash / leachate solutions was selected to evaluate the effects on waste chemistry and treatment processes from mixing an Envelope D wash / leachate solutions with an Envelope C feed. The proposed feed delivery sequence[5] to the RPP-WTP indicates that AN-102 waste will likely be mixed with AY-102/C-106 leachate and wash solutions. The proposed feed delivery sequence to the RPP-WTP also indicates that AN-107 waste will likely be mixed with AY-101/C-104 leachate and wash solutions. However, this feed sequence can be modified, if necessary. Valuable information on the affects to process chemistry can still be gained from testing the AN-102 mixture with C-104 wash / leachate solution.

The AN-102 composite slurry and HLW permeate solution mixture will be characterized for the same analytes as the as-received composite sample. Follow on testing with the mixture of AN-102 composite sample and HLW permeate solutions will consist of strontium / transuranic precipitation, crossflow ultrafiltration, cesium and technetium ion exchange, and low-activity waste product qualification tests. The River Protection Project Waste Treatment Plant (RPP-WTP) project will issue separate test specifications for these process verification and product qualification tests.

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[4] Characterization, Washing, Leaching, and Filtration of C-104, BNFL-RPT-030, May 2000, Battelle, Richland Washington.

[5] Letter No. 00-OPD-013, "Contract No. DE-AC06-99RL14047 – Modification to Feed Delivery Sequence", dated January 26, 2000 from R. T. French, Manager Office of River Protection to M. P. DeLozier, President CH2M Hill Hanford Group, Inc.

Task Specification Title: AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate

DRD Reference

Task 1.0 of the Development Requirements Document identifies characterization requirements for low-activity waste (LAW) and high-level waste (HLW) samples. Task 9.2.2 of the Development Requirements Document identifies activities for Active Integrated Pretreatment Testing (i.e., mixing AN-102 with C-104 HLW permeate and subsequent process verification testing).

Schedule Reference

The RPP-WTP Research and Technology schedule for fiscal year 2001 identifies LAW and HLW characterization activities as R20250, LAW & HLW Feed Characterization (PNNL). The corresponding activity on the PNNL schedule for fiscal year 2001 is BN.02.01.02, LAW & HLW Feed Characterization, AP-101.

CHG Statement of Work / RPP-WTP Request for Proposal Reference

Characterization of tank waste samples is identified in the CHG Statement of Work section 5.2.2, *Characterization of LAW and HLW Feeds* and the RPP-WTP Request for Proposal standard 2, item (a)(3)(i).

1.1 Justification

This task provides information for assessing tank AN-102 waste for compliance with the LAW and HLW (for solids component) feed specifications. It is critical that the individual samples of AN-102 waste shipped to PNNL are homogeneous before sub-sampling to ensure the analysis of the sub-samples provides information representative of the bulk sample. Additionally, this task prepares a sample of AN-102 solids for gas generation rate and energetics testing and a mixture of AN-102 sample and HLW permeate for integrated testing of the pretreatment processes.

1.2 Objectives

The objectives of this task are to:

- Receive and composite sample bottles that contain AN-102 waste,
- Thoroughly mix the composite AN-102 sample,
- Verify sample homogeneity,
- Sub-sample the AN-102 composite sample,
- Analyze the liquid fraction to determine compliance with specification 7 of RFP solicitation DE-RP27-00RV14136 and analyze the solid fraction to determine compliance with specification 8 of RFP solicitation DE-RP27-00RV14136,
- 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 summary analytical report in electronic and paper format within 90-days of completing all analyses.
- Separate a sub-sample of AN-102 solids for gas generation, energetics studies, and Sr/TRU reaction mechanism studies
- Mix the AN-102 composite sample with C-104 HLW permeate solutions

1.3 Success Criteria

For analytes that are detected above the estimated quantitation level (EQL), the relative percentage difference between duplicate analyses should be less than 20%. For laboratory analyses where matrix spikes, blank spikes, post-spikes, process blanks, and serial dilutions are used, the following criteria should be met.

Quality Control Method	Tolerance Limit
Matrix spikes recoveries	75% to 125%
Blank spikes recoveries	80% to 120%
Post spikes recoveries	75% to 125%
Process blanks analyses	analytes of interest below the EQL or less than 5% of the sample concentration
Serial Dilutions	± 10% of the sample analysis, where the analyte concentration exceeds the EQL

Laboratory control standards (LCS) shall be traceable to the National Institute of Standards and Technology (NIST) and all analytes above the EQL should exhibit recoveries within acceptance criteria.

The acceptance criteria for calibration verification check standards are ± 10% of true value.

1.4 Quality Assurance

The RPP-WTP *Quality Assurance Program*, BNFL-5193-QAP-01 revision 6, 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 6).

PNNL implements the RPP-WTP quality requirements in a quality assurance plan (QAP) as approved by the RPP-WTP QA organization. PNNL 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 PNNL QAP for important to safety items and activities.

Additionally, CHG personnel may use the PCB analysis results for regulatory submittals (i.e., environmental data). For the PCB analysis, PNNL is to required to use the following US EPA documents as guidance: "EPA Guidance for Quality Assurance Project Plan" (EPA QA/G-5) and "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846). PNNL is to provide an explanation in the test report if deviation from the recommended EPA methods are necessary. Analyses performed to generate environmental data are also subject to the requirements of the RPP-*WTP Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data* (PL-W375-QA00002, current revision). Justifications or rationale will be provided to the RPP-WTP QA manager and technical manager if alternative methods are proposed. In cases where constituents of concern are not to be sampled / prepared / analyzed by the standard SW-846 (EPA 1997) methods, alternative methods will be provided to RPP-WTP QA manager and technical manager for approval <u>prior to the commencement of sampling and analysis</u>. This includes the use of modified SW-846 methods.

The *Quality Assurance Requirements and Description* (DOE/RW-00333P), the principal quality assurance document for the Civilian Radioactive Waste Management Program, <u>does not</u> apply to activities conducted as part of this task.

1.5 Test Conditions

1. PNNL personnel are to report on the visual appearance of the liquid and solid / crystalline phases present in each AN-102 sample bottle.

2. PNNL personnel are to maintain a material balance for the AN-102 sample throughout the process steps defined by this test specification. Items typically to be recorded include: sample bottle identification number, the mass

(or volume) of sample received in each bottle (or the gross weight of each bottle if the tare weights are not provided by the 222-S laboratory), loss of sample due to residual sample left in each bottle, mass (or volume) of composite AN-102 sample, mass and volume of chemical additions, mass of sample removed for analysis, and any other significant activities that add or remove mass from the AN-102 sample.

3. PNNL personnel are to composite the contents of all AN-102 sample bottles into a clean, single vessel that contains a mechanical agitator and bottom sample port.

   a. The vessel used to composite the contents of the AN-102 sample bottles should have a capacity of at least 3.0-liters, since this same vessel will be used for mixing the composite AN-102 sample with C-104 HLW permeate solutions.

   b. Contents of each AN-102 sample bottle shall be passed through a ~1/8-inch screen to remove large particles prior to transferring to the compositing vessel. This is to avoid plugging the sampling valve on the vessel. Any solids collected on the screen are to be crushed so that they will pass through the 1/8-inch screen and added to the compositing vessel.

   c. The vessel agitator should be operated at least one-hour prior to removing any sub-samples from the AN102 composite sample.

4. While operating the vessel agitator, PNNL personnel are to extract three 100± 1-ml sub-samples from the AN102 composite sample to evaluate sample homogeneity.

   a. PNNL personnel are to record the sample temperature (± 1°C) and measure the density of these three sub-samples to ±0.01-gm/ml.

   b. Then, these three sub-samples are to sit undisturbed for a minimum of 24-hours.

   c. PNNL personnel are to record the volume of settle solids / crystalline material and supernate present in each of the three AN-102 sub-samples. The volume measurement shall be reported to an accuracy of ±1.0-ml.

   d. PNNL personnel are to calculate the relative volume percentage of settle solids / crystalline material present in each of the three AN-102 sub-samples.

   e. If the absolute standard deviation is less than 5 percentage units for the calculated volume percent of settle solids / crystalline material present in each of the three AN-102 sub-samples, the AN-102 composite sample shall be considered homogenous. The sub-samples are to be returned to the composite vessel.

   f. If the three AN-102 sub-samples fail criteria (e), the sub-samples are to be returned to the composite vessel, agitation increased / continued, three new sub-samples are to be obtained, and step 4 repeated.

5. PNNL personnel are to analyze in duplicate, homogenous sub-samples of the as-received AN-102 composite sample to determine the concentration of liquid and solid fraction analytes listed in Table 1 and 2, as well as PCBs in the liquid and solid phases. Quality Control parameters are defined in Tables 3 and 4.

   a. The AN-102 sub-sample(s) is to be centrifuged to separate the damp solids / crystalline material.

   b. Do not wash the damp solids / crystalline material, since this will cause solids dissolution.

   c. If the solids do not completely dissolve as part of the sub-sample preparation for analysis, do not proceed with the solids analysis. Contact the RPP-WTP technical representative to discuss using an alternative method for solids preparation.

   d. Solids are defined as the product of centrifuging the AN-102 sub-sample, separating and drying the solids at 100 ± 5°C for 24 ± 1-hour, and removing the dissolved solids contribution.

6. PNNL personnel are to compare the liquid and solid fraction analytical results for the AN-102 composite sample to determine compliance with Specification 7, Low-Activity Waste Envelopes Definition, and Specification 8, High-Level Waste Envelope Definition, of *WTP Request for Proposal Solicitation No. DE-RP27-00RV14136.*

7. PNNL personnel are to remove sufficient solids from the AN-102 composite sample to create a slurry that contains 2 to 5 weight percent undissolved solids. PNNL personnel are to determine the wt % centrifuged solids, particle size distribution (less than 0.1 micron and 0.12 to 700 micron ranges), viscosity versus shear rate (0.7cP minimum), heat capacity (expected range 0.5 to 2.0 Cp), thermal conductivity (if method available; expected range 0.2 to 0.8 kW/m-°C) for the AN-102 composite slurry.

   Note: Removal of cesium from the liquid fraction to conduct viscosity measurement of the slurry or suspension of the solid fraction in a simulant to conduct the particle size analysis of the slurry are acceptable methods to reduce personnel radiation dose rate.

8. PNNL personnel are to retain the solids that were separated from the AN-102 composite sample for gas generation and energetics studies. The solids should be maintained in a sealed bottle with sufficient supernate to cover the solids and keep the solids damp. A separate test specification will be issued for the gas generation and energetics studies.

9. PNNL personnel are to obtain a 100-ml homogeneous sub-sample of the AN-102 composite sample. PNNL personnel are to retain this sample for Sr/TRU reaction mechanism tests that will be discussed in a separate test specification.

10. PNNL personnel are to obtain two, 25 ± 1-ml homogeneous sub-sample of the AN-102 composite slurry. Personnel are to measure and record the sample volume (or mass) and amount of solids present to ± 0.1-ml (or ± 0.1-gm) in each sub-sample.

11. PNNL personnel are to mix the following amounts of C-104 initial permeate, wash, caustic leachate, and caustic wash solutions with one of the 25 ± 1-ml sub-samples of AN-102 composite slurry.

    a. C-104 initial permeate (bottle labeled as "C-104 Filtrate #1): 18.2 ± 0.1-ml.
    b. C-104 initial permeate (bottle labeled as "C-104 Filtrate #2): 18.2 ± 0.1-ml.
    c. C-104 wash (bottle labeled as "C-104 Wash (H20) Filtrate Composite): 12.3 ± 0.1-ml.
    d. C-104 caustic-leach permeate (bottle labeled as "C-104 caustic leach permeate"): 11.7 ± 0.1-ml.
    e. C-104 caustic wash solution (bottle labeled as "C-104 caustic wash composite"): 6.3 ± 0.1-ml.

12. After a minimum of 24-hours, PNNL personnel are to examine the mixture of AN-102 composite slurry and C104 wash / leachate solution and record the time since mixing, visual appearance and amount of solids present.

    a. PNNL personnel shall observe and record observations during the mix to identify signs of chemical reaction.

    b. **PNNL personnel are not to proceed with the remainder of this test specification if this mix of AN-102 composite slurry and C-104 wash / leachate solution has formed a gel (or viscous solution), solidified, or the amount of solids has increased by more than 25% of the original AN-102 solids volume.**

13. PNNL personnel are to the following amounts of C-104 initial permeate, wash,'caustic leachate, and caustic wash solutions with the AN-102 composite slurry. Volumes of each C-104 solutions are per 100-ml of AN-102 composite slurry.

    a. C-104 initial permeate (bottle labeled as "C-104 Filtrate #1): 72.8 ± 0.1-ml.
    b. C-104 initial permeate (bottle labeled as "C-104 Filtrate #2): 72.8 ± 0.1-ml.
    c. C-104 wash (bottle labeled as "C-104 Wash (H20) Filtrate Composite): 49.1 ± 0.1-ml.
    d. C-104 caustic-leach permeate (bottle labeled as "C-104 caustic leach permeate"): 46.9.2 ± 0.1-ml.
    e. C-104 caustic wash solution (bottle labeled as "C-104 caustic wash composite"): 25.3 ± 0.1-ml.

14. PNNL personnel are to analyze in duplicate, homogenous sub-samples of the mixture of AN-102 composite slurry and C-104 wash / leachate solution to determine the concentration of liquid and solid fraction analytes listed in Table 1 and 2. Quality Control parameters are defined in Tables 3 and 4.

15. PNNL is to archive residual C-104 wash/leachate solutions for potential use in later studies.

1.6 Reporting

PNNL personnel are to report all process verification and 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 CHG.

PNNL personnel are to issue the draft test results to RPP-WTP within 30 calendar days after completing the analyses identified in this test specification. PNNL personnel are to issue a final test report within 30 calendar days after receiving comments on the draft report from RPP-WTP. PNNL 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, and ensure this information is added to the Tank Waste Information System (TWINS).

Table 1
Liquid Fraction Analyses

Analyte	Minimum Reportable Quantity	Analysis Method
	μg/ml	
Al	7.5E+01	ICP-AES (AA may be used for Na, K, Cs, Rb or ICP-MS for Cs and Rb)
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Cs	1.5E+00	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Rb	1.0E+00 (if method available)	
S	3.0E+01	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TIC	1.5E+02	Furnace Oxidation
TOC	1.5E+03	
TOC	1.5E+03	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02	Silver catalyze persulfate and furnace oxidation method
Hg[1]	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03	
SO_4	2.3E+03	
	μCi/ml (except as noted)	
^{237}Np	2.7E-02	ICP-MS
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	5.1E-02	
^{99}Tc	1.5E-03	
^{99}Tc	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{90}Sr	1.5E-01	Separations / Liquid Scintillation
^{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	1.0E-02	
^{137}Cs	9.0E+00	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
total and free OH	7.5E+04 μg/ml	Titration
Ammonia[1]	1.4E+02 μg/ml	ISE

Table 1
Liquid Fraction Analyses

Analyte	Minimum Reportable Quantity	Analysis Method
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Organic Analytes	μg/ml	
EDTA	1.5E+03	HPLC / LC-MS (if methods are available)
HEDTA	1.5E+03	
Oxalate	1.5E+03	
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
D2EHPA	1.5E+03	
NTA	1.5E+03	
IDA	1.5E+03	

[1] These analyses are only required for the as-received AN-102 composite sample.

Table 2. HLW Solids Analyses

Analyte	Minimum Reportable Quantity	Analysis Method
	ug/gm	
Ag	9.0E+02	ICP-AES
Al	3.3E+02	
Ba	6.0E+02	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
La	6.0E+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	
Pd	3.0E+02	
Rh	3.0E+02	
Ru	3.0E+02	
S	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E-02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Hg[1]	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
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[1]	3.0E+00	CN analysis
NH_3[1]	6.0E+01	ISE
	µg/gm	ICP-MS
As	3.0E+00	
B	3.0E+00	
Be	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Li	3.0E+01	
Mo	3.0E+01	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
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	
W	6.0E+00	

Table 2. HLW Solids Analyses[1]		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Y	6.0E+00	
^{127}I[1]	1.5E+00	
^{129}I[1]	3.0E+01	
^{233}U	6.0E+00	
^{234}U	6.0E+00	
^{235}U	6.0E+00	
^{236}U	6.0E+00	
^{238}U	6.0E+00	
^{237}Np	1.8E+00	
^{99}Tc	6.0E+00	
	µCi/gm	
^{3}H[1]	1.5E-02	Separations / Liquid Scintillation
^{14}C[1]	1.8E-03	Separations / Liquid Scintillation
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}$Pu	6.0E+00	
^{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[1]	6.0E+00	
^{126}Sn[1]	6.0E-02	
^{134}Cs[1]	9.0E-01	
^{135}Cs[1]	5.3E-03	
^{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	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
^{241}Pu[1]	1.2E+00 µCi/gm	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Mineral types / structures		X-ray diffraction

[1] These analyses are only required for the as-received AN-102 composite sample.

Table 3. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[(a)]	**Spike %Recovery[(b)]**	**Duplicate RSD[(c)]**
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[(d)]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, 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%
Cs[(i)], Eu[(i)]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH-	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A[(e)]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[(f)]	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	ICP/MS	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{125}Sb	GEA	to be obtained		
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A[(g)]	<15%
^{137}Cs	GEA	NP	N/A[(g)]	<15%
^{152}Eu[(i)]	GEA	NP	N/A[(g)]	<15%
^{154}Eu[(i)]	GEA	NP	N/A[(g)]	<15%
^{155}Eu[(i)]	GEA	NP	N/A[(g)]	<15%
^{231}Pa	ICP/MS	Developed by Laboratory		
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np[(e)]	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu[(h)]	Separation/AEA	NP	N/A[(e)]	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[(e)]	<15%
^{242}Cm	Separation/AEA	NP	N/A[(e)]	<15%
^{243}Am/Cm	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A[(e)]	<15%
Total Alpha[(h)]	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids	Gravimetric	80 - 120%	N/A	<21%

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 of the mean/mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{93}Zr.

(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.

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

Table 4. Quality Control Parameters for Solids Analysis

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery(a)	Spike % Recovery(b)	Duplicate RSD(c)
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, La, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90(d)	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs(e)	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A(g)	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{59}Ni(h)	Separation/GEA	NP	N/A(g)	<15%
^{63}Ni(h)	Isotopic specific separation/beta-liq scintillation	NP	N/A(g)	<15%
^{60}Co(i)	GEA	NP	N/A(k)	<15%
^{90}Sr(j)	Isotopic specific separation/beta count	75 - 125%	N/A(g)	<15%
^{90}Y(j)	Isotopic specific separation/beta count	75 - 125%	N/A(g)	<15%
^{93}Zr(h),(l)	beta-liq scintillation	NP	N/A(g)	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m}Sn(l)	Separation/GEA	NP	N/A(g)	<15%
^{125}Sb(m)	GEA	to be obtained		
^{126}Sb(l),(n)	Separation/GEA	NP	N/A(g)	<15%
^{126m}Sb(k),(n)	Separation/GEA	NP	N/A(g)	<15%
^{126}Sn(n)	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A	<15%
^{133}Cs(h)	ICP/MS	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{151}Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A(g)	<15%
^{152}Eu(i)	GEA	NP	N/A(k)	<15%
^{154}Eu(i)	GEA	NP	N/A(k)	<15%
^{155}Eu(i)	GEA	NP	N/A(k)	<15%
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}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}Am/Cm	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A(g)	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids	Gravimetric	80 - 120%	N/A	<21%

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 of the mean/mean) x 100

(d) ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{93}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.

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.

.6.1.1.1 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		

REFERENCE COPY

PNNL Test Plan

Document No.: TP-41500-005
Rev. No.: 0

Title: AN-102 Sample Compositing for Process Testing

Work Location:
HLRF

Page 1 of 19

Author: Paul Bredt

Effective Date: Upon final signature
Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:
_ Radiological
_ Hazardous Materials
_ Physical Hazards
_ Hazardous Environment
_ Other:

Required Reviewers:
X Technical Reviewer
_ Building Manager
_ Radiological Control
_ ES&H
X Quality Engineer
X Project Manager
_ RPL Manager
X Client

Are One-Time Modifications Allowed to this Procedure?
X Yes _ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ___ **Yes or** X **No**
FOR REVISIONS:
Is retraining to this procedure required? ___ Yes X No
Does the OJT package associated with this procedure require revision to reflect procedure changes? __ Yes __ No X N/A

Approval Signature Date

Author Paul Bredt 12/7/00

Technical Reviewer 12/7/00

SFO Manager 12/7/00

Project Manager 12/7/00

Quality Engineer Almeida 12/7/00

Client 12/7/00

Applicability

This Test Plan describes work to be performed under Project 41500, Technical Support to CHG for Phase B2. Samples of actual waste from Hanford tank AN-102 will be received at PNNL's Radiochemical Processing Laboratory for testing under this project. In addition, caustic leach solutions and wash solutions from tank C-104, generated during testing last fiscal year, are currently archived in the HLRF. This Test Plan describes how those materials will be composited, homogenized, and sub-sampled for process testing and characterization tasks. This Test Plan does not cover work under Task 2.9, the "Regulatory Data Quality Objective".

This Test Plan provides details regarding the implementation of Test Plan 41500-004, "Sample Compositing". Client expectations for successful achievement of project data needs have already been established via Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification". This Test Plan is a mechanism for the cognizant scientist to communicate to technical staff and the client specifics on Test Plan 41500-004 implementation. Therefore, this Test Plan 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 a cognizant scientist.

Work with actual tank material will be performed in radiological hot cells.

Justification/Test Objectives/Success Criteria

The objective of this Test Plan is to provide homogeneous feed to tasks within the project. Samples will be transferred to the Radiochemical Processing Laboratory in sample jars that could range in size between approximately 60 to 500 ml. Under this Test Plan, these materials will be combined in a ratio meeting client specifications. The materials will be homogenized followed by addition of diluents and secondary waste products or appropriate additives as specified by the client in the current revision to the Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification".

Given the expected variability of samples (viscosity, yield stress, solids content, etc.), the cognizant scientist is responsible for selecting the appropriate impeller speed for the homogenization. A successful homogenization will result in uniform sub-samples of the composite. This Test Plan contains administrative hold points. Under these hold points, data on the success or failure of the homogenization process will be assessed before proceeding.

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 cognizant scientist and will be documented by crossing out the original information with a

M&TE List:

Balance 1:	Calibration ID 388-06-01-020 Calibration Expiration 8/01	Location "C" cel
Balance 2: (if needed)	Calibration ID 362-06-01-051 Calibration Expiration 8/2001	Location "B" Cell
Thermocouple:	Calibration ID 2971 Type K Calibration Expiration 5/01	Location C-Cell
Digital Thermometer:	Calibration ID PNL0002731 Calibration Expiration 7-2001	Location 601

Record daily balance check information below:

Loc	Date	Actual (g)	Measured (g)	Tolerance (g)	Acceptable (Y/N)
"C"	12/21/00	200.000	200.008	±.01	y
"C"	12/27/00	100	99.999	± .01	y
C	1/9/01	200.000	200.002	±.01	y
C	1/10/01	200.000	200.002	±.002	y
B	1/10/01	1000.00	999.97	±.01	y
C	1/16/01	200.000	200.006	±.01	y

single line, and recording, initialing, and dating the changes. The results of all measurements will be recorded in this Test Plan, a Laboratory Record Book (LRB), or bench sheet. Copies of the completed Test Plan, 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.

General Requirements

1) Personnel are to document the visual appearance of the liquid and solid/crystalline phases present in each sample bottles prior to homogenization. Visual inspection should include any indication of a separate organic layer.

2) Personnel are to maintain a material balance for the samples/composites throughout the steps defined by this Test Plan. Items typically to be recorded include: sample bottle identification number, 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 samples, mass of sample removed for analysis, and any other significant activities that add or remove mass from the samples/composites.

3) Video images are to be collected wherever practical to document observations.

4) Balance Checks should be performed each workday and documented in the M&TE section of this test plan.

Work Instructions

1) It is anticipated that 8 sample jars (containing approximately 500 ml each) will be shipped from 222-S to the RPL for process testing. Fill in the sample IDs in Table 1 based on the samples that are to be used for this compositing task.

2) Weigh the full sample jars to ±0.01 g and record the data in Table 1

3) Install the 1/8" (1/8" to 3/32" is acceptable) screen on the 7 L stainless steel mixing vessel.

> 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.

4) Transfer all material from the jars 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 in the jars or on the screen into the kettle. Record on a separate piece of paper from which sample jars any ≥1/8 inch particles are observed to be removed. Attach these observations to this test plan.

5) Remove the screen and place an airtight cover on the mixing vessel.

6) Reweigh the empty jars and record the empty masses to ± 0.01 g in Table 1.

7) Transfer any large particles retained on the screen to an appropriately sized glass jar of known mass labeled **"AN-102 >1/8"**. Weigh the jar to ±0.01g.

PRB 10/12/01

AN-102 >1/8

Total 306.61 g
Tare 298.79 g
Dry ~~Wet~~ Solids 7.82 g ←

Large particles retained in JAR 30 as received from BNFL
rgs

8) Allow the particles in **"AN-102 >1/8"** to air dry for at least 8 hours, then reweigh "AN-102 >1/8" (The goal of this step is to dry the material enough to allow for crushing, not to remove all water from the solids). Present this data to the cognizant scientist and have the cognizant scientist view the solids before proceeding. Additional drying may be required.

AN-102 >1/8

Lynette J. & Ralph did not record wet wt
rgs

Total ________g
Tare ________g
Dry Solids ________g

9) Transfer the particles in "**AN-102 >1/8"**" to a clean stainless steel pan. Crush the particle as fine as practical using a ~~stainless steel rod or similar stainless steel object.~~ • glass stopper

10) Remove the lid from the mixing vessel and install the 1/8" screen.

11) Transfer the crushed particle through the screen and into the mixing vessel.

12) If any particles are retained on the screen, then return them to the stainless steel pan and repeat steps 9 and 11 until all particles pass into the composite, then remove the screen. Forceps cleaned with DI H_2O & used to remove Chunks to > 1/8" bottle

Table 1. AN-102 Sample Information

order	Sample ID	Full Mass (g)	Empty Mass (g)	Mass Transferred (g)
①	2AN-00-21	967.05	295.88	671.17
3	2AN-00-24	964.41	295.10	669.31
5	2AN-00-25	* ~~954.31~~ 817.29	295.95	521.34*
8	2AN-00-26	983.90	297.28	686.62
②	2AN-00-27	965.42	296.58	~~668.84~~
4	2AN-00-28	973.15	300.16	672.99
6	2AN-00-29	973.97	303.42	670.05
7	2AN-00-30	989.07	298.79	690.28
				12/21/00
				Jar 30+ Dried solids = 306.61
✗	2AN-00-30	Reused for	>1/8" partcls	Empty TARE Wt: 298.79
				Net. 7.82g

* Bottle #25 had 137.02g removed for 102-Sr-TRU, hence Full wt was 817.29

Note:
Observations of the solids will be difficult due to the color dark color of the supernatant and vessel construction, but use of the in-cell video camera should give a good indication of when the mixer is generating a strong vortex.

13) 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 9.35pm Date 12/21/00 Temperature 31.5 °C Speed 4 on mixer

12/20/00
- very little air in 21 for mixing, so sucked ~20ml off to make room for mixing
- 21 still would not mix with shaking, so used glass rod.
- Some solids stuck in pile on seive. used rod to mush it around. looks like lots of chunks.
- used glass stopper to ~~try~~ try to push through sieve and went right. through.
- 27 had big chunk pile on sieve, pushed right through with glass stopper except ~2-3 chunk
- 24 had several chunks, but could not get clear shot on video ~~[illegible]~~ due to inability to rinse off the

> Note:
> Samples in Step 22 may be collected at the same time as samples in step 14. If these steps are performed at the same time, then all samples collected must be included in the hold point in step 20.

14) While operating the vessel agitator, extract one sub-sample of sufficient size 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 and record the time. Then collect three sub-samples of approximately 100 ml sub-samples from the mixing vessel into volume graduated glass jars of known mass labeled **"AN-102 AR-A"**, **"AN-102 AR-B"** and **"AN-102 AR-C"**. Weigh the jars to ±0.01g and measure the volumes to ±1ml. Record the mass and volume data below. Record the time the final sample was collected. 12-21-00

Time of Valve Clear 10:43 pm Time of final collection 10 : 55

10:55

	AN-102 AR-A		AN-102 AR-B		AN-102 AR-C	
Target	100 ml		100 ml		100 ml	
Total	297.533	g	300.407	g	295.781	g
Tare	134.05	g	134.25	g	134.90	g
Slurry	163.483	g	166.157	g	160.88	g
Slurry	114	ml	116	ml	113	ml

15) Using the mass and volume data collected above, calculate the density of the slurries in **"AN-102 AR-A"**, **"AN-102 AR-B"** and **"AN-102 AR-C"**.

AN-102 AR-A	AN-102 AR-B	AN-102 AR-C
ρ 1.434 g/ml	ρ 1.432 g/ml	ρ 1.424 g/ml

16) Turn off the stirring motor and place an airtight cover on the mixing vessel.

17) Record the date and time and allow **"AN-102 AR-A"**, **"AN-102 AR-B"** and **"AN-102 AR-C"** to settle for a minimum of 24 hours.

Day 12/27/00 Time 11 Am

Bottles ~~the~~ tare wts outside cell – Checked with 50.0g wt read 49.99g
Balance 362-06-01-051
Cal Date – 8/29/00
Due 4-2001

Cal Due Date 8/01
Cert # 1138

18) Record the date and time, and total volume of the slurries and volume of the settled solids to ±1ml in **"AN-102 AR-A", "AN-102 AR-B"** and **"AN-102 AR-C".**

Day 12/27/00 Time 11 Am

AN-102 AR-A	AN-102 AR-B	AN-102 AR-C
Total 114 ml	Total 116 ml	Total 113 ml
Solids 83 ml	Solids 84 ml	Solids 83 ml

19) Using the data in the previous step, calculate the volume percent settled solids in **"AN-102 AR-A", "AN-102 AR-B"** and **"AN-102 AR-C".** Document the calculation in Table 2.

Table 2. Volume Percent Settled Solids In Composite Samples

AN-102 AR-A	AN-102 AR-B	AN-102 AR-C
$83/114 \times 100 = 72.8\%$	$84/116 \times 100 = 72.4\%$	$83/113 \times 100 =$ 73.5%

20) **Administrative Hold Point**: If the standard deviation is less than 5 percentage units for the calculated volume percent of settled solids present in each of the three AN-102 sub-samples, the AN-102 composite samples shall be considered homogenous. These samples may be retained for further homogenization tests (ICP, GEA, TDS, etc.), otherwise return these sub-samples to the mixing vessel. If the three AN-102 sub-samples fail this criteria, then the sub-samples are to be returned to the composite vessel, agitation increased/continued and/or other modification deemed necessary to obtain improved mixing, three new sub-samples are to be obtained, and steps 13-20 repeated.

21) Stir the sample by slowly increasing the motor speed until it is similar to that used successfully in step 13. Stir for a minimum of one hour. Record the sample temperature to ±1.1°C.

Time ________ Date ________ Temperature ________°C

See step 13 PRB 1/08/01

Note:
Samples in Step 22 may be collected at the same time as samples in step 14. If these steps are performed at the same time, then all samples collected must be included in the hold point in step 20.

22) While operating the vessel agitator, extract one sub-sample of sufficient size 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 and record the time. Then collect sub-samples for characterization of the as-received material. The number and volume of these samples will be determined by the cognizant scientist. Use the necessary spaces below to record the sample masses. Record the time the final sample was collected. 12/21/00

Time of Valve Clear 10:43 pm Time of final collection 11:04

12/28/00 Balance checked

AN-102 AR-D
Target 110 ml
Total 293.846 g
Tare 137.01 g
Slurry 159.84 g

AN-102 AR-E
Target 110 ml
Total 302.053 g
Tare 134.26 g
Slurry 167.79 g

AN-102 AR-F
Target 110 ml
Total 282.806 g
Tare 133.92 g
Slurry 148.89 g

11:04

PRB 12/18/00

AN-102 AR-G
Target 110 ml
Total 296.376 g
Tare 134.60 g
Slurry 161.78 g

AN-102 AR-H
Target ~~400~~ 100 ml PRB 12/18/00
Total 281.388 g
Tare 134.21 g
Slurry 147.18 g
Final vol = 102 mL
> ρ = 1.44

AN-102 AR-I
Target ~~400~~ 110 ml
Total 291.413 g
Tare 130.87 g
Slurry 160.54 g

23) Record the weight percent undissolved solids in the AN-102 composite sub-sampled under step 14 or 22.

a. If this analysis has already been conduced, document when and how this analysis was conducted.

b. If this analysis has not been conducted, use Technical Procedure 29953-010 to measure the following value in duplicate.

(a) Wt% dissolved solids in the supernatant
(b) Wt% centrifuged solids in the composite
(c) Wt% water in the centrifuged solids

Assuming all of the water in the centrifuged solids is from interstitial supernatant, calculate the wt% undissolved solids in the centrifuged solids.

Take X_r X_o Dean →Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Undissolved Solids 38.2 wt% Source of this data See pg 9.5 Attached

AN-102 D
F
G
} to SAL on 1/25/01

ENGINEERING WORKSHEET

Page 9.5 of 19

…ared By: Swoboda | Date: 12/29/00 | Project: TP-41500-005 (data from TP-41500-015)

Title/Subject: CALC of Wt % Solids

From Calc MACRO via PAUL Bredt

Wt % Solids undissolved

$$= \left[\frac{\text{Sam Solids wet wT}}{\text{Total sample wet wt}} \times \left[1 - \left(\frac{1 - \left(\frac{\text{Sample dry solids}}{\text{Sample WET Solids}} \right)}{1 - \left(\frac{\text{Sam Liq dry wt}}{\text{Sam Liq wetwt}} \right)} \right) \right] \right] \times 100$$

Hence,

$$AR\text{-}C = \left[\frac{6.526\,g}{13.842\,g} \times \left[1 - \frac{\left(1 - \frac{5.95}{6.526}\right)}{\left(1 - \frac{3.819}{7.096}\right)} \right] \right] \times 100$$

$$= \boxed{38.14\%}$$

$$AR\text{-}D = \left[\frac{6.639}{14.016} \times \left[1 - \frac{\left(1 - \frac{6.047}{6.639}\right)}{\left(1 - \frac{3.798}{7.089}\right)} \right] \right] \times 100$$

$$= \boxed{38.27\%}$$

$$\text{Ave} = \boxed{38.2\%}$$

rgS 12/29/00

Checked by DE Kurath 12/29/00

attachment ~~to~~ test instruction 41500-005 2/[illegible]

24) Determine the volume of settled solids and supernatant needed to generate a slurry of ~~794~~ 894 ml containing 2 wt% undissolved solids. Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Target Volume of Settled Solids =__________ml

Target Volume of Supernatant =__________ml

25) Calculate the volume (and mass) of homogenous composite material that would contain the volume of settled solids calculated in the previous step. Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Target Volume of Homogenous Composite Material to Remove =__________ml

__________g

26) Calculate the volume (and mass) of additional supernatant needed to reach the total volume target of 794 ml. (This is 794 ml – volume of composite in the previous step). Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Calculated Volume of Additional Supernatant =__________ml

__________g

27) Stir the sample by slowly increasing the motor speed until it is similar to that used successfully in step 13. Stir for a minimum of one hour. Record the sample temperature to ±1.1°C.

Time __________ Date __________ Temperature __________°C

see attachment
IRO 01/08/01

28) While operating the vessel agitator, extract one sub-sample of sufficient size 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 and record the time. Then collect the mass of composite calculated in step 25 in volume graduated glass jars of known mass. Use the necessary spaces below to record the sample masses.

Time of Valve Clear__________ ~ 11:05 pm on 12/21/00

AN-102 AR-J

Total 896.71 g
Tare 311.15 g
Slurry 585.56 g

29) While continuing to operate the vessel agitator, extract the remaining material from the mixing vessel into volume graduated glass jars of known mass. Use the necessary spaces below to record the sample masses. Record the time the final sample was collected.

Time of final collection 12/21/00 11:18pm

All 500 ml Jars

AN-102 AR-K
Total 901.12 g
Tare 311.42 g
Slurry 589.70 g

AN-102 AR-L
Total 908.61 g
Tare 311.50 g
Slurry 597.11 g

AN-102 AR-M
Total 915.76 g
Tare 311.13 g
Slurry 604.63 g

AN-102 AR-N
Total 915.70 g
Tare 311.08 g
Slurry 604.62 g

AN-102 AR-O — 11:18
Total 907.77 g
Tare 311.30 g
Slurry 596.47 g

AN-102 AR-P
~~Total ______ g~~
~~Tare ______ g~~
~~Slurry ______ g~~
PRB 10/13/01

End AR Series

30) Record the date and time and allow the sample collected in the previous step to settle for a minimum of 3 days.

Day 12/21/00 Time 11:18 pm

31) Record the date and time, and total volume of the slurries and volume of the settled solids. Calculate the volume of settled solids in the samples.

Day 12/28/00 Time ~ 11:00am

AN-102 AR-J OZ
Total ~~110~~ 13.3 ml
Solids 9.0 ml
Vol % Solids 67.7

AN-102 AR-K OZ
Total 13.4 ml
Solids 9.4 ml
Vol % Solids 70.1

AN-102 AR-L OZ
Total 13.6 ml
Solids 9.3 ml
Vol % Solids 68.4

AN-102 AR-M OZ
Total 13.8 ml
Solids 9.4 ml
Vol % Solids 68.1

AN-102 AR-N OZ
Total 13.7 ml
Solids 9.2 ml
Vol % Solids 67.2

AN-102 AR-O OZ
Total 13.6 ml
Solids 8.9 ml
Vol % Solids 65.4

AN-102 AR-P
~~Total ______ ml~~
~~Solids ______ ml~~
~~Vol % Solids ______~~

Not needed
PRB 1/2/01

32) **Administrative Hold Point**: If the standard deviation is less than 5 percentage units for the calculated volume percent of settled solids present in the AN-102 sub-samples, the AN-102 composite samples shall be considered homogenous. If the AN-102 sub-samples fail this criteria, then the sub-samples are to be returned to the composite vessel, agitation increased/continued and/or other modification deemed necessary to obtain improved mixing, and new sub-samples are to be obtained, and steps 27-31 repeated.

33) Transfer the mass of supernatant calculated in step 26 from one or more jars collected in step 29 to a glass jar of known mass labeled **"AN-102 SUP A"** being careful not to disturb the settled solids. Leave approximately 10 ml of standing liquid on top of any remaining settled solids. Weigh **"AN-102 SUP A"** as well as the slurry remaining in the jars after removal of supernatant.

AN-102 SUP A		**AN-102 AR-__**		**AN-102 AR-__**	
Total	________g	Total	________g	Total	________g
Tare	________g	Tare	________g	Tare	________g
Sup	________g	Slurry	________g	Slurry	________g

34) Based on the data collected, confirm the wt% undissolved solids in the composite will be between 2 and 2.5 wt% following compositing of **"AN-102 SUP A"** and **"AN-102 AR-J"**. Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Estimated Wt% Undissolved solids in the Composite __________

35) Confirm the mixing vessel is empty (does not contain visible solids or liquid). If present, document the approximate volumes and types of materials retained in the vessel. As needed, rinse the vessel with DI water then air dry. Transfer the material in **"AN-102 AR-J"** and **"AN-102 SUP A"** to the mixing vessel.

See attachment
PRB
1/08/01

36) Stir the sample by slowly increasing the motor speed until it is similar to that used successfully in step 13. Stir for a minimum of one hour. Record the sample temperature to ±1.1°C.

Time 14:30 Date 1-9-01 Temperature 31.8 °C

BLANK

37) While operating the vessel agitator, extract one sub-sample of sufficient size 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 and record the time. Then collect three ~100 ml sub-samples in volume graduated glass jars of known mass. Record the sample masses. Samples "AN-102 TRU 1" and "AN-102 TRU 2" are to be retained for Sr/TRU reaction mechanism tests. Record the time the final sample was collected.

For Physical property work

Time of Valve Clear 15:20 hr Time of final collection 15:30

	~~AN-102 TRU 1~~	~~AN-102 TRU 2~~	AN-102 MOD 1	AN-102 MOD 2
Total	~~____ g~~	~~____ g~~	298.889 g	~~167.450~~ g
Tare	~~____ g~~	~~____ g~~	133.933 g	134.067 g
Slurry	~~____ g~~	~~____ g~~	160.657 g	167.450 g

Not needed 1/09/01 PRB

133.933
+ 4.299

305.922

PRB 1/3/00

+4.4048 g

38) Turn off the stirring motor and place an airtight cover on the mixing vessel.

39) Stir "AN-102 MOD" MOD 1 as directed by the cognizant scientist (depending on solids content, a magnetic stir bar is preferred) and transfer 25 ml in duplicate to 100 ml volume graduated glass centrifuge tubes ~~of known~~ mass (Kimax 45241-100 VWR 21113-005 or similar). Record the mass of the new samples.

MOD 1 Stir Bar 4.2990

stir bar 4.4048 g MOD 2

	AN-102 MOD A		AN-102 MOD B
+ stopper Total	121.402 g	Total	116.630 g
+ stopper Tare	85.870 g	Tare	84.927 g
Slurry	35.532 g	Slurry	31.703 g

1-10-01 @ 2pm

← 1-9-01

40) Record the date and time and allow the sample collected in the previous step to settle for a minimum of 24 hours.

Day 1/10/01 Time 14:00

MOD A $d = \frac{35.53}{26.5} = 1.34$

41) Record the date and time, and total volume of the slurries and volume of the settled solids.

MOD B $d = \frac{31.703}{24.2\,mL} = 1.31$

Day 1/11/01 Time 11:00

AN-102 MOD A		AN-102 MOD B	
Total	27.0 ml	Total	24.5 ml
Solids	7.2 ml	Solids	6.5 ml

Day 1/11/01 Time 14:00

Total	26.5 mL	Total	24.2 mL
solids	6.3 mL	Solids	5.9 mL

@ 1/12/00 Total 24.2 mL
Solids 5.0 mL

AN-102 MOD 2

$$\frac{167.5\,g}{\bar{x}\ \text{density} \sim 1.32} \cong 125\,mL \rightarrow 127\,mL$$

Assume 26 mL × 1.32 g/mL ≅ 34.3 g AN-102 MOD 2 Needs to go back into AN-102 Supernatant Composite in Mixing Vessel

rys 1/12/01

42) Fill in the original C-104 gross mass data to Table 3 along with the source of this data (TI, LRB, or other). Reweigh the bottles and record these current masses in Table 3. Add de-ionized water as needed to return the samples to their original gross masses (±1 g).

Table 3. Data on C-104 additives.
Source of original mass data: Email from Kriston Brooks 12/04/00

Additive	Original Mass (g) [solution + jar]	Current Mass (g)	DI Water Added (g) Target	Actual	~ Adjusted Net wt g
C-104 Filtrate #1	~~360.35~~ 809.90	796.87	13.0	13.2 ✓	449.55
C-104 Filtrate #2	~~360.72~~ ~~9.~~ 819.14 ~~819.~~	816.95	2.2	2.17 ✓	458.42
C-104 Wash (H2O) Filtrate Composite	~~508.03~~ 1128.36	1123.16	5.2	5.31 ✓	620.4
C-104 Caustic Leach Permeate	~~509.19~~ 1024.13	1022.87	1.3	1.5 ✓	514.9
C-104 Caustic Wash Composite	~~5.6~~ ~~506.5~~ ~~506.6~~ 1134.98	1132.67	2.3	2.3 ✓	628.4

PRB 1/09/01

43) Record the density of the liquid in "**C-104 Filtrate #1**", "**C-104 Filtrate #2**", "**C-104 Wash (H2O) Filtrate Composite**", "**C-104 Caustic Leach Permeate**", and "**C-104 Caustic Wash Composite**". If the density is not known, determine it in duplicate by measuring the mass in a 5 to 25 ml glass volumetric flask.. Record this data or previously collected data and source below.

RJS 1/12/01

A density data from Kriston Brooks

C-104 Filtrate #1

Mass ________ g　　Mass ________ g
Vol ________ ml　　Vol ________ ml
ρ ________ g/ml　　ρ ________ g/ml

ρ ave 1.0212 g/ml

C-104 Filtrate #2

Mass ________ g　　Mass ________ g
Vol ________ ml　　Vol ________ ml
ρ ________ g/ml　　ρ ________ g/ml

ρ ave 1.0602 g/ml

C-104 Wash (H2O) Filtrate Composite

Mass	________g	Mass	________g
Vol	________ml	Vol	________ml
ρ	________g/ml	ρ	________g/ml

ρ ave 1.013 g/ml

C-104 Caustic Leach Permeate

Mass	________g	Mass	________g
Vol	________ml	Vol	________ml
ρ	________g/ml	ρ	________g/ml

ρ ave 1.0948 g/ml

C-104 Caustic Wash Composite

Mass	________g	Mass	________g
Vol	________ml	Vol	________ml
ρ	________g/ml	ρ	________g/ml

ρ ave 1.028 g/ml

44) Using the densities in the previous step, fill in Table 3. Have calculations checked by the cognizant scientist or technical reviewer.

AN 102 MOD A
= 27 mL, Not 25 mL
rgs 1/11/01

Table 4. Volume of Additive to AN-102 MOD A

Additive	Required Volume (ml)	Density (g/ml)	Equivalent Mass (g) 25 mL	Equivalent mass (g) 27 mL
C-104 Filtrate #1	18.2/25 mL = 19.7/27 mL	1.0212	18.59	20.07 g
C-104 Filtrate #2	18.2 = 19.7	1.0602	19.30	20.8 g
C-104 Wash (H2O) Filtrate Composite	12.3 = 13.3	1.013	12.50	13.5 g
C-104 Caustic Leach Permeate	11.7 = 12.64	1.0948	12.81	13.85 g
C-104 Caustic Wash Composite	6.3 = 6.80	1.028	6.48	7.00 g

25.3

rgs 1/12/01

45) Transfer the mass of **"C-104 Filtrate #1"** calculated in Table ~~3~~ 4 rgs to **"AN-102 MOD A"**. Reweigh **"AN-102 MOD A"**.

AN-102 MOD A

121.402g minus the stopper → 113.157

113.157	Total	~~153.931~~ g ~~133.931~~
133.544	Tare+ Slurry	~~20.387~~ g ~~133.544~~ ~~153.544~~
	C-104 Filtrate #1	20.387 g

rgs 1/12/01

46) Transfer the mass of **"C-104 Filtrate #2"** calculated in Table ~~3~~ 4 to **"AN-102 MOD A"**. Reweigh "AN-102 MOD A".

AN-102 MOD A

Total	133.544 ~~153.931~~ g
Tare+ Slurry	153.884 g
C-104 Filtrate #2	20.34 g

47) Transfer the mass of **"C-104 Wash (H2O) Filtrate Composite"** calculated in Table 3 to **"AN-102 MOD A"**. Reweigh "AN-102 MOD A".

AN-102 MOD A

Total	153.884 g
Tare+ Slurry	167.392 g
C-104 Wash (H2O) Filtrate Composite	13.508 g

48) Transfer the mass of **"C-104 Caustic Leach Permeate"** calculated in Table ~~3~~ 4 to **"AN-102 MOD A"**. Reweigh "AN-102 MOD A".

AN-102 MOD A

Total	167.392 g
Tare+ Slurry	180.904 g
C-104 Caustic Leach Permeate	13.512 g

49) Transfer the mass of **"C-104 Caustic Wash Composite"** calculated in Table ~~3~~ 4 to **"AN-102 MOD A"**. Reweigh "AN-102 MOD A".

AN-102 MOD A

Total	180.904 g
Tare+ Slurry	188.002 g
C-104 Caustic Wash Composite	7.098 g

rgs 1/24/01

50) Thoroughly agitate "**AN-102 MOD A**" by inverting the sample at least 10 times.

51) Record the date and time and allow the sample collected in the previous step to settle for a minimum of 24 hours.

Prior to mixing sample fractions added did not happen to disturb the mix (See other AN 102 Start date 12/27/00)

Day 1/12/01 Time 15:15 hr

52) Record the date and time, and total volume of the slurries, volume of the settled solids as well as visual appearance of the solids and solution. Collect video images of the sample.

Day 1/15/01 Time 11:00 rgs

AN-102 MOD A + C-104 additions		MOD B	
Total	96 ml	Total	24.0
Solids	1.80 ml	Solids	6.5

rgs

53) <u>Administrative Hold Point</u>: Do not proceed with the remainder of this test specification and consult the client if "**AN-102 MOD A**" has formed a gel (or viscous solution), solidified, or the amount of solids has increased by more than 25% of the original AN-102 solids volume.

Supernate Layer Appears to be all Liquid Phase rgs 1/15/01

54) Transfer material from "**AN-102 MOD B**" and "**AN-102 MOD**" MOD 2 PRB 1/9/01 to the mixing vessel.

55) Using data collected to this point, calculate the volume of AN-102 slurry in the mixing vessel. This should be approximately <u>569 ml</u> (569 ml = 594 ml – 25 ml in **AN-102 MOD A**). Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Volume of AN-102 Slurry 569 mL See pg 17a & 17b attached rgs

56) Using the volume calculated in the previous step, complete Table 4. Have calculations checked by the cognizant scientist or technical reviewer.

Table 5. Volume of C-104 Solutions for Addition to AN-102 Slurry

Solution	Volume per 100 ml of AN-102 (ml)	Volume for Addition (ml) x density	wt for addition (g)
C-104 Filtrate #1	72.8 × 5.69 =	414.23 · 1.0212 =	423.0
C-104 Filtrate #2	72.8 =	414.23 · 1.0602 =	439.2
C-104 Wash (H2O) Filtrate Composite	49.1 =	279.38 · 1.013	283
C-104 Caustic Leach Permeate	46.9	266.86 · 1.0948	292.2
C-104 Caustic Wash Composite	25.3	143.96 · 1.028	148.0

54) The target wt of 17.4 g from AN-102 MOD 2 was transferred to AN-102 MOD B rgs 1/18/01

Final wt MOD B -

TP-41500-005

[illegible]
1/15/01

Pg 17a of 19

Total in mixing vessel

All of E	167.8 g
decant from J	160.565
" K	147.~~497~~ → 863 RB 1/15/01
" L	175.497
" M	173.346
" N	168.471
	~~993.176~~

total = 993.54 g

removed

AN-102 MOD 1	160.657
AN-102 MOD 2	167.450
	328.107 g

Checked by
1/30/01

993.54
− 328.107
665.433 g on 1/15/01

all Backs
in mod B 31.703
MOD 1 [(160.657)−(35.532)−(31.703)] = 93.422
125.125 g

Total will be 665.433
125.125
790.558 g $\xrightarrow{1.42 g/ml}$ 556.73 ml

TP-41500-005

[illegible]
1/15/01

Pg 176 of A

Need 569 ml in mixing vessel
⇓
807.98g

difference is 807.98g - 790.558 = 17.4g

So remove 17.4g from AN-102 MOD2
and transfer to mixing vessel

PRB
1/15/01

check
1/30/01

Pg 17c of 17

Sample Bottle Name	C-104 Filtrate #1	C-104 Filtrate #2	C-104 Wash (H2O) Filtrate Composite	C-104 Caustic Leach Permeate	C-104 Caustic Wash Composite
Description	Permeate removed after initial material was added to CUF	Permeate removed after second slurry sample was added to CUF	Combined permeate from three 0.1M NaOH washes	Permeate from caustic leach at 85°C	Combined permeate from 2 washes after caustic leach
Density (g/mL)	1.0212	1.0602	1.013	1.0948	1.028
Mass (g)	449.55	458.42	620.33	514.94	628.31
Volume (mL)	440	432	612	470	611
maximum estimated vol of as-received feed, mL	604.7	593.9	1246.6	1002.9	2415.8
volume increase factor	3.669	3.669	3.669	3.669	3.669
maximum estimated vol of diluted feed, mL	2219	2179	4574	3680	8863
volume of C-104 solution needed, mL	432.4	432.4	291.6	278.6	150.3

volumes

#1 5.94 · 72.8 = 432

#2 5.94 = 432

(H2O) 5.94 · 49.1 = 291

Leach 5.94 · 46.9 = ~~278~~ 278

W. 1 5.94 · 25.3 " 150

Prepared By Dean Kurath

Checked By

1/30/01

57) Stir the sample by slowly increasing the motor speed until it is similar to that used successfully in step 13. Stir for a minimum of 10 minutes. Record the sample temperature to ±1.1°C.

Start Time 09:15 Date 1-18-01 Temperature 32.1 °C

End transfer Time 09:45

58) While stirring the mixing vessel, transfer the material in **"AN-102 MOD A"** to the mixing vessel.

59) While continuing to operate the vessel agitator, use Alternately estimate wt & record wt added. rgs 1/11/01 a graduated cylinder of appropriate size with a tolerance of ≤1% of full scale (Corning style 63024 or similar) to add the volumes of C-104 solutions listed in Table 4 to the AN-102 slurry in the mixing vessel. Record the masses for each of these additions below.

* AN-102 TRAN 3 → TARE = 218.496

to Temporary Hold Bottle	Solution	Gradu~~ated~~ ~~Cylinder~~ Mass (g)	Total Mass (g)	Actual Solutions Mass (g)	Projected TARGET wt g	
DF-B	C-104 Filtrate #1	218.496 tared to zero	188.40 + 178.21 + 48.46	415.07 **	423	short 8 g
DF-C	C-104 Filtrate #2		162.91 + 157.64 + 107.15	427.70 **	439	short 12 g
DF-D	C-104 Wash (H2O) Filtrate Composite		142.28 + 142.12	284.4	283	
DF-E	C-104 Caustic Leach Permeate		171.53 + 121.10	292.6	292.2	
DF-A	C-104 Caustic Wash Composite	DF-A tared to 0	149.1	149.1	148	

Total ~ 1568.9 g + MODA volume (75.3) = 1643.3 g

60) Continue to stir for a minimum of one hour. Record the sample temperature to ±1.1°C.

Time 11:30 Date 01-18-01 Temperature 32.1 °C

* Since TARE wt of transfer is 218.496 g & Balance total cap ~ 400 g we will need to do a couple wt transfer to get to projected target wts

** - entire Sample was used here.

59) - Lost ~ 5mL to 10mL of C-104 Filtrate #2 ~~Wash~~ when pouring to the mixer rgs (1-18-01)

60) - Interrupted stirring to raise vessels off ground to be able to collect into 500mL bottles. Increase stirring speed to compensate.

61) While operating the vessel agitator, extract one sub-sample of sufficient size 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 and record the time. Then collect all the material in the mixing vessel in pre-weighed volume graduated glass jars. The number and volume of samples will be determined by the cognizant scientist based on the needs of the analytical tasks. Use the necessary spaces below to record the sample masses. Record the time the final sample was collected. These sub-samples will be used for characterization of the diluted feed, and process testing. Analytical testing will be documented on an Analytical Sample Request form (ASR). All remaining samples are to be stored in the hot cells in appropriate secondary containment until required for further testing.

To SAL
1-25-01

Time of Valve Clear 1-18-01 @ 11:33   Time of final collection 1-18-01@

AN-102 DF A
Target 150 ml
Total 409.690 g
Tare 218.342 g
Slurry 191.35 g

AN-102 DF B
Target 500 ml
Total 822.28 g
Tare 308.442 g
Slurry 513.84 g

AN-102 DF C
Target 500 ml
Total 813.21 g
Tare 308.160 g
Slurry 505.05 g

AN-102 DF D
Target 500 ml
Total 831.58 g
Tare 308.956 g
Slurry 522.62 g

AN-102 DF E
Target 500 ml
Total 812.76 g
Tare 308.778 g
Slurry 503.98 g

AN-102 ~~DF F~~ TRANS 3
Target recirculation ml ~ 50-100ml
Total 337.228 g
Tare 217.616 g
Slurry 119.61 g

Total wt.
DF composite
2356.45 g

AN-102 DF G
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

AN-102 DF H
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

AN-102 DF I
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

AN-102 DF J
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

AN-102 DF K
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

AN-102 DF L
Target ______ ml
Total ______ g
Tare ______ g
Slurry ______ g

YBurner 1/18/00

Total wts Need to be taken in "B" cell - manipulators are down currently
1/19/00

Attachment to Test Plan 41500-005

Author Pa B 1/08/01   Technical Reviewer D E Kurath 1/8/01

Applicability

This attachment to Test Plan 41500-005 provides details on the preparation of the 2 to 5 wt% feed. This attachment is needed due to a minor deviation from the Test Plan resulting from power outages in the 325 building. As a result of these outages, the entire initial composite was collected from the pot. Therefore, sample AN-102 AR-J was not collected as planned. The following steps detail how to prepare the ~~3-5~~ 2 to 5 PRB 1/8/01 wt% feed based on the current state of the sub-samples.

This document provides details regarding the implementation of Test Plan 41500-004, "Sample Compositing". Client expectations for successful achievement of project data needs have already been established via Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification". This document is a mechanism for the cognizant scientist to communicate to technical staff specifics on Test Plan 41500-004 implementation. 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 a cognizant scientist.

Work with actual tank material will be performed in radiological hot cells.

Work Instructions

1) Weigh two clean 250 ml jars labeled "**AN-102 TRANS 1**" and "**AN-102 TRANS 2**".

AN-102 TRANS 1	**AN-102 TRANS 2**
Tare 218.237 g	Tare 219.215 g

2) Install the baffles into the mixing vessel.

(See pg 1c following) **Note:**

In the following steps, 825 g of supernatant are to be separated from the AN-102 composite sub-samples and added into the mixing vessel along with sub-sample AN-102 AR-E to prepare the ~~3-5~~ wt% feed. The volumes of supernatant added to the mixing vessel are to be recorded in Table 1. The following steps are provided to document how much supernatant is removed from each sub-sample. The total supernatant mass of 825 g may be reach without the need to remove supernatant from all sub-samples indicated. Discontinue supernatant collection once the required supernatant mass is reached.

2 + 5 PRB

Attachment to Test Plan 41500-005

Page ______ of ______

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: P Bredt | Date: 1/8/01 | Project: 41500-005

Title/Subject: Attachment to Test Plan 41500-005
steps 24-26

Need 694 ml of 3.5 wt% feed

Supernatant ρ = 1.43 g/ml

Slurry ρ = 1.46 g/ml

Settled solids concentration = 70.8 vol%, 71.5 wt%

Undissolved Solids concentration ≅ 18 wt%

694 ml · 1.43 g/ml = 992.4 3wt% slurry needed

992.4 g · 0.035 = 34.7 g of undissolved solids needed

$$\frac{34.7\text{ g}}{x} = \frac{18\text{ g solids}}{100\text{ g original slurry}}$$

$$x = \boxed{192.7\text{ g of original slurry needed}}$$

$$192.7\text{ g slurry} \cdot \frac{71.5\text{ g settled solids}}{100\text{ g original slurry}} = \boxed{137.8\text{ g of settled solids needed}}$$

~~settled~~

s

$$992.4\text{ g} - 137.8\text{ g} = \boxed{854.6\text{ g of excess supernatant needed}}$$

- - - - - - - - - - - - - - - - - - - -

Sample AN-102 ARE contains 167.79 g of original slurry

167.79 · 0.18 = 30.20 g undissolved solids

$$\frac{30.2\text{ g}}{992.4\text{ g}} = 3\text{ wt\% undissolved solids}$$

992.4 - 167.79 g = 824.6 g of excess supernatant needed

If we use AN-102 ARE
for solids, and decant
remaining samples
for excess supernatant,
then we will generate
~3wt% slurry.

checked by D E Kurath 1/8/01

> Note:
> Confirm that the analytical balance in C-Cell has the appropriate capacity to weigh the items in the following steps.

3) Without disturbing the settled solids in **AN-102 AR-J**, transfer as much standing liquid from **AN-102 AR-J** as possible to **AN102 TRANS 1**. Weigh **AN-102 AR-J** and **AN-102 TRANS 1**. Enter the liquid value in **AN-102 TRANS 1** into Table 1.

AN-102 AR-J		AN-102 TRANS 1	
Total	735.24 g	Total	378.802 g
Tare	311.15 g	Tare	218.237 g
Slurry	424.09 g	Liquid	160.565 g

Table 1. Mass of liquid removed from composite sub-samples to prepare a 2-5 wt% feed.

Sample	Mass of Liquid (g)
AN-102 AR-J	160.565
AN-102 AR-K	147.863
AN-102 AR-L	175.497
AN-102 AR-M	173.346
AN-102 AR-N	168.471
AN-102 AR-O	—

} see following page

Total = 825.742 g

AN-102 Supernatant + 167.8 g of AN-102 AR-E
= 993.54 g of DF-comp.
÷ d of 1.43
≈ 695 mL

4) Without disturbing the settled solids in **AN-102 AR-K**, transfer approximately 170 g of standing liquid from **AN-102 AR-K** to **AN102 TRANS 2**. Leave at least 10 ml of standing liquid on the settled solids. Weigh **AN-102 AR-K** and **AN-102 TRANS 2**. Enter the liquid value in **AN-102 TRANS 2** into Table 1.

AN-102 AR-K		AN-102 TRANS 2	
Total	752.84 g	Total	367.078 g
Tare	311.42 g	Tare	219.215 g
Slurry	441.42 g	Liquid	147.863 g

5) Transfer the material in **AN-102 AR-E** to the mixing vessel. Use the supernatant in **AN-102 TRANS 1** and **AN-102 TRANS 2** to aid in this transfer.

6) Transfer the material in **AN-102 TRANS 1** and **AN-102 TRANS 2** to the mixing vessel.

7) Set the lid on the mixing vessel.

8) Without disturbing the settled solids in **AN-102 AR-L**, transfer approximately 170 g of standing liquid from **AN-102 AR-L** to **AN102 TRANS 1**. Leave at least 10 ml of standing liquid on the settled solids. Weigh **AN-102 AR-L** and **AN-102 TRANS 1**. Enter the liquid value in **AN-102 TRANS 1** into Table 1.

AN-102 AR-L		AN-102 TRANS 1	
Total	733.72 g	Total	393.734 g
Tare	311.50 g	Tare	218.237 g
Slurry	422.22 g	Liquid	175.497 g

9) Without disturbing the settled solids in **AN-102 AR-M**, transfer approximately 170 g of standing liquid from **AN-102 AR-M** to **AN102 TRANS 2**. Leave at least 10 ml of standing liquid on the settled solids. Weigh **AN-102 AR-M** and **AN-102 TRANS 2**. Enter the liquid value in **AN-102 TRANS 2** into Table 1.

AN-102 AR-M		AN-102 TRANS 2	
Total	743.30 g	Total	392.561 g
Tare	311.13 g	Tare	219.215 g
Slurry	432.17 g	Liquid	173.346 g

10) Remove the cover from the mixing vessel and transfer the material in **AN-102 TRANS 1** and **AN-102 TRANS 2** to the mixing vessel. Then recover the mixing vessel.

11) Without disturbing the settled solids in **AN-102 AR-N**, transfer approximately 170 g of standing liquid from **AN-102 AR-N** to **AN102 TRANS 1**. Leave at least 10 ml of standing liquid on the settled solids. Weigh **AN-102 AR-N** and **AN-102 TRANS 1**. Enter the liquid value in **AN-102 TRANS 1** into Table 1.

AN-102 AR-N		AN-102 TRANS 1	
Total	749.46 g	Total	~~391.115~~ g 386.708
Tare	311.08 g	Tare	218.237 g
Slurry	438.38 g	Liquid	168.471 g

1-9-01

12) Without disturbing the settled solids in **AN-102 AR-O**, transfer approximately 170 g of standing liquid from **AN-102 AR-O** to **AN102 TRANS 2**. Leave at least 10 ml of standing liquid on the settled solids. Weigh **AN-102 AR-O** and **AN-102 TRANS 2**. Enter the liquid value in **AN-102 TRANS 2** into Table 1.

AN-102 AR-O		**AN-102 TRANS 2**	
Total	______ g	Total	______ g
Tare	311.30 g	Tare	______ g
Slurry	______ g	Liquid	______ g

Not needed 1/9/01

13) Remove the cover from the mixing vessel and transfer the material in **AN-102 TRANS 1** and **AN-102 TRANS 2** to the mixing vessel. Then recover the mixing vessel.

14) Proceed to step 36 of the test plan.

BLANK

REFERENCE COPY

PNNL Test Instruction

Document No.: TP-41500-015
Rev. No.: 0

102 rgs 12/12/00

Title: AN-~~107~~ Integrated Test: Solids Analysis

Work Location: Radiochemical Processing Laboratory

Page 1 of 11

Author: Paul Bredt

Use Category Identification: Information

Effective Date: Upon Final Approval
Supersedes Date: New

Identified Hazards:
__ Radiological
__ Hazardous Materials
__ Physical Hazards
__ Hazardous Environment
__ Other:

Required Reviewers:
_X_ Technical Reviewer _X_ SFO Manager

Are One-Time Modifications Allowed to this Procedure? _X_ Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or _X_ No

FOR REVISIONS:
Is retraining to this procedure required? ____ Yes _X_ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____ Yes ____ No _X_ N/A

Approval	**Signature**	**Date**
Author	Paul B	12/12/00
Technical Reviewer		12/12/00
FSO Manager	Not Needed PRB 12/12/00	

102 12/12/00 [illegible]

AN-~~107~~ Integrated Test: Solids Analysis

This test instruction defines work to be conducted on slurry samples collected during the integrated testing of materials from tanks C-104 and AN-102. The samples are to be prepared by task 2.1 under test plan 41500-005, "AN-107 Sample Compositing for Process Testing". It is anticipated that solids analyses will be conducted on the as-received composite, and the diluted feed. This test instruction provides detail and a process for conducting the solids analysis.

This test instruction provides details regarding the implementation of Technical Procedure 29953-010. This test instruction is an internal mechanism for the cognizant scientist, to communicate to staff specifics on procedure implementation. Therefore, this document qualifies as a Test Instruction under Part B section 16 of the RPL Operations Manual.

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 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 Plan, bench sheets, and LRBs will be transferred to the project file upon completion of the project.

General Instructions:

1) Keep the sample in a sealed container as much as possible to prevent it from drying.
2) Sign and date the bottom of each page when the requested analyses are complete.
3) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.

Signature ____________ Start Date 12/27/00

M&TE List:

_____ Balance 1:

Calib ID 388-06-01-020 Calib Exp Date 8/01

Location C-cell

~~_____ Balance 2:~~

~~Calib ID ____________~~ Calib Exp Date________

~~Location ________~~

Not Needed PRB 10/13/01

Record balance check information below:

Loc
C-cell
↓

Date	Actual (g)	Measured (g)	Tolerance (g)	Acceptable (Y/N)
12/27/00	100.001	99.999	±.01	y
12/28/00	100.001	99.999	↓	y
12/29/00	100.001	100.000		y
1/3/01	100.001	99.998		y
1/4/01	100.001	99.999		y
1/5/01	100.001	100.000	±0.01	y
01/10/01	200.000	200.002	± ~~0.002~~ .01	y
01/16/01	100.001	100.000	± .0025 .01	y
01/16/01	200.001	200.006	± .01	y

rg> 1-10-01

Signature ______________ Date 1-16-01

PR Bredt
December 11, 2000

Test Instruction: TI-41500-015
Page 4 of 11

As-Received Solids Testing

After 4 days + settling
rgs 12/27/00
AR-H Total 102 mL
solids 77 mL
wt%. ≅ $\boxed{75\%}$

1) Record the identification number for the sample of as-received initial slurry provided by the compositing task.

Sample ID AN-102AR-H from TP-005 pg 9 Slurry = 147.18g

2) Thoroughly agitate the sample provided and transfer ~8 ml of each in 4 quadruplicate ~~duplicate into~~ preweighed graduated 10 ml centrifuge cones. These cones need to be rate for use at ~105°C. Weigh the loaded centrifuge cones. rgs 12/27/00

12/27/00
1300hr

	AN102 AR A		AN102 AR ~~A~~ B	C	D
Total	32.860 g	Total	32.928 g	32.844 g	33.313 g
Tare	19.118 g	Tare	19.054 g	19.002 g	19.297 g
Slurry	13.742 g	Slurry	13.874 g	13.842 g	14.016 g

PRB 12/19/00

3) Agitate the cones and record the time and date.

Time 13:30 Date 12/27/00

immediately run samples C & D through steps 12-17

~~Hold C & D for steps~~

4) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date 12/27/00 Time 14:30

	AN102 AR A		AN102 AR B
Total	9.6 ml	Total	9.50 ml
Solids	9.4 ml	Solids	9.35 ml
Liquid	0.2 ml	Liquid	.15 ml

5) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date 12/27/00 Time 16:15

	AN102 AR A		AN102 AR B
Total	9.60 ml	Total	9.5 ml
Solids	9.40 ml	Solids	9.35 ml
Liquid	0.2 ml	Liquid	0.15 ml

Signature Date 12/27/00

6) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 12/28/00 Time 07:55

	AN102 AR A			AN102 AR B	
Total	9.40	ml	Total	9.4	ml
Solids	7.4	ml	Solids	7.4	ml
Liquid	2.0	ml	Liquid	2.0	ml

7) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 12/28/00 Time 3:45 pm

	AN102 AR A			AN102 AR B	
Total	9.4	ml	Total	9.4	ml
Solids	6.9	ml	Solids	7.0	ml
Liquid	2.5	ml	Liquid	2.4	ml

8) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 12/29/00 Time 08:05

	AN102 AR A			AN102 AR B	
Total	9.40	ml	Total	9.40	ml
Solids	6.80	ml	Solids	6.70	ml
Liquid	2.6	ml	Liquid	2.7	ml

9) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 12/29/00 Time 9:49 Am

	AN102 AR A			AN102 AR B	
Total	9.4	ml	Total	9.4	ml
Solids	6.7	ml	Solids	6.6	ml
Liquid	2.7	ml	Liquid	2.8	ml

PRB 1/03/01

$A = 2.7 / \cancel{9.4}\ 9.4 = \cancel{29}\ 28.7$ vol% <u>supernatant</u>

$B = \frac{2.8}{9.4} = 29.8$

Av = 29.2 vol%

Signature Date 12/29/00

10) At the beginning of the next day (day 4), record the volume of the total sample and the volume of settled solids.

DAY 4
Not done New Yr's Weekend
12/29/00

Date 1-3-01 Time 07:43

AN102 AR A			AN102 AR B		
Total	9.4	ml	Total	9.4	ml
Solids	6.7	ml	Solids	6.6	ml
Liquid	2.7	ml	Liquid	2.8	ml

this DATA is OK but taken on 1-3-01

11) Transfer a sample of the settled supernatant into a preweighed graduated cylinder and record the mass and volume of the supernatant. Return the supernatant to the centrifuge cone.

AN102 AR A			AN102 AR B		
Total	42.947	g	Total	40.552	g
Tare	39.264	g	Tare	~~19.054~~ 36.889	g
Liquid	3.683	g	Liquid	3.663	g
Liquid	2.65	ml	Liquid	2.60	ml
ρ = 1.39 g/mL			ρ = 1.41 g/mL		

12/29/00

12) 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.

AN102 AR A			AN102 AR B			C		D	
Total	32.595	g	Total	32.692	g	32.844	g	33.313	g
Tare	19.118	g	Tare	19.054	g	19.002		19.297	
Sample	13.477	g	Sample	13.638	g	13.842		14.016	
Total	9.20	ml	Total	9.25	ml	9.50	mL	9.40	mL
Solids	4.35	ml	Solids	4.50	ml	4.3		4.1	
Liquid	4.85	ml	Liquid	4.75	ml	5.2		5.3	

PRB 12/19/00

12/27/00 @ 3:00pm

For C & D centrifuging
Start 12/27/00 @ 14:10
End " 15:10

For A & B centrifuging
Start 1-3-01 @ 10:00
End " @ 11:00

Signature ____________ Date 12/29/00

13) Decant as much of the centrifuged supernatant as possible to a preweighed graduated cylinder and record the mass and volume of the supernatant. Weigh the solids left in the centrifuge cone.

C		AN102 AR A		AN102 AR B	D
46.235 g	Liquid + Grad	45.939 g	Liquid + Grad	43,630 g	44.378
38.990 g	Grad	39.264 g	Grad	36.889 g	37.122 g
7.245 g	Liquid	6.675 g	Liquid	6.741 g	7.256 g
5.05 ml	Liquid	~~5.05~~ 4.75 ml	Liquid	4.70 ml	5.10 ml
d: 1.436		d = 1.405		d = 1.434	d ≅ 1.423
25.528 g	Solids + cone	25.859 g	Solids + cone	26.122 g	25.936 g
19.002 g	Cone	19.118 g	Cone	19.054 g	19.297 g
6.526 g	Solids	6.741 g	Solids	7.068 g	6.639 g

rg 1-3-01

14) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers.

CL		AN102 AR AL		AN102 AR BL	DL
23.908 g	Total	23.486 g	Total	23.452 g	24.157 g
16.812 g	Tare	16.917 g	Tare	16.885 g	17.068 g
7.096 g	Supernatant	6.569 g	Supernatant	6.567 g	7.089 g

15) 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.

C & D Start drying 12/28/00 @ 9 am

16) Transfer the solids and liquids to an oven at 105°C for 24 hours.

End drying 12/29/00 @ 9 Am

17) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~~~1 hour~~ Preliminary and reweigh.

1/4/00 @ 10 am - weighed without cooling

1/4/01

	CL		AN102 AR AL		AN102 AR BL	DL	
20.568 20.556	20.631 g	Total	21.092 20.861 g (F)	Total	20.512 20.462 g (F)	20.866 g	20.807 20.795 (F)
	16.812 g	Tare	16.917 g	Tare	16.885 g	17.068 g	
(F) 3.744	3.819 g	sample	4.175 3.944 g	sample	3.627 3.577 g	3.798 g	3.727

	C		AN102 AR A (F)		AN102 AR B (F)	D	
24.480	24.952 g	Total	25.202 25.101 g	Total	25.233 25.068 g	25.344 g	24.840
24.395	19.002 g	Tare	19.118 g	Tare	19.054 g	19.297 g	24.744
(F) 5.393	5.95 g	sample	6.084 5.983 g	sample	6.179 6.014 g	6.047 g	5.447

(F) = final

(See reverse side)
rgs

A & B Start drying on 1-3-01 @ 13:50 hr

Signature rg Date 1-3-01

This test material Does NOT contain C-104 solutions PRB 2/28/01

3 wt%

18) Record the identification number for the sample of ~~as received initial~~ slurry provided by the compositing task.

Sample ID AN-102 MOD 2 PRB 1/10/01

19) Thoroughly agitate the sample provided and transfer ~~~8~~ ml of each in duplicate into preweighed graduated 10 ml centrifuge cones. These cones need to be rate for use at ~105°C. Weigh the loaded centrifuge cones.

5 mL for "C" sample

	AN102 DF A		AN102 DF B		AN102 DF C
Total	27.975 g	Total	28.001 g		26.228 g
Tare	19.347 g	Tare	19.200 g	← 1-10-01	19.158 g
Slurry	8.628 g	Slurry	8.801 g		7.070 g

20) Agitate the cones and record the time and date.

Time 11:45 Date 1/10/01

21) One hour after agitation, record the volume of the total sample and the volume of settled solids.

~~Date~~ Time 14:00 ~~Time~~ DATE 1/10/01

	AN102 DF A		AN102 DF B	AN-102 DF-C
Total	6.25 ml	Total	6.30 ml	5.1
Solids	6.25 ml	Solids	6.30 ml	5.1
Liquid	— ml	Liquid	— ml	—
d =	1.38		wet ρ = 1.40	ρ = 1.386

22) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date 01-10-01 Time 15:30

	AN102 DF A		AN102 DF B
Total	6.25 ml	Total	6.30 ml
Solids	6.25 ml	Solids	6.30 ml
Liquid	— ml	Liquid	— ml

Signature ________ Date 1-10-01

23) At the start of the next day (Thurs day 2), record the volume of the total sample and the volume of settled solids.

Date 1/11/01 Time 11:00 am

	AN102 DF A			AN102 DF B	
Total	6.15	ml	Total	6.20	ml
Solids	1.52	ml	Solids	1.48	ml
Liquid	4.63	ml	Liquid	4.72	ml

24) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 1/11/01 Time 4:15 pm

	AN102 DF A			AN102 DF B	
Total	6.2	ml	Total	6.2	ml
Solids	1.5	ml	Solids	1.5	ml
Liquid	4.7	ml	Liquid	4.7	ml

25) At the start of the next day (Friday day 3), record the volume of the total sample and the volume of settled solids.

Date 1/12/01 Time 09:00

	AN102 DF A			AN102 DF B	
Total	6.2	ml	Total	6.2	ml
Solids	1.3	ml	Solids	1.4	ml
Liquid	4.9	ml	Liquid	4.8	ml

26) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date______ Time______

	AN102 DF A			AN102 DF B	
Total		ml	Total		ml
Solids		ml	Solids		ml
Liquid		ml	Liquid		ml

Set up for centrifuging & drying at this pt. So we can dry over the weekend
RB 1/12/01

Signature ______ Date 1-12-01

27) At the beginning of the next day (~~day 4~~ Monday – day 6), record the volume of the total sample and the volume of settled solids.

Date________ Time________

Not done, went to centrifuging & drying 1/12/01 rqs

AN102 DF A		AN102 DF B	
Total	________ml	Total	________ml
Solids	________ml	Solids	________ml
Liquid	________ml	Liquid	________ml

28) Transfer a sample of the settled supernatant into a preweighed graduated cylinder and record the mass and volume of the supernatant. Return the supernatant to the centrifuge cone.

AN102 DF A		AN102 DF B	
Total	________g	Total	________g
Tare	________g	Tare	________g
Liquid	________g	Liquid	________g
Liquid	________ml	Liquid	________ml

Not needed 1/10/01 PRB

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.

	AN102 DF A	AN102 DF B	AN102 DF C
Total	27.975 g	28.001 g	26.224 g
Tare	19.347 g	19.200 g	19.158 g
Sample	8.628 g	8.801 g	7.066 g
Total	6.15 ml	6.15 ml	5.05 ml
Solids	.85 ml	0.80 ml	0.58 ml
Liquid	5.30 ml	5.35 ml	4.47 ml

Centrifuging ↓	DF A	DF B	DFC
Start	01/12/01 @ 9:00	01/12/01 @ 9:00	01/11/01 8:35
Stop	01/12/01 @ 10:00	01/12/01 @ 10:00	↓ 9:42

rqs 1/11/01

Signature ____________ Date 1/12/01

30) ~~Decant as much of the centrifuged supernatant as possible to a preweighed graduated cylinder and record the mass and volume of the supernatant.~~ Weigh the solids left in the centrifuge cone.

Not needed 1/10/01 PRB

	AN102 DF A		AN102 DF B	AN102 DF C
~~Liquid + Grad~~	~~g~~	~~Liquid + Grad~~	~~g~~	
~~Grad~~	~~g~~	~~Grad~~	~~g~~	
~~Liquid~~	~~g~~	~~Liquid~~	~~g~~	
~~Liquid~~	~~ml~~	~~Liquid~~	~~ml~~	
	AN-102 DFA		AN-102 DFB	
Solids + cone	20.583 g	Solids + cone	20.421 g	20.146 g
Cone	19.347 g	Cone	19.200 ~~19.258~~ [illegible] g	19.158 g
Solids	1.236 g	Solids	1.221 g	0.988 g

31) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers, and centrifuge cones.

	AN102 DF AL		AN102 DF BL	AN102 DF C
Total	23.666 g	Total	21.896 g	22.901 g
Tare	16.846 g	Tare	17.022 g	16.865 g
Supernatant	6.82 g	Supernatant	4.874 g	6.036 g

Lost some supernatant on floor

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.

33) Transfer the solids and liquids to an oven at 105°C for 24 hours.

34) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh.

1/15/01 @ 11:45

See opposite page

	AN102 DF AL		AN102 DF BL	AN102 DF CL
Total	20.292 g	Total	20.138 g	19.901 g
Tare	16.846 g	Tare	17.022 g	16.865 g
~~sample~~	~~g~~	~~sample~~	~~g~~	~~g~~

Not stable PRB 3/16/01

	AN102 DF A		AN102 DF B	AN-102 DFC
Total	21.369 g	Total	20.117 g	20.001 g
Tare	19.347 g	Tare	19.200 g	19.158 g
~~sample~~	~~g~~	~~sample~~	~~g~~	~~g~~
				g

Signature ____ Date 1-12-01

Blank

Test Plan: BNFL-TI-41500-015

Physical Testing: Feed Characterization Summary Report.
Tank and Test Matrix ---- Date: 10/31/2001
AN-102 AR-H (as received)

Step #	Description	units	AN-102-H -AR A	AN-102-H -AR B	AN-102-H -AR C	AN-102-H -AR D
	Centrifuge cone tare	g	19.118	19.054	19.002	19.297
	Total Wt after transfer	g	32.860	32.928	32.844	33.313
	Total Initial Volume	mL	9.60	9.50	9.50	9.40
	72 hr total Vol	mL	9.40	9.40	9.50	9.40
	72 hr total settled solids	mL	6.70	6.60		
	Total Wt after centrifuging	g	32.595	32.692	32.844	33.313
	Total Volume after centrifuging	ml	9.20	9.25	9.50	9.40
	Solids volume mark after cent.	ml	4.35	4.50	4.30	4.10
	Tare Wt of Graduate	g	39.264	36.889	38.990	37.122
	Liquid plus Graduate wt.	g	45.939	43.630	46.235	44.378
	Liquid Volume in graduate	mL	4.75	4.70	5.05	5.10
	Total Wet Solids Wt. + cone	g	25.859	26.122	25.528	25.936
	Tare Wt of 20mL Vial	g	16.917	16.885	16.812	17.068
	Liquid plus 20mL Vial wt.	g	23.486	23.452	23.908	24.157
	Final Dry Wt. of Cone	g	23.411	23.610	22.940	23.531
	Final Dry Wt. of 20mL Vial	g	20.240	20.181	20.379	20.631

							AN-102-H -AR	
Calc. Step	Description	units	A	B	C	D	AVE	std.dev
1	Bulk Density	g/mL	1.494	1.500	1.457	1.491	1.485	0.02
2	Vol % Settled Solids	%	71.3%	70.2%			71%	1%
3	Density of Centrifuged Solids	g/mL	1.550	1.571	1.518	1.619	1.564	0.04
4	Vol % Centrifuged Solids	%	47.3%	48.6%	45.3%	43.6%	46.2%	2.2%
5	Wt. % Centrifuged Solids	%	50.0%	51.8%	47.1%	47.4%	49.1%	2.2%
6	Supernatant Density	g/mL	1.405	1.434	1.435	1.423	1.424	0.01
7	Density of Settled Solids	g/mL	1.485	1.494			1.489	0.01
8	Wt. % Settled Supernatant	%	27.6%	28.9%			28.3%	0.9%
9	Wt% dissolved solids in supernatant	%	50.6%	50.2%	50.3%	50.3%	50.3%	0.2%
10	Wt% total solids in Centrifuged Sludge	%	63.7%	64.5%	60.3%	63.8%	63.1%	1.8%
11	Wt.% Total Solids	%	57.2%	57.6%	55.1%	56.8%	56.7%	1.1%
12	Wt. % Undissolved Solids	%	13.26%	14.85%	9.55%	12.87%	13%	2%

some sample loss PRB 11/01/01

Analyst: 11/01/01 Reviewer: 11/01/01

Test Plan: BNFL-TI-41500-015

Physical Testing: Feed Characterization Summary Report.
Tank and Test Matrix ----
AN-102- E- DF (diluted feed)

Date: 10/31/2001

			MOD-2	MOD-2	MOD-2
			DF-	DF-	DF-
Step #	Description	units	A	B	C
	Centrifuge cone tare	g	19.347	19.200	19.158
	Total Wt after transfer	g	27.975	28.001	26.228
	Total Initial Volume	mL	6.15	6.15	5.05
	72 hr total Vol	mL	6.15	6.15	5.05
	72 hr total settled solids	mL	1.30	1.40	
	Total Wt after centrifuging	g	27.975	28.001	26.224
	Total Volume after centrifuging	ml	6.15	6.15	5.05
	Solids volume mark after cent.	ml	0.85	0.80	0.58
	Tare Wt of Graduate	g			
	Liquid plus Graduate wt.	g			
	Liquid Volume in graduate	mL			
	Total Wet Solids Wt. + cone	g	20.583	20.421	20.146
	Tare Wt of 20mL Vial	g	16.846	17.022	16.865
	Liquid plus 20mL Vial wt.	g	23.666	21.896	22.901
	Final Dry Wt. of Cone	g	20.105	19.959	19.714
	Final Dry Wt. of 20mL Vial	g	20.485	19.574	19.811

						MOD-2	
						DF-	
Calc. Step	Description	units	A	B	C	AVE	std.dev
1	Bulk Density	g/mL	1.403	1.431	1.400	1.411	0.02
2	Vol % Settled Solids	%	21.1%	22.8%		22.0%	1.1%
3	Density of Centrifuged Solids	g/mL	1.454	1.526	delete ~~1.703~~	1.49 ~~1.561~~	5% RPD ~~0.13~~
4	Vol % Centrifuged Solids	%	13.8%	13.0%	~~11.6%~~	13% ~~12.8%~~	6% RPD ~~1.2%~~
5	Wt. % Centrifuged Solids	%	14.3%	13.9%	~~14.0%~~	14% ~~14.1%~~	3% RPD ~~0.2%~~
6	Supernatant Density	g/mL					
7	Density of Settled Solids	g/mL					
8	Wt. % Settled Supernatant	%					
9	Wt% dissolved solids in supernatant	%	53.4%	52.4%	48.8%	51.5%	2.4%
10	Wt% total solids in Centrifuged Sludge	%	61.3%	62.2%	56.3%	59.9%	3.2%
11	Wt.% Total Solids	%	54.6%	54.3%	49.9%	52.9%	2.7%
12	Wt. % Undissolved Solids	%	2.45%	2.85%	2.04%	2.4%	0.4%

11/1/01

Analyst: 11/01/01

Reviewer: 11/01/01

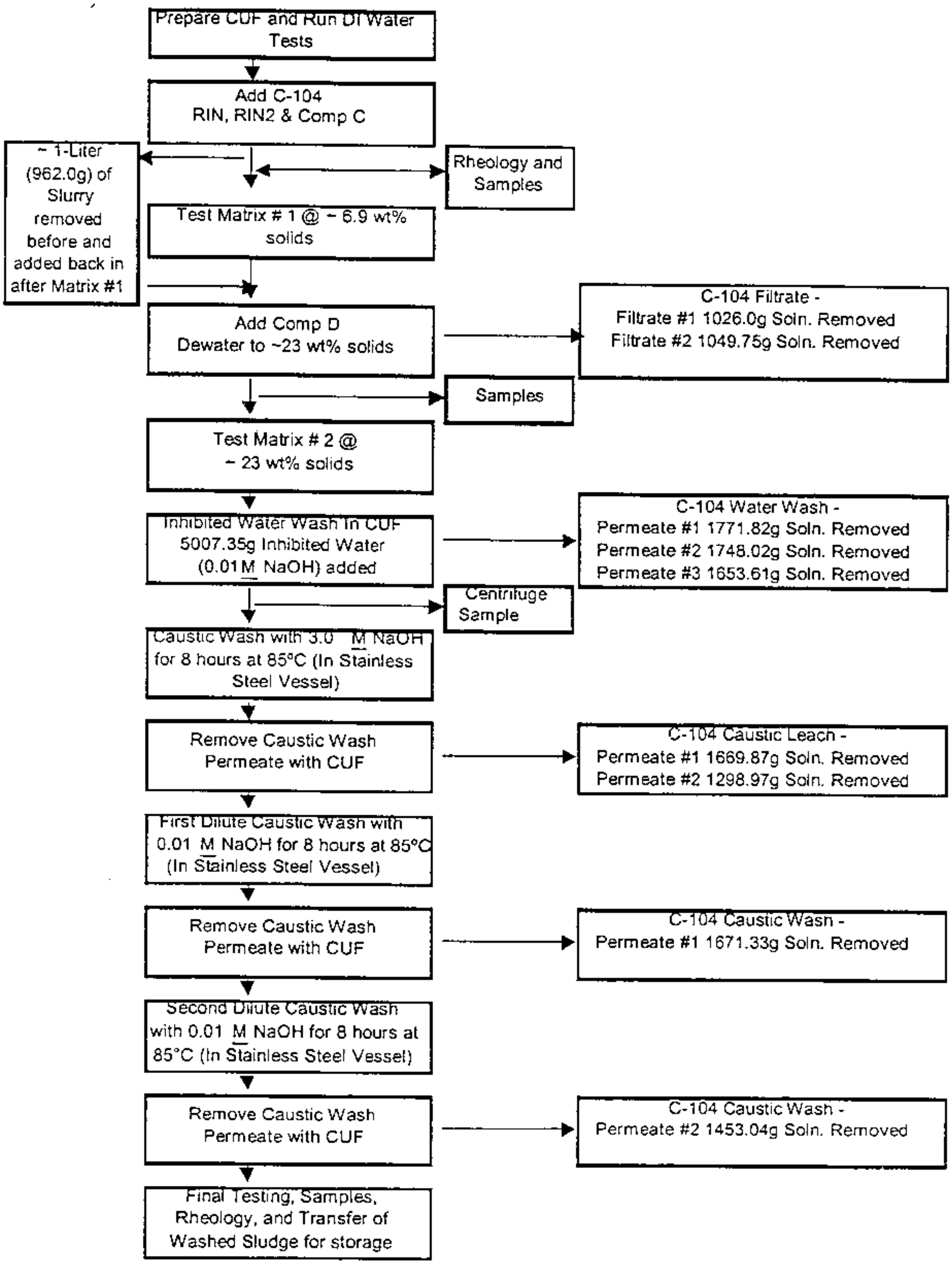

Figure 2.3. C-104 Crossflow Filtration Test Experimental Steps

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Appendix B

- ASR 6025 and 6025.01 and two Addendums
- ICP-AES Results
- ICP-MS Results
- Radiochemical Results and U KPA Results
- IC Results
- Hydroxide Results
- TOC/TIC Results
- Organic Acids Results
- Organic Phosphate Results
- Chelator Results
- Heat Capacity Results
- Particle Size Results
- Rheology Results

RECORD COPY

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 Michael W Urie
Print Name MICHAEL W URIE
Phone 376-9454 MSIN P7-22

PNNL Project #: 42040
Charge Code: F22616 + MULTIPLE
Date Required: 3-31-01

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) ✓
- **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 # 6011
  or Previous RPL ID # ___

Does the Waste Designation Documentation Indicate Presence of PCBs?
No ✓ **Yes** ___

Additional or Special Instructions Attached

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: IN HLRF AS OF 1-23-01
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) ___
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

Received By: ___
ASR Number: 6025
RPL Numbers: (01-00441)-(01-00442)

RPG/CMC Work Accepted By: M. Urie **Signature/Date:** MW Urie 1-23-01

LRG, CZS, JJW, MWU, RTS, MJS, RGS, JAC, EWH, M-LPT, OTF, LPD

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
	SEE AHAched SPECIAL				
	INSTRUCTIONS FOR PREPARING				
	PHASE SEPARATED SAMPLES FROM				
	SAMPLE "AN-102 DF A"				
01-00441	AN-102-SUP-DF	DILUTED AN-102	SEE AHachment	SEE SPECIAL	
		SUPERNATANT	TABLES 1 & 3	INSTRUCTIONS	
01-00442	AN-102-CS-DF	DILUTED AN-102	SEE AHachment	SEE SPECIAL	
		CENTRIFUGED SOLIDS	TABLES 2 & 4.	INSTRUCTIONS	
		(wet)			

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____; On Attachment ☑]

ASR # 6025

Page ________ of ________

Special Instructions: ASR 6025
Diluted Feed Analyses for AN-102 Supernatant Sample 01-00441 and Centrifuged Solids Sample 01-00442

AN-102 Supernatant Sample 01-00441 – LIMS Tests

Density/Solution	SAL	-- Use F22618
Wt% Solids/TDS	SAL	-- Use F22618
Digestion-128	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
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
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Sub-Sample	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC (w/o oxid.)	RAD	-- Use CMC K88421
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use F22620
Acids/Organic	ORG	-- Use F22620
Phosphates/Organic	ORG	-- Use F22620

AN-102 Centrifuged Solids Sample 01-00442 – LIMS Tests

Wt% Solids/Total (after phase separation)	SAL	-- Use F22618
Digestion-129	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORTG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
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
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Fusion-116	SAL	-- Use F22618
ICP/MS (Pt,Pd,Rh,Ru only)	ADV INORG	-- Use F22619
Leach/Water-103	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
Sub-Sample	SAL	-- Use F22618
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409

Phase Separation –Bottle "AN-102 DF A":

The AN-102 Diluted Feed composite sample **AN-102 DF A** containing approximately 150 mL of feed slurry is to be used for the characterization analyses for the AN-102 Diluted Feed. The content of **AN-102 DF A** are to be phase separated into composite supernatant sample **AN-102 SUP DF (01-00441)** and a composite wet centrifuged sample which is to remain in the original container. Following phase separation bottle **AN-102 DF A** shall be relabeled as **AN-102 CS DF (01-00442).**

General 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.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in duplicate.

Preparative or analysis QC is to include, preparation blank (**in duplicate for acid digestion and fusion preparations**), sample duplicate, matrix spike, matrix spike duplicate where appropriate, and an 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.

Phase Separation

For container **AN-102 DF A**:

1. Centrifuge container at approximately 1000 rpm for 1 hour. (with secondary containment)
2. Decant supernatant into tared 250-mL glass bottle labeled **AN-102 SUP DF 01-00xxx**, close immediately. Weigh and record gross weight.
3. Immediately close container **AN-102 DF A**, weigh, and record gross weight.
4. Leave wet centrifuged solids in orginal container and relabel container **AN-102 CS DF 01-004xx**, weigh, and record gross weight.
5. Determine total supernatant and wet centrifuge solids net weight.

Administrative Holdpoint:

To perform all analyses in duplicate, with full QC, required a minimum of 60 mL (approximately 75 g) of supernatant and 7 grams of wet centifuged solids. If this quantity of material is not available, notify M. Urie prior to proceeding with any preparations or distributions.

SAL Preparation/Analysis Preferred Order

During all sub-sampling activities, minimize the time that the supernatant and wet centrifuged solids containers are open to the hot cell atmosphere.

1. Perform a Wt% solids on the wet centrifuged solids sample and a TDS on the supernatant.
2. Determine density of supernatant using 10-mL volumetrics (note: this material can be used for other analyses following the density determination)
3. Sub-sample and distribution supernatant and solids samples requiring no preparation in SAL.
4. Acid Digestions (supernatant and solids)
5. Fusions for ICP/MS (solids only)
6. Water Leaches (solids only)
7. Organic preparation for dose reduction (supernatant only), per test plan provided by J. Campbell.

Laboratory Analyses

All analyses are to be conducted per approved PNNL procedures or test plans.

The attached Table 1 and Table 2 define the analytes of interest and the method to be used for reporting the analysis and Table 3 and Table 4 define the QC parameters (acceptance criteria) for each of the analyses. Please note the required QC criteria and the reporting units.

Results from each analysis shall be accompanied by a full narrative detailing the method used, the sample results, the QC results, and any unusual observations.

For total Cs and Rb in the supernatant sample, ICP-MS will be used for reporting the results.

For TOC and TIC, both the hot persulfate and furnace method shall be used. The furnace method is to report both the TOC and TC.

For organic analytes, the matrix spike, matrix spike duplicate, blank, and LCS shall be subjected to the same processing (e.g., IX for dose reduction) as the samples.

Table 1 Supernatant Analyses

Analyte	Minimum Reportable Quantity	Analysis Method
	µg/ml	
Al	7.5E+01	ICP-AES (AA may be used for Na, K, Cs, Rb or ICP-MS for Cs and Rb)
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Cs	1.5E+00	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Rb	1.0E+00	
S	3.0E-01	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TIC	1.5E+02	Silver catalyze persulfate oxidation method
TOC	1.5E+03	
TOC	1.5E-03	Furnace oxidation method
TIC	1.5E+02	
Hg[1]	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03	
SO_4	2.3E+03	
	µCi/ml (except as noted)	
^{237}Np	2.7E-02	ICP-MS
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	5.1E-02	
^{99}Tc	1.5E-03	
^{99}Tc	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{90}Sr	1.5E-01	Separations / Liquid Scintillation
^{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	1.0E-02	
^{137}Cs	9.0E+00	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
total and free OH	7.5E+04 µg/ml	Titration
Ammonia[1]	1.4E+02 µg/ml	ISE

Table 1 Supernatant Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Organic Analytes	**µg/ml**	
EDTA	1.5E+03	HPLC / LC-MS (if methods are available)
HEDTA	1.5E+03	
Oxalate	1.5E+03	
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
D2EHPA	1.5E+03	
NTA	1.5E+03	
IDA	1.5E+03	

[1] These analyses are only required for the as-received AN-102 composite sample

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
	µg/gm	
Ag	9.0E+02	ICP-AES
Al	3.3E+02	
Ba	6.0E+02	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
La	6.0E+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	
Pd	3.0E+02	
Rh	3.0E+02	
Ru	3.0E+02	
S	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Hg[1]	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
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)	

Table 2. HLW Solids Analyses

Analyte	Minimum Reportable Quantity	Analysis Method
CN[1]	3.0E+00	CN analysis
NH_3[1]	6.0E+01	ISE
As	3.0E+00	ICP-MS
B	3.0E+00	
Be	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Li	3.0E+01	
Mo	3.0E+01	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
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	
W	6.0E+00	
Y	6.0E+00	
^{127}I[1]	1.5E+00	
^{129}I[1]	3.0E+01	
^{233}U	6.0E+00	
^{234}U	6.0E+00	
^{235}U	6.0E+00	
^{236}U	6.0E+00	
^{238}U	6.0E+00	
^{237}Np	1.8E+00	
^{99}Tc	6.0E+00	
	μCi/gm	
^{3}H[1]	1.5E-02	Separations / Liquid Scintillation
^{14}C[1]	1.8E-03	Separations / Liquid Scintillation
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239+240}$Pu	6.0E+00	
^{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[1]	6.0E+00	
^{126}Sn[1]	6.0E-02	
^{134}Cs[1]	9.0E-01	
^{135}Cs[1]	5.3E-03	
^{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	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
^{241}Pu[1]	1.2E+00 µCi/gm	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Mineral types / structures		X-ray diffraction

[1] These analyses are only required for the as-received AN-102 composite sample.

Table 3. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Duplicate RSD[c]
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, 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%
Cs[f], Eu[f]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH-	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO_3^{-2}	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[f]	GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[g]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{125}Sb	GEA	to be obtained		
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A[g]	<15%
^{137}Cs	GEA	NP	N/A[g]	<15%
^{152}Eu[f]	GEA	NP	N/A[g]	<15%
^{154}Eu[f]	GEA	NP	N/A[g]	<15%
^{155}Eu[f]	GEA	NP	N/A[g]	<15%
^{231}Pa	ICP/MS	Developed by Laboratory		
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np[e]	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu[h]	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[e]	<15%
^{242}Cm	Separation/AEA	NP	N/A[e]	<15%
^{243}Am/Cm	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A[e]	<15%
Total Alpha[h]	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids	Gravimetric	80 - 120%	N/A	<21%

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 of the mean/mean) x 100

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

(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 1 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.

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

Table 4. Quality Control Parameters for Solids Analysis

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[a]	Spike % Recovery[b]	Duplicate RSD[c]
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, La, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs[e]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^{3}H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{59}Ni[h]	Separation/GEA	NP	N/A[g]	<15%
^{63}Ni[h]	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{60}Co[f]	GEA	NP	N/A[f]	<15%
^{90}Sr[f]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{90}Y[f]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{93}Zr[h][i]	beta-liq scintillation	NP	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m}Sn[h]	Separation/GEA	NP	N/A[g]	<15%
^{125}Sb[h]	GEA	to be obtained		
^{126}Sb[h][i]	Separation/GEA	NP	N/A[g]	<15%
^{126m}Sb[h][i]	Separation/GEA	NP	N/A[g]	<15%
^{126}Sn[h]	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or	NP	N/A	<15%
^{135}Cs[h]	ICP/MS	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{151}Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{152}Eu[f]	GEA	NP	N/A[g]	<15%
^{154}Eu[f]	GEA	NP	N/A[g]	<15%
^{155}Eu[f]	GEA	NP	N/A[g]	<15%
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}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%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[g]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%

Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids	Gravimetric	80 - 120%	N/A	<21%

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 of the mean/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.

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 ____
Phone ____ MSIN ____

PNNL Project #: 42365
Charge Code: W5791,2
Date Required: 6-15-01

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) ____
- **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 ____

Same as Original ASR

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** ____

MW Urie

Additional or Special Instructions ____

Send Report To ____ **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: ____
Delivered By (optional) ____
Time Delivered (optional) ____
Group ID (optional) ____
CMC Waste Sample? No ____ Yes ____
Cost Estimate, if requested: $ ____

Received By: 6025

ASR Number: 6025.01
RPL Numbers: 01-00441

RPG/CMC Work Accepted By: MW Urie **Signature/Date:** MW Urie 5/29/01

MWU, JAC

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
01-0991	AN-102 SUP-DF		Add Succinic Acid AND ED3A TO ORGANIC ANALYTE LIST TABLE 2 MRQs set at 1.5 E3 mg/L		

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___; On Attachment ___]

ASR # 6025.01

Page ______ of ______

Urie, Michael W

From: Johnson, Michael E
Sent: Wednesday, January 24, 2001 11:38 AM
To: Urie, Michael W; Johnson, Michael E
Cc: Kurath, Dean E
Subject: RE: AN-102 Dilute Feed analysis

Mike - If we don't have sufficient solids in the diluted AN-102 sample, I would like to reduce the analyte list, but perform all analyses with full QC. My order for conducting the requested analyses is as follows:

1) Wt % solids of centrifuged solids samples (after phase separation).
2) Acid Digestion Preparation:
   - ICP-AES analytes
   - GEA
   - Sr-90
3) Water Leach for Soluble Anions (F, Cl, NO2, NO3, PO4, SO4)
4) Direct TOC/TIC analyses (by two methods)

5) AEA for Pu, Am, Cm
6) Total U by Kin-Phos.
7) ICP-MS analytes (except platinum metals)
8) Gross Alpha
9) Fusion for Platinum Metals

Michael E. Johnson
Research and Technology Manager
CH2MHill Hanford Group

-----Original Message-----
From: Urie, Michael W [mailto:mike.urie@pnl.gov]
Sent: Tuesday, January 23, 2001 8:59 AM
To: Johnson, Michael E
Subject: AN-102 Dilute Feed analysis

Mike,

We are going to begin the Dilute Feed Characterization for AN-102. We probably will not have enough solids to perform all the analytical work identified in Table 2 of the Test Specification. We have the following options depending on the total solids available.

1) Reduce the analyte list (i.e., prioritize the analyses, but perform analyses with full QC)
2) Perform all analyses with reduced QC (i.e., duplicates and matrix spikes omitted - this will not allow us to demonstrate QC compliance with Table 4).
3) Combination of both is only minimal solids available.

Here is how the sample can be process and the analytes from each processing

A) Wt % solids of centrifuged solids samples (after phase separation). We need this to establish the actual solids composition (i.e., after adjusting for interstitial liquid by TDS)
B) Acid Digestion Preparation (experience has shown that everything goes into solution with an acid digest). From acid digest we can perform the following:

- ICP-AES analytes
- ICP-MS analytes (except platinum metals)
- GEA
- Gross Alpha
- AEA for Pu, Am, Cm
- Total U by Kin-Phos.
- Sr-90

C) Fusion for Platinum Metals
D) Water Leach for Soluble Anions (F, Cl, NO2, NO3, PO4, SO4)
E) Direct TOC/TIC analyses (by two methods)

The question is ---- If we insufficient sample for all analyses per the TS QC requirements, what analyses and/or QC can we eliminate? and in what order (e.g., QC reduction before analyte reduction)?

Mike

Michael W. Urie
Radiochemical Processing Group
RPL P7-22
Voice: (509) 376-9454
Fax: (509) 373-9675

Pacific Northwest National Laboratory
Battelle Boulevard
PO Box 999
Richland, Washington 99352
e-mail: mike.urie@pnl.gov

SAL ADDENDUM TO ASR 6025

January 29, 2001
By:

The SAL is to maintain material balance on the two AN-102 Diluted Feed containers **AN-102-SUP-DF (01-00441) and AN-102-CS-DF (01-00442)** by recording gross weight following each daily operation.

TO:
RTS, FVH, FILE

BLANK

ADDENDUM
ASR 6019, ASR 6025, ASR 6031

The SAL is instructed to use the attached procedures (PNL-ALO- 501 and PNL-ALO-504) to perform the following:

Total Weight Percent Solids
Total Dissolved Solids
Density (supernatant)

January 30, 2000
By MW Urie
MW Urie

Distributed to: RTS, FVH, ASR files

Use category of procedures is Reference Use.
K. Smith 1/30/01

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-501 Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges.

APPLICABILITY

This procedure will be used to measure the following physical properties of solutions, slurries, and sludges:

- Density or Specific Gravity
- pH of solutions
- Physical Appearance
- Settling Rate
- Shear Strength
- Shear Stress versus Shear Rate
- Vol% Centrifuged Solids
- Vol% Settled Solids
- Wt% Dissolved Solids
- Wt% Centrifuged Solids
- Wt% Total Oxides
- Wt% Total Solids
- Yield Stress

This procedure is applicable to samples that are homogenous and representative of the bulk sample with the exception of the sample used for shear strength. If not homogenous, the sample will be mixed and the procedure documented in or on the appropriate Laboratory Record Book (LRB), data record, or test instruction.

DEFINITIONS

Apparent Viscosity - Viscosity of a fluid defined as a ratio of the shear stress (at a particular shear rate) and the shear rate.

Density or Specific Gravity - Density is the mass per unit volume and specific gravity is the ratio of the density of the sample and a reference material.

Interstitial Solution - The solution contained within the original sludge sample.

Author RD Scheele Date 3/9/92	Project Mgr. TE Jones Date 3/9/92	QAD Representative GK Gerke Date 3/9/92
Technical Reviewer Tingey JM Tingey Date	Line Mgr. GH Bryan Date 3/10/92	Other Date

Procedure No.	Revision No.	Effective Date	Page
PNL-ALO-501	0	MAR 10 1992	1 of 11

Newtonian - A fluid with no yield stress and a viscosity that is independent of shear rate.

Non-Newtonian - A fluid whose viscosity varies with shear rate.

pH - pH is -log [H^+]. pH is applicable to aqueous solutions or slurries only.

Physical Appearance - The physical state (solid, liquid, sludge, slurry), color, and consistency.

Rheogram/Flow Curve - A plot of a fluid's shear response as a function of the shear rate. A plot of shear stress versus shear rate.

Sludge - Wet solids having little or no standing liquid, i.e. mud-like.

Slurry - A thin mixture of solids and solution.

Solution - A liquid phase containing dissolved material.

Solids Settling Rate - The rate at which the solids in a homogenized sample settle.

Vol% Settled Solids - The percentage of the volume of the sample that the settled solids occupy.

Vol% Centrifuged Solids - The percentage of the volume of the sample that the solids occupy after being centrifuged for a specific time and force.

Wt% Total Oxides - The percentage of the mass of the sample that remains after converting all non-volatile elements to oxides. Some volatile elements such as cesium might be lost in this process.

Wt% Centrifuged Solids - The percentage of the mass of the sample that the solids occupy after being centrifuged.

Wt% Dissolved Solids - The percentage of the mass of sample that is dissolved in the aqueous portion of the sample.

Wt% Total Solids - The percentage of the mass of the sample that remains after removing volatiles including free water by drying at 105 ± 5°C for 24 h.

Yield Stress - The minimum stress required to initiate fluid movement.

RESPONSIBLE STAFF

Analyst
Cognizant Scientist

PROCEDURE

1.0 General Requirements

The COGNIZANT SCIENTIST is responsible for preparing the test instructions to implement each step of this procedure and supplying the analyst with the test instructions. Because of the nature and diversity of samples that will be analyzed using this procedure, the cognizant scientist is responsible for assuring that the test instructions satisfy the requirements of this procedure and provide a unique sample or container identifier to insure sample traceability and control.

All measurements will be made at the temperature specified (± 5°C) in the test instructions or at ambient temperature if unspecified. Temperature will be measured using a calibrated thermocouple or RTD and digital thermometer or a mercury thermometer. If the test instructions specify a temperature for any measurement except, vol% centrifuged solids, wt% solids, wt% dissolved solids, or wt% total oxides; a constant temperature bath/circulator will be used.

The results of all measurements will be recorded in the appropriate LRB or on the test instructions or data sheet. A copy of the test instructions sheet will be entered into the LRB or the project records. If used, the LRB will be the responsibility of the COGNIZANT SCIENTIST. The ANALYST or other authorized personnel may also record information in the LRB, test instruction, or data sheet.

The calibrated instrumentation used will be recorded in the LRB, on the test instruction, or data sheet.

2.0 Procedure for Physical Appearance Measurement

This procedure will be used to observe some general physical properties for solutions, sludges, and slurries.

2.1 Equipment and Materials

None

2.2 Determination of Physical Appearance

Describe the sample in terms of its physical state (solution, slurry, or sludge), its color, its ability to hold its shape, and any other observable physical characteristics.

The ANALYST'S ability to observe these properties may be limited by work location. For example if the sample is located in the 325A shielded facility, the actual color and the observed color may be different due to the yellow leaded glass used in the shielded cell observation windows. The properties observed in this procedure are qualitative not quantitative.

3.0 Procedure for Settling Rate and Vol% Settled Solids Measurement

3.1 Equipment and Materials

- 15 or 50 mL Graduated Plastic Centrifuge Tube with Cap
- Analytical Balance, 2-place (minimum sensitivity)
- Plastic Rod or Plastic Spatula
- Centrifuge

3.2 Determination of Solids Settling Rate and Vol% Settled Solids

Label and weigh the centrifuge tube to be used. Determine the cm/mL for the centrifuge tube by measuring the distance between the 4 and 14 mL graduations for the 15 mL tube or between the 10 and 20 mL graduations for the 50 mL tube. Calculate cm/mL.

Transfer the sample to the preweighed, labeled centrifuge tube and seal. Reweigh and determine the sample mass by difference. Measure the volume of the total sample using the graduations on the centrifuge tube. Begin the settling rate measurements on a Monday, Tuesday, or Wednesday and no later than 1 h after the start of the working day. Record the start time and the times that all succeeding measurements are taken. Allow the solids in the sample to settle and measure the total sample and the solids volumes. The time between settling measurements is dependent upon the settling rate of the sample. The cognizant scientist will determine the time period between measurements to obtain appropriate settling rate data. The solids and total sample volumes should be measured at time increments no longer than every two hours during a the working day. Also, measure the total sample and solids volumes at the end of the working day and at the beginning of the next working day. Measure every two hours until no change is observed between three successive measurements or until 3 days have transpired.

Volumes will normally be measured to ± 0.2 mL when using the 15 mL centrifuge cone and to ± 1 mL when using the 50 mL centrifuge cone. The recommended sample volumes are ≥10 mL for the 15 mL tube and ≥30 mL for the 50 mL tube.

Present the settling rate as the solids height or vol% solids versus time. The solids height will be calculated by multiplying the solids volume by the height of 1 mL. Calculate Vol% Settled Solids by dividing the solids volume by the total volume and multiplying by 100%.

Some samples may stick to the side of the centrifuge tube. Centrifuge these samples briefly to drive them to the bottom of the tube to measure the sample volume. If the centrifuging causes separation of the solids and liquid, shake or blend with a plastic rod or spatula or as directed by the <u>COGNIZANT SCIENTIST</u> before beginning the settling procedure. Reweigh the tube and sample and calculate the new sample weight by difference.

The upper solids level may be difficult to determine because of different solids settling rates. The solids volume will be defined as the uppermost volume in which solids are present or the solids-solution interface which is the point where the sample becomes opaque.

4.0 Procedure for Density or Specific Gravity Measurement

This procedure will be used to measure the density or specific gravity of sludges, solutions, and slurries. This procedure will refer only to the measurement of density. If the user desires specific gravity, divide the measured density by the density of the reference material.

4.1 Equipment and Materials

- Graduated Plastic Centrifuge with Cap
- Centrifuge
- Analytical Balance, 2-place (minimum sensitivity)
- Volumetric Flask (Option for Solutions)

4.2 Determination of Density

Transfer the sample to a preweighed, labeled centrifuge tube and seal. Reweigh and calculate the sample mass by difference. As an alternative to a new sample, the sample from 4.3.2 may be used. The sealed centrifuge tube will be centrifuged for 1 h at ~1000 gravities. Measure the total volume of sample using the graduations on the centrifuge tube.

Calculate the density by dividing the sample mass by the sample volume. The sample mass will normally be measured to ± 0.02 g and the sample volume will normally be measured to ± 0.2 mL for the 15 mL tube or ± 1 mL for the 50 mL tube. The recommended sample volumes are 10 and 30 mL for the 15 and 50 mL centrifuge tubes, respectively.

With sludge samples the solids will often slope because of the centrifuging, the volume of the sample will be the average of the high and low volumes observed for the sample.

An alternative method of determining a solution's density will be to transfer a sample into a preweighed, labeled volumetric flask. Determine the sample mass by difference. This alternative method is preferable to the use of a graduated centrifuge tube because of increased accuracy for the volume measurement. The volume of a volumetric flask will normally be known to ± 0.04 mL.

5.0 Procedure for Determination of Vol%, Wt% and Densities of Centrifuged Solids and Supernate

This procedure will be used for determining the vol%, wt%, and the densities of centrifuged solids and centrifuged supernates for sludges and slurries.

5.1 Equipment and Materials

- Graduated Plastic Centrifuge Tube with Cap
- Clinical Centrifuge
- Analytical Balance, 2-place (minimum sensitivity)
- graduated cylinder
- Transfer Pipet and tips

5.2 Determination of Vol%, Wt%, and Densities of Centrifuged Solids and Supernate.

Transfer sample to a preweighed, labeled centrifuge tube and reweigh. Calculate the mass of the sample by difference. The sample and tube from Part4.2 may be used as the centrifuged sample. Centrifuge the sample for 1 h at maximum speed on the centrifuge. Record gravities apply. Measure the total sample and solids volume using the centrifuge tube graduations. The recorded solids volume will be the average of the high and low solids volume measurements. The supernate volume is the total sample volume less the solids volume. Volume is normally measured to ± 0.2 or ± 1 mL for the 15 and 50 mL centrifuge tubes, respectively.

The vol% centrifuged solids is the solids volume divided by the total volume multiplied by 100%. The vol% centrifuged supernate is 100% - vol% centrifuged solids.

To determine the wt% centrifuged solids, decant or transfer with a pipet the centrifuged supernate into a labeled, preweighed graduated cylinder. Measure the mass of the centrifuge tube and solids. Calculate the centrifuged solids mass by difference. Measure the volume of the supernate in the graduated cylinder and weigh the recovered centrifuged supernate andgraduated cylinder. Calculate the mass of the centrifuged supernate by difference.

The wt% centrifuged solids is the solids' mass divided by the total sample mass multiplied by 100%. The wt% centrifuged supernate is the mass of the supernate divided by the total sample mass multiplied by 100%. The centrifuged solids density is the mass of the solids divided by the solids volume and the centrifuged supernate density is the supernate's mass divided by its volume.

6.0 Procedure for Determination of Wt% Total Solids, Wt% dissolved Solids, and Wt% Total Oxides.

This procedure will be used for determining the wt% total solids, wt% dissolved solids, and wt% total oxides in solutions, sludges, and slurries.

6.1 Equipment and Materials

- Ceramic Crucibles
- Glass Vials
- Analytical Balance, 2-place
- Desiccator Containing Dry Indicating Desiccant
- Muffle Furnace (100 to >1000°C)
- Drying Oven

6.2 Determination of Wt% Total Solids, Wt% Dissolved Solids, and Wt% Total Oxides.

Transfer sample to a preweighed, labeled crucible or vial and weigh the sample in the vial or crucible. Use crucibles for samples for which total oxides will be determined and use vials for samples for which only wt% solids are to be determined. Calculate the sample mass by difference. Allow the sample to air dry at least overnight to remove free liquid and to prevent splattering in the oven.An alternate method for removing free liquid, such as using a heat lamp, may be designated by the COGNIZANT SCIENTIST. When the free liquid has evaporated, transfer the sample and container to a 105 ± 5°C drying oven or furnace. Hold at temperature for 24 h. Remove the dried samples and containers and place in the desiccator to cool to room temperature. It should take at least 1 h to cool the sample. Reweigh the dried sample and container. Determine the residual sample weight by difference. The mass of the sample will normally be measured to ± 0.02 g.

The wt% total solids is the residual mass divided by the initial sample mass multiplied by 100%. For solutions the wt% dissolved solids is the residual mass divided by the initial sample mass multiplied by 100%. For slurries or sludges with interstitial solutions that are recoverable using centrifugation, the wt% dissolved solids is the weight fraction dissolved solids in the centrifuged supernate times the weight fraction centrifuged supernate in the sludge/slurry determined using the procedure found in Section5 times 100%.

Place the sample and crucible from the wt% total solids procedure into the furnace. Do not perform this procedure with the glass vials. Heat the furnace to >1000°C and hold for 30 min between 1000 and 1050°C. Turnoff the furnace and cool to 150°C. Transfer the crucibles using tongs to the desiccator and cool to room temperature. It should take 1 to 2 h to cool the sample. Once cool weigh the sample and crucible. Determine the residual oxide mass by difference. Consider that volatile elements may also be lost.

The wt% total oxide is the residual oxide mass divided by the initial sample mass multiplied by 100%.

7.0 Procedure for Determining pH

This procedure will be used to determine the pH of a solution or slurry.

7.1 Equipment and Materials

- pH Meter and Electrode
- pH 4, 7 and 10 Buffers
- Plastic beakers or vials

7.2 Determination of pH

Standard Laboratory procedure will be used to measure the pH of solutions or slurries. Prior to use daily calibration of the pH meter and electrode will be performed using a two or three standard method. The temperature of the standards and samples will be taken into account. The electrode calibration will be done using pH buffers that are traceable to the manufacturers lot number. The expiration date for the buffers used in the check must not have lapsed. The electrode will be prepared for use and stored in accordance with the manufacturer's directions.

Sufficient sample will be added to a beaker or vial to cover the sensing portion of the electrode. Remove the probe from the storage solution, rinse with deionized (DI) water, and blot dry with a tissue. Insert the probe into the sample. Once the reading has stabilized, record the pH value and rinse the electrode with DI water. After the pH for the last sample has been determined, rinse and blot dry the electrode, and return it to its storage fluid.

The pH meter must have an accuracy of ±0.02 pH units. Data will normally be reported to the nearest 0.1 pH unit.

8.0 Procedure for Determination of Shear Stress vs Shear Rate and Yield Stress

This procedure will be used for determining the shear stress versus shear rate, yield stress, and shear strength for solutions, sludges, and slurries.

8.1 Equipment and Materials

- Viscometer
- Certified Newtonian Viscosity Standards
- Constant Temperature Bath/Circulator

8.2 Performance Requirements

Performance checks of the viscometer will be performed monthly or within approximately 30 days of measurement using certified Newtonian viscosity standards traceable to the manufacturer's lot number. The viscometer used must have a demonstrated accuracy of ± 15% at apparent viscosity measurements less than 10 CP and ± 10% at apparent viscosity measurements greater than 10 CP.

The data will be generated following the instructions provided in the instrument's Instruction Manual. The data will be in the form of a rheogram or flow curve, which is a plot of shear stress as a function of shear rate.

8.3 Measurement of Shear Stress vs Shear Rate and Yield Stress

Select and install the Sensor. For measurements at elevated temperatures, adjust the temperature setting for the constant temperature circulator to the desired level. Install the temperature control shroud. Transfer the sample by weight into the viscometer beaker. If the MV I Rotor is used transfer 40 mL. If the MV II Rotor is used transfer 55 mL. The weight transferred equals the density times the volume. After the coolant in the shroud reaches the desired temperature, stir the sample, and install the sample and beaker in the viscometer. Allow the sample to remain in position for 5 min to bring the sample to temperature unless instructed otherwise by the <u>cognizant scientist</u>. Obtain a rheogram. Adjust the temperature controller to the next temperature. Once the temperature of the coolant in the shroud reaches the desired level, allow the sample to equilibrate for 5 min before obtaining a rheogram. Repeat as necessary to obtain rheograms at the temperatures of interest.

It is important that the time between installation of the sample and beaker in the viscometer and the acquisition of the rheogram be as short as possible to minimize the effects of any solids settling.

To calculate the apparent viscosity, measure the shear stress (from the y axis) of the sample at a selected shear rate (from the x axis) and divide the shear stress by shear rate.

The fluid yield stress is obtained from the rheogram. A rheogram for a material with a yield stress will have two portions to it. The first appears as a nearly vertical line beginning at the origin and running up the ordinate. This line forms an angle, ß, with the ordinate. This portion of the rheogram is recording the behavior of the material as it acts like a solid or gel. When sufficient force is transmitted to the material to break the gel or make it yield, the rheogram will angle sharply to the right, and from then on the behavior of the material as a fluid will be recorded. This sharp angle that is recorded as the material becomes fluid is the yield point. The height of this break measured in Pascals along the ordinate is the value of the material's yield stress.

Some samples exhibit a creeping transition from solid to liquid behavior. this creep is recorded as a rounded yield point that is more difficult to identify. In cases where creep is exhibited, the yield point is defined as the intersection of a tangent drawn along the solid portion of the rheogram with a tangent drawn along the fluid portion of the rheogram.

Yield stress data is normally reported with a degree of precision of ± C.1 Pa.

9.0 Procedure for Determination of Shear Strength

This procedure will be used to measure the shear strength of sludge samples. Shear strength is a semi-quantitative/qualitative/relative measure of the force that is required to move the material. No calibration standards are currently available to calibrate the shear vane.

9.1 Equipment and Materials

- Viscometer
- Shear Vane

9.2 Measurement of Shear Strength

Install shear vane into the viscometer. Position sample beneath the shear vane. Raise the sample slowly using the lab jack, centering the shear vane in the sample. Submerge the vane with at least 3 mm of sample above and below the vane. Based on the appearance of the sample, the COGNIZANT SCIENTIST or ANALYST will select the stress scale and the shear rate. Obtain the rheogram. Note: Shear Strength is dependent on sample history. The sample should be disturbed as little as possible before measurement or should be allowed to age for at least 2 days before the shear strength is determined.

The size and geometry of the sample is important when measuring shear strength. Try to select a sample geometry with a diameter of at least 4 shear vane diameters and a depth such that there is at least 3 mm of sample above and below the shear vane. The shear vane should be located at least two shear vane diameters away from the container walls.

The rheogram should show a peak at the beginning, level off, and finally drop off. The shear strength is calculated by taking the peak torque at a fraction of the full-scale reading on the chart, multiplying by %τ, multiplying by the full scale τ, and dividing by the surface area of the shear vane fins. The shear strength is reported in dynes/cm^2.

10.0 Quality Assurance

Work performed using this technical procedure will meet Impact Level II requirements as described in PNL-MA-70. All data generated using this procedure will be recorded on test instructions or directly in a LRB. The completed test instructions will later be entered into the appropriate LRB.

Changes to the test instructions can be made only by Cognizant Scientist and will be documented by crossing out the original information with a single line, and recording, initialling, and dating the changes. Major changes will be made by revision of the test instruction.

PNL Operating Procedure

Org. Code: D9T41
Procedure No.: PNNL-ALO-504
Rev. No.: 2

Title: PERCENT SOLIDS DETERMINATION OF SOILS/SLUDGES/SOLIDS

Work Location: 325 Building -- General

Page 1 of 6

Author: RT Steele

Effective Date: 07/14/97
Supersedes Date: 09/26/90

Identified Hazards:
☒ Radiological
☐ Hazardous Materials
☒ Physical Hazards
☐ Hazardous Environment
☐ Other:

Was a Procedure Worksheet Completed?
☐ Yes ☒ No

Are One-Time Modifications Allowed to this Procedure?
☒ Yes ☐ No

Signatures:

Author ____________ RT Steele (Signature) 7/21/97 (Date)

Technical Reviewer ____________ MW Urie (Signature) 7/21/97 (Date)

Approval:

Line Manager ____________ KJ Kuhl-Klinger (Signature) 8/22/97 (Date)

Concurrences, as appropriate:

Building Manager ____________ (Signature) 8-8-97 (Date)

Health & Safety ____________ (Signature) 7/28/97 (Date)

Quality Programs ____________ TL Almeida (Signature) 8/18/97 (Date)

Radiological Control ____________ (Signature) 8/13/97 (Date)

____________ (Other) (Signature) (Date)

PNL Operating Procedure	Org. Code: D9T41 Procedure No.: PNNL-ALO-504 Rev. No.: 2
Title: PERCENT SOLIDS DETERMINATION OF SOILS/SLUDGES/SOLIDS	

PURPOSE/SCOPE

This procedure provides a uniform method for determining the weight percent solids fraction of samples. The methodology is consistent with the Contract Laboratory Program (ILM02.1) percent solids determination method.

APPLICABILITY

This procedure applies to solid, soil, sludge, slurry, and mixed-phase aqueous samples that are stable after drying to 105 °C.

HAZARD ASSESSMENT/HAZARD MITIGATION SUMMARY

Typically, the sample materials analyzed are chemically hazardous (e.g., corrosive, carcinogenic, toxic, etc.) and/or radioactive. When handling radioactive materials, the principles of ALARA shall be applied to minimize exposure and precautions taken to mitigate the spread of radioactive contamination. When hazardous materials are handled, laboratory staff are alerted to the potential hazards via the PNNL Sample Information Check List. Personnel protective clothing shall be specified in the applicable Chemical Safety Protocol or Radiological Work Permit (RWP).

Extreme care must be taken when removing the hot sample containers from the drying oven to avoid burns or sample spills. The use of tongs is mandatory when removing containers from the drying oven.

EMERGENCY RESPONSE

Emergency response actions can be initiated by 1) the activation of an emergency signal such as a fire gong, evacuation siren, or radiation detection/monitoring device alarms, 2) loss of building electrical power, and 3) from an unforeseen accident during sample handling or analysis activities. In all cases, proceed in accordance with the Building Emergency Procedure. Emergency response actions shall be reported to the Single Point of Contact at 375-2400. Notify Line Management immediately of emergency actions taken.

PREREQUISITES

Laboratory staff shall be trained to this procedure, prior to conducting any of the activities described herein.

Based on the samples handled (e.g., radioactive or non-radioactive, hazardous or non-hazardous), one or more of the following prerequisite training classes may be required:

- Laboratory Standard Hazard Communication
- Radiological Worker I or Radiological Worker II
- Laboratory Fume Hood Safety (including Radiological Fume Hood Safety)
- Safe Operating Procedure (SOP) "Routine Management, Storage, and Disposal of Hazardous, Low-Level Radioactive, or Mixed Waste", GEN-325-WM1

PRECAUTIONS AND LIMITATIONS

This procedure is applicable to determining weight percent solids on drying, or percent weight loss on drying (i.e., 100 - weight percent solids). In general, this procedure is not applicable to the determination of water content, since any component volatile at 105 °C can not be differentiated from water and all water may not be released at a temperature of 105 °C. However, with Cognizant Scientist approval, this procedure may be used to evaluate the absorbed moisture content (moisture loss on drying) for applicable sample of known chemical composition.

QUALITY CONTROL INSTRUCTIONS

Sample weights stated in this procedure may be varied by the Cognizant Scientist. This option is especially useful when the quantity of sample is limited or ALARA principles apply. Use of a sample data sheet or Analytical Chemistry Laboratory Record Book (ACL RB) is required for recording sample data and analyst's comments. A weight percent solids determination on a sample duplicate shall be performed with each analytical batch of samples. An analytical batch may range in size from 1 to 20 analytical samples. Calibration data for all Measuring and Test Equipment used in this procedure (i.e., balance and oven thermometer/thermocouple) must documented and retrievable.

EQUIPMENT AND REAGENTS

- Analytical Balance capable of weighing to at least 0.001 g.
- Drying Oven capable of maintaining a temperature of 105 ± 2 °C.
- Weighing Containers (e.g., porcelain crucibles, glass beakers)
- Lids for Weighing Containers (optional)
- Desiccator

WORK INSTRUCTIONS

1.0 PERCENT SOLIDS DETERMINATION

A sample is weighed into tared containers, dried at 105 °C for 12 to 24 hours, and reweighed to determine the solids fraction of the original sample. Weight data is recorded on a sample data sheet (or ACL RB).

1.1 Thoroughly mix the sample to be analyzed; mixing can be either manual or mechanical.

1.2 Label and weigh the weighing container to be used for the determination. Record this container tare weight (CW_t) to nearest 0.001 gram on a sample data sheet (See Exhibit 1 for example). Note: Be sure to include one duplicate analysis per each analytical batch.

1.3 Add sample to be analyzed to the weighing container, and weigh. Record this wet gross weight (GW_w) to the nearest 0.001 grams on the data sheet.

> Typical sample size is 5 to 10 grams. However, sample availability may limit the quantity of sample to be analyzed to less than 5 grams. When less than 2 grams of sample are available, consult the Cognizant Scientist prior to proceeding with the analysis.

1.4 Repeat Steps 1.1 through 1.4 until all samples being processed in the analytical batch have been weighed, then continue with Step 1.5.

1.5 Place the samples (i.e., entire analytical batch) into a drying oven maintained at 105 ± 2°C. Record the date, time, and oven temperature at the time the samples are placed into the oven. Note: When used, weighing container lids must be tipped to allow evaporating moisture to escape.

1.6 Maintain the samples at 105 ± 2 °C for 12 to 24 hours.

1.7 Remove the dried samples from the oven, and record date, time, and temperature on the data sheet. Immediately transfer the samples to a desiccator, close the desiccator, and allow samples to cool.

1.8 Once cool, remove a sample from the desiccator (close desiccator) and weigh immediately. Record this dry gross weight (GW_d) to the nearest 0.001 gram on the data sheet. Repeat until all samples have been weighed.

When obtaining the dry gross weight, an unstable balance reading is indicative of:

- Attempting to obtain the weight before sample has reached ambient temperature. This condition results in a trendless, unstable balance reading. If this condition occurs, return the sample to the desiccator for 20 minutes, then repeat Step 1.8. Repeat this process until two consecutive weight readings are within 0.003 gram.
- The sample being hygroscopic. This condition results in a steadily increasing balance reading. If this condition occurs, return the sample to the drying oven, dry for 1 hour, cool, and reweigh immediately. Repeat this process until two consecutive weight readings are within 0.003 gram. If the sample absorbs moisture too rapidly to obtain a stable reading, consult Cognizant Scientist.

2.0 CALCULATIONS

$$\text{Wt\% Solids} = \frac{(GW_d - CW_t)}{(GW_w - CW_t)} * 100$$

Where,

$Wt\%$ = Weight Percent Solids
GW_d = Dry Gross Weight (Step 1.8)
GW_w = Wet Gross Weight (Step 1.3)
CW_t = Container Tare Weight (Step 1.2)

SOURCE REQUIREMENTS

Quality Assurance Plan for Activities Conducted by the Analytical Chemistry Laboratory (ACL), MCS-033, (Section 8)

Quality Assurance, PNNL-MA-70, 1997, (PAP-70-1201).

Hanford Analytical Services Quality Assurance Requirement Documents, Volume 4, (Section 4.0).

USEPA CLP SOW for Inorganic Analyses, Document Number ILM02.0 (1990), ILM02.1 (1991). Exhibit D, Part F.

EXHIBITS/ATTACHMENTS

Exhibit 1: "Wt% Solids Data Sheet"

EXHIBIT 1 - WT% SOLIDS DATA SHEET

Pacific Northwest National Laboratory
Analytical Chemistry Laboratory

Page 1 of 1

WT% SOLIDS DATA SHEET

CLIENT: ____________ WORK PACKAGE: ____________ ASR/LOI/TI: ____________

QA PLAN: ____________ IMPACT LEVEL: ____________ PROCEDURE NO: PNNL-ALO-504

SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	CONTAINER TARE WEIGHT (CW_t)	GROSS WET WEIGHT (GW_w) [Wet Sample + Container]	GROSS DRY WEIGHT (GW_d) [Dry Sample + Container]	WEIGHT % SOLIDS

EXAMPLE

Alternate forms may be used, provided that the content and method of processing is consistent with this procedure.

$$\text{WT\% SOLIDS} = \frac{(GW_d - CW_t) * 100}{(GW_w - CW_t)}$$

DATE/TIME IN: ____________ OVEN TEMPERATURE: ________ °C

DATE/TIME OUT: ____________ OVEN TEMPERATURE: ________ °C

THERMOCOUPLE ID: ____________ M&TE ID: ____________

Analyst: ____________ Date: ____________ Reviewer: ____________ Date: ____________

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Project / WP#:	42365 ~~G42040~~ / F22626
ASR#:	6019 & 6025
Client:	M. Urie
Total Samples:	4

	From	To
RPL#:	01-00429, -00430	01-00441, -00442
Client ID:	AN-102-SUP-AR, -CS-AR	AN-102-SUP-DF, -CS-DF

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 02-15-01 (A0645)

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 7-6-01
Reviewed by

MW Urie 7-12-01
Concur

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Two samples, "AN-102-SUP-AR" and "AN-102-SUP-DF" (RPL ID# 01-00429 and 01-00441), were processed by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-128 acid digestion of as-received and diluted-feed supernatant liquid. Approximately 1 mL of material (density 1.437 g/mL (supernatant liquid, as-received) and 1.159 g/mL (supernatant liquid, diluted-feed), prepared in duplicate; duplicate digestion process blanks (reagents only) and a blank-spike were processed and analyzed by ICPAES. Each sample aliquot was transferred to digestion vessel using a 1 mL pipet and weighed. Sample volumes were estimated by dividing sample aliquot weight by sample density. (Sample density data for wet centrifuged solids and supernatant liquids were determined from earlier sample measurements.) Following acid digestion each sample was diluted to approximately 25 mL and weighed. Final volume was estimated by dividing the final weight of prepared solution by its density. Density of solution was determined by weighing a 1 mL aliquot. ICPAES data is reported as µg/mL.

Also, two samples, "AN-102-CS-AR" and "AN-102-CS-DF" (RPL ID# 01-00430 and 01-00442), were processed by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-129 acid digestion of as-received (wet solids) and diluted-feed (dry solids) centrifuged material. Approximately 0.5 to 0.8 g of wet solids material (AN-102-CS-AR) was prepared in duplicate, duplicate digestion process blanks (reagents only), matrix-spikes (each sample), and blank-spike were processed and analyzed by ICPAES. After acid digestion each sample was diluted to a final volume approximately 25 mL and weighed. Approximately 0.22 to 0.29 g of dry solids material (AN-102-CS-DF) was prepared but reported as wet sample (µg/g-wet) based upon 35.1 wt% solids of original wet material. Sample aliquot and final volumes were weighed and volumes determined from estimated density measurements as noted above.

ICPAES results have been corrected for process dilution resulting from acid digestion of the supernatant and centrifuged solids material and analytical dilution during ICPAES analysis session. Wet solids and supernatant sample material was transferred to digestion vessels using a 1 mL pipette and weighed. Final sample volume was determined by dividing weight of final sample volume by estimated density. Density of final liquid was estimated by weighing a 1 mL aliquot of final diluted sample.

Analytes of interest and MRQ's listed in Table 1. Supernatant Analyses of Special Instructions include the following: Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Na, Ni, P, Pb, and U. Analytes of interest and MRQ's listed in Table 2. HLW Solids Analysis include all of the analytes listed in Table 1, (except K and U) plus Ag, Bi, Cu, Mn, Nd, Pd, Rh, Ru, Si, Sr, Ti, Zn, and Zr. EQL limits, adjusted for sample preparation and analytical dilution, were generally below MRQ's except for potassium. MRQ in Table 1 for potassium is 75 µg/mL. However, potassium in the supernatant samples measured about 18,800 µg/mL, well above MRQ. The following analytes required for HLW solids (centrifuged solids) did not meet MRQ specifications: Ca, Cd, Cu, La, Na, Pd, Rh, and Ru. Data report lists EQL's for samples and MRQ specifications.

See attached ICPAES Data Report for final results.

5/30/2001

Quality control check-standard results met tolerance requirements for all analytes of interest reported except where noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan.

Five fold serial dilution:

All analytes of interest were within tolerance limit of 10% after correcting for dilution except for the following: Calcium varied by about 25% to 50% in supernatant duplicate and matrix-spike samples (RPL# 01-00429) and by about 150% in the centrifuged solids sample (RPL# 01-00430). Barium differed by about 11% and sodium differed by about 20% between 10-fold and 2-fold dilution of centrifuged solids in matrix-spike sample (RPL# 01-00442-MS1).

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 15% RPD for all analytes of interest greater than EQL except for nickel (40%) and zirconium (51%) in centrifuge solids (DF) sample RPL#01-00442. The original and duplicate sample was not within specification limit of ≤ 3% RPD for sodium greater than EQL except in centrifuge solids sample RPL#01-00442 (1% RPD). Sodium RPD generally varied from 3.8% to 7.8%.

Post-Spiked Samples (Group A):

All analytes of interest were recovered within tolerance of 75% to 125% post-spike recovery.

Post-Spiked Samples (Group B):

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

All analytes of interest were recovered within tolerance of 80% to 120% except silver. Silver recovery was low (about 22%). Low recovery may be due to the amount of hydrochloric acid used to process the sample (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples. Potassium recovery was also low (72% to 75%) for both sample preparation procedures (PNL-ALO-128 and –129). No apparent reason for the discrepancy in known.

Matrix Spiked Sample:

All analytes of interest were recovered within tolerance of 75% to 125% except low recovery of silver (23%), and potassium (65%) in matrix-spiked supernatant sample RPL#01-00429. Recovery of silver (26% and 35%), calcium (49%), copper (129%), magnesium (131%), nickel (163%)

and zirconium (54%) exceeded tolerance limits in the matrix-spiked centrifuged solids. Low recovery for silver is likely due to the amount of hydrochloric acid used to process samples (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples. Low or over recovery of the other analytes may be due to non-homogeneity of the centrifuged solids (RPL#01-00442).

Quality Control Check Standards:

Concentration of all analytes of interest is within tolerance limit of ± 10% accuracy in the check standards QC_MCVA, QC_MCVB, and QC_SSTMCV with the following exceptions. Sodium (11% to 14%), silicon (10% to 12%), and potassium (11% to 17%) measurements of QC_MCVA exceed tolerance limit of 10%. Potassium (15%, 18%) and sodium (15% & 16%) was also high in check standard QC_SSTMCV. High recovery for these analytes may be due to sample carry-over and require longer clean-out time.

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 one of two measurements of potassium. One of the two potassium measurements was 13% high. All other analytes measured were within tolerance.

Process Blank:

Concentration of all analytes of interest is within tolerance limit of ≤EQL or less than ≤5% of the sample.

Laboratory Control Standard (LCS):

A blank-spike containing all analytes of interest was prepared and analyzed instead of an LCS (See "Blank Spike" above for details).

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=		40.3		38.0		84.5		100.2		63.6		62.5
		RPL/LAB #=		01-00430-PB1		01-00430-PB2		01-0430 @2		01-0430-DUP @2		01-00442 @2		01-0442-DUP @2
		Client ID=		Proc.Blank-1 (ALO-129)		Proc.Blank-2 (ALO-129)		AN-102-CS-AR (ALO-129)		AN-102-CS-AR (dup) (ALO-129)		AN-102-CS-DF (ALO-129)		AN-102-CS-DF (dup) (ALO-129)
Det. Limit	MRQ$^{(1)}$	Run Date=	EQL	2/15/2001	EQL	2/15/2001	EQL	2/15/2001	EQL	2/15/2001	EQL	2/15/2001	EQL	2/15/2001
(ug/mL)	(ug/g)	(Analyte)	(ug/g)	ug/g-wet	(ug/g)	ug/g-wet	(ug/g)	ug/g-wet	(ug/g)	ug/g-wet	(ug/g)	ug/g-wet	(ug/g)	ug/g-wet
0.025	900	Ag	10.1	--	9.5	--	21.1	--	25.1	--	15.9	--	15.6	--
0.060	330	Al	24	--	23	[2.6]	51	28,000	60	29,000	38	58,100	37	53,100
0.250		As	101	--	95	--	211	--	251	--	159	[17]	156	--
0.050		B	20	50.1	19	60.2	42	89.8	50	97.1	32	59.8	31	54.3
0.010	600.0	Ba	4.0	--	3.8	--	8.5	26.9	10.0	28.0	6.4	78.6	6.2	78.6
0.010		Be	4.0	--	3.8	--	8.5	--	10.0	--	6.4	7.12	6.2	7.03
0.100	6000	Bi	40	--	38	--	85	--	100	--	64	[12]	62	[11]
0.250	180	Ca	101	--	95	--	211	434	251	407	159	441	156	405
0.015	11.0	Cd	6	--	6	--	13	37.4	15	38.5	10	20.0	9	19.6
0.200		Ce	81	--	76	--	169	[17]	200	--	127	[72]	125	[70]
0.050		Co	20	--	19	--	42	--	50	--	32	--	31	[3.1]
0.020	120	Cr	8	--	8	--	17	1,800	20	1,850	13	4,550	12	4,580
0.025	18	Cu	10	--	9	--	21	[19]	25	[19]	16	[11]	16	[13]
0.050		Dy	20	--	19	--	42	--	50	--	32	--	31	--
0.100		Eu	40	--	38	--	85	--	100	--	64	--	62	--
0.025	140	Fe	10	[4.9]	9	[1.2]	21	1,150	25	1,180	16	3,260	16	3,640
2.000		K	806	--	760	--	1690	[1,100]	2005	[1,100]	1271	[580]	1249	[550]
0.050	60	La	20	--	19	--	42	[32]	50	[32]	32	106	31	105
0.030		Li	12	--	11	--	25	--	30	--	19	[11]	19	[11]
0.100	450	Mg	40	--	38	--	85	[11]	100	--	64	[55]	62	[53]
0.050	300	Mn	20	--	19	--	42	254	50	261	32	764	31	765
0.050		Mo	20	--	19	--	42	[33]	50	[33]	32	[15]	31	[16]
0.150	150	Na	60	73.1	57	86.5	127	160,000	150	173,000	95	58,700	94	59,300
0.100	600	Nd	40	--	38	--	85	[65]	100	[64]	64	181	62	179
0.030	160	Ni	12	--	11	--	25	254	30	261	19	105	19	157
0.100	600	P	40	--	38	--	85	1,390	100	1,420	64	1,060	62	1,060
0.100	600	Pb	40	--	38	40.3	85	245	100	244	64	412	62	400
0.750	300	Pd	302	--	285	--	634	--	752	--	477	--	469	--
0.300	300	Rh	121	--	114	--	254	--	301	--	191	--	187	--
1.100	300	Ru	443	--	418	--	930	--	1103	--	699	--	687	--
0.500		Sb	201	--	190	--	423	--	501	--	318	--	312	--
0.250		Se	101	--	95	--	211	--	251	--	159	--	156	--
0.500	3000	Si	201	[120]	190	[130]	423	[190]	501	[210]	318	3,630	312	3,630
1.500		Sn	604	--	570	--	1268	--	1504	--	953	--	937	--
0.015	300	Sr	6	--	6	--	13	[6.1]	15	[6.2]	10	23.8	9	23.8
1.500		Te	604	--	570	--	1268	--	1504	--	953	--	937	--
1.000		Th	403	--	380	--	845	--	1002	--	636	[160]	625	[160]
0.025	150	Ti	10	--	9	--	21	--	25	--	16	[3.2]	16	[3.3]
0.500		Tl	201	--	190	--	423	--	501	--	318	--	312	--
2.000		U	806	--	760	--	1690	--	2005	--	1271	[330]	1249	[310]
0.050		V	20	--	19	--	42	--	50	--	32	--	31	--
2.000		W	806	--	760	--	1690	--	2005	--	1271	--	1249	--
0.050		Y	20	--	19	--	42	[9.4]	50	[9.5]	32	[26]	31	[26]
0.050	6	Zn	20	--	19	--	42	[21]	50	[23]	32	79.5	31	77.9
0.050	600	Zr	20	--	19	--	42	46.3	50	[47]	32	58.2	31	97.6

(1) MRQ's are for un-dissolved solids (Table 2. ILW Solids Analyses).
Measurement results are for un-dissolved solids plus interstitial liquids.

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

		Multiplier=		26.2		26.3		136.0		52.3		50.4		49.9
		RPL/LAB #=		01-00429-PB1		01-00429-PB2		01-00429 @5		01-00429-DUP @2		01-00441 @2		01-00441-DUP @2
		Client ID=		Proc.Blank-1 (ALO-128)		Proc.Blank-2 (ALO-128)		AN-102-SUP-AR (ALO-128)		AN-102-SUP-AR-dup (ALO-128)		AN-102-SUP-DF (ALO-128)		AN-102-SUP-DF-DUP (ALO-128)
Det. Limit (ug/mL)	MRQ (ug/mL)	Run Date= (Analyte)	EQL (ug/mL)	2/15/2001 (ug/mL)	EQL (ug/mL)	2/15/2001 (ug/mL)	EQL (ug/mL)	2/15/2001 (ug/mL)	EQL (ug/mL)	2/15/2001 (ug/mL)	EQL (ug/mL)	2/15/2001 (ug/mL)	EQL (ug/mL)	2/15/2001 (ug/mL)
0.025		Ag	6.6	--	6.6	--	34.0	--	13.1	--	12.6	--	12.5	--
0.060	75	Al	16	[5.0]	16	[4.9]	82	12,100	31	12,400	30	5,390	30	5,280
0.250		As	66	--	66	--	340	--	131	--	126	--	125	--
0.050		B	13	42.9	13	43.8	68	77.8	26	81.8	25	51.5	25	48.0
0.010	2.3	Ba	2.6	--	2.6	--	13.6	--	5.2	--	5.0	--	5.0	--
0.010		Be	2.6	--	2.6	--	13.6	--	5.2	--	5.0	--	5.0	--
0.100		Bi	26	--	26	--	136	--	52	--	50	--	50	--
0.250	150	Ca	66	--	66	--	340	465	131	513	126	169	125	137
0.015	7.5	Cd	4	--	4	--	20	61.2	7.9	62.3	8	16.9	7	16.4
0.200		Ce	52	--	53	--	272	--	105	--	101	--	100	--
0.050		Co	13	--	13	--	68	--	26	[3.9]	25	--	25	--
0.020	15	Cr	5	--	5	--	27	216	10	213	10	69.9	10	68.2
0.025		Cu	7	--	7	--	34	[23]	13	23.9	13	[6.8]	12	[6.7]
0.050		Dy	13	--	13	--	68	--	26	--	25	--	25	--
0.100		Eu	26	--	26	--	136	--	52	--	50	--	50	--
0.025	150	Fe	7	[1.6]	7	--	34	37.6	13	37.3	13	[6.7]	12	[6.4]
2.000	75	K	525	--	525	--	2720	[2,000]	1047	1,960	1009	[540]	998	[550]
0.050	35	La	13	--	13	--	68	[16]	26	[15]	25	[3.2]	25	[3.3]
0.030		Li	8	--	8	--	41	--	16	--	15	--	15	[1.6]
0.100	300	Mg	26	--	26	--	136	--	52	--	50	--	50	--
0.050		Mn	13	--	13	--	68	[17]	26	[16]	25	--	25	--
0.050		Mo	13	--	13	--	68	[53]	26	52.0	25	[16]	25	[15]
0.150	75	Na	39	55.2	39	58.3	204	180,000	79	188,000	76	74,100	75	72,300
0.100		Nd	26	--	26	--	136	[33]	52	[30]	50	[6.2]	50	[6.7]
0.030	30	Ni	8	[0.96]	8	[0.89]	41	419	16	413	15	123	15	120
0.100	600	P	26	--	26	--	136	1,800	52	1,830	50	607	50	594
0.100	300	Pb	26	--	26	--	136	188	52	183	50	[49]	50	[49]
0.750		Pd	197	--	197	--	1020	--	393	--	378	--	374	--
0.300		Rh	79	--	79	--	408	--	157	--	151	--	150	--
1.100		Ru	289	--	289	--	1496	--	576	--	555	--	549	--
0.500		Sb	131	--	131	--	680	--	262	--	252	--	250	--
0.250		Se	66	--	66	--	340	--	131	--	126	--	125	--
0.500		Si	131	151	131	139	680	[290]	262	[180]	252	290	250	270
1.500		Sn	393	--	394	--	2040	--	785	--	757	--	749	--
0.015		Sr	4	--	4	--	20	[2.3]	8	[2.3]	8	--	7	--
1.500		Te	393	--	394	--	2040	--	785	--	757	--	749	--
1.000		Th	262	--	263	--	1360	--	523	--	504	--	499	--
0.025		Ti	7	--	7	--	34	--	13	--	13	--	12	--
0.500		Tl	131	--	131	--	680	--	262	--	252	--	250	--
2.000	600	U	525	--	525	--	2720	--	1047	--	1009	--	998	--
0.050		V	13	--	13	--	68	--	26	--	25	--	25	--
2.000		W	525	--	525	--	2720	--	1047	[130]	1009	--	998	--
0.050		Y	13	--	13	--	68	--	26	[2.8]	25	--	25	--
0.050		Zn	13	--	13	--	68	--	26	[6.2]	25	[3.3]	25	--
0.050		Zr	13	--	13	--	68	[8.4]	26	[7.9]	25	--	25	--

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).

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Battelle, PNNL / AIAL
Inorganic Analysis / ICP-MS Data Report

Project / WP#:	42040 / W59711
ASR#:	6025
Client:	Mike Urie
Total Samples:	2

RPL #	Client ID
01-00441	AN-102-SUP-DF
01-00442	AN-102-CS-DF

Procedure: PNL-ALO-280 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

Analyst: James P Bramson

Analysis	Analysis Date	Analysis Files		
		Experiment	Procedure	Element Menu
Supernatant				
Cs, Rb, Tc	3/27/01	27MAR01	010327a	CsRbTc
Actinides	4/4/01, 4/12/01	4APR01 12APR01	010404a 010412a	actinides
Wet Centrifuged Solids				
Mass 0-100	4/23/01, 4/25/01	23APR01 25APR01	010423a 0104255b	7-100
Mass 100-200	4/19/01	19APR01	010419a	100-210
I-127/129	4/27/01 4/30/01	27APR01 30APR01	010427a 010430a	iodine
Actinides	4/3/01, 4/4/01 4/11/01	3APR01 4APR01 11APR01	010403a 010404a 010411a	UTh actinides
U Isotopics	4/11/01	11APR01	010411a	100-210

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

8-23-01
Reviewed By

James Bramson 8/24/01
Concur

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Two samples, Diluted Feed AN-102 Supernatant and Wet Centrifuged Solids, were analyzed on a radioactive material contained ICP/MS for the requested analytes.

1. Analysis

See attached ICP/MS data reports for final results and run order for the analytical batches. The final results have been corrected for all laboratory dilutions performed on the samples during analysis. Preparation blanks, blank spikes, duplicates, and matrix spikes were submitted with the samples. The preparation blanks and blank spike are given RPL ID#s associated with ASR 6019. The matrix spike of As Received AN-102 supernatant (01-00429) and wet centrifuged solids (01-00430) of ASR 6019 serve as the matrix spike QC samples for these analyses. The analyses were separated into several analytical batches:

A. Supernatants
  A.1. Cs, Rb, Tc
  A.2. Actinides

B. Wet Centrifuged Solids
  B.1. Mass 0-100
  B.2. Mass 100-200
  B.3. I-127/129
  B.4. Actinides
  B.5. Uranium Isotopics

Lithium, beryllium, and boron are at the end of the detector range and the instrument response tends to have a wide variation in this region. Arsenic, selenium, and vanadium may also have a wide variation in the results due to the extensive chain of isobaric interference corrections. The calibration curves of four analytes had correlation coefficients < 0.995: lithium and I-127 (during the analysis of the preparation blanks), boron (during the analysis of the samples), and selenium (during the analysis of preparation blanks and samples).

2. Quality Control

Duplicates (DUP). Duplicates were submitted with the samples. In addition, replicate analyses were also performed. The QC acceptance criterion was an RPD of < 15%.

A. Supernatants
  A.1. Cs, Rb, Tc. The duplicate (and replicate) analyses met the QC criterion.
  A.2. Actinides. The duplicate (and replicate) analyses met the QC criterion.

B. Wet Centrifuged Solids
  B.1. Mass 0-100. The duplicate QC criterion was not met for Co. The replicate analysis was just outside the duplicate QC criterion for Mo.
  B.2. Mass 100-200. The QC criterion was not met for Sb, Ta, and Tl. The replicate analysis does not meet the duplicate QC criterion for Tl. The Ta concentration is comparable to that found in the preparation blanks. The Tl is near the detection limit.

- B.3. I-127/129. The QC criterion was not met for the duplicate analysis of I-127. The replicate analysis met the duplicate QC criterion.
- B.4. Actinides. The QC criterion was met in both duplicate and replicate analysis.
- B.5. Uranium Isotopics. The QC criterion was met in both duplicate and replicate analysis.

Matrix Spikes (MS). Matrix spikes were submitted with the samples. The matrix spike of As Received AN-102 supernatant (01-00429) and wet centrifuged solids (01-00430) of ASR 6019 serve as the matrix spike QC samples for these analyses. In addition post spike (PS) analyses were also performed.

- A. Supernatants
  - A.1. Cs, Rb, Tc. The QC criterion of 70-130% was met in both MS and PS analysis.
  - A.2. Actinides. These analytes did not appear to have been spiked into the sample, however the PS met the MS QC criterion of 75-125%
- B. Wet Centrifuged Solids
  - B.1. Mass 0-100. The MS did not meet the QC criterion met for Li and B. The PS did not meet the MS QC criterion for Li and Se.
  - B.2. Mass 100-200. The MS did not meet the QC criterion for W. The PS did not meet the MS QC criterion for Ce. The cause for the PS failure is unknown.
  - B.3. I-127/129. These analytes did not appear to be spiked into the samples. Although there is no QC acceptance criterion for these analytes, the PS was within 75-125%.
  - B.4. Actinides. The QC criterion was met.
  - B.5. Uranium Isotopics. These analytes did not appear to be spiked into the samples. No PS was performed.

Preparation Blank (PB) and Blank Spike (BS). Two preparation blanks and a blank spike were submitted with the samples. In addition a post spike (PS) of one of the preparation blanks was performed.

- A. Supernatants
  - A.1. Cs, Rb, Tc. The PBs were below detection level and the recovery for both BS and PS met the QC criterion of 70-130%.
  - A.2. Actinides. The PBs were below detection level. The analytes did not appear to be spiked into the BS, however the PS met the MS QC criterion of 75-125%.
- B. Wet Centrifuged Solids
  - B.1. Mass 0-100. Most of the elements were below the detection level of the instrument except for B, V, As, and Y. Boron was well above the EQL and MRQ. Vanadium was above the EQL but below the MRQ. The arsenic was below the EQL. Yttrium was above detection level in PB2, but below the EQL. The BS did not meet the QC criterion for B. The PS did not meet the MS QC criterion for Li and Se.
  - B.2. Mass 100-200. All analytes were below the detection level except for Ta, which was slightly above the EQL but below the MRQ.
  - B.3. I-127/129. The PB was below detection level. These analytes did not appear to be spiked into the BS. Although there is no spike QC acceptance criterion, the PS was within 75-125%.

- B.4. Actinides. The PBs were below detection level. The BS (and PS) met the spike QC criterion.
- B.5. Uranium Isotopics. The PBs were below detection level. These analytes did not appear to be spiked into the BS. A PS was not performed.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criterion of less than the estimated quantitation limit (< EQL) was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV).

- A. Supernatants
  - A.1. Cs, Rb, Tc. The ICV/CCVs met the QC criterion.
  - A.2. Actinides. Some of the ICV/CCV QC criterion was not met for Pu-239 (87%), Pu-240 (81%, 81%) and AM-241 (89.8%, 89.8%, 81%) during the analysis of the PBs and for Pu-240 (115%, 83%) and Am-241 (112%) during the analysis of the samples. For Pu-240 and Am-241, the count rates are very small.

- B. Wet Centrifuged Solids
  - B.1. Mass 0-100. Some of the ICV/CCV QC criterion were not met for B (113%, 112%), V (111%, 111%, 113%), As (115%), Se (119%-228%), Rb (114%), and Mo (110%-120%). For the selenium analysis, all of the samples including the spikes have selenium concentrations below the MRQ.
  - B.2. Mass 100-200. Some of the ICV/CCV QC criterion were not met for W (120%), Ta (89.5%, 80%), and Tl (113%).
  - B.3. I-127/129. The QC criterion was not met for the final CCV during the I-129 (87%) analysis of the samples.
  - B.4. Actinides. Some of the QC criterion were not met for Pu-239 (86%) and Pu-240 (82%, 81%) during the analysis of the PBs and Pu-239 (116%) during the analysis of the samples.
  - B.5. Uranium Isotopics. N/A

Internal Standard (IS). The ISs met the QC criterion of 30% to 120%.

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6025
Log-In: 01-00441
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/ml of original sample for radioisotopes; µg analyte/ml of original sample for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/ml	Rb µg/ml	±	1SD	MDL µCi/ml	^{99}Tc µCi/ml	±	1SD	MDL µg/ml	^{133}Cs µg/ml	±	1SD
	1%HNO3		ICB	<	1.32E-06			<	6.91E-08			<	2.45E-06		
	1%HNO3		CCB1	<	1.47E-06			<	7.70E-08			<	2.73E-06		
	1%HNO3		CCB2	<	1.49E-06			<	7.78E-08			<	2.76E-06		
	1%HNO3		CCB3	<	1.22E-06			<	6.40E-08			<	2.27E-06		
	ICV		ICV1		2.01E-04	±	2.11E-06		3.29E-06	±	2.69E-08		1.94E-04	±	1.29E-06
	CCV		CCV1a		2.01E-04	±	3.10E-07		3.20E-06	±	3.71E-08		1.87E-04	±	2.57E-06
	CCV		CCV2a		2.10E-04	±	5.36E-06		3.28E-06	±	5.30E-08		1.93E-04	±	2.12E-06
	CCV		CCV3a		2.13E-04	±	2.74E-06		3.04E-06	±	4.40E-08		1.85E-04	±	6.43E-07
True Value					**2.00E-04**				**3.39E-06**				**2.00E-04**		
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	2.88E-01 <	1.20E-01			6.29E-03 <	6.29E-03			2.23E-01 <	2.23E-01		
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	2.63E-01	1.73E+01	±	3.13E-01	5.73E-03	2.37E-01	±	3.79E-03	2.03E-01	1.50E+01	±	1.71E-01
Spike Concentration					2.04E-04				2.80E-06				1.77E-04		
Spike Recovery					**102%**				**83%**				**88%**		
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	3.08E-01 <	1.28E-01			6.71E-03 <	6.71E-03			2.38E-01 <	**2.38E-01**		
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS	1.09E+00	2.18E+01	±	1.90E-01	2.37E-02 <	2.37E-02			8.39E-01	3.00E+01	±	4.65E-01
Spike Concentration					7.73E-05								1.06E-04		
Spike Recovery					**103%**								**94%**		
01-00441	01-00441	AN-102-SUP-DF	Sample1	7.97E-02	2.28E+00	±	1.26E-02	1.74E-03	4.20E-02	±	3.35E-04	6.16E-02	2.59E+00	±	1.36E-02
01-00441-DUP	01-00441-DUP	AN-102-SUP-DF	Sample2	7.93E-02	2.27E+00	±	4.85E-03	1.73E-03	4.28E-02	±	4.84E-04	6.13E-02	2.65E+00	±	5.04E-02
01-00441	01-00441 duplicate	AN-102-SUP-DF	Sample3	7.33E-02	2.28E+00	±	4.30E-02	1.60E-03	3.84E-02	±	1.46E-03	5.67E-02	2.45E+00	±	3.13E-02
Duplicate %RPD					**0.1%**				**8.8%**				**5.4%**		
01-00429	01-00429	AN-102-SUP-AR	Sample4	2.37E-01	8.91E+00	±	7.69E-02	5.16E-03	1.48E-01	±	1.97E-03	1.83E-01	9.60E+00	±	1.05E-01
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	9.91E-02	2.67E+01	±	2.16E-01	5.18E-03	4.08E-01	±	7.97E-03	1.84E-01	2.50E+01	±	5.16E-02
Spike Concentration					2.17E-04				3.18E-06				1.88E-04		
Spike Recovery					**109%**				**94%**				**94%**		
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	3.42E-01	3.21E+01	±	3.36E-01	1.79E-02	1.68E-01	±	3.85E-03	6.35E-01	3.98E+01	±	9.41E-01
Spike Concentration					8.19E-05								1.07E-04		
Spike Recovery					**117%**								**102%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01
Instrument Filenames. Experiment (27MAR01), Procedure(010327a), Element Menu (CsRbTc)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6025
Log-In: 01-00441
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/ml of original sample for radioisotopes; µg analyte/ml of original sample for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL ppm		^{241}Am ppm	±	1SD
	1%HNO3 (blanks)		ICB		<	2.46E-07		
	1%HNO3 (blanks)		CCB1		<	3.04E-07		
	1%HNO3 (blanks)		CCB2		<	2.13E-07		
	1%HNO3 (samples)		ICB		<	3.60E-07		
	1%HNO3 (samples)		CCB1		<	3.76E-07		
	1%HNO3 (samples)		CCB2		<	3.75E-07		
	ICV (blanks)		ICV			4.49E-05	±	1.01E-06
	CCV (blanks)		CCV1			4.49E-05	±	1.74E-06
	CCV (blanks)		CCV2			4.03E-05	±	1.12E-06
	CCV (blanks)		CCV3			4.78E-05	±	1.50E-06
True Value						**5.00E-05**		
	ICV (samples)		ICV			5.17E-05	±	9.44E-07
	CCV (samples)		CCV1			5.10E-05	±	9.97E-07
	CCV (samples)		CCV2			5.59E-05	±	1.12E-06
True Value						**5.00E-05**		
	20ppb U030 uranium (blanks)		U030a		<	2.38E-07		
	30ppb 4321B uranium (blanks)		4321b1		<	2.72E-07		
	30ppb 4321B uranium (blanks)		4321b2		<	2.21E-07		
	10ppb 4321B uranium (samples)		4321b		<	4.59E-07		
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	1.74E-03	<	1.74E-03		
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	1.84E-03		2.60E-01	+	1.27E-02
Spike Concentration						4.59E-05		
Spike Recovery						**92%**		
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	1.72E-03	<	1.72E-03		
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS1	1.23E-03	<	1.23E-03		
01-00441	01-00441	AN-102-SUP-DF	Sample1	1.28E-03		1.15E-02	±	1.40E-03
01-00441-DUP	01-00441-DUP	AN-102-SUP-DF	Sample2	1.36E-03		1.22E-02	+	8.35E-04
01-00441	01-00441 duplicate	AN-102-SUP-DF	Sample3	1.30E-03		1.30E-02	±	7.09E-04
Duplicate %RPD						**12.0%**		
01-00429	01-00429	AN-102-SUP-AR	Sample4	1.58E-03		4.86E-02	+	3.11E-05
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	1.55E-03		3.10E-01	±	3.90E-03
Spike Concentration						4.80E-05		
Spike Recovery						**96%**		
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	1.54E-03		5.07E-02	±	4.48E-04

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01
Instrument Filenames: Experiment (27MAR01), Procedure(010327a), Element Menu (CsRbTc)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6025
Log-In: 01-00441
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-1-01

Analyst: 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/ml of original sample for radioisotopes; µg analyte/ml of original sample for all other elements.

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Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/ml	^{237}Np µCi/ml	±	1SD	MDL µCi/ml	^{239}Pu µCi/ml	±	1SD	MDL µCi/ml	^{240}Pu µCi/ml	±	1SD
	1%HNO3 (blanks)		ICB		< 1.58E-10				< 2.85E-08				< 4.37E-08		
	1%HNO3 (blanks)		CCB1		< 1.95E-10				5.90E-08	±	5.76E-09		< 5.40E-08		
	1%HNO3 (blanks)		CCB2		< 1.37E-10				< 2.47E-08				< 3.79E-08		
	1%HNO3 (samples)		ICB		< 6.21E-10				< 8.51E-08				< 1.56E-07		
	1%HNO3 (samples)		CCB1		< 6.48E-10				< 8.88E-08				< 1.63E-07		
	1%HNO3 (samples)		CCB2		< 6.46E-10				< 8.85E-08				< 1.63E-07		
	ICV (blanks)		ICV		3.24E-08	±	5.70E-10		3.22E-06	±	1.94E-07		1.90E-07	±	1.55E-08
	CCV (blanks)		CCV1		3.26E-08	±	1.00E-09		3.04E-06	±	4.15E-08		2.25E-07	±	2.75E-08
	CCV (blanks)		CCV2		3.18E-08	±	6.00E-10		2.64E-06	±	6.37E-08		1.89E-07	±	3.50E-09
	CCV (blanks)		CCV3		3.77E-08	±	1.25E-09		3.17E-06	±	5.08E-08		2.19E-07	±	1.28E-08
True Value					**3.52E-08**				**3.04E-06**				**2.34E-07**		
	ICV (samples)		ICV		3.70E-08	±	3.93E-10		2.97E-06	±	2.84E-08		2.34E-07	±	2.92E-09
	CCV (samples)		CCV1		3.55E-08	±	4.04E-10		3.19E-06	±	6.69E-08		2.68E-07	±	4.82E-08
	CCV (samples)		CCV2		3.86E-08	±	2.46E-10		2.89E-06	±	1.66E-07		1.94E-07	±	5.76E-09
True Value					**3.52E-08**				**3.04E-06**				**2.34E-07**		
	20ppb U030 uranium (blanks)		U030a		< 1.53E-10				< 2.76E-08				< 4.23E-08		
	30ppb 4321B uranium (blanks)		4321b1		< 1.75E-10				7.82E-08	±	2.87E-09		< 4.83E-08		
	30ppb 4321B uranium (blanks)		4321b2		< 1.42E-10				3.46E-08	±	1.52E-08		< 3.92E-08		
	10ppb 4321B uranium (samples)		4321b		< 7.91E-10				< 1.08E-07				< 1.99E-07		
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	1.12E-06	< 1.12E-06			2.01E-04	< 2.01E-04			3.09E-04	< 3.09E-04		
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	1.18E-06	1.90E-04	±	9.44E-06	2.13E-04	1.59E-02	±	1.21E-03	3.27E-04	1.01E-03	±	1.23E-04
Spike Concentration					3.36E-08				2.81E-06				1.79E-07		
Spike Recovery					**95%**				**92%**				**77%**		
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	1.10E-06	< 1.10E-06			1.99E-04	< 1.99E-04			3.05E-04	< 3.05E-04		
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS1	7.89E-07	< 7.89E-07	±	4.66E-08	1.42E-04	< 1.42E-04			2.18E-04	< 2.18E-04		
01-00441	01-00441	AN-102-SUP-DF	Sample1	2.21E-06	5.20E-05	±	2.26E-06	3.03E-04	1.79E-03	±	4.91E-05	5.56E-04	6.80E-04	±	4.86E-05
01-00441-DUP	01-00441-DUP	AN-102-SUP-DF	Sample2	2.35E-06	5.22E-05	±	9.14E-07	3.21E-04	1.65E-03	±	4.35E-05	5.90E-04	6.23E-04	±	1.13E-04
01-00441	01-00441 duplicate	AN-102-SUP-DF	Sample3	2.24E-06	5.82E-05	±	2.28E-06	3.07E-04	1.71E-03	±	1.80E-05	5.64E-04	< 5.64E-04		
Duplicate %RPD					**11.2%**				**4.6%**						
01-00429	01-00429	AN-102-SUP-AR	Sample4	2.73E-06	1.21E-04	±	3.76E-06	3.74E-04	6.75E-03	±	1.38E-04	6.86E-04	2.07E-03	±	1.13E-04
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	2.67E-06	2.97E-04	±	6.02E-06	3.66E-04	2.72E-02	±	2.01E-04	6.72E-04	3.35E-03	±	2.82E-04
Spike Concentration					3.23E-08				3.75E-06				2.35E-07		
Spike Recovery					**92%**				**124%**				**101%**		
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	2.65E-06	1.24E-04	±	3.51E-06	3.62E-04	6.15E-03	±	6.16E-05	6.66E-04	2.03E-03	±	1.40E-04

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01
Instrument Filenames: Experiment (27MAR01), Procedure(010327a), Element Menu (CsRbTc)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	V µg/g	±	1SD	MDL µg/g	Co µg/g	±	1SD	MDL µg/g	As µg/g	±	1SD
	1%HNO3 (blanks)		ICB		< 3.96E-07				< 5.64E-06				< 1.34E-05		
	1%HNO3 (blanks)		CCB1		< 5.25E-07				< 7.47E-06				< 1.78E-05		
	1%HNO3 (blanks)		CCB2		< 5.55E-07				< 7.90E-06				< 1.88E-05		
	1%HNO3 (samples)		ICB		< 2.65E-06				< 1.08E-06				< 3.06E-05		
	1%HNO3 (samples)		CCB1		< 4.21E-06				< 1.72E-06				< 5.87E-05		
	1%HNO3 (samples)		CCB2		< 4.78E-06				< 1.95E-06				< 5.95E-05		
	ICV (blanks)		ICV		2.22E-04	±	2.55E-06		2.11E-04	±	2.75E-06		2.19E-04	±	4.35E-06
	CCV (blanks)		CCV1		1.98E-04	±	5.79E-06		2.00E-04	±	9.36E-06		2.08E-04	±	9.53E-06
	CCV (blanks)		CCV2		2.20E-04	±	4.89E-06		2.11E-04	±	5.18E-06		2.12E-04	±	1.16E-05
True Value					**2.00E-04**				**2.00E-04**				**2.00E-04**		
	ICV (samples)		ICV		5.30E-04	±	1.53E-05		5.25E-04	±	9.62E-06		5.35E-04	±	3.13E-05
	CCV (samples)		CCV1		5.55E-04	±	1.98E-05		5.07E-04	±	1.05E-05		5.77E-04	±	1.70E-05
	CCV (samples)		CCv2		5.64E-04	±	1.41E-05		5.48E-04	±	2.19E-05		5.05E-04	±	3.72E-05
True Value					**5.00E-04**				**5.00E-04**				**5.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	2.78E-02	5.14E+00	±	9.18E-02	3.95E-01	< 3.95E-01			9.39E-01	3.52E+00	±	3.46E-01
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	4.91E-02	3.23E+01	±	2.21E+00	6.98E-01	2.32E+01	±	7.37E-01	1.66E+00	2.67E+01	±	1.90E+00
Spike Concentration					2.56E-04				2.19E-04				2.19E-04		
Spike Recovery					**128%**				**109%**				**110%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	2.85E-02	4.01E+00	±	3.25E-01	4.06E-01	< 4.06E-01			9.66E-01	2.29E+00	±	1.10E-01
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	1.53E-01	7.05E+01	±	3.13E+00	2.18E+00	3.40E+01	±	5.65E-01	5.18E+00	3.28E+01	±	1.06E+00
Spike Concentration					2.47E-04				1.28E-04				1.11E-04		
Spike Recovery					**104%**				**109%**				**94%**		
01-00430	01-00430	AN-102-CS-AR	Sample4	1.90E-01	6.49E+00	±	9.90E-02	7.74E-02	2.57E+00	±	8.81E-02	2.40E+00	5.88E+00	±	6.05E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	2.58E-01	6.72E+00	±	1.62E-01	1.05E-01	2.15E+00	±	1.98E-01	3.31E+00	7.19E+00	±	7.77E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	2.05E-01	6.42E+00	±	5.40E-02	8.37E-02	2.77E+00	±	1.83E-01	2.60E+00	6.37E+00	±	8.27E-01
Duplicate %RPD					**1.1%**				**7.6%**				**8.1%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	2.17E-01	2.49E+01	±	7.51E-01	8.82E-02	2.13E+01	±	8.62E-01	2.77E+00	2.52E+01	±	1.82E+00
Spike Concentration					4.36E-04				4.43E-04				4.56E-04		
Spike Recovery					**87%**				**89%**				**91%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	1.35E+00	8.19E+01	±	2.25E+00	5.50E-01	4.03E+01	±	6.46E-01	1.86E+01	2.09E+02	±	2.05E+01
Spike Concentration					2.85E-04				1.42E-04				7.68E-04		
Spike Recovery					**126%**				**126%**				**102%**		
01-00442	01-00442	AN-102-CS-DF	Sample1	1.61E-01	5.10E+00	±	1.39E-01	6.56E-02	8.61E-01	±	2.22E-02	1.60E+00	6.43E+00	±	4.16E-01
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	2.01E-01	5.65E+00	±	2.69E-01	8.17E-02	1.89E+00	±	1.10E-01	2.01E+00	7.32E+00	±	1.29E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	1.82E-01	5.60E+00	±	1.28E-01	7.40E-02	8.68E-01	±	2.47E-02	1.81E+00	6.88E+00	±	9.46E-01
Duplicate %RPD					**9.4%**				**0.8%**				**6.7%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reviewed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Li µg/g	±	1SD	MDL µg/g	Be µg/g	±	1SD	MDL µg/g	B µg/g	±	1SD
	1%HNO3 (blanks)		ICB		< 5.63E-06				< 1.43E-06				< 5.63E-06		
	1%HNO3 (blanks)		CCB1		< 7.46E-06				< 1.90E-06				< 7.46E-06		
	1%HNO3 (blanks)		CCB2		< 7.89E-06				< 2.01E-06				< 7.90E-06		
	1%HNO3 (samples)		ICB		< 4.09E-06				< 4.52E-06				< 3.00E-05		
	1%HNO3 (samples)		CCB1		< 6.52E-06				< 7.20E-06				< 4.77E-05		
	1%HNO3 (samples)		CCB2		< 7.39E-06				< 8.17E-06				< 5.41E-05		
	ICV (blanks)		ICV		1.88E-04	±	4.64E-06		2.12E-04	±	9.29E-07		1.88E-04	±	7.64E-06
	CCV (blanks)		CCV1		2.10E-04	±	1.89E-05		2.03E-04	±	6.54E-06		2.18E-04	±	2.48E-05
	CCV (blanks)		CCV2		2.12E-04	±	1.05E-05		1.99E-04	±	1.96E-06		2.26E-04	±	6.22E-06
True Value					**2.00E-04**				**2.00E-04**				**2.00E-04**		
	ICV (samples)		ICV		4.91E-04	±	2.41E-05		4.74E-04	±	2.24E-05		5.14E-04	±	3.45E-05
	CCV (samples)		CCV1		4.73E-04	±	7.42E-06		4.85E-04	±	3.43E-05		5.62E-04	±	2.93E-05
	CCV (samples)		CCV2		5.10E-04	±	6.52E-05		5.28E-04	±	4.09E-05		5.03E-04	±	6.90E-06
True Value					**5.00E-04**				**5.00E-04**				**5.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	3.94E-01	< 3.94E-01			1.00E-01	< 1.00E-01			3.94E-01	2.30E+01	±	3.14E+01
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	6.97E-01	3.06E+01	±	8.77E+00	1.77E-01	2.60E+01	±	2.93E+00	6.98E-01	5.04E+01	±	1.03E+01
Spike Concentration					2.89E-04				2.46E-04				2.59E-04		
Spike Recovery					**145%**				**123%**				**129%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	4.06E-01	< 4.06E-01			1.03E-01	< 1.03E-01			4.06E-01	1.86E+01	±	7.11E-01
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	7.23E+00	3.81E+02	±	1.04E+02	5.53E-01	3.65E+01	±	3.85E+00	2.18E+00	3.94E+01	±	1.56E+00
Spike Concentration					3.60E-04				1.38E-04				6.19E-05		
Spike Recovery					**122%**				**117%**				**52%**		
01-00430	01-00430	AN-102-CS-AR	Sample4	2.94E-01	5.74E-01	±	4.00E-02	3.25E-01	1.26E+00	±	3.40E-01	2.15E+00	2.76E+01	±	3.03E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	3.99E-01	3.56E+01	±	6.99E-01	4.41E-01	9.83E-01	±	5.36E-02	2.92E+00	5.13E+01	±	9.16E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	3.18E-01		±	3.34E-01	3.51E-01	9.57E-01	±	1.17E-01	2.33E+00	2.76E+01	±	1.90E-01
Duplicate %RPD									**27.3%**				**0.01%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	3.35E-01	2.99E+01	±	3.38E+00	3.70E-01	2.79E+01	±	3.68E+00	2.45E+00	5.11E+01	±	1.52E+00
Spike Concentration					6.95E-04				6.30E-04				5.55E-04		
Spike Recovery					**139%**				**126%**				**111%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	3.97E+00	1.92E+03	±	3.10E+01	2.31E+00	1.98E+02	±	3.35E+00	1.53E+01	3.74E+02	±	6.40E+00
Spike Concentration					3.63E-03				7.44E-04				1.31E-03		
Spike Recovery					**241%**				**99%**				**35%**		
01-00442	01-00442	AN-102-CS-DF	Sample1	2.49E-01	2.41E+01	±	1.81E-01	2.75E-01	1.47E+01	±	4.03E-01	1.82E+00	1.72E+01	±	8.87E-02
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	3.10E-01	2.35E+01	±	5.30E-01	3.43E-01	1.39E+01	±	7.87E-01	2.27E+00	1.76E+01	±	2.68E-01
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	2.81E-01	2.47E+01	±	3.61E-01	3.10E-01	1.40E+01	±	7.12E-01	2.06E+00	1.83E+01	±	4.46E-01
Duplicate %RPD					**2.6%**				**4.9%**				**6.2%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Se µg/g	±	1SD	MDL µg/g	Rb µg/g	±	1SD	MDL µg/g	Y µg/g	±	1SD
	1%HNO3 (blanks)		ICB	<	1.78E-04			<	3.70E-06			<	1.75E-07		
	1%HNO3 (blanks)		CCB1	<	2.36E-04			<	4.90E-06			<	2.32E-07		
	1%HNO3 (blanks)		CCB2	<	2.50E-04			<	5.18E-06			<	2.46E-07		
	1%HNO3 (samples)		ICB	<	1.45E-04			<	2.31E-06			<	2.47E-06		
	1%HNO3 (samples)		CCB1	<	2.74E-04			<	4.44E-06			<	3.93E-06		
	1%HNO3 (samples)		CCB2	<	2.90E-04			<	4.50E-06			<	4.46E-06		
	ICV (blanks)		ICV		2.34E-04	±	2.09E-05		2.20E-04	±	7.09E-07		2.17E-04	±	3.61E-06
	CCV (blanks)		CCV1		3.63E-04	±	3.20E-04		2.05E-04	±	9.54E-06		1.98E-04	±	8.24E-06
	CCV (blanks)		CCV2	<	2.27E-04				2.13E-04	±	8.01E-06		2.10E-04	±	3.88E-06
True Value					2.00E-04				2.00E-04				2.00E-04		
	ICV (samples)		ICV		5.97E-04	±	2.31E-04		5.40E-04	±	4.78E-06		5.17E-04	±	1.87E-05
	CCV (samples)		CCV1		1.14E-03	±	1.47E-04		5.50E-04	±	7.03E-06		5.14E-04	±	8.33E-06
	CCV (samples)		CCV2		6.98E-04	±	3.34E-04		5.71E-04	±	2.15E-05		5.19E-04	±	5.00E-06
True Value					5.00E-04				5.00E-04				5.00E-04		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	1.25E+01 <	1.25E+01			2.59E-01 <	2.59E-01			1.23E-02 <	1.23E-02		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	2.20E+01	3.31E+01	±	1.88E+01	4.58E-01	2.12E+01	±	7.33E-01	2.17E-02	2.13E+01	±	1.37E+00
Spike Concentration					3.13E-04				2.00E-04				2.01E-04		
Spike Recovery					156%				100%				100%		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	1.28E+01 <	1.28E+01			2.66E-01 <	2.66E-01			1.26E-02	3.78E-02	±	9.44E-03
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	2.29E+02	2.62E+02	±	1.83E+02	1.43E+00	5.59E+01	±	1.80E+00	6.77E-02	5.97E+01	±	1.78E+00
Spike Concentration					9.89E-04				2.11E-04				2.25E-04		
Spike Recovery					84%				89%				95%		
01-00430	01-00430	AN-102-CS-AR	Sample4	1.20E+02 <	1.20E+02			1.81E-01	4.77E+00	±	2.32E-01	1.77E-01	9.59E+00	±	1.39E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	1.68E+01 <	1.68E+01			2.50E-01	4.64E+00	±	4.22E-01	2.41E-01	9.02E+00	±	2.39E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	1.31E+01 <	1.31E+01			1.96E-01	5.08E+00	±	1.50E-01	1.92E-01	9.67E+00	±	1.12E-01
Duplicate %RPD									6.3%				0.8%		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	9.29E+00	2.85E+01	±	1.11E+01	2.09E-01	2.37E+01	±	4.82E-01	2.02E-01	2.71E+01	±	2.44E-01
Spike Concentration					6.74E-04				4.49E-04				4.14E-04		
Spike Recovery					135%				90%				83%		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	8.74E+01 <	8.74E+01			1.41E+00	7.77E+01	±	3.15E+00	1.26E+00	6.67E+01	±	1.03E+00
Spike Concentration									2.75E-04				2.16E-04		
Spike Recovery									122%				95%		
01-00442	01-00442	AN-102-CS-DF	Sample1	1.10E+01 <	1.10E+01			1.21E-01	1.11E+00	±	1.65E-01	1.50E-01	2.61E+01	±	3.41E-01
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	1.40E+01 <	1.40E+01			1.52E-01	1.24E+00	±	5.59E-02	1.87E-01	2.55E+01	±	9.67E-01
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	1.22E+01 <	1.22E+01			1.37E-01	1.14E+00	±	1.47E-01	1.69E-01	2.68E+01	±	1.11E-01
Duplicate %RPD									2.5%				2.6%		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: EMP 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g		Mo µg/g	±	1SD	MDL µCi/g		99Tc µCi/g	±	1SD
	1%HNO3 (blanks)		ICB		<	1.50E-05				<	3.88E-08		
	1%HNO3 (blanks)		CCB1		<	1.89E-05				<	4.89E-08		
	1%HNO3 (blanks)		CCB2		<	2.14E-05				<	5.54E-08		
	1%HNO3 (samples)		ICB		<	2.87E-05				<	2.87E-08		
	1%HNO3 (samples)		CCB1		<	5.43E-05				<	5.42E-08		
	1%HNO3 (samples)		CCB2		<	5.76E-05				<	5.76E-08		
	ICV (blanks)		ICV			2.26E-04	±	2.14E-05			3.63E-06	±	8.06E-08
	CCV (blanks)		CCV1			2.18E-04	±	1.85E-05			3.60E-06	±	1.76E-07
	CCV (blanks)		CCV2			2.12E-04	±	7.59E-05			3.71E-06	±	2.74E-07
True Value						**2.00E-04**					**3.39E-06**		
	ICV (samples)		ICV			5.52E-04	±	2.91E-05			8.45E-06	±	4.64E-07
	CCV (samples)		CCV1			6.05E-04	±	4.16E-05			9.31E-06	±	3.66E-07
	CCV (samples)		CCv2			5.61E-04	±	2.23E-05			9.04E-06	±	1.84E-07
True Value						**5.00E-04**					**8.48E-06**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	2.47E+00	<	1.05E+00			1.84E-03	<	2.72E-03		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	4.36E+00		2.26E+01	±	7.23E-01	3.68E-01		3.68E-01	±	2.45E-02
Spike Concentration						2.13E-04					2.05E-04		
Spike Recovery						**107%**					**103%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	2.54E+00	<	1.08E+00			7.72E-04	<	2.79E-03		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	5.66E+00		3.17E+02	±	7.60E+00	1.44E-02	<	5.26E-02		
Spike Concentration						1.20E-03							
Spike Recovery						**101%**							
01-00430	01-00430	AN-102-CS-AR	Sample4	2.38E+00		3.34E+01	±	1.28E+00	2.37E-03		9.50E-02	±	5.11E-03
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	3.34E+00		3.26E+01	±	2.66E+00	3.34E-03		1.03E-01	±	3.13E-03
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	2.60E+00		3.41E+01	±	2.47E+00	2.60E-03		1.04E-01	±	1.67E-03
Duplicate %RPD						**2.1%**					**8.7%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	2.91E+00		4.91E+01	±	1.65E+00	2.90E-03		4.53E-01	±	5.80E-03
Spike Concentration						3.73E-04					4.99E-04		
Spike Recovery						**75%**					**100%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	1.73E+01		3.58E+02	±	1.34E+01	1.73E-02		8.75E-02	±	1.66E-02
Spike Concentration						1.22E-03							
Spike Recovery						**108%**							
01-00442	01-00442	AN-102-CS-DF	Sample1	2.19E+00		1.19E+01	±	4.35E-01	2.19E-03		3.39E-02	±	2.37E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	2.78E+00		1.35E+01	±	1.12E+00	2.78E-03		3.03E-02	±	2.54E-03
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	2.42E+00		1.02E+01	±	1.09E+00	2.42E-03		3.16E-02	±	3.45E-04
Duplicate %RPD						**15.4%**					**6.8%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reviewed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Ca µg/g	±	1SD	MDL µg/g	Pr µg/g	±	1SD	MDL µg/g	W µg/g	±	1SD
	1%HNO3		ICB	<	8.17E-06			<	2.66E-06			<	1.18E-05		
	1%HNO3		CCB1	<	8.08E-06			<	2.63E-06			<	1.17E-05		
	1%HNO3		CCB2	<	8.08E-06			<	2.63E-06			<	1.17E-05		
	1%HNO3		CCB3	<	8.11E-06			<	2.64E-06				2.14E-05	±	3.60E-07
	ICV		ICV		2.17E-04	±	6.15E-06		1.94E-04	±	3.02E-06		1.92E-04	±	9.37E-06
	CCV		CCV1		2.20E-04	±	2.91E-06		2.03E-04	±	8.41E-06		2.03E-04	±	1.16E-05
	CCV		CCV2		1.91E-04	±	5.13E-06		1.97E-04	±	6.48E-06		1.92E-04	±	9.74E-06
	CCV		CCV3		2.07E-04	±	7.16E-06		2.16E-04	±	4.85E-06		2.39E-04	±	1.03E-05
True Value					2.00E-04				2.00E-04				2.00E-04		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	4.15E-01 <	4.15E-01			1.35E-01 <	1.35E-01			6.02E-01 <	6.02E-01		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	4.34E-01	1.05E+01	±	5.01E-01	1.41E-01	1.07E+01	±	3.62E-01	6.29E-01	1.13E+01	±	5.52E-01
Spike Concentration					1.98E-04				2.02E-04				2.13E-04		
Spike Recovery					99%				101%				107%		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	4.36E-01 <	4.36E-01			1.42E-01 <	1.42E-01			6.32E-01 <	6.32E-01		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	4.00E+00	5.34E+01	±	8.73E-01	1.30E+00	5.76E+01	±	6.81E-01	5.80E+00	4.30E+01	±	4.90E+00
Spike Concentration					1.01E-04				1.09E-04				8.11E-05		
Spike Recovery					85%				92%				69%		
01-00430	01-00430	AN-102-CS-AR	Sample1	3.78E-01	2.25E+01	±	8.87E-01	1.23E-01	1.75E+01	±	2.77E-01	5.48E-01	9.28E+01	±	4.56E+00
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample2	4.54E-01	2.35E+01	±	2.43E-01	1.48E-01	1.76E+01	±	2.20E-01	6.58E-01	1.01E+02	±	2.46E+00
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample3	3.64E-01	2.32E+01	±	4.51E-01	1.19E-01	1.79E+01	±	4.25E-01	5.28E-01	9.91E+01	±	5.09E+00
Duplicate %RPD					2.9%				2.0%				6.6%		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	6.20E-01	4.53E+01	±	6.45E-01	2.02E-01	3.45E+01	±	6.10E-01	8.99E-01	1.40E+02	±	3.56E+00
Spike Concentration					2.70E-04				2.01E-04				5.54E-04		
Spike Recovery					135%				101%				111%		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	3.82E+00	4.21E+02	±	4.01E+00	1.24E+00	7.53E+01	±	9.34E-01	5.54E+00	1.32E+02	±	5.40E+00
Spike Concentration					7.52E-04				1.09E-04				7.45E-05		
Spike Recovery					100%				97%				55%		
01-00442	01-00442	AN-102-CS-DF	Sample4	2.30E-01	7.14E+01	±	1.03E+00	7.50E-02	4.71E+01	±	2.11E-01	3.34E-01	4.26E+01	±	1.86E+00
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample5	2.20E-01	6.90E+01	±	8.26E-01	7.18E-02	4.67E+01	±	1.05E+00	3.19E-01	4.20E+01	±	2.48E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample6	2.51E-01	7.22E+01	±	5.19E-01	8.17E-02	4.78E+01	±	1.42E+00	3.64E-01	4.64E+01	±	1.03E+00
Duplicate %RPD					1.1%				1.5%				8.7%		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Revelwed by: kmP 6-1-01

Analyst: JPB 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Sb µg/g	±	1SD	MDL µg/g	Te µg/g	±	1SD	MDL µg/g	Ta µg/g	±	1SD
	1%HNO3		ICB	<	9.89E-06			<	1.29E-04				3.59E-05	±	5.49E-04
	1%HNO3		CCB1	<	9.78E-06			<	1.28E-04				3.74E-05	±	3.98E-05
	1%HNO3		CCB2	<	9.78E-06			<	1.28E-04				3.64E-05	±	5.43E-05
	1%HNO3		CCB3	<	9.82E-06			<	1.29E-04				3.87E-05	±	1.56E-05
	ICV		ICV		2.16E-04	±	1.72E-05		2.04E-04	±	4.85E-06		1.79E-04	±	1.11E-05
	CCV		CCV1		2.19E-04	±	2.24E-05		1.93E-04	±	4.97E-06		1.85E-04	±	2.23E-06
	CCV		CCV2		2.03E-04	±	8.92E-06		2.04E-04	±	6.20E-06		1.59E-04	±	9.43E-06
	CCV		CCV3		1.94E-04	±	7.75E-06		2.06E-04	±	2.66E-05		1.97E-04	±	4.69E-06
True Value					**2.00E-04**				**2.00E-04**				**2.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	5.03E-02 <	5.03E-02			6.58E-01 <	6.58E-01			3.24E-02	1.99E-01	±	9.14E-02
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	5.25E-01	1.09E+00	±	7.77E-02	6.87E-01	1.07E+00	±	1.41E-01	3.38E-02	9.80E-01	±	4.22E-02
Spike Concentration					2.06E-04				2.02E-04				1.47E-04		
Spike Recovery					**103%**				**101%**				**74%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.28E-02 <	5.28E-02			6.90E-01 <	6.90E-01			3.40E-02	2.00E-01	±	5.72E-02
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	4.84E+00	1.23E+02	±	2.21E+00	6.33E+01 <	6.33E+01			3.12E+00	4.73E+01	±	2.90E+00
Spike Concentration					2.33E-04				1.20E-04				8.94E-05		
Spike Recovery					**99%**				**101%**				**76%**		
01-00430	01-00430	AN-102-CS-AR	Sample9	4.31E-02	1.16E-01	±	3.36E-02	5.64E-01	1.48E+00	±	1.77E-01	2.77E-02	2.45E-01	±	1.18E-02
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample10	4.77E-02	1.99E-01	±	3.05E-02	6.25E-01	1.66E+00	±	5.40E-01	3.07E-02	2.61E-01	±	5.97E-02
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample11	4.27E-02	1.81E-01	±	4.85E-02	5.59E-01	1.37E+00	±	1.30E-01	2.75E-02	2.22E-01	±	2.75E-02
Duplicate %RPD					**44.0%**				**7.4%**				**10.0%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	7.51E-01	2.12E+01	±	1.30E+00	9.83E+00	1.95E+01	±	1.21E+00	4.84E-01	1.33E+01	±	3.94E-01
Spike Concentration					2.50E-04				2.13E-04				1.55E-04		
Spike Recovery					**125%**				**107%**				**77%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	4.63E+00	1.23E+02	±	6.45E+00	6.05E+01	6.15E+01	±	1.31E+00	2.98E+00	4.21E+01	±	1.18E+00
Spike Concentration					2.32E-04				1.13E-04				7.91E-05		
Spike Recovery					**103%**				**100%**				**70%**		
01-00442	01-00442	AN-102-CS-DF	Sample12	2.96E-02	1.34E-01	±	2.39E-02	3.87E-01	4.38E+00	±	4.31E-01	1.90E-02	2.65E-01	±	2.28E-02
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample13	3.03E-02	1.56E-01	±	2.86E-02	3.97E-01	4.07E+00	±	2.28E-01	1.95E-02	1.89E-01	±	1.70E-02
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample14	3.05E-02	1.16E-01	±	4.73E-03	4.00E-01	4.23E+00	±	5.12E-02	1.97E-02	2.78E-01	±	4.97E-03
Duplicate %RPD					**14.2%**				**3.3%**				**4.9%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reviewed by: tmP3 6-1-01

Analyst: JCBryan 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	127I µg/g	± 1SD	MDL µCi/g	129I µCi/g	± 1SD
	1%HNO3 (blanks)		ICB		< 1.05E-05			< 1.81E-09	
	1%HNO3 (blanks)		CCB1		< 4.62E-05			< 1.96E-09	
	1%HNO3 (blanks)		CCB2		< 1.01E-05			< 1.78E-09	
	1%HNO3 (blanks)		CCB3		< 1.01E-05			< 1.78E-09	
	1%HNO3 (samples)		ICB		< 1.33E-05			< 2.46E-09	
	1%HNO3 (samples)		CCB1		< 1.53E-05			< 2.86E-09	
	1%HNO3 (samples)		CCB2		< 1.84E-05			< 3.42E-09	
	ICV (blanks)		ICV1		5.48E-05 ±	6.36E-06		8.35E-09 ±	6.04E-10
True Value					**5.19E-05**			**8.71E-09**	
	CCV (blanks)		CCV1		4.62E-05 ±	7.61E-07		9.25E-09 ±	4.67E-10
	CCV (blanks)		CCV3		5.12E-05 ±	5.91E-06		7.76E-09 ±	2.16E-10
	CCV (blanks)		CCV5		5.35E-05 ±	1.66E-06		8.09E-09 ±	8.30E-10
True Value					**5.11E-05**			**8.71E-09**	
	ICV (samples)		ICV1&2		4.80E-05 ±	5.02E-06		8.22E-09 ±	1.20E-09
True Value					**5.19E-05**			**8.71E-09**	
	CCV (samples)		CCV1&2		4.93E-05 ±	4.42E-06		8.40E-09 ±	4.81E-10
	CCV (samples)		CCV3&4		4.78E-05 ±	3.17E-06		7.61E-09 ±	6.08E-10
True Value					**5.11E-05**			**8.71E-09**	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB5	1.15E-01	< 1.15E-01		2.04E-05	< 2.04E-05	
01-00430-PB1	01-00430-PB1+ spike	PROCESS BLANK	PBS2&5	1.29E-01	1.15E+00 ±	7.18E-02	2.17E-05	7.03E-05 ±	1.43E-05
Spike Concentration					1.09E-04			6.64E-09	
Spike Recovery					**106%**			**76%**	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB6	1.13E-01	< 1.13E-01		2.00E-05	< 2.00E-05	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.06E-01	< 1.06E-01		1.87E-05	< 1.87E-05	
01-00430	01-00430	AN-102-CS-AR	Sample4	5.09E-01	2.43E+00 ±	1.17E-01	5.79E-05	1.53E-04 ±	8.35E-06
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample3	5.44E-01	2.46E+00 ±	4.59E-01	6.20E-05	1.70E-04 ±	2.76E-05
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample2	4.59E-01	2.35E+00 ±	4.40E-01	5.22E-05	1.46E-04 ±	1.76E-05
Duplicate %RPD					**3.3%**			**4.7%**	
01-00430	01-00430 + spike	AN-102-CS-AR	Sample6&7	8.35E-01	4.43E+00 ±	3.02E+00	4.94E-05	4.23E-04 ±	2.76E-05
Spike Concentration					5.91E-05			1.60E-08	
Spike Recovery					**116%**			**92%**	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample5	6.37E-01	2.27E+00 ±	1.83E-01	7.25E-05	1.51E-04 ±	1.07E-05
01-00442	01-00442	AN-102-CS-DF	Sample8	1.87E-01	8.37E-01 ±	1.45E-01	2.13E-05	6.10E-05 ±	1.02E-05
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.82E-01	6.12E-01 ±	3.62E-02	2.07E-05	5.50E-05 ±	1.69E-06
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample9	1.80E-01	7.57E-01 ±	3.00E-02	2.05E-05	6.80E-05 ±	5.01E-06
Duplicate %RPD					**10.1%**			**10.8%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6-01-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Pt µg/g	±	1SD	MDL µg/g	Tl µg/g	±	1SD
	1%HNO3		ICB	<	1.11E-05			<	5.15E-06		
	1%HNO3		CCB1	<	1.06E-05			<	5.09E-06		
	1%HNO3		CCB2	<	1.10E-05			<	5.09E-06		
	1%HNO3		CCB3	<	9.58E-06			<	5.11E-06		
	ICV		ICV		2.19E-04	±	1.38E-05		1.92E-04	±	8.62E-06
	CCV		CCV1		2.13E-04	±	1.93E-05		2.06E-04	±	7.06E-06
	CCV		CCV2		1.92E-04	±	9.43E-06		1.80E-04	±	3.42E-06
	CCV		CCV3		1.93E-04	±	6.02E-06		2.26E-04	±	1.69E-06
True Value					**2.00E-04**				**2.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	5.48E-02 <	5.48E-02			2.62E-02 <	2.62E-02		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	5.64E-02	1.06E+00	±	2.31E-02	2.73E-02	1.02E+00	±	3.28E-02
Spike Concentration					2.00E-04				1.93E-04		
Spike Recovery					**100%**				**96%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.66E-02 <	5.66E-02			2.75E-02 <	2.75E-02		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	5.54E+00 <	5.54E+00			2.52E+00	5.75E+02	±	1.33E+01
Spike Concentration									1.09E-03		
Spike Recovery									**92%**		
01-00430	01-00430	AN-102-CS-AR	Sample9	4.41E-02 <	4.41E-02			2.24E-02 <	2.24E-02		
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample10	5.02E-02 <	5.02E-02			2.48E-02 <	2.48E-02		
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample11	4.52E-02 <	4.52E-02			2.22E-02 <	2.22E-02		
Duplicate %RPD											
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	8.19E-01	1.73E+01	±	2.51E-01	3.91E-01	4.31E+01	±	1.17E+00
Spike Concentration					2.05E-04				5.10E-04		
Spike Recovery					**102%**				**102%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	5.05E+00 <	5.05E+00			2.41E+00	5.79E+02	±	8.97E+00
Spike Concentration									1.09E-03		
Spike Recovery									**97%**		
01-00442	01-00442	AN-102-CS-DF	Sample12	3.20E-02 <	3.20E-02			1.54E-02	2.03E-02	±	5.88E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample13	3.17E-02 <	3.17E-02			1.58E-02	5.81E-02	±	5.08E-03
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample14	3.25E-02 <	3.25E-02			1.59E-02	4.80E-02	±	1.14E-02
Duplicate %RPD									**81.0%**		

Analytical Equipment: ICP/MS VG WB35913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
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Reveiwed by: MPD 6-1-01

Analyst: JPBrown 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g		Th µg/g	±	1SD	MDL µg/g		U µg/g	±	1SD
	1%HNO3		ICB		<	2.55E-06				<	1.92E-06		
	1%HNO3		CCB1		<	2.36E-06				<	1.78E-06		
	1%HNO3		CCB2		<	2.27E-06				<	1.71E-06		
	1%HNO3		CCB3		<	2.09E-06				<	1.58E-06		
	1%HNO3		CCB4		<	2.11E-06				<	1.59E-06		
	ICV		ICV			1.03E-04	±	5.35E-06			1.02E-04	±	1.97E-06
	CCV		CCV1			1.03E-04	±	4.47E-06			1.08E-04	±	5.71E-06
	CCV		CCV2			1.06E-04	±	1.98E-06			1.02E-04	±	2.16E-06
	CCV		CCV3			1.00E-04	±	1.89E-06			9.70E-05	±	1.81E-06
	CCV		CCV4			1.00E-04	±	3.72E-06			9.72E-05	±	1.94E-06
True Value						**1.00E-04**					**1.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	6.75E-01	<	6.75E-01			5.09E-01	<	5.09E-01		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	5.97E-01		3.02E+01	±	7.35E-01	4.51E-01		2.96E+01	±	4.03E-01
Spike Concentration						1.14E-04					1.12E-04		
Spike Recovery						**114%**					**112%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	6.92E-01	<	6.92E-01			5.23E-01	<	5.23E-01		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	5.73E-01		8.49E+01	±	4.71E+00	4.33E-01		7.43E+01	±	2.21E+00
Spike Concentration						3.21E-04					2.81E-04		
Spike Recovery						**81%**					**71%**		
01-00430	01-00430	AN-102-CS-AR	Sample6	4.30E-01		2.61E+01	±	1.28E+00	3.25E-01		6.70E+01	±	3.46E+00
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	5.56E-01		2.67E+01	±	1.27E+00	4.20E-01		6.39E+01	±	1.11E+00
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	4.45E-01		2.79E+01	±	5.90E-01	3.36E-01		6.47E+01	±	1.49E+00
Duplicate %RPD						**6.7%**					**3.5%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample14	4.25E-01		4.47E+01	±	7.80E-01	3.21E-01		8.73E+01	±	1.43E+00
Spike Concentration						8.79E-05					9.61E-05		
Spike Recovery						**88%**					**96%**		
01-00430-MS	01-00430-MS	AN-102-CS-AR	Sample12	6.03E+00		5.34E+02	±	5.97E+00	4.55E+00		6.60E+02	±	1.62E+01
Spike Concentration						1.92E-04					2.24E-04		
Spike Recovery						**102%**					**119%**		
01-00442	01-00442	AN-102-CS-DF	Sample9	1.39E+00		6.40E+01	±	1.21E+00	1.05E+00		2.80E+02	±	2.93E+00
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.40E+00		6.18E+01	±	7.10E-01	1.06E+00		2.77E+02	±	2.34E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	1.37E+00		6.89E+01	±	7.24E-01	1.03E+00		2.81E+02	±	7.43E+00
Duplicate %RPD						**7.4%**					**0.5%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
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Reveiwed by: 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	U233 µCi/g	± 1SD	MDL µCi/g	U234 µCi/g	± 1SD	MDL µCi/g	U235 µCi/g	± 1SD
	1%HNO3		ICB		< 3.23E-10			< 3.50E-10			< 1.55E-13	
	1%HNO3		CCB1		< 1.20E-10			< 1.58E-10			< 3.02E-14	
	1%HNO3		CCB2		< 1.13E-11			< 3.24E-11			< 4.29E-14	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	7.61E-06	< 7.61E-06		3.03E-05	< 3.03E-05			< 1.12E-08	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.67E-06	< 5.67E-06		6.75E-06	< 6.75E-06			< 1.59E-08	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	7.95E-06	< 7.95E-06		6.50E-06	< 6.50E-06		1.12E-08	4.32E-07 ±	2.21E-08
01-00430	01-00430	AN-102-CS-AR	Sample6	7.61E-06	6.13E-05 ±	1.21E-05	3.24E-05	< 3.24E-05		1.12E-08	9.89E-07 ±	3.72E-08
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	7.61E-06	5.07E-05 ±	8.95E-06	2.91E-05	< 2.91E-05		1.12E-08	9.84E-07 ±	3.12E-08
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	7.61E-06	4.77E-05 ±	7.17E-06	2.61E-05	< 2.61E-05		1.12E-08	1.01E-06 ±	3.62E-08
Duplicate %RPD					25.1%						1.6%	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	7.61E-06	1.14E-04 ±	1.81E-05	3.03E-05	8.60E-05 ±	1.38E-05	1.12E-08	4.45E-06 ±	1.90E-07
01-00442	01-00442	AN-102-CS-DF	Sample9	7.61E-06	1.09E-03 ±	5.88E-05	3.03E-05	1.33E-04 ±	2.06E-05	1.12E-08	4.47E-06 ±	8.62E-08
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	7.61E-06	1.15E-03 ±	6.85E-05	3.03E-05	1.20E-04 ±	5.14E-06	1.12E-08	4.33E-06 ±	1.55E-07
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	7.61E-06	1.06E-03 ±	5.67E-05	3.03E-05	1.20E-04 ±	1.85E-05	1.12E-08	4.44E-06 ±	8.53E-08
Duplicate %RPD					2.8%			10.9%			0.6%	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
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Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: tmPS 6·1·01

Analyst: JBrown 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	U236 µCi/g	± 1SD	MDL µCi/g	U238 µCi/g	± 1SD
	1%HNO3		ICB		< 2.52E-12			< 7.02E-13	
	1%HNO3		CCB1		< 1.07E-12			< 6.00E-13	
	1%HNO3		CCB2		< 2.91E-14			< 5.66E-13	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1		< 1.75E-07			< 1.67E-07	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2		< 1.27E-07			< 1.72E-07	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.75E-07	< 1.75E-07		1.67E-07	2.49E-05 ±	1.34E-06
01-00430	01-00430	AN-102-CS-AR	Sample6	1.75E-07	1.34E-06 ±	8.29E-08	1.67E-07	2.24E-05 ±	8.87E-07
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	1.75E-07	1.12E-06 ±	4.56E-08	1.67E-07	2.13E-05 ±	5.75E-07
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	1.75E-07	1.33E-06 ±	2.46E-07	1.67E-07	2.16E-05 ±	9.45E-07
Duplicate %RPD					**0.7%**			**3.5%**	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	1.75E-07	1.23E-06 ±	1.47E-07	1.67E-07	2.21E-04 ±	1.26E-05
01-00442	01-00442	AN-102-CS-DF	Sample9	1.75E-07	6.62E-06 ±	1.59E-07	1.67E-07	9.34E-05 ±	6.57E-07
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.75E-07	6.02E-06 ±	2.45E-07	1.67E-07	9.24E-05 ±	1.44E-06
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	1.75E-07	6.08E-06 ±	4.70E-07	1.67E-07	9.37E-05 ±	2.17E-06
Duplicate %RPD					**8.4%**			**0.4%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Revelwed by: mPD 6-1-01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	237Np µCi/g	± 1SD	MDL µCi/g	239Pu µCi/g	± 1SD	MDL µCi/g	240Pu µCi/g	± 1SD
	1%HNO3 (blanks)		ICB		< 1.58E-10			< 1.78E-08			< 4.37E-08	
	1%HNO3 (blanks)		CCB1		< 1.95E-10			5.90E-08 ±	5.76E-09		< 5.40E-08	
	1%HNO3 (blanks)		CCB2		< 1.37E-10			< 1.54E-08			< 3.79E-08	
	1%HNO3 (samples)		ICB		< 5.00E-10			< 7.99E-08			< 5.15E-08	
	1%HNO3 (samples)		CCB1		< 5.21E-10			< 8.32E-08			< 5.36E-08	
	1%HNO3 (samples)		CCB2		< 6.43E-10			< 1.03E-07			< 6.62E-08	
	1%HNO3 (samples)		CCB3		< 6.25E-10			< 9.98E-08			< 6.43E-08	
	1%HNO3 (samples)		CCB4		< 6.29E-10			3.83E-07 ±	3.88E-08		< 6.48E-08	
	1%HNO3 (samples)		CCB5		< 1.03E-09			4.34E-07 ±	3.61E-08		< 1.06E-07	
	ICV (blanks)		ICV		3.24E-08 ±	5.70E-10		3.22E-06 ±	1.94E-07		1.90E-07 ±	1.55E-08
	CCV (blanks)		CCV1		3.26E-08 ±	1.00E-09		3.04E-06 ±	4.15E-08		2.25E-07 ±	2.75E-08
	CCV (blanks)		CCV2		3.18E-08 ±	6.00E-10		2.64E-06 ±	6.37E-08		1.89E-07 ±	3.50E-09
	CCV (blanks)		CCV3		3.77E-08 ±	1.25E-09		3.17E-06 ±	5.08E-08		2.19E-07 ±	1.28E-08
True Value					**3.52E-08**			**3.06E-06**			**2.33E-07**	
	ICV (samples)		ICV		3.79E-08 ±	2.19E-09		3.39E-06 ±	3.91E-08		2.08E-07 ±	1.09E-08
	CCV (samples)		CCV1		3.58E-08 ±	1.51E-09		3.54E-06 ±	5.71E-08		2.46E-07 ±	3.15E-08
	CCV (samples)		CCV2		3.72E-08 ±	1.07E-09		3.27E-06 ±	1.11E-07		2.35E-07 ±	5.70E-08
True Value					**3.52E-08**			**3.06E-06**			**2.33E-07**	
	30ppb 4321B uranium (blanks)		4321b1	1.75E-10 <	1.75E-10		1.97E-08	7.82E-08 ±	2.87E-09	4.83E-08 <	4.83E-08	
	30ppb 4321B uranium (blanks)		4321b2	1.42E-10 <	1.42E-10		1.60E-08	3.46E-08 ±	1.52E-08	3.92E-08 <	3.92E-08	
	10ppb U030 uranium (samples)		U030e		< 1.06E-09			< 1.70E-07			< 1.10E-07	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB3	1.94E-06 <	1.94E-06		2.19E-04 <	2.19E-04		5.38E-04 <	5.38E-04	
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS2	2.20E-06	7.49E-04 ±	2.38E-05	2.48E-04	6.38E-02 ±	6.13E-04	6.09E-04	5.01E-03 ±	3.02E-05
Spike Concentration					7.07E-08			6.03E-06			4.73E-07	
Spike Recovery					**100%**			**99%**			**101%**	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB4	1.89E-06 <	1.89E-06		2.13E-04 <	2.13E-04		5.22E-04 <	5.22E-04	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.54E-06 <	1.54E-06		1.74E-04	2.23E-04 ±	1.91E-05	4.27E-04 <	4.27E-04	
01-00430	01-00430	AN-102-CS-AR	Sample11	5.64E-06	9.04E-04 ±	2.49E-05	9.01E-04	5.60E-02 ±	2.16E-03	5.81E-04	1.51E-02 ±	3.55E-04
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample12	8.10E-06	9.38E-04 ±	1.81E-05	1.29E-03	5.51E-02 ±	6.15E-04	8.34E-04	1.50E-02 ±	1.19E-03
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample13	6.13E-06	8.94E-04 ±	2.66E-05	9.78E-04	5.15E-02 ±	1.14E-03	6.31E-04	1.39E-02 ±	3.83E-04
Duplicate %RPD					**1.2%**			**8.4%**			**8.1%**	
01-00430	01-00430 + spike	AN-102-CS-AR	Sample15	6.51E-06	1.19E-03 ±	3.15E-05	1.04E-03	8.83E-02 ±	2.35E-03	6.70E-04	1.65E-02 ±	6.04E-04
Spike Concentration					3.38E-08			3.82E-06			1.61E-07	
Spike Recovery					**96%**			**126%**			**69%**	
01-00430-MS	01-00430-MS2	AN-102-CS-AR	Sample14	6.86E-06	7.33E-04 ±	1.04E-05	1.10E-03	2.93E-02 ±	8.93E-04	7.06E-04	7.55E-03 ±	5.50E-04
01-00442	01-00442	AN-102-CS-DF	Sample8	4.42E-05	6.30E-04 ±	3.08E-05	7.05E-03	1.43E-01 ±	2.89E-03	4.55E-03	3.88E-02 ±	4.01E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample9	4.24E-05	5.71E-04 ±	2.77E-05	6.77E-03	1.34E-01 ±	4.00E-03	4.37E-03	3.66E-02 ±	3.64E-04
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample10	4.07E-05	5.70E-04 ±	3.15E-05	6.50E-03	1.39E-01 ±	5.00E-03	4.19E-03	3.51E-02 ±	4.35E-03
Duplicate %RPD					**10.1%**			**2.7%**			**10.0%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Battelle – PNNL / AIAL
Inorganic Analysis / 320-ICP/MS Data Report

Project / WP#: 42040 / W59711
ASR#: 6025
Client: Mike Urie
Total Samples: 2

RPL #	Client ID
01-00441	AN-102-SUP-DF
01-00441 DUP	AN-102-SUP-DF

Procedure: PNL-SC-01 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

Analyst: Orville Thomas Farmer III

Report Written by: Orville Thomas Farmer III

Analysis Date(s): 9/23/01

Analysis Files:
Experiments – 21SEP01
Procedures – CS-ISO
Element Menus – CS-ISO

Laboratory Record Book (LRB):
LRB BNW 56465: Sample login: p.59
LRB BNW 56465: Cs Isotopics assay: p.59

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

9-25-01
Reviewed By

Concur
9-25-01

1. Analysis

Two liquid samples for Cs isotopic analysis were received from the client and were analyzed by HPIC-ICP/MS. All samples were diluted by a factor of 100 as received and screened to determine an estimate of the Cs concentration. The screening results were used to determine optimum Cs response during the on-line separation into the ICP/MS.

A Dionex HPIC system was configured on-line with the ICP/MS for the separation of Ba and Cs isotopes. A CS3 cation column was used as the stationary phase and a gradient eluent of nitric acid was used as the mobile phase for this separation. Data was collected using peak-hopping data acquisition parameters.

ICP/MS results for all solutions are reported in **atomic abundance** and are instrument blank corrected. In Table 1, reports results are in atomic abundance. In Table 2, the precision of the analysis was determined by analyzing three separate aliquots of a single unknown tank sample. These three solutions were analyzed to measure the separation and analysis procedures accuracy and precision.

Table 1: Cs Atomic Abundance

File Name	Cs-133 +/- 1 sigma	Cs-135 +/- 1 sigma	Cs-137 +/- 1 sigma
01-00441	0.603 +/- 0.002	0.166 +/- 0.002	0.231 +/- 0.001
01-00441 DUP	0.603 +/- 0.002	0.165 +/- 0.001	0.232 +/- 0.002

No Blanks were submitted for analysis.

Table 2: Cs Atomic Abundance Check solution

File Name	Cs-133 +/- 1 sigma	Cs-135 +/- 1 sigma	Cs-137 +/- 1 sigma
Check 1	0.6033 +/- 0.0023	0.1646 +/- 0.0008	0.2321 +/- 0.0020
Check 2	0.6044 +/- 0.0018	0.1634 +/- 0.0003	0.2322 +/- 0.0021
Check 3	0.6022 +/- 0.0023	0.1643 +/- 0.0002	0.2335 +/- 0.0021
Avg ratio	0.6033	0.1641	0.2326
STDEV	0.00089	0.00051	0.00064
%RSD	0.15	0.31	0.28

Currently there does not exist a known total Cs check sample and therefore a triplicate is used to measure precision of the procedure.

QC Summary:

Duplicate (DUP): A duplicate was submitted and analyzed. The duplicate RPD was determined to be 0.021% for Cs-133, 0.97% for Cs-135 and 0.64% for Cs-137.

Matrix Spike (MS): No post matrix spike was analyzed for this work.

Post Spike (PS): Not a QC requirement.

Preparation Blank (PB) and Laboratory Control Standard (LCS/BS): No blanks submitted.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB): Not a QC requirement.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): Not a QC requirement.

Internal Standard (ISTD): Not a QC requirement.

BLANK

Battelle

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Project No. 42040

Internal Distribution
File/LB

Date June 29, 2001

To M. W. Urie

From L. R. Greenwood LRGreenwood

Subject Radiochemical Analyses for AN-102 –ASR 6019, 25

Samples of the supernates and centrifuged solids from tank AN-102 were analyzed for gamma emitters, ^{90}Sr, total alpha, U, Pu, and Am/Cm according to ASRs 6019 and 6025. Sample AN-102-CS-AR was also analyzed for ^{3}H and ^{241}Pu. The samples were acid digested in the hot cells according to procedures PNL-ALO-128 and -129 and aliquots were delivered to the laboratory for analysis. The attached reports list measured analyte activities in the original sample material in units of μCi/ml for the supernates. The solids results are reported per gram of wet weight. Sample AN-102-CS was dried prior to analysis and the results were corrected for a wt% solids value of 35.1%. 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.

The hot cell sample preparations were performed in two separate batches, as indicated by the horizontal lines separating the results in the attached tables. However, all of the samples were initially run together in one batch in the laboratory. The hot cell process blanks prepared with each batch of samples showed significant alpha contamination, as discussed below.

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. Initially, the samples were diluted prior to gamma analysis. However, in order to meet the requested detection limits, direct aliquots of the hot cell preparations were counted for periods of 4 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. The AN-102 samples also showed the presence of ^{60}Co, ^{154}Eu, ^{155}Eu, and ^{241}Am in most of the samples. The MRQ values for extended counting time GEA were met in all cases except for ^{155}Eu for sample AN-102-SUP, where the detection limit was only slightly above the MRQ value of 9.E-2 μCi/ml. Detection limits are listed in the tables. All of the hot cell process blanks showed the presence of ^{137}Cs and some of them also showed ^{134}Cs. However, in all cases, the activities in the blanks were negligible with respect to the samples. Sample duplicates showed excellent repeatability with RPD or RSD values less than 10% except for ^{155}Eu and ^{241}Am for sample AN-102-SUP-DF where the RPD values are in reasonable agreement considering the counting uncertainties.

Total Alpha

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. The sums of the individual alpha emitters, as discussed below, are in very good agreement with the total alpha data indicating minimal losses due to alpha self-absorption. As mentioned previously, all of the hot cell preparation batches showed significant alpha contamination. Although this effect is not so evident in the total alpha data, the data for individual alpha emitters is compromised in some cases, as discussed in the following section. The LCS and matrix spike recoveries with ^{239}Pu were 103% and 100%, respectively. No alpha contamination was detected in the laboratory blank. RPD and RSD values are less than 3% in all cases.

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 excellent, averaging about 95%. Neither Pu, Am nor Cm were detected in the laboratory preparation blank. However, all of the hot cell process blanks indicated significant contamination for some of the isotopes. For the first hot cell batch with samples AN-102-SUP-AR and AN-102-SUP-DF, the process blank contamination is 20% and 100% of the sample activities, respectively, for ^{238}Pu, and 10% and 37% of the sample activities, respectively, for $^{243/244}$Cm. For the second hot cell batch with samples AN-102-CS-AR and AN-102-CS-DF, the process blank contamination levels are 5% and 12% of the sample activities, respectively, for $^{243/244}$Cm. For both batches, the process blank contamination levels are not significant for the other alpha emitting isotopes.

The LCS and matrix spike recoveries were 96 to 107% for both Pu and Am. RPD and RSD values were generally quite good except for ^{243,244}Cm in sample AP-101-DF, where the counting uncertainties are quite high.

The ^{241}Pu beta activity was measured in sample AN-102-CS-AR by placing the precipitation plates from the Pu/AEA measurements in glass scintillation vials, adding scintillation cocktail, and liquid scintillation counting according to procedure PNL-ALO-474. 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. Radiochemical yields were taken from the Pu/AEA tracer measurements. Blank spike and sample spike yields were determined in the Pu/AEA measurements and were not separately measured for ^{241}Pu. No ^{241}Pu was detected in the sample or duplicate and the detection limit was well below the MRQ value of 1.2 µCi/g. Weak ^{241}Pu activity was seen in the hot cell process blanks, although the level was negligible with respect to the MRQ value. No activity was seen in the laboratory 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). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. However, this correction was negligible with respect to the activity in the samples. No ^{90}Sr was found in the laboratory preparation blank. All of the hot cell process blanks showed ^{90}Sr activities, although the levels are not significant. RPD values are less than 7% in all cases indicating good sample reproducibility. The LCS and matrix spike recoveries were 88% and 91%, respectively.

Total Uranium

Total uranium was measured according to procedure PNNL-ALO-4014 using Kinetic Phosphoresence Analysis (KPA). Sample solutions from the hot cell were evaporated dry with nitric acid, then re-dissolved in dilute nitric acid for uranium analysis. No uranium separation was done. Results are reported in µg/ml of supernate. The uranium content is well below the MRQ value of 780 µg/ml. The repeatability of duplicate samples is excellent with RPD values of 2%. Uranium was detected in the hot cell blanks, but at a concentration less than 1% of the sample uranium concentration.

The instrument blanks had no detectable uranium. All the instrument standards except the lowest one gave results within 3% of their known values. The lowest standard is at 2.5 times the detection limit, and has an inherently higher uncertainty.

Tritium

Sample AN-102-CS-AR was analyzed in duplicate for tritium using a water leach performed in the hot cells. The water in the sample was separated by distillation, using procedure PNL-ALO-418, and the tritium was measured by liquid scintillation counting according to procedure PNL-ALO-474. Due to the anticipated high level of ^{137}Cs in this sample, two successive distillations were performed. In spite of this precaution, the beta energy spectrum clearly showed the presence of ^{137}Cs contamination. However, using the counts in the tritium energy region of the beta spectrum as an upper limit on the tritium activity results in detection limits of 2 to 3E-3 µCi/g, a factor of 5 or more below the requested MRQ value of 1.5E-2 µCi/g. The hot cell process blank shows weak tritium contamination well below the MRQ value. However, the process blank did not show any contamination from high-energy beta emitters. No beta activities were detected in the laboratory blank and the detection limit was < 2.E-4 µCi/g. The LCS and matrix spike tritium recoveries were excellent at 99% and 100%, respectively.

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 fractions were then counted according to procedure RPG-CMC-408. The samples were also counted by liquid scintillation counting according to procedure PNL-ALO-474 to confirm that the beta energy spectra matched that of ^{99}Tc and that no other beta emitters were present. The LCS blank spike recovery of a ^{99}Tc standard was 92%. However, a matrix spike gave a standard recovery of 248%. The reason for this high recovery is not known. Sample duplicates and lab replicates gave good agreement with RPD values less than 1%. The ^{99}Tc activities in the hot cell and laboratory blanks were negligible or non-detectable and well below the requested MRQ values.

Review:
C. Soderquist 6-29-01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurement Center

01-0429

8/23/01

Client : Mike Urie

Cognizant Scientist: LR Greenwood   Date : 8/23/01

Concur : T Trang-Le   Date : 8/23/01

PNL-ALO-476 (Sr-90) PNL-ALO-450 (GEA)

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID Client ID		Sr-90 Error %	Cs-134 Error %	Cs-137 Error %	Co-60 Error %	Sb-125	SnSb-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0429PB1		1.47E-2	4.58E-4	6.42E-3							
Process Blank		3%	16%	3%							
	MDA	<4.E-4	<2.E-4	<2.E-4	<3.E-4	<6.E-4	<2.E-4	<9.E-4	<6.E-4	<6.E-4	<6.E-4
01-0429PB2		4.47E-3		2.17E-3							
Process Blank		5%		7%							
	MDA	<4.E-4	<3.E-4	<3.E-4	<3.E-4	<5.E-4	<2.E-4	<9.E-4	<6.E-4	<6.E-4	<6.E-4
01-0429		5.58E+1		3.66E+2	8.58E-2				2.30E-1		
AN-102-SUP-AR		3%		2%	3%				3%		
	MDA	<7.E-1	<8.E-3	<5.E-2	<2.E-3	<2.E-1	<1.E-1	<9.E-3	<9.E-3	<1.E-1	<1.E-1
01-0429 DUP		5.86E+1		3.71E+2	8.39E-2				2.31E-1		1.65E-1
AN-102-SUP-AR		3%		2%	3%				3%		31%
	MDA	<7.E-1	<8.E-3	<5.E-2	<3.E-3	<2.E-1	<1.E-1	<8.E-3	<1.E-2	<1.E-1	<1.E-1
	RPD	5%		1%	2%				1%		
01-0441		1.41E+1		1.02E+2	2.55E-2				5.83E-2	4.00E-2	4.17E-2
AN-102-SUP-DF		3%		2%	2%				2%	10%	14%
	MDA	<4.E-1	<1.E-3	<7.E-3	<4.E-4	<3.E-2	<1.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
01-0441 DUP		1.34E+1		1.02E+2	2.53E-2				5.80E-2	3.53E-2	3.22E-2
AN-102-SUP-DF		3%		2%	2%				2%	10%	16%
	MDA	<4.E-1	<1.E-3	<7.E-3	<3.E-4	<3.E-2	<1.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
	RPD	5%		0%	1%				1%	12%	26%
01-0441 REP		1.33E+1									
AN-102-SUP-DF		3%									
	MDA	<4.E-1									
01-0430PB1		7.84E-2		7.32E-3							
Process Blank		3%		6%							
	MDA	<2.E-3	<5.E-4	<6.E-4	<4.E-3	<1.E-3	<5.E-4	<2.E-3	<2.E-3	<2.E-3	<1.E-3
01-0430PB2		4.59E-2	9.60E-4	2.48E-2							
Process Blank		3%	13%	3%							
	MDA	<2.E-3	<4.E-4	<5.E-4	<4.E-3	<1.E-3	<5.E-4	<2.E-3	<2.E-3	<2.E-3	<2.E-3
01-0430		1.40E+2		2.16E+2	5.76E-2				5.08E-1	3.14E-1	4.12E-1
AN-102-CS-AR		3%		2%	3%				2%	9%	12%
	MDA	<1.E+0	<6.E-3	<4.E-2	<3.E-3	<2.E-1	<7.E-2	<1.E-2	<9.E-3	<2.E-1	<2.E-1
01-0430 DUP		1.47E+2		2.16E+2	5.66E-2				5.15E-1	3.26E-1	4.29E-1
AN-102-CS-AR		3%		2%	4%				2%	10%	12%
	MDA	<2.E+0	<6.E-3	<4.E-2	<4.E-3	<2.E-1	<8.E-2	<1.E-2	<9.E-3	<2.E-1	<2.E-1
	RPD	5%		0%	2%				1%	4%	4%
01-0442**		4.31E+2		8.49E+1	2.07E-2			2.15E-2	1.22E+0	7.27E-1	1.16E+0
AN-102-CS-DF		3%		2%	2%			5%	2%	3%	4%
	MDA	<7.E+0	<2.E-3	<6.E-3	<6.E-4	<2.E-2	<1.E-2	<3 E-3	<3.E-3	<2.E-2	<2.E-2
01-0442 DUP**		4.02E+2		8.39E+1	2.11E-2			2.18E-2	1.21E+0	7.16E-1	1.15E+0
AN-102-CS-DF		3%		2%	2%			5%	2%	3%	4%
	MDA	<7.E+0	<2.E-3	<6.E-3	<7.E-4	<2.E-2	<1.E-2	<3.E-3	<3.E-3	<2.E-2	<2.E-2
	RPD	7%		1%	2%			2%	1%	1%	0%
Sample spike		88%									
Blank spike		91%									
Blank		<1.E-4									

*Supernates are reported per ml and solids are reported per gram.

**Sample AN-102-CS-DF is reported per gram of wet weight using a value of 35.1% wt% solids.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurement Center

01-0429

08/20/01

Client : Mike Urie

Cognizant Scientist: L R Greenwood Date : 8/20/01

Concur : T Trang-le Date : 8/23/01

RPG-CMC-408 (Total Alpha)
PNL-ALO-417,496 (Pu, Am/Cm)

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID Client ID		Alpha Error %	Pu-238 Error %	Pu-239/240 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	Sum of Alpha Error %
01-0429PB1		1.10E-3	3.20E-4	4.07E-5	1.06E-4	5.71E-4		1.04E-3
Process Blank		5%	3%	6%	6%	3%		4%
	MDA	<6.E-5	<2.E-6	<2.E-6	<4.E-6	<4.E-6	<2E-6	
01-0429PB2		3.60E-4	9.49E-5	1.67E-5	4.45E-5	1.56E-4		3.12E-4
Process Blank		10%	3%	6%	4%	3%		4%
	MDA	<6.E-5	<6.E-7	<5.E-7	<1.E-6	<1.E-6	<5E-7	
01-0429		1.66E-1	1.66E-3	6.00E-3	1.49E-1	6.51E-3	6.53E-4	1.64E-1
AN-102-SUP-AR		4%	14%	6%	2%	6%	20%	3%
	MDA	<5.E-3	<3.E-4	<2.E-4	<2.E-4	<2.E-4	<2.E-4	
01-0429 DUP		1.66E-1	1.63E-3	5.80E-3	1.52E-1	6.91E-3	6.04E-4	1.67E-1
AN-102-SUP-AR		4%	11%	5%	2%	7%	23%	3%
	MDA	<6.E-3	<1.E-4	<1.E-4	<3.E-4	<2.E-4	<2.E-4	
	RPD	0%	2%	3%	2%	6%	8%	2%
01-0441		4.60E-2	6.75E-4	1.40E-3	3.83E-2	2.26E-3	1.79E-4	4.28E-2
AN-102-SUP-DF		7%	14%	9%	2%	6%	23%	3%
	MDA	<4.E-3	<1.E-4	<9.E-5	<2.E-4	<7.E-5	<7.E-5	
01-0441 DUP		4.64E-2	3.03E-4	1.58E-3	3.66E-2	1.57E-3	1.30E-4	4.02E-2
AN-102-SUP-DF		8%	26%	8%	2%	8%	28%	3%
	MDA	<6.E-3	<2.E-4	<9.E-5	<2.E-4	<7.E-5	<6.E-5	
	RPD	1%	76%	12%	5%	36%	32%	6%
01-0430PB1		3.92E-3	1.27E-3	1.52E-4	3.33E-4	1.97E-3		3.73E-3
Process Blank		4%	2%	3%	5%	3%		4%
	MDA	<2.E-4	<2.E-6	<2.E-6	<5.E-6	<5.E-6	<4E-6	
01-0430PB2		3.11E-3	8.59E-4	9.49E-5	2.15E-4	1.61E-3		2.78E-3
Process Blank		4%	2%	4%	6%	3%		4%
	MDA	<2.E-4	<2.E-6	<2.E-6	<6.E-6	<5.E-6	<4E-6	
01-0430		4.93E-1	1.09E-2	4.16E-2	4.44E-1	1.81E-2		5.15E-1
AN-102-CS-AR		3%	11%	5%	3%	10%		4%
	MDA	<8.E-3	<2.E-3	<2.E-3	<2.E-3	<2.E-3	<1E-3	
01-0430 DUP		5.06E-1	1.29E-2	4.17E-2	4.38E-1	1.62E-2		5.09E-1
AN-102-CS-AR		3%	11%	6%	3%	11%		4%
	MDA	<2.E-2	<2.E-3	<2.E-3	<3.E-3	<2.E-3	<2E-3	
	RPD	3%	17%	0%	1%	11%		1%

Battelle Pacific Northwest Laboratory 01-0429
Radiochemical Processing Group-325 Building
Chemical Measurement Center 08/20/01

Client : Mike Urie

Cognizant Scientist: LRGreenwood Date : 8/20/01

Concur : T Trang-le Date : 8/26/01

PNL-ALO-432 (Tc-99)

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID		Pertechnetate Tc-99 Error %	MDA
01-0429 CB Process Blank			<2.E-6
01-0429 AN-102-SUP-AR		5.55E-2 4%	<6.E-6
01-0441 AN-102-SUP-DF		2.20E-2 4%	<4.E-6
01-0441 Rep AN-102-SUP-DF		2.17E-2 4%	<4.E-6
	RPD	1%	
Sample spike		248%	
Blank spike		92%	
Blank		7.89E-06 23%	<6.E-6

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID		Alpha	Pu-238	Pu-239/240	Am-241	Cm-243/244	Cm-242	Sum of Alpha
Client ID		Error %	Error %	Error %	Error %	Error %	Error %	Error %
01-0442**		1.35E+0	2.61E-2	1.16E-1	1.08E+0	6.80E-2	3.52E-3	1.29E+0
AN-102-CS-DF		2%	6%	3%	2%	3%	14%	3%
	MDA	<6.E-3	<7.E-4	<8.E-4	<5.E-4	<4.E-4	<4.E-4	
01-0442 DUP**		1.33E+0	2.64E-2	1.08E-1	1.08E+0	4.32E-2	2.48E-3	1.26E+0
AN-102-CS-DF		2%	6%	3%	2%	5%	22%	3%
	MDA	<7.E-3	<6.E-4	<5.E-4	<1.E-3	<9.E-4	<7.E-4	
	RPD	2%	1%	7%	0%	45%	35%	3%
Sample spike		103%		105%	97%			
Blank spike		100%		107%	96%			
Blank		<4.E-3	<6.E-6	<6.E-6	<8.E-6	<6.E-6	<4.E-6	

*Supernates are reported per ml and solids are reported per gram.

**Sample AN-102-CS-DF is reported per gram of wet weight using a value of 35.1% wt% solids.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurement Center

01-0429

08/27/01

Client : Mike Urie

Cognizant Scientist: LR Greenwood Date : 8/27/01

Concur : C. Soderquist Date : 8.27.01

RPG-CMC-4014 (Uranium KPA)

Measured Activities (ug/ml) with 1-sigma error

ALO ID Client ID	Uranium Error %	MDL
01-0429PB1 Process Blank	6.08E-2 2%	6.E-3
01-0429PB2 Process Blank	3.23E-2 2%	6.E-3
01-0429 AN-102-SUP-AR	1.15E+1 5%	6.E-3
01-0429 DUP AN-102-SUP-AR	1.23E+1 5%	6.E-3
RPD	7%	
01-0441 AN-102-SUP-DF	1.44E+1 4%	5.E-3
01-0441 Rep AN-102-SUP-DF	1.38E+1 4%	5.E-3
01-0441 DUP AN-102-SUP-DF	1.55E+1 4%	5.E-3
RSD	6%	
Lab Blank	<6.E-3	

Instrument Standards

	Observed	Expected	Spike Yield
Blank 1	5.05E-6	-	
Blank 2	0.00E+0	-	
Blank 3	1.30E-5	-	
IV 5.E-1	5.07E-1	5.05E-1	100%
IV 5.E-1	5.07E-1	5.05E-1	100%
*IV 5.E-5	6.20E-5	5.07E-5	122%

*This standard is near the detection limit, where uncertainty is higher.
Sample solutions ranged from 1.06E-4 to 6.22E-2 µg/mL

BLANK

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	M Urie	Charge Code/Project:	F22616-F22626/42040
ASR Number:	6019, 6025 Supernatants	Sample Receipt Date:	6019 - 00/29/2001 6025 – 01/23/2001
Sample Prep Date:	N/A	Sample Analysis Date:	04/03-27/2001 (01-00429 Rerun) 05/10/2001
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.

Raw data, standards information, preparation benchsheets filed with ASR 6019 Urie: Project 42365

Samples 01-00429, 01-00441 Final Results:

RPL Number	Sample ID	F[1] µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	0.25	0.25	0.50	0.25	0.500	0.500	0.500	0.500
01-00429 PB	Hot Cell Blank	< 0.25	< 0.25	< 0.50	< 0.25	< 0.50	< 0.50	< 0.50	< 0.50
	EQL	*2,500*	*2,500*	*5,000*	*2,500*	*5,000*	*5,000*	*5,000*	*5,000*
01-00429	*AN-102-SUP-AR*	*< 2500*	*8,560*	*19,100*	*< 2500*	*404,000*	*< 5000*	*32,800*	*< 5000*
01-00429 Dup	*AN-102-SUP-AR*	*< 2500*	*5,080*	*96,100*	*< 2500*	*233,000*	*< 5000*	*18,500*	*< 5000*
	RPD	*n/a*	*36*	*47*	*n/a*	*38*	*n/a*	*40*	*n/a*
01-00429 MS % Rec	*AN-102-SUP-AR MS*	*136*	*108*	*87*	*95*	*132*	*102*	*91*	*102*
	EQL	2,500	2,500	5,000	2,500	5,000	5,000	5,000	5,000
Rerun 01-00429	AN-102-SUP-AR	< 2500	4,780	85,900	< 2500	223,000	< 5000	16,800	< 5000
Rerun 01-00429 Dup	AN-102-SUP-AR	< 2500	4,860	84,800	< 2500	219,000	< 5000	16,900	< 5000
	RPD	n/a	2	1	n/a	2	n/a	1	n/a
Rerun 01-00429 MS %Rec	AN-102-SUP-AR MS	96	94	104	96	115	95	93	102
	EQL	200	200	500	250	2,000	200	400	400
01-00441	AN-102-SUP-DF	3,700	1,170	25,900	< 250	61,400	< 200	4,470	2,300
01-00441 Dup	AN-102-SUP-DF	3,700	1,130	25,800	< 250	60,600	< 200	4,390	2,270
	RPD	0	3	1	n/a	1	n/a	1	1
LCS %Rec	60/160 Spike Solution	107	105	105	106	101	104	102	107

n/a = not applicable since either or both the sample and duplicate results are not above the EQL.

[1] The 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), possibly formate or acetate.

The sample was prepared for ion chromatography anion analysis by dilution from 800-fold to 10,000-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample as dilutions less than 800-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the table. Poor reproducibility of the AN-102 supernatant analysis conducted April 3 to April 27 prompted the need for reanalysis. The AN-102 sample, duplicate, and matrix spike were reanalyzed on May 10, with the reanalysis providing good QC and reasonable comparison to the original AN-102 duplicate. The 'Rerun' results are considered the most reliable, and essentially meet all QC criteria.

Q.C. Comments:

Duplicates: The samples AN-102 Sup AR and AN-102 Sup DF were sub-sampled in the hot cell. The samples and duplicates were aliquotted at the IC workstation. The initial AN-102 Sup AR sample and duplicate analysis did not meet the RPD acceptance criteria of < 20%. The results appeared to differ by about a factor of two, suggesting the possibility of a dilution error. Additional sample and duplicate samples for AN-102 Sup AR were prepared and analyzed. The RPD for the reanalysis meets the acceptance criteria of <20%.

Matrix Spike (010207 MS High, 60/160): A matrix spike was prepared from each sample. The fluoride and nitrate matrix spikes for the initial AN-102 Sup AR sample produced very high recoveries (i.e., 36% and 36%, respectively). Like the sample and duplicate, the matrix spike for the AN-102 Sup AR sample was prepared again and reanalyzed. The AN-102 Sup DF and the reanalyzed AN-102 Sup AR matrix spiked had anion recoveries were within the 75% to 125% recovery acceptance criteria.

Laboratory Control Sample-LSC/BS (100207 MS High, 60/160): 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 within the 80% to 120% acceptance criteria.

System Blank/Processing Blanks: Thirty-four system blanks were analyzed throughout the analysis runs (including the reruns). No anions were detected in the system blanks above the estimate quantitation level. Also, no anions were detected in the hot cell blank (01-00429).

Quality Control Calibration Verification Check Standards: Twenty-two mid-range verification standards were analyzed throughout the analysis runs (including the reruns). Except for one chloride measurement and seven oxalate measurement, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The chloride measurement that failed produced a recovery of 89% and the oxalate measurements produced recoveries of from 110% to 112%.

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 Urie Date 6-18-01

Review/Approval: MJ Steele Date 7-19-01

Archive Information:

Files: ASR 6019 6025 Sup Urie.doc ASR 6014 6019 6025 6031 6057.xls

Battelle
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Date: 03/20/01

Subject: Hydroxide Analyses for: AN-102 composite
ASR: 6019.01
ASR: 6025

To: Mike Urie

From: Bob Swoboda 3/22/01

As recieved composites from tank **AN-102 Supernatant (01-00429)** and from **AN-102 Diluted Feed Supernatant (01-00441)** were analyzed in duplicate or triplicate 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 solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. The duplicate results on 01-00429 gave an average OH molarity of 0.25, which equals 4.23E+3 ug/ml, with a standard deviation of 25%. The triplicate results on 01-00441 gave an average OH molarity of 0.28, which equals 4.56E+3 ug/ml, with a relative standard deviation of 9%. The RSD value for sample 01-00429 is higher than the QC acceptance criteria of 15%; however, the hydroxide level is rather low in these samples at only 6% of the MRQ value. The standard recovery averaged 98% and a sample spike recovered at 97%, thus satisfying the QC acceptance criteria. No hydroxide was detected in the cell blank or reagent blank. Second and third inflection points, which are frequently associated with carbonate and bicarbonate, were detected in sample 01-00429 at 1.92 moles with a standard deviation of 3% and 1.20 moles with a standard deviation of 10%. For sample 01-00441, these points were at 0.76 moles, with a standard deviation of 14%, and at 0.56 moles, with a standard deviation of 8%. The titration curves are included with the report.

Reviewer: JRGreenwood 3/22/01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6019.01

WP# k88426

Hydroxide and Alkalinity Determination
Procedure: PNL-ALO-228 Equip # WB76843

Analyst: 3/22/01

Reviewer: 3-22-01

Report Summary

			Concentration, moles		
RPG #	Client ID		First Point	Second Point	Third Point
01-0429-CB	Cell Blank		0	0	0
01-0429	AN-102-SUP-AR		0.28	1.89	1.26
01-0429	AN-102-SUP-AR	Rep	0.22	1.94	1.13
		RPD	25%	3%	10%
Average hydroxide concentration in ug/ml			4.23E+3		
01-0441	AN-102-SUP-DF		0.25	0.81	0.54
01-0441	AN-102-SUP-DF	Rep	0.30	0.70	0.58
01-0441	AN-102-SUP-DF	Trip.	0.26	0.76	0.55
		RSD	9%	7%	4%
Average hydroxide concentration in ug/ml			4.56E+3		
Reagent Blank			0		
Standard 1			98%		
Standard 2			97%		
MS 01-0441	Matrix spike		97%		

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.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client: M. Urie
RPL Numbers: 01-0429/0430 & 01-0441
Analyst: MJ Steele

Charge Code/Project: W57911 / 42365
ASR Number: 6019 and 6025
Analysis Date: 7/24, 8/01, 8/22, & 8/23 2001

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).

AN-102 As-Received and AN-102/C-104 Diluted Feed Supernatant Results

RPL Number	Sample ID	TOC MDL µgC/mL	TOC µgC/mL	TOC RPD %	TC MDL µgC/mL	TC µgC/mL	TC RPD %	TIC MDL (b) µgC/mL	TIC (c) µgC/mL
01-00429	AN102-SUP AR	140	18,200		140	41,400		140	23,200
01-00429 Dup	AN102-SUP AR	60	17,900	2%	120	40,800	1%	120	22,900
01-00429 MS	AN102-SUP AR		(a)			114%			
01-00441	AN102-SUP DF	140	15,800		140	16,000		140	200
01-00441 Dup	AN102-SUP DF	60	15,700	1%	60	16,100	1%	60	400
01-00441 MS	AN102-SUP DF		85%			95%			
BS/LCS	Blank Spike		97%			99%			

(a) 01-00441 MS is the analysis batch MS QC sample for the TOC determination

(b) TIC MDL set to TC MDL

(c) TIC results obtained by difference (TC – TOC): analysis performed on two independent sample aliquots

AN-102 As-Received Wet Centrifuged Solids Results

RPL Number	Sample ID	TOC MDL µgC/g	TOC µgC/g	TOC RPD %	TC MDL µgC/g	TC µgC/g	TC RPD %	TIC MDL (b) µgC/g	TIC (c) µgC/g
01-00430	AN102-CS AR	300	17,400		560	35,900		560	18.500
01-00430 Dup	AN102-CS AR	390	21,900	23%	200	36,000	0%	200	14,100
01-00430 MS	AN102-CS AR		100% (a)			82%			
BS/LCS	Blank Spike		99%			104%			

(a) Sample 01-01017MS is the analysis batch MS QC sample for the TOC Determination.

(b) TIC MDL set to TC MDL

(c) TIC results obtained by difference (TC – TOC); analysis performed on two independent sample aliquots

The TOC/TIC analyses of the samples submitted under ASRs 6019 and 6025 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 750 °C for 30 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 30 minutes. The total inorganic carbon is obtained by difference.

The tables 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-380.

For the centrifuged solids, the TOC and TC analysis were only performed on the AN-102 CS AR, since there was insufficient AN-102 CS DF available for analysis. Per the client's request the AN-102 CS DF TOC/TC analysis was cancelled.

Q.C. Comments:

The calibration and QC standards for TC and TOC analysis are liquid or solid carbon standards or pure chemicals from Ricca, Aldrich, Sigma, Mallinckrodt and VWR. The identification of the standards and their Chemical Management System (CMS) numbers and expiration dates are included on the raw data benchsheets.

The coulometer analysis system calibration is checked by analyzing calibration standards at the beginning, middle, and end of the analysis run. The average recovery from these calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for each of the four analysis days was 92%, 98%, 99%, and 100%.

System blanks were analyzed similarly to the calibration check, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The TOC determination produced average blanks of 5 and 15 µC. The TC determination produced average blanks of 4 and 55 µC. The 55 µC blank level is unusually high; however, the reproducibility of the blank was reasonably good (i.e., 49 to 59 µC) and the magnitude of the blank is less than 5% of the sample results (i.e., samples produced 1600 to 3100 µC for the samples sizes analyzed).

For each days analysis run, the QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes.

Blank Spike/Laboratory Control Sample: The BS/LCS was within acceptance criteria of 80% to 120% required by the client's Test Specification for both the TC and TOC analysis of the supernatant and solids.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is acceptable for the supernatant samples. The TC and TOC supernatant RPDs meet the acceptance criteria of <15% established by the client's Test Specification. However, the TOC RPD for the AN-102 as-received centrifuged solids was outside the acceptance criteria. The reason for the poor RPD on the wet centrifuged solids may be attributed to heterogeneity of small sub-samples used for the analysis.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes (for AN-102 SUP AR, AN-102 SUP DF, and AN-102 CS AR) demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery. Matrix spikes were not prepared for every AN-102 sample for both the TC and TOC analyses; however, when a matrix spike was not prepared from the AN-102 samples, a matrix spike from other samples in the batch was analyzed per the QA plan requirements.

Furnace Results Compared to Hot Persulfate Results

		TIC		TOC		TC	
		Persul.	Furn. (a)	Persul.	Furn.	Persul. (b)	Furn.
RPL Number	Sample ID	µgC/mL	µgC/mL	µgC/mL	µgC/mL	µgC/mL	µgC/mL
01-0429	AN102-SUP-AR	10,900	23,200	29,400	18,200	40,300	41,400
01-0429 Dup	AN102-SUP-AR	11,000	22,900	29,100	17,900	40,000	40,800
01-0441	AN102-SUP-DF	6,000	200	9,300	15,800	15,300	16,000
01-0441 Dup	AN102-SUP-DF	6,000	400	9,700	15,700	15,700	16,100
		µgC/g	µgC/g	µgC/g	µgC/g	µgC/g	µgC/g
01-0430	AN-102-CS-AR	17,100	18,500	25,900	17,400	43,000	35,900
01-0430 Dup	AN-102-CS-AR	17,600	14,100	25,300	21,900	42,800	36,000

Persul. = Results from hot persulfate method

Furn. = Results from furnace combustion method – 750 °C for TOC and 1000 °C for TC

(a) Results are obtained by difference (TC – TOC); analysis performed on two independent sample aliquots

(b) Results are obtained by summation (TIC + TOC); analyses performed on same sample aliquot

The two methods appear to produce comparable results for TC for the AN-102 supernatants; however, there is about a 15% difference between the TC results for the AN-102 centrifuged solids, with the furnace producing the lower results. However, there are significant differences between the TIC and TOC results reported by each method. Based on the AN-102 as-received results, the persulfate results appear to have a higher likelihood of being accurate; that is, it is entirely plausible that there are organic compounds in both the solids and the supernatant that are difficult to oxidize at 750 °C, leading to low TOC and high TIC for the furnace method. For the AN-102 diluted with washes and leaches from C-104, the reason for the discrepancy between the persulfate method and furnace method is unknown, but it appears that the inorganic carbon, perhaps in the form of easily oxidized metal carbonate, is being combusted at 750 °C with the furnace method.

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** 9/14/01

Review/Approval by: ______________________ **Date** 9/14/01

Raw Data Calculation/Archive Information: File: ASR 6019L&S 6025L 6104L 6015L.xls

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie	Temp.	Balance M&TE: 360-06-01-023
Project :	42040	1000 Degree C	TOC Cal Std: VWR CMS#161356 0.10% Carbon <<[G]
Work Pkg:	CMC	Run time	TIC Cal Std: VWR CMS#161355 0.10% Carbon <<[G]
Analyzed:	July 24, 2001 MJ Steele 9/12/01	30 Minutes	LCS/BS/MS TOC Std: RICCA CMS#161730 0.10% Carbon <<[C]
ASR:	6019.01, 6025, 6031		LCS/BS/MS TIC Std: RICCA CMS#161734 0.10% Carbon <<[C]

		Raw TC (ug C)	
Blanks:	Calibration blank (start of batch)	4.6	
	Calibration blank (start of batch)	5.2	
	Calibration blank	3.7	
	Calibration blank (end of batch)	4.2	

4.4	<<< Blank Average (ug C)
0.6	<<< Blank Std Dev (ug C)
4	<<< *# of Blanks analyzed*
2.4	<<< *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)	447.5	4	0.50	88.6	0.25 ml TIC and TOC Cal Std
	Calibration Standard (start of batch)	467.3	4	0.50	92.6	0.25 ml TIC and TOC Cal Std
	Calibration Standard	237.4	4	0.25	93.2	0.25 ml TOC Cal Std
	Calibration Standard (end of batch)	468.3	4	0.50	92.8	0.25 ml TIC and TOC Cal Std
QC	Blank Spike/LCS	457.2	4	0.50	98.7	0.25 ml TIC and TOC LCS/BS/MS Std

91.8	<<< [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".

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie	Temp. 1000 Degree C	Balance M&TE: 360-06-01-023
Project :	42040		TOC Cal Std: VWR CMS#161356 0.10% Carbon <<[G]
Work Pkg:	CMC	Run time	TIC Cal Std: VWR CMS#161355 0.10% Carbon <<[G]
Analyzed:	July 24, 2001	30 Minutes	LCS/BS/MS TOC Std: RICCA CMS#161730 0.10% Carbon <<[C]
ASR:	6019.01, 6025, 6031		LCS/BS/MS TIC Std: RICCA CMS#161734 0.10% Carbon <<[C]

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)	Vol. (ml)	(ug C/ml)	RPD (%)	
01-00429	AN102-SUP AR	1903	4	0.050	41,368		
01-00429 Dup	AN102-SUP AR	940	4	0.025	40,771	1	
01-00429 MS	AN102-SUP AR	1465	4	0.025	see below		0.25 ml TOC and TIC Std
01-00441	AN102-SUP DF	738	4	0.050	15,984		
01-00441 Dup	AN102-SUP DF	742	4	0.050	16,071	1	
01-00441 MS	AN102-SUP DF	960	4	0.050	see below		0.25 ml TOC Std
01-00520PB	Hot Cell Blank	15	4	0.500	23		
01-00520PB Du	Hot Cell Blank	10	4	0.500	11	n/a	
01-00520A	AP101-DF-A	1353	4	0.200	7,346		
01-00520A Du	AP101-DF-A	1355	4	0.200	7,357	0	
01-00520B	AP101-DF-B	689	4	0.100	7,458		
01-00520B Du	AP101-DF-B	700	4	0.100	7,578	2	
01-00520B MS	AP101-DF-B	1165	4	0.100	see below		0.25 ml TOC and TIC Std

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
01-00429 MS	Total Carbon Recovery (TC)	1465	4	40771	0.03	1019	0.5000	500	**114.4**
01-00441 MS	Total Carbon Recovery (TC)	960	4	16071	0.05	804	0.2500	250	**95.0**
01-00520B MS	Total Carbon Recovery (TC)	1165	4	7578	0.10	758	0.5000	500	**101.3**

Reviewer/date: MW Urie 9-12-01

PNNL Radiochemical Processing Group: TOC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie	Temp. 750 Degree C	Analyzer M&TE: WD13071 -- 701 Balance M&TE: 360-06-01-023
Project :	42040		
Work Pkg:	CMC	Run time 30 Minutes	Cal Std: VWR CMS#161356 0.10% Carbon <<[G]
Analyzed:	August 1, 2001 MJ Steele 9/12/01		BS MS Std: RICCA CMS#161730 0.10% Carbon <<[C]
ASR:	6019.01, 6025		

		TOC (ug C)	
Blanks:	Calibration blank (start of batch)	3.6	
	Calibration blank (start of batch)	6.5	
	Calibration blank (end of batch)	6.0	

5.4	<<< Blank Average (ug C)
1.6	<<< Blank Std Dev (ug C)
3	<<< *# of Blanks analyzed*
7	<<< ***Method Det. Limit (ug C)*** [M]

		TOC			
		[A] Raw	[B]	[D] Std	TOC
		TOC (ug)	Blk (ug)	Vol (ml)	% Rec
Standards:	Calibration Standard (start of batch)	206	5	0.20	100.3
	Calibration Standard (start of batch)	203	5	0.20	98.8
	Calibration Standard (end of batch)	199	5	0.20	96.8
QC	Blank Spike/LCS	196	5	0.20	96.6

98.7 <<< [L] Average TOC % Rec

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 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".

Sample Results		[I] Raw	[J]	[K] Sam	TOC	
RPL Number	Sample ID	TOC (ug C)	Blk (ug C)	Vol. (ml)	(ug C/ml)	RPD (%)
01-00429	AN102-SUP AR	905	5	0.050	18,239	
01-00429 Dup	AN102-SUP AR	890	5	0.050	17,935	2
01-00441	AN102-SUP DF	785	5	0.050	15,806	
01-00441 Dup	AN102-SUP DF	778	5	0.050	15,664	1
01-00441 MS	AN102-SUP DF	1030	5	0.050	see below	

0.3 ml TOC Std

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	Client Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol. (ml)	(ug C)	Vol (ml)	(ug C)	% Recovery
01-00441 MS	Total Organic Carbon Recovery	1030	5	15664	0.05	783	0.30	300	85.1

Reviewer/date: 9-12-01

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client:	M. Urie	**Charge Code/Project:**	W57911 / 42365
RPL Numbers:	01-0429/0430 & 01-0441	**ASR Number:**	6019 and 6025
Analyst:	MJ Steele	**Analysis Date:**	July 24, 2001

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).

AN-102 As-Received and AN-102/C-104 Diluted Feed Supernatant Results

RPL Number	Sample ID	TIC MDL µg C/mL	TIC µg C/mL	TIC RPD (%)	TOC MDL µg C/mL	TOC µg C/mL	TOC RPD (%)	TC µg C/mL	TC RPD (%)
01-0429	AN102-SUP-AR	33	10,900		87	29,400		40,300	
01-0429 Dup	AN102-SUP-AR	65	11,000	1%	170	29,100	1%	40,000	1%
01-0429 MS %Rec	AN102-SUP-AR		101%			92%		95%	
01-0441	AN102-SUP-DF	33	6,000		87	9,300		15,300	
01-0441 Dup	AN102-SUP-DF	33	6,000	0%	87	9,700	4%	15,700	3%
01-0441 MS %Rec	AN102-SUP-DF		99%			98%		98%	
BS/LCS %Rec	Blank Spike		100.3%			104.4%			

AN-102 As-Received Wet Centrifuged Solids Results

RPL Number	Sample ID	TIC MDL µg C/g	TIC µg C/g	TIC RPD (%)	TOC MDL µg C/g	TOC µg C/g	TOC RPD (%)	TC µg C/g	TC RPD (%)
01-0430	102-AN-CS-AR	66	17,100		180	25,900		43,000	
01-0430 Dup	102-AN-CS-AR	76	17,600	3%	200	25,300	2%	42,800	0%
01-0430 MS %Rec	102-AN-CS-AR		44%			160%		95%	
01-0430 MSD %Rec	102-AN-CS-AR		75%			119%		94%	
BS/LCS %Rec	Blank Spike		100%			101%			

The TOC/TIC analyses of the samples submitted under ASRs 6019 and 6025 were 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 tables 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.

TIC/TOC analysis was only performed on the AN-102 CS AR, since there was insufficient AN-102 CS DF available for TIC/TOC analysis. Per the client's request the AN-102 CS DF TIC/TOC analysis was cancelled.

Q.C. Comments:

The standards for TIC and TOC supernatant analysis are liquid carbon standards from Ricca Chemical and VWR, respectively. The lot numbers and expiration dates are included on the raw data benchsheets.

For TIC and TOC solids analysis pure chemical compounds are used as the calibration, as well as matrix spiking, standards. The TIC analysis uses calcium carbonate and the TOC uses α-Glucose (JT Baker and Aldrich lot numbers are provided on the raw data benchsheets).

The QC for the methods involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes.

Supernatant Analysis:

Laboratory Control Sample/Blank Spike: The LCS/BS was within acceptance criteria of 80% to 120% required by the client's Test Specification.

Calibration Blanks: The five calibration blanks run at the beginning, middle, and end of the analysis run averaged 21 μgC TIC and 74 μgC TOC. These calibration/system blanks are considered acceptable for the concentration of TIC and TOC measured in the samples. However, the standard deviation for the TIC blanks was outside the historical pooled standard deviation used to establish the method detection limits; this indicates that there is significantly more variability in the blank than normal.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is acceptable for the samples. All the TIC and TOC RPDs meet the acceptance criteria of <15% established by the client's Test Specification.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes (for both AN-102 SUP AR and AN-102 SUP DF) demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery.

Solids Analysis:

Laboratory Control Sample/Blank Spike: The LCS/BS was within acceptance criteria of 80% to 110% required by the client's Test Specification.

Calibration Blanks: The three calibration blanks run at the beginning, middle, and end of the analysis run averaged 11 μgC TIC and 69 μgC TOC. These calibration/system blanks are considered acceptable for the concentration of TIC and TOC measured in the samples. The standard deviation for the TIC and TOC blanks are within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: The TIC and TOC RPDs meet the acceptance criteria of <15% established by the client's Test Specification.

Matrix Spike: The matrix spike (MS) recovery was very low for TIC and very high for the TOC spiked sample. A matrix spike duplicate (MSD) recovered within the acceptance criteria of 75% to 125% recovery; albeit the TIC recovery was still lower than normal and the TOC higher than normal. The total carbon recovery for both the MS and MSD is calculated at about 95%, suggesting that the quantity of the sulfuric acid added to the sample for analysis of TIC was insufficient to fully acidify the sample. Based on the MS and MSD recoveries, there is a possibility that the reported TIC for the solids is bias low and the TOC is bias high.

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: ____________________ **Date** 8-21-01

Review/Approval by: ____________________ **Date** 8-23-01

Archive Information:

Files: ASR 6019 6025 Urie P.doc ASR 6019L&S 6025L 6104L 6015L.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie
Project :	42040
Work Pkg:	CMC
Analyzed:	July 24, 2001
ASR:	6019.01, 6025

Analyzer M&TE:	WC01713 -- 701	
Balance M&TE:	360-06-01-023	
TOC STD: VWR CMS#161356	0.10% Carbon	<<[G]
TIC STD: VWR CMS# 161355	0.10% Carbon	<<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch) 7-24-01	13.0	68.0
	Calibration blank (start of batch)7-24-01	14.0	53.0
	Calibration blank	15.0	80.0
	Calibration blank	23.0	81.0
	Calibration blank (end of batch) 7-24-01	40.0	87.0

TIC	TOC	
21.0	73.8	<<< Blank Average (ug C)
11.3	13.5	<<< 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	**No**
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) 7-24-01	991	21	1.0000	97.0		1036	74	1.0000	96.2	
	Calibration Standard (start of batch) 7-24-01	995	21	1.0000	97.4		1035	74	1.0000	96.1	
	Calibration Standard CCV	1015	21	1.0000	99.4		1100	74	1.0000	102.6	
	Calibration Standard	999	21	1.0000	97.8		1032	74	1.0000	95.8	
	Calibration blank (end of batch) 7-24-01	1035	21	1.0000	101.4		1128	74	1.0000	105.4	
		[L] Average TIC % Rec >>>>			98.6	<<[L]	[P] Average TOC % Rec >>>>			99.2	<<[P]
QC	Blank Spike/LCS	1024	21	1.0000	100.3		1118	74	1.0000	104.4	

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.

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie
Project :	42040
Work Pkg:	CMC
Analyzed:	July 24, 2001
ASR:	6019.01, 6025

Analyzer M&TE: WC01713 -- 701		
Balance M&TE: 360-06-01-023		
TOC STD: VWR CMS#161356	0.10% Carbon	<<[G]
TIC STD: VWR CMS# 161355	0.10% Carbon	<<[C]

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 (%)
01-00429	An102-AR	2175	21	0.20	10,923		5905	74	0.20	29,379		40,302	
01-00429 Dup	An102-AR	1102	21	0.10	10,963	0	2960	74	0.10	29,083	1	40,047	1
01-00429 MS	An102-AR	1060	21	0.05	see below		2430	74	0.05	see below		see below	
01-00441	An102-DF	1197	21	0.20	5,963		1925	74	0.20	9,327		15,290	
01-00441 Dup	An102-DF	1212	21	0.20	6,040	1	2000	74	0.20	9,705	4	15,744	3
01-00441 MS	An102-DF	1590	21	0.10	see below		2012	74	0.10	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	
01-00429 MS	TIC Recovery	1060	21	10963	0.05	548	0.5000	500	**101.1**	TIC
	TOC Recovery	2430	74	29083	0.05	1454	1.0000	1000	**92.0**	TOC
	Total Carbon Recovery (TIC + TOC)							1500	**95.0**	TC
01-00441 MS	TIC Recovery	1590	21	6040	0.10	604	1.0000	1000	**98.7**	TIC
	TOC Recovery	2012	74	9705	0.10	970	1.0000	1000	**98.3**	TOC
	Total Carbon Recovery (TIC + TOC)							2000	**98.5**	TC

Reviewer/date: 8-23-01

FINAL Report
Organic Acids-AN-102 Supernate and Diluted Feed

GM Mong, R Niederer

Introduction

The desired analytes for WTP samples AN-102 supernate and diluted feed included a component (gluconate) which is not listed in standard separations for organic acids by ion chromatography (Dionex and Alltech catalog references). Gluconate is the first oxidation step in transformation of glucose α-carbon to a carboxylic acid from an aldehyde/hemiacetal functionality. This represents our first attempt to analyze tank waste materials for gluconate by ion exchange chromatography

Several separation mechanisms were attempted to define a separation of gluconate from the other desired analytes (glycolate, formate, oxalate, citrate). A useful separation was discovered for acetate from glycolate using the Dionex AS-14 column and 2mM sodium borate as the eluent. This affords a near separation of glycolate from acetate; unfortunately gluconate does not elute from this system. The reasons for gluconate's failure to elute from this column was not pursued. The best separation scheme for gluconate inclusive of the other analytes was to employ our established AS-11 (Dionex) separation using 0.08mM NaOH as the initial eluent. This affords gluconate as a peak just beyond the unretained volume of the column at a retention time that overlaps with fluoride. No other separations (AS-14, AS-9, AS-4) appeared to generate fruitful separations for gluconate from the other monovalent analytes (glycolate, formate). Due to the low ionic strength of the initial eluent, any samples require extensive dilution to prevent retention time variation, and long equilibrations (20 minutes) were a necessity.

Once these limitations were defined, a workable separation of gluconate, glycolate, formate, oxalate, and citrate was accomplished from the other tank ions (chiefly nitrite, nitrate, and sulfate). Surprisingly, a peak with the retention characteristics of gluconate was observed in the 441 samples. A test to distinguish this peak from fluoride was done by adding calcium oxide to sample 441S. The peak's observed area upon dilution was not diminished; therefore, this material does not have the characteristics of fluoride, and matches the retention time of gluconate.

Analytical efforts were undertaken to extend the retention time for gluconate and glycolate as far from the unretained volume as possible. The peak found in the samples very near the unretained volume had the same retention time as gluconate in the standards.

Instrumentation and sample preparation:

Dionex system (WD24293) was used with a sodium hydroxide gradient elution, AS-11 anion exchange column with AG-11 guard column. The elution ramp was 0.08mM were further diluted 1:500 for analysis. BS, MS and MSD samples, obtained as part of the sample batch, were prepared by spiking final dilutions with known amounts of a mixed standard of gluconate, glycolate, formate, oxalate, and citrate.

Results:

Results of analysis are summarized below. The calculations are corrected to the original sample concentration.

1. Analysis 01-429 AN-102-Sup and 01-441-AN-102-DF

Sample No.	Gluconate µg/mL	Glycolate µg/mL	Formate µg/mL	Oxalate µg/mL	Citrate µg/mL
01-00429S	<MDL (<1000)	11,000	8000	510	4400
01-00429D	<MDL (<1000)	10,000	8000	410	4400
01-00441S	41,000	3200	2800	1800	1200
01-00441D	40,000	3400	2800	1800	1200

Note: Gluconate values are represented from a material peak eluting near the void volume. This peak matches the retention time for gluconate, and does not respond chemically as fluoride (evidenced by addition of calcium oxide to the concentrated sample). Elution times near the void volume only indicate identity. An exact match of retention time near the void volume may not constitute firm identity of the material.

Method Detection Limits:

Estimated detection limits are based on the IDL multiplied by the approximate dilution factor subjected to each sample. This value represents the minimum detectability per sample prior to any handling in the hot cell. Use these values for any estimation of net detectability in the raw sample prior to dilutions in the hot cell or instrumental workup.

Gluconate µg/mL	Glycolate µg/mL	Formate µg/mL	Oxalate µg/mL	Citrate µg/mL
1000	100	100	200	200

2. Quality Control:

Matrix spikes were produced using the final dilution volumes; spikes were added which approximately doubled the size of the native material in each sample (determined by separate analysis of the MS and MSD samples). Spike added amounts are in **µg/mL**.The values reported DO NOT take into account any dilutions of sample materials and represent spikes on diluted samples.

Sample No.	Gluconate Spike added % recovery	Glycolate Spike added % recovery	Formate Spike added % recovery	Oxalate Spike added % recovery	Citrate Spike added % recovery
01-00441MS	1.1 (121%)	0.49 (105%)	0.25 (123%)	0.33 (89%)	0.60 (112%)
01-00441MSD	1.1 (107%)	0.49 (105%)	0.25 (109%)	0.33 (86%)	0.60 (116%)

Blank Sample Data: An analysis was done on sample 429 B. IDL values indicate a value estimated for a clean sample without dilution.

Sample No.	Gluconate	Glycolate	Formate	Oxalate	Citrate
01-00429B	<IDL (1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.2 µg/mL)	<IDL (0.2 µg/mL)

Blank Spike Sample Data: An analysis was done on sample 429 BS, a small peak was observed for oxalate. The reported value is indicative of the amount present in the delivered sample for analysis. This sample was spiked with analytes at 2 times the LLS value and analyzed.

Sample No.	Gluconate (spk added, %)	Glycolate (spk added, %)	Formate (spk added, %)	Oxalate (spk added, %)	Citrate (spk added, %)
01-00429BS initial analysis	<IDL (1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.1 µg/mL)	0.3µg/mL	<IDL (0.2 µg/mL)
01-00429BS, spiked	1.1µg/mL, 106%	0.49µg/mL, 100%	0.25µg/mL, 108%	0.33µg/mL, 78%	0.60µg/mL, 89%

ICB and CCB data: All collected data was less than IDL for all analytes

ICV, CCV data: Blank water spiked at 2 times the LLS value; recovery is percentage calculated from calibration curves. ICV was prepared from independent materials, with the exception of gluconate.

	Gluconate	Glycolate	Formate	Oxalate	Citrate
ICV1	102%	100%	97%	101%	96%
ICV2	106%	92%	102%	102%	99%
CCV1	110%	99%	104%	102%	101%
CCV2	106%	99%	104%	102%	101%

LCS data: sample prepared at 2 times the LLS level.

	Gluconate	Glycolate	Formate	Oxalate	Citrate
LCS	104%	103%	103%	100%	100%

LLS data:

	Gluconate 0.75μg/mL	Glycolate 0.33μg/mL	Formate 0.17μg/mL	Oxalate 0.18μg/mL	Citrate 0.4μg/mL
LLS	96%	102%	101%	98%	95%

Comments: Almost all of the quality control data meets the success criteria. Blank spike recovery for oxalate and MSD recovery for oxalate are out of the ±15% range. All other data are acceptable.
If Gluconate is a continued concern for analysis, a more rigorous search for an analytical method (ion chromatography or other means) should be undertaken to improve analytical confidence in its identity in tank materials.

Prepared by GM Mong 10/23/01

Reviewed by JA Campbell JA Campbell 10/23/01

Blank

Organic Phosphate Analysis

Introduction

A derivatization gas chromatography (GC) method was developed to analyze bis-(2-ethylhexyl)phosphoric acid (D2EHP) under the auspices of the Organic Tanks Safety Program (Campbell 1996). Derivatization gas chromatography/mass spectrometry (GC/MS) and GC/flame ionization detection (FID) for the analysis of D2EHP has been performed only twice in the past. Samples associated with tank 241-C-106 were obtained during washing and centrifuging experiments and appeared to contain a waxy or oily substance. Initial direct GC analysis of methylene chloride extracts of the matrix did not reveal the reason for high total organic carbon (TOC) values associated with these samples; tributylphosphate (TBP) and a later eluting component identified as butyl bis-(2-ethylhexyl) phosphate were found. Treatment of an aliquot of the sample with ethereal hydrochloric acid followed by diazomethane revealed a large quantity of D2EHP to be present in the sample. Quantitation of these three components afforded a good match to the measured TOC values; these three components make up about 75% of the total mass of the sample (Mong and Campbell 1999). Samples from tank 241-C-103 were taken from a layer amounting to at least 16 m^3 floating on top of aqueous waste to resolve safety concerns. The organic liquid was found to consist primarily of paraffin hydrocarbons and TBP in an approximately 1:2 ratio. Direct analysis of the liquid revealed smaller quantities of dibutylbutyl phosphonate (DBBP) and butyl bis-(2-ethylhexyl) phosphate. Treatment of an acidified portion of this sample did not reveal the presence of D2EHP (Mong and Campbell 1999, Campbell 1996). The previously mentioned studies focused primarily on identification and semi-quantitation.

The current analysis of the tank waste samples AN-102 as-received and AN-102/C-104 blended feed represents the first attempt at using diphenylphosphate (DPP) as a surrogate standard. Diphenylphosphate was chosen as a surrogate compound because its structure and potential reactivity are similar to D2EHP.

The analysis of the AN-102 samples for D2EHP has been conducted on a best effort basis. The samples were analyzed according test plan TP-RPP-WTP-047, "Identification and Quantification of D2EHP in Tank Waste". D2EHP was not detected in any of the samples.

Instrumentation

The derivatized phosphate samples were analyzed using a GC 5890 Series II (Hewlett Packard) equipped with a flame ionization detector and a fused silica column (DB-5MS [J&W] 30 m X 0.25 mm X0.25 µm film thickness). The temperature program used was the following: 40° C for 1 min, 40° C-260°C at 8°C/min and 260° C for 7 min. The injector was at 265°C and the detector was at 260°C. A 1 µL splitless injection was performed for the analyses. The retention times and identities of DPP and D2EHP were confirmed using GC/MS.

Standard and Sample preparation

Standards were prepared for DPP (2.3 μg/mL to 116 μg/mL) and for D2EHP (2.6 μg/mL to 130 μg/mL) in methylene chloride and derivatized using diazomethane prepared as discussed below.. A five-point calibration curve was constructed for both DPP and D2EHP.

Each sample, including quality control samples, was prepared from 5-mL aliquots, which were placed in a beaker and weighed. A 5-ml aliquot of water was added, and the sample was transferred to a separatory funnel. The pH was adjusted to 2 (measured with pH paper) with 6 N nitric acid. Each sample was then extracted three times with 25 mL of methylene chloride. Each sample was then extracted with 25 mL of butanol. The methylene chloride and butanol extracts were stored separately. The methylene chloride extracts were concentrated to a volume of less than 1 mL. Diazomethane was prepared by stirring an ethereal slurry of N-methyl-N-nitrourea (Pfalz and Bauer Chemical Company) over an ice-cold 40% KOH solution. The yellow diazomethane/ether solution is simply decanted off for refrigerated storage. Diazomethane, though hazardous if handled incautiously, is a reagent for methylation.

Approximately 100 μL of the diazomethane/ether solution was added to each extract. Contact of an acid phosphate (e.g. D2EHP) with diazomethane solution results in instantaneous evolution of nitrogen gas; excess diazomethane is indicated by the characteristic yellow color of the solution. Additional diazomethane is added until the yellow color persists with slow evolution of bubbles. The reaction is left covered, in a hood, for an additional hour to ensure complete conversion of the acid phosphates to the methyl esters. The volume of the samples was then adjusted to 1 mL using nitrogen blow-down techniques and analyzed using GC/FID. (The highly acidic nature of alkyl phosphates (pKa ca. 1.0-1.5) and the ability of phosphoric acid to dehydrate alcohols makes phosphates intractable to esterification in the laboratory with acidic reagents, such as mineral acids or BF_3, in conjuction with alcohols.)

The laboratory control sample (LCS) extracted in the hot cell was inadvertently spilled during the concentration procedure. Another LCS was prepared outside the hot cell by adding D2EHP and DPP to 5 mL of Milli-Q water adjusted to a pH of 12 using 6M NaOH. The pH of the sample adjusted to 2 using 6N nitric acid. The pH was measured with pH paper. The sample was then extracted in a separatory funnel 3 times with 25 mL of methylene chloride with vigorous shaking. The methylene chloride extracts were combined and concentrated to approximately 1 mL with K-D apparatus. The sample was derivatized with diazomethane following the procedure above, and the final volume was brought to 1 mL prior to analysis. The sample was analyzed using GC/FID.

Analysis Results

The results of analyses for the samples and recoveries for DPP and D2EHP are listed below in Table 1. The estimated quantitation limit (EQL) for D2EHP in the sample, sample duplicate, and process blank are based on the concentration of the lowest calibration standard converted to the appropriate volume of sample (ca 5 mL). D2EHP was not detected in any of the samples.

Table 1. Organic Phosphate Sample and QC Results

		DPP		D2EHP	
RPL Number	**Sample ID**	**µg/mL**	**Recovery**	**µg/mL (1)**	
01-00429	AN-102-SUP AR	31.9	144%	<0.54	
01-00429-D	AN-102-SUP AR Dup	39.4	187%	<0.51	
01-00441	AN-102-SUP DF	17.7	83%	<0.52	
01-00441-D	AN-102-SUP DF Dup	18.4	87%	<0.51	
01-00429-PB	Process Blank	0.62	3%	<0.51	
QC Samples	**Sample ID**	**µg/mL**	**Recovery**	**µg/mL**	**Recovery**
LCS	Lab Control Sample	1.04	5%	10.3	42%
01-00441-MS	AN-102-SUP DF Matrix Spike	15.4	73%	20.3	84%
01-00441-MSD	AN-102-SUP DF Matrix Spike Dup	19.4	93%	25.6	107%

(1) The ‘less than results’ for D2EHP are the EQL based on the concentration of the lowest standard used to generate the calibration curve adjusted to the sample volume and dilutions.

Neither the Test Specification nor the governing QA Plan provides QC acceptance criteria for the organic phosphate analysis. However, to assess method performance the results of the QC samples (i.e.,, LCS and matrix spikes) were compared to the QC acceptance criteria for anions by ion chromatography. Recoveries for both the DPP and D2EHP from water matrices (i.e., process blank and LCS) were very poor. The reason for the high DPP recoveries in the AN-102-SUP AR samples is not apparent, but it is possible that the DPP spike may have been added twice. The low DPP recoveries observed in the blank and LCS and low D2EHP recovery in the LCS can not be explained.

To indicate the variance in derivatization, three replicate samples (i.e. calibration verification check standards) containing D2EHP and DPP were derivatized and analyzed. The recoveries ranged from 62-121% for DPP and 73-84% for D2EHP. Each sample contained 58 µg/mL (DPP) and 65 µg/mL for D2EHP. The results are shown in Table 2.

Table 2. DPP and D2EHP Standard Mixture Results

	DPP		D2EHP	
Sample	**µg/mL**	**Recovery**	**µg/mL**	**Recovery**
1	36	62	47	73
2	57	98	53	82
3	70	121	54	84

Recommendations

The analysis of tank waste samples for D2EHP was performed on a best effort basis, since this technique has not been thoroughly validated and qualified. From this work the appears to hold promise for producing reliable results for D2EHP; however, further methods development is required. The apparent inconsistency in recoveries must be addressed. One of the chromatographic problems encountered was tailing of the peaks which may have contributed to high recoveries with certain samples. As a result of peak tailing, manual integration of the peaks was required. The peak-tailing problem may be solved in additional analyses by simply adjusting the chromatographic parameters such as the column temperature program and injection port temperature or changing the column.

In addition, in order to make this technique reliable and robust, a systematic study must be undertaken to evaluate the extraction process and the derivatization process to determine the reason for low recoveries in the control sample. It may be that the choice of DPP as a surrogate compound was not a good one. Also, it would be advisable to add a known methylated compound to examine the extraction efficiency.

Since the analyses of these sample, additional work has been done to evaluate the poor recoveries demonstrated by the process blank and LCS. Sodium sulfate has been added to an aqueous sample to form a saturated solution, then spiked with known volumes of DPP and D2EHP. The recovery for DPP was 60% and for D2EHP was 91%. The increased recovery is most likely due to a salting out effect. In addition, stability of the derivatized standard over time has been examined. The responses (i.e., apparent concentration) tended to decrease.

A test plan is being written that proposes testing on various matrices (e.g., water, simulated tank waste, and tank waste) to identify the cause of the low recovery problems and to develop a more rugged, reliable, consistent technique for the analysis of D2EHP. Areas to be addressed include derivative stability, ionic strength, pH, and method of agitation. Once the method has been verified with a water matrix, the method will be extended to simulated wastes.

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Archive Information
Sample data and calculations located in LRB 14059 pages 7-8 and 28-31.
Analysis data and supporting data and documentation located in ASR 6016/6025 for Project 42020 (or 43265).

Prepared By: J. A. Campbell Date: 5/2/02
Reviewed By: Steven C. Goheen Date: 5/2/02

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Derivatization GC/FID Analysis of Chelators and Degration Products Report

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ASR Number: 6019, 6025
Preparation Date: 6/04/02
Analyst: J.A. Campbell

Charge Code/Project: W57912
Sample Receipt Date:
Analysis Date: 8/15/01

Preparation Procedure: TPR-PNNL-WTP-049
Analysis Procedure: TP-RPP-WTP-048
M&TE: WD14807, WD 14805 –GC
WD 30902, WD 30903 GC/MSD

Final Results

See attached report

Summary of Deviations

Spiking solutions for the MS and MSD were performed with citric acid and EDTA only. There were no standards available for nitrosoiminodiacetic acid (NIDA) and ED3A. The concentrations were calculated on the basis of the response compared to EDTA. In addition, the concentration of HEDTA was based on the response of EDTA.

Quality Control Sample Comments

See attached report.

General Comments

See attached report.

Report Prepared by: J.A. Campbell J.A. Campbell **Date** 6-28-02

Review/Approval by: R.B. Lucke R.B. Lucke **Date** 6-28-02

Archive Information

Chelator and Degradtion Product Analysis Using Derivatization GC/FID of AN-102 Supernate and Diluted Feed

J.A. Campbell

Introduction

Chelators such as ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl) ethylenediaminetriacetic acid (HEDTA), nitrilotriacetic acid (NTA), iminodiacetic acid (IDA), succinic acid (SA), ethylenediaminetriacetic acid (ED3A), and citric acid (CA) are not part of the Environmental Protection Agency's (EPA) list of targeted, hazardous chemicals; nonetheless, they are receiving renewed environmental interest. For chelators and chelator fragments, there is no accepted SW-846 type accepted methodology that we are aware of. As a result, derivatization gas chromatography (GC) was developed to analyze chelators under the auspices of the Organic Tanks Safety Program and Flammable Gas Safety Program several years ago. Derivatization gas chromatography/mass spectrometry (GC/MS) and GC/flame ionization detection (FID) for the analysis of chelators has been performed several times in the past. Samples associated with tank 241-SY-101, C-104, and C-201 (Grant, Mong, Lucke, Campbell 1996; Campbell et al. 1998; Campbell et al. 1994) and others have been analyzed.

The chelators are a class of compounds whose low volatility and high polarity preclude analysis by GC/MS or GC/FID without prior derivatization. Derivatization techniques that have been applied to the analysis of chelators include 1) reaction with boron trifluoride (BF_3/methanol) to form the methyl esters, 2) reaction with diazomethane to form methyl esters, 3) reaction with butanol/HCl to form butyl esters, and 4) reaction with silylation derivatizing reagents to form silyl derivatives (Campbell et al. 1998). The most useful method appears to be the reaction with BF_3/methanol, with the other methods giving, at best, modest results (Grant, Mong, Lucke and Campbell 1996; Campbell et al. 1994).

The analysis of tank waste samples for chelators and chelator degradation products was on a best effort basis. The samples were analyzed according test plan TP-PNNL-WTP-048 entitled "Derivatization GC/FID Analysis of Chelators and Degradation Products". In addition, the test plan TPR-RPP-WTP-049 entitled "Ion Exchange for Activity Reduction" was also employed to reduce the radioactivity level of the samples.

Chelators and degradation products were detected in the samples and duplicates. These include EDTA, HEDTA, SA, NTA, ED3A, and CA. As a result of the variability in the concentrations obtained, additional work is required to make this a reliable, robust technique.

Instrumentation

The derivatized samples containing the methyl esters were analyzed using a GC 5890 Series II (Hewlett Packard) equipped with a flame ionization detector and a capillary fused silica column (30 m X 0.25 mm X 0.25 μm film thickness). The temperature program used was the following: 40°C for 1 min, 40°C-260°C at 8°C/min and 260°C for 7 min. The injector was at 265°C and the detector was at 260°C. A 1 μL splitless injection was performed for the analyses.

The retention times and identities of the chelators and degradation products were confirmed using gas chromatography/mass spectrometry (Agilent 5973).

Standard and Sample preparation

Standards of EDTA, HEDTA, adipic acid (AA), CA, SA, NTA, and IDA were prepared ranging in concentration from approximately 500 to 30 µg/mL and subsequently derivatized with BF_3/methanol as discussed below. Adipic acid is used as a monitor of derivatization efficiency. The standards were analyzed using GC/FID.

Approximately 5 mL of supernate was diluted with 5 mL of H_2O and then eluted through a bed of AG50W-X8 resin (Biorad), 50-100 mesh, prepared in the sodium form. The resin must not be used in the usual H^+, since the chelator analytes tend to precipitate from solution as insoluble free acids when the matrix is exposed to the acid form of the resin. The sodium form of the cation exchange resin is conveniently prepared by stirring the resin with sufficient 6N NaOH until the solution remains basic to pH paper. The resin is then washed with Milli-Q water. After elution through the cation exchange resin, the net reduction of fission products (chiefly ^{137}Cs and ^{90}Sr) was sufficient that the sample could be removed from the hot cell environment and safely worked within a fume hood (Mong, Orr, Hoopes, Campbell 1998).

A 2-mL aliquot of tank waste material obtained after elution through a cation exchange resin was placed in a Reactivial and concentrated to dryness using nitrogen blow down techniques. Adipic acid was added to each of the samples as a check on derivatization efficiency. After the sample was completely dry, 2 ml of a 12% (w/v) BF_3 in methanol (Aldrich) was added and the sample was capped tightly. The Reactivial was placed into the heat block and heated to a temperature of 100° C for 1 hour. The sample was agitated twice during the reaction period. After one hour, the sample was allowed to cool to room temperature. While the sample was cooling, 15 mL of a 0.4 M K_2HPO_4 was poured into a sample vial. After the reaction mixture was cooled to room temperature, 2 mL of chloroform was added to the Reactivial and shaken. The solution was then added to the vial containing the phosphate buffer, shaken, and the aqueous and chloroform layers were allowed to separate. The pH of the aqueous layer was checked with 0-14 range pH paper and was in the range of 7-8. The top aqueous layer was decanted to waste. The bottom layer contains the derivatized organics for analysis. The chloroform solution was added to a vial containing approximately 200 mg of anhydrous Na_2SO_4 to collect the entrained water. The chloroform solution was added to a GC autosampler vial and capped. Prior to removal of the sample from the contaminated area fume hood, it was surveyed by the RCT. The samples were then analyzed using GC/FID. Confirmation of the identification of the analytes (SA, CA, EDTA, HEDTA, NIDA, and NTA) was performed using GC/MS. Tentative identification of nitroso-ED3A and ED3A was based on mass spectral data.

Results of Analyses

Standards were prepared for EDTA, HEDTA, CA, SA, IDA, and NTA (approximately 500-30 µg/mL) and derivatized using the method described above. A five-point calibration curve was constructed for EDTA, HEDTA, CA, SA, IDA, and NTA. HEDTA is also very susceptible to the final pH during the addition of phosphate buffer. This may be the reason the calibration curve for HEDTA was not linear and the results were highly variable. IDA suffers from a similar fate or is susceptible to hydrolysis and the calibration curve is not linear, and the results were highly variable as well.

The results of analyses for the samples are shown in Table 1. In view of the fact no standards exist for nitroso-IDA (NIDA), nitroso-ED3A, or ED3A, the concentrations of these components were based on the assumption of equivalent response to EDTA. Both of these compounds are not commercially available and require independent synthesis. The results for these compounds would be termed qualitative based on the fact that no standards are readily available.

In the reaction of chelators with BF_3/methanol, methylation occurs at the carboxylic acid sites. However, methylation does not occur at hydroxy sites in the reaction of BF_3/methanol. The mass spectrum of methylated HEDTA shows a parent ion at m/z 288. The hydoxyethyl group of HEDTA resists methylation by BF_3/methanol and, instead, forms a cyclic, or intramolecular lactone with one of the neighboring acetate ligands. The MW 288 lactone accounts for only approximately 30% of the species detected. Trimethylated HEDTA with an unreacted, or free, hydroxyethyl group accounts for the remaining 70% of the HEDTA. It is speculated that the species is simply too polar to migrate through the GC column (Lokken et al. 1986; Grant, Mong, Lucke, and Campbell 1996). Due to nonlinearity and variabiltity of the calibration curve for HEDTA, the concentration of HEDTA was based on the assumption the response is similar to EDTA.

ED3A, when derivatized with BF_3/methanol, forms a lactam. In addition, ED3A also forms a nitroso-ED3A under derivatizing conditions. IDA also forms a nitroso-IDA (NIDA) under these conditions. The nitroso compounds are formed under acidic conditions (BF_3, the derivatizing reagent, is a Lewis acid) by the reaction of an analyte with a free amine hydrogen, as in the case of IDA and ED3A, with the high concentration of nitrite present in the waste samples. The identity of these compounds was confirmed using GC/MSD. It should be emphasized that the nitroso compounds are not present in the tank waste; they are artifacts of the derivatization procedure (Grant, Mong, Lucke, and Campbell 1996).

Table 1. Results of the Analyses for the AN-102 Samples and AN-AN-102 Diluted Feed (Concentrations are expressed in μg/mL of sample)

sample	SA	IDA	CA	NTA	EDTA	HEDTA	ED3A(a)
01-00429-s	35.9	3464.6	429.6	255.6	602.8	<120.1	1520.1
01-00429-d	<34.3	1388.5	<126.2	113.6	237.2	<120.1	556.7
01-00441-s	<34.3	1562.2	574.4	196.0	772.1	<120.1	761.7
01-00441-d	<34.3	1118.1	284.6	133.2	463.7	<120.1	535.7
Blank	<34.3	<120.1	<126.2	<97.2	<120.1	<120.1	<120.1

* The analyte is detected at the required retention time but the calculated concentration is below the calculated EQL. This is based on the lowest concentration of standard used in calibration.

SA- succinic acid
IDA- iminodiacetic acid
CA- citric acid
NTA- nitrilotriacetic acid
EDTA – ethylenediaminetetraacetic acid
HEDTA – N-(2-hydroxyethyl)ethylenediaminetriacetic acid
ED3A – ethylenediaminetriacetic acid
(a) Nitroso-ED3A and ED3A were tentatively identified based on mass spectral interpretation.
The values for the blank were calculated using the lowest concentration of the standards used in calibration converted to the sample.

The ED3A category in Table 1 includes a combination of both ED3A and nitroso-ED3A. The concentration of IDA is based on the formation of NIDA. The concentrations of both ED3A and IDA are based on the assumption of a similar response to EDTA. Nitroso-ED3A and ED3A were tentatively identified using mass spectral data. Actual standards would have to be synthesized and compared with the retention times to positively confirm their identification.

The recovery for AA in the blank was 86%. The percent recoveries for AA for 429-s (91 %), 429-d (80%), 441-s (98%), and 441-d (85 %). The data were not corrected based on recovery of AA. Due to the fact that the chelators are very similar in structure and reactivity to EDTA and the acids similar to CA, only EDTA and CA were added to both the matrix spike and duplicate. For the matrix spike and matrix spike duplicate, the following results were obtained. The recoveries for the matrix spike and duplicate are listed in Table 2. This represents the first time MS and MSD samples have been analyzed with this technique.

Table 2. Recoveries for the MS and MSD

	CA	EDTA
01-00441-MS	47%	66%
01-00441-MSD	50%	58%

The recovery for adipic acid in these two samples are 107% (441-MS) and 103% (441-MSD). Chelators were not found in the blank.

The laboratory control sample (LCS) was compromised during sample preparation in the contamination hood, and a LCS was prepared and the corresponding sample preparation was performed outside of the hot cell facilities. The sample was eluted through cation exchange resin column. An aliquot was concentrated, derivatized, and analyzed using GC/FID. The corresponding recoveries for AA were 106%, SA 69%, EDTA 78%, NTA 71%, CA 83%, and HEDTA 38%. The concentration of HEDTA was not corrected.

Neither the Test Specification nor the governing QA Plan provides QC acceptance criteria for the chelator analysis. In addition, the target values for recoveries listed in the Test Plan are target values. There is no historical QC data for this analysis technique.

Recommendations

The analysis of tank waste samples for chelators and chelator degradation products was performed on a best effort basis. This a very complex procedure due to moisture and safety considerations. In addition, the formation of compounds (e.g. methyl ester of HEDTA) appears to be pH dependent and the recoveries are variable. The variation in recoveries and reproducibility has also been evident in other studies as well (Campbell et al. 1994; Campbell et al. 1998; Grant, Mong, Lucke, Campbell 1996). A problem encountered with these tank samples involved the separation of the chloroform and aqueous layer during derivatization. The solution was very cloudy which resulted in a lack of phase distinction. This may be an explanation for the variability in the results. The degree of variability will have to be addressed. One of the ways of possibly improving the variability is using smaller sample sizes. As the sample dries it tends to form a concrete-like material which may be impervious to the derivatizing reagent. Using a smaller sample may provide a thiner crusty level, and the derivatization process may be better. Heating at lower temperatures for longer periods of time during the derivatization process may also provide less variability in results. Further work with simulants to perfect the technique would be advisable; additional spiking experiments would also be beneficial. The derivatization method for chelators and degradation products, in its present form, requires additional methods development. A direct analysis technique, with no derivatization, potentially would be a more robust and reliable technique. Preliminary results with capillary electrophoresis shows promise as a direct analysis technique for chelators and degradation products; however, additional methods development is required for this technique as well.

The results for HEDTA, IDA, and ED3A should be considered semiquantitative in view of the fact that no standards exist for the nitroso-IDA, ED3A, and nitroso-ED3A and the variability observed with the results of HEDTA. It should be emphasized that this technique has not been thoroughly verified in terms of QA and QC requirements. However, I do think the potential is there for this technique to be useful for the analysis of chelators. Prior to this technique becoming a reliable technique, additional work is required. Work with tank waste simulants would lessen the cost of methods development.

It is estimated the additional methods development time to do spiking studies and solve the variability problems would require approximately 4-5 weeks. Simulated waste studies would be suggested to lessen the cost. In addition, under the auspices of another project, methods development involving capillary electrophoresis is underway. With this technique, the samples would be analyzed directly; no derivatization would be required prior to analysis. However, capillary electrophoresis methods development is just underway. Within several months, a capillary electrophoresis instrument capable of analyzing radioactive samples will be available. In order to detect smaller concentrations of chelators, GC/MS may be a more sensitive technique. The problems associated with sample preparation would still be present.

The concentration of citric acid as determined by ion chromatography is approximately 10 times the concentration determined by derivatization GC/MS. The IC method is a direct analysis method; no derivatization is required prior to analysis. As a result, the result obtained by IC may be more reliable at this point.

The concentration of citric acid as determined by ion chromatography is approximately 10 times the concentration determined by derivatization GC/MS. The IC method is a direct analysis method; no derivatization is required prior to analysis. As a result, the result obtained by IC may be more reliable at this point.

In view of the fact there is a discrepancy in the TOC accounted for, I would like to suggest that other aliquots be dried, derivatized, and analyzed. The problem associated with the drying process has been tentatively identified and corrected. As a result, the drying time should be much less. The samples are already available and would not have to go through the hot cell facilities.

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Reviewer

Heat Capacity Measurement of AN-102 MOD2-1

RD Scheele 12/6/01
R. D. Scheele, Author Date

C.F. Wend 06Dec2001
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Analytical Approach

To measure heat capacity, we used a differential scanning calorimeter (DSC) and the instrument manufacturer's prescribed approach for measuring heat capacity. Differential scanning calorimetry measures enthalpy (heat) changes as the temperature is increased at a known and constant rate or at constant temperature by measuring differences between heat requirements between a sample and a reference located in a second sample holder. Sample sizes range from a couple mg up to 100 mg depending on density. For these analyses, we used nominal 10 mg sample sizes.

For the heat capacity measurement, a three-step approach is used. First the empty sample pan is heated to an operator selected starting temperature, held for 10 min, then heated over the temperature range of interest at a controlled rate, and then held at the final temperature for 10 minutes. Second, the sample pan is filled with roughly the same amount of reference material as will be used for the sample and the same temperature program repeated. The reference material is removed and replaced with the sample and the same temperature program repeated. The sample's heat capacity is calculated based on differences between the reference and the baseline and differences between the sample and the baseline taking into account differences in reference and sample masses. Typically, the reference material is similar in state and mass to the samples to be analyzed. For solid samples, sapphire is recommended by the equipment manufacturer as the reference. For aqueous samples, water is recommended by the equipment manufacturer as the reference; we used commercially available high purity water supplied for use with inductively coupled argon plasma (ICP) with a mass spectrometer (MS) detector.

For these measurements, the temperature program used was to 1) heat to 30°C, 2) hold at 30°C for 10 minutes, 3) heat to 120°C at 5°C/min, and 4) hold at 120°C for 10 min. The analysis of aqueous or slurry samples complicates heat capacity measurements because of water's volatility between room temperature and 100°C and above. To accommodate water's volatility and eliminate enthalpy changes due to evaporation, we used sealed gold sample pans for the water reference and the sample. The use of sealed sample pans prevented us from using the same sample pan for baseline, water reference standard, and sample.

To accommodate the complication due to water volatility, we used a single separate sealed gold pan containing nominally 10 mg of the high purity water as the reference standard for each analysis and used another sealable gold pan for the baseline and sample. We assumed that the gold pans containing the water and the sample were equivalent.

The sample characterized was AN-102_MOD2-1. This material was principally an aqueous samples with the possibility of some solids, which presented a particular challenge because of the temperature range of interest (room temperature to above 100°C) and the volatility of water in that range. To prevent water evaporation and eliminate associated heat losses, which would mask measurement of the heat capacity, we encapsulated the deionized water (DIW) reference and the sample in hermetically sealed gold pans. The DIW reference was encapsulated in its own gold pan and reused for each analysis. We used a linear least squares regression of water's heat capacity from 300 K (30°C) to 473 K (100°C) (Weast 1984) to model the DIW reference standard's heat capacity at constant pressure (C_p); e.g. $C_p = 0.000562*T+3.9998$ J/(g K). We used this model to extrapolate above water's boiling point based on the assumption that the bulk of the water remained as liquid until the sealed pan ruptures. The model was used to calculate the reference C_p at each data point's temperature; data points were taken by the instrument every 0.4 °C. The sample was encapsulated in the sample pan used for the baseline; we assumed equivalent behavior for the pan used for the DIW and the baseline. This assumption was necessary since sealing the pan rendered the pan useless for other samples.

Triplicate analyses of the two samples were performed. The results presented are averages of the three analyses.

Results

AN-102_MOD2-1

Table 1 and Figure 1 provide the average measured heat capacity of the AN-102_MOD sample. Figure 1 also provides a linear regression of the average heat capacity provided in the figure. A linear least squares regression of the three analyses data yields:

$$C_p = (-0.0018 \pm 0.0002)\, T + (3.42 \pm 0.08) \text{ J/(g K)}$$

Where C_p is the heat capacity at constant pressure, T is temperature in degrees Kelvin. The 95% confidence interval is ± 0.12 J/(g K).

As shown in Table 1, 102-AN_MOD's heat capacity is lower than water's heat capacity for the same temperature range. This is likely due to the solids in the 102-AN waste, which typically have much lower heat capacities than water. For example, sodium nitrate at 300 K has a heat capacity of 1.10 J/(g K) compared to liquid water's C_p of 4.18 J/(g K). The slight negative slope is unexpected and may arise from dissolution of any solids having an exothermic heat of solution or from changes in the distribution of gaseous and liquid water or from some other unidentified possibilities. Gaseous water at 300 K has a C_p of 1.88 J/(g K) or about half that of liquid water. These two possibilities are conjecture at best.

References

Weast, R.C. (Editor). 1984 *CRC Handbook of Chemistry and Physics.* CRC Press, Inc., Boca Raton, Florida.

Table 1. Measured Heat Capacity of AN-102 MOD2-1

T, K	Analysis 1 Cp, J/(g K)	Analysis 2 Cp, J/(g K)	Analysis 3 Cp, J/(g K)	Average Cp, J/(g K)	Water Cp, J/(g K)
310	2.86	2.89	2.75	2.83	4.17
315	2.87	2.89	2.75	2.83	4.18
320	2.84	2.87	2.74	2.82	4.18
325	2.84	2.89	2.72	2.82	4.18
330	2.83	2.86	2.73	2.80	4.19
335	2.81	2.83	2.80	2.82	4.19
340	2.79	2.84	2.75	2.79	4.19
345	2.82	2.83	2.75	2.80	4.19
350	2.78	2.82	2.67	2.76	4.20
355	2.76	2.82	2.63	2.74	4.20
360	2.73	2.82	2.66	2.74	4.20
365	2.74	2.81	2.61	2.72	4.20
370	2.73	2.72	2.67	2.71	4.21
375	2.73	2.75	2.67	2.72	4.21
380	2.77	2.74	2.69	2.74	4.21
382	2.83	2.83	2.68	2.78	4.22

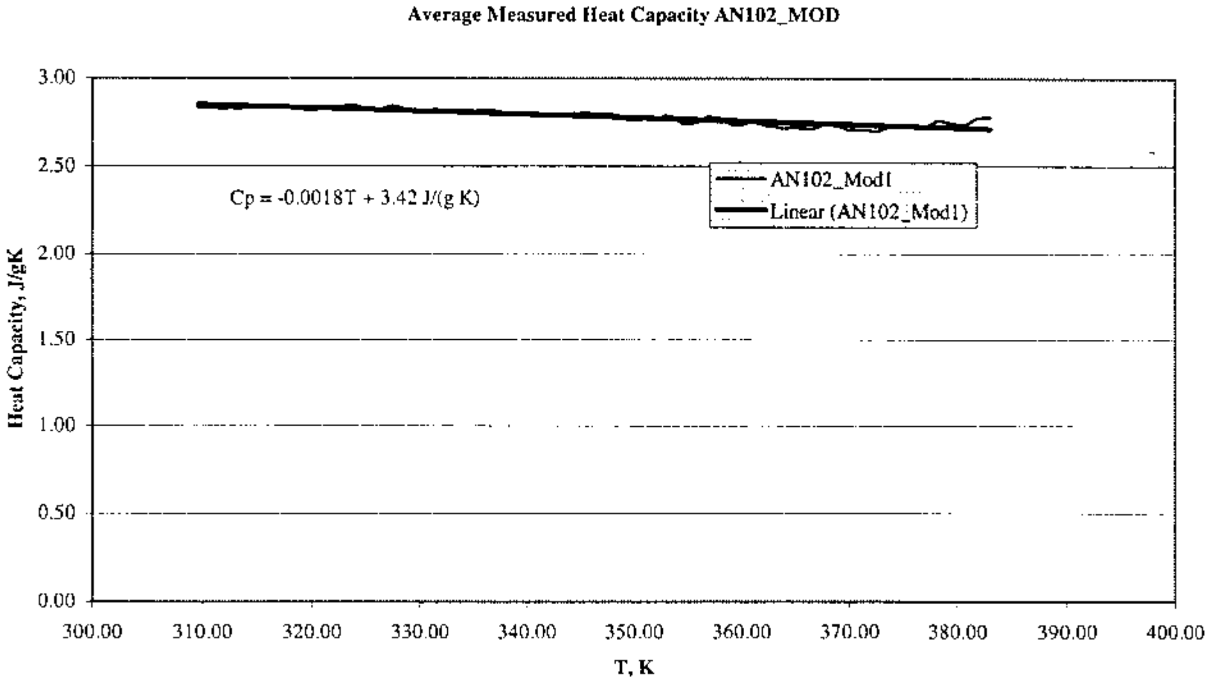


Figure 1. Average Measured Heat Capacity of AN-102_MOD

Particle Size Distribution of AN-102 MOD2 Sample

Hanford Tank 241-AN-102

Adam P Poloski 11/29/01

Adam P Poloski, Ph.D., Author Date

11/29/01

Paul R Bredt, Ph.D., Technical Reviewer Date

Particle Size Distribution

The particle size distribution of a 2 wt. % solids sample from Hanford Tank 241-AN-102 labeled as AN-102 MOD2 is described in this report. A Microtrac X-100 Particle Analyzer and a Microtrac Ultrafine Particle Analyzer (UPA) were both used to measure the particle size distribution of the tank samples. The Microtrac X-100 Particle Analyzer 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 is 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 and 700 μm. The Microtrac UPA measures particle diameter by Doppler shifted scattered light. This method is limited to particles with diameters between 3 nm and 6.5 μm.

The particle size distribution of the AN-102 MOD2 sample was measured in the Microtrac X-100 at a flow rate of 40 ml/s. The flow rate was then increased to 60 ml/s and the particle size distribution was measured. The sample was then sonicated with 40W ultrasonic waves for 90 sec at a flow rate of 60 ml/sec and the particle size distribution was measured. Finally, the sample was then sonicated a second time with 40W ultrasonic waves for 90 sec at a flow rate of 60 ml/sec and the particle size was measured. Sample was withdrawn from twice from the X100, once prior to sonication and once after sonication. These two samples were then analyzed in the Microtrac UPA. In the X100 instrument, analyses were performed in triplicate on each sample under all flow/sonication conditions. The average of these triplicate measurements are reported in this document. In the UPA instrument, only one measurement and a duplicate measurement are reported per sample.

The suspending medium for these analyses was a surrogate supernatant based on the ICP and IC data obtained for the 241-AN-102 supernatant liquid. The composition of this supernatant liquid is reported below (see Table 1).

Table 1: Surrogate Supernatant Composition

Component	Concentration (M)
$NaNO_3$	1.51E+00
NaOH	4.39E+00
$Al(NO_3)_3 \cdot 9H_2O$	1.03E+00
Na2SO4	1.35E-01
$Na_2HPO_4 \cdot 7H_2O$	4.53E-02
NaCl	1.07E-01
$NaNO_2$	2.31E+00
$NaCO_3$	1.00E+00
$Na_2C_2O_4$	6.37E-03
NaF	1.20E-02

The instrument performance was checked against a range of NIST traceable standards from Duke Scientific Corporation. These standards are polymer microspheres dispersed in a 1 mM KCl solution. These standards were run prior to analysis of the sample. To show that the instrument worked properly throughout the experiment, the standards were run again after the analysis of the sample. Results from these standard tests are presented in Tables 2 & 3. The percentile data shown in the table represent the given percent of the volume (or weight 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.

Table 2: X-100 Calibration Standards

X-100 (Prior to Analyses)		
	20 µm Duke Scientific Standard (Lot #19411)	**301 µm Duke Scientific Standard (Lot# 19136)**
Percent by volume less than the specified particle size	**Size (µm)**	**Size (µm)**
10	17.74	236.7
50	23.47	300.8
90	32.63	476
Mean Particle Size Calculated on a Volume Basis	24.47	328.7
X-100 (After Analyses)		
	20 µm Duke Scientific Standard (Lot #19411)	**301 µm Duke Scientific Standard (Lot# 19136)**
Percent by volume less than the specified particle size	**Size (µm)**	**Size (µm)**
10	17.26	249.6
50	23.5	282.4
90	36.64	332.8
Mean Particle Size Calculated on a Volume Basis	25.63	299.5

Table 3. UPA Calibration Standards

UPA (Prior to Analyses)		
	96 nm Duke Scientific Standard (Lot #16339)	**895 nm Duke Scientific Standard (Lot# 15924)**
Percent by volume less than the specified particle size	**Size (nm)**	**Size (nm)**
10	87.2	745.3
50	100.1	884.9
90	117.4	1085
Mean Particle Size Calculated on a Volume Basis	101.3	1178
UPA (After Analyses)		
	96 nm Duke Scientific Standard (Lot #16339)	**895 nm Duke Scientific Standard (Lot# 15924)**
Percent by volume less than the specified particle size	**Size (nm)**	**Size (nm)**
10	82.5	712.3
50	100.1	901.3
90	119.7	1110
Mean Particle Size Calculated on a Volume Basis	100.6	906.9

Tables 4 & 5 contain a summary of the particle size analysis from the X100 instrument on a volumetric and numeric basis respectively. This data presented in these tables 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 data presented in Table 4 indicate a large volume of particles in the 0.5 to 15.0 micron range. The peak value is at about 4.0 microns. At the low shear rates (i.e. 40 mL/sec recirculation flow rate), larger particles in the range of 15 to 300 microns are present. At this shear rate these particles represent about 26% of the total volume. As the shear rate increases (due to increasing the flow rate from 40 mL/sec to 60 mL/sec), the volume of large particles is reduced significantly to about 6% of the total volume in the 15 to 40 micron size range. Another reduction in large particles occurs due to the large shearing forces applied through sonication.

Table 5 presents the same data as in Table 4 but on a particle number basis. Obviously, a large difference in particle sizes reported between the volume basis and the number basis distributions exist. These differences can be explained by recognizing that as particle size increases the volume contribution of a particle increases cubically. This weighs the volume distribution more heavily towards larger particles (i.e. a few of the larger particles will greatly effect the volume basis but will have no significant effect on the number basis). Hence, Table 5 suggests that a large number of particles at approximately 0.9 microns exist. The bulk of these particles do not change due to changes in the shear force applied. However, small changes in the number of larger particles are greatly amplified on a volume basis (see Table 4).

Table 4: Particle Size Distribution (Volume) of Hanford Tank 241-AN-102 MOD2 Sample

Sample Conditions	Peak/Mode Number	Peak/ Mode Particle Size (μm)	Peak/Mode Width (μm)	Approximate Volume Percent of Particles in Peak/Mode
X-100 at 40 ml/s	1	230	78	5%
	2	68	95	21%
	3	3.9	6.4	74%
X-100 at 60 ml/s	1	30	10	6%
	2	4.0	6.7	94%
X-100 at 60 ml/s with 90 sec sonication at 40W #1	1	4.1	8.0	100%
X-100 at 60 ml/s with 90 sec sonication at 40W #2	1	4.1	7.9	100%

Table 5: Particle Size Distribution (Number) of Hanford Tank 241-AN-102 MOD2 Sample

Sample Conditions	Peak/Mode Number	Peak/ Mode Particle Size (μm)	Peak/Mode Width (μm)	Approximate Number Percent of Particles in Peak/Mode
X-100 at 40 ml/s	1	0.85	0.84	100%
X-100 at 60 ml/s	1	0.88	0.85	100%
X-100 at 60 ml/s with 90 sec sonication at 40W #1	1	0.89	0.84	100%
X-100 at 60 ml/s with 90 sec sonication at 40W #2	1	0.89	0.84	100%

The particle size distributions on a volume basis are presented graphically in Figures 1–3. In these figures, the left y-axis represents the volume percent of particles in a small, discrete range. The right y-axis represents the sum of these data from small particles to large particles and is referred to as the cumulative volume percent. These data represent the volume percent of particles smaller than a given particle size. The volume distribution data indicate that most of the particles are in the 0.5 to 15 micron particle size. As the shear rate in the instrument increased (due to increasing the flow rate from 40 mL/sec to 60 mL/sec) some of the larger particles (i.e. particles greater than 40 micron) appear to break apart, resulting in a larger number of smaller particles. When sonication is applied (i.e. 40W for 90 seconds) at the same flow rate (60 mL/sec), the particles in the 20 to 40 micron size appeared to further break apart. A second sonication resulted in no significant change.

The particle size distributions on a number basis are presented graphically in Figures 3–6. As discussed above, only small changes are observed in the number basis at each shearing condition.

Lastly, samples from the X100 recirculation system were taken and analyzed in the UPA before and after sonication. Unfortunately, the UPA indicated a low solids loading from both of these samples. Consequently, data from this instrument should be considered suspect. However, the UPA consistently indicated a large volume/number of particles in the 5 to 20 nm range.

Figure 1: AN-102 MOD2 Particle Size Analyses at Various Flow Rates (Volume Basis)

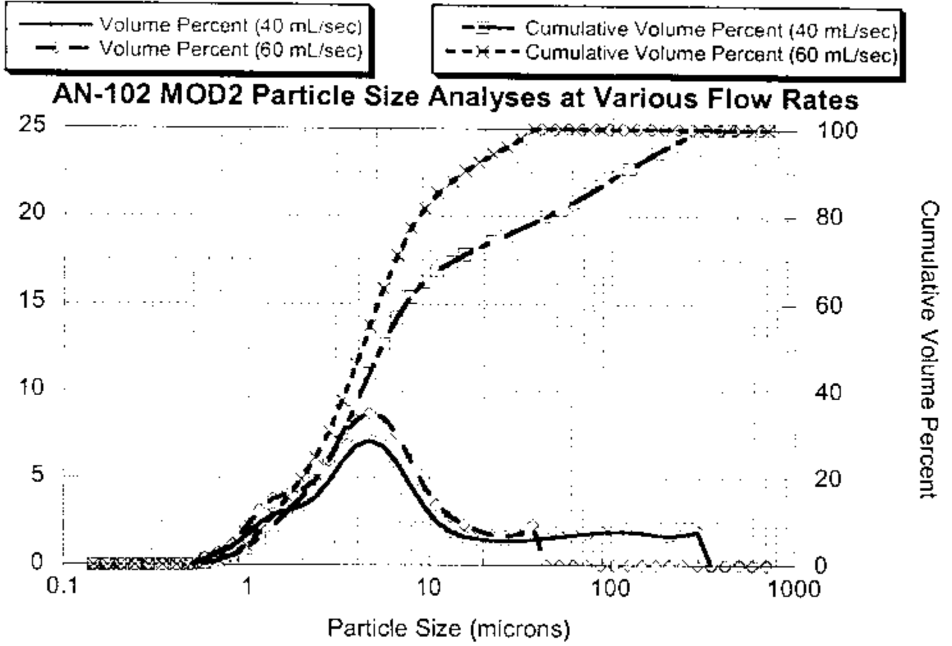


Figure 2: AN-102 MOD2 Particle Size Analyses at Before and After Sonication (Volume Basis)

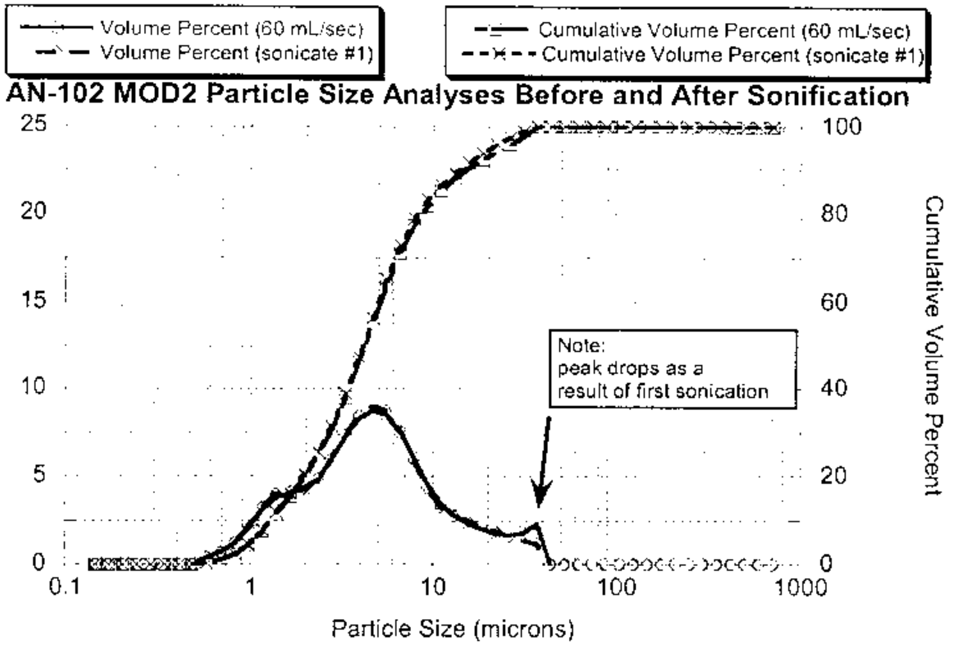


Figure 3: AN-102 MOD2 Particle Size Analyses at Before and After 2nd Sonication (Volume Basis)

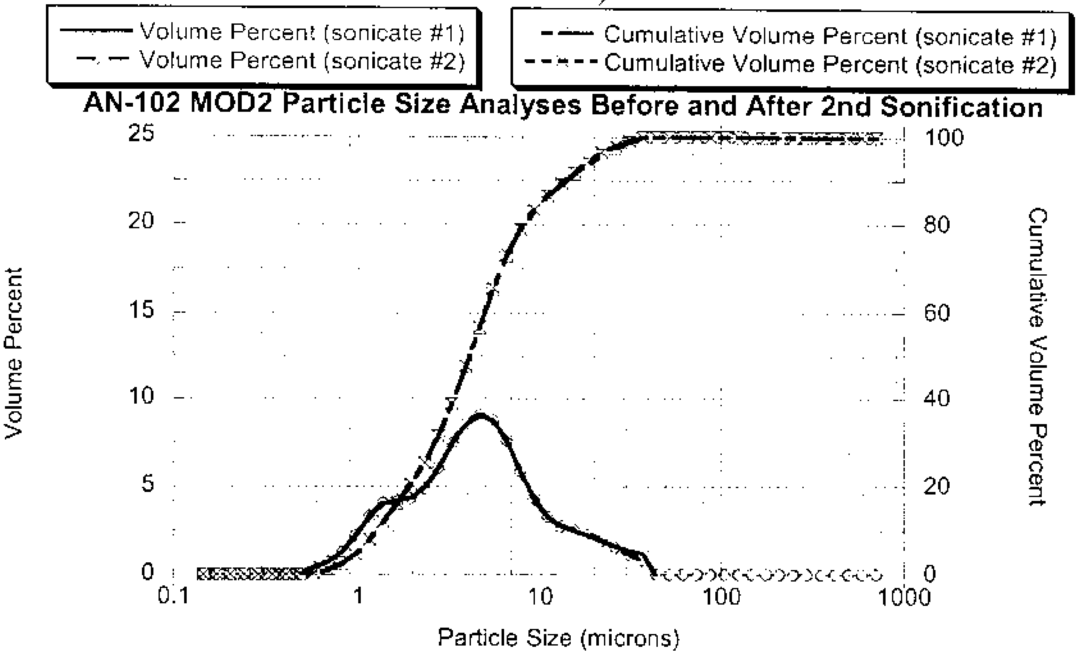


Figure 4: AN-102 MOD2 Particle Size Analyses at Various Flow Rates (Number Basis)

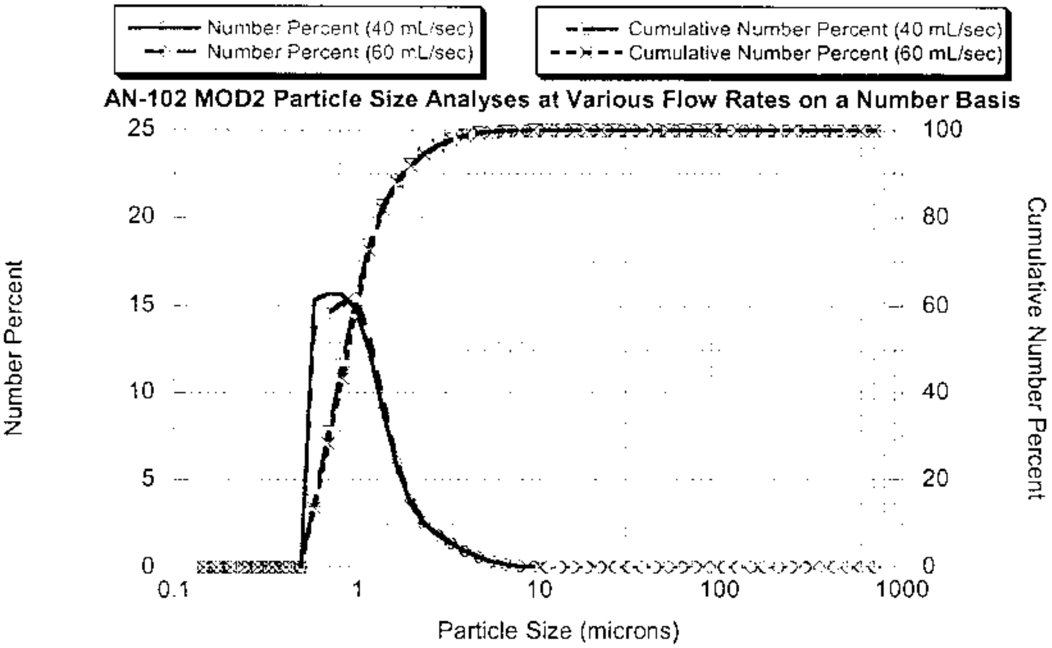


Figure 5: AN-102 MOD2 Particle Size Analyses at Before and After Sonication (Number Basis)

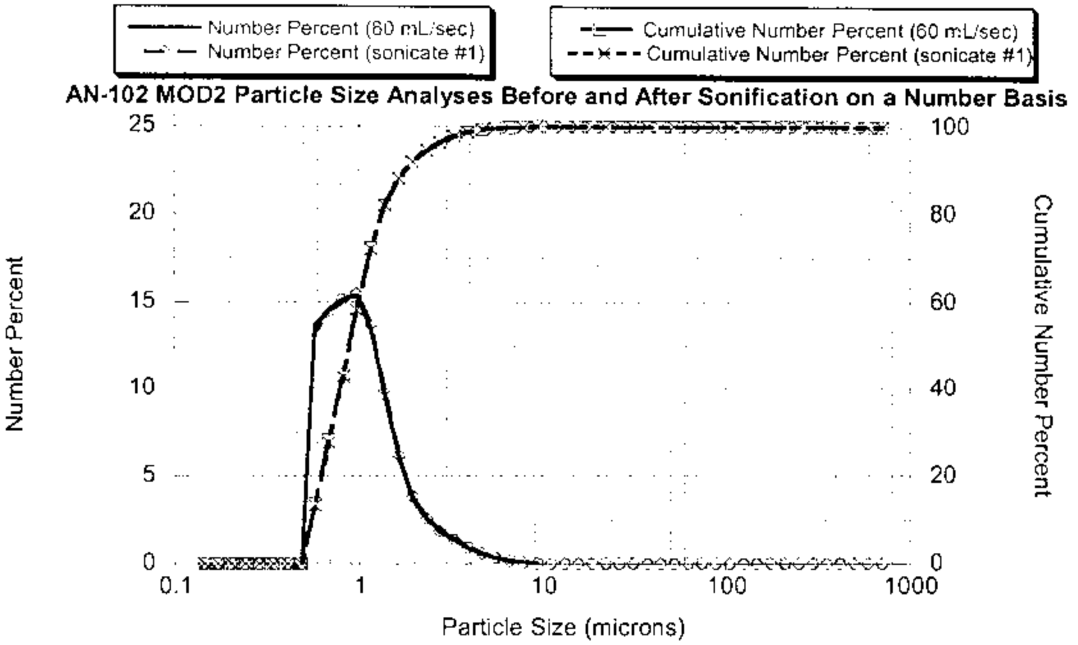
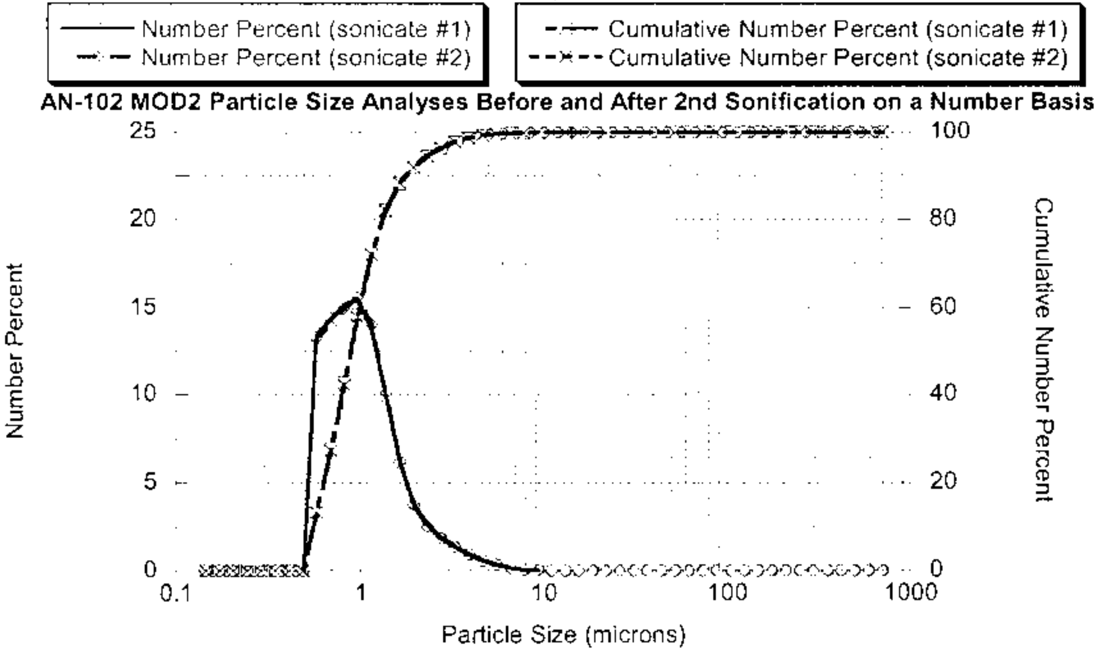


Figure 6: AN-102 MOD2 Particle Size Analyses at Before and After 2nd Sonication (Number Basis)



1.1.1 Rheology

The following Rheology Report and Data package was reviewed by:

signature

11/30/01
Date

Rheology testing of the 2-wt% UDS AN-102 slurry (AN-102 Mod 2) was performed with the Haake M5 head in the HLRF. The primary tests were standard shear stress vs. shear rate curves.

1.1.1.1 Background

Viscosity is the internal resistance to flow of a fluid against external forces. Viscosity is mathematically defined as the shear stress divided by the shear rate. For a Newtonian fluid this ratio is constant. For non-Newtonian fluids this ratio can change based on flow conditions and shear history. The rheological data most often requested and provided is 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 a rheogram viscosity data, yield stress data and flow curve information are obtained. Viscosity is usually reported in centipoises (cP). One cP is equal to a millipascal second. There are several types of 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:

Prepared By

11/29/01

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

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

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

4) Herschel-Bulkley Equation
(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, $^1/_s$)
η = Viscosity (Pascal-seconds, Pa-s; reported in cP)
η_p = coefficient related to flow resistance, similar to 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 but 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 pseudo-plastic, or power law fluid, has a viscosity that varies with stress in a non-linear fashion. A yield pseudo-plastic is a power law fluid with a yield point.

1.1.1.2 Equipment Capabilities and Sensor Selection

The Haake 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 M5 head's design specifications give it a maximum defection of 1% of full torque with a sensitivity range of up to 0.001° 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 viscosity and shear rate ranges available for any given measurement. For the 2-wt% UDS AN-102 slurry samples, the NV sensor system was utilized. This sensor system has the most available surface area and therefore yields the highest sensitivity, which is important for low viscosity fluids. The NV sensor's small gap size limits use to material with low solids concentrations. This measurement head and senor combination has an optimum effective viscosity range of 1 cP to 10,000 cP over a shear rate range of 0 $^1/_s$ to 2700 $^1/_s$. A 100 cP standard oil is used to validate the calibration of the machine.

1.1.1.3 Sample Testing Parameters

For the 2-wt% UDS AN-102 slurry samples a standard 0 to 1000 $^1/_s$ ramp test over 6 minutes was completed for each sample. Several samples were ramped over a period of 20 minutes with no discernable difference in observed behavior; therefore, 6 minutes was chosen as the ramp time for the remaining analyses. Each sample was tested at least twice. The pertinent resulting rheograms and viscosity curves are included in Appendix B.

The samples were loaded and then ramped up from 0 to 1000 $^1/_s$ in 3 minutes and then 1000 to 0 $^1/_s$ in 3 minutes. There was a repeat of this ramp cycle at least once for each sample. Thus each sample was tested through a minimum of two complete ramp cycles from 0 to 1000 $^1/_s$ over a total time of 6 minutes. If the second run data was a close overlay of the first run data, then the testing for that sample was considered complete. If there was a noticeable variation in the data, then the sample was ramped though this cycle again until two consecutive similar data sets were obtained. This repetition is 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.

The purpose of this set of testing parameters was to identify the rheological behavior and shear sensitivity of the materials. 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 with the used sample shows the effects of a shear history with a short time of recovery for the sample.

The 2-wt% UDS AN-102 slurry samples were tested at three separate temperatures, 25 °C, 35 °C and 50 °C to evaluate the temperature effects on viscosity and flow curves at possible expected process conditions.

1.1.1.4 Sample Rheology Results

Each run of the 2-wt% UDS AN-102 slurry samples showed a pseudoplastic nature with little or no yield point. 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. Table 2.5 presents viscosity data calculated at specific shear rates.

As shown by Figures 2.2 and 2.3, there was a drop in the fluid resistance (i.e., viscosity) with increasing temperatures. The apparent viscosity decreased approximately 50% when the temperature was increased from 25 °C to 35 °C. The continued rise in temperature up to 50 °C resulted in a further decrease of the viscosity but not such a significant one. The change in temperature did not change the flow profile to any relevant extent.

Table Error! No text of specified style in document..**1. Viscosity at Specific Shear Rates; 2-Wt% UDS AN-102**

	Yield [a]	**Viscosity (cP)** [b]					
Material	**(Pa)**	**@33 $^{1}/_{s}$**	**@150 $^{1}/_{s}$**	**@300 $^{1}/_{s}$**	**@500$^{1}/_{s}$**	**@750 $^{1}/_{s}$**	**@990 $^{1}/_{s}$**
AN-102 25 °C Run 1	<2	34.4	22.6	22.0	19.7	17.2	15.4
AN-102 25 °C Run 2	<2	26.5	22.3	21.5	18.5	16.9	15.5
AN-102 35 °C Run 1	<2	33.3	15.6	15.4	12.7	11.8	10.7
AN-102 35 °C Run 2	<2	16.5	13.2	12.9	11.3	11.0	10.2
AN-102 50 °C Run 1	<2	19.6	15.7	12.5	11.6	10.7	9.9
AN-102 50 °C Run 2	<2	17.8	13.0	12.5	11.1	10.3	9.9

[a] Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits, the numbers are not exact, and when there is a disparity between multiple runs the highest yield is reported to be conservative. A <2 results indicates the material may have a small yield but it is insignificant.

[b] Viscosity data is in cP and is a specific number selected at a specific shear rate for each run.

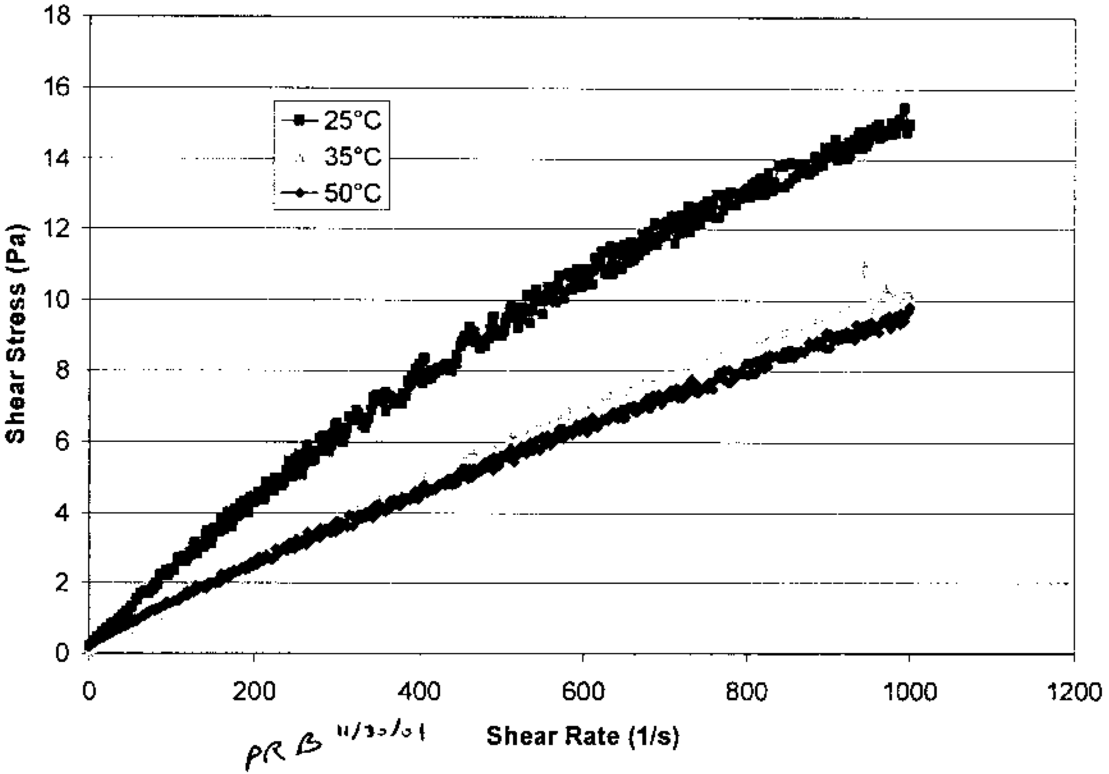


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Figure ~~Error! No text of specified style in document.~~1. Shear Stress Versus Shear Rate for 2-wt% UDS AN-102 Slurry

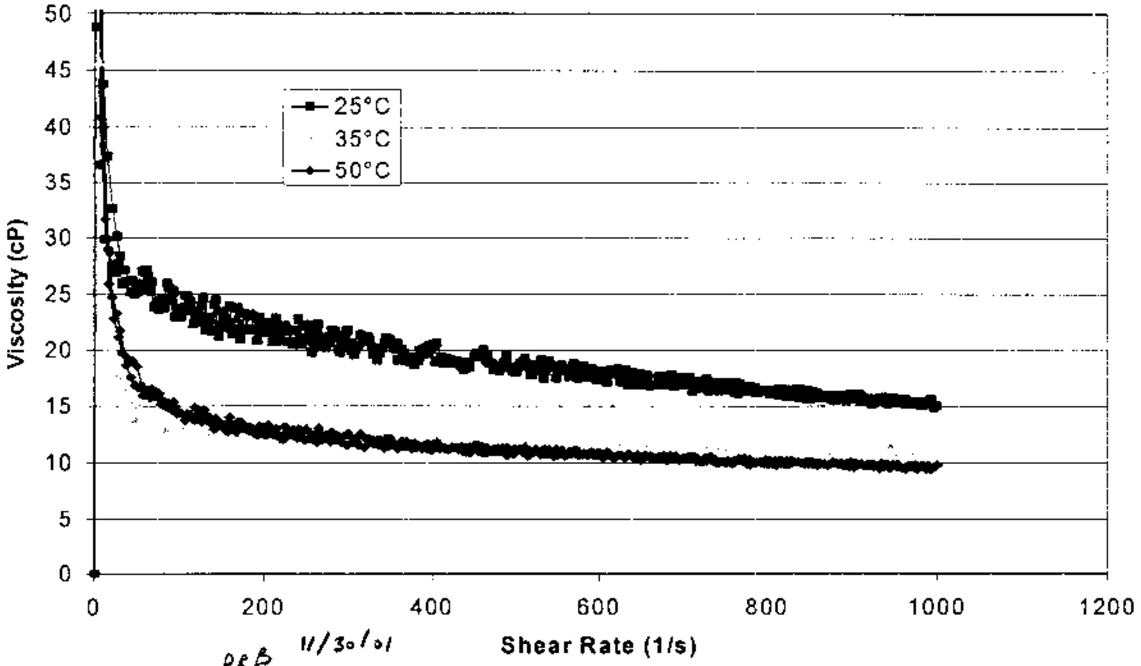

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Figure ~~Error! No text of specified style in document~~.2. Viscosity Versus Shear Rate for 2-wt% UDS AN-102 Slurry

The Haake M5 with NV sensor 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 itself rather than actual fluid resistance. It is likely that this contributed to the small "yield stress" seen 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. For example, at 33 $^{1}/_{s}$ shear rate the 35 °C samples had vastly different "single point" values (i.e., 33 cP and 16 cP). However the overall curve data is almost identical. It is better to use the curve equations to predict the viscosities at any given shear rate and singular data points.

1.1.1.5 Rheology Curve Fits

As can be seen in Table 2.6, the best curve fits for the material were found to be Herschel-Bulkley or Ostwald fits with an 'n' factor of > 0.8 and often above 0.9 for most of the fits. The 'n' factor is a measure of the degree of pseudo-plasticity or deviation from Newtonian the fluid exhibits. For Newtonian fluids the 'n' factor equals unity. The 'n' factor of greater than 0.8 for the 2-wt% UDS AN-102 slurry composite indicates the pseudo-plastic nature of this material is present, but is slight. The Herschel-Bulkley model does have a yield factor, but in this case it is most likely an artifact of the Haake system due to the need to overcome inertial mechanical forces and can be neglected. Given the lack of the observed yield stress in the Herschel-Bulkley model, an Ostwald model is the most accurate overall model for this material. The Newtonian model was not evaluated since all samples demonstrated some degree of pseudo-plasticity.

Table Error! No text of specified style in document..**2. Model Fit Parameters**

Sample [a]	**Ostwald**			**Herschel-Bulkley**				**Bingham**		
	η_p	n	R^2	τ_o	η_p	n	R^2	τ_o	η_p	R^2
25 °C Run 1	0.05943	0.8135	1.00	-0.1162	0.07828	0.7713	1.00	1.592	0.01478	0.98
25 °C Run 2	0.05726	0.8166	1.00	0.0840	0.04164	0.8666	0.99	1.486	0.01473	0.98
35 °C Run 1	0.08108	0.7081	0.97	0.2102	0.03547	0.8358	0.91	1.390	0.009876	0.98
35 °C Run 2	0.03030	0.8443	0.99	0.05816	0.02214	0.8931	1.00	0.6015	0.01006	0.99
50 °C Run 1	0.1207	0.6236	0.88	0.8383	0.0121	0.9631	0.76	1.220	0.00891	0.94
50 °C Run 2	0.05002	0.7581	0.98	0.2362	0.0158	0.9362	1.00	0.7177	0.009415	0.99

[a] Results for first and second run at each temperature presented. Data for additional runs is included in Appendix B.

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