PNWD-3229
WTP-RPT-020, Rev 1

Chemical Analysis and Physical Property Testing of 241-AN-102 Tank Waste—Supernatant and Centrifuged Solids

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

LEGAL NOTICE

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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) 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, characterization, combination with C-104 permeate solutions, additional characterization, Sr/TRU (transuranic) precipitation, Cs and Tc removal using ion exchange, and vitrification of the resulting low-activity waste and high-level waste streams. This report summarizes sample receipt, compositing, homogenization, and initial characterization activities of the AN-102 tank waste.

Samples taken in August 2000 from Tank AN-102 were provided to the PNWD. Eight of these samples, taken 190 cm from the tank floor at the top of the sludge layer, were homogenized into a single slurry or composite and carefully separated into 15 sub-samples. The solids and supernatant phases were separated and tested for physical properties, including density and percent solids. Four composite sub-samples were selected for inorganic, radiochemical, and selected organic analysis, including polychlorinated biphenyls (PCBs). Most analyses were performed in duplicate on both the solid and supernatant phases.

The characterization of the representative AN-102 composite sub-samples for both supernatant and wet centrifuged solids included but was not limited to:

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

Table S.1 summarizes the physical properties measured on the AN-102 as-received material.

Table S.1. AN-102 As-Received Composite—Physical Properties

Test	Matrix/Processing	Average	RPD
Density (g/mL)	Slurry	1.464	1%
	Supernatant	1.406	0.5%
	Wet Centrifuged Solids	1.528	2%
Weight Percent Solids (%)	Solids Wt% in Supernatant After Drying at 105 °C	49.2	0.2%
	Solids Wt% in Centrifuged Solids After Drying at 105 °C	58.8	0.3%
Weight Percent Oxide (%)	Solids Wt% in Supernatant After Firing at 1050 °C	24.7	4%
	Solids Wt% in Centrifuged Solids After Firing at 1050 °C	43.4	0.5%
RPD = Relative Percent Difference			

Tables S.2 and S.3 present the supernatant and wet centrifuged solids results. The wet centrifuged solids results contain interstitial supernatant. The supernatant contribution to the wet centrifuged solids results has been mathematically removed and is reported as undissolved solids (UDS) analyte concentration. The calculated UDS results are reported as micrograms per gram on both a wet-weight and dry-weight basis. Because the solids were not washed, they contain components that may be removed during pretreatment.

Table S.2. AN-102 As-Received Composite—Analyte Summary

		Supernatant		**Wet Centrifuged Solids**		**Calculated UDS**	
Analyte	**Measure Method**	**Average µg/mL**	**Data Flag(g)**	**Average µg/g**	**Data Flag(g)**	**Average µg/g (wet)**	**Average µg/g (dry)**
Ag	ICP-AES	*2.E+00*	*UX*	2.3E+00	UX	<2E+00	< 1E+01
Al	ICP-AES	1.23E+04		2.85E+04		2.14E+04	1.13E+05
As	ICP-MS	NM		6.53E+00	JB	6.53E+00	3.46E+01
B	ICP-MS	NM		3.95E+01	BX	3.95E+01	2.09E+02
Ba	ICP-AES	*9.E-01*	*U*	2.75E+01		2.75E+01	1.46E+02
Be	ICP-MS	NM		1.12E+00	J	1.12E+00	5.93E+00
Bi	ICP-AES	9.4E+00	U	9.4E+00	U	<9E+00	<5E+01
Ca	ICP-AES	4.89E+02		4.21E+02		1.38E+02	7.30E+02
Cd	ICP-AES	6.2E+01		3.8E+01		2.38E+00	1.26E+01
Ce	ICP-MS	NM		2.30E+01		2.30E+01	1.22E+02
Citrate	Organic/IC	4.40E+03		NM		(a)	(a)
Cl	IC	4.80E+03		3.3E+03 (b)	B	5.50E+02	2.91E+03
Cyanide	Dist./Color.	NM		3.3E+01		3.3E+01	1.74E+02
Co	ICP-MS	NM		2.36E+00	X	2.36E+00	1.25E+01
Cr	ICP-AES	2.15E+02		1.83E+03		1.70E+03	9.00E+03
^{133}Cs	ICP-MS	9.60E+00		NM		(a)	(a)
Cs (total)	ICP-MS and GEA	1.60E+01		9.36E+00(i)		1.51E-01	7.99E-01
Cu	ICP-AES	*2.3E+01*	*J*	1.9E+01	J	5.44E+00	2.88E+01
D2EHP(j)	Deriv/GC/FID	5.E-01	UX	NM		(a)	(a)
ED3A(j)	Deriv/GC/FID	1.00E+03	X	NM		(a)	(a)
EDTA(j)	Deriv/GC/FID	4.20E+02	JX	NM		(a)	(a)
F (c)	IC	3.E+03	U	3.80E+03		3.80E+03	2.01E+04
Fe	ICP-AES	3.7E+01		1.17E+03		1.14E+03	6.03E+03
Formate	Organic/IC	8.00E+03		NM		(a)	(a)
Gluconate	Organic/IC	1.E+03	U	NM		(a)	(a)
Glycolate	Organic/IC	1.05E+04		NM		(a)	(a)
HEDTA(j)	Deriv/GC/FID	1.E+02	UX	NM		(a)	(a)
Hg	CVAA	1.40E-04	U	1.90E-03		1.85E-03	9.79E-03

Table S.2. (Cont'd)

		Supernatant		Wet Centrifuged Solids		Calculated UDS	
Analyte	**Measure Method**	**Average µg/mL**	**Data Flag(g)**	**Average µg/g**	**Data Flag(g)**	**Average µg/g (wet)**	**Average µg/g (dry)**
^{127}I	ICP-MS	NM		2.45E+00	J	2.45E+00	1.30E+01
^{127}I (d)	ICP-MS	NM		6.E-01	U	<6E-01	<3E+00
^{129}I	ICP-MS	NM		9.12E-01	J	9.12E-01	4.83E+00
^{129}I (d)	ICP-MS	NM		1.E+00	U	<1E+00	<5E+00
IDA(j)	Deriv/GC/FID	2.5E+03	X	NM		(a)	(a)
K	ICP-AES	1.98E+03	JX	1.10E+03	JX	<2E+02	<1E+03
La	ICP-AES	1.6E+01	J	3.2E+01	J	2.31E+01	1.22E+02
Li	ICP-MS	NM		1.81E+01	JX	1.81E+01	9.58E+01
Mg	ICP-AES	9.4E+00	U	1.1E+01	J	1.10E+00	5.82E+00
Mn	ICP-AES	1.7E+01	J	2.58E+02		2.48E+02	1.31E+03
Mo	ICP-MS	NM		3.30E+01		3.30E+01	1.75E+02
Na	ICP-AES	1.84E+05	X(h)	1.67E+05	X(h)	6.04E+04	3.20E+05
Nd	ICP-AES	*3.2E+01*	*J*	6.5E+01	J	4.63E+01	2.45E+02
NH_3	ISE	1.52E+02		8.90E+01		1.61E+00	8.52E+00
Ni	ICP-AES	4.16E+02		2.58E+02		1.75E+01	9.26E+01
NO_2	IC	8.54E+04		5.51E+04		5.82E+03	3.08E+04
NO_3	IC	2.21E+05		1.33E+05		5.02E+03	2.66E+04
^{237}Np	ICP-MS	NM		1.35E+00		1.35E+00	7.20E+00
NTA(j)	Deriv/GC/FID	1.90E+02	JX	NM		(a)	(a)
OH	Titration	4.30E+03	X	NM		(a)	(a)
Oxalate	Organic/IC	4.60E+02	X	2.88E+04	X	2.85E+04	1.51E+05
P	ICP-AES	1.82E+03		1.41E+03		3.58E+02	1.89E+03
Pb	ICP-AES	1.86E+02		2.45E+02	B	1.37E+02	7.25E+02
Pd	ICP-AES	*7.1E+01*	*U*	6.9E+01	U	<7E+01	<4E+02
PO_4	IC	5.0E+03	U	8.20E+03		8.18E+03	4.33E+04
Pr	ICP-MS	NM		1.76E+01		1.76E+01	9.31E+01
Pt	ICP-MS	NM		5.E-02	U	<5E-02	<3E-01
Pt (d)	ICP-MS	NM		8.E-02	U	<8E-02	<4E-01
Rb	ICP-MS	8.74E+00		4.70E+00		<2E-01	<1E+00
Rh	ICP-AES	*2.8E+01*	*U*	2.8E+01	U	<3E+01	<2E+02
Ru	ICP-MS	NM		1.52E+01	U	<2E+01	<8E+01
Sb	ICP-MS	NM		1.57E-01	J	1.57E-01	8.36E-01
Se	ICP-MS	NM		7.E+01	U	<7E+01	<4E+01
Si	ICP-AES	*2.35E+02*	*JBX*	2.00E+02	JB	6.44E+01	3.41E+02
SO_4	IC	1.69E+04		1.65E+04		6.73E+03	3.56E+04
Sr	ICP-AES	*2.3E+00*	*J*	6.2E+00	J	4.82E+00	2.55E+01
Succinic acid(j)	Deriv/GC/FID	3.6E+01	JX	NM		(a)	(a)
Ta	ICP-MS	NM		2.53E-01	JBX	2.53E-01	1.34E+00

Table S.2. (Cont'd)

		Supernatant		Wet Centrifuged Solids		Calculated UDS	
Analyte	Measure Method	Average µg/mL	Data Flag(g)	Average µg/g	Data Flag(g)	Average µg/g (wet)	Average µg/g (dry)
^{99}Tc	ICP-MS	NM		5.81E+00		5.81E+00	3.08E+01
Te	ICP-MS	NM		1.57E+00	J	1.57E+00	8.31E+00
Th	ICP-MS	NM		2.64E+01		2.64E+01	1.40E+02
Ti	ICP-AES	*2.00E+00*	*U*	2.3E+00	U	<2E+00	<1E+01
TIC-F (e)	TIC Furnace	2.30E+04		1.63E+04	X	3.00E+03	1.59E+04
TIC-P (e)	TIC Hot Pers.	1.10E+04		1.74E+04		1.10E+04	5.82E+04
Tl	ICP-MS	NM		2.E-02	U	<2E-02	<1E-01
TOC-F (e)	TOC Furnace	1.80E+04		1.96E+04	X	9.24E+03	4.89E+04
TOC-P (e)	TOC Hot Pers.	2.93E+04		2.56E+04		8.73E+03	4.62E+04
U	ICP-AES	1.90E+02	U	1.90E+02	U	<2E+02	<1E-01
U	KPA/ICP-MS(f)	1.19E+01		6.55E+01	X	5.86E+01	3.10E+02
^{233}U	ICP-MS	NM		5.75E-03	J	5.75E-03	3.04E-02
^{234}U	ICP-MS	NM		5.E-03	U	<5E-03	<3E-02
^{235}U	ICP-MS	NM		4.56E-01		4.56E-01	2.41E+00
^{236}U	ICP-MS	NM		1.90E-02	J	1.90E-02	1.01E-01
^{238}U	ICP-MS	NM		6.49E+01		6.49E+01	3.43E+02
V	ICP-MS	NM		6.61E+00	B	6.61E+00	3.50E+01
W	ICP-MS	NM		9.68E+01	X	9.68E+01	5.13E+02
Y	ICP-MS	NM		9.31E+00		9.31E+00	4.93E+01
Zn	ICP-AES	*6.50E+00*	*U*	2.2E+01	J	1.84E+01	9.74E+01
Zr	ICP-AES	*8.20E+00*	*J*	4.7E+01		4.18E+01	2.21E+02
Aroclor 1016/1242	GC/ECD	2.E-04	U	8.E-03	U	<8E-03	<4E-02
Aroclor 1221	GC/ECD	2.E-04	U	8.E-03	U	<8E-03	<4E-02
Aroclor 1232	GC/ECD	2.E-04	U	8.E-03	U	<8E-03	<4E-02
Aroclor 1248	GC/ECD	2.E-04	U	8.E-03	U	<8E-03	<4E-02
Aroclor 1254	GC/ECD	2.E-04	U	1.7E-02		1.70E-02	9.00E-02
Aroclor 1260	GC/ECD	2.E-04	U	3.0E-02		3.00E-02	1.59E-01
Total PCB	GC/ECD	1.E-03	U	7.9E-02		7.9E-02	4.18E-01

Table S.2. (Cont'd)

		Supernatant		Wet Centrifuged Solids		Calculated UDS	
Analyte	Measure Method	Average µg/mL	Data Flag[g]	Average µg/g	Data Flag[g]	Average µg/g (wet)	Average µg/g (dry)

WCS = wet centrifuged solids; UDS = undissolved solids; NM= 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; PCB= polychlorinated biphenyl

Italic results represent results for analytes not listed on the test specification (TS); opportunistic analytes.

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

(b) The chloride results are considered qualitative or at best the upper bound for the chloride. When corrected for the differences in dilution, the hot cell blank contribution to the reported results is about 15%.

(c) 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.

(d) ^{129}I and ^{127}I results from fusion preparation prescribed for I analysis; Pt results from fusion preparation prescribed for platinum group metals. All other results for I and Pt are from acid digestion preparations.

(e) For TOC and TIC: P=by hot persulfate method; F=by furnace method

(f) Uranium measured in the supernatant and centrifuged solids by KPA and ICP-MS, respectively

(g) 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 8.1)

(h) The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 4.3%, the solids RPD value obtained for Na was 7.8%. The serial dilution for this analyte was not measured.

(i) 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 GEA ^{137}Cs in the solids.

(j) Results are qualitative (See Section 9.12 and 9.13).

Table S.3. AN-102 As-Received Composite – Radioisotope Summary

		Supernatant		Wet Centrifuged Solids		Calculated UDS	
Analyte	Measure Method	Average µCi/mL	Data Flag (a)	Average µCi/g	Data Flag (a)	µCi/g (wet)	µCi/g (dry)
^{3}H (b)	Rad	NM		3.E-03	U	<3E-03	<2E-02
^{14}C	Rad	NM		6.32E-04	J	6.32E-04	3.4E-03
^{60}Co	Rad	8.49E-02		5.71E-02		8.16E-03	4.32E-02
^{90}Sr	Rad	5.72E+01		1.44E+02		1.11E+02	5.87E+02
^{99}Tc	ICP-MS	1.48E-01		9.88E-02		1.36E-02	7.20E-02
^{99}Tc	Rad	5.55E-02	X	NM		(c)	(c)
^{125}Sb	GEA	NM		2.E-01	U	<2E-01	<1E+00
^{126}SnSb	GEA	NM		8.E-02	U	<8E-02	<4E-01
^{129}I	ICP-MS	NM		1.62E-04	J	1.62E-04	8.57E-04
^{129}I (d)	ICP-MS	NM		2.E-04	U	<2E-04	<1E-03
^{134}Cs	GEA	NM		6.E-03	U	<6E-03	<3E-02
^{137}Cs	GEA	3.69E+02		2.16E+02		3.44E+00	1.82E+01
^{152}Eu	GEA	NM		1.E-02	U	<1E-02	<5E-02
^{154}Eu	GEA	2.31E-01		5.12E-01		5.12E-01	2.71E+00

Table S.3. (Cont'd)

Analyte	Measure Method	Supernatant Average µCi/mL	Supernatant Data Flag [(a)]	Wet Centrifuged Solids Average µCi/g	Wet Centrifuged Solids Data Flag [(a)]	Calculated UDS µCi/g (wet)	Calculated UDS µCi/g (dry)
^{155}Eu	GEA	1.00E-01	U	3.20E-01		3.20E-01	1.69E+00
^{233}U	ICP-MS	NM		5.60E-05	J	5.60E-05	2.96E-04
^{234}U	ICP-MS	NM		3.E-05	U	<3E-05	<2E-04
^{235}U	ICP-MS	NM		9.86E-07		9.86E-07	5.22E-06
^{236}U	ICP-MS	NM		1.23E-06	J	1.23E-06	6.51E-06
^{238}U	ICP-MS	NM		2.18E-05		2.18E-05	1.16E-04
^{237}Np	ICP-MS	1.20E-04		9.21E-04		8.51E-04	4.50E-03
^{238}Pu	Rad	1.65E-03	BJ	1.19E-02	BJ	1.10E-02	5.82E-02
^{239}Pu	ICP-MS	6.47E-03		5.56E-02		5.18E-02	2.74E-01
^{240}Pu	ICP-MS	2.01E-03	J	1.50E-02		1.39E-02	7.36E-02
$^{239/240}$Pu	Rad	5.90E-03		4.17E-02		3.82E-02	2.02E-01
^{241}Pu	Rad	NM		1.E-01	U	<1E-01	<5E-01
^{241}Am [(e)]	ICP-MS	1.58E-01		NM		[(c)]	[(c)]
^{241}Am	GEA	*1.65E-01*	*J*	*4.21E-01*	*J*	3.25E-01	1.72E+00
^{241}Am	Rad	1.51E-01		4.41E-01		3.54E-01	1.87E+00
^{242}Cm	Rad	6.29E-04	J	2.E-03	U	<2E-03	<1E-02
$^{243/244}$Cm	Rad	6.71E-03	B	1.72E-02	BJ	1.33E-02	7.04E-02
Alpha	Rad	1.66E-01		5.00E-01		4.04E-01	2.14E+00
Sum of Alpha	Rad	1.65E-01		5.12E-01		4.17E-01	2.21E+00

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

Italic results represent results for analytes not listed on the test specification (TS).

(a) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria; X = quality control (QC) deficiency (See Section 8.1)

(b) The tritium samples showed weak contamination with ^{137}Cs above the detection limit of <2E-04 µCi/g.

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

(d) ^{129}I result from fusion preparation prescribed for I analysis; other I result from acid digestion preparation.

(e) ^{241}Am calculated by applying the specific activities to the mass-241 response (using ^{241}Am for the calibration of the mass).

Decay correction reference dates extend from March 2001 to May 2001.

The AN-102 analytical results for the supernatant and UDS are compared to the WTP specifications in Table S.4 and S.5, respectively. The supernatant composite was compared to the Specification 7 Low-Activity Waste Envelope C Definition; the UDS component was compared to Specification 8, High-Level Waste Definition. The Specification 7 limits were exceeded for sulfate and ^{60}Co concentrations, although ^{60}Co is expected to decay to below the specification limit by the time of actual waste processing. The Specification 8 limits were exceeded for Cr, Na, and S concentrations, with Na and S exceeding the limits by approximately 40% and 50%, respectively.

Table S.4. AN-102 As-Received Supernatant – Specification 7 Envelope C Summary

Analytes Measured above MDL		Analytes not Detected above MDL	
Results % of Limit	**Analyte**	**% of Limit based on MDL**	**Analyte**
Table TS 7.1 Analytes			
110	SO_4	(<) 18	F
80	TIC-F(b)	(<) 17	PO_4
61	NO_2	(<) 8	U
60	TOC-P(b)	(<) 5 or less(a)	Ba, Hg
56	NO_3		
46	Cl		
38	TOC-F(b)		
37	TIC-P(b)		
30	Ni		
23	Al		
19	PO_4 (as P)(c)		
17	La		
16	Pb		
8	Cr		
5 or less(a)	Ca, K, Cd, Fe		
Table TS 7.2 Analyte			
106	^{60}Co		
40	^{137}Cs		
33	^{90}Sr		
25	TRU(d)		
25	^{154}Eu		
10	^{99}Tc		

Bolded and Outlined Values exceed Specification 7 Envelope C criteria.

(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) Phosphate based on ICP-AES average total P result.
(d) 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.

Table S. 5. AN-102 As-Received UDS – Specification 8 Summary

Analytes Measured above MDL		**Analytes not Detected above MDL**	
Results % of Limit	**Analyte**	**% of Limit based on MDL**	**Analyte**
Table TS 8.1 Analytes			
17	W	(<) 10 or less (a)	Se, Rb, Tl
10 or less (a)	V, Li, Pr, Y, Zn, As, Mn, B, Mo, Ce, Nd, Be, Te, Pu, Cu, La, Sr, Ta, Co, Cs, Hg, Sb		
Table TS 8.2 Analytes			
79	TIC-P as CO_3 (b)		
73	Cl		
36	TOC-F (b)		
34	TOC-P (b)		
21	TIC-F as CO_3 (b)		
13	$NO_3 + NO_2$		
10 or less (a)	Cyanide, Ammonia		
Table TS 8.3 Analytes			
24	^{129}I	(<) 20	^{126}Sn-Sb
10 or less (a)	^{14}C, ^{233}U, ^{238}Pu, ^{239}Pu, ^{90}Sr, ^{155}Eu, ^{237}Np, ^{154}Eu, ^{235}U, ^{241}Am, $^{234/244}$Cm, ^{137}Cs, ^{60}Co, ^{99}Tc	(<) 10 or less (a)	^{3}H (c), ^{152}Eu, ^{125}Sb, ^{241}Pu
Table TS 8.4 Analytes			
148	SO_4 as S(d)	(<) 23	Pd
136	Na	(<) 10 or less (a)	K, Ag, Bi, Ti
107	Cr		
65	Al		
47	F (e)		
10 or less (a)	P, Pd(f), Pb, Ru, Fe, Rh(f), Ca, Ni, Ba, Mg, Si, Th, U, Zr, Cd		

Bolded and Outlined Values exceed Specification 8 criteria.

(a) Analytes are presented from 10% in descending order.
(b) For TOC and TIC: P=by hot persulfate method; F=by furnace method/TIC by difference (TIC = TC-TOC).
(c) Tritium results possibly biased high by contamination from ^{137}Cs.
(d) Sulfur not measured. Sulfate from ion chromatography analysis converted to sulfur and compared to the specification.
(e) Fluoride results may be biased high due to interferences from co-eluting organic anions.
(f) Results are from fusion preparation; other results from acid digestion preparation.

Terms and Abbreviations

AEA	Alpha Energy Analysis
AMU	atomic mass unit
ASR	Analytical Service Request
BNI	Bechtel National Inc.
BS	blank spike
CN	cyanide
COC	chain of custody
CS	centrifuged solids
CVAA	cold vapor atomic absorption
D2EHP	bis-(2-ethylhexyl) phosphate
DCB	decachlorobiphenyl
DI	deionized water
DPP	diphenylphosphate
DS	dry solids
EDTA	ethylenediaminetetraacetic acid
ED3A	ethylenediaminetriacetic acid
EHT	extended homogenized test
EQL	estimated quantitation limit
GC/ECD	gas chromatography/electron capture detector
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatography/mass spectrometry
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
HPIC	high-performance ion chromatography
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
IDA	iminodiacetic acid
IDL	instrument detection limit
ISE	ion specific electrode
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
NA	not applicable
NIST	National Institute of Standards and Technology
NM	not measured
NR	not recovered
NTA	nitrilotriacetic acid
%D	percent difference
PB	process blank
PCB	polychlorinated biphenyl
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
SRM	Standard Reference Material
TC	total carbon
TCX	tetrachloroxylene
TDS	total dissolved solids
TI	test instruction
TIC	total inorganic carbon
TOC	total organic carbon
TP	test plan
TRU	transuranic
TS	test specification
UDS	undissolved solids
WCS	wet centrifuged solids
WTP	Waste Treatment Plant
XRD	X-ray diffraction

Unit Abbreviations

°C	degrees Centigrade
Bq	Becquerel
g	gram
G	gravitational force
h	hour
in	inch
L	liter
μCi	microcurie
μg	microgram
M	molarity
mL	milliliter
mm	millimeter
mmole	millimole
N	normality
nCi	nanocurie
pCi	picocurie
Vol%	volume percent
Wt%	weight percent

Definitions

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

Centrifuged Solids – the solids layer that separates from the bulk slurry after centrifugation (typically for 1 hour at 1000 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.

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1.0 Introduction

Battelle – Pacific Northwest Division is conducting physical property testing and inorganic, radiochemical, and selected organic waste characterization of waste from underground storage tank number 241-AN-102 (AN-102) for Bechtel National, Inc. (BNI). This effort supports the operation of the River Protection Project-Waste Treatment Plant (RPP-WTP). Tank wastes must be compared to analyte and radioisotopic compositions described in envelope ranges Specification 7 and Specification 8 (Appendix A, Test Specification TSP-W375-00-00007). It is important to characterize the compositions of tank wastes as specified in the envelope to define pretreatment and separations processes before final stabilization (vitrification) of the waste.

The RPP-WTP design flowsheets intend to use filtration and process the liquids and solids separately. Therefore, it is important to fully characterize both the liquids and solids in the tank waste before proceeding with final process design.

AN-102 wastes have been previously characterized. In 1998, the Savannah River Technology Center analyzed supernatant and solid phases from 16 L of AN-102 sample retrieved at the Hanford Site (Hay 2000). The Westinghouse Hanford Company also analyzed AN-102 solids and the incidental supernatant (Lambert 1996). The current characterization activity is to provide information for assessing Tank AN-102 waste for compliance with the low-activity waste (LAW) and high-level waste (HLW) feed specifications and to provide additional information for process testing.

Approximately 4 L of AN-102 waste that was retrieved from the tank in August 2000 was used as starting material for this characterization task. Test Specification TSP-W375-00-00007, *Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate*,[1] hereafter referred to as TS, defined the work scope. Test plan TP-41500-005, *AN-102 Sample Compositing for Process Testing,* was prepared by the PNWD to conduct the work scope defined in the TS. The four objectives of this work are to:

- ensure that sample material used for analysis is homogenized
- separate homogenized composite 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 to the WTP specifications. For liquids, the analytical results are compared to Specification 7. For solids, the analytical results for the "insoluble solids" are compared to Specification 8. For the purposes of comparison to the specification, the TS identifies "insoluble solids" as the total dried solids remaining after phase separating centrifuged solid minus the contribution to the dried solids from the interstitial liquid. For this report, "insoluble solids" are assumed to be the "undissolved solids," although some of the solids may be the result of salt precipitates from the saturated supernatant.

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[1] TSP-W375-00-00007, Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate, Rev. 0, November 1, 2000, CH2MHill Hanford Group, Richland Washington.

The PNWD Quality Assurance Program Plan “Conducting Analytical Work in Support of Regulatory Program” was used in support of all analytical operations and is compliant with the Hanford Analytical Service Quality Assurance Requirements Documents (HASQARD) DOE/RL-96-68. The inorganic, radioisotopic, and organic analytes tested were identified in the TS. The quality requirements for both the liquid and solid fractions were included in the TS and transmitted to the laboratory staff via the Analytical Service Request (ASR) 6019.01.

Data and results limitations are described. Initial sample processing, QC sample results, method detection limits (MDL), and other QC indicators are described in the Section 9, Procedures, Quality Control, and Data Evaluation.

2.0 Sample Receiving

Thirty samples were obtained from Hanford waste tank 241-AN-102 (AN-102) in August 2000 from Riser 22 at six different tank waste heights. These samples were shipped to the 222-S Laboratory in Hanford's 200 West Area and were stored. Twenty-seven samples were received by the PNWD from the 222-S laboratory in November 2000, but only eight samples were used for characterization and process testing. The additional 19 samples received in this shipment, some containing slurry and other supernatant only, were not used in support of this report. The eight samples were obtained from the sampling location 190 cm (76 in.) measured from the bottom of the tank at the sludge/supernatant interface and contained a large (nominally 50% by volume) solids/sludge fraction. The samples were not necessarily representative of the complete tank contents.

As described in the memorandum "Shipment of AN-102 to Pacific Northwest National Laboratory" (Appendix A), the net weight of each of the samples retrieved from the tank was between 600 and 700 g. Each sample was contained in 500-mL bottles and consisted of wet solids plus supernatant. Upon receipt at the High Level Radiation Facility (HLRF), the AN-102 waste samples were visually inspected, and the inspection was documented through Test Instruction 41500-009, "AN-102 Sample Inspection," contained in Appendix B. All bottles and lids were in good condition. All samples were similar in appearance, containing a large amount of light brown settled solids with a dark brownish/black liquid. An organic layer could not be discerned because of the dark color of the supernatant. No specific crystalline phases were apparent. The results of the inspection are provided in Table 2.1.

Table 2.1. Received AN-102 Waste Samples

Bottle Label	222-S ID	Net Mass Recovered, g	Visual Appearance Settled Solids	Approximate Solids Volume, mL	Visual Appearance Supernatant	Approximate Supernatant Volume, mL
2AN-00-21	S00T001598	671	Light Brown Solids	100	Dark Brown to Black	400
2AN-00-24	S00T001602	669	Light Brown Solids	275	Dark Brown to Black	225
2AN-00-25	S00T001664	658 (a)	Light Brown Solids	100	Dark Brown to Black	400
2AN-00-26	S00T001665	687	Light Brown Solids	300	Dark Brown to Black	200
2AN-00-27	S00T001666	669	Light Brown Solids	200	Dark Brown to Black	300
2AN-00-28	S00T001667	673	Light Brown Solids	200	Dark Brown to Black	300
2AN-00-29	S00T001662	670	Light Brown Solids	300	Dark Brown to Black	200
2AN-00-30	S00T001663	690	Light Brown Solids	250	Dark Brown to Black	250
Total mass received		5388				

No crystalline phases were present in any of the samples received.

(a) 137 g of supernatant were removed from this sample, leaving 521 g. This supernatant was used for Sr/TRU (transuranic) removal testing.

3.0 Compositing and Sub-Sampling

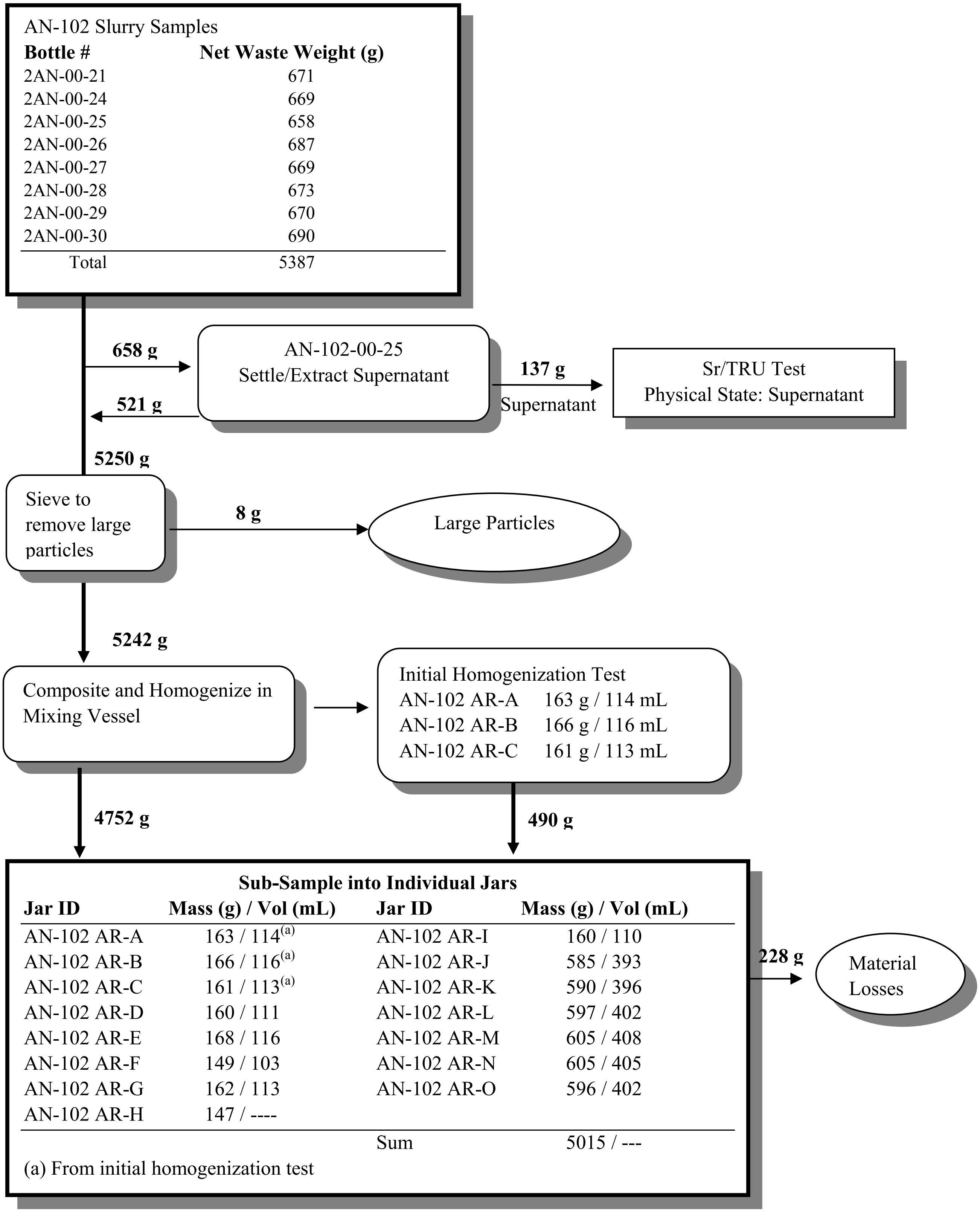
The objective of compositing the AN-102 samples is to provide homogeneous feed to tasks within the project. The homogenized feed is used to support the characterization task as well as process testing. The homogenization and sub-sampling activity was performed according to the Test Plan TP-41500-005, "AN-102 Sample Compositing for Process Testing," contained in Appendix B. The overall sample processing is summarized in Figure 3.1.

Before compositing, 137 g of supernatant were removed from bottle number 2AN-00-25 to conduct Sr/TRU process testing (separate report). Then all materials in each of the eight bottles were passed through a 2.38 mm (3/32 in.) sieve to remove large particles. The transferred material was collected in an 8-L stainless steel mixing vessel. Clumps of agglomerated material were retained on the sieve. Most of this material was pushed through the sieve with a glass stopper. A total of 7.8 g (mass after >8 h drying time) of large particles were trapped on the screen. These large particles (See Figure 3.2) could not be crushed with a glass stopper to pass through the screen. Characteristics of these particles were not inconsistent with gravel; however, definitive identification could not be made. The particles were later discarded.

The AN-102 waste was homogenized in the mixing vessel equipped with a dual-bladed impeller. Material was stirred with the impeller for about 70 min at a temperature of 31.5°C (the ambient hot-cell temperature) to thoroughly homogenize the material. With the impeller running, a 100-mL portion was removed through the ¾ in. 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. Three 100-mL samples were collected from the mixing vessel for homogenization evaluation into volume-graduated glass jars, AN-102 AR-A through AN-102 AR-C ("AR" indicates as-received). The volume percent (vol%) settled solids was determined on each and found to be consistent within 1%. The remaining contents of the mixing vessel were stirred and collected into 12 additional volume-graduated glass jars labeled AN-102 AR-D through AN-102 AR-O. Sample AN-102 AR-H was used immediately for physical-property testing. The remaining 14 jars were left undisturbed for 7 days and did not show any sign of additional precipitation or organic layer formation following the 7-day hold time.

The total volume of material in each of the 14 jars and the volume of settled solids were recorded after the 7-day settling period. These data were used to calculate the volume percent (vol%) settled solids. Table 3.1 lists the calculated mass, volume, and vol% settled solids for each sub-sample. According to the TS, the absolute standard deviation of the vol% settled solids of the collection of 14 sub-sample jars must be less than 5%. Constant vol% settled solids is one measure of homogeneity. The average vol% settled solids of as-received homogenized sub-samples in the jars was 70% with a standard deviation of ±2.5%. As shown in Table 3.1, the sub-samples collected in the jars from the compositing vessel met the TS criteria for homogeneity.

Sub-samples AN-102 AR-A, AR-B, AR-C, AR-D, AR-F, AR-G, and AR-I were selected for inorganic, radiochemical, and organic characterization analysis and were transferred from the HLRF to the Shielded Analytical Laboratory (SAL) for processing and analysis (See Section 4 and 5).



AN-102 Slurry Samples

Bottle #	Net Waste Weight (g)
2AN-00-21	671
2AN-00-24	669
2AN-00-25	658
2AN-00-26	687
2AN-00-27	669
2AN-00-28	673
2AN-00-29	670
2AN-00-30	690
Total	5387

Initial Homogenization Test

AN-102 AR-A	163 g / 114 mL
AN-102 AR-B	166 g / 116 mL
AN-102 AR-C	161 g / 113 mL

Sub-Sample into Individual Jars

Jar ID	Mass (g) / Vol (mL)	Jar ID	Mass (g) / Vol (mL)
AN-102 AR-A	163 / 114[(a)]	AN-102 AR-I	160 / 110
AN-102 AR-B	166 / 116[(a)]	AN-102 AR-J	585 / 393
AN-102 AR-C	161 / 113[(a)]	AN-102 AR-K	590 / 396
AN-102 AR-D	160 / 111	AN-102 AR-L	597 / 402
AN-102 AR-E	168 / 116	AN-102 AR-M	605 / 408
AN-102 AR-F	149 / 103	AN-102 AR-N	605 / 405
AN-102 AR-G	162 / 113	AN-102 AR-O	596 / 402
AN-102 AR-H	147 / ----		
		Sum	5015 / ---

(a) From initial homogenization test

Figure 3.1. Flow Diagram of the AN-102 Tank Slurry Receiving, Homogenizing, and Sub-Sampling

Figure 3.2. Residual Large Particles/Agglomerates Not Forced Through Sieve (Approximately 8 g of a Total of 5251 g Processed)

Table 3.1. AN-102 As-Received Composite Sub-Samples Collected From Homogenization Vessel

Jar ID	Sample Mass, g	Sample Volume, mL	Vol% Settled Solids, Visual
AN-102 AR-A	163	114	73
AN-102 AR-B	166	116	72
AN-102 AR-C	161	113	74
AN-102 AR-D	160	111	70
AN-102 AR-E	168	116	71
AN-102 AR-F	149	103	73
AN-102 AR-G	162	113	71
AN-102 AR-H	147	NA	NA
AN-102 AR-I	160	110	72
AN-102 AR-J	585	393	68
AN-102 AR-K	590	396	70
AN-102 AR-L	597	402	68
AN-102 AR-M	605	408	68
AN-102 AR-N	605	405	67
AN-102 AR-O	596	402	65
		Average	70
		Standard Deviation (1σ)	3
NA- Not available, sample H was immediately removed for physical testing, so data on settled volumes were not collected.			

4.0 Physical Measurement

The composite material from Jar AN-102 AR-H was selected for further physical-properties measurements. These include density (slurry, supernatant, settled solids, and wet centrifuged solids), vol% solids (settled and centrifuged), and weight percent (wt%) solids (dried supernatant, settled solids, centrifuged solids, and dried centrifuged solids). Testing was conducted in accordance with Test Instruction TI-41500-015, "AN-102 Integrated Test: Solids Analysis," contained in Appendix B. Additionally, samples were taken for determination of fired oxide content and crystal structure.

4.1 Density and Weight Percent Solids

Four 9.4 mL (~14 g) aliquots (labeled A, B, C, and D) were removed from the stirred contents of sub-sample AN-102-AR-H and used for density and percent solids characterization. The aliquots taken were placed into 10-mL volume-graduated centrifuge cones, and the bulk slurry densities were determined from the total mass (m_t) and the total volume. The solids in samples A and B were allowed to settle for 6 days, and the solids volume was recorded. A fraction of the supernatant was removed into another volume-graduated centrifuge cone, and the supernatant density was calculated. From the total supernatant volume and corresponding density, the supernatant mass was calculated. The supernatant fractions were returned to the corresponding solids sample. The settled solids mass (m_{ss}) was calculated by the difference of the gross mass and the sum of the tare and calculated supernatant mass. The wt% settled solids (wt $\%_{ss}$) was calculated according to Equation 4.1:

$$wt\,\%_{ss} = \frac{m_{ss}}{m_t} * 100 \tag{4.1}$$

All four aliquots were centrifuged at approximately 1000 times the force of gravity (G) for 1 h. All of the centrifuged supernatant from each of the samples was then transferred to separate graduated cylinders, and the masses and volumes were recorded for both the supernatant and the solids fractions. The centrifuged solids densities and wt% solids were similarly calculated as those for the settled solids.

The centrifuged solids and supernatants were then dried at 105°C to constant mass. The solids drying time lasted about 3 weeks. The mass of the centrifuged solids (m_{cs}) and the mass of the dried centrifuged solids (m_{dcs}) were recorded, as were the mass of the centrifuged supernatant and dried centrifuged supernatant. The wt% solids in the centrifuged solids were determined according to Equation 4.2:

$$wt\%_{dcs} = \frac{m_{dcs}}{m_{cs}} * 100 \tag{4.2}$$

The wt% solids in the supernatant were similarly determined. Percent solids of the slurry, supernatant, and centrifuged solids, and density of the solids and supernatant phases are reported in Table 4.1. The relative standard deviation was generally less than 4%.

Table 4.1. 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	1.49	1.50	1.46	1.49	1.49	0.02	1%
Supernatant	1.40	1.43	1.43	1.42	1.42	0.02	1%
Settled Solids	1.48	1.49	NM	NM	1.49	NA	NA
Wet Centrifuged Solids	1.55	1.57	1.52	1.62	1.56	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	NM	NM	70.8	NA	NA
Centrifuged Slurry	47.3	48.6	45.3	43.6	46.2	2	5%
Weight Percent Solids (%)							
Wt% Solids in Supernatant After Drying at 105 °C	50.6	50.2	50.3	50.3	50.4	0.2	0.4%
Solids Wt% in Centrifuged Solids After Drying at 105 °C	63.7	64.5	60.3	63.8	63.1	2	3%

RSD = relative standard deviation; NM = not measured; NA = not applicable

4.2 Weight Percent Fired Oxide

Following the completion of the density and vol% and wt% solids, it was determined that in order to compare the analyte concentration of the undissolved solids fraction in the centrifuged solids to Specification 8, the wt% fired oxide of both the supernatant and centrifuged solids was required. Additional samples from AN-102 AR-H were processed to obtain these results according to test instruction TI-RPP-WTP-098, "AN-102 Weight Percent Oxides" found in Appendix B. The samples were centrifuged and dried as previously described. The dried salts were then fired at 1000 to 1050°C for 1 hour. From the mass of centrifuged solids (m_{cs}) and mass of fired oxide from the centrifuged solids (m_{sfo}), the centrifuged solids wt% fired oxide (wt%$_{\text{sfo}}$) was determined according to Equation 4.3:

$$wt\%_{sfo} = \frac{m_{sfo}}{m_{cs}} * 100 \qquad (4.3)$$

The wt% fired oxide in the supernatant was similarly determined. The results are presented in Table 4.2.

The densities of the slurry, settled solids, and centrifuged solids are virtually identical to the initial results. As can be seen from the wt% solids from the supernatant and centrifuged solids, there is a slight difference in the result obtained from the initial testing. However, this difference is considered insignificant, since it has only a minor effect on the concentration of the undissolved solids on a per 100-g oxide basis. The data from Table 4.2 are used in this report to calculate the analyte concentrations in the undissolved solids on both a per g slurry basis and a per g dried solids basis and to calculate the analyte concentration on a per 100 g of oxide basis.

4.3 Crystalline Phases

The only physical-property measurement specified by the TS to be performed on the as-received AN-102 composite is mineral type/structure by x-ray diffraction (XRD) on the centrifuged solids. All other physical measurements specified in the TS (i.e., particle size, viscosity versus shear rate, and heat capacity) are performed on the AN-102 composite following adjustment of the as-received composite to 2 wt% undissolved solids. These physical measurements are reported in the characterization report for the 241-AN-102 (2% undissolved solids) blended with 241-C-104 wash/leachate solutions, (Urie, 2002).

An aliquot of the wet centrifuged solids from AN-102 AR-H was examined using procedure PNNL-RPG-268, Solids Analysis; X-ray Diffraction, for identification of crystalline phases present (See Appendix B, *XRD Analysis of Sample AN-102 AR-H*). The sample was prepared as a thin sample on a glass slide using collodian as a binder. Corundum, Al_2O_3, was added as an internal standard for line positioning, and a long count time (20 sec per step) was used in an attempt to obtain better counting statistics. The only crystalline phases identified were sodium nitrate ($NaNO_3$) and sodium nitrite ($NaNO_2$). The raw data exhibited a significant amorphous background, indicating the presence of non-crystalline material that cannot be identified by XRD.

Table 4.2. AN-102 Homogenized Slurry Composite Sample—Percent Oxide Measurements

	AN-102-AR-H A-2	**AN-102-AR-H B-2**	**Average**	**RPD**
Density (g/mL)				
Slurry	1.474	1.454	1.464	1%
Supernatant	1.410	1.403	1.406	0.5%
Wet Centrifuged Solids	1.546	1.510	1.528	2%
Weight Percent Solids (%)				
Wt% Solids in Supernatant After Drying at 105 °C	49.1	49.2	49.2	0.2%
Wt% Solids in Centrifuged Solids After Drying at 105 °C	58.9	58.7	58.8	0.3%
Weight Percent Oxide (%)				
Wt% Oxide in Supernatant After Firing at 1050 °C	24.2	25.1	24.7	4%
Wt% Oxide in Centrifuged Solids After Firing at 1050 °C	43.5	43.3	43.4	0.5%
RPD = Relative Percent Difference				

5.0 Extended Homogenization Test

5.1 Objective

The initial WTP proposal[2] identified an extended homogenization test (EHT) to be completed on the first three waste tanks that contained significant quantities of undissolved solids. The AN-102 as-received composite was the first of these three tanks; however, due to changes in the contract, additional EHTs have been removed from the future scope of work. The objective of the EHT is to analyze sub-samples of the composite such that an evaluation can be made as to the reproducibility of the sub-sampling activities. There are two sub-sampling activities; i.e., sub-sampling of the composite from the compositing/homogenizing vessel and extracting analytical aliquots from these composite sub-samples.

It is important to assess the concentration variations in the composite sub-samples as well as the variations in the concentration of analytical aliquots. Concentration variations identified in the composite sub-sample may suggest that the method for collecting the composite does not ensure representative sub-sampling. Concentration variations identified between the analytical aliquots taken from the same bottle may indicate the need for an alternate method of aliquoting for analytical processing or distribution.

5.2 Sample Preparation

To assess the reproducibility of the composite sub-sampling activity and the analytical sub-sampling activity, four 125-mL bottles obtained during the sub-sampling of the composite (i.e., the emptying of the composite vessel) were selected for evaluation. These four bottles (AR-A, AR-B, AR-C, and AR-I) include the first (AR-A) and last (AR-I) 125-mL bottle collected for analytical work from the compositing vessel. Additional 250-mL bottles (beyond sample AR-I) were collected from the compositing vessel but were not included in the EHT. The four 125-mL bottles were transferred to the SAL for analytical sub-sampling and processing to assess the reproducibility of the analytical aliquoting process. The sub-sampling and analytical requirements were documented in ASR 6011 and 6011.01; the samples were assigned Radiochemical Processing Laboratory (RPL) identifications 01-386, -387, -388, and -420. To assess the analytical aliquoting process, seven 0.5-mL slurry samples were extracted from each of the bottles (AR-A, AR-B, AR-C, and AR-I) containing the AN-102 slurry composite. The sub-samples were taken with a pipet while the slurry was mixed with an impeller stirrer. The samples were acid digested per PNL-ALO-129, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*, and then analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) for metals and gamma energy analysis (GEA) for gamma-emitting radioisotopes. The AN-102 as-received slurry samples appeared to be fully digested by this procedure (i.e., no solids residue remained following digestion).

Figure 5.1 provides a flowchart of the sub-sampling, digestion, and analysis activities for the EHT analytical aliquots processed for ICP-AES and GEA. The remainder of the slurry material in bottles AR-A, AR-B, AR-C, and AR-I were processed for additional analytical activities described in Section 6 and Section 7.

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[2] CH2M Hill Hanford Group Part B2 Development Workscope, September 2000, Pacific Northwest National Laboratory, Richland, Washington.

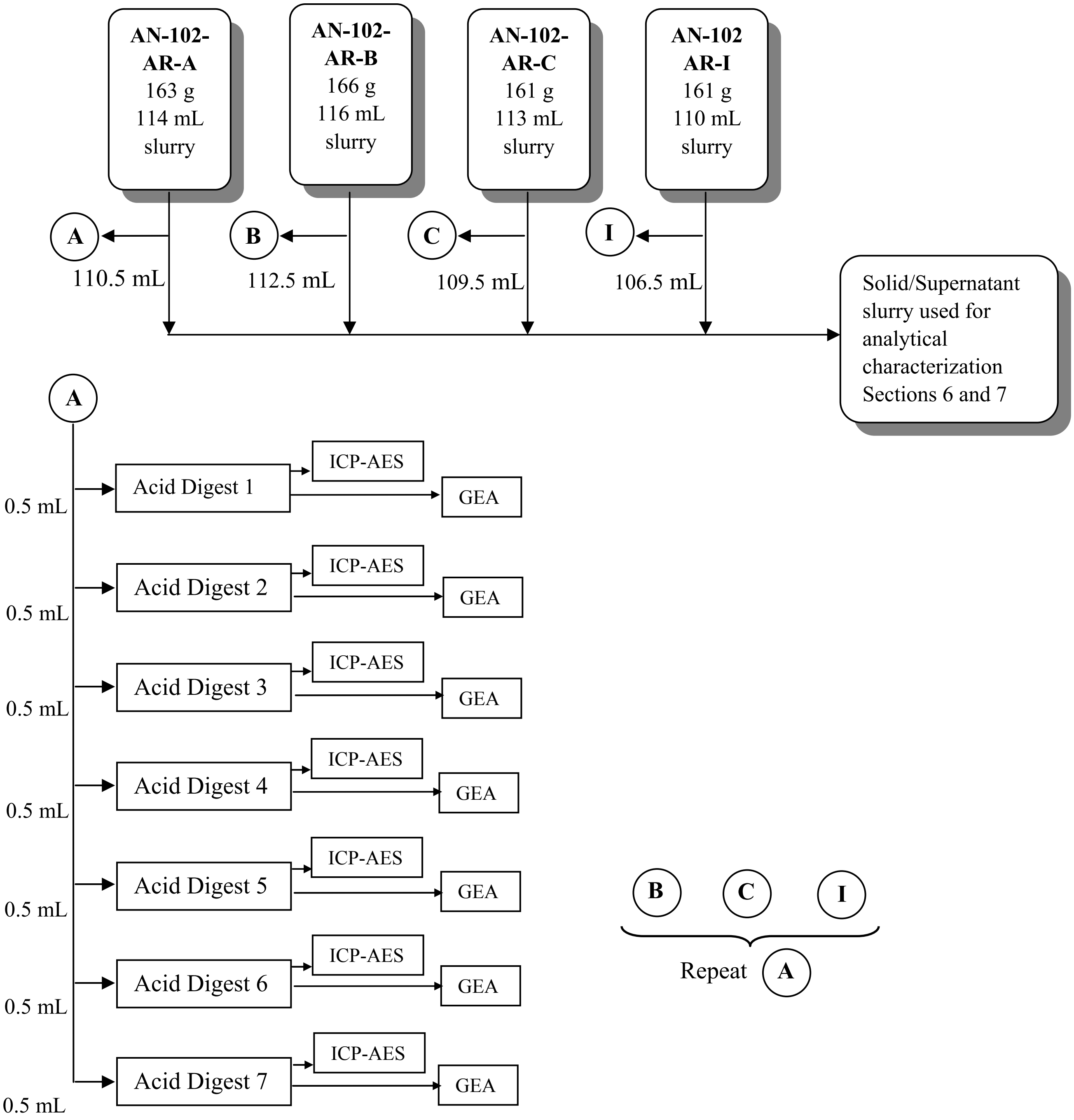

Figure 5.1. Flowchart of EHT Analytical Sub-Sampling and Analysis

5.3 ICP-AES and GEA Composite Slurry Results

The results of the seven ICP-AES analyses and GEA from each of the four composite sub-samples are reported in Table 5.1 through Table 5.4. The first column of data in each of the four tables provides the results for the process blanks analyzed concurrently with the samples. In each of the cases, no analytes were detected in the process blanks, except for sodium and boron. Also, before performing the ICP-AES and GEA measurements, the digested samples were randomized; i.e., the seven samples from each composite sub-sample were not analyzed in sample number order.

Appendix C provides additional information on the ICP-AES analysis using PNL-ALO-211, *Determination of Elements by Inductively Coupled Argon Plasma-Atomic Emission Spectrometry*, and on the GEA analysis using PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Spectrometry*.

Besides the analyte concentration measured from each of the seven analytical aliquots (#1 through #7), the average concentration for each of the 17 analytes measured by ICP-AES and ^{137}Cs measured by GEA is calculated and presented in Table 5.1 through Table 5.4. Eleven of the 18 measured analytes were measured at less than the estimated quantitation limit (EQL), and the precision at these concentration levels is typically greater than 15% relative standard deviation (RSD). However, except in a few cases, these analytes appeared to reproduce as well as those analytes above the EQL. For those analytes above the EQL, the RSD for each analyte from the seven replicates is less than 5%, except for bottle AR-C. Two aliquots (#3 and #5) produced significantly different concentrations for all analytes than were obtained for the other five aliquots in AR-C (or in the other 21 aliquots measured from AR-A, AR-B, and AR-I). In evaluating the raw data, by switching the sample weights for #3 and #5, the results track the other aliquot results (i.e., the RSD from the seven replicates for those analytes above the EQL is less than 5%). Although it appears, based on the results, that the Aliquots #3 and #5 were switched in the SAL during the acid-digestion preparation, the results in Table 5.3 (AR-C) are reported using the sample weights recorded on the preparation benchsheets. Due to aliquot #3 and #5, the RSDs for the analyte in AR-C are at the 12% level; however, the averages are basically identical to those obtained from AR-A, AR-B, and AR-I for all analytes. Based on the good reproducibility of the seven aliquots taken from individual bottles, the method of sub-sampling for analytical processing is considered acceptable.

The average concentrations measured for each of the bottles (AR-A, AR-B, AR-C, and AR-I) are used to evaluate the sub-sampling of the AN-102 as-received material from the compositing vessel. Table 5.5 presents these averages and the resulting average (average of the averages), standard deviation, and RSD from the four bottles. For those analytes measured above the EQL, the RSD is 1.1% or less (and less than 7% for all detected analytes), indicating excellent sub-sampling of the composite. That is, mixing by impeller agitation while sub-sampling from a port at the bottom of the compositing vessel ensures reproducible sub-samples for tank material with physical characteristics similar to AN-102 tank material.

Table 5.1. Extended Homogeneity Test Results - AN-102-AR-A (RPL# 01-386)

Analyte	PB	#1	#2	#3	#4	#5	#6	#7	Average	Std Dev	RSD
	µg/mL										%
Al	n.d.	27,900	29,900	27,700	28,700	27,000	27,700	27,800	28,100	936	3
B	50 J	94 J	91 J	98 J	120 J	92 J	98 J	110 J	100 J	11	11
Ba	n.d.	22 J	21 J	22 J	22 J	21 J	22 J	22 J	22 J	0.5	2
Ca	n.d.	540 J	520 J	550 J	560 J	530 J	570 J	580 J	550 J	22	4
Cd	n.d.	59	61	59	60 J	58	59	59 J	59 J	1.1	2
Cr	n.d.	1,490	1,560	1,490	1,530	1,460	1,480	1,450	1,490	39	3
Cu	n.d.	28 J	25 J	29 J	28 J	29 J	29 J	29	28 J	1.5	5
Fe	n.d.	895	917	894	927	870	901	896	900	18	2
K	n.d.	2,200 J	1,600 J	2,400 J	1,900 J	2,500 J	2,300 J	2,200 J	2,160 J	310	14
La	n.d.	34 J	28 J	35 J	32 J	35 J	35 J	35 J	33 J	2.6	8
Mn	n.d.	206	218	207	210 J	202	208	210 J	209 J	4.9	2
Mo	n.d.	51 J	50 J	50 J	51 J	50 J	51 J	52 J	51 J	0.8	1
Na	98 J	225,000	228,000	220,000	234,000	214,000	221,000	226,000	224,000	6,400	3
Ni	n.d.	401	418	395	403	390	400	405	402	8.8	2
P	n.d.	1,950	2,040	1,930	1,960	1,890	1,940	1,970	1,950	46	2
Pb	n.d.	280 J	260 J	280 J	280 J	280 J	290 J	290 J	280 J	10	4
Zr	n.d.	40 J	38 J	40 J	39 J	39 J	40 J	40 J	39 J	0.8	2
	µCi/mL										%
^{137}Cs	0.02	153	155	156	155	155	156	155	155	0.9	1

Notes:
n.d. = not detected
PB = process blank
J = Results are less than 10 times the MDL, and uncertainty is estimated at greater than 15 percent.
^{137}Cs decay correction reference date is January 2001.

Table 5.2. Extended Homogeneity Test Results - AN-102-AR-B (RPL# 01-387)

Analyte	PB	#1	#2	#3	#4	#5	#6	#7	Average	Std Dev	RSD
	µg/mL										%
Al	n.d.	27,900	29,000	27,500	30,100	28,900	27,900	27,800	28,400	927	3
B	43 J	84 J	96 J	99 J	130 J	110 J	84 J	140 J	106 J	22	21
Ba	n.d.	22 J	21 J	22 J	22 J	23 J	22 J	22 J	22 J	0.6	3
Ca	n.d.	550 J	470 J	550 J	530 J	580 J	560 J	610 J	550 J	44	8
Cd	n.d.	60	59	60 J	63 J	62 J	60 J	60 J	60 J	1.5	3
Cr	n.d.	1,490	1,490	1,450	1,550	1,520	1,460	1,450	1,490	38	3
Cu	n.d.	28 J	24 J	29 J	24 J	29 J	19 J	29 J	26 J	3.8	15
Fe	n.d.	893	883	896	1,010	925	890	893	913	45	5
K	n.d.	2,100 J	1,500 J	2,500 J	1,300 J	1,900 J	2,200 J	2,200 J	1,960 J	424	22
La	n.d.	34 J	27 J	36 J	26 J	34 J	34 J	35 J	32 J	4.0	12
Mn	n.d.	207	214	210 J	220 J	215 J	210 J	210 J	212 J	4.3	2
Mo	n.d.	51 J	47 J	50 J	50 J	52 J	46 J	51 J	50 J	2.2	4
Na	82 J	224,000	219,000	219,000	230,000	236,000	225,000	230,000	226,000	6,260	3
Ni	n.d.	401	403	397	424	413	400	400	405	9.6	2
P	n.d.	1,970	1,930	1,930	2,060	2,010	1,910	1,930	1,960	54	3
Pb	n.d.	280 J	250 J	290 J	260 J	280 J	280 J	280 J	274 J	14	5
Zr	n.d.	39 J	37 J	40 J	37 J	40 J	39 J	39 J	39 J	1.3	3
	µCi/mL										%
^{137}Cs	0.04	156	154	158	157	157	155	155	156	1.5	1

Notes:

n.d. = not detected

PB = process blank

J = Results are less than 10 times the MDL, and uncertainty is estimated at greater than 15 percent.

^{137}Cs decay correction reference date is January 2001.

Table 5.3. Extended Homogeneity Test Results - AN-102-AR-C (RPL# 01-388)

Analyte	PB	#1	#2	#3	#4	#5	#6	#7	Average	Std Dev	RSD
	µg/mL										%
Al	n.d.	27,900	29,700	35,600	29,100	24,900	27,200	27,600	28,900	3,350	12
B	59 J	83 J	84 J	130 J	110 J	97 J	100 J	85 J	98 J	17	17
Ba	n.d.	22 J	22 J	28 J	23 J	20 J	21 J	20 J	22 J	2.8	12
Ca	n.d.	540 J	52 J	710 J	600 J	490 J	490 J	510 J	485 J	206	43
Cd	n.d.	59	59	77	62 J	53 J	56 J	56	60 J	7.8	13
Cr	n.d.	1,480	1,550	1,910	1,550	1,330	1,440	1,450	1,530	184	12
Cu	n.d.	28 J	25 J	38 J	30 J	26 J	27 J	24 J	28 J	4.7	17
Fe	n.d.	890	911	1,150	940	802	866	850	916	112	12
K	n.d.	2,100 J	1,500 J	3,100 J	2,300 J	2,000 J	2,100 J	1,500 J	2,090 J	543	26
La	n.d.	33 J	28 J	47 J	36 J	31 J	32 J	27 J	33 J	6.7	20
Mn	n.d.	207	216	268	220 J	190 J	200 J	203	215 J	25.5	12
Mo	n.d.	50 J	49 J	67 J	54 J	46 J	48 J	46 J	51 J	7.4	14
Na	97 J	222,000	223,000	285,000	235,000	200,000	220,000	206,000	227,000	28,000	12
Ni	n.d.	395	400	516	420	358	383	391	409	50.7	12
P	n.d.	1,930	1,980	2,500	2,040	1,740	1,880	1,870	1,990	243	12
Pb	n.d.	280 J	250 J	370 J	300 J	260 J	270 J	240 J	281 J	44	16
Zr	n.d.	39 J	38 J	52 J	42 J	36 J	38 J	35 J	40 J	5.7	14
	µCi/mL										%
^{137}Cs	0.03	155	154	197	161	140	151	147	158	18.5	12

Notes:

n.d. = not detected
PB = process blank
J = Results are less than 10 times the MDL, and uncertainty is estimated at greater than 15 percent.
^{137}Cs decay correction reference date is January 2001.

Table 5.4. Extended Homogeneity Test Results - AN-102-AR-I (RPL# 01-420)

Analyte	PB	#1	#2	#3	#4	#5	#6	#7	Average	Std Dev	RSD
	µg/mL										%
Al	n.d.	30,000	28,900	28,800	27,400	28,300	28,000	29,500	28,700	887	3
B	68 J	95 J	130 J	110 J	120 J	100 J	110 J	94 J	108 J	13	12
Ba	n.d.	21 J	22 J	20 J	22 J	21 J	22 J	21 J	21 J	0.8	4
Ca	n.d.	520 J	560 J	510 J	580 J	520 J	570 J	520 J	540 J	29	5
Cd	n.d.	60	60 J	59 J	58 J	59	59 J	61	59 J	1.0	2
Cr	n.d.	1,550	1,540	1,500	1,470	1,500	1,490	1,550	1,510	32	2
Cu	n.d.	24 J	29 J	22 J	29 J	25 J	29 J	25 J	26 J	2.9	11
Fe	n.d.	916	932	886	891	882	899	921	904	19	2
K	n.d.	1,200 J	2,000 J	n.d.	2,500 J	1,600 J	2,300 J	1,500 J	1,850 J	501	27
La	n.d.	26 J	34 J	n.d.	36 J	28 J	35 J	28 J	31 J	4.3	14
Mn	n.d.	219	220 J	210 J	200 J	209	210 J	217	212 J	7.1	3
Mo	n.d.	48 J	52 J	47 J	51 J	49 J	50 J	50 J	50 J	1.7	3
Na	88 J	230,000	233,000	222,000	223,000	212,000	226,000	223,000	224,000	6,720	3
Ni	n.d.	411	405	404	398	401	400	413	405	5.6	1
P	n.d.	2,010	1,960	1,960	1,910	1,960	1,950	2,010	1,970	35	2
Pb	n.d.	250 J	280 J	240 J	290 J	260 J	280 J	260 J	266 J	18	7
Zr	n.d.	37 J	39 J	35 J	39 J	37 J	40 J	38 J	38 J	1.7	4
	µCi/mL										%
^{137}Cs	0.06	157	155	158	158	156	156	156	156	1.4	1

Notes:

n.d. = not detected

PB = process blank

J = Results are less than 10 times the MDL, and uncertainty is estimated at greater than 15 percent.

^{137}Cs decay correction reference date is January 2001.

Table 5.5. Extended Homogenizing Testing Results - Four AN-102 Slurry Sub-Samples

	Average AR-A	Average AR-B	Average AR-C	Average AR-I	Four Bottle Average	Four Bottle Std Dev	Four Bottle RSD
Analyte	**µg/mL**				**µg/mL**		**%**
Al	28,100	28,400	28,900	28,700	28,500	291	1.0
B	100 J	106 J	98 J	108 J	103 J	4.8	4.6
Ba	22 J	22 J	22 J	21 J	22 J	0.4	1.8
Ca	550 J	550 J	485 J	540 J	547 J	4.8	0.9
Cd	59 J	60 J	60 J	59 J	60 J	0.5	0.9
Cr	1,490	1,487	1,530	1,514	1,510	17	1.1
Cu	28 J	26 J	28 J	26 J	27 J	1.2	4.4
Fe	900	913	916	904	907	6.1	0.7
K	2,160 J	1,957 J	2,090 J	1,850 J	2,010 J	134	6.6
La	33 J	32 J	33 J	31 J	33 J	1.0	3.2
Mn	209 J	212 J	215 J	212 J	212 J	2.2	1.0
Mo	51 J	50 J	51 J	50 J	50 J	0.8	1.6
Na	224,000	226,000	227,000	224,000	225,000	1230	0.5
Ni	402	405	409	405	405	2.2	0.5
P	1,950	1,960	1,990	1,970	1,970	11.6	0.6
Pb	280 J	274 J	281 J	266 J	275 J	6.8	2.5
Zr	39 J	39 J	40 J	38 J	39 J	0.9	2.2
	µCi/mL				**µCi/mL**		**%**
^{137}Cs	155	156	158	156	156	1.0	0.6

J = Results are less than 10 times the MDL, and uncertainty is estimated at greater than 15 percent.
^{137}Cs decay correction reference date is January 2001.

6.0 Analytical Sample Preparation (Phase Separation)

Following the compositing, homogenization, and sub-sampling of the AN-102 as-received material reported in Section 3, the bottles identified for characterization were transferred from the HLRF to the SAL. Before any characterization analyses defined by the TS, the EHT reported in Section 5 was completed. Based on the excellent compositing sub-sampling and analytical aliquoting results, the remainder of the AN-102 as-received material in bottles AN-102-AR-A, AN-102-AR-B, AN-102-AR-C, and AN-102-AR-I and the bottles sub-sampled to provide sufficient sample for polychlorinated biphenyl (PCB) analysis (i.e., AN-102-AR-D, AN-102-AR-F, and AN-102-AR-G) were phase separated.

Phase separation and supernatant and solids compositing were conducted according to instructions delineated in ASR 6019. The phase separation was performed by centrifuging each of the AN-102-AR bottles at 1000 G for 1 h (with secondary containment in case of breakage). The supernatant was then decanted into a bottle labeled AN-102 SUP AR; the wet-centrifuged solids were transferred by spatula into a bottle labeled AN-102 CS AR. The decanting and transfer operations were performed as rapidly as possible with remote manipulators to minimize the time that the supernatant and wet centrifuged solids were exposed to the SAL environment (i.e., to reduce potential of cross contamination and weight change due to evaporation/drying). Table 6.1 identifies the masses of supernatant and wet-centrifuged solids collected for analytical characterization.

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

Bottles Sub-sampled for Characterization		**Sample Mass (g)**	**Total Mass (g)**	**Mass used for EHT (g)**	**Mass Lost during Phase Separation (g)**	**Percent Lost during Phase Separation (%)**
RPL Number	**Bottle ID**					
01-386	AN-102-AR-A	163	1121	21	132	12
01-387	AN-102-AR-B	166				
01-388	AN-102-AR-C	161				
None [(a)]	AN-102-AR-D	160				
None [(a)]	AN-102-AR-F	149				
None [(a)]	AN-102-AR-G	162				
01-420	AN-102-AR-I	160				
Supernatant and Wet Centrifuged Solids Phase Separated Samples		**Bottle Tare Mass (g)**	**Bottle Gross Mass (g)**		**Sample Mass (g)**	**Total Mass (g)**
RPL Number	**Bottle ID**					
01-429	AN-102 SUP AR	449.7	937.8		488.1	968.3
01-430	AN-102 CS AR	308.7	788.9		480.2	

(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 total dissolved solids (TDS) of the supernatant, and the wt% solids of the wet centrifuged solids were measured to compare with results obtained from the composite sub-sample AN-102-AR-H (i.e., Table 4.1 and Table 4.2). These limited physical-property measurement on the phase-separated supernatant and wet-centrifuged solids were performed in duplicate; the results are presented in Table 6.2. One of the TDS results on the supernatant did not match well with previous data, and the supernatant was re-sampled to confirm the nominal TDS value.

Table 6.2. Density and Percent Solids on Analytical Characterization Samples

RPL Number	Sample ID	Analysis	Sample	Duplicate	Average	RPD
01-0429	AN-102 SUP AR	Density (g/mL)	1.436	1.438	1.437	0%
		TDS (%) [(a)]	53.5 [(b)]	52.6	53.1	1.7%
01-0430	AN-102 CS AR	Wt% Total Solids (%) [(a)]	58.5	59.3	58.9	1.4%

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

(b) Initial result was 58.9% and reanalysis was performed since the results did not match previous TDS values.

The supernatant density values reported in Table 6.2 compare well with the average densities obtained in Section 4 (i.e., 1.42 and 1.406 g/mL), as does the TDS (i.e., 50.4% and 49.2%) and the wt% solids on the wet-centrifuged solids (i.e., 63.1% and 58.8%). Based on the physical-measurements results, the supernatant and wet-centrifuged solids phases separated in the SAL (i.e., AN-102 SUP AR and AN-102 CS AR) 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 Specification 8 (Envelope D for the undissolved solids).

7.0 Analytical Sample Processing

Following the phase separation of the AN-102 as-received composite into supernatant and wet-centrifuged solids, each phase was analyzed for the target analytes defined in the TS. The analytical processing of the supernatant and wet-centrifuged solids and distribution of the unprocessed and processed sample aliquots are detailed in Figure 7.1. ASRs 6019, 6019.01, 6019.02, and 6019.03 and three addenda provided instructions to the laboratory to successfully complete the analytical and QC requirements defined in the TS.

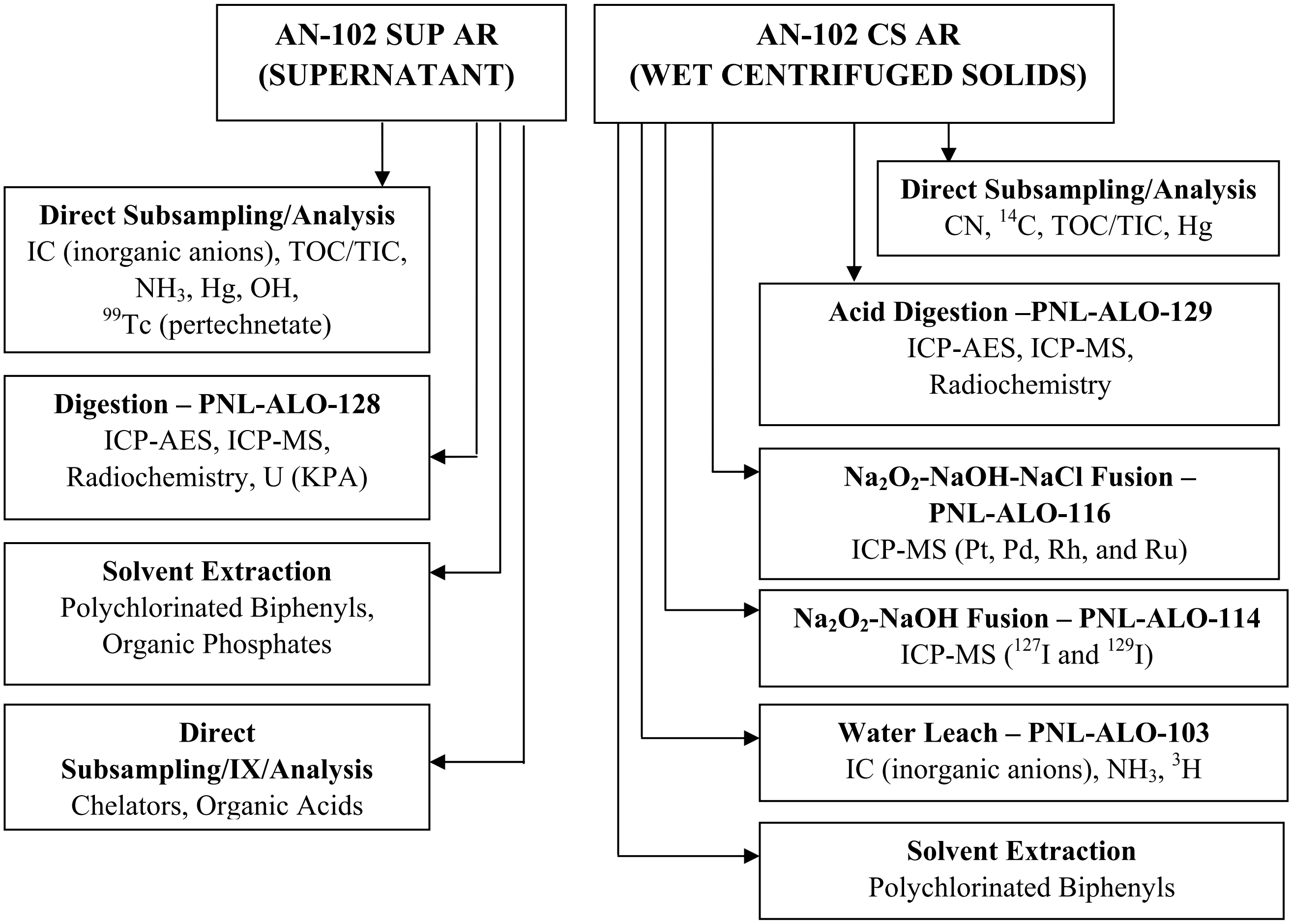

Figure 7.1. Flow Diagram for Analytical Processing of Supernatant and Wet-Centrifuged Solids

7.1 Supernatant

7.1.1 Direct Sub-Sampling/Analysis

The AN-102 as-received supernatant was sub-sampled in the SAL hot cells and then delivered to the RPL analytical workstations for various measurements including inorganic anion, hydroxide, ammonia, mercury, total organic and inorganic carbon (TOC/TIC), and ^{99}Tc (pertechnetate). For these sub-samples, the staff at the analytical workstation were responsible for ensuring that the appropriate batch and analytical QC samples were analyzed and for providing any additional processing to the sub-samples that might be required (e.g., digestions for mercury analysis).

7.1.2 Direct Sub-sampling/IX/Analysis

The AN-102 as-received supernatant was sub-sampled and subjected to ion exchange (IX) procedures (Test Plan TP-RPP-WTP-049, *Ion Exchange for Activity Reduction,* and TI-RPP-WTP-059, *Organic Acids Sample Preparation of Tank AN-102 Supernatant*) in the SAL to reduce the sample dose levels. The resulting effluents from the IX procedures 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).

7.1.3 Acid Digestion

The AN-102 as-received 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 inductively coupled plasma-mass spectrometry (ICP-MS) and to various RPL analytical workstations for ICP-AES, total U by kinetic phosphorescence analysis (KPA) and the following radiochemical analyses: total alpha, gamma emitters by 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, two blank spikes (one for ICP-AES and one for ICP-MS), and two matrix spikes (one for ICP-AES and one for ICP-MS). Aliquots of the blank spike, matrix spike, and the process blanks 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 blank spikes and matrix spikes were prepared at the time of radiochemical separation, except for GEA, which did not require any additional sample preparation.

7.1.4 Solvent Extraction for Organic Phosphates

The AN-102 as-received supernatant was sampled and extracted in the SAL for analysis of bis-(2-ethylhexyl)phosphoric acid (D2EHP) according to the Test Plan (TP) 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 matrix-spike samples (surrogate and D2EHP spike) adjusted to pH <2. A process blank consisting of distilled deionized (DI) water (surrogate spike only) and a blank spike (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 blank-spike sample was inadvertently spilled during the concentration procedure, so an additional BS was synthesized outside the hot cell. The analysis was performed per test plan TP-RPP-WTP-047 using gas chromatography with a flame ionization detector (GC/FID). Since the recoveries from the methylene chloride extracts were good, the butanol extracts were not processed and analyzed.

7.1.5 Solvent Extraction for Polychlorinated Biphenyl

The AN-102 as-received supernatant was sampled and extracted in the SAL for PCB analysis according to the extraction procedure outlined in the ASR 6019.01. Sub-samples consisted of duplicate 100-mL aliquot samples diluted with 120 mL of organic-free water (with surrogate spike only), and duplicate 50-mL aliquot matrix spike samples were diluted with 60-mL organic free water (with surrogate and Aroclor spike). A processing blank consisting of organic-free water (surrogate spike only) and a BS consisting of PCB-spiked organic free water were processed with the sample batch. All samples were extracted three times with methylene chloride. The extracts were combined, and each passed through a sodium sulfate drying column. The extracts were then transferred from the SAL in RPL to the PCB workstation in the 329 Facility under chain of custody (COC) to meet the more stringent QC requirements established for PCBs by the TS. The AN-102 supernatant and QC sample extracts were analyzed for PCBs by gas chromatography/electron capture detection (GC/ECD) per test instruction TI-RPP-WTP-072, *Analysis for PCBs and Pesticides*.

7.2 Wet Centrifuged Solids

7.2.1 Direct Sub-Sampling/Analysis

The AN-102 as-received wet-centrifuged solids were sub-sampled in the SAL and then delivered to the RPL analytical workstations for various measurements, including cyanide, mercury, TOC/TIC, and ^{14}C. For these sub-samples, the staff at the analytical workstation were responsible for ensuring that the appropriate batch and analytical QC samples were analyzed and for providing any additional processing to the sub-samples that might be required (e.g., digestions for mercury analysis or distillation for the cyanide).

7.2.2 Water Leach

The AN-102 as-received wet-centrifuged solids were leached with distilled DI water according to procedure PNL-ALO-103, *Water Leach of Sludges, Soils, and other Solids Samples*, in the SAL hot cells and delivered to various RPL workstations for inorganic anions, ammonia, and tritium analyses.

The SAL prepared duplicate samples of the centrifuged solids for anions and tritium analysis, and on a separate occasion duplicate samples for ammonia analysis, by leaching the samples with distilled DI water at a ratio of ~1:100 (sample:water). Along with the samples for anions and tritium analyses, the SAL prepared a process blank, matrix spike (anions only), and a blank spike (anions only). The blank spike, matrix spike, and process blank were sent with the duplicate samples for anion analysis. For tritium analysis, only the process blank was sent with the samples for analysis. A post water-leach blank spike and matrix spike were prepared at the time of radiochemical separation. Along with the samples for ammonia analysis, the SAL prepared a process blank, blank spike, and duplicate matrix spikes.

7.2.3 Acid Digestion

The AN-102 as-received wet-centrifuged solids were acid digested according to procedure PNL-ALO-129, *HNO_3-HCl Acid Extraction of Solids Using a Dry-Block Heater*, in the SAL. 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, total U by KPA, and the following radiochemical analyses: total alpha, GEA, $^{239+240}$Pu, ^{241} Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm, and ^{90}Sr.

The SAL processed ~0.5 to 0.8-g aliquots of the wet centrifuged solids in duplicate according to PNL-ALO-129. 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, two blank spikes (one for ICP-AES and one for ICP-MS) and two matrix spikes (one for ICP-AES and one for ICP-MS). Aliquots of the blank spike, matrix spike, and the process blanks were sent with aliquots of the duplicate samples for ICP-AES and ICP-MS analyses. For radiochemical analyses, only the duplicate process blanks were sent with the duplicate samples for analysis. Post-digestion blank spikes and matrix spikes were prepared at the time of radiochemical separation, except for GEA, which did not require any additional sample preparation.

7.2.4 Fusion: ALO-116

The AN-102 as-received wet-centrifuged solids were prepared according to procedure PNL-ALO-116, *Solubilization of Platinum Group Metals from Solids Using a Na_2O_2-NaOH-NaCl Fusion*, in the SAL and delivered to the 329 Facility for analysis of Pt, Pd, Rh, and Ru by ICP-MS. Along with the duplicate samples, the SAL prepared duplicate process blanks, a blank spike, and a matrix spike.

7.2.5 Fusion: ALO-114

The AN-102 as-received wet-centrifuged solids were prepared for iodine analysis according to procedure PNL-ALO-114, *Solubilization of Metals from Solids Using a Na_2O_2-NaOH Fusion*, in the SAL and then delivered to the 329 Facility for analysis of ^{127}I and ^{129}I by ICP-MS. Along with the samples, the SAL prepared duplicate process blanks, a blank spike, and a matrix spike.

7.2.6 Solvent Extraction for Polychlorinated Biphenyl

The AN-102 as-received wet-centrifuged solids were sampled and extracted in the SAL for PCB analysis according to the extraction procedure outlined in the ASR 6019.01. Sub-samples consisting of duplicate 2.5 g of wet centrifuged solids (surrogate spike only) and duplicate matrix-spike samples (with surrogate and Aroclor spikes) were extracted with water. The liquid from this extraction was subjected to the process used for the supernatant samples. The solids remaining after the aqueous extraction were dried using granular sodium sulfate and extracted three times with methylene chloride/acetone while subjected to ultrasonication. The methylene chloride residue from the water extract was combined with the methylene chloride/acetone residue produced from the extract of non-aqueous soluble solids. A blank consisting of granular sodium sulfate (surrogate spike only) and a laboratory control standard (LCS) consisting of PCB-spiked granular sodium sulfate were processed with the sample set. The extracts were then transferred from the SAL in the RPL to the PCB workstation in the 329 Facility under COC to meet the more stringent QC requirements established for PCBs by the TS. The AN-102 wet-centrifuged solids and QC sample extracts were analyzed for PCBs by GC/ECD per test plan TI-RPP-WTP-072, *Analysis for PCBs and Pesticides*.

8.0 Analytical Results

8.1 Introduction

Table 8.1 through **Table 8.11** provide inorganic, radioisotopic, and organic analytical results for the AN-102 as-received 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 tables (Table 8.1 through Table 8.11) and the QC tables (Table 9.2 to Table 9.12) include a Data Flag column (i.e., a "Data Qualifier Code") and the analyte concentrations or averages are flagged, as appropriate. The codes utilized were taken from the QA Plan and are defined below, as they relate to this report:

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

The term MDL used in this report is an 'estimated' MDL. That is, the MDLs have not been determined on the AN-102 tank waste matrix per SW-846[3] protocol; however, a few MDLs have been determined for reagent water per the SW-846 protocol (e.g., cyanide and mercury). 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

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[3] 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.

response near background levels. The estimated quantitation limit (EQL) is typically set at 10 times the estimated MDL adjusted for dilution factors resulting from digestion or leaching processing. For a few methods (e.g., IC and ammonia), no ‘estimated’ MDL is determined and the EQL is based on the lowest calibration standard; no results are reported below the EQL for these methods. For radiochemical methods, 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 9.0.

8.2 Analyte List Modifications

The supernatant and “insoluble solids” analyte list were defined by the TS, TSP-W375-00-00007. 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.
- Analysis for ^{135}Cs in the solids was not performed by gamma spectrometry as requested. There are no gamma-emissions associated with this isotope. The concentration may be estimated from the Cs isotopic distribution, assuming the solids have the same isotopic distribution as the supernatant.
- 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 ion chromatography (IC), organic phosphates by GC/FID following derivatization, and chelators by GC/FID following derivatization, not by HPLC/LC-MS as defined by the TS.
- A weight percent fired (i.e., heated in air at 1000 to 1050 °C) oxide physical-property test was added for both the supernatant and wet-centrifuged solids. This test is required in order to be able to convert the undissolved solids analyte concentration from per gram of undissolved solids basis to a per 100 grams of oxide basis, for comparison of the undissolved solids, i.e., “insoluble solids” results with Specification 8 required by the TS.
- Succinic acid and ethylenediaminetriacetic acid (ED3A) 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 total Cs is calculated relative to the ^{137}Cs measured in the solids by GEA.

8.3 Data Limitations

- The reported fluoride results represent the summation of fluoride, acetate, and formate concentrations, as these were not resolvable on the inorganic anion analysis IC system. Thus, the F may be biased high.
- The ICP-MS result at atomic mass unit AMU-241 is either ^{241}Am, ^{241}Pu, or a combination of both. However, comparing the ^{241}Am activities with those found by radiochemical methods [GEA and alpha energy analysis (AEA)] indicates the AMU-241 is largely, if not completely, attributable to ^{241}Am.
- Concentrations of numerous elements are reported by ICP-MS (Table 8.7). Element concentrations are determined by comparison of a selected isotopic mass response for a given element to the calibration curve generated for that element. However, the calibration curve is based on natural abundance, and many of the analytes measured may not have a natural isotopic distribution. Elements such as Rb, Ru, Pd, Sb, Se, Mo, Ce, Te, and AMU-151 (Sm) likely have significantly altered isotopic ratios. For accurate analysis of elements with altered isotopic distributions, chemical separation of the element is required so that individual atomic masses can be quantified. No chemical separations were performed on the AN-102 as-received sample.
- PCBs have a low solubility and tend to plate out on vessel walls. Typically sample vessels are rinsed with a PCB solvent (methylene chloride) to remove potentially plated PCBs. No vessel rinsing was performed during the sampling, homogenization, or sub-sampling steps. This could cause a low bias for the PCB results.
- The reported total PCB MDL represents the MDL summation of six Aroclors. For the supernatant analysis the individual Aroclor MDL is 2E-04 µg/mL and for the wet centrifuged solids is 8E-03 µg/g, making the total PCB MDL for the supernatant and solids 1.2E-3 µg/mL and 4.8E-02 µg/g , respectively.
- Iodine was measured in the wet centrifuged solids by ICP-MS from a fusion preparation and acid digestion. The fusion preparation resulted in undetected ^{127}I with an MDL five times less than the ^{127}I found in the acid digest samples. The fusion prep LCS resulted in acceptable iodine recovery. The fusion preparation for I analysis should keep iodine in solution. However, in this case, it appears to have been lost.
- The pertechnetate QC (process duplicate and MS) failed so this analysis is compromised. However, the ratio of total technetium to pertechnetate is approximately 3, results consistent with analysis from another Envelope C waste, AN-107 (Blanchard 2000).
- The IC phosphate results for the supernatant are reported as less than the MDL (i.e., 5,000 µg/mL based on the dilution required at the IC). This result is consistent with the ICP-AES phosphorous results (average 1,800 µg/mL); i.e., the ICP-AES result on a phosphate basis is about 5600 µg/mL). However, the IC phosphate results on a leach of the wet centrifuged solids averages 8,200 µg/g, as compared to the ICP-AES result of 4,300 µg/g (or 1,400 µg/g phosphorus). The acid digestion preparation (for ICP-AES) should provide a better dissolution of the phosphate than the water leach (for IC). The two most likely explanations for the discrepancy are 1) the IC results are biased high due to the presence of co-eluting organic anions or 2) the ICP-AES results are biased low due to precipitation of phosphate (e.g., as zirconium phosphate).

- 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.

8.4 General Observations

- The total ^{99}Tc measured by ICP-MS is approximately three times the $^{99}TcO_4^{-1}$ (pertechnetate) analysis measured by separations and beta counting. This suggests about a third of the ^{99}Tc is in the pertechnetate state (consistent with Envelope C tank AN-107 results reported by Blanchard, Rapko, and Kurath, 2000). However, the pertechnetate analysis exhibited extremely unrealistic batch matrix spike recovery and the pertechnetate results have been flagged as having a QC deficiency.
- Three analytes (Ce, Mo, and Y) were analyzed by both ICP-AES and ICP-MS on the wet centrifuged solids. The agreement between the results is excellent. The average results from ICP-AES are 19, 33, and 9.4 µg/g for Ce, Mo, and Y, respectively. For ICP-MS, the average results were 25, 33, and 9.3 µg/g for Ce, Mo, and Y, respectively.
- The ^{137}Cs concentration in the supernatant, calculated from ICP-MS measured Cs isotopic distribution as related to ^{133}Cs concentration, agreed within 15% of the ^{137}Cs determined by GEA (converted to µg/mL).
- The TOC measured by both the furnace and the hot persulfate methods is greater than the TOC calculated by summing the individual measured organic species. This indicates either other organic species are present, or one or more of the individually measured organic analyte concentrations is/are underestimated.
- The $^{239+240}$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 difference). The supernatant average being 6.0E-03 µg/mL from AEA versus the sum of 8.5E-03 µg/mL from ICP-MS. Comparable results were obtained on the wet centrifuged solids, where the averages are 4.2E-02 µg/g for AEA versus 7.0E-02 µg/g for ICP-MS.
- The ^{137}Cs concentration (average 3.23E+02 µCi/mL) determined by ICP-MS isotopic distribution compares reasonably well with the ^{137}Cs concentration (average 3.69E+02 µCi/mL) determined by GEA. The difference between the reported values is 14%.

- 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.

8.5 Analytical Results Tables

The analytical results for the AN-102 as-received supernatant are presented in Table 8.1 through Table 8.5 and the AN-102 as-received wet centrifuged solids results are presented in Table 8.6 through Table 8.11. The undissolved solids results corrected for interstitial supernatant analyte contribution are presented in Section 8.6. Comparison of the supernatant to Specification 7 and undissolved solids to Specification 8 are presented in Section 8.7 and Section 8.8, respectively.

Table 8.1. AN-102 As-Received Supernatant – ICP-AES Metals Results

	Process Blank 1			Process Blank 2			Sample			Duplicate		
	MDL	01-429 PB1	Data	MDL	01-429 PB2	Data	MDL	01-429	Data	MDL	01-429 D	Data
Analyte	µg/mL		Flag(c)	µg/mL		Flag(c)	µg/mL		Flag(c)	µg/mL		Flag(c)
ICP-AES Test Specification Analytes (a)												
Al	1.6	5.0	J	1.6	4.9	J	8.2	12,100		3.1	12,400	
Ba	0.3	0.3	U	0.3	0.3	U	1.4	1.4	U	0.5	0.5	U
Ca	6.6	6.6	U	6.6	6.6	U	34	465		13	513	
Cd	0.4	0.4	U	0.4	0.4	U	2.0	61.2		0.8	62.3	
Cr	0.5	0.5	U	0.5	0.5	U	2.7	216		1.0	213	
Fe	0.7	1.6	J	0.7	0.7	U	3.4	37.6		1.3	37.3	
K	52	52	U	53	53	U	270	2,000	JX	110	1,960	X
La	1.3	1.3	U	1.3	1.3	U	6.8	16.0	J	2.6	15.0	J
Mg	2.6	2.6	U	2.6	2.6	U	14	13.6	U	5.2	5.2	U
Na	3.9	55		3.9	58		41	180,000	X (b)	39	188,000	X (b)
Ni	0.8	1.0	J	0.8	0.9	J	4.1	419		1.6	413	
P	2.6	2.6	U	2.6	2.6	U	14	1,800		5.2	1,830	
Pb	2.6	2.6	U	2.6	2.6	U	14	188		5.2	183	
U	52	52	U	53	53	U	270	270	U	110	110	U
Other Analytes Measured												
Ag	0.7	0.7	U	0.7	0.7	U	3.4	3.4	UX	1.3	1.3	UX
As	6.6	6.6	U	6.6	6.6	U	34	34	U	13	13	U
B	1.3	43		1.3	44		6.8	78	B	2.6	82	B
Be	0.3	0.3	U	0.3	0.3	U	1.4	1.4	U	0.5	0.5	U
Bi	2.6	2.6	U	2.6	2.6	U	14	14	U	5.2	5.2	U
Ce	5.2	5.2	U	5.3	5.3	U	27	27	U	10	10	U
Co	1.3	1.3	U	1.3	1.3	U	6.8	6.8	U	2.6	3.9	J
Cu	0.7	0.7	U	0.7	0.7	U	3.4	23	J	1.3	24	
Dy	1.3	1.3	U	1.3	1.3	U	6.8	6.8	U	2.6	2.6	U
Eu	2.6	2.6	U	2.6	2.6	U	14	14	U	5.2	5.2	U

Table 8.1. (Cont'd)

Analyte	Process Blank 1			Process Blank 2			Sample			Duplicate		
	MDL	01-429 PB1	Data Flag[c]	MDL	01-429 PB2	Data Flag[c]	MDL	01-429	Data Flag[c]	MDL	01-429 D	Data Flag[c]
	µg/mL			µg/mL			µg/mL			µg/mL		
Li	0.8	0.8	U	0.8	0.8	U	4.1	4.1	U	1.6	1.6	U
Mn	1.3	1.3	U	1.3	1.3	U	6.8	17	J	2.6	16	J
Mo	1.3	1.3	U	1.3	1.3	U	6.8	53	J	2.6	52	
Nd	2.6	2.6	U	2.6	2.6	U	14	33	J	5.2	30	J
Pd	20	20	U	20	20	U	100	100	U	39	39	U
Rh	7.9	7.9	U	7.9	7.9	U	41	41	U	16	16	U
Ru	29	29	U	29	29	U	150	150	U	58	58	U
Sb	13	13	U	13	13	U	68	68	U	26	26	U
Se	6.6	6.6	U	6.6	6.6	U	34	34	U	13	13	U
Si	13	151		13	139		68	290	JBX	26	180	JBX
Sn	39	39	U	39	39	U	200	200	U	79	79	U
Sr	0.4	0.4	U	0.4	0.4	U	2.0	2.3	J	0.8	2.3	J
Te	39	39	U	39	39	U	200	200	U	79	79	U
Th	26	26	U	26	26	U	140	140	U	52	52	U
Ti	0.7	0.7	U	0.7	0.7	U	3.4	3.4	U	1.3	1.3	U
Tl	13	13	U	13	13	U	68	68	U	26	26	U
V	1.3	1.3	U	1.3	1.3	U	6.8	6.8	U	2.6	2.6	U
W	52	52	U	53	53	U	270	270	U	110	130	J
Y	1.3	1.3	U	1.3	1.3	U	6.8	6.8	U	2.6	2.8	J
Zn	1.3	1.3	U	1.3	1.3	U	6.8	6.8	U	2.6	6.2	J
Zr	1.3	1.3	U	1.3	1.3	U	6.8	8.4	J	2.6	7.9	J

(a) All ICP-AES analytes reported except sulfur (See Section 8.2)

(b) The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 4.3%. The serial dilution for this analyte was not measured.

(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 8.1).

Table 8.2. AN-102 As-Received Supernatant - ICP-MS Results

Analytes	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-429-PB1	± 1SD	Data Flag(d)	MDL	01-429-PB2	± 1SD	Data Flag(d)	MDL	01-429	± 1SD	Data Flag(d)	MDL	01-429 D	± 1SD	Data Flag(d)
	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)		6E-03	1.15E+01	5% (a)		6E-03	1.23E+01	5% (a)	
^{133}Cs	2E-01	2E-01		U	2E-01	2E-01		U	2E-01	9.60E+00	1.05E-01		2E-01	9.61E+00	6.47E-02	
^{135}Cs(b)		NA				NA				2.65E+00	4.5E-02			2.65E+00	2.4E-02	
^{137}Cs(b)		NA				NA				3.72E+00	6.2E-02			3.70E+00	4.1E-02	
Rb	1E-01	1E-01		U	1E-01	1E-01		U	1E-01	8.91E+00	7.69E-02		9E-02	8.56E+00	3.50E-02	
AMU-241 (c)	2E-03	2E-03		U	2E-03	2E-03		U	2E-03	4.86E-02	3.11E-05		1E-03	4.93E-02	7.94E-04	
	µCi/mL				µCi/mL				µCi/mL				µCi/mL			
^{99}Tc	6E-03	6E-03		U	7E-03	7E-03		U	5E-03	1.48E-01	1.97E-03		5E-03	1.48E-01	1.87E-03	
^{237}Np	1E-06	1E-06		U	1E-06	1E-06		U	3E-06	1.21E-04	3.76E-06		2E-06	1.20E-04	1.32E-06	
^{239}Pu	2E-04	2E-04		U	2E-04	2E-04		U	4E-04	6.75E-03	1.38E-04		3E-04	6.18E-03	1.82E-04	
^{240}Pu	3E-04	3E-04		U	3E-04	3E-04		U	7E-04	2.07E-03	1.13E-04	J	6E-04	1.96E-03	2.61E-04	J
AMU-241 (c)	6E-03	6E-03		U	6E-03	6E-03		U	5E-03	1.57E-01	1.00E-04		5E-03	1.59E-01	2.56E-03	

NA = not applicable

(a) Uranium results by KPA; the ±1 SD is 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 the sample and duplicate are 3.66E+02 µCi/mL (4.20E00 µg/mL) and 3.71E+02 µCi/mL (4.26E00 µg/mL).

(c) ^{241}AMU 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 8.1).

Decay correction reference date is nominally April 2001.

Table 8.3. AN-102 As-Received Supernatant - Radioisotope Results

	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDA	01-429-PB1	Err(a)	Data	MDA	01-429-PB2	Err	Data	MDA	01-429 Sample	Err	Data	MDA	01-429 Dup	Err	Data
Analyte	µCi/mL		%	Flag(c)	µCi/mL		%	Flag(c)	µCi/mL		%	Flag(c)	µCi/mL		%	Flag(c)
Alpha	6E-05	1.10E-03	5		6E-05	3.60E-04	10		5E-03	1.66E-01	4		6E-03	1.66E-01	4	
^{238}Pu	2E-06	3.20E-04	3		6E-07	9.49E-05	3		3E-04	1.66E-03	14	BJ	1E-04	1.63E-03	11	BJ
$^{239/240}$Pu	2E-06	4.07E-05	6		5E-07	1.67E-05	6		2E-04	6.00E-03	6		1E-04	5.80E-03	5	
^{241}Am	4E-06	1.06E-04	6		1E-06	4.45E-05	4		2E-04	1.49E-01	2		3E-04	1.52E-01	2	
^{241}Am (GEA)	6E-04	6E-04		U	6E-04	6E-04		U	1E-01	1E-01		U	1E-01	1.65E-01	31	J
$^{243/244}$Cm	4E-06	5.71E-04	3		1E-06	1.56E-04	3		2E-04	6.51E-03	6	B	2E-04	6.91E-03	7	B
^{242}Cm	2E-06	2E-06		U	5E-07	5E-07		U	2E-04	6.53E-04	20	J	2E-04	6.04E-04	23	J
Sum of Alpha		1.04E-03	4			3.12E-04	4			1.64E-01	3			1.67E-01	3	
^{90}Sr	4E-04	1.47E-02	3		4E-04	4.47E-03	5		7E-01	5.58E+01	3		7E-01	5.86E+01	3	
^{99}Tc	2E-06	2E-06		U					6E-06	5.55E-02	4	X		NM(b)		
^{137}Cs	2E-04	6.42E-03	3		3E-04	2.17E-03	7		5E-02	3.66E+02	2		5E-02	3.71E+02	2	
^{60}Co	3E-04	3E-04		U	3E-04	3E-04		U	2E-03	8.58E-02	3		3E-03	8.39E-02	3	
^{154}Eu	6E-04	6E-04		U	6E-04	6E-04		U	9E-03	2.30E-01	3		1E-02	2.31E-01	3	
^{155}Eu	6E-04	6E-04		U	6E-04	6E-04		U	1E-01	1E-01		U	1E-01	1E-01		U

NM = not measured

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

(b) The duplicate sample was not measured (See Section 9.4.5 and Table 9.6)

(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 8.1).

Decay correction reference date is nominally May 2001

Table 8.4. AN-102 As-Received Supernatant - Other Analytes Results

	Process Blank			Sample			Duplicate		
	EQL/ MDL (a)	01-429-PB	Data Flag(h)	EQL/ MDL (a)	01-429	Data Flag(h)	EQL/ MDL (a)	01-429 D	Data Flag(h)
Analyte	µg/mL			µg/mL			µg/mL		
Test Specification Analytes									
F (b)	0.25	0.25	U	2,500	2,500	U	2,500	2,500	U
Cl	0.25	0.25	U	2,500	4,780		2,500	4,860	
NO_2	0.5	0.5	U	5,000	85,900		5,000	84,800	
NO_3	0.5	0.5	U	5,000	223,000		5,000	219,000	
PO_4	0.5	0.5	U	5,000	5,000	U	5,000	5,000	U
SO_4	0.5	0.5	U	5,000	16,800		5,000	16,900	
OH	170	170(d)	U	170	4,800		170	3,700	
NH_3	70	70(g)	U	70	153		70	150	
Hg	0.00014	0.00014	U	0.00014	0.00014	U	0.00014	0.00014	U
TOC-F (c)		n/a		140	18,200		60	17,900	
TIC-F (c)		n/a		140	23,300		120	22,900	
TOC-P (c)		n/a		87	29,400		170	29,100	
TIC-P (c)		n/a		33	10,900		65	11,000	
Gluconate	1	1	U	1,000	1,000	U	1,000	1,000	U
Glycolate(e)	0.1	0.1	U	100	11,000		100	10,000	
Formate	0.1	0.1	U	100	8,000		100	8,000	
Oxalate	0.2	0.2	U	200	510	X	200	410	X
Citrate	0.2	0.2	U	200	4,400		200	4,400	
Other Analytes Measured									
Br	0.25	0.25	U	2,500	2,500	U	2,500	2,500	U
Oxalate(f)	0.5	0.5	U	5,000	5,000	U	5,000	5,000	U

Table 8.4. (Cont'd)

Analyte	Process Blank			Sample			Duplicate		
	EQL/ MDL (a)	01-429-PB	Data Flag(h)	EQL/ MDL (a)	01-429	Data Flag(h)	EQL/ MDL (a)	01-429 D	Data Flag(h)
	µg/mL			µg/mL			µg/mL		

(a) F, Cl, NO_2, NO_3, PO_4, SO_4, Br, Oxalate (Other) and ammonia are reported only 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 (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 to report oxalate results from organic IC analysis.

(g) Ammonia detected in the blank at 3 µg/mL, well below the linear calibration range and the EQL.

(h) 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 8.1).

Table 8.5. AN-102 As-Received Supernatant - Other Organic Analytes Results

			Process Blank		**Sample**		**Duplicate**	
Analyte	**CAS #**	**MDL µg/mL**	**01-429-PB µg/mL**	**Data Flag(c)**	**01-429 Sample µg/mL**	**Data Flag(c)**	**01-429 Dup µg/mL**	**Data Flag(c)**
Polychlorinated Biphenyl Analytes								
Aroclor 1016/1242	12674-11-2/ 53429-21-9	0.0002	0.0002	U	0.0002	U	0.0002	U
Aroclor 1221	11104-28-2	0.0002	0.0002	U	0.0002	U	0.0002	U
Aroclor 1232	11141-16-5	0.0002	0.0002	U	0.0002	U	0.0002	U
Aroclor 1248	12672-29-6	0.0002	0.0002	U	0.0002	U	0.0002	U
Aroclor 1254	11097-69-1	0.0002	0.0002	U	0.0002	U	0.0002	U
Aroclor 1260	11096-82-5	0.0002	0.0002	U	0.0002	U	0.0002	U
Total PCB		0.0012	0.0012	U	0.0012	U	0.0012	U
TCX (surrogate)	877-09-8		66% (a)		89% (a)		78% (a)	
DCB (surrogate)	2051-24-3		62% (a)		65% (a)		26% (a)	
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)		144% (a)		187% (a)	
Chelators and Degradation Products								
EDTA	60-00-4	120	120	UX	600	JX	240	JX
HEDTA(d)	150-39-0	120	120	UX	120	UX	120	UX
ED3A(d)		120	120	UX	1,500	X	560	X
NTA	139-13-9	100	100	UX	260	JX	110	JX
IDA (as NIDA)(d)	142-73-4	120	120	UX	3,500	X	1,400	X
Citric acid(b)	77-92-9	130	130	U	430	J	130	J
Succinic acid	110-15-6	34	34	UX	36	JX	34	JX
AA (surrogate)			86% (a)		91% (a)		80% (a)	

Table 8.5. (Cont'd)

Analyte	CAS #	MDL µg/mL	Process Blank 01-429-PB µg/mL	Data Flag[c]	Sample 01-429 Sample µg/mL	Data Flag[c]	Duplicate 01-429 Dup µg/mL	Data Flag[c]

TCX= 2,4,5,6-tetrachloroxylene; DCB= decachlorobiphenyl; D2EHP= bis-(2-ethylhexyl)phosphate; DDP= diphenylphosphate;
EDTA= ethylenediaminetetraacetic acid; ED3A=ethylenediaminetriacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetetraacetic 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) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 8.1).
(d) Concentration results based on EDTA calibration; results are considered qualitative (See Section 9.12).
(e) D2EHP analysis performed on a best effort basis; results are considered qualitative (See Section 9.13).

Table 8.6. AN-102 As-Received Wet Centrifuged Solids[a] - ICP-AES Metals Results

	Process Blank 1			**Process Blank 2**			**Sample**			**Duplicate**		
	MDL	**01-430 PB1**	**Data**	**MDL**	**01-430 PB2**	**Data**	**MDL**	**01-430**	**Data**	**MDL**	**01-430 D**	**Data**
Analyte	**µg/g**		**Flag[d]**	**µg/g**		**Flag[d]**	**µg/g**		**Flag[d]**	**µg/g**		**Flag[d]**
ICP-AES Test Specification Analytes[b]												
Ag	1.0	1.0	U	0.9	0.9	U	2.1	2.1	UX	2.5	2.5	UX
Al	2.4	2.4	U	2.3	2.6	J	5.1	28,000		6.0	29,000	
Ba	0.4	0.4	U	0.4	0.4	U	0.8	27		1.0	28	
Bi	4.0	4.0	U	3.8	3.8	U	8.5	8.5	U	10	10	U
Ca	10	10	U	9.5	9.5	U	21	434		25	407	
Cd	0.6	0.6	U	0.6	0.6	U	1.3	37		1.5	39	
Cr	0.8	0.8	U	0.8	0.8	U	1.7	1,800		2.0	1,850	
Cu	1.0	1.0	U	0.9	0.9	U	2.1	19	J	2.5	19	J
Fe	1.0	4.9	J	0.9	1.2	J	2.1	1,150		2.5	1,180	
La	2.0	2.0	U	1.9	1.9	U	4.2	32	J	5.0	32	J
Mg	4.0	4.0	U	3.8	3.8	U	8.5	11	J	10	10	U
Mn	2.0	2.0	U	1.9	1.9	U	4.2	254		5.0	261	
Na	6.0	73	X	5.7	87		63	160,000	X[c]	75	173,000	X[c]
Nd	4.0	4.0	U	3.8	3.8	U	8.5	65	J	10	64	J
Ni	1.2	1.2	U	1.1	1.1	U	2.5	254		3.0	261	
P	4.0	4.0	U	3.8	3.8	U	8.5	1,390		10	1,420	
Pb	4.0	4.0	U	3.8	40.3		8.5	245	B	10	244	B
Pd	30	30	U	28	28	U	63	63	U	75	75	U
Rh	12	12	U	11	11	U	25	25	U	30	30	U
Ru	44	44	U	42	42	U	93	93	U	110	110	U
Si	20	120	J	19	130	J	42	190	JB	50	210	JB
Sr	0.6	0.6	U	0.6	0.6	U	1.3	6.1	J	1.5	6.2	J
Ti	1.0	1.0	U	0.9	0.9	U	2.1	2.1	U	2.5	2.5	U
Zn	2.0	2.0	U	1.9	1.9	U	4.2	21	J	5.0	23	J
Zr	2.0	2.0	U	1.9	1.9	U	4.2	46		5.0	47	J

Table 8.6. (Cont'd)

Analyte	Process Blank 1			Process Blank 2			Sample			Duplicate		
	MDL	01-430 PB1	Data Flag[(d)]	MDL	01-430 PB2	Data Flag[(d)]	MDL	01-430	Data Flag[(d)]	MDL	01-430 D	Data Flag[(d)]
	µg/g			µg/g			µg/g			µg/g		
ICP-MS Test Specification Analytes Measured by ICP-AES												
As	10	10	U	9.5	9.5	U	21	21	U	25	25	U
B	2.0	50		1.9	60		4.2	90	B	5.0	97	B
Be	0.4	0.4	U	0.4	0.4	U	0.8	0.8	U	1.0	1.0	U
Ce	8.1	8.1	U	7.6	7.6	U	17	17	J	20	20	U
Co	2.0	2.0	U	1.9	1.9	U	4.2	4.2	U	5.0	5.0	U
K	81	81	U	76	76	U	170	1,100	JX	200	1,100	JX
Li	1.2	1.2	U	1.1	1.1	U	2.5	2.5	U	3.0	3.0	U
Mo	2.0	2.0	U	1.9	1.9	U	4.2	33	J	5.0	33	J
Sb	20	20	U	19	19	U	42	42	U	50	50	U
Se	10	10	U	9.5	9.5	U	21	21	U	25	25	U
Te	60	60	U	57	57	U	130	130	U	150	150	U
Th	40	40	U	38	38	U	85	85	U	100	100	U
Tl	20	20	U	19	19	U	42	42	U	50	50	U
U	81	81	U	76	76	U	170	170	U	200	200	U
V	2.0	2.0	U	1.9	1.9	U	4.2	4.2	U	5.0	5.0	U
W	81	81	U	76	76	U	170	170	U	200	200	U
Y	2.0	2.0	U	1.9	1.9	U	4.2	9.4	J	5.0	9.5	J
Other Analytes Measured												
Dy	2.0	2.0	U	1.9	1.9	U	4.2	4.2	U	5.0	5.0	U
Eu	4.0	4.0	U	3.8	3.8	U	8.5	8.5	U	10	10	U
Sn	60	60	U	57	57	U	130	130	U	150	150	U

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

(b) All ICP-AES analytes reported except sulfur (See Section 8.2).

(c) The RPD required for Na was <3.5%; the supernatant RPD value obtained for Na was 7.8%. The serial dilution for this analyte was not measured.

(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 8.1)

Table 8.7. AN-102 As-Received Wet Centrifuged Solids(a) - ICP-MS Metals Results

	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-430-PB1	± 1SD	(d) Data	MDL	01-430-PB2	± 1SD	(d) Data	MDL	01-430	± 1SD	(d) Data	MDL	01-430 D	± 1SD	(d) Data
Analytes	µg/g			Flag	µg/g			Flag	µg/g			Flag	µg/g			Flag
As	9.4E-01	3.52E+00	3.46E-01	J	9.7E-01	2.29E+00	1.10E-01	J	2.4E+00	5.88E+00	6.05E-01	JB	3.3E+00	7.19E+00	7.77E-01	JB
B	3.9E-01	2.30E+01	3.14E-01		4.1E-01	1.86E+01	7.11E-01		2.2E+00	2.76E+01	3.03E-01	BX	2.9E+00	5.13E+01	9.16E-01	BX
Be	1E-01	1E-01		U	1E-01	1E-01		U	3.2E-01	1.26E+00	3.40E-01	J	4.4E-01	9.83E-01	5.36E-02	J
Ce	4E-01	4E-01		U	4E-01	4E-01		U	3.8E-01	2.25E+01	8.87E-01		4.5E-01	2.35E+01	2.43E-01	
Co	4E-01	4E-01		U	4E-01	4E-01		U	7.7E-02	2.57E+00	8.81E-02		1.1E-01	2.15E+00	1.98E-01	
Cs$^{(b)}$		3.2E-04	2.0E-05			1.1E-03	4.0E-05			9.36E+00	2.83E-01			9.36E+00	2.83E-01	
^{127}I	1E-01	1E-01		U	1E-01	1E-01		U	5.1E-01	2.43E+00	1.17E-01	J	5.4E-01	2.46E+00	4.59E-01	J
^{127}I$^{(c)}$	1E+00	1E+00		U	1E+00	1E+00		U	5.5E-01	5.49E-01		U	6E-01	6E-01		U
Li	4E-01	4E-01		U	4E-01	4E-01		U	2.9E-01	5.74E-01	4.00E-02	JX	4.0E-01	3.56E+01	6.99E-01	X
Mo	2E+00	1E+00		U	3E+00	1E+00		U	2.4E+00	3.34E+01	1.28E+00		3.3E+00	3.26E+01	2.66E+00	J
Pd$^{(c)}$	1E-01	1E-01		U	1E-01	1E-01		U	1.5E-01	1.30E+01	2.62E-01		1.6E-01	1.35E+01	1.78E-01	
Pr	1E-01	1E-01		U	1E-01	1E-01		U	1.2E-01	1.75E+01	2.77E-01		1.5E-01	1.76E+01	2.20E-01	
Pt	5E-02	5E-02		U	6E-02	6E-02		U	4E-02	4E-02		U	5E-02	5E-02		U
Pt$^{(b)}$	7E-02	7E-02		U	7E-02	7E-02		U	7E-02	7E-02		U	8E-02	8E-02		U
Rb	3E-01	3E-01		U	3E-01	3E-01		U	1.8E-01	4.77E+00	2.32E-01		2.5E-01	4.64E+00	4.22E-01	
Rh$^{(c)}$	6E-02	6E-02		U	6E-02	6E-02		U	6.7E-02	4.90E+00	2.63E-02		7.4E-02	5.31E+00	1.11E-01	
Ru$^{(c)}$	1E-01	1E-01		U	1E-01	1E-01		U	1.4E-01	1.47E+01	1.21E-02		1.6E-01	1.56E+01	4.89E-01	
Sb	5E-02	5E-02		U	5E-02	5E-02		U	4.3E-02	1.16E-01	3.36E-02	J	4.8E-02	1.99E-01	3.05E-02	J
Se	1E+01	1E+01		U	1E+01	1E+01		U	1E+02	1E+02		U	2E+01	2E+01		U
Ta	3.2E-02	1.99E-01	9.14E-02	J	3.4E-02	2.00E-01	5.72E-02	J	2.8E-02	2.45E-01	1.18E-02	JBX	3.1E-02	2.61E-01	5.97E-02	JBX
Te	7E-01	7E-01		U	7E-01	7E-01		U	5.6E-01	1.48E+00	1.77E-01	J	6.2E-01	1.66E+00	5.40E-01	J
Tl	3E-02	3E-02		U	3E-02	3E-02		U	2E-02	2E-02		U	2E-02	2E-02		U
Th	7E-01	7E-01		U	7E-01	7E-01		U	4.3E-01	2.61E+01	1.28E+00		5.6E-01	2.67E+01	1.27E+00	
U	5E-01	5E-01		U	5E-01	5E-01		U	3.2E-01	6.70E+01	3.46E+00	X	4.2E-01	6.39E+01	1.11E+00	X
V	2.8E-02	5.14E+00	9.18E-02		2.8E-02	4.01E+00	3.25E-01		1.9E-01	6.49E+00	9.90E-02	B	2.6E-01	6.72E+00	1.62E-01	B
W	6E-01	6E-01		U	6E-01	6E-01		U	5.5E-01	9.28E+01	4.56E+00	X	6.6E-01	1.01E+02	2.46E+00	X
Y	1E-02	1E-02		U	1.3E-02	3.78E-02	9.44E-03	J	1.8E-01	9.59E+00	1.39E-01		2.4E-01	9.02E+00	2.39E-01	

Table 8.7. (Cont'd)

	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-430-PB1	± 1SD	(d) Data	MDL	01-430-PB2	± 1SD	(d) Data	MDL	01-430	± 1SD	(d) Data	MDL	01-430 D	± 1SD	(d) Data
Analytes	µg/g			Flag	µg/g			Flag	µg/g			Flag	µg/g			Flag

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

(b) Total Cs by ICP-MS was not required by the TS. The total Cs concentration is estimated based on the assumption the Cs isotopic distribution in the solids is equal to the Cs isotopic distribution in the liquids. The total Cs concentration is determined using the ^{137}Cs measured by GEA in the solids and solids preparation blanks. The uncertainty is derived from the propagated error.

(c) ^{127}I results from fusion preparation prescribed for I analysis; Pd, Pt, Rh, and Ru results from fusion preparation prescribed for platinum group metals. All other results are from acid digestion preparations.

(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 8.1).

Table 8.8. AN-102 As-Received Wet Centrifuged Solids[(a)] - ICP-MS Radioisotope Results [(b)]

	Process Blank 1				**Process Blank 2**				**Sample**				**Duplicate**			
	MDL	**01-430-PB1**	**± 1SD**	[(d)] **Data**	**MDL**	**01-430-PB2**	**± 1SD**	[(d)] **Data**	**MDL**	**01-430**	**± 1SD**	[(d)] **Data**	**MDL**	**01-430 D**	**± 1SD**	[(d)] **Data**
Analytes	**µg/g**			**Flag**	**µg/g**			**Flag**	**µg/g**			**Flag**	**µg/g**			**Flag**
^{99}Tc	1E-01	2E-01		U	5E-02	2E-01		U	1.4E-01	5.59E+00	3.01E-01		2.0E-01	6.04E+00	1.84E-01	
^{129}I	1E-01	1E-01		U	1E-01	1E-01		U	3.3E-01	8.63E-01	4.71E-02	J	3.5E-01	9.61E-01	1.56E-01	J
^{129}I [(c)]	1E+00	1E+00		U	1E+00	1E+00		U	1E+00	1E+00		U	1E+00	1E+00		U
^{233}U	8E-04	8E-04		U	6E-04	6E-04		U	7.8E-04	6.29E-03	1.24E-03	J	7.8E-04	5.20E-03	9.18E-04	J
^{234}U	5E-03	5E-03		U	1E-03	1E-03		U	5E-03	5E-03		U	5E-03	5E-03		U
^{235}U	5E-03	5E-03		U	7E-03	7E-03		U	5.2E-03	4.57E-01	1.72E-02		5.2E-03	4.55E-01	1.44E-02	
^{236}U	3E-03	3E-03		U	2E-03	2E-03		U	2.7E-03	2.08E-02	1.28E-03	J	2.7E-03	1.73E-02	7.05E-04	J
^{238}U	5E-01	5E-01		U	5E-01	5E-01		U	5.0E-01	6.64E+01	2.63E+00		5.0E-01	6.33E+01	1.71E+00	
^{237}Np	3E-03	3E-03		U	3E-03	3E-03		U	8.3E-03	1.33E+00	3.66E-02		1.2E-02	1.38E+00	2.66E-02	
^{239}Pu	4E-03	4E-03		U	3E-03	3E-03		U	1.4E-02	9.02E-01	3.47E-02		2.1E-02	8.88E-01	9.90E-03	
^{240}Pu	2E-03	2E-03		U	2E-03	2E-03		U	2.6E-03	6.65E-02	1.56E-03		3.7E-03	6.59E-02	5.24E-03	
	µCi/g			**Flag**	**µCi/g**			**Flag**	**µCi/g**			**Flag**	**µCi/g**			**Flag**
^{99}Tc	2E-03	3E-03		U	8E-04	3E-03		U	2.4E-03	9.50E-02	5.11E-03		3.3E-03	1.03E-01	3.13E-03	
^{129}I	2E-05	2E-05		U	2E-05	2E-05		U	5.8E-05	1.53E-04	8.35E-06	J	6.2E-05	1.70E-04	2.76E-05	J
^{129}I [(c)]	2E-04	2E-04		U	2E-04	2E-04		U	2E-04	2E-04		U	2E-04	2E-04		U
^{233}U	8E-06	8E-06		U	6E-06	6E-06		U	7.6E-06	6.13E-05	1.21E-05	J	7.6E-06	5.07E-05	8.95E-06	J
^{234}U	3E-05	3E-05		U	7E-06	7E-06		U	3E-05	3E-05		U	3E-05	3E-05		U
^{235}U	1E-08	1E-08		U	2E-08	2E-08		U	1.1E-08	9.89E-07	3.72E-08		1.1E-08	9.84E-07	3.12E-08	
^{236}U	2E-07	2E-07		U	1E-07	1E-07		U	1.8E-07	1.34E-06	8.29E-08	J	1.7E-07	1.12E-06	4.56E-08	J
^{238}U	2E-07	2E-07		U	2E-07	2E-07		U	1.7E-07	2.24E-05	8.87E-07		1.7E-07	2.13E-05	5.75E-07	
^{237}Np	2E-06	2E-06		U	2E-06	2E-06		U	5.6E-06	9.04E-04	2.49E-05		8.1E-06	9.38E-04	1.81E-05	
^{239}Pu	2E-04	2E-04		U	2E-04	2E-04		U	9.0E-04	5.60E-02	2.16E-03		1.3E-03	5.51E-02	6.15E-04	
^{240}Pu	5E-04	5E-04		U	5E-04	5E-04		U	5.8E-04	1.51E-02	3.55E-04		8.3E-04	1.50E-02	1.19E-03	

Table 8.8. (Cont'd)

Analytes	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-430-PB1	± 1SD	(d) Data Flag	MDL	01-430-PB2	± 1SD	(d) Data Flag	MDL	01-430	± 1SD	(d) Data Flag	MDL	01-430 D	± 1SD	(d) Data Flag
	µg/g				µg/g				µg/g				µg/g			

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

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

(c) ^{129}I results from fusion preparation. All other results are from acid digestion preparations.

(d) Data flags: U = not detected above reported MDL; J = estimated value (See Section 8.1).

Decay correction reference data is nominally April 2001.

Table 8.9. AN-102 As-Received Wet Centrifuged Solids[(a)] - Radioisotope Results

	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDA	01-430-PB1	Err[(b)]	[(d)] Data	MDA	01-430-PB2	Err[(b)]	[(d)] Data	MDA	01-430	Err[(b)]	[(d)] Data	MDA	01-430 D	Err[(b)]	[(d)] Data
Analyte	µCi/g		%	Flag	µCi/g		%	Flag	µCi/g		%	Flag	µCi/g		%	Flag
Alpha	2E-04	3.92E-03	4		2E-04	3.11E-03	4		8E-03	4.93E-01	3		2E-02	5.06E-01	3	
^{238}Pu	2E-06	1.27E-03	2		2E-06	8.59E-04	2		2E-03	1.09E-02	11	B	2E-03	1.29E-02	11	BJ
$^{239/240}$Pu	2E-06	1.52E-04	3		2E-06	9.49E-05	4		2E-03	4.16E-02	5		2E-03	4.17E-02	6	
^{241}Am	5E-06	3.33E-04	5		6E-06	2.15E-04	6		2E-03	4.44E-01	3		3E-03	4.38E-01	3	
^{241}Am (GEA)	1E-03	1E-03		U	1E-03	1E-03		U	2E-01	4.12E-01	12		2E-01	4.29E-01	12	J
$^{243/244}$Cm	5E-06	1.97E-03	3		5E-06	1.61E-03	3		2E-03	1.81E-02	10	B	2E-03	1.62E-02	11	BJ
^{242}Cm	4E-06	4E-06		U	4E-06	4E-06		U	1E-03	1E-03		U	2E-03	2E-03		U
Sum of Alpha		3.73E-03	4			2.78E-03	4			5.15E-01	4			5.09E-01	4	
^{90}Sr	2E-03	7.84E-02	3		2E-03	4.59E-02	3		1E+00	1.40E+02	3		2E+00	1.47E+02	3	
^{134}Cs	5E-04	5E-04		U	4E-04	9.60E-04	13	J	6E-03	6E-03		U	6E-03	6E-03		U
^{137}Cs	6E-04	7.32E-03	6		5E-04	2.48E-02	3		4E-02	2.16E+02	2		4E-02	2.16E+02	2	
^{60}Co	4E-03	4E-03		U	4E-03	4E-03		U	3E-03	5.76E-02	3		4E-03	5.66E-02	4	
^{152}Eu	2E-03	2E-03		U	2E-03	2E-03		U	1E-02	1E-02		U	1E-02	1E-02		U
^{154}Eu	2E-03	2E-03		U	2E-03	2E-03		U	9E-03	5.08E-01	2		9E-03	5.15E-01	2	
^{155}Eu	2E-03	2E-03		U	2E-03	2E-03		U	2E-01	3.14E-01	9		2E-01	3.26E-01	10	
^{125}Sb	1E-03	1E-03		U	1E-03	1E-03		U	2E-01	2E-01		U	2E-01	2E-01		U
^{126}SnSb	5E-04	5E-04		U	5E-04	5E-04		U	7E-02	7E-02		U	8E-02	8E-02		U
^{241}Pu	2E-04	8.17E-03	6		2E-04	6.18E-03	6		1E-01	1E-01		U	1E-01	1E-01		U
^{3}H[(c)]	2E-04	7.46E-04	7			NA			2E-03	2E-03		U	3E-03	3E-03		U
^{14}C	4E-04	4E-04				NA			3E-04	5.51E-04	27	J	4E-04	7.29E-04	24	J

NA = not applicable, only one process blank was supplied with the duplicate samples.

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

(b) The % error represents the uncertainty at 1-σ.

(c) The tritium samples showed weak contamination with ^{137}Cs above the detection limit of <2E-04 µCi/g.

(d) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 8.1).

Decay correction reference date is nominally May 2001.

Table 8.10. AN-102 As-Received Wet Centrifuged Solids(a) - Other Analytes Results

	Process Blank			**Sample**			**Duplicate**		
Analyte	**EQL/ MDL (b)**	**01-430-PB**	**Data Flag(h)**	**EQL/ MDL (b)**	**01-430**	**Data Flag(h)**	**EQL/ MDL (b)**	**01-430 D**	**Data Flag(h)**
	µg/g			**µg/g**			**µg/g**		
Test Specification Analytes									
F (c)	12	12	U	250	3,900		250	3,700	
Cl (d)	12	44		250	3,520	B	250	3,140	B
NO_2	24	24	U	500	57,400		500	52,700	
NO_3	24	114		500	137,000		500	128,000	
PO_4	24	24	U	500	8,970		500	7,380	
SO_4	24	24	U	500	17,500		500	15,400	
Cyanide	0.065	0.065	U	1.1	33.1		0.6	32.5	
NH_3	70	70(g)	U	70	87		70	91	
Hg	0.0007	0.0007	U	0.0006	0.0019		0.0007	0.0018	
TOC-F (e)		n/a		300	17,400		390	21,900	
TIC-F (e)		n/a		560	18,500		200	14,100	
TOC-P (e)		n/a		180	25,900		200	25,300	
TIC-P (e)		n/a		66	17,100		76	17,600	
Other Analytes									
Br	12	12	U	250	250	U	250	250	U
Oxalate (f)	24	24	U	500	30,000	X	500	27,600	X

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

(b) F, Cl, NO2, NO3, PO4, SO4, Br, oxalate, and NH_3 are resulted only above the EQL; therefore, the EQL is presented in this column. For all other analytes, the MDL is presented.

(c) Fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggest the presence of co-eluting anion(s), possibly formate or acetate.

(d) The chloride results are considered qualitative or at best the upper bound for the chloride. When corrected for the difference in dilution, the SAL hot cell blank contribution to the reported results is about 15%.

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

(f) Oxalate was analyzed by IC with other inorganic anions. The LCS processed from the SAL hot cell demonstrates no recovery for oxalate; thus the results provided are for information only and for comparison with organic IC results.

(g) Ammonia detected in the blank at 3 µg/mg, well below the linear calibration range and the EQL.

(h) 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 8.1).

Table 8.11. AN-102 As-Received Wet Centrifuged Solids(a) - Other Organic Analytes Results

			Process Blank		**Sample**		**Duplicate**	
Analyte	**CAS #**	**MDL µg/g**	**01-430-PB**	**Data Flag(c)**	**01-430 Sample µg/g**	**Data Flag(c)**	**01-430 Dup µg/g**	**Data Flag(c)**
Polychlorinated Biphenyl Analytes								
Aroclor 1016/1242	12674-11-2/ 53429-21-9	0.008	0.008	U	0.008	U	0.008	U
Aroclor 1221	11104-28-2	0.008	0.008	U	0.008	U	0.008	U
Aroclor 1232	11141-16-5	0.008	0.008	U	0.008	U	0.008	U
Aroclor 1248	12672-29-6	0.008	0.008	U	0.008	U	0.008	U
Aroclor 1254	11097-69-1	0.008	0.008	U	0.008	U	0.017	
Aroclor 1260	11096-82-5	0.008	0.008	U	0.008	U	0.030	
Total PCB		0.048	0.048	U	0.048	U	0.079	
TCX (surrogate)	877-09-8		50% (b)		35% (b)		62% (b)	
DCB (surrogate)	2051-24-3		119% (b)		77% (b)		114% (b)	

TCX= 2,4,5,6-tetrachloroxylene; DCB= decachlorobiphenyl

(a) Concentrations are based on the mass of wet centrifuged solids so the results contain a contribution from interstitial supernatant.
(b) Value represents percent recovery of the surrogate standard
(c) Data flags: U = not detected above reported MDL (See Section 8.1).

8.6 Undissolved Solids Results

Table 8.12 and Table 8.13 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 4.2 have been used to calculate the concentration of each analyte in the UDS per Equation 8.1.

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

Where: X = UDS concentration (μg/g or μCi/g) on a per g of WCS (i.e., UDS concentration on a wet-weight basis)

C = average measured concentration of analyte in WCS (μg/g or μCi/g)

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

D = density of supernatant (1.406 g/mL)

W = fractional solids weight in WCS after drying at 105°C (0.588)

T = fractional solids weight in supernatant after drying at 105°C (0.492).

Also reported in Table 8.12 and Table 8.13 are the analyte concentrations calculated on a dry-weight basis. This is calculated by subtracting the supernatant from the solids fraction according to Equations 8.2 and 8.3.

$$Y = \frac{X}{F} \tag{8.2}$$

Where: Y = UDS 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 of WCS) and F is calculated according to Equation 8.3.

$$F = 1 - \left[\frac{(1-W)}{(1-T)} \right] \tag{8.3}$$

The following apply to the both UDS calculations:

a) If the analyte is not measured on the supernatant, X = C (i.e., assumes no analyte contribution from the supernatant).

b) If the analyte is measured on the supernatant, but not detected above the MDL, "S" is set to zero (*0*).

c) If the analyte is measured on the WCS but is not detected, "X" is set equal to "<C", even if the analyte is measured on the supernatant.

d) If X results in less than or equal to zero, the analyte is indicated to come only from the supernatant within uncertainty of the method. The UDS is then set to the MDL of the WCS.

e) If only one of the duplicate sample results was above the MDL, its value was conservatively used in place of the "average" concentration.

Table 8.12. AN-102 Undissolved Solids (Wet-weight and Dry-weight Basis) - µg/g

	WCS				Supernatant (a)		UDS (b)	
Analyte	**Measure Method**	**MDL µg/g**	**Average µg/g**	**Data Flag(f)**	**Measure Method**	**Average µg/mL**	**Average µg/g (wet)**	**Average µg/g (dry)**
Ag	ICP-AES	2E+00	2E+00	UX	ICP-AES		<2E+00	<1E+01
Al	ICP-AES	5E+00	2.85E+04		ICP-AES	1.23E+04	2.14E+04	1.13E+05
As	ICP-AES	2E+01	2E+01	U	ICP-AES	*0*	<2E+01	<1E+02
As	ICP-MS	2E+00	6.54E+00	JB	NM		6.54E+00	3.46E+01
B	ICP-AES	4E+00	9.35E+01	B	ICP-AES	8.00E+01	4.74E+01	2.51E+02
B	ICP-MS	2E+00	3.95E+01	BX	NM		3.95E+01	2.09E+02
Ba	ICP-AES	8E-01	2.75E+01		ICP-AES	*0*	2.75E+01	1.46E+02
Be	ICP-AES	8E-01	9E-01	U	ICP-AES	*0*	<9E-01	<5E+00
Be	ICP-MS	3E-01	1.12E+00	J	NM		1.12E+00	5.93E+00
Bi	ICP-AES	9E+00	9E+00	U	ICP-AES	*0*	<9E+00	<5E+01
Br	IC	3E+02	3E+02	U	IC	*0*	<3E+02	<1E+03
Ca	ICP-AES	2E+01	4.21E+02		ICP-AES	4.89E+02	1.38E+02	7.33E+02
Cd	ICP-AES	1E+00	3.80E+01		ICP-AES	6.18E+01	2.38E+00	1.26E+01
Ce	ICP-AES	2E+01	1.85E+01	J	ICP-AES	*0*	1.85E+01	9.79E+01
Ce	ICP-MS	4E-01	2.30E+01		NM		2.30E+01	1.22E+02
Cl	IC	3E+02	3.33E+03	B	IC	4.82E+03	5.50E+02	2.91E+03
CN	Dist./Color.	1E+00	3.28E+01		NM		3.28E+01	1.74E+02
Co	ICP-AES	4E+00	5E+00	U	ICP-AES	3.90E+00	<5E+00	<2E+01
Co	ICP-MS	8E-02	2.36E+00		NM		2.36E+00	1.25E+01
Cr	ICP-AES	2E+00	1.83E+03		ICP-AES	2.15E+02	1.70E+03	9.00E+03
Cu	ICP-AES	2E+00	1.90E+01	J	ICP-AES	2.35E+01	5.44E+00	2.88E+01
Dy	ICP-AES	4E+00	5E+00	U	ICP-AES	*0*	<5E+00	<2E+01
Eu	ICP-AES	9E+00	9E+00	U	ICP-AES	*0*	<9E+00	<5E+01
F	IC	3E+02	3.80E+03		IC	*0*	3.80E+03	2.01E+04
Fe	ICP-AES	2E+00	1.17E+03		ICP-AES	3.75E+01	1.14E+03	6.05E+03
Hg	CVAA	6E-04	1.85E-03		CVAA	*0*	1.85E-03	9.79E-03
K	ICP-AES	2E+02	1.10E+03	JX	ICP-AES	1.98E+03	<2E+02 (c)	<9E+02 (c)
La	ICP-AES	4E+00	3.20E+01	J	ICP-AES	1.55E+01	2.31E+01	1.22E+02
Li	ICP-AES	3E+00	3E+00	U	ICP-AES	*0*	<3E+00	<1E+01
Li	ICP-MS	3E-01	1.81E+01	JX	NM		1.81E+01	9.57E+01
Mg	ICP-AES	9E+00	1.10E+01	J	ICP-AES	*0*	1.10E+01	5.82E+01
Mn	ICP-AES	4E+00	2.58E+02		ICP-AES	1.65E+01	2.48E+02	1.31E+03
Mo	ICP-AES	4E+00	3.30E+01	J	ICP-AES	5.25E+01	2.72E+00	1.44E+01
Mo	ICP-MS	2E+00	3.30E+01		NM		3.30E+01	1.75E+02
Na	ICP-AES	6E+01	1.67E+05	X	ICP-AES	1.84E+05	6.04E+04	3.19E+05
Nd	ICP-AES	9E+00	6.45E+01	J	ICP-AES	3.15E+01	4.63E+01	2.45E+02
NH_3	ISE	7E+01	8.90E+01		ISE	1.52E+02	1.61E+00	8.52E+00
Ni	ICP-AES	3E+00	2.58E+02		ICP-AES	4.16E+02	1.75E+01	9.28E+01
NO_2	IC	5E+02	5.51E+04		IC	8.54E+04	5.82E+03	3.08E+04
NO_3	IC	5E+02	1.33E+05		IC	2.21E+05	5.02E+03	2.66E+04

Table 8.12. (Cont'd)

Analyte	WCS				Supernatant (a)		UDS (b)	
	Measure Method	MDL µg/g	Average µg/g	Data Flag(f)	Measure Method	Average µg/mL	Average µg/g (wet)	Average µg/g (dry)
Oxalate	IC	5E+02	2.88E+04	X	IC	4.60E+02	2.85E+04	1.51E+05
P	ICP-AES	9E+00	1.41E+03		ICP-AES	1.82E+03	3.58E+02	1.89E+03
Pb	ICP-AES	9E+00	2.45E+02	B	ICP-AES	1.86E+02	1.37E+02	7.28E+02
Pd	ICP-AES	6E+01	7E+01	U	ICP-AES	*0*	<7E+01	<4E+02
Pd (d)	ICP-MS	1E-01	1.33E+01		NM		1.33E+01	7.01E+01
PO_4	IC	5E+02	8.18E+03		IC	*0*	8.18E+03	4.33E+04
Pr	ICP-MS	1E-01	1.76E+01		NM		1.76E+01	9.29E+01
Pt	ICP-MS	4E-02	5E-02	U	NM		<5E-02	<2E-01
Pt (d)	ICP-MS	7E-02	8E-02	U	NM		<8E-02	<4E-01
Rb	ICP-MS	2E-01	4.71E+00		ICP-MS	8.74E+00	<2E-01 (c)	<1E+00 (c)
Rh	ICP-AES	3E+01	3E+01	U	ICP-AES	*0*	<3E+01	<1E+02
Rh (d)	ICP-MS	7E-02	5.11E+00		NM		5.11E+00	2.70E+01
Ru	ICP-AES	9E+01	1E+02	U	ICP-AES	*0*	<1E+02	<5E+02
Ru (d)	ICP-MS	1E-01	1.52E+01		NM		1.52E+01	8.02E+01
Sb	ICP-AES	4E+01	5E+01	U	ICP-AES	*0*	<5E+01	<2E+02
Sb	ICP-MS	4E-02	1.58E-01	J	NM		1.58E-01	8.33E-01
Se	ICP-AES	2E+01	2E+01	U	ICP-AES	*0*	<2E+01	<1E+02
Se	ICP-MS	1E+02	7E+01	U	NM		<7E+01	<4E+02
Si	ICP-AES	4E+01	2.00E+02	JB	ICP-AES	2.35E+02	6.44E+01	3.41E+02
Sn	ICP-AES	1E+02	1E+02	U	ICP-AES		<1E+02	<7E+02
SO_4	IC	5E+02	1.65E+04		IC	1.69E+04	6.73E+03	3.56E+04
Sr	ICP-AES	1E+00	6.15E+00	J	ICP-AES	2.30E+00	4.82E+00	2.55E+01
Ta	ICP-MS	3E-02	2.53E-01	JBX	NM		2.53E-01	1.34E+00
Te	ICP-AES	1E+02	1E+02	U	ICP-AES	*0*	<1E+02	<7E+02
Te	ICP-MS	6E-01	1.57E+00	J	NM		1.57E+00	8.31E+00
Th	ICP-AES	9E+01	9E+01	U	ICP-AES	*0*	<9E+01	<5E+02
Th	ICP-MS	4E-01	2.64E+01		NM		2.64E+01	1.40E+02
Ti	ICP-AES	2E+00	2E+00	U	ICP-AES	*0*	<2E+00	<1E+01
TIC-F (e)	TOC-TC	6E+02	1.63E+04		TOC-TC	2.31E+04	3.00E+03	1.59E+04
TIC-P (e)	TIC-TOC	7E+01	1.74E+04		TIC-TOC	1.10E+04	1.10E+04	5.84E+04
Tl	ICP-AES	4E+01	5E+01	U	ICP-AES	*0*	<5E+01	<2E+02
Tl	ICP-MS	2E-02	2E-02	U	NM		<2E-02	<1E-01
TOC-F (e)	TOC-TC	4E+02	1.97E+04		TOC-TC	1.81E+04	9.24E+03	4.89E+04
TOC-P (e)	TIC-TOC	2E+02	2.56E+04		TIC-TOC	2.93E+04	8.73E+03	4.62E+04
U	ICP-AES	2E+02	2E+02	U	ICP-AES	*0*	<2E+02	<1E+03
U	ICP-MS	3E-01	6.55E+01	X	U(KPA)	1.19E+01	5.86E+01	3.10E+02
V	ICP-AES	4E+00	5E+00	U	ICP-AES	*0*	<5E+00	<2E+01
V	ICP-MS	2E-01	6.61E+00	B	NM		6.61E+00	3.50E+01
W	ICP-AES	2E+02	2E+02	U	ICP-AES	*0*	1.85E+02	9.79E+02
W	ICP-MS	5E-01	9.69E+01	X	NM		9.69E+01	5.13E+02
Y	ICP-AES	4E+00	9.45E+00	J	ICP-AES	2.80E+00	9.45E+00	5.00E+01
Y	ICP-MS	2E-01	9.31E+00		NM		9.31E+00	4.92E+01

Table 8.12. (Cont'd)

Analyte	WCS				Supernatant (a)		UDS (b)	
	Measure Method	MDL µg/g	Average µg/g	Data Flag(f)	Measure Method	Average µg/mL	Average µg/g (wet)	Average µg/g (dry)
Zn	ICP-AES	4E+00	2.20E+01	J	ICP-AES	6.20E+00	2.20E+01	1.16E+02
Zr	ICP-AES	4E+00	4.65E+01		ICP-AES	8.15E+00	4.18E+01	2.21E+02
Organics								
Aroclor 1016/1242	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	*0*	<8E-03	<4E-02
Aroclor 1221	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	*0*	<8E-03	<4E-02
Aroclor 1232	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	*0*	<8E-03	<4E-02
Aroclor 1248	GC/ECD	8.00E-03	8.00E-03	U	GC/ECD	*0*	<8E-03	<4E-02
Aroclor 1254	GC/ECD	8.00E-03	1.70E-02		GC/ECD	*0*	1.70E-02	9.00E-02
Aroclor 1260	GC/ECD	8.00E-03	3.00E-02		GC/ECD	*0*	3.00E-02	1.59E-01
Total PCB	GC/ECD	4.80E-02	7.90E-02		GC/ECD	*0*	7.90E-02	4.18E-01

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

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured, 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) When the calculated UDS results is <0 or =0, the UDS results is set to <MDL of the WCS.

(d) Results from fusion preparation; other Pd, Pt, Rh, and Ru results from acid digestion.

(e) P= carbon from persulfate method; F= carbon from furnace method-TIC by difference (TIC=TC-TOC).

(f) 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 8.1).

Table 8.13. AN-102 Undissolved Solids (Wet-weight and Dry-weight Basis) - µCi/g

Analyte	WCS				Supernatant (a)		UDS (b)	
	Measure Method	MDL/MDA µCi/g	Average µCi/g	Data Flag(e)	Measure Method	Average µCi/mL	Average µCi/g WCS	Average µCi/g DS
^{3}H (c)	Rad	2E-03	3E-03	U	NM		<3E -03	<1E -02
^{14}C	Rad	4E-04	6.40E-04		NM		6.40E -04	3.39E -03
^{60}Co	GEA	3E-03	5.71E-02		GEA	8.49E-02	8.16E -03	4.32E -02
^{90}Sr	Rad	1E+00	1.44E+02		Rad	5.72E+01	1.11E +02	5.85E +02
^{99}Tc	ICP-MS	2E-03	9.90E-02		ICP-MS	1.48E-01	1.36E -02	7.21E -02
^{125}Sb	GEA	2E-01	2E-01	U	NM		<2E -01	<1 E +00
^{126}SnSb	GEA	7E-02	8E-02	U	NM		<8E-02	<4E-01
^{129}I	ICP-MS	6E-05	1.62E-04	J	NM		1.62E-04	8.55E-04
^{129}I (d)	ICP-MS	2E-04	2E-04	U	NM		<2E-04	<1E-03
^{134}Cs	GEA	6E-03	6E-03	U	NM		<6E-03	<3E-02
^{137}Cs	GEA	4E-02	2.16E+02		GEA	3.69E+02	3.44E+00	1.82E+01
^{152}Eu	GEA	1E-02	1E-02	U	NM		<1E-02	<5E-02
^{154}Eu	GEA	9E-03	5.12E-01		NM		5.12E-01	2.71E+00
^{155}Eu	GEA	2E-01	3.20E-01		GEA	*0*	3.20E-01	1.69E+00

Table 8.13. (Cont'd)

	WCS				Supernatant [(a)]		UDS [(b)]	
Analyte	**Measure Method**	**MDL/MDA µCi/g**	**Average µCi/g**	**Data Flag[(e)]**	**Measure Method**	**Average µCi/mL**	**Average µCi/g WCS**	**Average µCi/g DS**
^{233}U	ICP-MS	8E-06	5.60E-05	J	NM		5.60E-05	2.96E-04
^{234}U	ICP-MS	3E-05	3E-05	U	NM		<3E-05	<2E-04
^{235}U	ICP-MS	1E-08	9.87E-07		NM		9.87E-07	5.22E-06
^{236}U	ICP-MS	2E-07	1.23E-06	J	NM		1.23E-06	6.51E-06
^{238}U	ICP-MS	2E-07	2.19E-05		NM		2.19E-05	1.16E-04
^{237}Np	ICP-MS	6E-06	9.21E-04		ICP-MS	1.21E-04	8.51E-04	4.51E-03
^{238}Pu	Rad	2E-03	1.19E-02	BJ	Rad	1.65E-03	1.10E-02	5.79E-02
^{239}Pu	ICP-MS	9E-04	5.56E-02		ICP-MS	6.47E-03	5.18E-02	2.74E-01
^{240}Pu	ICP-MS	6E-04	1.51E-02		ICP-MS	2.02E-03	1.39E-02	7.35E-02
$^{239/240}$Pu	Rad	2E-03	4.17E-02		Rad	5.90E-03	3.82E-02	2.02E-01
^{241}Pu	Rad	1E-01	1E-01	U	NM		<1E-01	<5E-01
^{241}Am	GEA	2E-01	4.21E-01	J	GEA	1.65E-01	3.25E-01	1.72E+00
^{241}Am	Rad	2E-03	4.41E-01		Rad	1.51E-01	3.54E-01	1.87E+00
^{242}Cm	Rad	1E-03	2E-03	U	NM		<2E-03	<8E-03
$^{243/244}$Cm	Rad	2E-03	1.72E-02	BJ	Rad	6.71E-03	1.33E-02	7.03E-02
Alpha	Rad	8E-03	5.00E-01		Rad	1.66E-01	4.04E-01	2.14E+00
Sum of Alpha	Rad		5.12E-01		Rad	1.66E-01	4.17E-01	2.20E+00

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

(a) If the analyte is measured but not detected above the MDL, the supernatant analyte concentration is set to 0 (zero). If the analyte is not measured, 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) Results from water leach.

(d) Results from fusion preparation; other ^{129}I result from acid digestion.

(e) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 8.1).

8.7 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 8.14 and Table 8.15 present the ratio limits and the as-measured ratios. The mole or Bq analyte to moles Na ratio did not exceed the limits defined in Specification 7 for Envelope C, except for sulfate and ^{60}Co, which were both slightly above the Specification 7 limit. However, ^{60}Co is expected to be below the Specification limit at the time of actual waste processing due to its relatively short half-life (5.3 years). In 2007, the estimated processing time for AN-102 material, the ^{60}Co Bq/mole sodium ratio will be approximately half that reported in Table 8.15.

Table 8.14. AN-102 As-Received Supernatant – Mole Analyte per Mole Na Ratio

Analytes	MRQ µg/mL	MDL/ EQL (a) µg/mL	01-429 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	5.7	12,300		5.7E-02	2.5E-01	22.8	Yes
Ba	2.3	0.9	0.9	U	<8.6E-07	1.0E-04	< 0.9	Yes
Ca	150	24	489		1.5E-03	4.0E-02	3.8	Yes
Cd	7.5	1.4	62		6.9E-05	4.0E-03	1.7	Yes
Cl	300	2,500	4,820		1.7E-02	3.7E-02	45.9	Yes
Cr	15	1.9	215		5.2E-04	6.9E-03	7.5	Yes
F	150	2,500	2,500	U	<1.6E-02	9.1E-02	< 17.6	Yes
Fe	150	2.4	37		8.4E-05	1.0E-02	0.8	Yes
Hg	1.5	0.00014	0.00014	U	<8.7E-11	1.4E-05	< 0.0	Yes
K	75	190	1,980	JX	6.3E-03	1.8E-01	3.5	Yes
La	35	4.7	16	J	1.4E-05	8.3E-05	16.9	Yes
Na	75	40	184,000	X	1	NA		
Ni	30	2.8	416		8.9E-04	3.0E-03	29.7	Yes
NO_2	3,000	5,000	85,350		2.3E-01	3.8E-01	60.5	Yes
NO_3	3,000	5,000	221,000		4.5E-01	8.0E-01	56.3	Yes
Pb	300	9.4	186		1.1E-04	6.8E-04	16.2	Yes
PO_4 (as P) (b)	600	9.4	5,580		7.3E-03	3.8E-02	19.2	Yes
PO_4	2,500	5,000	5,000	U	<6.6E-03	3.8E-02	< 17.4	Yes
SO_4	2,300	5,000	16,850		**2.2E-02**	2.0E-02	**110.0**	**No**
TIC-P (c)	150	49	10,950		1.1E-01	3.0E-01	36.7	Yes
TOC-P (c)	1,500	129	29,250		3.0E-01	5.0E-01	60.0	Yes
TIC-F (c)	150	130	23,100		2.4E-01	3.0E-01	80.0	Yes
TOC-F (c)	1,500	100	18,000		1.9E-01	5.0E-01	38.0	Yes
U	600	190	190	U	<9.9E-05	1.2E-03	< 8.3	Yes

Bolded and Outlined Values exceed Specification 7 criteria.

(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 1820 µg/mL.

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

(d) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 8.1).

Table 8.15. AN-102 As-Received Supernatant – Bq Radioisotope per Mole Na Ratio

Radioisotopes	MRQ µCi/mL	MDA/MDL [(a)] µCi/mL	01-429 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)]	NA	1.65E-01		7.62E+05	3.0E+06	25.4	Yes
^{137}Cs	9.0E+00	5.0E-02	3.69E+02		1.70E+09	4.3E+09	39.5	Yes
^{90}Sr	1.5E-01	7.0E-01	5.72E+01		2.64E+08	8.0E+08	33.0	Yes
^{99}Tc	1.5E-03	5.0E-03	1.48E-01		6.86E+05	7.1E+06	9.7	Yes
^{60}Co	1.0E-02	2.5E-03	8.49E-02		**3.92E+05**	3.7E+05	**105.9**	**No**
^{154}Eu	2.0EE-03	9.5E-03	2.31E-01		1.07E+06	4.3E+06	24.9	Yes

Bolded and Outlined Values exceed Specification 7 criteria.

(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.

8.8 Comparison of Undissolved Solids Results to Specification 8

Specification 8 for Envelope D defines limits for several elemental or anion concentrations and radionuclide activities per 100 g equivalent non-volatile waste oxides (i.e., sodium oxide and silicon oxide). The g of analyte per gram of waste oxide is calculated according to Equation 8.4 when starting from a wet centrifuged solids (i.e., wet-weight basis), and according to Equation 8.5 when starting from an undissolved dry solid (i.e., dry-weight basis).

$$X_o = C * F_1 * \left[\frac{1-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * (1-W)\right)} \right] * F_2 \qquad (8.4)$$

$$X_o = U * F_1 * \left[\frac{W-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * (1-W)\right)} \right] * F_2 \qquad (8.5)$$

where X_o = analyte concentration per mass of oxide (g/100g)
C = undissolved solids analyte concentration in µg/g (wet-weight basis)
U = undissolved solids analyte concentration in µg/g (dry-weight basis)
F_1 = mass conversion factor (g/10^6µg)
W_{ocs} = fractional oxide mass of the centrifuged solids (0.434, Table 4.2)
W_{os} = fraction oxide mass of the supernatant (0.247, Table 4.2)
W = fractional mass of solids in centrifuged solids after drying (0.588, Table 4.2)
T = fractional mass of solids in supernatant after drying (0.492, Table 4.2)
F_2 = 100-g oxide mass conversion factor, 100.

The calculated results are based on the unwashed solids analysis results. Solids feed for vitrification will be washed removing soluble materials. For example, $NaNO_3$ and $NaNO_2$ were found in the solids fraction and would be dissolved during the solids washing processes. The calculated g analyte per 100 g waste oxide results are presented in Table 8.16; the calculated Ci analyte per 100 g waste oxide results are shown in Table 8.17. Except for chromium, sodium, and sulfur, the g or Ci of analyte to 100 g waste oxide ratios do not exceed the limits defined in Specification 8 for Envelope D. Chromium at 107% only slightly exceeds the specification limit, whereas sodium and sulfur are considerably above the specification limit at 148% and 136%, respectively.

Table 8.16. AN-102 Undissolved Solids – g / 100 g Waste Oxide

	Measure Method	Solids MDL µg/g	UDS Average µg/g (wet)	UDS Average µg/g (dry)	Data Flag(g)	UDS Average g/100 g waste oxide	UDS Spec 8 Max g/100 g waste oxide	% of Limit	Meet Spec 8? Y/N
Table TS 8.1 Analytes									
As	ICP-AES	21	23	122	U	< 0.01	0.16	< 6.3	Yes
As	ICP-MS	2.4	6.5	35	JB	0.003	0.16	1.9	Yes
B	ICP-AES	4.2	47.4	251	B	0.02	1.3	1.5	Yes
B	ICP-MS	2.2	39.5	209	BX	0.02	1.3	1.5	Yes
Be	ICP-AES	0.80	0.90	4.76	U	< 0.0004	0.065	< 0.6	Yes
Be	ICP-MS	0.33	1.12	5.93	J	0.0005	0.065	0.8	Yes
Ce	ICP-AES	17	19	98	J	0.01	0.81	1.2	Yes
Ce	ICP-MS	0.38	23	122		0.01	0.81	1.2	Yes
Co	ICP-AES	4.2	4.6	24.3	U	< 0.002	0.45	< 0.4	Yes
Co	ICP-MS	0.08	2.36	12.5		0.001	0.45	0.2	Yes
Cs (total)(a)	ICP-MS/GEA		0.151	0.799		0.000065	0.58	0.0	Yes
Cu	ICP-AES	2.1	5.4	28.8	J	0.002	0.48	0.4	Yes
Hg	CVAA	0.0006	0.0019	0.0098		0.000001	0.10	0.0	Yes
La	ICP-AES	4.2	23.1	122	J	0.01	2.6	0.4	Yes
Li	ICP-AES	2.5	2.8	14.6	U	< 0.001	0.14	< 0.7	Yes
Li	ICP-MS	0.29	18.1	95.7	JX	0.01	0.14	7.1	Yes
Mn	ICP-AES	4.2	248	1312		0.11	6.5	1.7	Yes
Mo	ICP-AES	4.2	2.7	14.4	J	0.001	0.65	0.2	Yes
Mo	ICP-MS	2.4	33.0	175		0.01	0.65	1.5	Yes
Nd	ICP-AES	8.5	46.3	245	J	0.02	1.7	1.2	Yes
Pr	ICP-MS	0.12	17.6	92.9		0.01	0.35	2.9	Yes
Pu	(b)		0.90	4.74		0.0004	0.054	0.7	Yes
Rb	ICP-MS	0.18	< 0.2	< 1		< 0.0001	0.19	< 0.1	Yes
Sb	ICP-AES	42	46	243	U	< 0.02	0.84	< 2.4	Yes
Sb	ICP-MS	0.04	0.16	0.83	J	0.0001	0.84	0.0	Yes
Se	ICP-AES	21	23	122	U	< 0.01	0.52	< 1.9	Yes
Se	ICP-MS	120	68	362	U	< 0.03	0.52	< 5.8	Yes
Sr	ICP-AES	1.3	4.8	25.5	J	0.002	0.52	0.4	Yes
Ta	ICP-MS	0.03	0.25	1.34	JBX	0.0001	0.03	0.3	Yes
Te	ICP-AES	130	140	741	U	< 0.06	0.13	< 46.2	Yes
Te	ICP-MS	0.56	1.57	8.31	J	0.001	0.13	0.8	Yes
Tl	ICP-AES	42	46	243	U	< 0.02	0.45	< 4.4	Yes
Tl	ICP-MS	0.02	0.02	0.12	U	< 0.00001	0.45	< 0.0	Yes
V	ICP-AES	4.2	4.6	24.3	U	< 0.002	0.032	< 6.3	Yes
V	ICP-MS	0.19	6.61	35.0	B	0.003	0.032	9.4	Yes
W	ICP-AES	170	185	979	U	< 0.08	0.24	< 33.3	Yes
W	ICP-MS	0.55	96.9	513	X	0.04	0.24	16.7	Yes
Y	ICP-AES	4.2	9.5	50.0	J	0.004	0.16	2.5	Yes
Y	ICP-MS	0.18	9.31	49.2		0.004	0.16	2.5	Yes
Zn	ICP-AES	4.2	22.0	116	J	0.01	0.42	2.4	Yes

Table 8.16. (Cont'd)

	Measure Method	Solids MDL µg/g	UDS Average		Data Flag$^{(g)}$	UDS Average	UDS Spec 8 Max	% of Limit	Meet Spec 8? Y/N
			µg/g (wet)	µg/g (dry)		g/100 g waste oxide			
Table TS 8.2 Analytes									
Cl	IC	250	550	2,910	B	0.24	0.33	72.7	Yes
Cyanide	Dist./Color.	1.10	33	174		0.01	1.6	0.6	Yes
NH_3	ISE	70	2	9		0.001	1.6	0.1	Yes
NO_2	IC	500	5,820	30,800			Incl. as NO_3		
NO_3	IC	500	5,020	26,600		4.6	36	12.8	Yes
TIC-F as CO_3 $^{(c)}$	TIC Furnace	600	15,000	79,500		6.4	30	21.3	Yes
TIC-P as CO_3 $^{(c)}$	TIC Hot Pers.	66	55,200	292,000		23.6	30	78.7	Yes
TOC-F $^{(c)}$	TOC Furnace	400	9,240	48,900		3.95	11	35.9	Yes
TOC-P $^{(c)}$	TOC Hot Pers.	180	8,730	46,200		3.74	11	34.0	Yes
Table TS 8.4 Analytes									
Ag	ICP-AES	2.1	2.3	12.2	UX	< 0.001	0.55	< 0.2	Yes
Al	ICP-AES	5.1	21,400	113,000		9.16	14	65.4	Yes
Ba	ICP-AES	0.80	27.5	145.5		0.01	4.5	0.2	Yes
Bi	ICP-AES	8.5	9.3	48.9	U	< 0.004	2.8	< 0.1	Yes
Ca	ICP-AES	21	138	733		0.06	7.1	0.8	Yes
Cd	ICP-AES	1.3	2.4	12.6		0.001	4.5	0.0	Yes
Cr	ICP-AES	1.7	1,700	9,000		$\boxed{\textbf{0.73}}$	0.68	$\boxed{\textbf{107.4}}$	**No**
F $^{(d)}$	IC	250	3,800	20,100		1.63	3.5	46.6	Yes
Fe	ICP-AES	2.1	1,140	6,050		0.49	29	1.7	Yes
K	ICP-AES	170	< 200	< 1,000	JX	< 0.09	1.3	< 6.9	Yes
Mg	ICP-AES	8.5	10.5	58.2	J	0.004	2.1	0.2	Yes
Na	ICP-AES	63	60,400	319,000	X	$\boxed{\textbf{25.8}}$	19	$\boxed{\textbf{135.8}}$	**No**
Ni	ICP-AES	2.5	17.5	92.8		0.01	2.4	0.4	Yes
P	ICP-AES	8.5	358	1,900		0.15	1.7	8.8	Yes
Pb	ICP-AES	8.5	137	728	B	0.06	1.1	5.5	Yes
Pd	ICP-AES	63	69	365	U	< 0.03	0.13	< 23.1	Yes
Pd $^{(e)}$	ICP-MS	0.15	13.3	70.1		0.01	0.13	7.7	Yes
Rh	ICP-AES	25	28	146	U	< 0.01	0.13	< 7.7	Yes
Rh $^{(e)}$	ICP-MS	0.07	5.11	27.0		0.002	0.13	1.5	Yes
Ru	ICP-AES	93	102	537	U	< 0.04	0.35	< 11.4	Yes
Ru	ICP-MS	0.1	15.2	80.2		< 0.007	0.35	2.0	Yes
SO_4 / S $^{(f)}$	IC	500	6,730 / 2,240	35,600 / 11,900		$\boxed{\textbf{0.96}}$	0.65	$\boxed{\textbf{147.7}}$	**No**
Si	ICP-AES	42	64	341	JB	0.03	19	0.2	Yes
Th	ICP-AES	85	93	489	U	< 0.04	5.0	< 0.8	Yes
Th	ICP-MS	0.43	26.4	140		0.01	5.0	0.2	Yes
Ti	ICP-AES	2.1	2.3	12.2	U	< 0.001	1.3	< 0.1	Yes
U	ICP-AES	170	185	979	U	< 0.08	14	< 0.6	Yes
U	KPA/ICP-MS	0.33	58.6	310	X	0.03	14	0.2	Yes
Zr	ICP-AES	4.2	41.8	221		0.02	15	0.1	Yes

Table 8.16. (Cont'd)

	Measure Method	Solids MDL µg/g	UDS Average		Data Flag[g]	UDS Average	UDS Spec 8 Max	% of Limit	Meet Spec 8? Y/N
			µg/g (wet)	µg/g (dry)		g/100 g waste oxide			

NM = not measured

Bolded and Outlined Values exceed Specification 8 criteria.

(a) The total Cs concentration in the solids is estimated based on the assumption the Cs isotopic ratio in the solids is equivalent to the isotopic ratio in the supernatant, and is calculated relative to the ^{137}Cs measured in the solids by GEA.

(b) Pu concentration summed for Pu isotopes measured by ICP-MS.

(c) P = by Hot Persulfate Method; F = by Furnace Method-TIC by difference (TIC=TC-TOC)

(d) Fluoride results may be biased high due to interference from co-eluting anions.

(e) Results are from fusion preparations, other Pd and Rh results from acid digestion preparations.

(f) Sulfur not measured. Sulfate from IC analysis converted to S and compared to specification.

(g) 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 8.1).

Table 8.17. AN-102 Undissolved Solids – Ci / 100 g Waste Oxide

Table		**Solids(a)**				**UDS**	**UDS**		**Meet**
TS 8.3	**Measure**	**MDL/MDA**	**UDS Average**		**Data**	**Average**	**Spec 8 Max**	**% of**	**Spec 8?**
Analytes	**Method**	**µCi/g**	**µCi/g (wet)**	**µCi/g (dry)**	**Flag(d)**	**Ci/100 g waste oxide**		**Limit**	**Y/N**
^{3}H (b)	Rad	3E-03	3E-03	1E-02	U	< 1E-06	6.5E-05	< 1.5	Yes
^{14}C	Rad	3E-04	6.40E-04	3.39E-03	J	2.74E-07	6.5E-06	4.2	Yes
^{60}Co	GEA	3E-03	8.16E-03	4.32E-02		3.49E-06	1.E-02	0.0	Yes
^{90}Sr	Rad	1E+00	1.11E+02	5.85E+02		4.73E-02	1.E+01	0.5	Yes
^{99}Tc	ICP-MS	2E-03	1.36E-02	7.21E-02		5.83E-06	1.5E-02	0.0	Yes
^{125}Sb	GEA	2E-01	2E-01	1E+00	U	< 9E-05	3.2E-02	< 0.3	Yes
^{126}SnSb	GEA	8E-02	8E-02	4E-01	U	< 3E-05	1.5E-04	< 20.0	Yes
^{129}I	ICP-MS	6E-05	1.62E-04	8.55E-04	J	6.91E-08	2.9E-07	23.8	Yes
^{129}I (c)	ICP-MS	2E-04	2E-04	1E-03	U	< 1E-07	2.9E-07	< 34.5	Yes
^{137}Cs	GEA	4E-02	3.44E+00	1.82E+01		1.47E-03	1.5E+00	0.1	Yes
^{152}Eu	GEA	1E-02	1E-02	5E-02	U	< 4E-06	4.8E-04	< 0.8	Yes
^{154}Eu	GEA	9E-03	5.12E-01	2.71E+00		2.19E-04	5.2E-02	0.4	Yes
^{155}Eu	GEA	2E-01	3.20E-01	1.69E+00		1.37E-04	2.9E-02	0.5	Yes
^{233}U	ICP-MS	8E-06	5.60E-05	2.96E-04	J	2.40E-08	9.0E-07	2.7	Yes
^{235}U	ICP-MS	1E-08	9.87E-07	5.22E-06		4.22E-10	2.5E-07	0.2	Yes
^{237}Np	ICP-MS	6E-06	8.51E-04	4.51E-03		3.64E-07	7.4E-05	0.5	Yes
^{238}Pu	Rad	2E-03	1.10E-02	5.79E-02	BJ	4.69E-06	3.5E-04	1.3	Yes
^{239}Pu	ICP-MS	9E-04	5.18E-02	2.74E-01		2.22E-05	3.1E-03	0.7	Yes
^{241}Pu	Rad	1E-01	1E-01	5E-01	U	< 4E-05	2.2E-02	< 0.2	Yes
^{241}Am	GEA	2E-01	3.25E-01	1.72E+00	J	1.39E-04	9.0E-02	0.2	Yes
^{241}Am	Rad	2E-03	3.54E-01	1.87E+00		1.52E-04	9.0E-02	0.2	Yes
$^{243/244}$Cm	Rad	2E-03	1.33E-02	7.03E-02	BJ	5.68E-06	3.0E-03	0.2	Yes

(a) MDL is presented for ICP-MS and MDA is presented for Rad (Radiochemical Analyses)

(b) ^{3}H results possibly biased high by contamination from ^{137}Cs.

(c) Solids prepared for ^{129}I by fusion, other ^{129}I result from acid digestion preparation.

(d) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 8.1).

Decay correction reference dates from March 2001 to May 2001.

9.0 Procedures, Quality Control, and Data Evaluation

A discussion of procedures, data quality, and QC is provided below for each analytical method. Analytical instrument calibration and calibration verification were performed in accordance with the QA 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 the Project 42365 file.

The sample average, MRQ, data flags, QC parameters and QC acceptance criteria are summarized in Table 9.1 through Table 9.12. In some cases, one sample value was reported as <EQL and the duplicate reported with a value >EQL. The reported average is conservatively estimated as the one reported value >EQL.

The QC and 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.

9.1 Inductively Coupled Plasma-Atomic Emission Spectroscopy

Table 8.1, Table 8.6, Table 9.2, and Table 9.3

Both the PNL-ALO-128 acid-digested samples of the supernatant and PNL-ALO-129 acid digested samples of the centrifuged solids 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, process blanks, matrix spikes, LCS or blank spikes, post spikes, serial dilution, calibration verification check standards, interference check standards, and linear range check standards. Matrix spike recovery, LCS (or blank spike) 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.

Except for sodium, duplicate sample analytes of interest with concentrations greater than the EQL for supernatant and wet centrifuged solids demonstrated a RPD within the acceptance criteria of <15% RPD. The sodium results failed to meet the more stringent RPD acceptance criterion of <3.5%. RPD values for sodium ranged from 4.3% to 7.8%.

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

Serial dilution was required for aluminum and sodium for the supernatant analysis and aluminum, chromium, and sodium in the wet centrifuged solids analysis. Except for sodium, the serial dilution results were within the acceptance criteria for percent difference (%D) of <±10%. The sodium in both the supernatant and wet centrifuged solids was so high that sufficient successive serial dilutions were not obtained during the analysis of the samples to calculate the %D. Since the 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.

All matrix-spiked analytes of interest, which were 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 (26%) and calcium (49%) in the wet 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. All analytes of interest (except for Na) found in the post-spiked samples were recovered within acceptance criteria of 75% to 125%.

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 LCS blank spike (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.

9.2 Inductively Coupled Plasma-Mass Spectrometry

Table 8.2, Table 8.7, Table 8.8, Table 9.4, Table 9.5, and Table 9.6

The PNL-ALO-128 and PNL-ALO-129 acid digested samples of the supernatant and wet centrifuged solids, respectively, and the PNL-ALO-114 and PNL-ALO-116 fusion samples of the centrifuged solids were submitted for ICP-MS analysis and analyzed according to procedure PNL-SC-01, *Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*. Except for the MS and LCS, the acid 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, process blanks, MS, LCS or blank spike, post spikes, and calibration verification check standards and blanks. Matrix spike recoveries, LCS recoveries, and precision (based on duplicate analyses) QC criteria are defined by the TS. Radionuclides were not spiked into the LCS or the MS samples. The required isotopic quantity needed would be inappropriate given the large dilutions necessary to perform the analysis. Radionuclides are instead post-spiked into samples. These QC criteria were evaluated in detail and are summarized below.

The analytes of interest were recovered from the LCS within acceptance criteria, except boron (52%), lithium (122%), tantalum (76%), uranium (71%), and tungsten (69%) in the wet centrifuged solids sample. For analytes with concentrations greater than the EQL for supernatant and wet centrifuged solids fractions the duplicates demonstrated RPDs within the acceptance criteria of <15%, except for lithium (194%), boron (60%), and cobalt (17%). 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 wet centrifuged solids samples. Boron, tantalum, and vanadium measured at comparable concentrations in the both the samples and the PBs.

The matrix-spiked analytes of interest were recovered within acceptance criteria except boron (35%), lithium (241%), and tungsten (66%) in the wet centrifuged solids sample. The post-spiked analytes of interest (including radionuclides) were recovered within the MS acceptance criteria except cerium (135%), ^{127}I by fusion (140%), lithium (139%), selenium (135%), and ^{240}Pu (69%).

Many of the QC failures are attributed to lithium, beryllium, boron, arsenic, selenium, and/or vanadium 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 less than optimum reproducibility in this region. Arsenic, selenium, and vanadium may have wide variations in the results due to the extensive chain of isobaric interference corrections. Also, selenium is difficult to ionize; therefore, the sensitivity for selenium is poor.

The Cs isotopic distribution (^{133}Cs, ^{135}Cs, and ^{137}Cs) was determined according to PNL-SC-01, *Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*. The Cs was separated from isobaric interferences using high-performance ion chromatography (HPIC) and the eluant was fed directly to the ICP-MS. Relative abundances of the Cs isotopes were measured and mass concentrations determined relative to the measured ^{133}Cs. QC acceptance criteria were not specified for Cs isotopic distribution. Duplicate samples results agreed within 1%. The ^{137}Cs measured by GEA differed only by 14% with the ICP-MS results.

9.3 U Analysis by KPA

Table 8.2 and Table 9.4

Acid digested (PNL-ALO-128) sample solutions of the supernatant and two preparation blanks from the SAL hot cell were evaporated to dryness 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 (KPA)*. A uranium-specific separation was not performed. The uranium content was well below the MRQ value of 780 µg/mL. The repeatability of duplicate samples was excellent with RPD values of 7%. Uranium was detected in the hot cell blanks, but at a concentration less than 1% of the sample uranium concentration. No QC criteria were specifically called out for U analysis by KPA. An LCS (i.e., a mid-range standard) prepared at the workstation recovered at 100%, within the nominal 80% to 120% acceptance criteria. A matrix spike sample was not analyzed.

9.4 Radiochemical Analyses

Table 8.3, Table 8.9, Table 9.7, and Table 9.8

The supernatant and wet centrifuged solids samples were acid digested according to procedures PNL-ALO-128 and -129, respectively. The supernatant digest was analyzed for gamma emitters, ^{90}Sr, total alpha, $^{239+240}$Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and $^{243+244}$Cm. The centrifuged solids digest was analyzed for gamma emitters, ^{90}Sr, total alpha, $^{239+240}$Pu, ^{241}Pu, ^{238}Pu, ^{241}Am, ^{242}Cm, and $^{243+244}$Cm. An aliquot of supernatant was provided directly (no digestion) for subsequent analysis of the pertechnetate form of ^{99}Tc. An aliquot of the centrifuged solids was leached with DI water and the leachate submitted for ^{3}H analysis. In all cases, the LCS and MS were prepared at the analytical workstation.

9.4.1 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 blank spikes and sample spikes were not prepared, nor required, for this analysis because 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 h. 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 except for ^{155}Eu for the supernatant sample, where the MDL was only slightly above the MRQ value of 9E-02 µCi/mL. All of the hot cell process blanks showed the presence of ^{137}Cs and some of them also showed ^{134}Cs. However, in all cases, the activities in the blanks were negligible with respect to the samples. Sample duplicates showed excellent repeatability with RPD values <5%.

9.4.2 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 total alpha contamination of 0.7%. 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 matrix spike recoveries with ^{239}Pu were 103% and 100%, respectively. No alpha contamination was detected in the laboratory reagent blank. The duplicate sample RPD values are < 3%.

9.4.3 Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417, *Separation of Am and Pu and Actinide Screen by Extraction Chromatography*. The separated fractions were precipitation plated according to PNL-ALO-496, *Precipitation Plating of Actinides for High-Resolution Alpha Spectrometry*, and counted by alpha spectrometry according to PNL-ALO-422, *Solution Analysis: Alpha Spectrometry*. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium chemistry and both these elements 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 a laboratory preparation blank. However, all of the SAL hot cell process blanks indicated significant contamination for some of the isotopes. For the first hot cell batch with samples of the supernatant, the process blank contamination levels were up to 20% of the sample activities for ^{238}Pu and $^{243/244}$Cm. For the second SAL hot cell batch with samples of the wet centrifuged solids, the process blank contamination levels were about 10% of the sample activities for $^{243+244}$Cm. Other alpha emitting isotope contamination levels were not significant in either process blank.

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. All RPD values were within the acceptance criteria of <15% except for ^{238}Pu of the wet centrifuged solids sample, which had a RPD value of 17%.

The ^{241}Pu beta activity was measured in the centrifuged solids sample by placing the precipitation plates from the Pu/AEA measurements in glass scintillation vials, adding scintillation cocktail, and liquid scintillation counting according to procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. A blank filter was used to determine the background counting rate and a blank filter was spiked with a ^{241}Pu standard to determine the beta counting efficiency. Radiochemical yields were taken from the Pu/AEA tracer measurements. Blank spike (LCS) and MS yields were determined in the Pu/AEA measurements and were not separately measured for ^{241}Pu. No ^{241}Pu was detected in the sample or duplicate and the MDL was well below the MRQ value of 1.2 µCi/g. Weak ^{241}Pu activity was seen in the SAL hot cell process blanks, although the level was negligible with respect to the MRQ value. No activity was detected in the laboratory blank.

9.4.4 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 beta counting the samples were gamma counted according to PNL-ALO-450, *Gamma Energy Analysis and Low-Energy Photon Spectrometry* (for ^{85}Sr yield determination and ^{137}Cs impurity assessment). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. However, this correction was negligible with respect to the activity in the samples. No ^{90}Sr was found in the laboratory preparation blank. The SAL hot cell process blanks showed ^{90}Sr activities, although the levels were not significant. RPD values of the duplicates were ≤5%. The LCS and MS recoveries were 88% and 91%, respectively, within the LCS acceptance criteria of 75% to 125% (MS was not required and criteria were not defined).

9.4.5 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 as-received supernatant (no digestion) was taken for analysis according to procedure PNL-ALO-432, *Separation of Technetium by Cation Exchange and Solution Extraction Prior to Measurement by Beta Counting*. This procedure normally requires the use of a sodium dichromate addition to oxidize 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 PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*, to confirm that the beta energy spectra matched that of ^{99}Tc and that no other beta emitters were present.

The LCS (BS) recovery of a ^{99}Tc standard was 92%. However, the MS gave a standard recovery of 248%, which was outside of the 70% to 130% acceptance criteria. The reason for this high recovery is not known. However, the quantity of pertechnetate added to the AN-102 as-received MS 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 solution 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 a third of the total ^{99}Tc (which is in the 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 blank were negligible or non-detectable and well below the requested MRQ value of 0.0015 µCi/mL. Precision could not be assessed on this sample because it was not run in duplicate. However, another sample, AN-102/C-104 blended feed supernatant (01-441), was run in duplicate with the analytical batch and demonstrated a RPD of 1%.

9.4.6 Tritium

The centrifuged solids sample was prepared in duplicate for tritium using a water leach method PNL-ALO-103, *Water Leach of Sludges, Soils and Other Solid Samples*. The leachates prepared from the centrifuged solids sample, were distilled using procedure PNL-ALO-418, *Tritium Determination in Soil and Water Using a Lachat Micro-Dist™ System*. Due to the anticipated high level of ^{137}Cs in this sample, two successive distillations were performed. The tritium was measured by liquid scintillation counting according to procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. In spite of the double distillation, the beta energy spectrum clearly showed the presence of ^{137}Cs contamination. However, using the counts in the tritium energy region of the beta spectrum as an upper limit on the tritium activity resulted in MDLs of 2E-3 to 3E-3 µCi/g, a factor of 5 or more below the requested MRQ value of 1.5E-2 µCi/g. The hot cell process blank showed weak tritium contamination well below the MRQ value. However, the process blank did not show any contamination from high-energy beta emitters. No beta activities were detected in a laboratory blank at the MDA of 2.E-04 µCi/g. The LCS and MS tritium recoveries were excellent at 99% and 100%, respectively.

9.4.7 Carbon-14

The wet centrifuged solids sample was sub-sampled in the SAL and prepared in duplicate for ^{14}C analysis according to procedure PNL-ALO-381, *Direct Determination of TC, TOC, and TIC in Radioactive Sludges, and Liquids by Hot Persulfate Method*. The trap solution generated from PNL-ALO-381 was measured by liquid scintillation counting according to procedure PNL-ALO-474, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*. Beside the sample and duplicate, an LCS (blank spike), MS, MSD, and processing blank were analyzed. The samples, duplicate, LCS, processing blank, and MSs were corrected for the recovery obtained on calibration standards that were processed and analyzed with the samples. The calibration standards demonstrated consistent, but somewhat low, recoveries; i.e., five calibration standards averaged 69% recovery with an RSD of 5%.

The ^{14}C measured in the sample and duplicate was only slightly above the MDA and no ^{14}C was detected above the MDA in the processing blank. The LCS at 98% recovery and the MS at 87% recovery were within the acceptance criteria; however, the MSD demonstrated an excessively high recovery. It was determined that the MSD was contaminated with unknown beta emitter (with a higher energy than ^{14}C). None of the other samples in the processing batch demonstrated any significant contamination from higher energy beta emitters; therefore, the MSD was discarded and not reported.

9.5 Inorganic Anions

Table 8.4, Table 8.10, Table 9.9, and Table 9.10

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 and water leaches of the wet centrifuged solids.

The wet centrifuged solids samples were prepared for analysis by water leach procedure PNL-ALO-103, *Water Leach of Sludges, Soils, and Other Solid Samples*. Once the leachates were prepared an additional 100-fold dilution was required in order to ensure that the anions were measured within the linear calibration range. Column overloading prohibited analysis of the centrifuged solids leachates at dilutions less than 100-fold.

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 as dilutions less than 800-fold.

Quality control for the anions analysis consisted of sample duplicates, process blanks, matrix spikes, blank spikes, post spikes, and calibration verification check standards and blanks. Matrix spike recovery, blank spike 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.

The initial supernatant sample and duplicate analysis did not meet the RPD acceptance criterion of <15%. The poor reproducibility between the initial supernatant sample and duplicate prompted the need for reanalysis. The results appeared to differ by about a factor of two, suggesting the possibility of a dilution error. Therefore, the sample, duplicate, and MS were reanalyzed with the reanalysis providing good QC and reasonable comparison to the original duplicate. The reanalysis results meet all QC criteria and are the reported results. The RPD of the re-prepared supernatant sample was <15%, as was the RPD for the centrifuged solids leachates.

The fluoride and nitrate MSs for the initial supernatant sample produced very high recoveries (>130%). The supernatant MS was re-prepared and reanalyzed, meeting the acceptance criteria of 75% to 125% recovery. The MS was prepared for the centrifuged solid by spiking the centrifuged solid during the water leaching. The spiking level was set assuming that the leachates would require up to a 5-fold additional dilution. However, once the leachates required a 100-fold dilution, the MS was diluted to below the EQL and is not reported. Post spikes on the leachates were performed to assess matrix interferences.

Since the MS for the centrifuged solids sample was lost due to excessive dilution, a post spike was prepared by adding the same spiking solution as used for the MS to the leachate, but after diluting the leachate by 100-fold. The post spike recoveries met the acceptance criteria.

For the supernatant samples, a blank spike was prepared and measured at the same time as the MS. The BS demonstrated recoveries within the acceptance criteria. For the centrifuged solids samples, a BS was prepared, processed, and analyzed like the sample. This BS demonstrated recoveries within the acceptance criteria, except for the oxalate. The oxalate demonstrated 0% recovery (i.e., there was no peak detected for oxalate). The oxalate results on the leachates are provided for information only; oxalate is not an analyte of interest in the centrifuged solids and is reported by another method (organic anions by IC) for the supernatant.

A SAL hot cell blank (i.e., water used in SAL and handled like the sample) was analyzed with the supernatant sample and no anions were detected. However, slight chloride and nitrate contamination was detected in the SAL hot cell leaching process blank (i.e., a water leach with no sample added) provided with the wet centrifuged solids leachates. This blank level, if actually present in the samples, contributed ~500 μg/mL and ~1,300 μg/mL to the chloride and nitrate results, respectively. The leaching process blank chloride contribution to the sample exceeded 5% and therefore the chloride results did not meet the acceptance criteria. Since the nitrate concentration in the sample is very high, the leach process blank nitrate concentration was well within the acceptance criteria.

9.6 TOC/TIC by Hot Persulfate and Furnace

Table 8.4, Table 8.10, Table 9.9, and Table 9.10

The AN-102 as-received supernatant and wet centrifuged solids were analyzed for total organic and inorganic carbon by two different procedures: Procedure PNL-ALO-381, *Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method*, and PNL-ALO-380, *Determination of Carbon in Solids Using the Coulometric Carbon Dioxide Coulometer*.

9.6.1 Hot Persulfate Method (PNL-ALO-381)

The hot persulfate wet oxidation method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. All sample results 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.

Liquid carbon standards were used for the TIC and TOC supernatant analysis. For the wet centrifuged solids analysis, pure chemical solid compounds were used for system calibration standards as well as matrix spikes. The TIC analysis used calcium carbonate and the TOC used α-Glucose. The QC for the method involves, sample duplicates, LCS/blank spike, a MS for the supernatant samples, and a MS for the wet centrifuged solids samples.

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. For the centrifuged solids analysis, three calibration blanks were run at the beginning, middle, and end of the analysis run averaging 11 μgC TIC and 69 μgC TOC. These calibration/system blanks are considered acceptable for the concentration of TIC and TOC measured in the samples. The standard deviation for the TIC and TOC blanks are within the historical pooled standard deviation used to establish the MDL.

All the TIC and TOC RPDs for both the supernatant and centrifuged solids analyses met the acceptance criteria of <15%.

The MS for the supernatant samples demonstrated recoveries well within the acceptance criteria of 75% to 125%. For the wet centrifuged solids samples, the MS recovery was very low for TIC and very high for the TOC spiked sample. An additional MS was prepared and analyzed. The second MS recovered within the acceptance criteria of 75% to 125%; albeit the TIC recovery was still lower than normal and the TOC higher than normal. The total carbon recovery for both the first MS and the second MS is calculated at about 95%, suggesting that the quantity of the sulfuric acid added to the sample for analysis of TIC was insufficient to fully acidify the sample. Based on the MS recoveries, there is a possibility that the reported TIC for the solids is biased low and the TOC is biased high.

The blank spike for the supernatant and wet centrifuged solids analyses were within acceptance criteria of 80% to 120% recovery.

9.6.2 Furnace Oxidation Method (PNL-ALO-380)

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

The calibration and QC standards for TC and TOC analysis are liquid or solid carbon standards or pure chemicals. The TIC analysis used calcium carbonate and the TOC used α-Glucose. The analytical QC include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes.

The coulometer analysis system calibration is checked by analyzing calibration check standards at the beginning, middle, and end of the analysis run. The average recovery from these calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for each of the four analysis days was 92%, 98%, 99%, and 100%.

System blanks were analyzed similarly to the calibration check standards, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The TOC determination produced average blanks of 5 and 15 μC. The TC determination produced average blanks of 4 and 55 μC. The 55 μC blank level is unusually high; however, the reproducibility of the blank was reasonably good (i.e., 49 to 59 μC) and the magnitude of the blank is less than 5% of the sample results (i.e., samples produced 1600 to 3100 μC for the samples sizes analyzed).

The BS/LCS was within acceptance criteria of 80% to 120% required by the client's TS for both the TC and TOC analysis of the supernatant and solids.

The precision between the duplicates (replicates), as demonstrated by the RPD, is acceptable for the supernatant samples. The TC and TOC supernatant RPDs met the acceptance criteria of <15% established by the client's TS. However, the TOC RPD for the AN-102 as-received centrifuged solids was outside the acceptance criteria. The reason for the poor RPD on the wet centrifuged solids may be attributed to heterogeneity of small sub-samples used for the analysis.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes (for supernatant and centrifuged solids) demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery.

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

Table 8.4 presents the TOC and TIC results obtained from the hot persulfate method and the furnace oxidation method for the AN-102 as-received 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 two methods appear to produce comparable results for TC for the supernatant; however, there is about a 15% difference between the TC results for the centrifuged solids, with the furnace producing the lower results. There are *significant* differences between the TIC and TOC results reported by each method. The persulfate results appear to have a higher likelihood of being accurate; it is entirely plausible that there are organic compounds in both the solids and the supernatant that are difficult to oxidize at 750 °C, leading to low TOC and high TIC for the furnace method.

Also, although the OH 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 12,000 μg C/mL (or approximately equivalent to the hot persulfate results for the supernatant).

9.7 Cyanide Analysis

Table 8.10 and Table 9.10

Per the TS, cyanide was only measured on the wet centrifuged solids. The centrifuged solids samples were micro-distilled according to PNL-ALO-287, *Midi and Micro Distillation of Cyanide in Liquid and Solid Samples*, with the addition of sulfamic acid to minimize interference from high nitrates present in the sample. The distillates were analyzed by automated spectrophotometry for cyanide (CN) concentration according to PNL-ALO-289, *Total Cyanide Determination with Spectrophotometry (Manual or Automated) or Argentometric Titration*. In addition to the analysis of the duplicate wet centrifuged solids samples, MS, process blank, and multiple QC check standards and blanks relative to the sample preparation and instrument performance were also analyzed.

The RPD of the duplicate samples was 2%, well within the <20% acceptance criteria. The MS recovery was 147%, which did not meet the acceptance criteria of 75% to 125%. However, the spiking level of the MS is less that 20% of the sample concentration, making the recovery of the spike very difficult to accurately assess (i.e., recovery is based on subtraction of two large numbers to obtain the spike level). The preparation blank met the acceptance criterion of <EQL. The LCS-solid was not within ±10% of the certified value, but it was within the 80.4 to 322 mg/kg vendor performance acceptance limits. Subsequent analysis of the LCS-solid found that the large mesh size of the material was the most likely cause of the variable cyanide recovery. The 98% recovery of an LCS-liquid prepared as an additional QC check did meet the acceptance criteria.

9.8 Mercury Analysis

Table 8.4, Table 8.10, Table 9.9, and Table 9.10

The AN-102 supernatant and centrifuged solids samples and associated batch QC samples were digested for mercury analysis per procedure RPG-CMC-131, *Mercury Digestion*, and analyzed by CVAA spectroscopy for inorganic mercury according to procedure RPG-CMC-201, *Mercury Analysis*. Concentrations of all samples and duplicates measured were at or near the MDL and several times lower than MRQ. The low calibration standard was defined as the MDL for the reported results and assumes non-complex aqueous matrices. Routine precision and bias is typically ± 15% (2-σ) or better for non-complex aqueous samples that are free of interference.

Quality control for the mercury analysis consisted of sample duplicates, preparation blanks, matrix spikes, LCS, and calibration verification check standards and blanks. Matrix spike recovery, LCS/blank recovery, and precision (based on duplicate analyses) QC criteria are defined by the TS.

The matrix spike recovery of the supernatant (33%) and wet centrifuged solids (20%) did not meet the acceptance criteria of 75% to 125%. Based on the excellent precision and recoveries from the other QC samples, the failure of the MS is considered to be from an unknown matrix effect. Further study is needed to understand the reason for these poor recoveries on the AN-102 matrix. Since the concentration of mercury in the supernatant was below the MDL, no RPD was calculated. The RPD of the wet centrifuged solids met the acceptance criteria of <15%. The liquid LCS (National Institute of Standards and Technology Standard Reference Material [NIST SRM]-1641d) for the supernatant and the solids LCS (NIST SRM 2709) for the wet centrifuged solids analyses met the acceptance criteria. Also, the preparation blanks for both the supernatant and wet centrifuged solids analyses met the acceptance criteria of ≤EQL or ≤5% of sample concentration.

9.9 Hydroxide Titration

Table 8.4 and Table 9.9

The AN-102 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 0.25, which equals 4,230 µg/mL, with a 25% 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 (i.e., 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 1.92 molar with a 3% RPD and 1.20 molar with a 10% 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.

9.10 Ammonia Analysis

Table 8.4, Table 8.10, Table 9.9, and Table 9.10

Duplicate samples of the AN-102 supernatant and water leachate of the wet centrifuged solids were diluted with water and acidified with sulfuric acid in the SAL hot cell then taken to the ammonia analytical workstation. The samples were then diluted an additional 10-fold and analyzed for ammonia by ion selective electrode (ISE) procedure RPG-CMC-226, Measurement of Ammonia in Aqueous Solutions. The LCS, MS, MSD, and a blank were also prepared in the SAL hot cell for ammonia analysis. The RPD of the duplicate samples for the supernatant (2%) and the wet centrifuged solids (3%) were within the acceptance criteria. Ammonia was detected in the hot cell blank, but at a concentration below the linear calibration range and at a concentration significantly ≤ EQL or ≤5% of the sample concentration. The LCS recovery of 120% was within the acceptance criteria. Recoveries of the MS (62%) and MSD (66%) did not meet the acceptance criteria of 75% to 125%; however, the MS level is less than 20% of the sample concentration making recovery of the MS very difficult to assess. Since the ammonia analysis uses standard addition for measurement of the ammonia concentration, post spikes were not analyzed.

9.11 Organic Acids

Table 8.4 and Table 9.9

Duplicate samples of the AN-102 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 1000-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 Hanford Tank Waste by Ion Chromatography*. A blank, blank spike, 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. No organic acids were detected in the SAL hot cell blank above the MDL. The blank spike 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 matrix MS and MSD recoveries met acceptance criteria of 75% to 125% for all organic acids measured.

9.12 Chelator Analysis and Degradation Products

Table 8.5 and Table 9.11

The analysis of the AN-102 supernatant samples for chelators and chelator-degradation products was performed on a best-effort basis. The chelators, ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), 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 nominal 5-mL sub-samples of the AN-102 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 analysis according to procedure TP-RPP-WTP-048, *Derivatization GC/FID Analysis of Chelators and Degradation Products*. Adipic acid was added to 2-mL aliquots of each sample (following a 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 the blended AN-102/C-104 samples from ASR 6025 (prepared and analyzed in the sample batch as the AN-102 as-received samples) with citric acid and EDTA. The citric acid, although not specifically requested, mimics the behavior of succinic acid and was used in the MS and MSD. EDTA mimics the behavior of HEDTA, NTA, and IDA and was also used in the MS and MSD.

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. The EQL is set at 10 times the MDL. Only ED3A and IDA were detected above the EQL, and the RPDs for ED3A (91%) and IDA (86%) far exceeded the standard 20% 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 citric acid concentration from this derivatization procedure is nearly a factor of ten lower than the citrate concentration determined from organic acids by IC. The organic carbon concentration (9,500 μg C/mL) determined by summing the individual organic analytes on a μg C/mL basis, is significantly less than the TOC determined by either the furnace oxidation method (18,100 μg C/mL) or the hot-persulfate method (29,300 μg C/mL). This indicates that there could be another organic component present in the AN-102 supernatant, the organic-specific analytical method(s) is (are) biased low, or the TOC methods are biased high, or some combination of these factors. It is probable that the reported chelator concentrations are highly underestimated as the method still needs further development, and as evidenced by the citrate result comparison between the IC method (4,400 μg/mL) and the derivatization method (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 show promise as a direct analysis technique for chelators and degradation products; however, additional development is required for this technique as well.

9.13 Organic Phosphates Analysis

Table 8.5 and Table 9.11

Following extraction of the AN-102 supernatant in the SAL for the organic phosphate analysis, the resulting extracts (both methylene chloride and butanol) were transferred to the Building 329 Facility for analysis. The butanol extracts were processed in case the recoveries demonstrated from the methylene chloride were very poor, but were not analyzed.

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

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 144% and 187%, respectively. The reason for the high recoveries is unknown. 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 9.1 for information.

Table 9.1. Recoveries for D2EHP and DPP Standard

Standard @ 58 μg/mL DPP and 65 μg/mL D2EHP					
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

9.14 Polychlorinated Biphenyls (PCB) Analysis

Table 8.5, Table 8.11, Table 9.11, and Table 9.12

Following extraction of the AN-102 supernatant and wet centrifuged solids in the SAL, the resulting methylene chloride (supernatants) or methylene chloride/acetone (solids) residues were transferred to the 329 Facility for analysis. At the 329 Facility PCB workstation, the residues were exchanged into hexane and concentrated to 2 mL. The residues were split, and 1.0 mL was subjected to sulfuric acid cleanup by agitation of the sample with the acid, and analyzed by GC/ECD per Test Plan TI-RPP-WTP-072, *Analysis of PCBs and Pesticides*. The remaining hexane fraction was tested later to confirm the original results.

Surrogate compounds were spiked at approximately 0.0004 µg/mL in the supernatant samples before extraction. The recoveries ranged from a high of 89.3% for 2,4,5,6-tetrachloroxylene (TCX) to a low of 26.3% for decachlorobiphenyl (DCB). The surrogate recoveries are noticeably higher in the MS and MSD samples, ranging from 80% to 163%, indicating that these may have been doubly spiked with the surrogate compounds. Surrogate recoveries of more than 100% exceeded the calibrated range of the instrument and are therefore estimates.

Surrogates were spiked at approximately 0.016 µg/g in the wet centrifuged solid samples before extraction. The surrogate recoveries in the solids were lower for TCX than to those observed in the supernatants and higher for DCB. Notably higher surrogate recoveries were observed in the solids MSs, indicating that these may have been doubly spiked with the surrogates as well. Several of the surrogate recoveries were lower than the initial target of 70 to 130% with the exception of the MSs mentioned previously. All of the results were verified by an analysis of the remaining 1.0-mL aliquot of sample residue that had been split during sample preparation. No further action was taken.

Samples of both the supernatant and solids were spiked in duplicate. Because limited sample was available, the quantity of supernatant sample used for the MS and MSD was approximately half of that used for unspiked sample analysis. The LCS consisted of PCB-spiked blank water for the liquid extractions and a PCB-spiked drying agent (granulated sodium sulfate) for the solids extractions. Several of the matrix-spike recoveries were lower than the initial target of 70 to 130%. All of the results were verified by analyzing the remaining 1.0-mL aliquot of sample residue that had been split during sample preparation. No further action was taken.

Five PCB congeners were used to quantify each of the Aroclors 1016 and 1260 to conduct performance assessments. The fifth congener used for Aroclor 1260 in the initial and continuing calibration checks demonstrated high recovery by about 5%. The difference can be attributed to the use of a different source of standard Aroclor 1260 material. All of the other calibration verification criteria were met for the remaining 9 PCB congeners and the two surrogate compounds.

Patterns of Aroclors 1254 and 1260 were identified in the wet-centrifuged solid samples. They were present above the MDL only in the wet centrifuged solids duplicate. The levels found were quite low (within three times the MDL), and the ratio of congeners that make up each given Aroclor varied substantially. The congener ratios are often affected as the detection limit is approached. Analysis using gas chromatography/mass spectrometry (GC/MS) for additional definitive confirmation was not performed, but is recommended. The quantity of PCBs found is well below conventional electron impact mass spectrometer detection limits.

Table 9.2. AN-102 As-Received Supernatant – ICP-AES QC Results

Analyte	MRQ µg/mL	MDL µg/mL	01-429 Average µg/mL	(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%*
Test Specification Analytes											
Al	75	5.7	12,300		2	<15	92	NR	NR		5.7
Ba	2.3	0.9	0.9	U	(a)	<15	90	87	97		
Ca	150	24	489		10	<15	97	NR	103		
Cd	7.5	1.4	62		2	<15	95	94	105		
Cr	15	1.9	215		1	<15	96	80	110		
Fe	150	2.4	37		1	<15	99	95	107		
K	75	190	1,980	JX	(a)	<15	**72**	**65**	91		
La	35	4.7	16	J	(a)	<15	91	88		97	
Mg	300	9.4	9.4	U	(a)	<15	99	99	110		
Na	75	40	184,000	X	**4.3 (b)**	<3.5	89	NR	NR		**NM**
Ni	30	2.8	416		1	<15	98	NR	119		
P	600	9.4	1,815		2	<15	94	NR	107		
Pb	300	9.4	186		3	<15	102	93	110		
U	600	190	190	U	(a)	<15	89	86		101	
Other Analytes											
Ag		2.4	2.4	UX	(a)		23	23	98		
As		24	24	U	(a)				112		
B		4.7	80	B	5				102		
Be		0.9	0.9	U	(a)				100		
Bi		9.4	9.4	U	(a)		91	91	99		
Ce		19	19	U	(a)					104	
Co		4.7	5.4	U	(a)				110		
Cu		2.4	23	J	(a)		94	93	99		
Dy		4.7	4.7	U	(a)					99	
Eu		9.4	9.4	U	(a)					107	

Table 9.2. (Cont'd)

Analyte	MRQ µg/mL	MDL µg/mL	01-429 Average µg/mL	(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%*
Li		2.8	2.8	U	(a)				89		
Mn		4.7	17	J	(a)		97	93	106		
Mo		4.7	53	J	(a)				105		
Nd		9.4	32	J	(a)		90	86		96	
Pd		71	71	U	(a)		89	100		82	
Rh		28	28	U	(a)		88	94		95	
Ru		100	100	U	(a)		93	113		111	
Sb		47	47	U	(a)				104		
Se		24	24	U	(a)				104		
Si		47	235	JBX	(a)		108	59	154		
Sn		140	140	U	(a)					87	
Sr		1.4	2.3	J	(a)		90	89	98		
Te		140	140	U	(a)					104	
Th		94	94	U	(a)					103	
Ti		2.4	2.4	U	(a)		91	87	97		
Tl		47	47	U	(a)				99		
V		4.7	4.7	U	(a)				101		
W		190	201		(a)						
Y		4.7	4.8	U	(a)				104		
Zn		4.7	6.5	U	(a)		97	110	109		
Zr		4.7	8.2	J	(a)		93	38	102		

Blank areas indicate QC not required for specified analyte.

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

NR = not recovered (spike concentration <20% of sample concentration); NM = not measured.

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

(b) An RPD of 3.5% was requested for Na analysis.

(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 8.1).

Table 9.3. AN-102 As-Received Wet Centrifuged Solids – ICP-AES QC Results

Analyte	MRQ µg/g	MDL µg/g	01-430 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	2.3	2.3	UX	(a)	<15	**22**	**26**	97		
Al	330	5.5	28,500		4	<15	92	NR	NR		-0.2
Ba	600	0.9	27.5		4	<15	90	94	99		
Bi	6,000	9.2	9.2	U	(a)	<15	90	97	99		
Ca	180	23	421		6	<15	93	**49**	102		
Cd	11	1.4	38		3	<15	92	102	101		
Cr	120	1.8	1,830		3	<15	94	NR	NR		-2.6
Cu	18	2.3	19	J	(a)	<15	92	111	100		
Fe	140	2.3	1,170		3	<15	98	NR	107		
La	60	4.6	32	J	(a)	<15	90	101		95	
Mg	540	9.2	11	J	(a)	<15	96	107	108		
Mn	300	4.6	258		3	<15	95	NR	111		
Na	150	69	167,000	X	**7.8 (b)**	<3.5	94	NR	NR		**NM**
Nd	600	9.2	65	J	(a)	<15	90	102		94	
Ni	160	2.8	258		3	<15	96	105	108		
P	600	9.2	1,410		2	<15	90	102	97		
Pb	600	9.2	245	B	0	<15	90	109	104		
Pd	300	69	69	U	(a)		86	103			
Rh	300	28	28	U	(a)		88	98		86	
Ru	300	100	100	U	(a)		89	107			
Si	3000	46	200	JB	(a)	<15	104	91	119		
Sr	300	1.4	6.2	J	(a)	<15	91	98	101		
Ti	150	2.3	2.3	U	(a)	<15	89	96	98		
Zr	600	4.6	47		(a)	<15	92	102	103		
Zn	6	4.6	22	J	(a)	<15	95	105	105		

Table 9.3. (Cont'd)

Analyte	MRQ µg/g	MDL µg/g	01-430 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-MS Test Specification Analytes Measured by ICP-AES											
As	3	23	23	U	(a)				106		
B	3	4.6	93	B	8				102		
Be	3	0.9	0.9	U	(a)				101		
Ce	6	18	19	J	(a)					97	
Co	3	4.6	4.6	U	(a)				106		
K	1500	190	1,100	JX	(a)		75	81	98		
Li	30	2.8	2.8	U	(a)				98		
Mo	30	4.6	33	J	(a)				102		
Sb	12	46	46	U	(a)				99		
Se	300	23	23	U	(a)				100		
Te	6	140	140	U	(a)					97	
Th	600	92	92	U	(a)					105	
Tl	600	46	46	U	(a)				97		
U	600	190	190	U	(a)		89	99		94	
V	6	4.6	4.6	U	(a)				99		
W	6	190	190	U	(a)						
Y	6	4.6	9.5	J	(a)				102		
Other Analytes											
Dy		4.6	4.6	U	(a)					100	
Eu		9.2	9.2	U	(a)					107	
Sn		140	140	U	(a)					71	

Blank areas indicate QC not required for specified analyte.

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

NR = not recovered (spike concentration <20% of sample concentration); NM = not measured.

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

(b) An RPD of 3.5% was requested for Na analysis.

(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 8.1).

Table 9.4. AN-102 As-Received Supernatant – ICP-MS QC Results

Analyte	MRQ µg/mL	MDL µg/mL	01-429 Average µg/mL	(e) 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%*
Test Specification Analytes									
U(KPA) (a)	7.8E+02	6E-03	1.19E+01		7		100 (b)		
^{133}Cs	1.5E+00	2E-01	9.60E+00		0		94	102	94
Rb	1.0E+00	1E-01	8.74E+00		4	<15	103	117	109
^{241}as Am	--	2E-03	4.90E-02		1		92 (c)		96
	µCi/mL	**µCi/mL**	**µCi/mL**						
^{241}as Am	5.1E-02	5E-03	1.58E-01		1	<15	92 (c)		96
^{241}as Pu	5.1E-02	2E-01	4.99E+00		1	<15	92 (c)		96
^{99}Tc	1.5E-03	5E-03	1.48E-01		0	<15	83 (c)		94
^{237}Np	2.7E-02	3E-06	1.20E-04		2	<15	95 (c, d)	(d)	92 (d)
^{239}Pu	3.0E-02	3E-04	6.47E-03		9		92 (c)		124
^{240}Pu	1.0E-02	7E-04	2.01E-03	J	6		77 (c)		101

Blank areas indicate QC not required for specified analyte.

(a) Uranium result by kinetic phosphorescence analysis (KPA).

(b) No acceptance criteria defined in the TS for U (KPA) LCS/BS.

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

(d) BS acceptance criteria are 90% to 110%; MS and PS acceptance criteria are 75% to 125%.

(e) Data flags: J = estimated value (See Section 8.1).

Table 9.5. AN-102 As-Received Wet Centrifuged Solids - ICP-MS Metals QC Results

Analyte	MRQ µg/g	MDL µg/g	01-430 Average µg/g	(e) 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%*
Test Specification Analytes									
As	3.0E+00	3E+00	6.53E+00	J	(a)	<15	94	102	91
B	3.0E+00	3E+00	3.95E+01	BX	**60**	<15	**52**	**35**	111
Be	3.0E+00	4E-01	1.12E+00	J	(a)	<15	117	99	126
Ce	6.0E+00	4E-01	2.30E+01		4	<15	85	100	**135**
Co	3.0E+00	9E-02	2.36E+00	X	**17**	<15	109	126	89
^{127}I	1.5E+00	5E-01	2.45E+00	J	1		106 (b, d)	(b)	116 (b)
^{127}I (c)	1.5E+00	6E-01	6E-01	U	(a)		101 (b, d)	(b)	**140 (b)**
Li	3.0E+01	4E-01	1.81E+01	J X	**194**	<15	**122**	**241**	**139**
Mo	3.0E+01	3E+00	3.30E+01		2	<15	101	108	75
Pd (c)	3.0E+02	2E-01	1.33E+01		4	<15	113	93	91
Pr	6.0E+00	1E-01	1.76E+01		0	<15	92	97	101
Pt	3.0E+00	5E-02	5E-02	U	(a)		100 (b, d)	(b)	102 (b)
Pt (c)	3.0E+00	8E-02	8E-02	U	(a)		99 (b)	105 (b)	100 (b)
Rb	6.0E+00	2E-01	4.70E+00		3	<15	89	122	90
Rh (c)	3.0E+02	7E-02	5.11E+00		8	<15	105	103	96
Ru (c)	3.0E+02	2E-01	1.52E+01		6	<15	104	101	86
Sb	1.2E+01	5E-02	1.57E-01	J	(a)	<15	99	103	125
Se	3.0E+02	7E+01	7E+01	U	(a)	<15	84		**135**
Ta	6.0E+00	3E-02	2.53E-01	JBX	(a)	<15	**76**	70	77
Te	6.0E+00	6E-01	1.57E+00	J	(a)	<15	101	100	107
Tl	6.0E+02	2E-02	2E-02	U	(a)	<15	92	97	102
Th	6.0E+02	5E-01	2.64E+01		2	<15	81	102	88
U	6.0E+02	4E-01	6.55E+01	X	5		71 (b)	119	96
V	6.0E+00	2E-01	6.61E+00	B	3	<15	104	126	87
W	6.0E+00	6E-01	9.68E+01	X	8	<15	**69**	**66**	111
Y	6.0E+00	2E-01	9.31E+00		6		95	95	83

Blank areas indicate QC not required for specified analyte.

Bold 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 defined in TS for BS, MS, or PS samples.

(c) ^{129}I results from fusion preparation prescribed for I analysis; Pd, Pt, Rh, and Ru results from fusion preparation prescribed for platinum group metals. All other results are from acid digestion preparations.

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

(e) 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 8.1).

Table 9.6. AN-102 As-Received Wet Centrifuged Solids - ICP-MS Radioisotope QC Results

Analyte	MRQ µg/g	MDL µg/g	01-430 Average µg/g	(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%*
Test Specification Analytes									
^{99}Tc	6.0E+00	2E-01	5.81E+00		8	<15	103 (b)		100
^{129}I	3.0E+01	3E-01	9.12E-01	J	(a)	<15	76 (b, c)	(c)	92 (c)
^{129}I (f)	3.0E+01	1E+00	1E+00	U	(a)	<15	88	84	87
^{233}U	6.0E+00	8E-04	5.75E-03	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{234}U	6.0E+00	5E-03	5E-03	U	(a)	<15	(d, e)	(d, e)	(d, e)
^{235}U	6.0E+00	5E-03	4.56E-01		0	<15	(d, e)	(d, e)	(d, e)
^{236}U	6.0E+00	3E-03	1.90E-02	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{238}U	6.0E+00	5E-01	6.49E+01		5	<15	(d)	(d)	(d)
^{237}Np	1.8E+00	1E-02	1.35E+00		4	<15	100 (b, e)	(e)	96 (e)
^{239}Pu	6.0E+00	2E-02	8.95E-01		2	<15	99 (b)		126
^{240}Pu	6.0E+00	3E-03	6.62E-02		1	<15	101 (b)		69
		µCi/g	**µCi/g**						
^{99}Tc		3E-03	9.88E-02		8	<15	103 (b)		100
^{129}I		6E-05	1.61E-04	J	(a)	<15	76 (b, c)	(c)	92 (c)
^{129}I (f)		2E-04	2E-04	U	(a)	<15	88	84	87
^{233}U		8E-06	5.60E-05	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{234}U		3E-05	3E-05	U	(a)	<15	(d, e)	(d, e)	(d, e)
^{235}U		1E-08	9.86E-07		0	<15	(d, e)	(d, e)	(d, e)
^{236}U		2E-07	1.23E-06	J	(a)	<15	(d, e)	(d, e)	(d, e)
^{238}U		2E-07	2.18E-05		5	<15	(d)	(d)	(d)
^{237}Np		7E-06	9.21E-04		4	<15	100 (b, e)	(e)	96 (e)
^{239}Pu		1E-03	5.56E-02		2	<15	99 (b)		126
^{240}Pu		7E-04	1.50E-02		1	<15	101 (b)		**69**

Blank areas indicate QC not required for specified analyte.

Bold 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 defined in TS for BS, MS, or PS.
(d) Individual isotopic QC samples for uranium not prepared, refer to uranium results in Table 9.5.
(e) BS acceptance criterion is 90% to 110%, MS, and PS acceptance criterion is 75% to 125%.
(f) ^{129}I results from fusion preparation prescribed for I analysis. All other results are from acid digestion preparations.
(g) Data flags: U = not detected above reported MDL; J = estimated value (See Section 8.1).

Table 9.7. AN-102 As-Received Supernatant - Radioisotope QC Results

Analyte	MRQ µCi/mL	MDA µCi/mL	01-429 Average µCi/mL	Err %	Data Flag (a)	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.
Acceptance Criteria								*(e)*	*(e)*
Alpha	2.3E-01	6E-03	1.66E-01	4		0	<15	100	103
^{238}Pu	1.0E-02	2E-04	1.65E-03	13	BJ	(b)	<15		
$^{239/240}$Pu	3.0E-02	2E-04	5.90E-03	6		3	<15	107	105
^{241}Am	3.0E-02	3E-04	1.51E-01	2		2	<15	96	97
^{241}Am by GEA		1E-01	1.65E-01	31	J	(b)			
$^{243/244}$Cm	1.5E-02	2E-04	6.71E-03	7	B	6	<15		
^{242}Cm	1.5E-01	2E-04	6.29E-04	22	J	(b)	<15		
Sum of Alpha			1.65E-01	3		2			
^{90}Sr	1.5E-01	7E-01	5.72E+01	3		5	<15	91	88
^{99}Tc(c)	1.5E-03	6E-06	5.55E-02	4	X	1 (d)	<15	92	**248**
^{137}Cs	9.0E+00	5E-02	3.69E+02	2		1	<15		
^{60}Co	1.0E-02	3E-03	8.49E-02	3		2	<15		
^{154}Eu	2.0E-03	1E-02	2.31E-01	3		1	<15		
^{155}Eu	9.0E-02	1E-01	1E-01		U	(b)	<15		

Blank areas indicate QC not required for specified analyte.

Bold 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; X = quality control (QC) deficiency (See Section 8.1).

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

(c) Measured as pertechnetate.

(d) Duplicate not sub-sampled by SAL. RPD determined on replicate of AN-102/C-104 blended feed sample (01-441) prepared and analyzed in the same batch as the AN-102 as-received sample (01-429)

(e) Acceptance criteria listed for total alpha (LCS 70% to 130%, MS 70% to 130%), ^{90}Sr (LCS 75% to 125%) and ^{99}Tc (LCS 80% to 120%, MS 70% to 130%) only.

Table 9.8. AN-102 As-Received Wet Centrifuged Solids - Radioisotope QC Results

Analyte	MRQ µCi/g	MDA µCi/g	01-430 Average µCi/g	Err %	Data Flag (a)	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.
Acceptance Criteria								(c)	(c)
Alpha	1.0E-03	1E-02	5.00E-01	3		3	<15	100	103
^{238}Pu	6.0E-02	2E-03	1.19E-02	11	BJ	(b)	<15		
$^{239/240}$Pu	6.0E+00	2E-03	4.17E-02	6		0	<15	107	105
^{241}Am	1.8E-02	3E-03	4.41E-01	3		1	<15	96	97
^{241}Am by GEA	6.0E+00	2E-01	4.21E-01	12	J	(b)			
$^{243/244}$Cm	1.2E-02	2E-03	1.72E-02	11	BJ	(b)	<15		
^{242}Cm	1.2E-02	2E-03	2E-03		U	(b)	<15		
Sum of Alpha			5.12E-01	4		1			
^{90}Sr	7.0E+01	2E+00	1.44E+02	3		5	<15	91	88
^{134}Cs	9.0E-01	6E-03	6E-03		U	(b)			
^{137}Cs	6.0E-02	4E-02	2.16E+02	2		0	<15		
^{60}Co	1.2E-02	4E-03	5.71E-02	4		2	<15		
^{152}Eu	6.0E-02	1E-02	1E-02		U	(b)	<15		
^{154}Eu	6.0E-02	9E-03	5.12E-01	2		1	<15		
^{155}Eu	6.0E-02	2E-01	3.20E-01	10		(b)	<15		
^{125}Sb	6.0E+00	2E-01	2E-01		U	(b)			
^{126}SnSb	6.0E-02	8E-02	8E-02		U	(b)	<15		
^{241}Pu	1.2E+00	1E-01	1E-01		U	(b)			
^{3}H	1.5E-02	3E-03	3E-03		U	(b)	<15	99	100
^{14}C	1.8E-03	4E-04	6.32E-04		J	(b)	<15	98	87

Blank areas indicate QC not required for specified analyte.

(a) Data flags: U = not detected above reported MDL; J = estimated value; B = analyte in blank above the blank acceptance criteria (See Section 8.1).

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

(c) Acceptance criteria listed for total alpha (LCS 70% to 130%, MS 70% to 130%), ^{90}Sr (LCS 75% to 125%), ^{3}H (LCS 80% to 120%), and ^{14}C (LCS 80% to 120%, MS 75% to 125%) only.

Table 9.9. AN-102 As-Received Supernatant – Other Analysis QC Results

Analyte	MRQ µg/mL	EQL/ MDL(h) µg/mL	01-429 Average µg/mL	(i) 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	2,500	2,500	U	(a)	<15	107	96	
Cl	300	2,500	4,800		2	<15	105	94	
NO_2	3,000	5,000	85,400		1	<15	105	104	
NO_3	3,000	5,000	221,000		2	<15	101	115	
PO_4	2,500	5,000	5,000	U	(a)	<15	104	95	
SO_4	2,500	5,000	16,900		1	<15	102	93	
OH	75,000	170	4,300		**25**	<15	98	97 (c)	
NH_3	140	70	152		2	<15	120	62 (d)	66 (d)
Hg	1.5	0.00014	0.00014	U	(a)	<15	96	**33**	
TOC-F (e)	1,500	60	18,000		2	<15	97	85 (c)	
TIC-F (e)	150	120	23,000		2	<15	99	114	
TOC-P (e)	1,500	129	29,300		1	<15	104	92	
TIC-P (e)	150	49	11,000		1	<15	100	101	
Gluconate (f)	1500	1,000	1,000	U	(a)	NA	106	121 (g)	107 (g)
Glycolate (f)	1500	100	10,500		10	NA	100	105 (g)	105 (g)
Formate (f)	1500	100	8,000		0	NA	108	123 (g)	109 (g)
Oxalate (f)	1500	200	460	X	(a)	NA	78	89 (g)	86 (g)
Citrate (f)	1500	200	4,400		0	NA	89	112 (g)	116 (g)
Other Analytes (IC)									
Br		2,500	2,500	U	(a)	<15	106	96	
C_2O_4	1500	5000	5000	U	(a)	NA	107	102	

NA = not applicable; RPD criteria not defined in TS
Blank areas indicate QC not required for specified analyte.
Bold 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) The OH and TOC-F matrix spikes were performed on sample 01-441, the AN-102/C-104 blended feed composite analyzed with the batch.
(d) The MS level <20% of the sample concentration; MS and MSD results included for information only.
(e) For TOC and TIC: P=by hot persulfate method; F=by furnace method.
(f) No QC acceptance criteria defined in TS
(g) The organic acids MS and MSD were performed on sample 01-441 (the AN-102/C-104 blended feed) analyzed with the analysis batch.
(h) Only values exceeding the EQL are reported for IC anions (fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate and oxalate).
(i) Data flags: U = not detected above reported MDL; X = quality control (QC) deficiency (See Section 8.1).

Table 9.10. AN-102 As-Received Wet Centrifuged Solids - Other Analyses QC Results

Analyte	MRQ µg/g	MDL/ EQL (b) µg/g	01-430 Average µg/g	(j) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Hot Cell Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Matrix Spike Duplicate (MSD) % Rec.	Post Matrix Spike (PS) % Rec.
Acceptance Criteria							*80% - 120%*	*80% - 120%*	*75% - 125%*	*75% - 125%*	*75% - 125%*
Test Specification Analytes											
F (c)	7,500	250	3,800		4	<15	107	104	NR		106
Cl (d)	230	250	3,300	B	8	<15	105	108	NR		102
NO_2	450	500	55,100		6	<15	105	93	NR		104
NO_3	450	500	133,000		5	<15	101	110	NR		110
PO_4	600 (i)	500	8,200		14	<15	104	94	NR		98
SO_4	1200 (i)	500	16,500		9	<15	102	99	NR		99
Cyanide	3.0	0.83	33		2	<15	98, 47 (f)		**147 (g)**		NA
NH_3	60	70	89		5	<15	120		62 (g)	66 (g)	NA
Hg	1.5	0.0007	0.0019		7	<15	96		**20**		NA
TOC-F (h)	60	300	19,600	X	**23**	<15	99		100		N/A
TIC-F (h)	30	200	16,300	X	**27**	<15	104		82		N/A
TOC-P (h)	60	190	25,600		2	<15	101		**160**	119	NA
TIC-P (h)	30	71	17,400		3	<15	100		**44**	75	NA
Other Analytes											
Br		250	250	U	(a)	<15	106	101	NR		104
C_2O_4 (e)		500	28,800		6	<15	107	NP	NR		106

NP = no peak detected; NR = not recovered; MS concentration either below EQL after analysis dilution or sample concentration exceeds MS by 5x.

Blank areas indicate QC not required for specified analyte

Bold 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) Only values exceeding the EQL are reported for IC anions (fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate and oxalate).

(c) 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.

(d) The chloride results are considered qualitative or at best the upper bound for the chloride. The hot-cell blank contribution to the reported results is about 15%.

(e) The oxalate results are from the inorganic anion IC analysis and are considered qualitative. The LCS processed from the hot cell demonstrates no recovery for oxalate; the reason is unknown.

(f) The recovery for liquid LCS/BS was 98%. The recovery for the solid LCS was 94 mg/kg (47%), within the vendor's certified limits of 80.4 to 322 mg/kg.

(g) The MS spike level is <20% of the sample concentration; MS and MSD results included for information only.

(h) For TOC and TIC: P=by hot persulfate method; F=by furnace method.

(i) MRQ for phosphate is as P and for sulfate is as S.

(j) Data flags: U = not detected above reported MDL; B = analyte in blank above the blank acceptance criteria; X = quality control (QC) deficiency (See Section 8.1).

Table 9.11. AN-102 As-Received Supernatant - Other Organic Analyses QC Results

Analyte	CAS #	MRQ µg/mL	MDL µg/mL	01-429 Average µg/mL	$^{(g)}$ Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	MS Duplicate (MSD) % Rec.
Acceptance Criteria								*NA*	NA	NA
Polychlorinated Biphenyl Analytes										
Aroclor 1016/1242	12674-11-2/ 53429-21-9		0.0002	0.0002	U	$^{(a)}$		85 $^{(b)}$	69 $^{(b)}$	50 $^{(b)}$
Aroclor 1221	11104-28-2		0.0002	0.0002	U	$^{(a)}$				
Aroclor 1232	11141-16-5		0.0002	0.0002	U	$^{(a)}$				
Aroclor 1248	12672-29-6		0.0002	0.0002	U	$^{(a)}$				
Aroclor 1254	11097-69-1		0.0002	0.0002	U	$^{(a)}$				
Aroclor 1260	11096-82-5		0.0002	0.0002	U	$^{(a)}$		89	74	59
Total PCB			0.0012	0.0012	U	$^{(a)}$				
TCX (surrogate)	877-09-8			84% $^{(c)}$		13		86%$^{(c)}$	74% $^{(c)}$	80% $^{(c)}$
DCB (surrogate)	2051-24-3			45% $^{(c)}$		84		91%$^{(c)}$	121% $^{(c)}$	126% $^{(c)}$
Organic Phosphate Analytes / Chelators and Degradation Products										
D2EHP $^{(i)}$	298-07-7	1500	0.5	0.5	UX	$^{(a)}$		42	84 $^{(d)}$	107 $^{(d)}$
DPP (surrogate)	838-85-7			166%$^{(c)}$		26		5%$^{(c)}$	73% $^{(c,d)}$	93% $^{(c,d)}$
EDTA	60-00-4	1500	120	420	JX	$^{(a)}$		78	66$^{(d)}$	58$^{(d)}$
HEDTA$^{(h)}$	150-39-0	1500	120	120	UX	$^{(a)}$		38	n/m	
ED3A$^{(h)}$	$^{(e)}$		120	1000	X	91		n/m	n/m	
NTA	139-13-9	1500	100	190	JX	$^{(a)}$		71	n/m	
IDA (as NIDA)$^{(h)}$	142-73-4	1500	120	2500	X	86		n/m	n/m	
Citric acid$^{(f)}$	77-92-9		130	430	J	$^{(a)}$		83	47$^{(d)}$	50$^{(d)}$
Succinic acid	110-15-6		34	36	JX	$^{(a)}$		69	n/m	
AA (surrogate)				86% $^{(c)}$				106% $^{(c)}$	107% $^{(c)}$	103% $^{(c)}$

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) Aroclor 1016 and 1260 were used for the MS, MSD, and LCS.

(c) Value represents percent recovery of surrogate standard.

(d) The D2EHP, EDTA and citric acid MS and MSD were performed on sample 01-441 (the AN-102/C-104 blended feed) analyzed with the analysis batch. The EDTA spike mimics the behavior of HEDTA, NTA, and IDA. The citric acid behavior mimics the behavior of succinic acid.

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

(f) Citric acid is reported by this technique for comparison with the IC results.

(g) Data flags: U = not detected above reported MDL; J = estimated value; X = quality control (QC) deficiency (See Section 8.1).

(h) Concentration results based on EDTA calibration; results are considered qualitative. See Section 9.12.

(i) D2EHP analyzed on a best effort basis; results are considered qualitative. See Section 9.13.

Table 9.12. AN-102 As-Received Wet Centrifuged Solids - Other Organic Analyses QC Results

Analyte	CAS #	MRQ µg/Kg	MDL µg/g	01-430 Average µg/g	(d) Data Flag	RPD %	Target RPD %	Lab Control (LCS/BS) % Rec.	Matrix Spike (MS) % Rec.	Matrix Spike Duplicate (MSD) % Rec.
Acceptance Criteria								*NA*	*NA*	*NA*
Polychlorinated Biphenyl Analytes										
Aroclor 1016/1242	12674-11-2/ 53429-21-9		0.008	0.008	U	(a)		57 (b)	85 (b)	54 (b)
Aroclor 1221	11104-28-2		0.008	0.008	U	(a)				
Aroclor 1232	11141-16-5		0.008	0.008	U	(a)				
Aroclor 1248	12672-29-6		0.008	0.008	U	(a)				
Aroclor 1254	11097-69-1		0.008	0.017		(a)				
Aroclor 1260	11096-82-5		0.008	0.030		(a)		53	80	44
Total PCB			0.048	0.079		(a)				
TCX (surrogate)	877-09-8			48% (c)		56		41% (c)	104% (c)	65% (c)
DCB (surrogate)	2051-24-3			95% (c)		40		66% (c)	160% (c)	127% (c)

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

(b) Aroclor 1016 and 1260 were used for the MS, MSD, and LCS.

(c) Value represents percent recovery of surrogate standard.

(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 8.1).

10.0 References

Blanchard, D. L., D. E. Kurath, B. M. Rapko. 2000. *Small Column Testing of Superlig 639 for Removing* ^{99}*Tc from Hanford Tank Waste Envelope C (Tank 241-AN-107)*, PNWD-3028, Rev. 0, Battelle Pacific Northwest Division, Richland, Washington.

Hay, M. S., M. G. Bronikowski, C. W. Hsu, and T. L. White. 2000. *Chemical Characterization of an Envelope C Sample from Hanford Tank 241-AN-102*, BNF-003-98-0250, Westinghouse Savannah River Company, Aiken, South Carolina.

Lambert, S. L. 1996. *Tank Characterization Report for Double-Shell Tank 241-AN-102*, WHC-SD-WM-ER-545, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Urie, M.W, P.R. Bredt, J.A. Campbell, O.T. Farmer, L.R. Greenwood, L.K. Jagoda, G.M. Mong, A.P. Poloski, L.L. Reed, R.D. Scheele, C.Z. Soderquist, R.G. Swoboda, M.P. Thomas, J.J. Wagner. 2002. *Chemical and Physical Properties Testing of 241-AN-102 Tank Waste Blended with 241-C-104 Wash/Leachate Solutions*, PNWD-3172, Rev. 0, Battelle Pacific Northwest Division, Richland Washington.

Appendix A

- Test Specification TSP-W375-00-00007, *Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate*

- Memorandum "Shipment of AN-102 to Pacific Northwest National Laboratory"

- Chains of Custody

Appendix B

- Test Instruction 41500-009, AN-102 Sample Inspection
- Test Plan TP-41500-005, AN-102 Sample Compositing for Process Testing
- Test Instruction TI-41500-015, AN-102 Integrated Test: Solids Analysis
- Test Instruction TI-RPP-WTP-098, AN-102 Weight Percent Oxides
- Solids XRD Report

Appendix C

- ASR-6011, 6011.01
- ICP-AES Results ASR 6011, 6011.01
- GEA Results ASR 6011, 6011.01

Appendix D

- ASR 6019, 6019.01, 6091.02, 6019.03 and 3 addenda
- ICP-AES Results
- ICP-MS Results
- Radiochemical Results and U KPA Results
- IC Results
- Cyanide Results
- Ammonia Results
- Mercury Results
- Hydroxide Results
- TOC/TIC Results
- Organic Acids Results
- PCB Results
- Organic Phosphate Results
- Chelator Results

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