PNWD-3228
WTP-RPT-021, Rev 1

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

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December 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
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 3M 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 (wt%) 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-wt% solids AN-102 waste are reported.

Filtrate, leachate, and wash solutions from tank C-104 processing were blended with the 2-wt% UDS 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 analysis (KPA) 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(k)	Deriv/GC/FID	5E-01	UX	n/m		(b)	(b)
Dy	ICP-AES	*3E+00*	*U*	*3E+00*	*U*	<3E+00	<2E+01
ED3A(k)	Deriv/GC/FID	6.50E+02	JX	n/m		(b)	(b)
EDTA(k)	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(k)	Deriv/GC/FID	1E+02	UX	n/m		(b)	(b)
IDA(k)	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(k)	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(k)	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.
(k) Results are qualitative (See Section 6.9 and 6.10).

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)		
50	PO_4(d)		
46	NO_2		
39	NO_3		
28	Cl		
25	Al		
22	Ni		
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 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

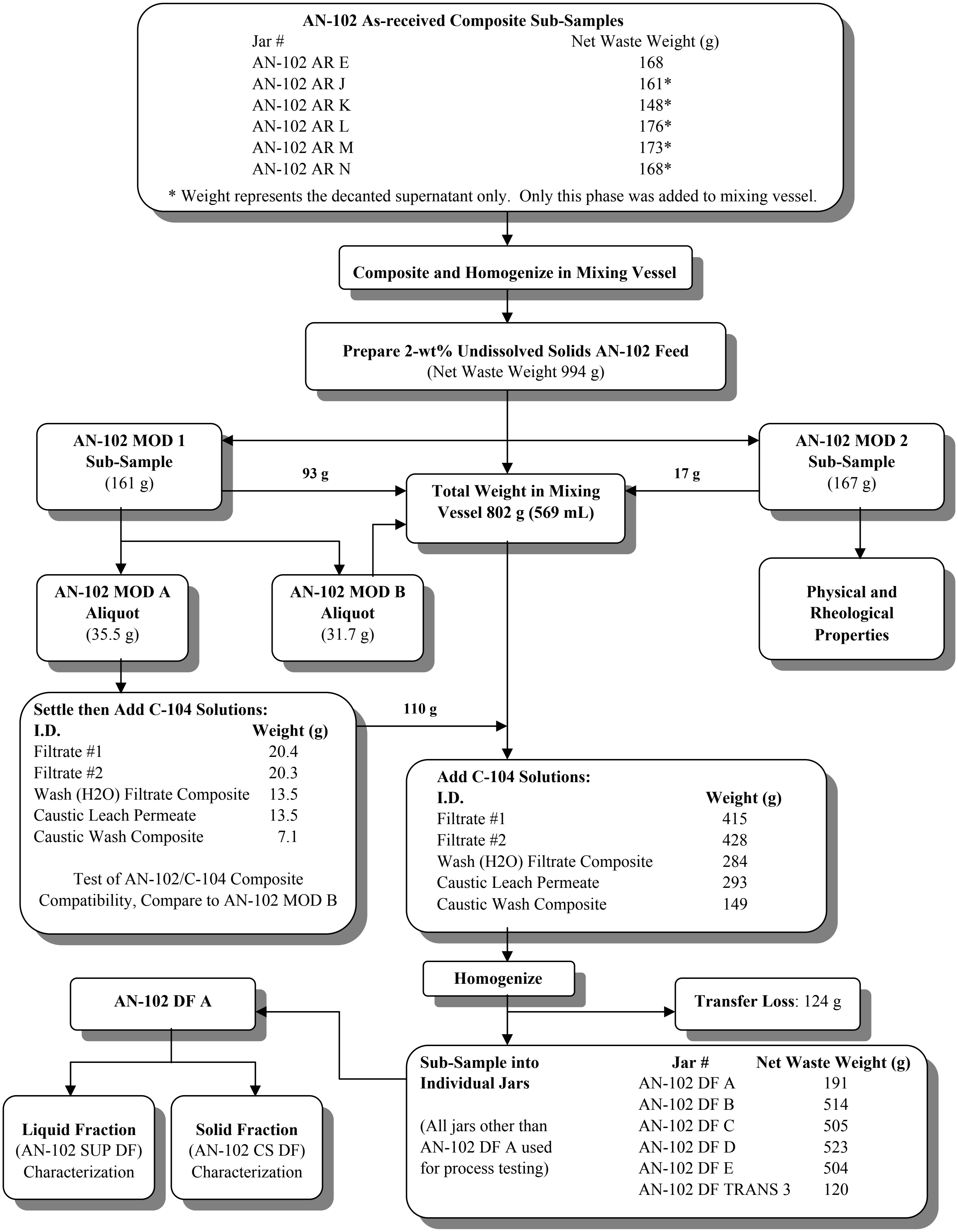
2.1 Overall Process Description

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.

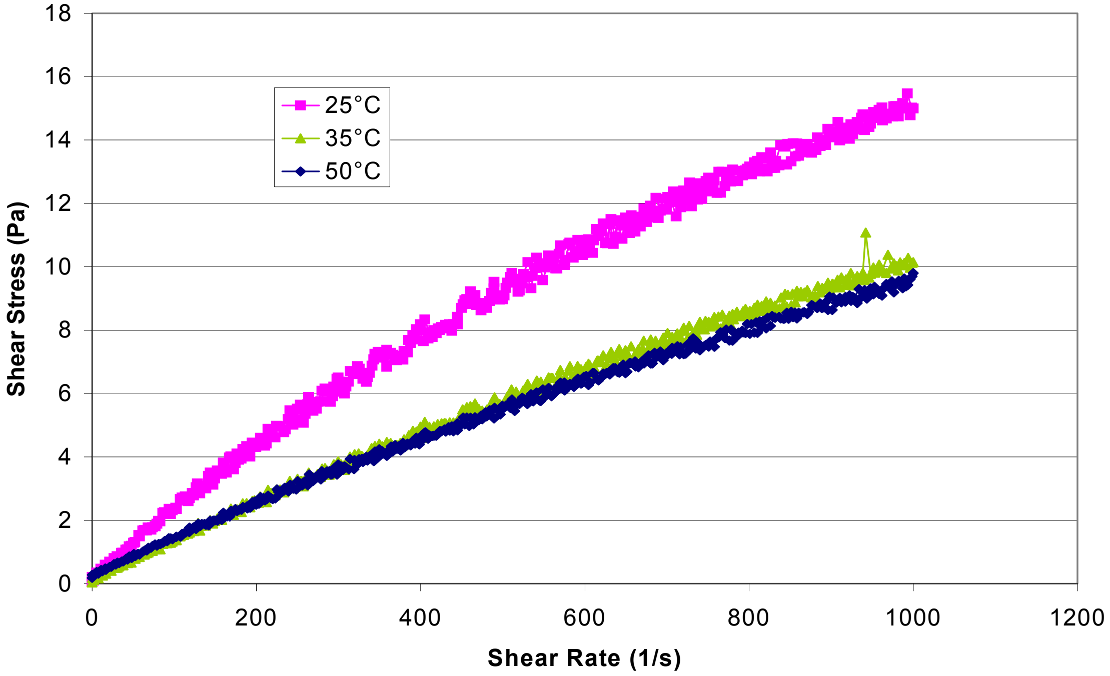


Figure 2.2. Shear Stress Versus Shear Rate for 2-Wt% UDS AN-102 Slurry

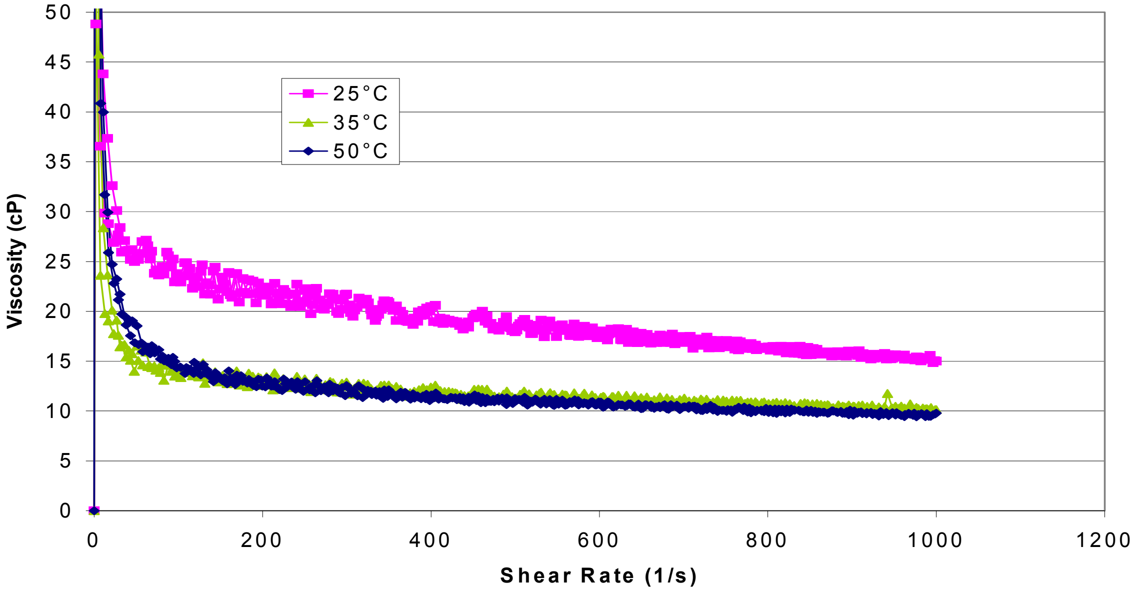


Figure 2.3. Viscosity Versus Shear Rate for 2-Wt% UDS AN-102 Slurry

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

	Ostwald			**Herschel-Bulkley**				**Bingham**		
Sample [(a)]	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 temperature 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	

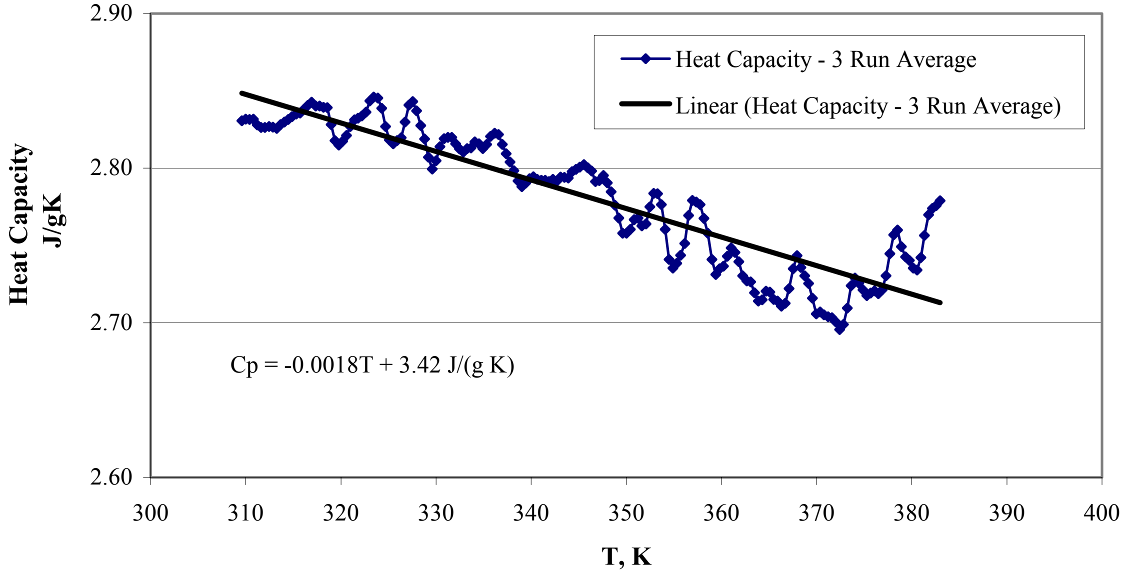


Figure 2.4. Average Heat Capacity of 2-Wt% UDS AN-102 Slurry

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 is 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
$Al(NO_3)_3•9H_2O$	1.03	$NaCO_3$	1.00
Na2SO4	0.135	$Na_2C_2O_4$	0.00637
$Na_2HPO_4•7H_2O$	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.

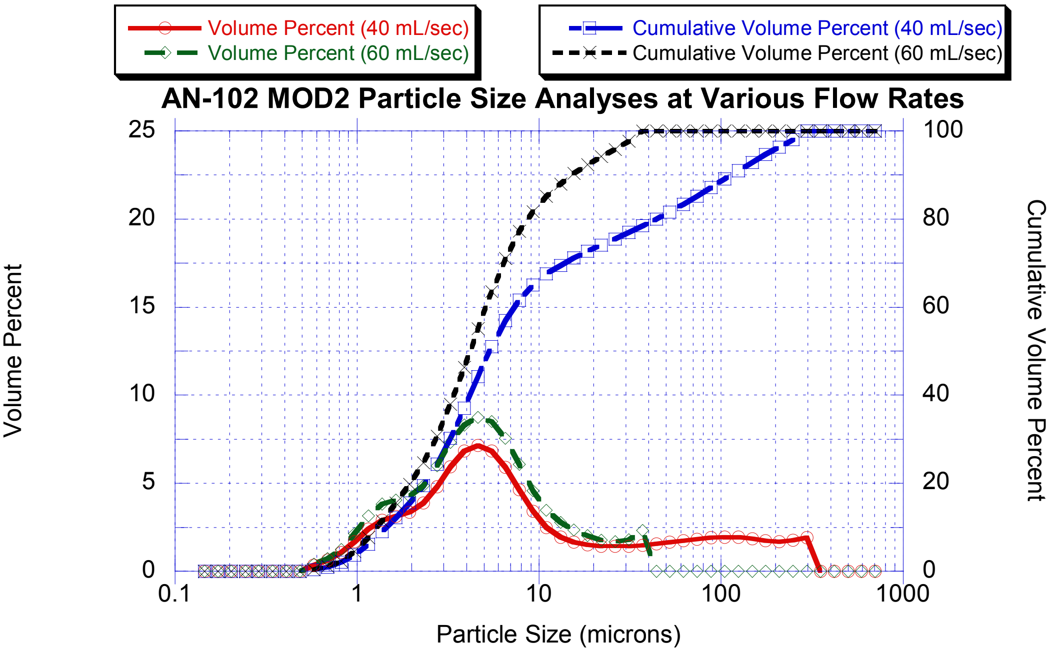


Figure 2.5. Particle Size Analyses at Various Flow Rates (Volume Basis) of 2-Wt% UDS AN-102 Slurry

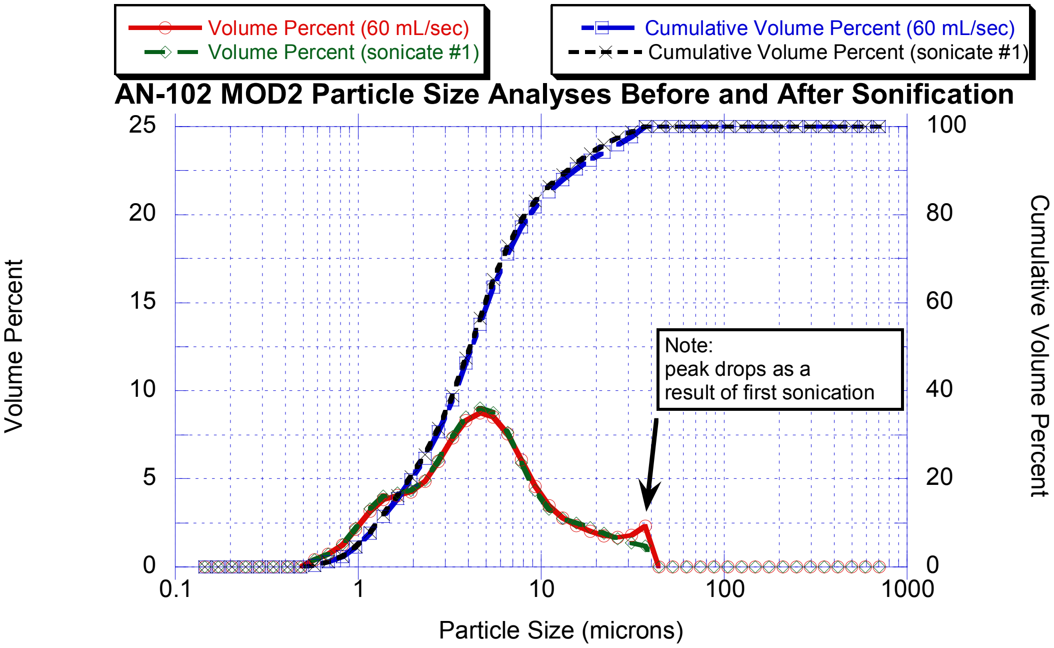


Figure 2.6. Particle Size Analyses Before and After Sonication (Volume Basis) of 2-Wt% UDS AN-102 Slurry

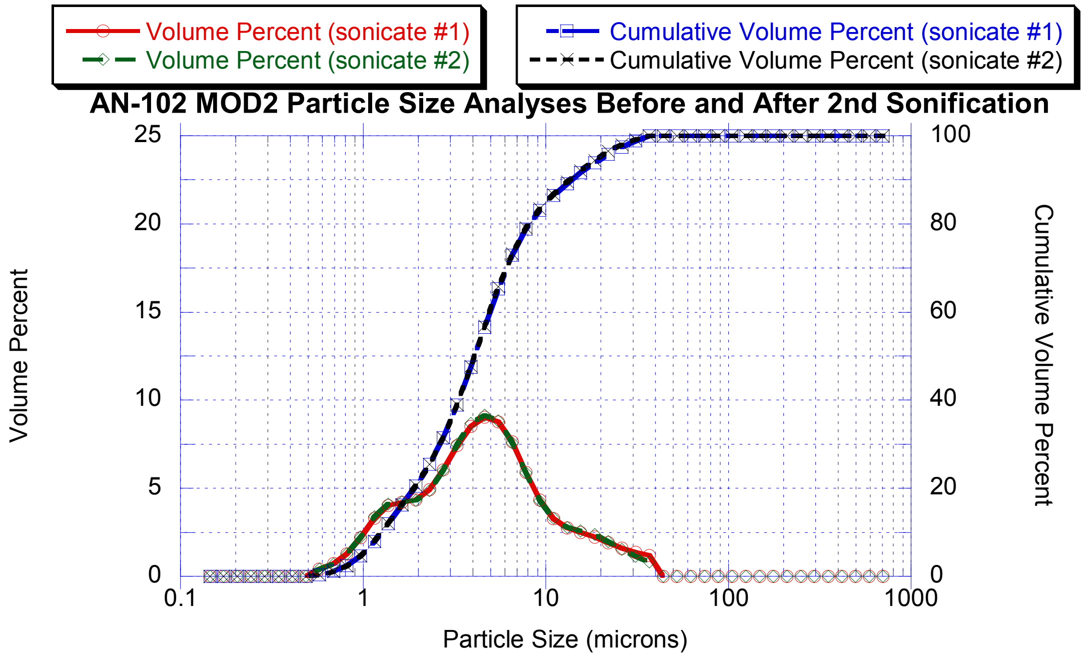


Figure 2.7. Particle Size Analyses Before and After 2nd Sonication (Volume Basis) of 2-Wt% UDS AN-102 Slurry

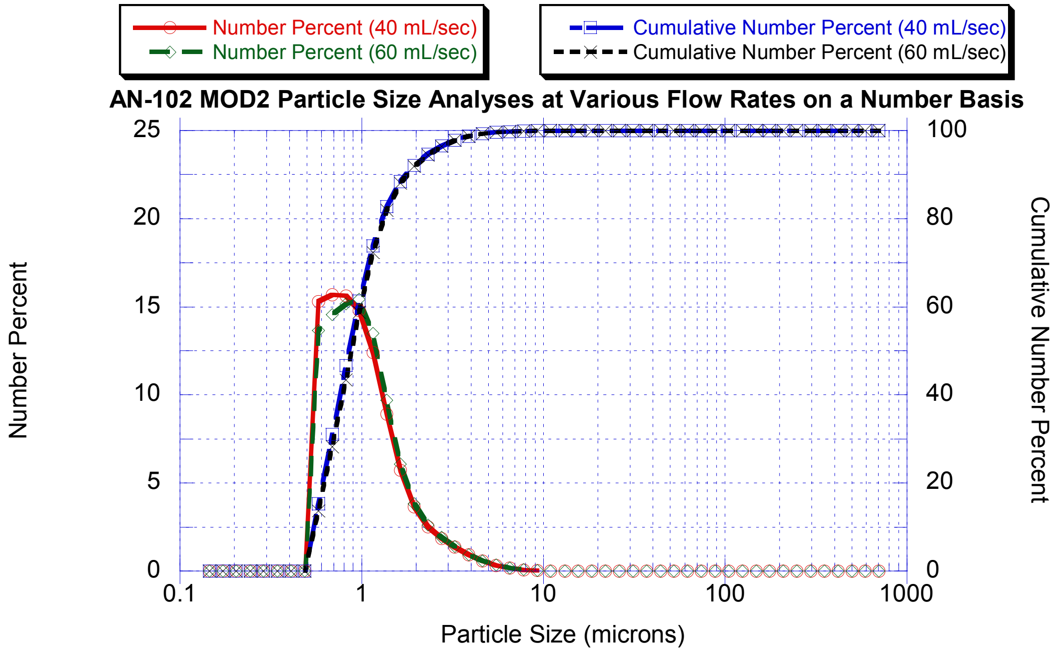


Figure 2.8. Particle Size Analyses at Various Flow Rates (Number Basis) of 2-Wt% UDS AN-102 Slurry

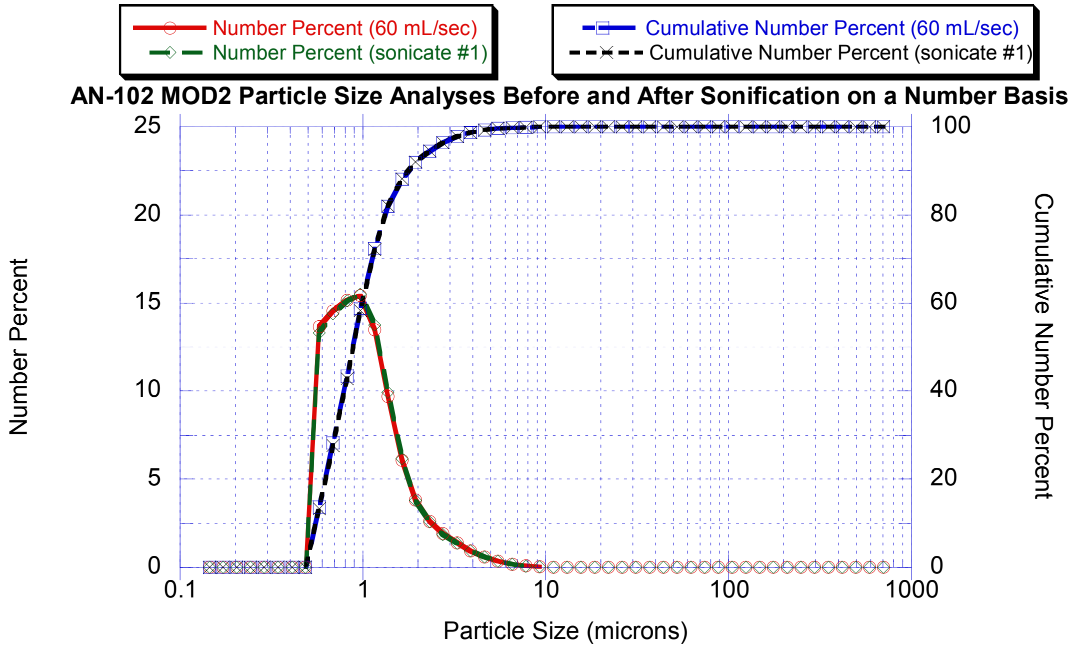


Figure 2.9. Particle Size Analyses Before and After Sonication (Number Basis) of 2-Wt% UDS AN-102 Slurry

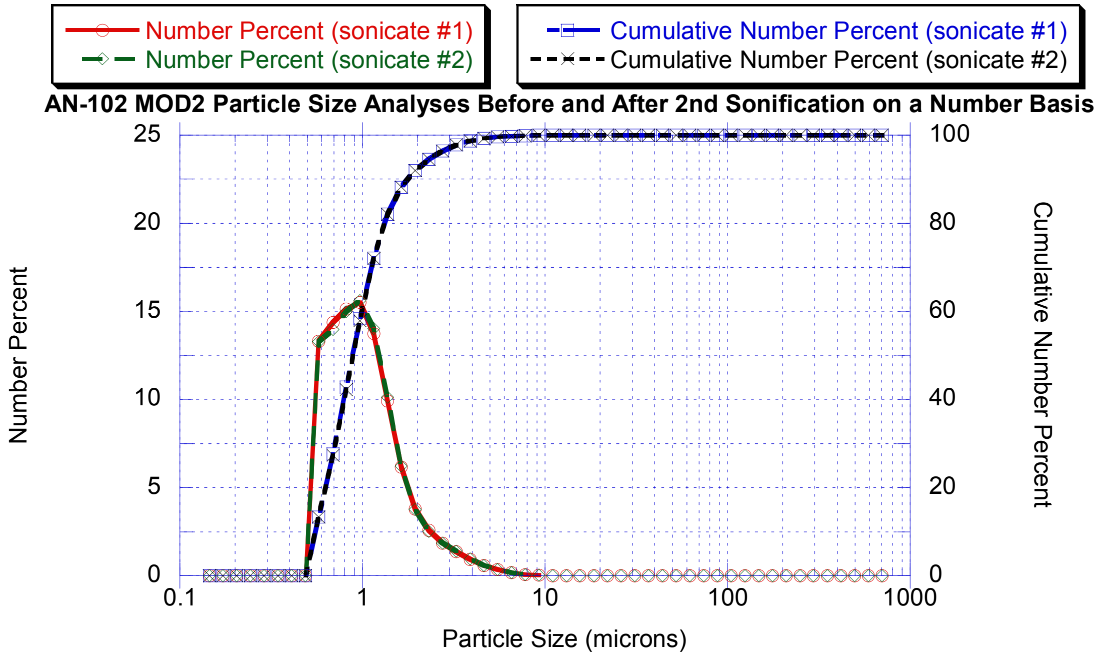


Figure 2.10. Particle Size Analyses Before and After 2nd Sonication (Number Basis) of 2-Wt% UDS AN-102 Slurry

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 (2-wt% 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**	**Mass Lost during Phase Separation**	**Percent Lost during Phase Separation**
RPL Number	**Bottle ID**	**(g)**	**(g)**	**(%)**
None [(a)]	AN-102-DF A	191.4	2.1	1

Supernatant and Wet Centrifuged Solids Phase Separated Samples		**Bottle Tare Mass**	**Bottle + Sample Mass**	**Sample Mass**	**Total Mass**
RPL Number	**Bottle ID**	**(g)**	**(g)**	**(g)**	**(g)**
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)

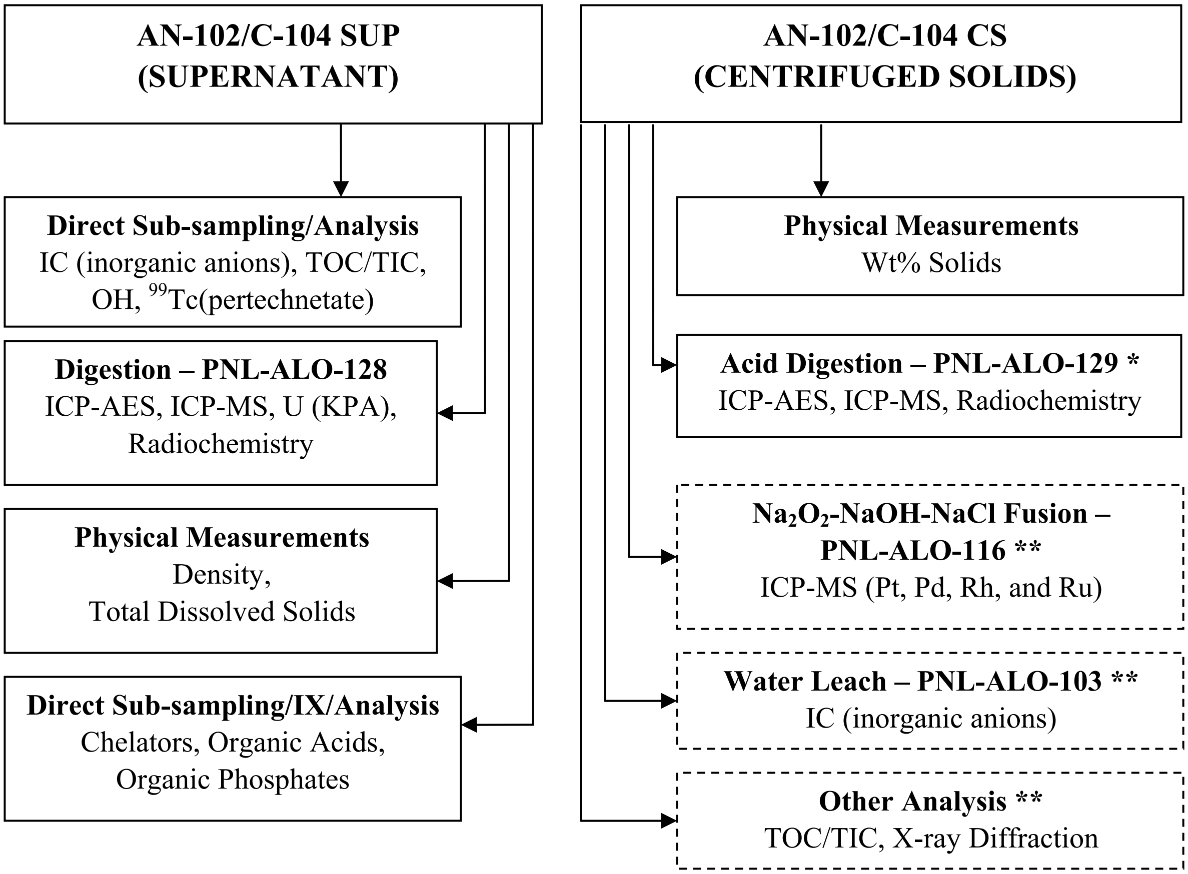
(a) After Drying at 105°C to constant weight

(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 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 EQL is typically set at 10 times the estimated

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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.

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 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/C-104 blended feed 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).
- The derivatization-GC/FID analysis for D2EHP was performed on a best effort basis. The results for D2EHP are considered qualitative, due primarily to the poor LCS/BS recoveries. Based on additional laboratory testing not presented in this report, the poor LCS/BS recoveries may be due to ionic strength variations, pH of the extraction, and/or stability of the derivative. Additional work in needed on the derivatization-GC/FID technique to develop a more robust method.
- Chelators and degradation products were measured by a derivatization-GC/FID method, with compounds being confirmed by GC/MS. However, the results of three compounds (HEDTA, ED3A, and IDA) are considered qualitative, since the results are estimates based on the EDTA calibration. The results are calculated from the compound response and the regression equation from the EDTA calibration, assuming that the response of HEDTA, ED3A, and IDA are equivalent (or at least similar) to that of EDTA. The EDTA calibration was used for estimating the HEDTA, ED3A, and IDA because lack of commercially available standards for ED3A and unsuccessful attempt to generate stable, linear calibrations from HEDTA and IDA standards.

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	

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
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
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**	**EQL/ MDL (a)**	**01-441**	**(g) Data**	**EQL/ MDL (a)**	**01-441D**	**(g) Data**
Analyte	**µg/mL**		**Flag**	**µg/mL**		**Flag**	**µg/mL**		**Flag**
Test Specification Analytes									
F (b)	0.25	0.25	U	200	3,700		200	3,700	
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	U	200	200	U
SO_4	0.5	0.5	U	400	4,470		400	4,390	
OH	170	170 (d)	U	170	4,230		170	5,030	
TOC-F (c)		n/a		140	15,800		60	15,700	
TIC-F (c)		n/a		140	200	J	60	400	J
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

Analyte	CAS #	MDL µg/mL	Process Blank 01-429-PB µg/mL	Process Blank Data Flag[(d)]	Sample 01-441 µg/mL	Sample Data Flag[(d)]	Duplicate 01-441D µg/mL	Duplicate Data Flag[(d)]
Organic Phosphate Analytes								
D2EHP[(e)]	298-07-7	0.5	0.5	UX	0.5	UX	0.5	UX
DPP (surrogate)	838-85-7		3% [(a)]		83% [(a)]		87% [(a)]	
Chelators and Degradation Products								
EDTA	60-00-4	120	120	UX	770	JX	460	JX
HEDTA[(c)]	150-39-0	120	120	UX	120	UX	120	UX
ED3A[(c)]		120	120	UX	760	JX	540	JX
NTA	139-13-9	100	100	UX	200	JX	130	JX
IDA (as NIDA)[(c)]	142-73-4	120	120	UX	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	UX	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; results are considered qualitative (See Section 6.9).
(d) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 5.1).
(e) D2EHP analysis performed on a best effort basis; results are considered qualitative (See Section 6.10).

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)

	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
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 Flag[(c)]	MDL	01-430-PB2	± 1SD	Data Flag[(c)]	MDL	01-442	± 1SD	Data Flag[(c)]	MDL	01-442D	± 1SD	Data Flag[(c)]
	µg/g				µg/g				µg/g				µg/g			
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^{o}C (0.351)
T = fractional solids weight in supernatant after drying at 105^{o}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 5.10. (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	600	15	1840		1.9E-02	3.8E-02	50	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), ethylenediaminetriacetic acid (ED3A), iminodiacetic acid (IDA), succinic acid, and nitrilotriacetic acid (NTA), have low volatility and high polarity precluding direct analysis by GC/FID. Derivatizing the chelators with a BF_3/methanol mixture results in a methyl ester product that is amenable to GC/FID separation and analysis.

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.

The identification of target analytes was confirmed by GC/MS; tentative identification of ED3A and nitroso-ED3A were based on mass spectral data. Since no standards are available for ED3A or the measured compound nitroso-ED3A, the concentration of ED3A is based on the EDTA calibration and assumes that an equivalent response to EDTA. HEDTA and IDA appeared to be very dependent on the final pH achieved during the addition of the phosphate buffer. This resulted in very high variability in the measured results and severely non-linear calibration curves. Based on these results, the HEDTA and IDA were also estimated based on the EDTA calibration; i.e., no calibration curve was generated for HEDTA or IDA (or the measured IDA compound, nitroso-IDA or NIDA). HEDTA, IDA, and E3DA results are considered qualitative.

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/BS recovery for citric acid was within the typical 80% to 120% recovery acceptance criterion. The NTA and EDTA LCS/BS recoveries of 71% and 78%, respectively, were slightly below the typical lower threshold of 80% recovery. The HEDTA LCS/BS 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.

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/BS and the low recovery of D2EHP in the LCS/BS cannot be confirmed. However, additional testing was conducted to evaluate these poor recoveries. This testing suggest that the low recoveries may be due to 1) ionic strength variations, 2) pH of extraction, and/or 3) stability of the derivative. Based on the low LCS recoveries and the lack of validation of the derivatization-GC/FID technique for tank waste materials, the reported results are considered qualitative.

Besides the routine QC samples (e.g., MS, MSD, LCS/BS, 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	**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	**72**	**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		**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		$\boxed{\textbf{17}}$	<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.	MS Duplicate (MSD) % Rec.
Acceptance Criteria								*n/a*	*n/a*	*n/a*
Organic Phosphate Analytes										
D2EHP (h)	298-07-7	1,500	0.5	0.5	UX	(a)		42	84	107
DPP (surrogate)	838-85-7			85% (b)		5		5%(b)	73%(b)	93%(b)
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; results are considered qualitative (See Section 6.9).
(g) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 5.1).
(h) D2EHP analyzed on a best effort basis; results are considered qualitative (See Section 6.10).

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) 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		**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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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)

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

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