PNWD-3173
WTP-RPT-020

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

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August 2002

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

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Battelle - Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by test specification TSP-W375-00-00007 and test plan TP-41500-005. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

_____ _____

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

Approved for RPP-WTP Project Use:

_____ _____

G. Todd Wright, Manager Date
Research and Technology

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	Deriv/GC/FID	5.E-01	U	NM		[a]	[a]
ED3A	Deriv/GC/FID	1.00E+03	X	NM		[a]	[a]
EDTA	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	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)

Analyte	Measure Method	Supernatant		Wet Centrifuged Solids		Calculated UDS	
		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	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	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	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)

Analyte	Measure Method	Supernatant		Wet Centrifuged Solids		Calculated UDS	
		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)

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

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

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)
^{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		Wet Centrifuged Solids		Calculated UDS	
		Average µCi/mL	Data Flag [a]	Average µCi/g	Data Flag [a]	µCi/g (wet)	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)]	U, 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.
For the 5% or less entries, the analytes are listed in ascending order (i.e., 5% to 0%).

(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) 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, $^{234/244Cm}$, ^{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.
For 10% or less entries, the analytes are listed in ascending order (i.e., 10% to 0%).

(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.

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).

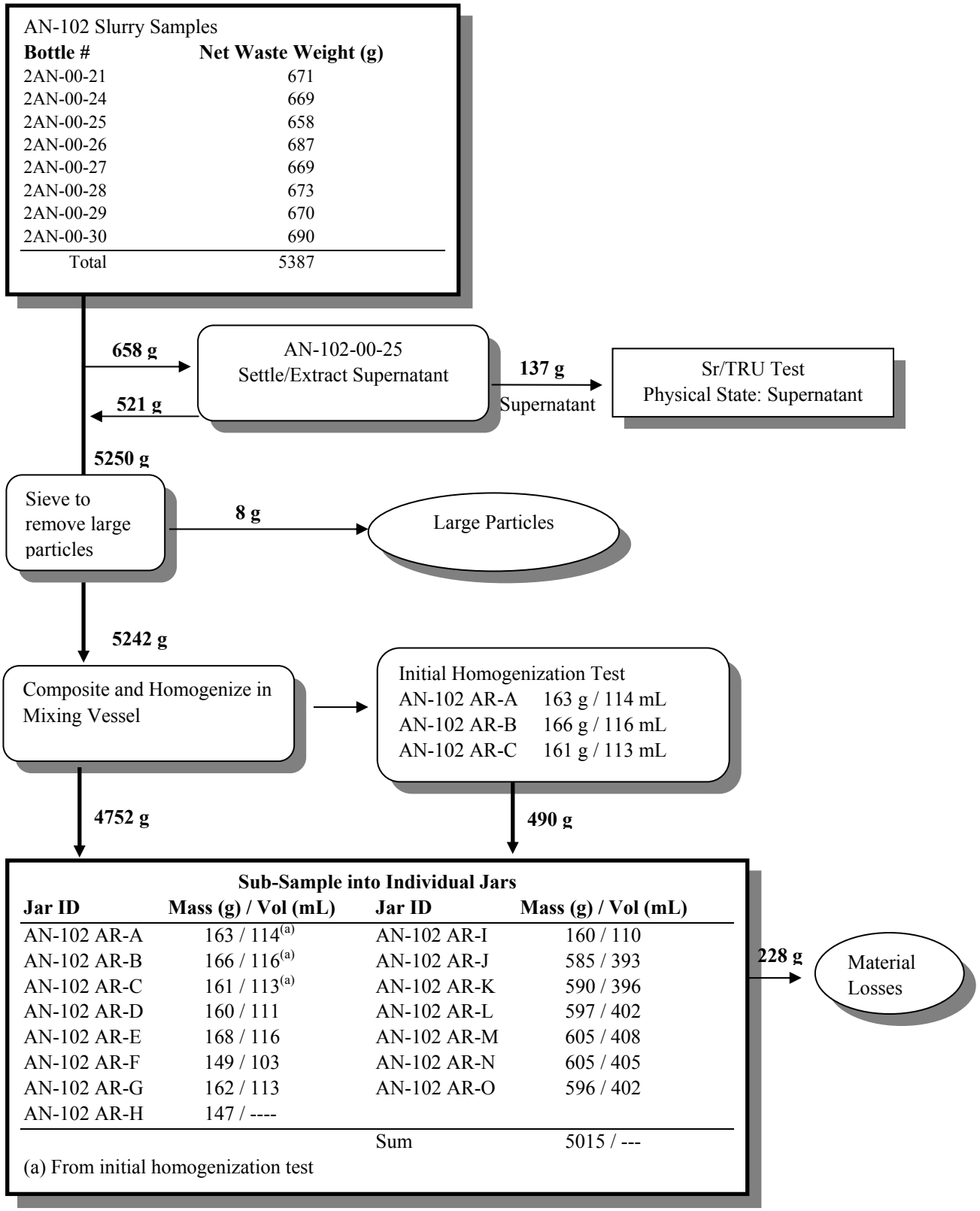


Figure 3.1. Flow Diagram of the AN-102 Tank Slurry Receiving, Homogenizing, and Sub-Sampling



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				Average	Std Dev (1-σ)	RSD
	AN-102-AR-H A	AN-102-AR-H B	AN-102-AR-H C	AN-102-AR-H D			
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 ($wt\%_{sfo}$) 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.

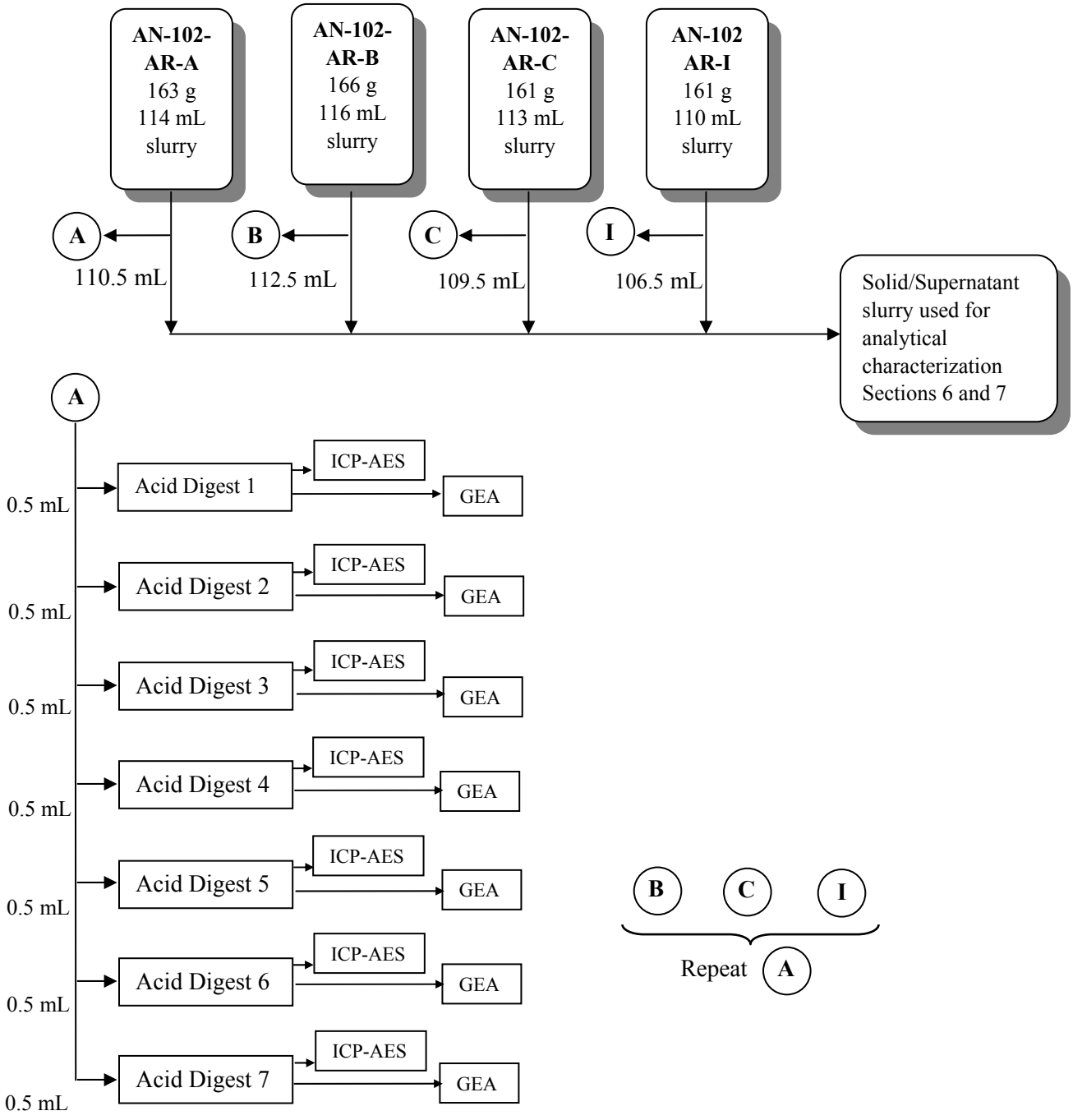


Figure 5.1. Flowchart of EHT Analytical Sub-Sampling and Analysis

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**	**Total Mass**	**Mass used for EHT**	**Mass Lost during Phase Separation**	**Percent Lost during Phase Separation**
RPL Number	**Bottle ID**	**(g)**	**(g)**	**(g)**	**(g)**	**(%)**
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**	**Bottle Gross Mass**		**Sample Mass**	**Total Mass**
RPL Number	**Bottle ID**	**(g)**	**(g)**		**(g)**	**(g)**
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.

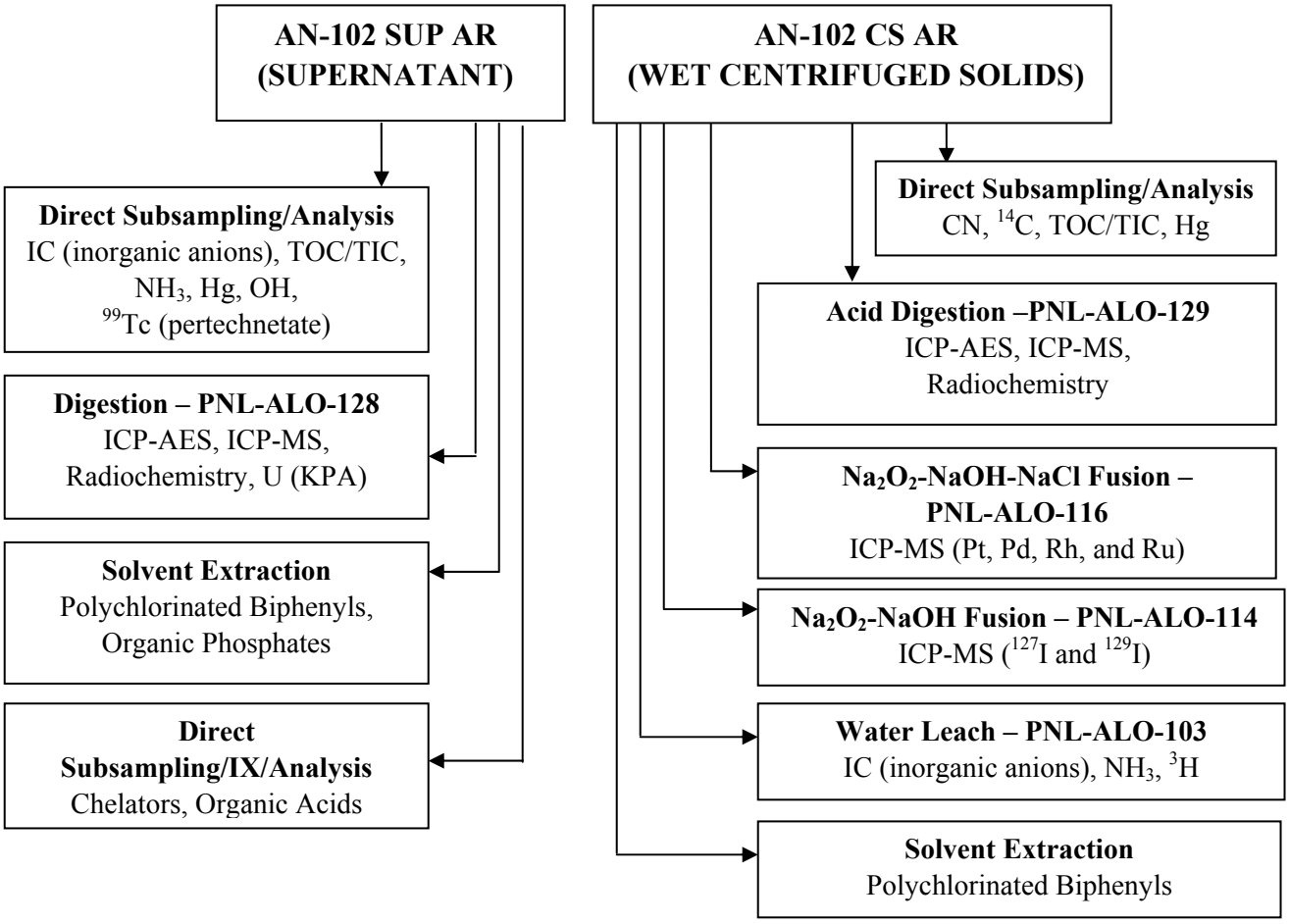


Figure 7.1. Flow Diagram for Analytical Processing of Supernatant and Wet-Centrifuged Solids

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

[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 minimum detectable activity (MDA) is calculated per the QA Plan and is based on the background counting statistics.

Specific QC and QA discussions are given in Section 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 Bechtel.
- 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).

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

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		
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

	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDL	01-429-PB1	± 1SD	Data	MDL	01-429-PB2	± 1SD	Data	MDL	01-429	± 1SD	Data	MDL	01-429 D	± 1SD	Data
Analytes	µg/mL			Flag[d]	µg/mL			Flag[d]	µg/mL			Flag[d]	µg/mL			Flag[d]
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

Analyte	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
	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

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		
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

Analyte	CAS #	MDL µg/mL	Process Blank 01-429-PB µg/mL	Process Blank Data Flag[c]	Sample 01-429 Sample µg/mL	Sample Data Flag[c]	Duplicate 01-429 Dup µg/mL	Duplicate 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	298-07-7	0.5	0.5	U	0.5	U	0.5	U
DPP (surrogate)	838-85-7		3% [a]		144% [a]		187% [a]	
Chelators and Degradation Products								
EDTA	60-00-4	120	120	U	600	JX	2400	JX
HEDTA[d]	150-39-0	120	120	U	120	UX	120	UX
ED3A[d]		120	120	U	1500	X	560	X
NTA	139-13-9	100	100	U	260	JX	110	JX
IDA (as NIDA)[d]	142-73-4	120	120	U	3500	X	1400	X
Citric acid[b]	77-92-9	130	130	U	430	J	130	J
Succinic acid	110-15-6	34	34	U	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	Process Blank Data Flag[c]	Sample 01-429 Sample µg/mL	Sample Data Flag[c]	Duplicate 01-429 Dup µg/mL	Duplicate 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								

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

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-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			
Analytes	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			
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 Flag	MDL	01-430-PB2	± 1SD	(d) Data Flag	MDL	01-430	± 1SD	(d) Data Flag	MDL	01-430 D	± 1SD	(d) Data Flag
Analytes	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) 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			
Analytes	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			
^{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				µCi/g				µCi/g				µCi/g			
^{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

Analyte	Process Blank 1				Process Blank 2				Sample				Duplicate			
	MDA	01-430-PB1	Err[b]	(d) Data Flag	MDA	01-430-PB2	Err[b]	(d) Data Flag	MDA	01-430	Err[b]	(d) Data Flag	MDA	01-430 D	Err[b]	(d) Data Flag
	µCi/g		%		µCi/g		%		µCi/g		%		µCi/g		%	
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

Analyte	Process Blank			Sample			Duplicate		
	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/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 8.11. AN-102 As-Received Wet Centrifuged Solids[a] - Other Organic Analytes Results

Analyte	CAS #	MDL	Process Blank 01-430-PB	Data Flag[c]	Sample 01-430 Sample	Data Flag[c]	Duplicate 01-430 Dup	Data Flag[c]
		µg/g			µg/g		µg/g	
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	<1e+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
^{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

Table 8.13. (Cont'd)

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
^{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).

$$X_o = C * F1 * \left[\frac{1-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * \left(1-W\right)\right)} \right] * F2 \qquad (8.4)$$

$$X_o = U * F1 * \left[\frac{W-T}{\left(W_{ocs} * (1-T)\right) - \left(W_{os} * (1-W)\right)} \right] * F2 \qquad (8.5)$$

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)
F1 = mass conversion factor ($g/10^6 \mu g$)
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)
F2 = 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 µg/g (wet)	UDS Average µg/g (dry)	Data Flag[g]	UDS Average g/100 g waste oxide	UDS Spec 8 Max	% of Limit	Meet Spec 8? Y/N
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 NO3		
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		**0.73**	0.68	**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	**25.8**	19	**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		**0.96**	0.65	**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 µg/g (wet)	UDS Average µg/g (dry)	Data Flag[(g)]	UDS Average g/100 g waste oxide	UDS Spec 8 Max	% of Limit	Meet Spec 8? Y/N

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 TS 8.3 Analytes	Measure Method	Solids[a] MDL/MDA µCi/g	UDS Average µCi/g (wet)	UDS Average µCi/g (dry)	Data Flag[d]	UDS Average Ci/100 g waste oxide	UDS Spec 8 Max Ci/100 g waste oxide	% of Limit	Meet Spec 8? 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.

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 recovery for citric acid was within the typical 80% to 120% recovery acceptance criterion. The NTA and EDTA LCS recoveries of 71% and 78%, respectively, were slightly below the typical lower threshold of 80% recovery. The HEDTA LCS was low at 38% recovery. The MS and MSD results for citric acid were 47% and 50% and for EDTA were 66% and 58%, showing fairly good precision, but a low bias. Confirmation of the identification of the analytes was performed using GC/MS.

The 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 shows promise as a direct analysis technique for chelators and degradation products; however, additional development is required for this techniques 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 and the low recovery of D2EHP in the LCS cannot be confirmed. However, additional testing was conducted to evaluate these poor recoveries. This testing suggest that 1) the low recoveries may be due to ionic strength variations, 2) pH of extraction, and/or 2) stability of the derivative.

Besides the routine QC samples (e.g., MS, MSD, LCS, and process blank), three replicates of a standard containing D2EHP and DPP were derivatized and analyzed like the samples. The concentrations and recoveries are included in Table 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	0.4	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	0.4	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.	Matrix Spike 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	74	80
DCB (surrogate)	2051-24-3			45% (c)		84		91	121	126
Organic Phosphate Analytes / Chelators and Degradation Products										
D2EHP	298-07-7	1500	0.5	0.5	U	(a)		42	84(d)	107(d)
DPP (surrogate)	838-85-7			166% (c)		26		5	73(d)	93(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.

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	104	65
DCB (surrogate)	2051-24-3			95% (c)		40		66	160	127

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

River Protection Project

Waste Treatment Plant

RPP-WTP

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

Test Specification

Revision 0

November 1, 2000

Michael E. Johnson
Pretreatment Technical Manager
CH2MHill Hanford Group

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

Revision Summary

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

Purpose

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

- Preparing a composite sample from multiple tank 241-AN-102 waste samples,
- Verifying the homogeneity of the composite sample,
- Analyzing the composite,
- Separating AN-102 sub-samples for other process verification tests,
- Mixing the AN-102 sample with C-104 wash / leachate solutions to simulate operations in the WTP,
- Analyzing the mixture of AN-102 sample and C-104 wash / leachate solutions,
- Comparing analytical results with the low-activity waste (LAW) and high-level waste (HLW) feed specifications for the waste treatment plant (WTP)
- Reporting analytical results

Background

Previous Tank 241-AN-102 Characterization Information

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

Fiscal Year 2001 Work Scope with AN-102 Sample

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

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

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

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

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

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

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

[4] Characterization, Washing, Leaching, and Filtration of C-104, BNFL-RPT-030, May 2000, Battelle, Richland Washington.
[5] Letter No. 00-OPD-013, "Contract No. DE-AC06-99RL14047 – Modification to Feed Delivery Sequence", dated January 26, 2000 from R. T. French, Manager Office of River Protection to M. P. DeLozier, President CH2M Hill Hanford Group, Inc.

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

DRD Reference

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

Schedule Reference

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

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

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

1.1 Justification

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

1.2 Objectives

The objectives of this task are to:

- Receive and composite sample bottles that contain AN-102 waste,
- Thoroughly mix the composite AN-102 sample,
- Verify sample homogeneity,
- Sub-sample the AN-102 composite sample,
- Analyze the liquid fraction to determine compliance with specification 7 of RFP solicitation DE-RP27-00RV14136 and analyze the solid fraction to determine compliance with specification 8 of RFP solicitation DE-RP27-00RV14136,
- Report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1).
- Provide a summary analytical report in electronic and paper format within 90-days of completing all analyses.
- Separate a sub-sample of AN-102 solids for gas generation, energetics studies, and Sr/TRU reaction mechanism studies
- Mix the AN-102 composite sample with C-104 HLW permeate solutions

1.3 Success Criteria

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

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

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

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

1.4 Quality Assurance

The RPP-WTP *Quality Assurance Program,* BNFL-5193-QAP-01 revision 6, requires the designation of a quality level for all work. The sample analysis results may be used directly for assessing the performance of important to safety equipment. Therefore, the work performed under this test specification is designated as important to safety (quality level QL-1 or QL-2 per the RPP-WTP *Quality Assurance Program,* BNFL-5193-QAP-01 revision 6).

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

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

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

1.5 Test Conditions

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

1.6 Reporting

PNNL personnel are to report all process verification and characterization results in metric units, in accordance with section 6.6.3, *Convention of Units of Measure*, of the DOE-ORP statement of work to CHG.

PNNL personnel are to issue the draft test results to RPP-WTP within 30 calendar days after completing the analyses identified in this test specification. PNNL personnel are to issue a final test report within 30 calendar days after receiving comments on the draft report from RPP-WTP. PNNL personnel shall also report liquid and solid analyses in accordance with *Standard Electronic Format Specification for Tank Waste Characterization Data Loader: Version 3.0*, (HNF-3638 revision 1), to the extent data field information is available, and ensure this information is added to the Tank Waste Information System (TWINS).

Table 1 Liquid Fraction Analyses		
Analyte	Minimum Reportable Quantity	Analysis Method
	µg/ml	
Al	7.5E+01	ICP-AES (AA may be used for Na, K, Cs, Rb or ICP-MS for Cs and Rb)
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Cs	1.5E+00	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Rb	1.0E+00 (if method available)	
S	3.0E+01	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TIC	1.5E+02	Furnace Oxidation
TOC	1.5E+03	
TOC	1.5E+03	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02	Silver catalyze persulfate and furnace oxidation method
Hg^1	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03	
SO_4	2.3E+03	
	µCi/ml (except as noted)	ICP-MS
^{237}Np	2.7E-02	
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	5.1E-02	
^{99}Tc	1.5E-03	
^{99}Tc	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{90}Sr	1.5E-01	Separations / Liquid Scintillation
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}Pu$	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}Cm$	1.5E-02	
^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	1.0E-02	
^{137}Cs	9.0E+00	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
total and free OH	7.5E+04 µg/ml	Titration
Ammonia[1]	1.4E+02 µg/ml	ISE

Table 1 Liquid Fraction Analyses		
Analyte	Minimum Reportable Quantity	Analysis Method
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Organic Analytes	µg/ml	
EDTA	1.5E+03	HPLC / LC-MS (if methods are available)
HEDTA	1.5E+03	
Oxalate	1.5E+03	
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
D2EHPA	1.5E+03	
NTA	1.5E+03	
IDA	1.5E+03	
[1]These analyses are only required for the as-received AN-102 composite sample.		

Table 2. HLW Solids Analyses		
Analyte	Minimum Reportable Quantity	Analysis Method
	µg/gm	
Ag	9.0E+02	ICP-AES
Al	3.3E+02	
Ba	6.0E+02	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
La	6.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Pd	3.0E+02	
Rh	3.0E+02	
Ru	3.0E+02	
S	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Hg^1	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	
CN^1	3.0E+00	CN analysis
$NH_3{}^1$	6.0E+01	ISE
	µg/gm	ICP-MS
As	3.0E+00	
B	3.0E+00	
Be	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Li	3.0E+01	
Mo	3.0E+01	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	
W	6.0E+00	

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Y	6.0E+00	
^{127}I[1]	1.5E+00	
^{129}I[1]	3.0E+01	
^{233}U	6.0E+00	
^{234}U	6.0E+00	
^{235}U	6.0E+00	
^{236}U	6.0E+00	
^{238}U	6.0E+00	
^{237}Np	1.8E+00	
^{99}Tc	6.0E+00	
	µCi/gm	
^{3}H[1]	1.5E-02	Separations / Liquid Scintillation
^{14}C[1]	1.8E-03	Separations / Liquid Scintillation
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}Pu$	6.0E+00	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}Cm$	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
^{125}Sb[1]	6.0E+00	
^{126}Sn[1]	6.0E-02	
^{134}Cs[1]	9.0E-01	
^{135}Cs[1]	5.3E-03	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
^{241}Pu[1]	1.2E+00 µCi/gm	Beta Liquid Scintillation Counting
Physical Property	Expected Range	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Mineral types / structures		X-ray diffraction

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

Table 3. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Duplicate RSD[c]
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-, PO_4^{-3}, SO_4^{-2}	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation colorimetric	80 - 120%	75 - 125%	<15%
Cs[i], Eu[i]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH-	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. Scintillation	80 - 120%	N/A[e]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
^{60}Co[f]	GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[e]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[e]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{125}Sb	GEA	to be obtained		
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A[g]	<15%
^{137}Cs	GEA	NP	N/A[g]	<15%
^{152}Eu[f]	GEA	NP	N/A[g]	<15%
^{154}Eu[f]	GEA	NP	N/A[g]	<15%
^{155}Eu[f]	GEA	NP	N/A[g]	<15%
^{231}Pa	ICP/MS	Developed by Laboratory		
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np[c]	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu[h]	Separation/AEA	NP	N/A[e]	<15%
$^{241}Pu/Am$, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[e]	<15%
^{242}Cm	Separation/AEA	NP	N/A[e]	<15%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[e]	<15%
Total Alpha[h]	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids	Gravimetric	80 - 120%	N/A	<21%

Acronyms:
AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

(a) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

(b) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

(c) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

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

(e) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.

(f) An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

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

(h) The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes that are defined as alpha-emitting TRU (e.g., ^{237}Np, ^{242}Pu, ^{242}Cm, ^{243}Am, and $^{243+244}Cm$) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination.

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

Table 4. Quality Control Parameters for Solids Analysis

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[a]	Spike % Recovery[b]	Duplicate RSD[c]
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, La, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs[e]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{59}Ni[h]	Separation/GEA	NP	N/A[g]	<15%
^{63}Ni[h]	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{60}Co[i]	GEA	NP	N/A[k]	<15%
^{90}Sr[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{90}Y[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{93}Zr[h], [l]	beta-liq scintillation	NP	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m}Sn[i]	Separation/GEA	NP	N/A[g]	<15%
^{125}Sb[m]	GEA	to be obtained		
^{126}Sb[i], [n]	Separation/GEA	NP	N/A[g]	<15%
^{126m}Sb[i], [n]	Separation/GEA	NP	N/A[g]	<15%
^{126}Sn[n]	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A	<15%
^{135}Cs[h]	ICP/MS	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{151}Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{152}Eu[i]	GEA	NP	N/A[k]	<15%
^{154}Eu[i]	GEA	NP	N/A[k]	<15%
^{155}Eu[i]	GEA	NP	N/A[k]	<15%
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu	Separation/AEA	NP	N/A[g]	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[g]	<15%
^{242}Cm	Separation/AEA	NP	N/A[g]	<15%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[g]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids	Gravimetric	80 - 120%	N/A	<21%

Acronyms:
AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

[a] LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

[b] For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

[c] RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

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

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

[f] Not used.

[g] Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.

[h] Radionuclide only required for WAPS justification.

[i] An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

[j] Combined analysis of ^{90}Sr and ^{90}Y.

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

[l] Combined analysis with ^{93m}Nb.

[m] Combined analysis with ^{125m}Te.

[n] Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

Specification 7: Low-Activity Waste Envelopes Definition

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

7.2 Requirements:

7.2.1 References:

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

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

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

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

7.2.2 Envelope Requirements:

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

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

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

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

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

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

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

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

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

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

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

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

(1) 2.6 units;or

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

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

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

(1) 1.15 units; or

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

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

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

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

Notes:
1 Mole of inorganic carbon atoms/mole sodium
2 Mole of organic carbon atoms/mole sodium

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

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

Notes:
[1] The activity limit shall apply to the feed certification date.
[2] TRU is defined as: Alpha-emitting radioncludes with an atomic number greater than 92 with half-life greater than 10 years.

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

Specification 8: High-Level Waste Envelope Definition

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

8.2 Requirements:

8.2.1 References:

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

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

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

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

8.2.2 High-Level Waste Slurry Description and Envelope Requirements:

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

Compositions for Envelope D unwashed solids (Tables TS-8.1, TS-8.2, TS-8.3, and TS-8.4) are defined in terms of elemental or anion concentrations and radionuclide activities per 100 grams equivalent non-volatile waste oxides. The non-volatile waste oxides include sodium oxide and silicon oxide.

The HLW feed components identified in Tables TS-8.1, TS-8.2 and TS-8.3 are waste components important to establishing the waste oxide loading in the HLW glass. Only these components have concentration limits, which will be used to provide the basis for certification that the HLW feed is within specification limits.

The HLW feed components identified in Table TS-8.4 are also important to HLW glass production. The concentrations of these components in the waste are not expected to exceed the maximum values listed in Table TS-8.4. Information on these components will be provided to support product and process qualification but will not be used as a basis for determining if the feed meets specification requirements.

All HLW feed (soluble and insoluble components) will meet the Tank Farm Operations specifications given in OSD-T-151-00007 (except for free hydroxide), the *Tank Waste Remediation System Final Safety Analysis Report* (HNF-SD-WM-SAR-067), and *Technical Safety Requirements* (HNF-SD-WM-TSR-006, Revision 1-D) as applicable. The radiochemical inventory of the waste feed at the time of delivery shall be compared to the specification limits to assess compliance.

Trace quantities of unspecified radionuclides, chemicals, and other impurities may be present in the waste feed. Feed will be delivered by pipeline in batches. Limits apply to the total retrievable contents of waste from a feed tank. Some elements, components, and isotopes are determined by calculation and not analytic measurement.

The HLW feed provided will not contain a visible separate organic layer.

The HLW waste provided will generate gases due to radiolysis including hydrogen and ammonia at a nearly constant rate and nearly uniform composition. The Contractor is responsible for the management of changes in gas release rate and distribution resulting from their waste processing activities.

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

Table TS-8.1 High-Level Waste Feed Unwashed Solids Maximum Non-Volatile Component Composition (grams per 100 grams non-volatile waste oxides)

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
As	0.16	Pu	0.054
B	1.3	Rb	0.19
Be	0.065	Sb	0.84
Ce	0.81	Se	0.52
Co	0.45	Sr	0.52
Cs	0.58	Ta	0.03
Cu	0.48	Tc	0.26
Hg	0.1	Te	0.13
La	2.6	Tl	0.45
Li	0.14	V	0.032
Mn	6.5	W	0.24
Mo	0.65	Y	0.16
Nd	1.7	Zn	0.42
Pr	0.35		

Table TS-8.2 High-Level Waste Feed Unwashed Solids Maximum Volatile Component Composition (grams per 100 grams non-volatile waste oxides)

Volatile Components	Maximum (grams / 100 grams waste oxides)
Cl	0.33
CO_3^{-2}	30
NO_2	36 (total NO_2/NO_3) as NO_3
NO_3	
TOC	11
CN	1.6
NH_3	1.6

Table TS-8.3 High-Level Waste Feed Unwashed Solids Maximum Radionuclide Composition (Curies per 100 grams non-volatile waste oxides)

Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)	Isotope	Maximum (Ci / 100 grams waste oxides)
^{3}H	6.5E-05	^{129}I	2.9E-07	^{237}Np	7.4E-05
^{14}C	6.5E-06	^{137}Cs	1.5E00	^{238}Pu	3.5E-04
^{60}Co	1E-02	^{152}Eu	4.8E-04	^{239}Pu	3.1E-03
^{90}Sr	1E+01	^{154}Eu	5.2E-02	^{241}Pu	2.2E-02
^{99}Tc	1.5E-02	^{155}Eu	2.9E-02	^{241}Am	9.0E-02
^{125}Sb	3.2E-02	^{233}U	9.0E-07	$^{243+244}Cm$	3.0E-03
^{126}Sn	1.5E-04	^{235}U	2.5E-07		

Table TS-8.4 Additional High-Level Waste Feed Unwashed Composition for Non-Volatile Components (grams per 100 grams non-volatile waste oxides)

Non-Volatile Element	Maximum (grams / 100 grams waste oxides)	Non-Volatile Element	Maximum (grams / 100 grams waste oxides)
Ag	0.55	Ni	2.4
Al	14	P	1.7
Ba	4.5	Pb	1.1
Bi	2.8	Pd	0.13
Ca	7.1	Rh	0.13
Cd	4.5	Ru	0.35
Cr	0.68	S	0.65
F	3.5	Si	19
Fe	29	Ti	1.3
K	1.3	U	14
Mg	2.1	Zr	15
Na	19		
Th	5.0		

BLANK

Fluor Hanford
P. O. Box 1000
Richland, WA 99352

FLUOR GLOBAL SERVICES

Memorandum

8G000-CMS-00-028

To:	D. J. Hart	T6-14	Date:	November 2, 2000
From:	C. M. Seidel	G1-32	Telephone:	373-5211
cc:	R. Akita	T6-50	T. R. Pauly	S7-70
	T. A. Brown	T6-14	J. R. Prilucik	T6-12
	R. L. Clawson	H1-11	J. F. Sickels	S7-03
	N. L. Hulse	R2-12	W. I. Winters	T6-07
	R. K. Fuller	T6-12	CMS File/LB	
	R. L. Myers	P7-28		

Subject: SHIPMENT OF AN-102 TO PACIFIC NORTHWEST NATIONAL LABORATORY

References: (1) BNFL-5193-ID-23, "Interface Control Document for Waste Treatability Samples," M. Johnson, June 22, 2000.

(2) Interoffice Memo, N. L. Hulse, CHG, to L. L. Lockrem, FH, and J. F. Sickels, CHG, "Request for Grab Samples from Tank 241-AN-102," 74B10-00-008 R1, dated August 1, 2000

Analytical Services has been instructed to ship twenty-seven grab samples from the August 2000 sampling event to the Pacific Northwest National Laboratory (PNNL). These samples are currently archived at 222-S. The samples are to be packaged in 500mL bottles and transported to PNNL at the 325 Building in the Hanford 300 Area, attention Ron Myers (372-1323). This shipment is to be preformed using the CH2M HILL Hanford Group, Inc. (CHG) Grab sample trucks under a Hanford Site SARP with closure of the public access highway between the Y-Barricade and the Hanford 300 Area. The 222-S Laboratory is directed to ship this material prior to November 15 or as soon as possible following that date. The 222-S Laboratory will contact the PNNL Technical Contact (R. L. Myers) 48 to 24 hours prior to delivery of this material to confirm the 325 Laboratory's ability and readiness to receive the shipment. Mr. R. L. Clawson of Waste Management Federal Services, Inc. or his appointed representative will be the certified shipper for this activity.

The U.S. Department of Energy, Office of River Protection (ORP) has directed CHG to deliver this material to PNNL by November 2000. ORP has instructed PNNL to perform the analytical work defined in the "Regulatory Data Quality Objectives Supporting Tank Waste Remediation System Privatization Project" (PNNL-12040), conduct waste treatment process verification testing, and to analyze crucible melt to demonstrate compliance with Specification 2.

Retrieval of this material from archive and packaging for shipment is to be charged directly to the Tank Characterization Grab sample analysis account at 222-S. Certified shipper support, actual transport and other activities supporting this shipment are to be charged to CACN: 114727. PNNL has requested shipment of the material in reverse order shipping bottle 30 first and bottle 1 last. A table of material to be shipped (Table #1) is attached to this letter. Samples 2AN-00-09, 2AN-00-22, and 2AN-00-23 are to be retained in archive at 222-S to support other CHG Programmatic Analytical needs.

Points of Contact:

Responsibility	Organization	Contact	Phone
Project Lead	AS, Client Services (FH)	C. M. Seidel	373-5211
Tank Coordinator	RPP (CHG) Process Engineering	N. L. Hulse	372-3409
222-S Operations	AS, 222-S Operations (FH)	T. A. Brown	372-0049
222-S Hot Cells	AS, 222-S Hot Cell Operations (FH)	R. Akita	373-2815
Certified Shipper	Transportation Services (WMTS)	R. L. Clawson	372-0653
Transport	Field Sampling (CHG)	J. F. Sickels	373-0259
PNNL Technical Contact	Chemical Separation and Slurry Process Group (PNNL)	R. L. Myers	372-1323

All applicable DOE contractual ES&H Requirements, sample handling, and transportation shall be performed per approved procedures or work packages. All DOE contractual and Contractor facility safety requirements apply to this work scope. No additional safety requirements are imposed by this Letter of Instruction.

mcr

Table #1 Table of Samples to be Shipped to PNNL

Jar/Vial.	Estimated Net Weight grams	Vial Size (mL)	Tank.	Grab.	Laboratory.	Sampling from tank bottom inches	Memo
2AN-00-01	666.9	500	AN-102	1	S00T001626	346.5	Riser 022
2AN-00-02	657.9	500	AN-102	2	S00T001668	346.5	Riser 022
2AN-00-03	662.1	500	AN-102	3	S00T001627	346.5	Riser 022
2AN-00-04	658.0	500	AN-102	4	S00T001649	346.5	Riser 022
2AN-00-05	664.1	500	AN-102	5	S00T001652	280.5	Riser 022
2AN-00-06	661.2	500	AN-102	6	S00T001628	280.5	Riser 022
2AN-00-07	647.5	500	AN-102	7	S00T001669	280.5	Riser 022
2AN-00-08	611.1	500	AN-102	8	S00T001629	280.5	Riser 022
2AN-00-10	659.6	500	AN-102	10	S00T001670	214.5	Riser 022
2AN-00-11	665.2	500	AN-102	11	S00T001631	214.5	Riser 022
2AN--00-12	651.2	500	AN-102	12	S00T001632	214.5	Riser 022
2AN-00-13	662.6	500	AN-102	13	S00T001647	148.5	Riser 022
2AN-00-14	676.8	500	AN-102	14	S00T001648	148.5	Riser 022
2AN-00-15	662.9	500	AN-102	15	S00T001650	148.5	Riser 022
2AN-00-16	668.3	500	AN-102	16	S00T001651	148.5	Riser 022
2AN-00-17	675.2	500	AN-102	17	S00T001653	82.5	Riser 022
2AN-00-18	671.8	500	AN-102	18	S00T001671	82.5	Riser 022
2AN-00-19	672.8	500	AN-102	19	S00T001672	82.5	Riser 022
2AN-00-20	681.0	500	AN-102	20	S00T001673	82.5	Riser 022
2AN-00-21	669.8	500	AN-102	21	S00T001598	76	Riser 022
2AN-00-24	667.6	500	AN-102	24	S00T001602	76	Riser 022
2AN-00-25	658.8	500	AN-102	25	S00T001664	76	Riser 022
2AN-00-26	686.4	500	AN-102	26	S00T001665	76	Riser 022
2AN-00-27	667.6	500	AN-102	27	S00T001666	76	Riser 022
2AN-00-28	676.7	500	AN-102	28	S00T001667	76	Riser 022
2AN-00-29	676.1	500	AN-102	29	S00T001662	76	Riser 022
2AN-00-30	692.2	500	AN-102	30	S00T001663	76	Riser 022

Table #2 The following samples are to remain in archive at 222-S to Support Tank Farms Operations.

Jar/Vial.	Estimated Net Weight grams	Vial Size (mL)	Tank.	Grab.	Laboratory.	Sampling from tank bottom inches	Memo
2AN-00-09	574.2	500	AN-102	9	S00T001630	214.5	Riser 022
2AN-00-22	689.1	500	AN-102	22	S00T001600	76	Riser 022
2AN-00-23	699.9	500	AN-102	23	S00T001601	76	Riser 022

BLANK

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler
2AN-00-30	see original chain-of-custody

(3) Tank	(4) Riser	(5) Cask/Pig/Serial No.
AN-102	022	460.30

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐

11/8/00

Retrieved Partial Sample Stroke Length: _____

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.

S00T001663

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R-T. STEELE	Don Reddoit	11/9/00 1405	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
Don Reddoit	R-T. Steele	11/9/00 1917	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler	(3) Tank	(4) Riser	(5) Cask/Pig Serial No.
2AN-00-29	see original chain-of-custody	AN-102	022	460.29

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

11/8/00

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.

S00T001662

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele	Don Reddart	11/9/00 1405	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
Don Reddart	R T Steele	11/9/00 1917	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler	(3) Tank	(4) Riser	(5) Cask/Pig Serial No.
2AN-00-28	see original chain-of-custody	AN-102	022	460.28

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

11/8/00

(8) Field Comments:

(9) Seal Intact Upon Release?	☒ Yes	☐ No	
(10) Seal Intact Upon Receipt?	☒ Yes	☐ No	
(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b)	☒ Yes	☐ No	

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.
500700 1667

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele R.T. STEELE	V. Matthews V. Matthews	11-9-00 1435	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
V. Matthews V. Matthews	R.T. Steele LT Steele	11/9/00 1917	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler
2AN-00-27	see original chain-of-custody

(3) Tank	(4) Riser	(5) Cask/Pig Serial No.
AN-102	022	460.27

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount: N/A per 11/8/00		
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.
500700 1666

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele R.T. STEELE	V. Matthews V. Matthews	11-9-00 1435	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
V. Matthews V. Matthews	R.T. Steele RT Steele	11/9/00 1917	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler	(3) Tank	(4) Riser	(5) Cask/Pig Serial No.
2AN-00-25	see original chain-of-custody	AN-102	022	460.25

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

RDE 11/8/00

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.
S00T001664

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele	V. Matthews	2010 11-7-00	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
V. Matthews	R.T. Steele	11/9/00 2230 hrs	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number: 2AN-00-26

(2) Supervisor/Sampler: see original chain-of-custody

(3) Tank: AN-102

(4) Riser: 022

(5) Cask/Pig Serial No.: 460-26

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☒
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
Retrieved Partial Sample Stroke Length		

11/8/00

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.

S00T001665

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele	V. Matthews	2010 11-9-00	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
V. Matthews	R.T. Steele	11-9-00 2130	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number	(2) Supervisor/Sampler
2AN-00-24	see original chain-of-custody

(3) Tank	(4) Riser	(5) Cask/Pig Serial No.
AN-102	022	460.24

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration	11/8/00	
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.
S00T001602

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
R.T. Steele R.T. Steele	Don Reddort Don Reddort	11/9/00 2025	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
Don Reddort Don Reddort	R.T. Steele R.T. Steele	11/9/00 2140	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

CHAIN-OF-CUSTODY RECORD FOR CPO

(1) Sample Number: 2AN-00-21

(2) Supervisor/Sampler: see original chain-of-custody

(3) Tank: AN-102

(4) Riser: 022

(5) Cask/Pig Serial No.: 460.23

(6) Shipment Description:

A. Work Package Number: N/A

B. Cask/Pig Seal Number: N/A

C. Date Sample Collected: N/A

D. Time Sample Collected: N/A

(7) Sampling Data

	Y	N
- Lithium Bromide	☐	☐
Amount	N/A	
Concentration		
- X-Ray	☐	☐
- Partial Sample	☐	☐
- Retrieved Partial Sample Stroke Length		

RTSteele 11/8/00

(8) Field Comments:

(9) Seal Intact Upon Release? ☒ Yes ☐ No

(10) Seal Intact Upon Receipt? ☒ Yes ☐ No

(11) Seal Number AND Cask/Pig SERIAL Number consistent with this record? (Block 5 & 6b) ☒ Yes ☐ No

(12) Laboratory Comments:

Field sampling information is provided on original chain-of-custody included with shipping paperwork.

222-S Laboratory I.D.

S00T001598

(13) Relinquished By (Sign and PRINT)	(14) Received By (Sign and PRINT)	(15) Date/Time	(16) Receiver Comments
RTSteele R.T. Steele	Don Reddart Don Reddart	11/6/00 2025	
(17) Relinquished By (Sign and PRINT)	(18) Received By (Sign and PRINT)	(19) Date/Time	(20) Receiver Comments
Don Reddart Don Reddart	Rick Steele R.T. Steele	11/6/00 2240	
(21) Relinquished By (Sign and PRINT)	(22) Received By (Sign and PRINT)	(23) Date/Time	(24) Receiver Comments
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments

DISTRIBUTION: Original - Laboratory Copy - Sampling Operations

BC-6001-326 (07/97)

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

REFERENCE COPY

PNNL Test Instruction

Document No.: TP-41500-009
Rev. No.: 0

Title: AN-102 Sample Inspection

Work Location:
HLRF

Page 1 of 7

Author: Paul Bredt

Use Category Identification: Reference

Effective Date: Upon final signature
Supersedes Date: New

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

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

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

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

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

Approval signature Date

Author Paul Bredt 12/4/00

Technical Reviewer [signature] 12/4/00

Applicability

This test instruction describes work to be performed under Project 41500 Tasks 2.1, 2.6 and 9.0. Samples of actual Hanford waste from Tank AN-102 were received in the 325 HLRF for testing. This test instruction provides a method for documenting the condition of the as-received samples in accordance with Technical Procedure 41500-004, "Sample Compositing". This document is a mechanism for the cognizant scientist to communicate to technical staff and the client specifics on procedure implementation. Therefore, this Test Plan qualifies as a test instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction a cognizant scientist.

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

Justification/Test Objectives/Success Criteria

The initial objective of this test instruction is to document to condition of the samples shipped from the Hanford 222-S laboratory to the 325 HLRF. Information to be documented includes the following:

1) Jar numbers
2) Condition of the jars and lids
3) Jar masses
4) Appearance of the solids (color, texture, gel, and/or other observations)
5) Appearance of the liquids (color, turbidity, and/or other observations)
6) Inspection for a separable organic layer

The second objective of test instruction is to obtain a subsample of AN-102 supernatant for Sr/TRU removal under task 2.6 . This supernatant will be decanted from one or more jars as needed to collect the required sample volume. Any remaining settled solids must be left with at least 10 ml of standing liquid to prevent drying of the solids. This supernatant will then be diluted to ~5.3M [Na] using 0.01M NaOH.

Quality Control

This work is to be conducted to the quality requirements in "Conducting Analytical Work in Support of Regulatory Programs." This quality program can be found on the PNNL internal web site at http://quality.pnl.gov/Guidance/QualityAssurancePlanning.

Changes to the test instructions can be made only by cognizant scientist and will be documented by crossing out the original information with a single line, and recording, initialing, and dating the changes.

The results of all measurements will be recorded in an LRB, test instruction, or bench sheet. Copies of the completed test instructions, bench sheets, and LRBs will be transferred to the project file upon completion of the project.

Spill Mitigation

When working with liquids and slurries, there is a risk of sample loss through inadvertent spills. During hot cell work, spills primarily result from dropped or tipped sample bottles, broken glassware, and the failure of transfer equipment. In most cases, spills can be controlled and material losses minimized through the use of secondary containment and other good laboratory practices. The cognizant scientist is responsible for working with hot cell staff to reduce the potential and programmatic impact of spills. Specific examples include:

1) Bottle holders to stabilize jars during sample transfers
2) Catch pans below homogenization vessels and primary sample containers
3) Safety coated jars and bottles (when this does not interfere with analytical requirements)
4) Plastic centrifuge liners

If a spill does occur, every practical effort will be made to recover as much of the sample as possible.

M&TE List

AN-102
For Sr-TRU Aliquot

Balance: Calibration ID 362-06-01-049 — 388-06-01-020

Expiration Date 8-2001 — 8/2001

Balance Location HLRF-"B" cell — HLRF - "C" Cell

Record daily balance check information below:

Date	Actual (g)	Measured (g)	Tolerance (g)	Acceptable (Y/N)	Balance Location
12/4/00	200 g	199.999 g	±.02	y	"C" cell
12/5/00	1000.01	999.99	±.02	y	"B" cell

Work Instructions

1) The following table contains a list of samples received from the 222-S laboratory. Video tape each of the bottles and complete the Table.
Tape Label: AN-102 Sample Inspection 12/4/00

** Jar ID	222-S ID	Gross Mass (g)	Bottle and Lid in good Condition (a)?	Organic Layer? (b)	* Appearance of Solids	* Appearance of Liquid	Order Viewed
2AN-00-01	S00T001626	964.64	Yes	?	None observed (light Brown Small Layer Likely)	Very Dark ~500 mL Dark Brown/Black.	5)
2AN-00-02	S00T001668	954.25	Yes	?	None observed (Small Layer Likely)	~ 500 mL Mostly All Dark Brown/Black	4)
2AN-00-03	S00T001627	960.08	Yes	?	None observed (Small Layer Likely)	~500mL Very dark Solution	12)
2AN-00-04	S00T001649	955.51	Yes	?	None observed (Small Layer Likely)	↓	13)
2AN-00-05	S00T001652	962.00	Yes	?	None observed (Small layer Likely)	↓	24)
2AN-00-06	S00T001628	958.80	Yes	?	None observed (Small Layer Likely)	~500mL Very Dark Solution	21)
2AN-00-07	S00T001669	945.01	Yes	?	None observed (Small Layer Likely)	~500mL Very Dark Solution	6)
2AN-00-08	S00T001629	909.01	Yes	?	None observed (Small Layer Likely)	<500mL ↓	9)
2AN-00-10	S00T001670	957.27	Yes	?	None observed (Small Layer Likely)	~500mL ↓	8)
2AN-00-11	S00T001631	963.40	Yes	?	Little Layer light Brown ppt	~500mL Very Dark Solution	17)
2AN--00-12	S00T001632	948.76	Yes	?	None observed (Small Layer likely)	~500mL ↓	18)
2AN-00-13	S00T001647	960.82	Yes	?	↓ ↓	↓ ↓	19)

* Very Dark supernate makes color differation and/or possible organic Layer virtually impossible to appraise

** - JAR IO's were engraved on Bottles

12/4/00 rgs

10) At this point, TAPE RAN thru Lunch ~45min.

Jar ID	222-S ID	Gross Mass (g)	Bottle and Lid in good Condition (a)?	Organic Layer? (b)	Appearance of Solids *	Appearance of Liquid *	Order Viewed
2AN-00-14	S00T001648	974.32	Yes	?	None observed (Possible small layer)	~500 mL Very Dark. Solution	10)
2AN-00-15	S00T001650	957.99	Yes	?	None observed (possibly Very small Layer)	~500 mL Very Dark	27)
2AN-00-16	S00T001651	966.65	Yes	?	None observed (possibly Very small layer)	~500 mL Very Dark	25
2AN-00-17	S00T001653	972.04	Yes	?	None observed (possible very Small Layer)	~500mL Very Dark	7)
2AN-00-18	S00T001671	972.00	Yes	?	↓ ↓	↓	26)
2AN-00-19	S00T001672	970.39	yes	?	Small Layer of Light Brown ppt	~500mL Very Dark	22)
2AN-00-20	S00T001673	978.66	Yes	?	None observed (Possibly a Small Layer)	~500mL Very Dark	15)
2AN-00-21	S00T001598	967.05	Yes	?	~ 100 mL of Light Brown ppt	Dark Brown/Black ~400 mL	3)
2AN-00-24	S00T001602	964.41	Yes	?	~275mL of Lite Brown ppt, some Cracking of ppt	Dark Brown/Black Solution ~225mL	23)
3) 2AN-00-25	S00T001664	817.29 + 137.02 = 954.31	Yes	?	~100 mL of Light Brown ppt	Dark Brown/Black ~400mL	11)
2AN-00-26	S00T001665	983.90	Yes	?	~300 mL of light Brown ppt	~200mL Dark Brown/Black solution	20)
2AN-00-27	S00T001666	965.42	Yes	?	Light ~200mL Brown/Black Solids	Dark Brown/Black ~300	1)
2AN-00-28	S00T001667	973.15	Yes	?	~200mL of Light Brown Solids	" ↓	2)
2AN-00-29	S00T001662	973.97	Yes	?	~300mL of Light Brown ppt	~200mL Dark Brown/Black Solution	6)

973.97 rgs 12/5/00

* Dark Supernate makes Actual color difficult, possible organic Layer virtually impossible to Appraise. 12/4/00 rgs

3) Removed 137.02g upfront for AN-102 Sr-TRU.

Jar ID	222-S ID	Gross Mass (g)	Bottle and Lid in good Condition (a)?	Organic Layer? (b)	Appearance of Solids	Appearance of Liquid
2AN-00-30	S00T001663	989.07	Y	?	~250 mL of Light Brown	Dark Brown/Black ~250

14)

(a) If bottle and lid are not in good condition, describe the condition below including sample ID. Replace lids as needed, and transfer material to new bottles if needed. Label any new lids or bottles with the old sample number and document the new tare for the bottle.

(b) If organic layer is found, describe below. Include depth of layer, appearance, and bottle ID

Comments and Additional Observations (add additional pages if necessary):

2) Decant 137 g (±0.5) of supernatant into a 250 ml glass jar of known mass from one or more of the AN-102 samples already inspected starting with jar 2AN-00-25. Consult the cognizant scientist on which jars to use if there is insufficient material in 2AN-00-25. Leave at least 10 ml of standing liquid on any remaining settled solids. Record the mass of material collected below. outside Cell Balance # 1113120467 Cal Date 2/2001

AN-102 SR/TRU – Bottle Tare = 219.55g in "C" cell Bal # 388-06-01-020 Cal. Expires: 8/2001

Total 356.572 g
Tare 219.550 g
Liquid 137.022 g

3) Record which bottles were used, and how much material was decanted in the following table. Removed 137.02g of 2AN-00-025 817.29 + 137.02 g = 954.31 (rgs)

Jar ID	Initial Mass (g)	Final Mass (g)	Net Removed (g)
2AN-00-25	954.31	817.29	137.022

4) Weigh a stir bar.

Stir bar 3.807 g → Stir Bar [illegible] g

Balance # 360-06-01-035 Cal Date 2/2001

5) Add the stir bar to "**AN-102 SR/TRU**".

6) Obtain at least 75 g of 0.01M NaOH from Rich Hallen. Record any data on the material label.

Label: See Attached 7a page on prep of 0.01m NaOH rgs 12/4/00

7) While stirring, add 75 (±0.5) grams of 0.01M NaOH to sample "**AN-102 SR/TRU**". Record the weight before and after caustic addition. Record any observation on dilution (are solids formed?). SEE AN-102 Sample Inspection Tape 12/4/00

Due to end of tape, I → inserted stirring video after Jar 14 inspection.

Stir bar ↓ AN-102 SR/TRU

356.572 − 137.022 + 3.807 = rgs 12/5/00

Final	434.41 g	← off Scale - Balance only weighs to 400+g, therefore weighed on "B" cell Balance on 12/5/00 rgs
Initial	360.379 g	
0.01 M NaOH	74.03 g	

Observation on dilution: None – except it went from Dark Brown to a Little lighter (See video starting after 2AN-00-14 inspection) No ppt formed.

8) Replace cap tightly to keep "**AN-102 SR/TRU**" from picking up CO_2 from the air.

[signature] 12/5/00

PSL 1425

Balance ID# 390-06-01-006 390-06-01-013
Calibration Date 2/2001 2/2001

Prep of 75 mL of 0.01M NaOH

Target Concentration of NaOH 0.01 M

Tare a 100 mL volumetric flask + Stopper 65.3515 grams Lot# Aldrich 04608MY

In a 100 mL volumetric add 1.0142 mL of 0.986 M NaOH
set 1.01 mL = 66.5993 grams

Dilute to mark and reweigh 164.9064 grams
164.9064

calculate density 0.995 ⇒ 1.00 g/mL

tare 100 mL storage bottle 160.34 grams
transfer 75 mL to a bottle and label: 0.01M NaOH
bottle plus NaOH 235.34 grams
weight of 0.01M NaOH 75.00 grams

0.01 M NaOH for AN-102 SR/TRU PREP

Prepared by: D D Heller
Date: 12/04/2000

attachment to Test Instruction 4500-009 PRB 2/20/01

REFERENCE COPY

PNNL Test Plan

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

Title: AN-102 Sample Compositing for Process Testing

Work Location: HLRF

Page 1 of 19

Author: Paul Bredt

Effective Date: Upon final signature
Supersedes Date: New

Use Category Identification: Reference

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

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

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

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

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

Approval	Signature	Date
Author	Paul Bredt	12/7/00
Technical Reviewer	Barry J. Kunath	12/7/00
SFO Manager	Richard J. Steele	12/7/00
Project Manager	D E Kurath	12/7/00
Quality Engineer	Talbert Almeida	12/7/00
Client	Michael E Johnson	12/7/00

Applicability

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

This Test Plan provides details regarding the implementation of Test Plan 41500-004, "Sample Compositing". Client expectations for successful achievement of project data needs have already been established via Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification". This Test Plan is a mechanism for the cognizant scientist to communicate to technical staff and the client specifics on Test Plan 41500-004 implementation. Therefore, this Test Plan qualifies as a Test Instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction of a cognizant scientist.

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

Justification/Test Objectives/Success Criteria

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

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

Quality Control

This work is to be conducted under the quality requirements of PNNL's Standards-Based Management System (SBMS). Changes to the test instructions can be made only by cognizant scientist and will be documented by crossing out the original information with a

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

Spill Mitigation

When working with liquids and slurries, there is a risk of sample loss through inadvertent spills. During hot cell work, spills primarily result from dropped or tipped sample bottles, broken glassware, and the failure of transfer equipment. In most cases, spills can be controlled and material losses minimized through the use of secondary containment and other good laboratory practices. The cognizant scientist is responsible for working with hot cell staff to reduce the potential and programmatic impact of spills. Specific examples include:

1) Bottle holders to stabilize jars during sample transfers
2) Catch pans below homogenization vessels and primary sample containers
3) Safety coated jars and bottles (when this does not interfere with analytical requirements)
4) Plastic centrifuge liners

If a spill does occur, every practical effort will be made to recover as much of the sample as possible.

General Requirements

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

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

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

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

M&TE List:

Balance 1:	Calibration ID 388-06-01-020	
	Calibration Expiration 8/01	Location "C" cell

Balance 2: (if needed)	Calibration ID 362-06-01-051	
	Calibration Expiration 8/2001	Location "B" Cell

Thermocouple:	Calibration ID 7971 Type K	
	Calibration Expiration 5/01	Location C-Cell

Digital Thermometer:	Calibration ID PNL000273	
	Calibration Expiration 7-2001	Location 601

Record daily balance check information below:

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

Work Instructions

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

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

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

> Note:
> While 1/8 inch is a fairly course mesh, samples with a high solids content may have a tendency to pile on the mesh. If this happens, rinse the material through the screens using the low solids content samples or using standing supernatant in the mixing vessel.

4) Transfer all material from the jars to the 7L mixing vessel using the 1/8" screen to remove the large particle. If necessary, use supernatant from the jars or mixing vessel to rinse the solids in the jars or on the screen into the kettle. Record on a separate piece of paper from which sample jars any ≥1/8 inch particles are observed to be removed. Attach these observations to this test plan.

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

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

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

10/12/01
AN-102 >1/8

PRB

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

Large particles retained in JAR 30 as received from BNFL
rgs

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

AN-102 >1/8

Lynette J. & Ralph did not record wet wt
rgs

Total	_____	g
Tare	_____	g
Dry Solids	_____	g

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

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

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

12) If any particles are retained on the screen, then return them to the stainless steel pan and repeat steps 9 and 11 until all particles pass into the composite, then remove the screen.

Forceps cleaned with DI H_2O & used to remove chunks to > 1/8" bottle

Table 1. AN-102 Sample Information

order	Sample ID	Full Mass (g)	Empty Mass (g)	Mass Transferred (g)
①	2AN-00-21	967.05	295.88	671.17
3	2AN-00-24	964.41	295.10	669.31
5	2AN-00-25	* ~~954.37~~ 817.29	295.95	521.34*
8	2AN-00-26	983.90	297.28	686.62
②	2AN-00-27	965.42	296.58	~~668.84~~
4	2AN-00-28	973.15	300.16	672.99
6	2AN-00-29	973.97	303.92	670.05
7	2AN-00-30	989.07	298.79	690.28
⊁	2AN-00-30	Reused for	>1/8" particles	

12/21/00
Jar 304 Dried solids = 306.61
Empty Tare Wt: 298.79
Net: 7.82g

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

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

13) The goal of this step is to homogenize the sample using as little force as possible. Stir the sample by slowly increasing the motor speed until the solids are mobilized. Stir for a minimum of one hour. Record time, date, sample temperature to ±1.1°C and impeller speed (RPM or controller setting).

Time 9:35 PM Date 12/21/00 Temperature 31.5 °C Speed 4 on mixer

12/20/00
- very little air in 21 for mixing, so sucked ~70ml off to make room for mixing
- 21 still would not mix with shaking, so used glass rod.
- some solids stuck in pile on sieve. used rod to mush it around. looks like lots of chunks.
- used glass stopper to try try to push through sieve and went right through.
-27 had big chunk pile on sieve, pushed right through with glass stopper except ~2-3 chunks
-21 had several chunks, but could not get clear shot on video due to inability to rinse off sides

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

14) While operating the vessel agitator, extract one sub-sample of sufficient size to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel and record the time. Then collect three sub-samples of approximately 100 ml sub-samples from the mixing vessel into volume graduated glass jars of known mass labeled **"AN-102 AR-A"**, **"AN-102 AR-B"** and **"AN-102 AR-C"**. Weigh the jars to ±0.01g and measure the volumes to ±1ml. Record the mass and volume data below. Record the time the final sample was collected. 12-21-00

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

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

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

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

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

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

Day 12/27/00 Time 11 Am

Bottles ~~tare~~ tare wts outside cell – checked w/ 450.0g & read 449.99g
Balance 362-06-01-051
Cal Data - 8/29/00
Due 4-2001
Cal Due Date 8/01
Cert # 1138

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

Day 12/27/00 Time 11 Am MJB

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

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

Table 2. Volume Percent Settled Solids In Composite Samples

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

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

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

Time _____ Date _____ Temperature _____ °C

See step 13 PRB 1/08/01

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

22) While operating the vessel agitator, extract one sub-sample of sufficient size to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel and record the time. Then collect sub-samples for characterization of the as-received material. The number and volume of these samples will be determined by the cognizant scientist. Use the necessary spaces below to record the sample masses. Record the time the final sample was collected. 12/21/00

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

12/28/00 Balance checked

AN-102 AR-D
Target 110 ml
Total 293.846 g
Tare 134.01 g
Slurry 159.84 g

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

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

11:04

AN-102 AR-G
Target 110 ml
Total 296.376 g
Tare 134.80 g
Slurry 161.78 g

AN-102 AR-H
Target ~~400~~ ml
100 PRB 12/18/00
Total 281.388 g
Tare 134.21 g
Slurry 147.18 g
Final V.R = 102 mL
R ≥ 1.99

AN-102 AR-I PRB 12/18/00
Target ~~400~~ 110 ml
Total 291.413 g
Tare 130.87 g
Slurry 160.54 g

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

a. If this analysis has already been conduced, document when and how this analysis was conducted.
b. If this analysis has not been conducted, use Technical Procedure 29953-010 to measure the following value in duplicate.
   (a) Wt% dissolved solids in the supernatant
   (b) Wt% centrifuged solids in the composite
   (c) Wt% water in the centrifuged solids

   Assuming all of the water in the centrifuged solids is from interstitial supernatant, calculate the wt% undissolved solids in the centrifuged solids.
   → Document this calculation and have it reviewed by the cognizant scientist or technical reviewer. Attach the calculation to this Test Plan.

Total x Dens

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

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

ENGINEERING WORKSHEET

Prepared By: [signature] Date: 12/29/00 Project: TP-41500-005 (data from TP-41500-015)

Title/Subject: CALC of Wt % Solids

From Calc MACRO via PAUL Bredt

Wt % Solids undissolved

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

Hence,

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

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

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

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

Ave = 38.2% rgB 12/29/00

Checked by DE Kurath 12/29/00

Attachment to test instruction 41500-005 2/[illegible]

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

Target Volume of Settled Solids =_____ml

Target Volume of Supernatant =_____ml

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

Target Volume of Homogenous Composite Material to Remove =_____ml

_____g

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

Calculated Volume of Additional Supernatant =_____ml

_____g

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

Time _____ Date _____ Temperature _____°C

see attachment IRO 01/08/01

28) While operating the vessel agitator, extract one sub-sample of sufficient size to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel and record the time. Then collect the mass of composite calculated in step 25 in volume graduated glass jars of known mass. Use the necessary spaces below to record the sample masses.

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

AN-102 AR-J

Total	896.71 g
Tare	311.15 g
Slurry	585.56 g

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

Time of final collection 12/21/00 11:14 pm All 500 ml Jars

AN-102 AR-K

Total 901.12 g
Tare 311.42 g
Slurry 589.70 g

AN-102 AR-L

Total 908.61 g
Tare 311.50 g
Slurry 597.11 g

AN-102 AR-M

Total 915.76 g
Tare 311.13 g
Slurry 604.63 g

AN-102 AR-N

Total 915.70 g
Tare 311.08 g
Slurry 604.62 g

AN-102 AR-O 11:18

Total 907.77 g
Tare 311.30 g
Slurry 596.47 g

End AR series

AN-102 AR-P

Total ____ g
Tare ____ g
Slurry ____ g

PRB 10/13/01

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

Day 12/21/00 Time 11:18 pm

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

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

AN-102 AR-J 0Z

Total ~~110~~ 13.3 ~~ml~~
Solids 9.0 ml
Vol % Solids 67.7

AN-102 AR-K 0Z

Total 13.4 ml
Solids 9.4 ml
Vol % Solids 70.1

AN-102 AR-L 0Z

Total 13.6 ml
Solids 9.3 ml
Vol % Solids 68.4

AN-102 AR-M 0Z

Total 13.8 ml
Solids 9.4 ml
Vol % Solids 68.1

AN-102 AR-N 0Z

Total 13.7 ml
Solids 9.2 ml
Vol % Solids 67.2

AN-102 AR-O 0Z

Total 13.6 ml
Solids 8.9 ml
Vol % Solids 65.4

AN-102 AR-P

Total ____ ml
Solids ____ ml
Vol % Solids ____

Not needed
PRB 1/2/01

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

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

AN-102 SUP A		AN-102 AR-__		AN-102 AR-__	
Total	_____g	Total	_____g	Total	_____g
Tare	_____g	Tare	_____g	Tare	_____g
Sup	_____g	Slurry	_____g	Slurry	_____g

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

Estimated Wt% Undissolved solids in the Composite _____

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

See attachment PRB 1/08/01

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

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

BLANK

37) While operating the vessel agitator, extract one sub-sample of sufficient size to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel and record the time. Then collect three ~100 ml sub-samples in volume graduated glass jars of known mass. Record the sample masses. Samples "AN-102 TRU 1" and "AN-102 TRU 2" are to be retained for Sr/TRU reaction mechanism tests. Record the time the final sample was collected.

For Physical property work

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

AN-102 TRU 1		AN-102 TRU 2		AN-102 MOD 1	
Total	g	Total	g	Total 298.889	g
Tare	g	Tare	g	133.933 Tare 133.933	g
Slurry	g	Slurry	g	+ 4.299 Slurry 160.657	g

Not needed 1/09/01 PRB

305.922
PRB 1/3/00
AN-102 MOD 2
~~167.450~~ g
134.067 g
167.450 g
+4.4048 g

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

MOD1

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

MOD 1 Stir Bar 4.2990

stir bar 4.4048 g MOD 2

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

1-10-01 @ 2pm

← 1-9-01

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

Day 1/10/01 Time 14:00

MOD A d = 35.53 / 26 = 1.34

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

MOD B d = 31.703 / 24.2 = 1.31

Day 1/11/01 Time 11:00

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

Day 1/11/01 Time 14:00

Total	26.5 ml	Total	24.2 ml
Solids	6.3 ml	Solids	5.9 ml

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

AN-102 MOD 2
167.5 g
÷ density 1.32
≅ 125 mL
→ 127 mL

Assume 26mL x 1.32 g/mL
≅ 34.3 g AN-102 MOD 2 Needs to go back into AN-102 Supernatant Composite in Mixing Vessel
RPB 1/12/01

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

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

Additive	Original Mass (g) [solution + jar]	Current Mass (g)	DI Water Added (g) Target	DI Water Added (g) Actual	~ Adjusted Net wt g
C-104 Filtrate #1	~~360.35~~ 809.90	796.87	13.0	13.2 ✓	449.55
C-104 Filtrate #2	~~360.29~~ ~~819.14~~ ~~819.~~	816.95	2.2	2.17 ✓	458.42
C-104 Wash (H2O) Filtrate Composite	~~508.03~~ 1128.36	1123.16	5.2	5.31 ✓	620.4
C-104 Caustic Leach Permeate	~~509.19~~ 1024.13	1022.87	1.3	1.5 ✓	514.9
C-104 Caustic Wash Composite	~~5.6~~ ~~506.3~~ ~~506.67~~ 1134.98	1132.67	2.3	2.3 ✓	628.4

PRB 1/09/01

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

rgs 1/12/01

~~density~~ density data from Kriston Brooks

C-104 Filtrate #1

Mass	_____ g	Mass	_____ g
Vol	_____ ml	Vol	_____ ml
ρ	_____ g/ml	ρ	_____ g/ml

ρ ave 1.0212 g/ml

C-104 Filtrate #2

Mass	_____ g	Mass	_____ g
Vol	_____ ml	Vol	_____ ml
ρ	_____ g/ml	ρ	_____ g/ml

ρ ave 1.0602 g/ml

C-104 Wash (H2O) Filtrate Composite

Mass	_____ g	Mass	_____ g
Vol	_____ ml	Vol	_____ ml
ρ	_____ g/ml	ρ	_____ g/ml

ρ ave 1.013 g/ml

C-104 Caustic Leach Permeate

Mass	_____ g	Mass	_____ g
Vol	_____ ml	Vol	_____ ml
ρ	_____ g/ml	ρ	_____ g/ml

ρ ave 1.0948 g/ml

C-104 Caustic Wash Composite

Mass	_____ g	Mass	_____ g
Vol	_____ ml	Vol	_____ ml
ρ	_____ g/ml	ρ	_____ g/ml

ρ ave 1.028 g/ml

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

AN102 MOD A = 27mL, Not 25mL
rvj 1/11/01

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

Additive	Required Volume (ml)	Density (g/ml)	Equivalent Mass (g) 25mL	Equivalent mass (g) 27mL
C-104 Filtrate #1	18.2 / 25mL = 19.7 / 27mL	1.0212	18.59	20.07 g
C-104 Filtrate #2	18.2 = 19.7	1.0602	19.30	20.8 g
C-104 Wash (H2O) Filtrate Composite	12.3 = 13.3	1.013	12.50	13.5 g
C-104 Caustic Leach Permeate	11.7 = 12.64	1.0948	12.81	13.83 g
C-104 Caustic Wash Composite	6.3 = 6.80	1.028	6.48	7.00 g
				25.3

rvj 1/12/01

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

121.402g minus the stopper → 113.157

133.544

AN-102 MOD A

Total	~~153.931~~ g	~~139.931~~
Tare+ Slurry	~~20.387~~ g	~~133.544~~ ~~153.544~~
C-104 Filtrate #1	20.387 g	

rgs 1/12/01

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

AN-102 MOD A

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

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

AN-102 MOD A

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

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

AN-102 MOD A

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

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

AN-102 MOD A

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

rgs 1/12/01

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

51) Record the date and time and allow the sample collected in the previous step to settle for a minimum of 24 hours. prior to mixing sample fractions. Added did not appear to disturb the mix (See Utility AN 102 Start date 12/27/00)

Day 1/12/01 Time 15:15 hr

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

Day 1/15/01 Time 11:00 rgs

AN-102 MOD A + C-104 Additions — MOD B

Total 96 ml — Total 24.0
Solids 1.80 ml — Solids ~~8~~ 6.5

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

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

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

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

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

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

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

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

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

Final wt MOD B -

Total in mixing vessel

All of	E	167.8 g
decant from	J	160.565
"	K	147.497 → 863 IRD 1/15/01
"	L	175.497
"	M	173.346
"	N	168.471
		~~993.176~~

total = 993.54 g

removed

AN-102 M.D 1	160.657
AN-102 Mo D 2	167.450
	328.107 g

Checked by [signature] 1/30/01

993.54
- 328.107
665.433 g on 1/15/01

all Barks
in m.d B 31.703
Mo D 1 [(160.657) - (35.532) - (31.703)] = 93.422
125.125 g

Total will be 665.433
125.125
790.558 g → 1.420 g/l 556.73 l

Need 569 ml in mixing vessel

⇓

807.98 g

difference is 807.98 g - 790.558 = 17.4 g

So remove 17.4 g from ANVOZ MOD 2 and transfer to mixing vessel

PRB 1/15/01

Sheet 1/30/01

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

volumes

#1 5.94 · 72.8 = 432

#2 5.94 = 432

(H2O) 5.94 · 49.1 = 291

Leach 5.94 · 46.9 = ~~276~~ 278

W.-1. 5.94 · 25.3 ≈ 150

Prepared By Dena Kruath

Checked By Paul Bu

1/30/01

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

Start Time 09:15 Date 1-18-01 Temperature 32.1 °C
End Transfer Time 09:45

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

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

Alternately estimate wt & record wt added. rgs 1/19/01

* AN-102 TRAN 3 → TARE = 218.496

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

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

Total ~ 1568.9 g + MOD A volume (75.3) = 1643.3 g

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

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

** - entire Sample was used here.

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

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

61) While operating the vessel agitator, extract one sub-sample of sufficient size to clear material from lowest portion of the vessel that may not be well mixed by the agitator. Personnel are then to pour this sample back into the mixing vessel and record the time. Then collect all the material in the mixing vessel in pre-weighed volume graduated glass jars. The number and volume of samples will be determined by the cognizant scientist based on the needs of the analytical tasks. Use the necessary spaces below to record the sample masses. Record the time the final sample was collected. These sub-samples will be used for characterization of the diluted feed, and process testing. Analytical testing will be documented on an Analytical Sample Request form (ASR). All remaining samples are to be stored in the hot cells in appropriate secondary containment until required for further testing.

To SAL 1-25-01

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

AN-102 DF A
Target 150 ml
Total 409.690 g
Tare ~~2.1~~ 218.342 g
Slurry 191.35 g

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

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

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

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

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

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

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

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

Total wt DF composite 2356.45 g

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

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

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

RJ Weaver 1/18/00

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

Attachment to Test Plan 41500-005

Author PAB 1/08/01 Technical Reviewer D E Kurath 1/8/01

Applicability

This attachment to Test Plan 41500-005 provides details on the preparation of the 2 to 5 wt% feed. This attachment is needed due to a minor deviation from the Test Plan resulting from power outages in the 325 building. As a result of these outages, the entire initial composite was collected from the pot. Therefore, sample AN-102 AR-J was not collected as planned. The following steps detail how to prepare the ~~3.5~~ 2 to 5 wt% feed based on the current state of the sub-samples. PRB 1/8/01

This document provides details regarding the implementation of Test Plan 41500-004, "Sample Compositing". Client expectations for successful achievement of project data needs have already been established via Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification". This document is a mechanism for the cognizant scientist to communicate to technical staff specifics on Test Plan 41500-004 implementation. Therefore, this document qualifies as a Test Instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction of a cognizant scientist.

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

Work Instructions

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

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

2) Install the baffles into the mixing vessel.

(See pg 1a following) Note:
In the following steps, 825 g of supernatant are to be separated from the AN-102 composite sub-samples and added into the mixing vessel along with sub-sample AN-102 AR-E to prepare the ~~3.5~~ 2 to 5 wt% feed. PRB The volumes of supernatant added to the mixing vessel are to be recorded in Table 1. The following steps are provided to document how much supernatant is removed from each sub-sample. The total supernatant mass of 825 g may be reach without the need to remove supernatant from all sub-samples indicated. Discontinue supernatant collection once the required supernatant mass is reached.

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: PBredt	Date: 1/8/01	Project: 41500-005

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

Need 694 ml of 3.5 wt% feed

Supernatant ρ = 1.43 g/ml

Slurry ρ = 1.46 g/ml

Settled solids concentration = 70.8 vol%, 71.5 wt%

Undissolved Solids concentration = 18 wt%

$694\,ml \cdot 1.43\,g/ml = 992.4\,g$ 3 wt% slurry needed

$992.4\,g \cdot 0.035 = 34.7\,g$ of undissolved solids needed

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

$x =$ **192.7 g of original slurry needed**

$192.7\,g\ slurry \cdot \frac{71.5\,g\ settled\ solids}{100\,g\ original\ slurry} =$ **137.8 g of ~~settled~~ settled solids needed**

$992.4\,g - 137.8\,g =$ **854.6 g needed of excess supernatant**

Sample AN-102 ARE contains 167.79 g of original slurry

$167.79 \cdot 0.18 = 30.20\,g$ undissolved solids

$\frac{30.22}{992.4\,g} = 3$ wt% undissolved solids

$992.4 - 167.79\,g = 824.6\,g$ of excess supernatant needed

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

checked by DG Kurath 1/8/01

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

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

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

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

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

} see following page

Total = 825.742g

AN-102 Supernatant + 167.8g of AN-102 AR-E = 993.54g of DF-comp.

÷ 2 of 1.43 = 695 mL

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

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

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

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

7) Set the lid on the mixing vessel.

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

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

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

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

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

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

AN-102 AR-N		AN-102 TRANS 1	
Total	749.46 g	Total	~~391.715~~ g 386.708
Tare	311.08 g	Tare	218.237 g
Slurry	438.38 g	Liquid	168.471 g

1-9-01

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

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

Not needed
RJB 1/9/01

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

14) Proceed to step 36 of the test plan.

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REFERENCE COPY

PNNL Test Instruction

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

102 rgs 12/12/00

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

Work Location: Radiochemical Processing Laboratory

Author: Paul Bredt

Use Category Identification: Information

Effective Date: Upon Final Approval
Supersedes Date: New

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

Required Reviewers:
_X_ Technical Reviewer _X_ SFO Manager

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

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

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

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

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

Approval	Signature	Date
Author	Paul Bredt	12/12/00
Technical Reviewer	[signature]	12/12/00
FSO Manager	Not Needed PRB 12/12/00	

102 12/12/00 rps

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

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

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

Quality Control

This work is to be conducted under the quality requirements of PNNL's Standards-Based Management System (SBMS). Changes to the test instructions can be made only by cognizant scientist and will be documented by crossing out the original information with a single line, and recording, initialing, and dating the changes. The results of all measurements will be recorded in this Test Instruction, a Laboratory Record Book (LRB), or bench sheet. Copies of the completed Test Plan, bench sheets, and LRBs will be transferred to the project file upon completion of the project.

General Instructions:

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

Start

Signature _____ Date 12/27/00

M&TE List:

_____ Balance 1:

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

Location C-cell

_____ Balance 2:

Calib ID _____ Calib Exp Date _____

Location _____ Not Needed PRB 10/13/01

Record balance check information below:

loc

Date	Actual (g)	Measured (g)	Tolerance (g)	Acceptable (Y/N)
C-cell 12/27/00	100.001	99.999	±.01	y
12/28/00	100.001	99.999	↓	y
12/29/00	100.001	100.000	↓	y
1/3/01	100.001	99.998	↓	y
1/4/01	100.001	99.999	↓	y
1/5/01	100.001	100.000	±0.01	y
01/10/01	200.000	200.002	~~±.002~~ ±.01	y
01/16/01	100.001	100.000	±.002 ±.01	y
01/16/01	200.001	200.006	±.01	y

(y) 1-10-01

Signature [signature] Date 1-16-01

As-Received Solids Testing

After 4 days + settling rgs 12/27/00

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

from TP-005 Pg 9 AR-H Total 102 mL solids 77 mL

Sample ID AN-102 AR-H Slurry = 147.18g

4 at % = 75%

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

12/27/00 1300hr

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

PRB 12/19/00

~~Hold C+D for steps~~

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

immediately run samples C+D through steps 12-17

Time 13:30 Date 12/27/00

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

Date 12/27/00 Time 14:30

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

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

Date 12/27/00 Time 16:15

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

Signature [signature] Date 12/27/00

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

Date 12/28/00 Time 07:55

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

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

Date 12/28/00 Time 3:45 pm

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

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

Date 12/29/00 Time 08:05

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

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

Date 12/29/00 Time 9:49 am

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

PRS 11/03/01

$A = 2.7 / \frac{9.4}{9.4} = 28.7$ vol% supernatant

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

AU = 29.2 vol%

Signature _____ Date 12/29/00

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

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

Date 1-3-01 Time 07:43

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

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

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

	AN102 AR A		AN102 AR B	
Total	42.947	g	40.552	g
Tare	39.264	g	~~19.054~~ 36.889	g
Liquid	3.683	g	3.663	g
Liquid	2.65	ml	2.60	ml

RJB 12/29/00

D = 1.39 g/mL

D = 1.41 g/mL

12) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

	AN102 AR A		AN102 AR B		C		D	
Total	32.595	g	32.692	g	32.844	g	33.313	g
Tare	19.888	g	19.054	g	19.002		19.297	
Sample	13.477	g	13.638	g	13.842		14.016	
Total	9.20	ml	9.25	ml	9.50	mL	9.40	mL
Solids	4.35	ml	4.50	ml	4.3		4.1	
Liquid	4.85	ml	4.75	ml	5.2		5.3	

For C & D centrifuging
Start 12/27/00 @ 14:10
End " 13:10

PRB 12/19/00

12/27/00 @ 3:00pm

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

Signature _____ Date 12/29/00

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

C

	AN102 AR A		AN102 AR B		D
46.235 g	Liquid+Grad	45.939 g	Liquid+Grad	43.630 g	44.378 g
38.990 g	Grad	39.264 g	Grad	36.889 g	37.122 g
7.245 g	Liquid	6.675 g	Liquid	6.741 g	7.256 g
5.05 ml	Liquid	~~5.05~~ 4.75 ml	Liquid	4.70 ml	5.10 ml
d= 1.435		d = 1.405		d = 1.434	d = 1.423
25.528 g	Solids+cone	25.859 g	Solids+cone	26.122 g	25.936 g
19.002 g	Cone	19.118 g	Cone	19.054 g	19.297 g
6.526 g	Solids	6.741 g	Solids	7.068 g	6.639 g

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

CL

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

15) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.

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

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

End drying 12/29/00 @ 9 Am

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

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

CL

	AN102 AR AL		AN102 AR BL		DL
20.631 g	Total	20.092 20.861 g (F)	Total	20.512 20.467 g (F)	20.866 g	20.807 20.795 (F)
16.812 g	Tare	16.917 g	Tare	16.885 g	17.068 g	
3.819 g	sample	4.175 3.944 g	sample	3.627 3.577 g	3.798 g	3.727 g

C

	AN102 AR A (F)		AN102 AR B (F)		D
24.952 g	Total	25.202 25.101 g	Total	25.233 25.068 g	25.344 g	24.840
19.002 g	Tare	19.118 g	Tare	19.054 g	19.297 g	19.744
5.95 g	sample	6.084 5.983 g	sample	6.179 6.014 g	6.047 g	5.447

(F) = final

(See reverse side)

rgs

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

Signature [signature] Date 1-3-01

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

Diluted Feed Solids Testing

3 wt%

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

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

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

5 ml for "C" sample

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

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

Time 11:45 Date 1/10/01

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

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

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

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

Date 01-10-01 Time 15:30

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

Signature [signature] Date 1-10-01

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

Thurs

Date 1/11/01 Time 11:00 am

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

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

Date 1/11/01 Time 4:15 pm

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

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

Friday

Date 1/12/01 Time 09:00

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

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

Date _____ Time _____

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

Set up for centrifuging & drying at this pt. So we can dry over the weekend

RB 1/12/01

Signature _____ Date 1-12-01

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

Date_____ Time_____ Not done, went to centrifuging & drying 1/12/01 rgs

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

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

AN102 DF A		AN102 DF B	
Total	g	Total	g
Tare	g	Tare	g
Liquid	g	Liquid	g
Liquid	ml	Liquid	ml

Not needed 1/10/01 PRB

29) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

AN102 DF A		AN102 DF B	
Total	27.995 g	Total	28.001 g
Tare	19.347 g	Tare	19.200 g
Sample	8.628 g	Sample	8.801 g
Total	6.15 ml	Total	6.15 ml
Solids	.85 ml	Solids	0.80 ml
Liquid	5.30 ml	Liquid	5.35 ml

AN102 DF C

26.224 g
19.158 g
7.066 g

5.05 mL
0.58 mL
4.47 mL

Centrifuging ↓

DF A
Start 01/12/01 @ 9:00
Stop 01/12/01 @ 10:00

DF B
01/12/01 @ 9:00
01/12/01 @ 10:00

DF C
01/11/01 8:35
↓ 9:42

rgs 1/11/01

Signature _____ Date 1/12/01

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

Not needed 1/9/01 PRB

	AN102 DF A		AN102 DF B	
Liquid + Grad		g		g
Grad		g		g
Liquid		g		g
Liquid		ml		ml
	AN-102 DFA		AN-102 DFB	
Solids + cone	20.583	g	20.421	g
Cone	19.347	g	19.200 ~~19.958~~ 19.200 1/9/01	g
Solids	1.236	g	1.221	g

AN 102 DF C
20.146 g
19.158 g
0.988 g

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

	AN102 DF AL		AN102 DF BL	
Total	23.666	g	21.896	g
Tare	16.846	g	17.022	g
Supernatant	6.87	g	4.874	g

Lost some Supernate on floor.

AN 102 DF CL
22.901 g
16.865 g
6.036 g

32) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.

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

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

1/15/01 @ 11:45

	AN102 DF AL		AN102 DF BL	
Total	20.292	g	20.138	g
Tare	16.846	g	17.022	g
sample		g		g

AN 102 DF CL
19.901 g
16.865 g

See opposite Page rev

Not stable IRB 3/16/01

	AN102 DF A		AN102 DF B	
Total	21.369	g	20.117	g
Tare	19.347	g	19.200	g
sample		g		g

AN-102 DFC
20.001 g
19.158 g

Signature _____ Date 1-12-01

Blank

Test Plan: BNFL-TI-41500-015

Physical Testing: Feed Characterization Summary Report.

Tank and Test Matrix ----

AN-102 AR-H (as received)

Date: 10/31/2001

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

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

some sample loss

PRB 11/01/01

Analyst: 11/01/01

Reviewer: 11/01/01

Test Plan: BNFL-TI-41500-015

Physical Testing: Feed Characterization Summary Report.

Tank and Test Matrix ----

Date: 10/31/2001

AN-102- E- DF (diluted feed)

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

Calc. Step	Description	units	A	B	C	MOD-2 DF- AVE	MOD-2 DF- std.dev
1	Bulk Density	g/mL	1.403	1.431	1.400	1.411	0.02
2	Vol % Settled Solids	%	21.1%	22.8%		22.0%	1.1%
3	Density of Centrifuged Solids	g/mL	1.454	1.526	~~1.703~~ delete	~~1.561~~ 1.49	~~0.13~~ 5% RPD
4	Vol % Centrifuged Solids	%	13.8%	13.0%	~~11.5%~~	~~12.8%~~ 13%	~~1.2%~~ 6% RPD
5	Wt. % Centrifuged Solids	%	14.3%	13.9%	~~14.0%~~	~~14.1%~~ 14%	~~0.2%~~ 3% RPD
6	Supernatant Density	g/mL					
7	Density of Settled Solids	g/mL					
8	Wt. % Settled Supernatant	%					
9	Wt% dissolved solids in supernatant	%	53.4%	52.4%	48.8%	51.5%	2.4%
10	Wt% total solids in Centrifuged Sludge	%	61.3%	62.2%	56.3%	59.9%	3.2%
11	Wt.% Total Solids	%	54.6%	54.3%	49.9%	52.9%	2.7%
12	Wt. % Undissolved Solids	%	2.45%	2.85%	2.04%	2.4%	0.4%

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

REFERENCE COPY

Battelle Test Instruction	Document No.: TI-RPP-WTP-098 Rev. No.: 0
Title: AN-102 Wt% Oxides W-57909	
Work Location: 325/SFO	**Page** 1 of 6
Author: PR Bredt	**Effective Date: Upon final signature** **Supersedes Date: New**
	Required Reviewers: X Author X Technical Reviewer ___ Quality Engineer ___ Project Manager ___ Client

Approval	Signature	Date
Author	Paul Bredt	7/27/01
Technical Reviewer	MW Thien	7-26-01

completed and reviewed
Paul Bredt
9/11/01

Applicability

This Test Instruction describes measurement of Wt% oxides on AN-102 as-received slurry. Client expectations for successful achievement of project data needs have been established via Task Specification TSP-W375-00-00007, "Tank 241-AN-102 Sample Composite, Homogeneity, Analysis, and Mixing with HLW Permeate Test Specification". This work is to be performed under procedure PRO-29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges". This document is a mechanism for the cognizant scientist to communicate to technical staff the specifics on procedure implementation and client expectations. Therefore, this document qualifies as a Test Instruction under Part B Section 16 of the RPL Operations Manual.

Work will be performed by RPL staff under the direction of cognizant project staff.

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

M&TE List:

Balance 1: 7/30/01 100g = 99.9989 g	Calibration ID JBG 14 AW-360 Calibration Expiration 2/2002 Location SAL Cell 2
Balance 2: (if needed)	Calibration ID _____ Calibration Expiration _____ Location _____
Thermocouple: See Last page	Calibration ID 1408 Type _____ Calibration Expiration 7/31/2002 Location SAL
Digital Thermometer:	Calibration ID _____ Calibration Expiration _____ Location SAL

AN-102-AR-H was transported from HLRF to SAL 7/30/01 - bottle was ~ 1/2 full.

Work Instructions

1) Transfer two ~9 ml sub-samples from **AN-102 AR-H** into pre-weighted glass graduated centrifuge cones. Record the mass data to ±0.001 g.

H- A		H- B	
Total	31.1926 g	Total	31.6992 g
Tare	18.6658 g	Tare	18.9037 g
Slurry	12.5268 g	Slurry	12.7955 g

Signature [signature] Date 7/30/01

- Started @ 7/30/01 @ 1400hr -

2) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

Signature [signature] Date 7/30/01 Time Finish 1500 hr

H- A			H- B		
Total	31.1918 g		Total	31.6989 g	
Tare	_____ g		Tare	_____ g	
Slurry	_____ g		Slurry	_____ g	
Total	8.50 ml	Wt solids only + cone	Total	8.8 ml	Wt solids only + cone
Solids	3.45 ml	~~24.7734~~	Solids	4.2 ml	~~23.9282~~ g / 25.2437 g
Liquids	_____ ml		Liquids	_____ ml	

calcs on spreadsheet PRB 8/9/01

3) Decanted supernatant to preweighed crucibles. Weigh the loaded crucibles.

Signature [signature] Date 7/30/01 Temperature 28 °C

AL		BL	
Total	36.0572 g	Total	42.7452 g
Tare	29.6429 g	Tare	36.2934 g
Liquid	6.4143 g	Liquid	6.4518 g

4) Transfer the solids to preweighed crucibles. Weigh the loaded crucibles.

Signature [signature] Date 7/30/01

Cone wt with residual solids
A solids – 20.9174 g
B solids – 21.0464 g

AS		BS	
Total	35.0566 g	Total	38.0742 g
Tare	31.9061 g	Tare	34.0626 g
Solids	3.1505 g	Solids	4.0116 g

5) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.

6) Transfer the solids and liquids to an oven at 105°C for 24 hours. Record the time and date.

Signature [signature] Time 14:00 Date 7/31/01

7) Remove the solids and liquids from the oven. Allow the ~~vial~~ crucible to cool ~10 minutes and reweigh. Record the time and date.

Signature [signature] Time 14:00 Date 8/1/01

AL		BL	
Total	33.0013 g	Total	40.1805 g
Tare	_____ g	Tare	_____ g
Liquid	_____ g	Liquid	_____ g

AS		BS	
Total	33.7907 g	Total	36.5540 g
Tare	_____ g	Tare	_____ g
Solids	_____ g	Solids	_____ g

8) Return the solids and liquids to an oven at 105°C for 24 hours. Record the time and date.

Signature [signature] Time 15:00 Date 8/1/01

9) Remove the solids and liquids from the oven. Allow the vial to cool ~10 minutes and reweigh. Record the time and date.

Signature RG Bowden Time 12:50 Date 8/2/1

AL		BL	
Total	32.7901 g	Total	39.6130 g
Tare	_____ g	Tare	_____ g
Liquid	_____ g	Liquid	_____ g

AS		BS	
Total	33.7553 g	Total	36.4103 g
Tare	_____ g	Tare	_____ g
Solids	_____ g	Solids	_____ g

10) As directed by the cognizant scientist, repeat heating and reweighing steps until the samples reach a stable mass. Record the mass data below or as needed on a bench sheet and attach the bench sheet to this test instruction.

Signature RG Bowden 8/6/01

? gained wt from previous data - suspect this pt.

Date	8/6/01 Tare wt	8/7/01			
AS	33.7620	33.7561			
BS	36.4127	36.4108			
AL	32.7921	32.7912			
BL	39.4691	39.4680			

11) Place the crucibles in the furnace. 8-7-01 @ 2pm rgb 8/7/01

12) Heat the furnace and hold for 30 minutes between 1000 and 1050°C. — See Calibration check — Temp was held @ 1020 → 1050° for 30 min. rgb

13) Turn off the furnace and cool to 150°C. ~ 1hr - Noticed crucible stuck to bottom of furnace.

14) Transfer the crucibles using tongs to a desiccator and cool to room temperature (1-2 hours).

15) Rewiegh the crucibles.

Signature [signature] Date 8/7/01 @ 16:00 hr

AL

Total 31.1942 g
Tare _____ g
Liquid _____ g
Oxides

BL

Total 37.9128 g
Tare _____ g
Liquid _____ g
Oxides

AS — Clean surface on bottom AL, BL, & BS all stuck to furnace bottom and had some slight amt. of furnace crudle adhering to bottom of crucible [signature] 8/7/01

AS

Total 33.2754 g
Tare _____ g
Solids _____ g
Oxides

BS

Total 35.7984 g
Tare _____ g
Solids _____ g
Oxides

CALIB – PNNL
EXPIRES: 7/31/2002
S/N: 14088
BY: EJG
DATE: 7/31/2001
LIMITATIONS:
100 – 650 deg C.
greater of
2.2 deg. C. or
0.75% of reading

← Copy of calibration tag on thermocouple used to check furnace needle temp. setting

T

Thermocouple Reading	Furnace Temp ± 5°C
100 °C	Not done
200 °C	~190 - 195°
600 °C	580 ± 5
650 °C	625° ± 5
1054 °C	1040 - 1045° ± 5°
1025°	1000° ± 5°

[signature] 8/7/01

Test Plan: TI-RPP-WTP-098

AN-102 Wt% Oxides

Date: 08/07/2001

Step #	Description	units	AN-102-H A	AN-102-H B
1a	Centrifuge cone tare	g	18.6658	18.9037
1b	Total Wt after transfer	g	31.1926	31.6992
1c	Net Wt Transferred	g	12.5268	12.7955
2a	Total Wt after centrifuging	ml	31.1918	31.6989
2b	Total Volume after centrifuging	ml	8.50	8.80
2c	Solids volume mark after cent.	ml	3.95	4.2
2d	Liquid fraction	ml	4.55	4.60
2e	Wt. Solids+ cone after cent.	g	24.7731	25.2437
	Net Wt Wet Solids	g	6.1073	6.3400

	Crucible Data		Liquid Fraction		Solids Fraction	
Step #	Description	units	A-L	B-L	A-S	B-S
3a,4a	Pre-fired Crucible Tare	g	29.6429	36.2934	31.9061	34.0626
3b,4b	Crucible + AN-102 wet material	g	36.0572	42.7452	35.0566	38.0742
3c, 4c	net wt. AN-102 wet material	g	6.4143	6.4518	3.1505	4.0116
10	Stable dry wt. @ 105 deg C.	g	32.7921	39.4691	33.7620	36.4177
10b	net dry wt. @ 105 deg C.	g	3.1492	3.1757	1.8559	2.3551
15a	Total Wt. after 1000-1050 deg C.	g	31.1942	37.9128	33.2754	35.7984
15b	Net Wt. after 1000-1050 deg C.	g	1.5513	1.6194	1.3693	1.7358

• °F

Step #	Description	units	A	B	AVE	std.dev
1	Bulk Density	g/mL	1.474	1.454	1.464	0.01
2	Centrifuged Solids Density	g/mL	1.546	1.510	1.528	0.03
3	Supernatant Density	g/mL	1.410	1.403	1.406	0.01
4	Wt.% Total Solids	%	52.3%	52.9%	52.6%	0.4%

			Liquid Fraction		Solids Fraction	
Calc. Step	Description	units	A-L	B-L	A-S	B-S
5	Wt% Sample after 105 deg C	%	49.1%	49.2%	58.9%	58.7%
		Ave	49.2%		58.8%	
		Std. Dev.	0.1%		0.1%	
6	Wt% Oxides after 1050 deg C	%	24.2%	25.1%	43.5%	43.3%
		Ave	24.6%		43.4%	
		Std. Dev.	0.6%		0.1%	

Analyst: [signature] 8/7/01

reviewed calculations and test instruction
[signature]
8/9/01

An102-wt%-oxides-8-01.xls

08/07/2001

BLANK

Date August 27, 2001

Analyst E D Jenson 8/27/01

To: P. R. Bredt

Reviewer [illegible] 8/29/01

From E. D. Jenson

Subject XRD Analysis of Sample AN-102 AR-H

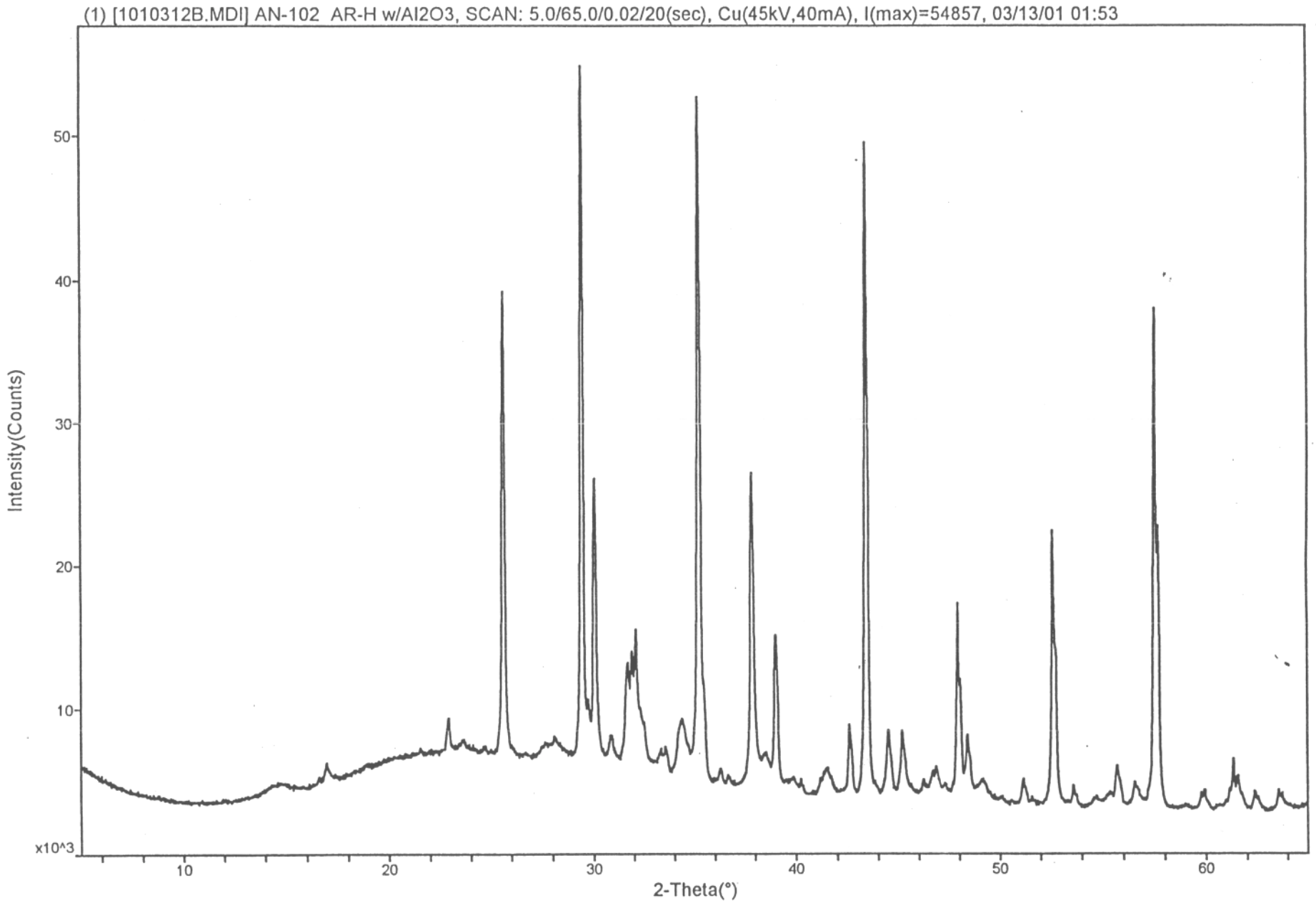
Sample AN-102 AR-H (file ID 1010312B) has been examined using X-ray Diffraction (XRD) for the identification of crystalline phases present. Major phases found were sodium nitrate ($NaNO_3$) and sodium nitrite ($NaNO_2$). Corundum, Al_2O_3, was also present as an internal standard.

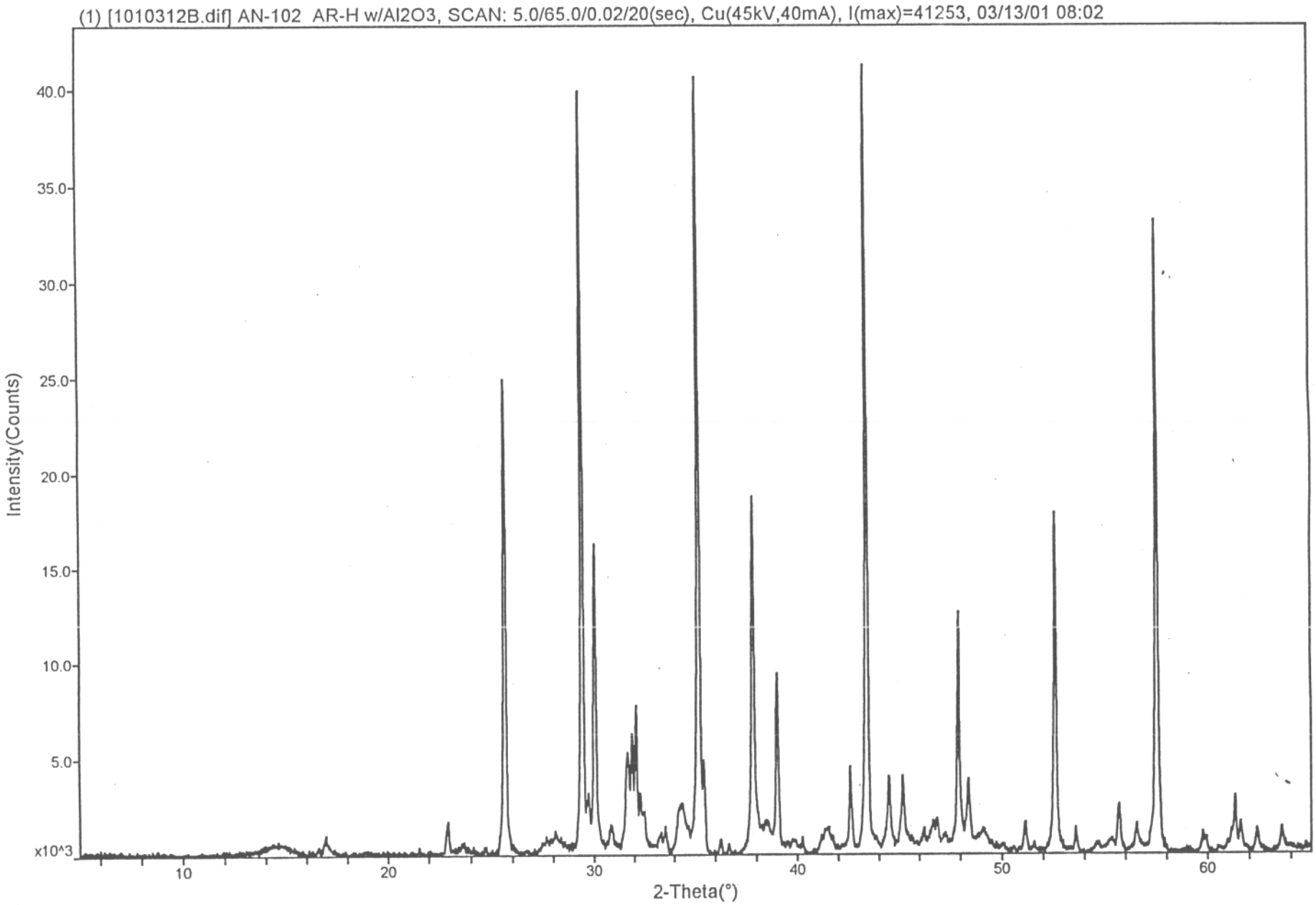
Plots are included with this report showing the raw data (.MDI file extension), background subtracted data (.DIF file extension), background subtracted and calibrated (2-theta) with stick figures of phases identified (.BIN) file extension.

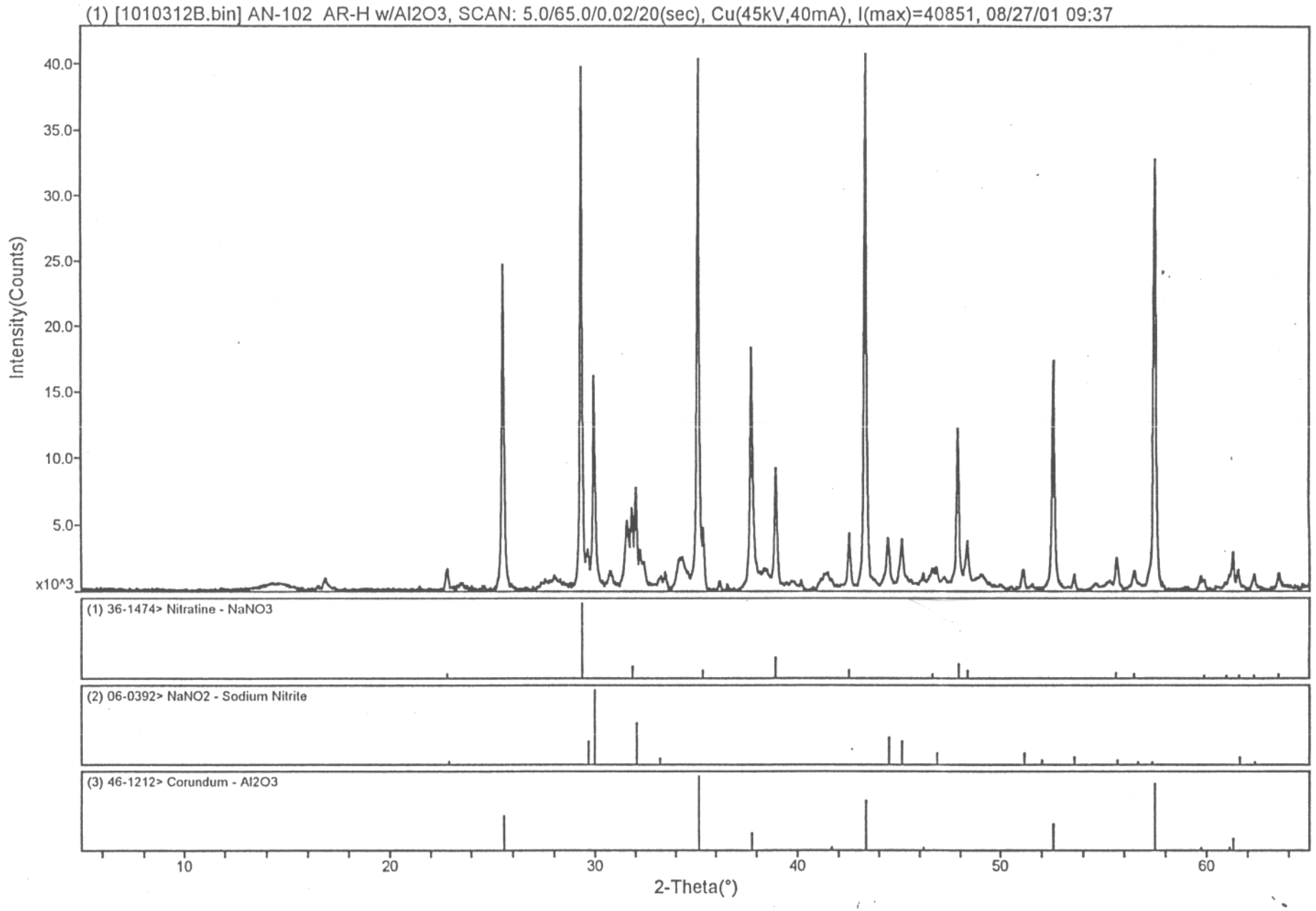
Major crystalline phases identified were sodium nitrate ($NaNO_3$, ICDD card number 36-1474) sodium nitrite ($NaNO_2$, ICDD card number 6-392). Corundum, (Al_2O_3, card number 46-1212) was also present, having been added as an internal line position standard. An amorphous "hump" is present in the raw data, indicating the presence of non-crystalline material which cannot be identified by XRD. This feature has been subtracted in the background subtracted files. Other low intensity lines were observed, but due to their very low intensity and small number, they could not be identified.

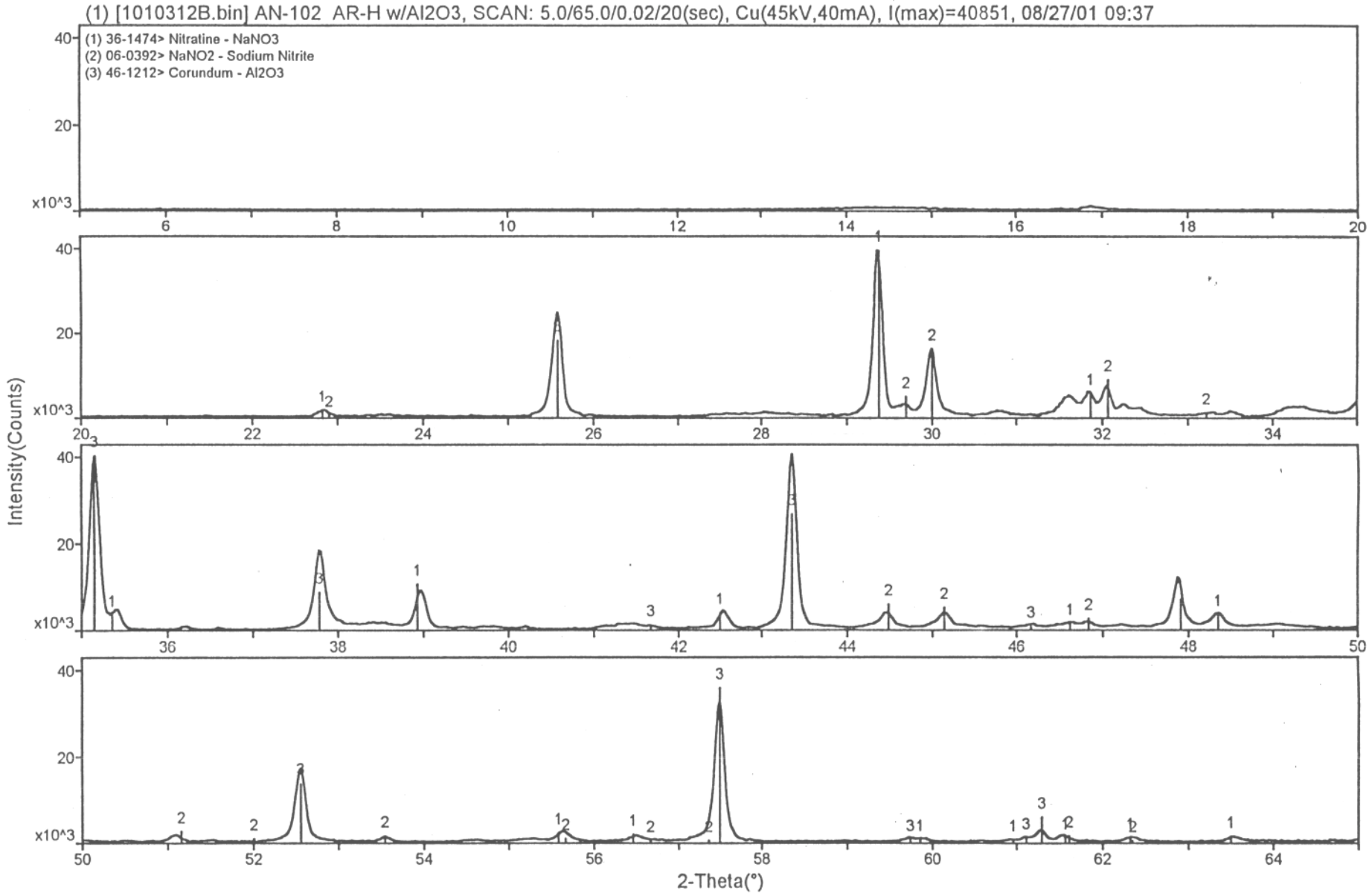
The sample was prepared as a thin sample on a glass slide using collodian as a binder. A long count time, 20 seconds per step, was used to get better counting statistics. Corundum, Al_2O_3, was added as an internal standard for line position.

This work was performed to technical procedure PNL-ALO-268, Solids Analysis, X-ray Diffraction. Calibration data are in PNL LRB 52334. The International Centre for Diffraction Data (ICDD) data base 49, through 1999 was used as the source of known 2-theta/intensity data. Daily calibration checks are in the operator's notebook.


(1) [1010312B.MDI] AN-102 AR-H w/Al2O3, SCAN: 5.0/65.0/0.02/20(sec), Cu(45kV,40mA), I(max)=54857, 03/13/01 01:53
Intensity(Counts)
x10^3
50
40
30
20
10
10
20
30
40
50
60
2-Theta(°)


(1) [1010312B.dif] AN-102 AR-H w/Al2O3, SCAN: 5.0/65.0/0.02/20(sec), Cu(45kV,40mA), I(max)=41253, 03/13/01 08:02
Intensity(Counts)
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
x10^3
10
20
30
40
50
60
2-Theta(°)


(1) [1010312B.bin] AN-102 AR-H w/Al2O3, SCAN: 5.0/65.0/0.02/20(sec), Cu(45kV,40mA), I(max)=40851, 08/27/01 09:37
Intensity(Counts)
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
x10^3
(1) 36-1474> Nitratine - NaNO3
(2) 06-0392> NaNO2 - Sodium Nitrite
(3) 46-1212> Corundum - Al2O3
10
20
30
40
50
60
2-Theta(°)


(1) [1010312B.bin] AN-102 AR-H w/Al2O3, SCAN: 5.0/65.0/0.02/20(sec), Cu(45kV,40mA), I(max)=40851, 08/27/01 09:37
(1) 36-1474> Nitratine - NaNO3
(2) 06-0392> NaNO2 - Sodium Nitrite
(3) 46-1212> Corundum - Al2O3
Intensity(Counts)
x10^3
2-Theta(°)

BLANK

Appendix C

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

RECORD COPY

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

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

PNNL Project #: 42039
Charge Code: F2616 + MULTIPLE
Date Required: 01-12-01

Matrix Type Information

- **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ✓ Sludge ___ Sediment ___ Glass ___ Filter ___ Metal ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- **QA Plan:** SBMS ✓ HASQARD (CAWSRP) ___
- **Additional QA Requirements?** No ✓ or Reference Doc # ___
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ✓ Yes ___
- **Hold Time:** None ✓ or RCRA ___ CERCLA ___ Other, Specify ___ & Date Sampled ___ Time Sampled ___
- **Special Storage Requirements:** None ✓ Refrigerate (4°C) ___ or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Disposal Information

- **Disposition of Virgin Samples:** Virgin samples are returned to requestor unless archiving provisions are made with receiving group! **If archiving, provide:** Archiving Reference Doc # ___
- **Disposition of Treated Samples:** Dispose ✓ Return ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ✓ or Reference Doc # ___ or Previous ASR # ___ or Previous RPL ID # ___

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

Additional or Special Instructions ___

Send Report To M. W. Urie **Phone** 376 9454

Phone ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12-20-00 FROM HLRF TO SHL
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) ___
CMC Waste Sample? No X Yes ___
Cost Estimate, if requested: $ ___

Received By: ___

ASR Number: 6011
RPL Numbers: (01-00386) - (01-00388)

RPG/CMC Work Accepted By: MW Urie **Signature/Date:** MW Urie 1-2-01

RG, CZS, RTR, JJW, MWU, RTS

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Library
01-00356	AN-102-AR-A	AN-102 As Received	Density-Slurry	See	
01-00357	AN-102-AR-B	"	Wt% TOTAL SOLIDS	Attachment	
01-00358	AN-102-AR-C	"	ICP *		
			GEA *		

Special Instructions: ASR 6011 (Extended Homogenization Test for AN-102)

AN-102 Slurry Composites (00-00386 through 00-00388) (1 Mill) (1 Mill)

LIMS Tests

Wt% Solids/Total	SAL	-- Use F22616
Density/Solution	SAL	-- Use F22616
Digestion-129	SAL	-- Use F22616
ICP-211-CMC	LAB	-- Use CMC WP Number
GEA-381/474-CMC	RAD	-- Use CMC WP Number

Sample received in the SAL for analytical testing are AN-102-AR-A through –G and AN-102-AR-I. Sample AN-102-A through AN-102-C are to be used for the AN-102 extended "homogenization test" being conducted under this ASR. The other containers are to be used for additional analytical work following completion of the "homogenization test" work. This ASR only covers the extended homogenization test analytical work.

Homogenization Test (2 Mill)
AN-102-AR-A --- RPL# 00-00386
AN-102-AR-B --- RPL# 00-00387
AN-102-AR-C --- RPL# 00-00388

Hold for Future Analyses
AN-102-AR-D --- RPL# TBD
AN-102-AR-E --- RPL# TBD
AN-102-AR-F --- RPL# TBD
AN-102-AR-G --- RPL# TBD
AN-102-AR-I --- RPL# TBD

FOR "EACH" SAMPLE 00-00386 THROUGH 00-00388

SAL

All "homogenization test" determinations and preparations are to be conducted on representative slurry sample. That is, the sample must be mixed/homogenized adequately while subsampling to ensure sample is homogenous. During all subsampling activities, the sample should be continuously mixed/agitated by an impeller-type mixer, and every effort should be made to obtain representative subamples.

No preparative matrix spikes or laboratory control standards are required for this work. For acid digestions, a blank should be carried through the digestion process.

1. Using at least 10 mL volumetric flask, determine density of slurry. (Note: Following the density determination, the sample material may be recombined with the original sample.)
2. Perform Wt% total solids determination in duplicate.

Hold Point: The precision of the density and Wt% solids determinations is to be evaluated prior to continuing. Contact either CZ Soderquist or MW Urie for this evaluation.

3. Acid digest seven (7) 0.5 mL subsamples per PNL-ALO-129, and record the sample weight. (To insure storage integrity for future analytical analyses, record gross weight of sample container after removing the seven subsamples for acid digestion.)
4. Document any observations from digestion (e.g., estimated residual solids quantity, color, etc.)
5. Distribute subsample of each of the seven digestates to the laboratory for GEA and ICP analysis. For GEA provide 2 mL of digestate in 20 mL scintillation vials, for ICP provide 10 mL in 20 mL scintillation vials.
6. Provide density results to laboratory staff conducting GEA and ICP analyses.

ICP and GEA Analyses

All instrumentation QC is to be performed for each analysis.

Results are to be reported in ug/mL or uCi/mL, as applicable. (Note: If 0.5 mL sample volumes are not consistent, use the sample mass and density to report results on a per mL basis.)

RPL Sample Information Checklist

(Please have client or sample owner complete to the best of their knowledge or provide the information obtained from the client.)

CLIENT

Name: M W. CRIG

Phone: 376 9454

Employer: BATTELLE

CHECKLIST PREPARED BY:

Print Name: M W Craig

Signature: M W Craig

Date: 12-20-00

(To be complete by Battelle research or laboratory staff)

Client Sample Number(s)	Description (Use page 2 if more than 4 samples)	RPL Unique Identifier(s)
AW02-HR-A	AW02 AS RECEIVED MATERIAL	01-00386
-B		01-00387
-C		01-00388

1. Is the sample from a commercial chemical product, manufacturing chemical intermediate, or off-specification commercial chemical product (i.e., P or U Listed)? (See 40 CFR 261.33) ☐ Yes ☒ No
   If yes, what chemicals or waste codes? _____

2. Is the sample from an F or K listed source? (See 40 CFR 261.31 and 261.32). ☐ Yes ☐ No
   If yes, what is the source and waste code? _____

3. Mark any of the following physical and chemical characteristics or constituents (and associated waste code) you suspect to be present:

☐ Explosive (D003)	☐ Pyrophoric (D003)	☐ Shock Sensitive (D003)	☐ Generates Toxic Gases in Water (D003)
☐ Oxidizer (D001)	☐ Asbestos	☐ Peroxide Former (D003)	☐ Halogenated Hydrocarbons (WP01 or WP02)
☐ Sulfides (D003)	☐ Cyanides (D003)	☐ Corrosive Solids(WSC2)	☐ Polycyclic Aromatic Hydrocarbons (WP03)
☐ Water Reactive (D003)	☐ Air Reactive (D003)	☐ Corrosive to Steel (D002)	
☒ Corrosive (D002); pH = 12		☐ Ignitable (D001); Flashpoint = _____ °F or _____ °C	

4. Mark any of the following constituents (and associated waste code) you suspect to be present (40 CFR 261.24):

☐ Arsenic (D004)	☐ Benzene (D018)	☐ 2,4-Dinitrotoluene (D030)	☐ Nitrobenzene (D036)
☒ Barium (D005)	☐ Carbon tetrachloride (D019)	☐ Endrin (D012)	☐ Pentachlorophenol (D037)
☐ Cadmium (D006)	☐ Chlordane (D020)	☐ Heptachlor (& its epoxide)(D031)	☐ Pyridine (D038)
☒ Chromium D007)	☐ Chlorobenzene (D021)	☐ Hexachlorobenzene (D032)	☐ Tetrachloroethylene (D039)
☒ Lead (D008)	☐ Chloroform (D022)	☐ Hexachlorobutadiene(D033)	☐ Toxaphene (D015)
☐ Mercury (D009)	☐ Cresol, o-, m-, or p- (D023-D025)	☐ Hexachloroethane (D034)	☐ Trichloroethylene (D040)
☐ Selenium (D010)	☐ 2,4,D (D016)	☐ Lindane (D013)	☐ 2,4,5-Trichlorophenol (D041)
☒ Silver (D011)	☐ 1,4-Dichlorobenzene (D027)	☐ Methoxychlor (D014)	☐ 2,4,6-Trichlorophenol (D042)
	☐ 1,2-Dichloroethane (D028)	☐ Methyl ethyl ketone (D035)	☐ 2,4,5-TP (Silvex) (D017)
	☐ 1,1-Dichloroethylene (D029)		☐ Vinyl chloride (D043)

5. List any known RCRA Underlying Hazardous Constituents: Unknown

6. Are there any state hazardous waste codes associated with the sample? ☐ Yes ☒ No
   If yes, please list: _____

7. Is the sample known to contain >2 ppm PCBs? ☐ Yes ☒ No
   If yes, is the sample concentration: ☐ <50 ppm ☐ >50 ppm?
   If <50 ppm, what is the source of PCBs (if known)? _____

8. Is the sample radioactive? ☒ Yes ☐ No If yes, list any known isotopes, activities, or dose rates associated with the sample:
   Sr-90, Cs 137, Pu-239, Am-241 (TOTAL ACTIVITY ≈ 0.1 Bq)

(To be completed by Battelle research or laboratory staff)

Battelle Project #: 47039

Charge Code #: F22616

Signature: _____ Date: _____

Receiving Inspection	
No Anomalies Found	☐
Anomalies Found, RPL POC Notified	☐

RECORD COPY

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature: Michael W Urie
Print Name: MICHAEL W. URIE
Phone: 376-9454 MSIN: P7-22

PNNL Project #: 42089 40 mod
Charge Code: F2616 + MULTIPLE
Date Required: 01-12-01

Matrix Type Information

- ♦ **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- ♦ **Solids:** ___ Soil ✓ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- ♦ **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ **QA Plan:**
  SBMS ✓
  HASQARD (CAWSRP) ___
- ♦ **Additional QA Requirements?** No ✓
  or Reference Doc # ___
- ♦ **Field COC?** No ✓ Yes ___
- ♦ **Lab COC Required?** No ✓ Yes ___
- ♦ **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- ♦ **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- ♦ **Data Quality Review Required?** No ✓ Yes ___

Disposal Information

- ♦ **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ___
- ♦ **Disposition of Treated Samples:**
  Dispose ✓ Return ___

Waste Designation Information

- ♦ **Sample Information Check List Attached?** Yes ___
  or Reference Doc # ___
  or Previous ASR # 6011
  or Previous RPL ID # ___

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

Additional or Special Instructions ___

Send Report To M. W. URIE **Phone** 376-9454
Phone ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12-20-00 FROM HLRF TO SAL
Received By: ___
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) ___
CMC Waste Sample? No ✓ Yes ___
Cost Estimate, if requested: $ ___

ASR Number: 6011.01
RPL Numbers: (01-00386) - (01-00388) (01-00420)

RPG/CMC Work Accepted By: MW URIE **Signature/Date:** MW Urie 1-5-01

: RG, CZS, RTR, JJW, MWU, RTS

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only: RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only: Test	Lab Staff Use Only: Library
01-00386	AN-102-AR-H	AN-102 As Received	Density-slurry	See	
01-00387	AN-102-AR-B	"	Wt% TOTAL SOLIDS	Attachment	
01-00388	AN-102-AR-C	"	ICP *		
01-00420	AN-102-AR-I	"	GEA *		

* ANALYTE LIST & DET LIMITS —
THIS WORK IS to evaluate homogeneity of Subsampling, REPORT ANY ANALYTES MEASURED ABOVE SYSTEM "ROUTINE" DETECTION LIMITS

* ASR REVISION .01 Adds SAMPLE 01-00420

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ✓; On Attachment ___]

ASR # 6011.01

Page _____ of _____

Special Instructions: ASR 6011.01 (Extended Homogenization Test for AN-102) , Revision .01: Addition of Sample 01-00420

AN-102 Slurry Composites (01-00386 through 01-00388, and 01-00420)
LIMS Tests

Wt% Solids/Total	SAL	-- Use F22616
Density/Solution	SAL	-- Use F22616
Digestion-129	SAL	-- Use F22616
ICP-211-CMC	LAB	-- Use CMC WP Number
GEA-381/474-CMC	RAD	-- Use CMC WP Number

Sample received in the SAL for analytical testing are AN-102-AR-A through –G and AN-102-AR-I. Sample AN-102-A through AN-102-C, and AN-102-AR-I are to be used for the AN-102 extended "homogenization test" being conducted under this ASR. The other containers are to be used for additional analytical work following completion of the "homogenization test" work. This ASR only covers the extended homogenization test analytical work.

Homogenization Test
AN-102-AR-A --- RPL# 00-00386
AN-102-AR-B --- RPL# 00-00387
AN-102-AR-C --- RPL# 00-00388
AN-102-AR-I --- RPL# 00-00420

Hold for Future Analyses
AN-102-AR-D --- RPL# TBD
AN-102-AR-E --- RPL# TBD
AN-102-AR-F --- RPL# TBD
AN-102-AR-G --- RPL# TBD

FOR "EACH" SAMPLE 01-00386 THROUGH 01-00388, and 01-00420

SAL
All "homogenization test" determinations and preparations are to be conducted on representative slurry sample. That is, the sample must be mixed/homogenized adequately while subsampling to ensure sample is homogenous. During all subsampling activities, the sample should be continuously mixed/agitated by an impeller-type mixer (or magnetic strirrer), and every effort should be made to obtain representative subamples. **Caution: Pipets used for transferring sample must have sufficiently large orfice to ensure that representative sampling of the full range of sample particle sizes.**

No preparative matrix spikes or laboratory control standards are required for this work. For acid digestions, a blank should be carried through the digestion process.

1. Using at least 10 mL volumetric flask, determine density of slurry. (Note: Following the density determination, the sample material may be recombined with the original sample.)
2. Perform Wt% total solids determination in duplicate.

Hold Point: The precision of the density and Wt% solids determinations is to be evaluated prior to continuing. Contact either CZ Soderquist or MW Urie for this evaluation.

3. Acid digest seven (7) 0.5 mL subsamples per PNL-ALO-129, and record the sample weight. (To insure storage integrity for future analytical analyses, record gross weight of sample container after removing the seven subsamples for acid digestion.)
4. Document any observations from digestion (e.g., estimated residual solids quantity, color, etc.)
5. Distribute subsample of each of the seven digestates to the laboratory for GEA and ICP analysis. For GEA provide 2 mL of digestate in 20 mL scintillation vials, for ICP provide 10 mL in 20 mL scintillation vials.
6. Provide density results to laboratory staff conducting GEA and ICP analyses.

ICP and GEA Analyses
All instrumentation QC is to be performed for each analysis.

Results are to be reported in ug/mL or uCi/mL, as applicable. (Note: If 0.5 mL sample volumes are not consistent, use the sample mass and density to report results on a per mL basis.)

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Project / WP#: 42039 / F22616+
ASR#: 6011, 6011.01
Client: M. Urie
Total Samples: 7 replicates of 4 slurries

	From	To
RPL#:	01-00386	01-00388, 01-00420
Client ID:	"AN-102-AR-A"	"AN-102-AR-C", "AN-102-AR-I"
Sample Preparation: PNL-ALO-129/SAL/vh (~0.5mL/25mL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 01-12-01 (A0633) & 01-17-01 (A0634)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

Jerry Wagner 7-12-01
Reviewed by

M.D. Urie 7-16-01
Concur

Four AN-102 slurry samples, "AN-102-AR-A" through "AN-102-AR-I" (RPL# 01-00386 through 01-00388 and 01-00420), was processed in four batches by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-129 acid digestion of slurry material. Seven aliquots (replicates) from each of four slurry samples were prepared using approximately 0.5mL (~0.75g) of material. After digestion the digestate was diluted to a final volume of approximately 25 mL. All sample aliquots and digestate solutions were weighed. Digestate solution volume was determined by dividing the weight of digestate by its density. Density of the four slurry samples was determined using 10mL or 25mL volumetric vessels. A 1mL pipette was used to determine the density of each digestate solution. The total digestate volume was calculated by dividing the weight of digestate by its density. Slurry density of AN-102 material is approximately 1.50 g/mL. Density of digestate solutions is about 1.06 g/mL.

Samples were diluted 10-fold because of high sodium (~5400 to 7400 µg/mL) and high aluminum (~650 to 900 µg/mL) concentration in the solution submitted for analysis. Prior to analysis, samples were randomized using computer generated random number sort routine. ICPAES results are reported in µg/mL and have been corrected for analytical dilution and sample processing. Analytes that are above EQL include Al, Cd, Cr, Fe, Mn, Na, Ni, and P. The process blanks appear to have a small amount of B and Na in them. The B may come from glassware used to digest the samples or from the digestion reagents (nitric acid or hydrochloric acid). The Na concentration in the process blanks is far below that found in the samples (< 0.05%). The source of Na may also come from glassware or reagents, or carryover from previous samples during analysis.

See attached ICPAES Data Report for final results.

Quality control check-standard results met tolerance requirements for all analytes of interest reported (analytes greater than EQL) except where noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. LCS, Matrix-spikes, and blank-spikes were not submitted for analysis, only process blanks were prepared with each batch of samples and analyzed in this report.

Only a small number of duplicate measurements were calculated in this report. Based upon the poor %RPD (≥ 20%) between two samples, RPL# 01-00388 (3) and 01-00388 (5), the two samples may have been mislabeled. The analytical result for the two samples is markedly different than all the other samples. If sample weights on the bench sheets are switched and re-calculated, the results agree much better with the overall average.

Five fold serial dilution:

All analytes were within tolerance limit of 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 20% RPD for all analytes greater than EQL except as follows. Sample

RPL# 01-00388 (3) @10 compared with sample 01-00388 (4) @10 differed by 20 %RPD or more for all analytes detected. This strongly suggests either mislabel or switched sample weights during processing. The suspect sample is RPL# 01-00388(3).

Post-Spiked Samples (Group A):

All post-spiked analytes were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

All post-spiked analytes were recovered within tolerance of 75% to 125%.

Blank Spike:

Either not prepared, not supplied or not identified as a Blank-Spike.

Matrix Spiked Sample:

Either not prepared, not supplied or not identified as a matrix-Spike.

Quality Control Check Standards:

Concentration of all analytes is within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV with the following exceptions.

Palladium results measured 5% to 15% greater than expected in QC_SSTMCV. Single element standards of Pd at 2.0 µg/mL measured 2.15 µg/mL at the start of analysis session and measured 2.00 µg/mL at the end of the analysis session. Palladium was not detected in any of the samples.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except for potassium. Potassium measurements were about 6% to 10% high in the QC_SST standard. All other analytes measured were within tolerance. Single element standards of potassium measured 98.4 µg/mL at the start and 98.9 µg/mL at the end of analytical session (A0633). Potassium measured 103 µg/mL at the start and 106 µg/mL at the end of analytical session (A0634). Potassium in the samples is < 7 µg/mL in the 10-fold diluted digestate solutions.

Process Blank:

Concentration of all analytes detected is within tolerance limit of ≤EQL or less than ≤5% of the sample.

Laboratory Control Standard (LCS):

Either not prepared, not supplied or not identified as an LCS.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	322.3	346.2	312.2	482.1	310.3
	RPL/LAB #=	01-00386-PB @10	01-00386-DUP @10	01-00387-DUP @10	01-00387 (4) @10	01-00388-DUP @10
	Client ID=	PB (Batch#1)	AN-102-A (Batch #1)	AN-102-B (Batch #1)	AN-102-AR-B (Batch #2)	AN-102-C (Batch #1)
Det. Limit	Run Date=	1/12/2001	1/12/2001	1/12/2001	1/12/2001	1/12/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	--	29,900	29,000	30,100	29,700
0.250	As	--	--	--	--	--
0.050	B	[43]	[91]	[96]	[130]	[84]
0.010	Ba	--	[21]	[21]	[22]	[22]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[520]	[470]	[530]	[520]
0.015	Cd	--	61.1	58.5	[63]	59.3
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	1,560	1,490	1,550	1,550
0.025	Cu	--	[25]	[24]	[24]	[25]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	917	883	1,010	911
2.000	K	--	[1,600]	[1,500]	[1,300]	[1,500]
0.050	La	--	[28]	[27]	[26]	[28]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	218	214	[220]	216
0.050	Mo	--	[50]	[47]	[50]	[49]
0.150	Na	--	228,000	219,000	230,000	223,000
0.100	Nd	--	[52]	[50]	--	[50]
0.030	Ni	--	418	403	424	400
0.100	P	--	2,040	1,930	2,060	1,980
0.100	Pb	--	[260]	[250]	[260]	[250]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	[5.6]	[5.4]	--	[5.6]
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	[19]
0.050	Zr	--	[38]	[37]	[37]	[38]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	368.2	391.4	520.5	393.6	393.6
	RPL/LAB #=	01-00388 (7) @10	01-00420 (1) @10	01-00420 (3) @10	01-00420 (5) @10	01-00420 (7) @10
	Client ID=	AN-102-AR-C (Batch #2)	AN-102-AR-I (Batch #2)	AN-102-AR-I (Batch #2)	AN-102-AR-I (Batch #2)	AN-102-AR-I (Batch #2)
Det. Limit (ug/mL)	Run Date= (Analyte)	1/12/2001 (ug/mL)	1/12/2001 (ug/mL)	1/12/2001 (ug/mL)	1/12/2001 (ug/mL)	1/12/2001 (ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	27,600	30,000	28,800	28,300	29,500
0.250	As	--	--	--	--	--
0.050	B	[85]	[95]	[110]	[100]	[94]
0.010	Ba	[20]	[21]	[20]	[21]	[21]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[510]	[520]	[510]	[520]	[520]
0.015	Cd	56.0	60.1	[59]	59.3	61.0
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	1,450	1,550	1,500	1,500	1,550
0.025	Cu	[24]	[24]	[22]	[25]	[25]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	850	916	886	882	921
2.000	K	[1,500]	[1,200]	--	[1,600]	[1,500]
0.050	La	[27]	[26]	--	[28]	[28]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	203	219	[210]	209	217
0.050	Mo	[46]	[48]	[47]	[49]	[50]
0.150	Na	206,000	230,000	222,000	212,000	223,000
0.100	Nd	[49]	[40]	--	[55]	[51]
0.030	Ni	391	411	404	401	413
0.100	P	1,870	2,010	1,960	1,960	2,010
0.100	Pb	[240]	[250]	[240]	[260]	[260]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[20]	--	--	--	--
0.050	Zr	[35]	[37]	[35]	[37]	[38]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	307.4	305.9	420.4	381.1	434.8
	RPL/LAB #=	01-00386-PB @10	01-00386 (Original) @10	01-00387-PB @10	01-00386 (3) @10	01-00386 (4) @10
	Client ID=	PB (Batch#1)	AN-102-AR-A (Batch#1)	PB (Batch#2)	AN-102-AR-A (Batch#2)	AN-102-AR-A (Batch#2)
Det. Limit (ug/mL)	Run Date= (Analyte)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	--	27,900	--	27,700	28,700
0.250	As	--	--	--	--	--
0.050	B	[50]	[94]	[43]	[98]	[120]
0.010	Ba	--	[22]	--	[22]	[22]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[540]	--	[550]	[560]
0.015	Cd	--	59.2	--	58.8	[60]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	1,490	--	1,490	1,530
0.025	Cu	--	[28]	--	[29]	[28]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	895	--	894	927
2.000	K	--	[2,200]	--	[2,400]	[1,900]
0.050	La	--	[34]	--	[35]	[32]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	206	--	207	[210]
0.050	Mo	--	[51]	--	[50]	[51]
0.150	Na	[98]	225,000	[82]	220,000	234,000
0.100	Nd	--	[71]	--	[76]	[63]
0.030	Ni	--	401	--	395	403
0.100	P	--	1,950	--	1,930	1,960
0.100	Pb	--	[280]	--	[280]	[280]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	[5.9]	--	[5.8]	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	[19]	--	[22]	--
0.050	Zr	--	[40]	--	[40]	[39]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

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3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	366.8	368.3	448.1	296.6	291.4
	RPL/LAB #=	01-00386 (5) @10	01-00386 (6) @10	01-00386 (7) @10	01-00386-PB @10	01-00387 (Original) @10
	Client ID=	AN-102-AR-A (Batch#2)	AN-102-AR-A (Batch#2)	AN-102-AR-A (Batch#2)	PB (Batch#1)	AN-102-AR-B (Batch#1)
Det. Limit (ug/mL)	Run Date= (Analyte)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	27,000	27,700	27,800	--	27,900
0.250	As	--	--	--	--	--
0.050	B	[92]	[98]	[110]	[49]	[84]
0.010	Ba	[21]	[22]	[22]	--	[22]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[530]	[570]	[580]	--	[550]
0.015	Cd	57.5	58.7	[59]	--	59.6
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	1,460	1,480	1,450	--	1,490
0.025	Cu	[29]	[29]	[29]	--	[28]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	870	901	896	--	893
2.000	K	[2,500]	[2,300]	[2,200]	--	[2,100]
0.050	La	[35]	[35]	[35]	--	[34]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	202	208	[210]	--	207
0.050	Mo	[50]	[51]	[52]	--	[51]
0.150	Na	214,000	221,000	226,000	[94]	224,000
0.100	Nd	[81]	[77]	[76]	--	[72]
0.030	Ni	390	400	405	--	401
0.100	P	1,890	1,940	1,970	--	1,970
0.100	Pb	[280]	[290]	[290]	--	[280]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	[5.7]	[5.9]	--	--	[5.9]
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[20]	[23]	--	--	[20]
0.050	Zr	[39]	[40]	[40]	--	[39]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

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3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

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Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	483.8 / 1/17/2001 (ug/mL)	419.3 (ug/mL)	415.8 (ug/mL)	455.9 (ug/mL)	2279.4 (ug/mL)
	RPL/LAB # =	01-00387-PB @10	01-00387 (3) @10	01-00387 (5) @10	01-00387 (6) @10	01-00387 (6) @50
	Client ID=	PB (Batch#2)	AN-102-AR-B (Batch#2)	AN-102-AR-B (Batch#2)	AN-102-AR-B (Batch#2)	AN-102-AR-B (Batch#2)
0.025	Ag	--	--	--	--	--
0.060	Al	--	27,500	28,900	27,900	29,900
0.250	As	--	--	--	--	--
0.050	B	[49]	[99]	[110]	[84]	--
0.010	Ba	--	[22]	[23]	[22]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[550]	[580]	[560]	--
0.015	Cd	--	[60]	[62]	[60]	[59]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	1,450	1,520	1,460	1,530
0.025	Cu	--	[29]	[29]	[19]	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	896	925	890	928
2.000	K	--	[2,500]	[1,900]	[2,200]	--
0.050	La	--	[36]	[34]	[34]	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	[210]	215	[210]	[210]
0.050	Mo	--	[50]	[52]	[46]	--
0.150	Na	[94]	219,000	236,000	225,000	260,000
0.100	Nd	--	[80]	[69]	[73]	--
0.030	Ni	--	397	413	400	[430]
0.100	P	--	1,930	2,010	1,910	[1,800]
0.100	Pb	--	[290]	[280]	[280]	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	[21]	--	--
0.050	Zr	--	[40]	[40]	[39]	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

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Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	586.3 / 01-00387 (7) @10 / AN-102-AR-B (Batch#2) / 1/17/2001 (ug/mL)	312.1 / 01-00386-PB @10 / PB (Batch#1) / 1/17/2001 (ug/mL)	311.1 / 01-00388 (Original) @10 / AN-102-AR-C (Batch#1) / 1/17/2001 (ug/mL)	427.4 / 01-00388-PB @10 / PB (Batch#2) / 1/17/2001 (ug/mL)	464.4 / 01-00388 (3) @10 / AN-102-AR-C (Batch#2) / 1/17/2001 (ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	27,800	--	27,900	--	35,600
0.250	As	--	--	--	--	--
0.050	B	[140]	[51]	[83]	[59]	[130]
0.010	Ba	[22]	--	[22]	--	[28]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[610]	--	[540]	--	[710]
0.015	Cd	[60]	--	58.6	--	76.7
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	1,450	--	1,480	--	1,910
0.025	Cu	[29]	--	[28]	--	[38]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	893	--	890	--	1,150
2.000	K	[2,200]	--	[2,100]	--	[3,100]
0.050	La	[35]	--	[33]	--	[47]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[210]	--	207	--	268
0.050	Mo	[51]	--	[50]	--	[67]
0.150	Na	230,000	[99]	222,000	[97]	285,000
0.100	Nd	[71]	--	[70]	--	[100]
0.030	Ni	400	--	395	--	516
0.100	P	1,930	--	1,930	--	2,500
0.100	Pb	[280]	--	[280]	--	[370]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	[5.7]	--	[7.6]
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	[21]	--	[29]
0.050	Zr	[39]	--	[39]	--	[52]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

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	Multiplier=	443.5	374.6	414.2	477.2	481.3
	RPL/LAB #=	01-00388 (4) @10	01-00388 (5) @10	01-00388 (6) @10	01-00420-PB @10	01-00420 (2) @10
	Client ID=	AN-102-AR-C (Batch#2)	AN-102-AR-C (Batch#2)	AN-102-AR-C (Batch#2)	PB-I (Batch#2)	AN-102-AR-I (Batch#2)
Det. Limit (ug/mL)	Run Date= (Analyte)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	29,100	24,900	27,200	--	28,900
0.250	As	--	--	--	--	--
0.050	B	[110]	[97]	[100]	[68]	[130]
0.010	Ba	[23]	[20]	[21]	--	[22]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[600]	[490]	[490]	--	[560]
0.015	Cd	[62]	[53]	[56]	--	[60]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	1,550	1,330	1,440	--	1,540
0.025	Cu	[30]	[26]	[27]	--	[29]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	940	802	866	--	932
2.000	K	[2,300]	[2,000]	[2,100]	--	[2,000]
0.050	La	[36]	[31]	[32]	--	[34]
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[220]	[190]	[200]	--	[220]
0.050	Mo	[54]	[46]	[48]	--	[52]
0.150	Na	235,000	200,000	220,000	[88]	233,000
0.100	Nd	[76]	[68]	[65]	--	[68]
0.030	Ni	420	358	383	--	405
0.100	P	2,040	1,740	1,880	--	1,960
0.100	Pb	[300]	[260]	[270]	--	[280]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	[250]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[24]	[19]	--	--	--
0.050	Zr	[42]	[36]	[38]	--	[39]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	526.5	445.7			
	RPL/LAB #=	01-00420 (4) @10	01-00420 (6) @10			
	Client ID=	AN-102-AR-I (Batch#2)	AN-102-AR-I (Batch#2)			
Det. Limit (ug/mL)	Run Date= (Analyte)	1/17/2001 (ug/mL)	1/17/2001 (ug/mL)			
0.025	Ag	--	--	--	--	--
0.060	Al	27,400	28,000	--	--	--
0.250	As	--	--	--	--	--
0.050	B	[120]	[110]	--	--	--
0.010	Ba	[22]	[22]	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[580]	[570]	--	--	--
0.015	Cd	[58]	[59]	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	1,470	1,490	--	--	--
0.025	Cu	[29]	[29]	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	891	899	--	--	--
2.000	K	[2,500]	[2,300]	--	--	--
0.050	La	[36]	[35]	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[200]	[210]	--	--	--
0.050	Mo	[51]	[50]	--	--	--
0.150	Na	223,000	226,000	--	--	--
0.100	Nd	[79]	[74]	--	--	--
0.030	Ni	398	400	--	--	--
0.100	P	1,910	1,950	--	--	--
0.100	Pb	[290]	[280]	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	[39]	[40]	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Group

01-0386
02/16/01

Client : Mike Urie

Cognizant Scientist: L R Greenwood Date : 2/16/01

Concur : T Trang-Le Date : 2/16/01

Procedure: PNL-ALO-450 - Gamma Energy Analysis

Measured Activities (uCi/ml)

ALO ID Client ID	Co-60 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Am-241 Error %
01-00386PB Process Blank	4.83E-4 8%	<2.E-4	2.23E-2 2%	<3.E-4	1.10E-3 17%
01-00386 AN-102-AR-A	<5.E-2	<3.E-2	1.53E+2 2%	<2.E-1	<5.E-1
01-00386 DUP AN-102-AR-A	<5.E-2	<3.E-2	1.55E+2 2%	<2.E-1	<5.E-1
01-00386 #3 AN-102-AR-A	<5.E-2	<3.E-2	1.56E+2 2%	<2.E-1	<5.E-1
01-00386 #4 AN-102-AR-A	<5.E-2	<3.E-2	1.55E+2 2%	<2.E-1	<5.E-1
01-00386 #5 AN-102-AR-A	<4.E-2	<3.E-2	1.55E+2 2%	<2.E-1	<5.E-1
01-00386 #6 AN-102-AR-A	3.75E-2 8%	<1.E-2	1.56E+2 2%	2.42E-1 5%	1.78E-1 38%
01-00386 #7 AN-102-AR-A	3.62E-2 17%	<2.E-2	1.55E+2 2%	2.43E-1 9%	<4.E-1
01-00387PB Process Blank	<2.E-4	<2.E-4	3.89E-2 2%	<5.E-4	9.25E-4 31%
01-00387 AN-102-AR-B	3.98E-2 12%	<2.E-2	1.56E+2 2%	2.37E-1 7%	<3.E-1
01-00387 DUP AN-102-AR-B	<3.E-2	<2.E-2	1.54E+2 2%	2.54E-1 7%	<3.E-1
01-00387 #3 AN-102-AR-B	3.26E-2 16%	<2.E-2	1.58E+2 2%	2.50E-1 7%	<3.E-1
01-00387 #4 AN-102-AR-B	<3.E-2	<2.E-2	1.57E+2 2%	1.99E-1 11%	<5.E-1
01-00387 #5 AN-102-AR-B	3.93E-2 14%	<2.E-2	1.57E+2 2%	2.48E-1 9%	<4.E-1
01-00387 #6 AN-102-AR-B	<3.E-2	<2.E-2	1.55E+2 2%	<1.E-1	<4.E-1

Procedure: PNL-ALO-450 - Gamma Energy Analysis

Measured Activities (uCi/ml)

ALO ID Client ID	Co-60 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Am-241 Error %
01-00387 #7 AN-102-AR-B	<4.E-2	<3.E-2	1.55E+2 2%	<1.E-1	<5.E-1
01-00388PB Process Blank	<2.E-4	7.98E-4 7%	3.14E-2 2%	<5.E-4	8.11E-4 28%
01-00388 AN-102-AR-C	3.20E-2 13%	<2.E-2	1.55E+2 2%	2.33E-1 8%	<3.E-1
01-00388 DUP AN-102-AR-C	4.50E-2 11%	<2.E-2	1.54E+2 2%	2.28E-1 8%	<3.E-1
01-00388 #3 AN-102-AR-C	<4.E-2	<3.E-2	1.97E+2 2%	<2.E-1	<5.E-1
01-00388 #4 AN-102-AR-C	<3.E-2	<3.E-2	1.61E+2 2%	2.44E-1 9%	<4.E-1
01-00388 #5 AN-102-AR-C	<3.E-2	<2.E-2	1.40E+2 2%	2.27E-1 8%	<4.E-1
01-00388 #6 AN-102-AR-C	3.41E-2 14%	<2.E-2	1.51E+2 2%	2.11E-1 8%	<3.E-1
01-00388 #7 AN-102-AR-C	3.58E-2 13%	<2.E-2	1.47E+2 2%	2.18E-1 9%	<4.E-1
01-00420PB Process Blank	<2.E-4	8.94E-4 6%	5.90E-2 2%	<5.E-4	9.77E-4 23%
01-00420 (1) AN-102-AR-I	4.57E-2 11%	<1.E-2	1.57E+2 2%	2.51E-1 8%	<4.E-1
01-00420 (2) AN-102-AR-I	<3.E-2	<2.E-2	1.55E+2 2%	2.20E-1 8%	<5.E-1
01-00420 (3) AN-102-AR-I	<4.E-2	<2.E-2	1.58E+2 2%	2.50E-1 8%	<5.E-1
01-00420 (4) AN-102-AR-I	<4.E-2	<2.E-2	1.58E+2 2%	<2.E-1	<5.E-1
01-00420 (5) AN-102-AR-I	2.93E-2 18%	<2.E-2	1.56E+2 2%	2.16E-1 9%	<4.E-1
01-00420 (6) AN-102-AR-I	<3.E-2	<2.E-2	1.56E+2 2%	<2.E-1	<4.E-1
01-00420 (7) AN-102-AR-I	3.48E-2 12%	<1.E-2	1.56E+2 2%	1.81E-1 10%	<3.E-1

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

RECORD COPY

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature: Michael W Urie
Print Name: MICHAEL W. URIE
Phone: 376-9454 MSIN: P7-22

PNNL Project #: 42040
Charge Code: F22616 + MULTIPLE
Date Required: 3-19-01

Matrix Type Information

- **Liquids:** ✓ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ✓ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- **QA Plan:**
  SBMS ___
  HASQARD (CAWSRP) ✓
- **Additional QA Requirements?** No ___
  or Reference Doc # ATTACHED TABLE 3&4
- **Field COC?** No ✓ Yes ___
- **Lab COC Required?** No ___ Yes ✓ PCB ONLY
- **Hold Time:** None ✓
  or RCRA ___ CERCLA ___
  Other, Specify ___
  & Date Sampled ___
  Time Sampled ___
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) ___
  or Other, specify ___
- **Data Quality Review Required?** No ✓ Yes ___

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # ___
- **Disposition of Treated Samples:**
  Dispose ✓ Return ___

Waste Designation Information

- **Sample Information Check List Attached?** Yes ___
  or Reference Doc # ___
  or Previous ASR # 6011
  or Previous RPL ID # ___

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

Additional or Special Instructions ATTACHED

Send Report To M. URIE **Phone** 376-9454
Phone ___

Preliminary results requested, as available? No ✓ Yes ___ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12-20-00 FROM HLEF TO SAL
Received By: ___
Delivered By (optional) ___
Time Delivered (optional) ___
Group ID (optional) ___
CMC Waste Sample? No X Yes ___
Cost Estimate, if requested: $ ___

ASR Number: 6019
RPL Numbers: (01-00429) (01-00430)

RPG/CMC Work Accepted By: MW URIE **Signature/Date:** MW Urie 1-15-00

LRG, CJS, JJW, MWU, RTS, MJS, RGS, JAC, M-L DT, EWH, OTF, LPD

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Library
	SEE ATTACHED SPECIAL INSTRUCTIONS FOLLOWING PHASE SEPARATION				
00-00429	AN-102-SUP-AR	As Received AN-102 Supernatant.	See Attached TABLE 1 *	See Attachment SPECIAL INSTRUCTIONS	
00-00430	AN-102-CS-AR	As Received AN-102 Centrifuged Solids (wet)	See Attached TABLE 2 *	See Attachment SPECIAL INSTRUCTIONS	
		* IN ADDITION TO THE ANALYTES OF INTEREST IDENTIFIED IN TABLES 1 & 2, PCB ANALYSIS IS REQUIRED ON BOTH THE SUPERNATANT & SOLIDS PHASE — SEE SPECIAL INSTRUCTIONS.			

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ___ ; On Attachment ✓]

ASR # 6019

Page _____ of _____

Special Instructions: ASR 6019
As Received Analyses for AN-102 Supernatant Sample 01-00429 and Centrifuged Solids Sample 01-00430

AN-102 Supernatant Sample 01-00429 – LIMS Tests

Density/Solution	SAL	-- Use F22618
Wt% Solids/TDS	SAL	-- Use F22618
Ext-Solvent (for PCBs)	SAL	-- Use F22618
PCB/Pesticides	ORG	-- Use F22620
Digestion-128	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Sub-Sample	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC (w/o oxid.)	RAD	-- Use CMC K88421
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
NH3-ISE	LAB	-- Use F22619
CN/Total	LAB	-- Use F22619
Hg-131/201-CMC	LAB	-- Use CMC K88408
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use F22620
Acids/Organic	ORG	-- Use F22620
Phosphates/Organic	ORG	-- Use F22620

AN-102 Centrifuged Solids Sample 01-00430 – LIMS Tests

Wt% Solids/Total (after phase separation)	SAL	-- Use F22618
Ext-Solvent (for PCBs)	SAL	-- Use F22618
PCB/Pesticides	ORG	-- Use F22620
Digestion-129	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORTG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
Pu241-417/474-CMC	RAD	-- Use CMC K88417
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Fusion-116	SAL	-- Use F22618
ICP/MS (Pt,Pd,Rh,Ru only)	ADV INORG	-- Use F22619
Leach/Water-103	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
NH3-ISE	LAB	-- Use F22619
H3-418/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use F22618
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
CN/Total	LAB	-- Use F22619
C14-381/474-CMC	RAD	-- Use CMC K88410
Hg-131/201-CMC	LAB	-- Use CMC K88408

Phase Separation and Samples for Compositing:

The sample bottles listed below are to be used for obtaining an AN-102 supernatant composite and an AN-102 Centrifuged Solids Composite. Four of the container have been issued RPL Numbers and were used for obtaining slurry samples for the Extended Homogenization Testing (ASR 6011). For this work, all material remaining in container used for the Extended Homogenization Testing and the material in the unused container are to be phase separated into a Supernatant Composite Sample labeled **AN-102 SUP AR (01-00429)** and a Centrifuged Solids Composite Sample labeled **AN-102 CS AR (01-00430).**

Homogenization Test RPL Numbers		Samples Held in SAL for this Work	
AN-102-AR-A --- RPL#	01-00386	AN-102-AR-D ---	none assigned
AN-102-AR-B --- RPL#	01-00387	AN-102-AR-E ---	none assigned
AN-102-AR-C --- RPL#	01-00388	AN-102-AR-F ---	none assigned
AN-102-AR-I --- RPL#	01-00420	AN-102-AR-G ---	none assigned

Administrative Hold Point:

The four containers used for the Extended Homogenization Testing (i.e., A, B, C, and I) and those in archive (i.e., D, E, F, and G) are to be weighed and compared to the weights of the containers following sub-sampling for the Extended Homogenization Testing and initial sub-sampling, respectively. If the "net" weight of the containers vary by more than 0.5% notify M. Urie prior to proceeding with any phase separation activities.

General QC Information

The analyses are to be conducted per PNNL's web-based Quality Assurance Planning Subject Area, "Conducting Analytical Work in Support of Regulatory Programs". The acceptance QC criteria for the supernatant and wet centrifuged solids are attached in Table 3 and Table 4, respectively.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in duplicate.

Preparative or analysis QC is to include, preparation blank (**in duplicate for acid digestion and fusion preparations**), sample duplicate, matrix spike, matrix spike duplicate where appropriate, and an LCS. If possible, the matrix spikes and LCS are to include all the analytes of interest to be reported for the specific analysis. The matrix spikes and LCS are to be provided to the SAL by the appropriate cognizant scientist.

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

Phase Separation

For each container above:

1. Centrifuge each of the containers at approximately 1000 rpm for 1 hour. (with secondary containment)
2. Decant supernatant into tared 1-L glass bottle labeled AN-102 SUP 00-00429, close immediately.
3. Transfer wet centrifuge solids to tared 500-ml glass jar labeled AN-102 CS 00-00430; close immediately.

Following phase separation of all containers, weigh 1-L glass bottle and 500-mL glass jar and record weight.

Administrative Holdpoint:

To perform all analyses in duplicate, with full QC, required approximately 360 mL of supernatant. If the volume of the supernatant is less than 360 mL, notify M. Urie prior to proceeding with any preparations or distributions.

SAL Preparation/Analysis Preferred Order

During all sub-sampling activities, minimize the time that the supernatant and wet centrifuged solids containers are open to the hot cell atmosphere.

1. Perform a Wt% solids on the wet centrifuged solids sample and a TDS on the supernatant.
2. Distribution of Supernatant and Solids samples requiring no preparation in SAL.
3. Acid Digestions (supernatant and solids)
4. Fusions for ICP/MS (solids only)
5. Water Leaches (solids only)
6. Organic preparation for dose reduction (supernatant only), per test plan provided by J. Campbell.
7. PCB extractions (supernatant and solids); special instructions included below for extraction.

Laboratory Analyses

All analyses are to be conducted per approved PNNL procedures or test plans.

The attached Table 1 and Table 2 define the analytes of interest and the method to be used for reporting the analysis and Table 3 and Table 4 define the QC parameters (acceptance criteria) for each of the analyses. Please note the required QC criteria and the reporting units.

Results from each analysis shall be accompanied by a full narrative detailing the method used, the sample results, the QC results, and any unusual observations.

For total Cs and Rb in the supernatant sample, ICP-MS will be used for reporting the results.

For TOC and TIC, both the hot persulfate and furnace method shall be used. The furnace method is to report both the TOC and TC.

For organic analytes, the matrix spike, matrix spike duplicate, blank, and LCS shall be subjected to the same processing (e.g., IX for dose reduction or PCB extraction) as the samples.

PCB Extractions

1.0 General Comments:

1.1 An extraction scheme for the PCB extraction is provided in Figure 1.

1.2 Since there is approximately 300 mL of AN-102 supernatant available for analysis, 100-mL aliquots will be used for the sample and duplicate and 50-mL aliquots used for the matrix spike and matrix spike duplicate. If less than 300 mL of AN-102 are available, divide the quantity available by 3 (1/3 for the sample, 1/3 for the duplicate, and 1/3 for splitting between the MS and MSD).

1.3 Teflon separatory funnels (125 mL and 250 mL), with FEP caps, are used for the liquid-liquid extraction processing of the supernatant samples or the centrifuged solids water-soluble samples.

1.4 For both the supernatant and centrifuged solids extractions, three extracts from each sample (duplicate, MS, MSD, LCS, and blank) are combined and transferred from the SAL to the 329 building organic laboratory. Volume reduction and any other pre-analysis processing are performed in the 329 building.

1.5 The target method detection limit (MDL) for total PCBs in the supernatant is 1 µg/L and in the centrifuged solids is 1000 µg/Kg.

1.6 Appropriate surrogate and matrix spiking solutions will be provided by E. Hoppe and are to be added prior to extractions.

2.0 Supernatant Extraction:

2.1 Transfer aliquot of sample into a 250-mL separatory funnel.

Note: If available, 100-mL aliquots of AN-102 SUP AR are to be used for the sample and duplicate, 50-mL aliquots of AN-102 SUP AR are to be used for the matrix spike and matrix spike duplicate, and 100-mL aliquot of organic-free water are to be used for the blank and LCS.

2.2 Add the surrogate spiking solution to all samples (including blank) and the target compound spiking solution to the MS and MSD.

Note: Use the entire contents of the vial(s) provided for spiking. After transferring the contents of the spiking vial to the sample, add approximately 0.2 mL of methylene chloride to the vial(s) and transfer this rinsate to the sample.

2.3 Perform three sequential separatory funnel shakeout extractions of the supernatant using 25-mL portions of methylene chloride. Collect and combine the three extracts in a 125-mL amber bottle labeled as:

AN-102 SUP AR PCB-x
Where, x = B for blank, S for sample, D for sample duplicate,
MS for matrix spike, MSD for matrix spike duplicate, and
LCS for lab control sample

2.4 Remove samples from SAL and transfer under COC to 329 building organic laboratory for further processing.

3.0 Wet Centrifuged Solids Extraction:

3.1 Transfer an aliquot of the centrifuged solids to a tared 200-mL centrifuge tube and weigh.

Note: For the solids, 2 g aliquots of AN-102 CS AR are to be used for the sample, duplicate, matrix spike, and matrix spike duplicate. For the processing blank and the LCS, 2 g of sodium sulfate (or alternate material selected by the cognizant scientist) is to be used.

3.2 Add the surrogate spiking solution to all samples (including blank and LCS) and the target compound spiking solution to the MS and MSD and LCS. Use the entire contents of the vial(s) provided for spiking. After transferring the contents of the spiking vial to the sample, add approximately 0.2 mL of methylene chloride to the vial(s) and transfer this rinsate to the sample.

3.3 Add 25 mL of organic-free water to the centrifuge tube and ultrasonicate (pulsed) for 2 minutes at 50% power.

Note: When working with multiple samples as a batch, rinse the sonic probe with several 5-mL portions of methylene chloride between samples and add rinsate to appropriate separatory funnel.

Note: If only PCBs are being analyzed (e.g., no pesticide analyses are required), the cognizant scientist may elect to use an acid dissolution in lieu of the water dissolution. If any acidification is performed, this must be documented on the appropriate extraction bench sheet.

3.4 Centrifuge the tube and decant the liquid into a 125-mL (or larger) separatory funnel.

3.5 Repeats Steps 3 and 4, combining all liquids into the separatory funnel. If any solids remain, set aside the wet centrifuged solids for ultrasonic extraction (step 3.7).

3.6 Perform a set of three sequential separatory funnel shakeout extractions of liquid using 25-mL portions of methylene chloride. Collect and combine the three extracts in the 250-mL amber bottle labeled as designated below.

AN-102 CS AR PCB-x
Where, x = B for blank, S for sample, D for sample duplicate,
MS for matrix spike, MSD for matrix spike duplicate, and
LCS for lab control sample

3.7 To any solids remaining from Step 3.5, add 2-3 times amount of anhydrous sodium sulfate desiccant and stir with a glass or metal rod until a sandy texture is obtained.

3.8 Add 25 ml of methylene chloride/acetone mixture (1:1) and ultrasonicate (pulsed) for 2 minutes. Settle (or centrifuge if necessary) and decant the extract into the 250-mL amber bottle labeled in Step 3.6.

3.9 Repeat Step 3.8 two additional times and combine the extracts.

For further guidance and questions regarding execution of these steps for PCB extraction, contact Eric W. Hoppe, 376-2126.

Figure 1: AN-102 As-Received PCB Extraction Scheme

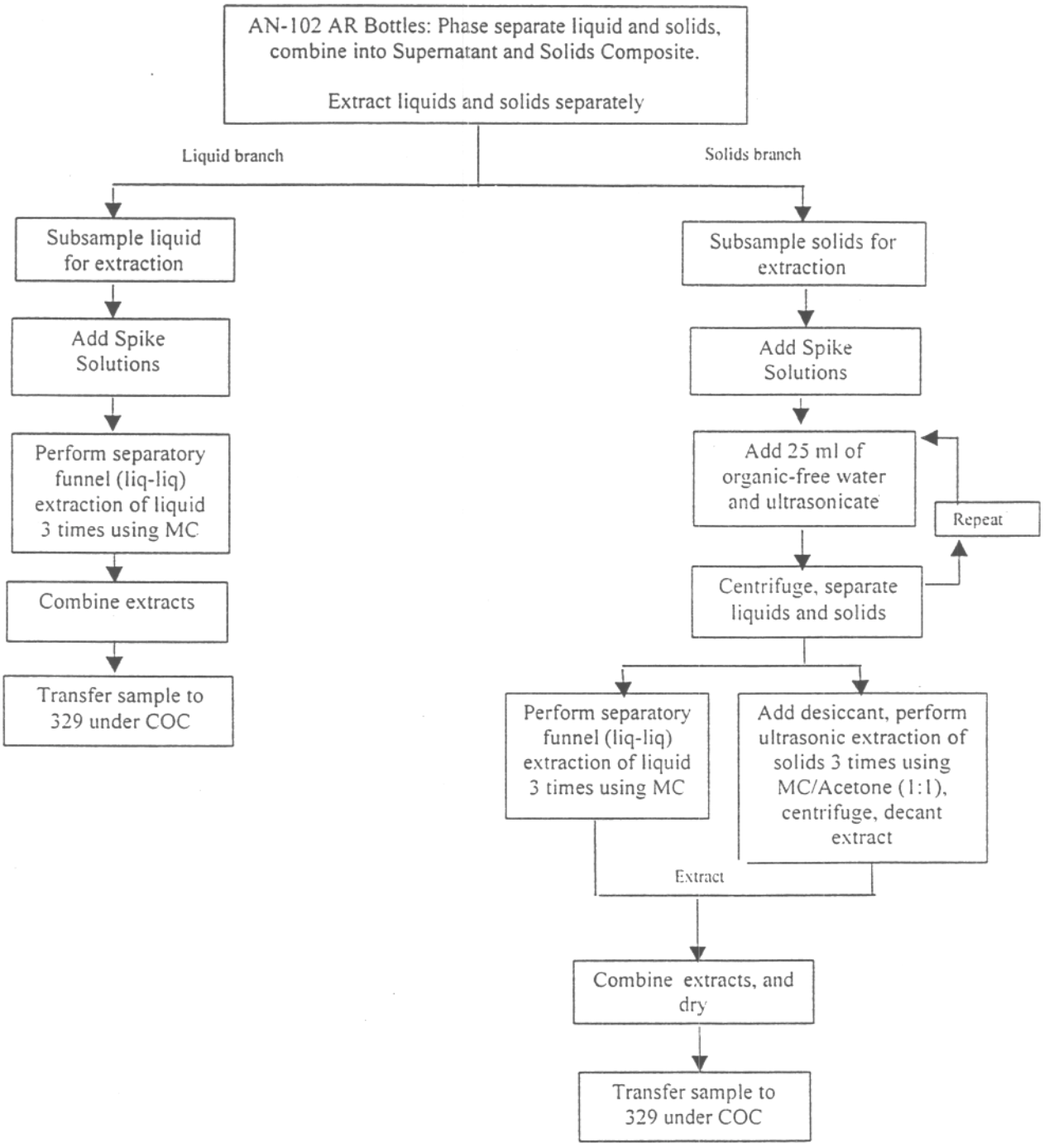


Table 1		
Analyte	Minimum Reportable Quantity	Analysis Method
	µg/ml	
Al	7.5E+01	ICP-AES (AA may be used for Na, K, Cs, Rb or ICP-MS for Cs and Rb)
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Cs	1.5E+00	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Rb	1.0E+00	
S	3.0E+01	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TIC	1.5E+02	
TOC	1.5E+03	
TOC	1.5E+03	Silver catalyze persulfate and furnace oxidation method
TIC	1.5E+02	Silver catalyze persulfate and furnace oxidation method
Hg^{1}	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03	
SO_4	2.3E+03	
	µCi/ml (except as noted)	
^{237}Np	2.7E-02	ICP-MS
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	5.1E-02	
^{99}Tc	1.5E-03	
^{99}Tc	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to
^{90}Sr	1.5E-01	Separations / Liquid Scintillation
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}Pu$	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}Cm$	1.5E-02	
^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	1.0E-02	
^{137}Cs	9.0E+00	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-
total and free OH	7.5E+04 µg/ml	Titration
Ammonia[1]	1.4E+02 µg/ml	ISE

Table 1 Supernatant Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Organic Analytes	**µg/ml**	
EDTA	1.5E+03	HPLC / LC-MS (if methods are available)
HEDTA	1.5E+03	
Oxalate	1.5E+03	
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
D2EHPA	1.5E+03	
NTA	1.5E+03	
IDA	1.5E+03	
[1]These analyses are only required for the as-received AN-102 composite sample.		

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
	µg/gm	
Ag	9.0E+02	ICP-AES
Al	3.3E+02	
Ba	6.0E+02	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
La	6.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Pd	3.0E+02	
Rh	3.0E+02	
Ru	3.0E+02	
S	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Hg[1]	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	
SO_4	1.2E+03 (as S)	

BLANK

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
SO_4	1.2E+03 (as S)	
CN^I	3.0E+00	CN analysis
NH_3^I	6.0E+01	ISE
	µg/gm	ICP-MS
As	3.0E+00	
B	3.0E+00	
Be	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Li	3.0E+01	
Mo	3.0E+01	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	
W	6.0E+00	
Y	6.0E+00	
$^{127}I^I$	1.5E+00	
$^{129}I^I$	3.0E+01	
^{233}U	6.0E+00	
^{234}U	6.0E+00	
^{235}U	6.0E+00	
^{236}U	6.0E+00	
^{238}U	6.0E+00	
^{237}Np	1.8E+00	
^{99}Tc	6.0E+00	
	µCi/gm	
$^3H^I$	1.5E-02	Separations / Liquid Scintillation
$^{14}C^I$	1.8E-03	Separations / Liquid Scintillation
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}Pu$	6.0E+00	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}Cm$	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
$^{125}Sb^I$	6.0E+00	
$^{126}Sn^I$	6.0E-02	
$^{134}Cs^I$	9.0E-01	
$^{135}Cs^I$	5.3E-03	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
^{241}Pu[1]	1.2E+00 µCi/gm	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Mineral types / structures		X-ray diffraction
[1]These analyses are only required for the as-received AN-102 composite sample.		

Table 3. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Duplicate RSD[c]
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-, PO_4^{-3}, SO_4^{-2}	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation colorimetric	80 - 120%	75 - 125%	<15%
$Cs^{[f]}$, $Eu^{[f]}$	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH-	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. Scintillation	80 - 120%	N/A[e]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
$^{60}Co^{[f]}$	GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[e]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[e]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{125}Sb	GEA	to be obtained		
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A[g]	<15%
^{137}Cs	GEA	NP	N/A[g]	<15%
$^{152}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
$^{154}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
$^{155}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
^{231}Pa	ICP/MS	Developed by Laboratory		
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
$^{237}Np^{[e]}$	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, $^{240}Pu^{[h]}$	Separation/AEA	NP	N/A[e]	<15%

Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids	Gravimetric	80 - 120%	N/A	<21%

Acronyms:
AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

[a] LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

[b] For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

[c] RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

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

[e] Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.

[f] An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

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

[h] The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes that are defined as alpha-emitting TRU (e.g., ^{237}Np, ^{242}Pu, ^{242}Cm, ^{243}Am, and $^{243+244}Cm$) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination.

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

Table 4. Quality Control Parameters for Solids Analysis

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[a]	Spike % Recovery[b]	Duplicate RSD[c]
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, La, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90[d]	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs[e]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{59}Ni[h]	Separation/GEA	NP	N/A[g]	<15%
^{63}Ni[h]	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{60}Co[i]	GEA	NP	N/A[k]	<15%
^{90}Sr[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{90}Y[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{93}Zr[h, i]	beta-liq scintillation	NP	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m}Sn[i]	Separation/GEA	NP	N/A[g]	<15%
^{125}Sb[m]	GEA	to be obtained		
^{126}Sb[i, m]	Separation/GEA	NP	N/A[g]	<15%
^{126m}Sb[i, m]	Separation/GEA	NP	N/A[g]	<15%
^{126}Sn[m]	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or	NP	N/A	<15%
^{135}Cs[h]	ICP/MS	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{151}Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{152}Eu[i]	GEA	NP	N/A[k]	<15%
^{154}Eu[i]	GEA	NP	N/A[k]	<15%
^{155}Eu[i]	GEA	NP	N/A[k]	<15%
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu	Separation/AEA	NP	N/A[g]	<15%
$^{241}Pu/Am$, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[g]	<15%
^{242}Cm	Separation/AEA	NP	N/A[g]	<15%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[g]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%

Acronyms:

AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

[a] LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

[b] For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

[c] RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

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

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

[f] Not used.

[g] Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.

[h] Radionuclide only required for WAPS justification.

[i] An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

[j] Combined analysis of ^{90}Sr and ^{90}Y.

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

[l] Combined analysis with ^{93m}Nb.

[m] Combined analysis with ^{125m}Te.

[n] Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

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RECORD COPY

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature: MW Thie
Print Name: _____
Phone _____ MSIN _____

PNNL Project #: 42040
Charge Code: F22616 + MULTIPLE
Date Required: 3-19-01

Matrix Type Information

- **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil ___ Sludge ___ Sediment ___ Glass ___ Filter ___ Metal ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- **QA Plan:** SBMS _____ HASQARD (CAWSRP) _____
- **Additional QA Requirements?** No _____ or Reference Doc # _____
- **Field COC?** No _____ Yes _____
- **Lab COC Required?** No X Yes _____
- **Hold Time:** None _____ or RCRA _____ CERCLA _____ Other, Specify _____ & Date Sampled _____ Time Sampled _____
- **Special Storage Requirements:** None _____ Refrigerate (4°C) _____ or Other, specify _____
- **Data Quality Review Required?** No _____ Yes _____

Disposal Information

- **Disposition of Virgin Samples:** Virgin samples are returned to requestor unless archiving provisions are made with receiving group! **If archiving, provide:** Archiving Reference Doc # _____
- **Disposition of Treated Samples:** Dispose _____ Return _____

Waste Designation Information

- **Sample Information Check List Attached?** Yes _____ or Reference Doc # _____ or Previous ASR # _____ or Previous RPL ID # _____

Does the Waste Designation Documentation Indicate Presence of PCBs? No _____ Yes _____

Additional or Special Instructions _____

Send Report To _____ **Phone** _____

_____ **Phone** _____

~~Preliminary results requested~~, as available? No ____ Yes ____ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: _____
Delivered By (optional) _____
Time Delivered (optional) _____
Group ID (optional) _____
CMC Waste Sample? No _____ Yes _____
Cost Estimate, if requested: $ _____

Received By: _____

ASR Number: 6019.01
RPL Numbers: 01-00429

RPG/CMC Work Accepted By: MW Thie **Signature/Date:** MW Thie 1-23-01

RTS, M-L PT, MWL, LRG, CZS, JJW, MJS, RGS, JAC, EWH, OTF, LPD

Special Instructions: ASR 6019
As Received Analyses for AN-102 Supernatant Sample 01-00429 and Centrifuged Solids Sample 01-00430

AN-102 Supernatant Sample 01-00429 – LIMS Tests

Density/Solution	SAL	-- Use F22618
Wt% Solids/TDS	SAL	-- Use F22618
Ext-Solvent (for PCBs)	SAL	-- Use F22618
PCB/Pesticides	ORG	-- Use F22620
Digestion-128	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Sub-Sample	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
Tc99-432/408-CMC (w/o oxid.)	RAD	-- Use CMC K88421
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
NH3-ISE	LAB	-- Use F22619
~~CN/Total~~	~~LAB~~	~~Use F22619~~
Hg-131/201-CMC	LAB	-- Use CMC K88408
OH-/Titration-228-CMC	LAB	-- Use CMC K88426
Chelators	ORG	-- Use F22620
Acids/Organic	ORG	-- Use F22620
Phosphates/Organic	ORG	-- Use F22620

6019.01

1) Delete CN on 01-00429

2) New Tables 1, 2, 3, 4

AN-102 Centrifuged Solids Sample 01-00430 – LIMS Tests

Wt% Solids/Total (after phase separation)	SAL	-- Use F22618
Ext-Solvent (for PCBs)	SAL	-- Use F22618
PCB/Pesticides	ORG	-- Use F22620
Digestion-129	SAL	-- Use F22618
ICP-211-CMC	LAB	-- Use CMC K88407
ICP/MS	ADV INORTG	-- Use F22619
GEA-381/474-CMC	RAD	-- Use CMC K88414
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411
Pu241-417/474-CMC	RAD	-- Use CMC K88417
U/KPA-4014-CMC	RAD	-- Use CMC K88423
Sr-90-476/408-CMC	RAD	-- Use CMC K88419
Fusion-116	SAL	-- Use F22618
ICP/MS (Pt,Pd,Rh,Ru only)	ADV INORG	-- Use F22619
Leach/Water-103	SAL	-- Use F22618
IC-212-CMC	LAB	-- Use CMC K88406
NH3-ISE	LAB	-- Use F22619
H3-418/474-CMC	RAD	-- Use CMC K88422
Sub-Sample	SAL	-- Use F22618
TOC/TIC-381-CMC	LAB	-- Use CMC K88409
TOC/TIC-380-CMC	LAB	-- Use CMC K88409
CN/Total	LAB	-- Use F22619
C14-381/474-CMC	RAD	-- Use CMC K88410
Hg-131/201-CMC	LAB	-- Use CMC K88408

Phase Separation and Samples for Compositing:

The sample bottles listed below are to be used for obtaining an AN-102 supernatant composite and an AN-102 Centrifuged Solids Composite. Four of the container have been issued RPL Numbers and were used for obtaining slurry samples for the Extended Homogenization Testing (ASR 6011). For this work, all material remaining in container used for the Extended Homogenization Testing and the material in the unused container are to be phase separated into a Supernatant Composite Sample labeled **AN-102 SUP AR (01-00429)** and a Centrifuged Solids Composite Sample labeled **AN-102 CS AR (01-00430).**

Homogenization Test RPL Numbers		Samples Held in SAL for this Work	
AN-102-AR-A --- RPL#	01-00386	AN-102-AR-D ---	none assigned
AN-102-AR-B --- RPL#	01-00387	AN-102-AR-E ---	none assigned
AN-102-AR-C --- RPL#	01-00388	AN-102-AR-F ---	none assigned
AN-102-AR-I --- RPL#	01-00420	AN-102-AR-G ---	none assigned

Administrative Hold Point:

The four containers used for the Extended Homogenization Testing (i.e., A, B, C, and I) and those in archive (i.e., D, E, F, and G) are to be weighed and compared to the weights of the containers following sub-sampling for the Extended Homogenization Testing and initial sub-sampling, respectively. If the "net" weight of the containers vary by more than 0.5% notify M. Urie prior to proceeding with any phase separation activities.

General QC Information

The analyses are to be conducted per PNNL's web-based Quality Assurance Planning Subject Area, "Conducting Analytical Work in Support of Regulatory Programs". The acceptance QC criteria for the supernatant and wet centrifuged solids are attached in Table 3 and Table 4, respectively.

Both the supernatant and wet centrifuged solids sample are to be processed and/or analyzed in duplicate.

Preparative or analysis QC is to include, preparation blank (**in duplicate for acid digestion and fusion preparations**), sample duplicate, matrix spike, matrix spike duplicate where appropriate, and an LCS. If possible, the matrix spikes and LCS are to include all the analytes of interest to be reported for the specific analysis. The matrix spikes and LCS are to be provided to the SAL by the appropriate cognizant scientist.

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

Phase Separation

For each container above:

1. Centrifuge each of the containers at approximately 1000 rpm for 1 hour. (with secondary containment)
2. Decant supernatant into tared 1-L glass bottle labeled AN-102 SUP 00-00429, close immediately.
3. Transfer wet centrifuge solids to tared 500-ml glass jar labeled AN-102 CS 00-00430; close immediately.

Following phase separation of all containers, weigh 1-L glass bottle and 500-mL glass jar and record weight.

Administrative Holdpoint:

To perform all analyses in duplicate, with full QC, required approximately 360 mL of supernatant. If the volume of the supernatant is less than 360 mL, notify M. Urie prior to proceeding with any preparations or distributions.

SAL Preparation/Analysis Preferred Order

During all sub-sampling activities, minimize the time that the supernatant and wet centrifuged solids containers are open to the hot cell atmosphere.

1. Perform a Wt% solids on the wet centrifuged solids sample and a TDS on the supernatant.
2. Distribution of Supernatant and Solids samples requiring no preparation in SAL.
3. Acid Digestions (supernatant and solids)
4. Fusions for ICP/MS (solids only)
5. Water Leaches (solids only)
6. Organic preparation for dose reduction (supernatant only), per test plan provided by J. Campbell.
7. PCB extractions (supernatant and solids); special instructions included below for extraction.

Laboratory Analyses

All analyses are to be conducted per approved PNNL procedures or test plans.

The attached Table 1 and Table 2 define the analytes of interest and the method to be used for reporting the analysis and Table 3 and Table 4 define the QC parameters (acceptance criteria) for each of the analyses. Please note the required QC criteria and the reporting units.

Results from each analysis shall be accompanied by a full narrative detailing the method used, the sample results, the QC results, and any unusual observations.

For total Cs and Rb in the supernatant sample, ICP-MS will be used for reporting the results.

For TOC and TIC, both the hot persulfate and furnace method shall be used. The furnace method is to report both the TOC and TC.

For organic analytes, the matrix spike, matrix spike duplicate, blank, and LCS shall be subjected to the same processing (e.g., IX for dose reduction or PCB extraction) as the samples.

PCB Extractions

1.0 General Comments:

1.1 An extraction scheme for the PCB extraction is provided in Figure 1.

1.2 Since there is approximately 300 mL of AN-102 supernatant available for analysis, 100-mL aliquots will be used for the sample and duplicate and 50-mL aliquots used for the matrix spike and matrix spike duplicate. If less than 300 mL of AN-102 are available, divide the quantity available by 3 (1/3 for the sample, 1/3 for the duplicate, and 1/3 for splitting between the MS and MSD).

1.3 Teflon separatory funnels (125 mL and 250 mL), with FEP caps, are used for the liquid-liquid extraction processing of the supernatant samples or the centrifuged solids water-soluble samples.

1.4 For both the supernatant and centrifuged solids extractions, three extracts from each sample (duplicate, MS, MSD, LCS, and blank) are combined and transferred from the SAL to the 329 building organic laboratory. Volume reduction and any other pre-analysis processing are performed in the 329 building.

1.5 The target method detection limit (MDL) for total PCBs in the supernatant is 1 µg/L and in the centrifuged solids is 1000 µg/Kg.

1.6 Appropriate surrogate and matrix spiking solutions will be provided by E. Hoppe and are to be added prior to extractions.

2.0 Supernatant Extraction:

2.1 Transfer aliquot of sample into a 250-mL separatory funnel.

Note: If available, 100-mL aliquots of AN-102 SUP AR are to be used for the sample and duplicate, 50-mL aliquots of AN-102 SUP AR are to be used for the matrix spike and matrix spike duplicate, and 100-mL aliquot of organic-free water are to be used for the blank and LCS.

2.2 Add the surrogate spiking solution to all samples (including blank) and the target compound spiking solution to the MS and MSD.

Note: Use the entire contents of the vial(s) provided for spiking. After transferring the contents of the spiking vial to the sample, add approximately 0.2 mL of methylene chloride to the vial(s) and transfer this rinsate to the sample.

2.3 Perform three sequential separatory funnel shakeout extractions of the supernatant using 25-mL portions of methylene chloride. Collect and combine the three extracts in a 125-mL amber bottle labeled as:

AN-102 SUP AR PCB-x
Where, x = B for blank, S for sample, D for sample duplicate,
MS for matrix spike, MSD for matrix spike duplicate, and
LCS for lab control sample

2.4 Remove samples from SAL and transfer under COC to 329 building organic laboratory for further processing.

3.0 Wet Centrifuged Solids Extraction:

3.1 Transfer an aliquot of the centrifuged solids to a tared 200-mL centrifuge tube and weigh.

Note: For the solids, 2 g aliquots of AN-102 CS AR are to be used for the sample, duplicate, matrix spike, and matrix spike duplicate. For the processing blank and the LCS, 2 g of sodium sulfate (or alternate material selected by the cognizant scientist) is to be used.

3.2 Add the surrogate spiking solution to all samples (including blank and LCS) and the target compound spiking solution to the MS and MSD and LCS. Use the entire contents of the vial(s) provided for spiking. After transferring the contents of the spiking vial to the sample, add approximately 0.2 mL of methylene chloride to the vial(s) and transfer this rinsate to the sample.

3.3 Add 25 mL of organic-free water to the centrifuge tube and ultrasonicate (pulsed) for 2 minutes at 50% power.

Note: When working with multiple samples as a batch, rinse the sonic probe with several 5-mL portions of methylene chloride between samples and add rinsate to appropriate separatory funnel.

Note: If only PCBs are being analyzed (e.g., no pesticide analyses are required), the cognizant scientist may elect to use an acid dissolution in lieu of the water dissolution. If any acidification is performed, this must be documented on the appropriate extraction bench sheet.

3.4 Centrifuge the tube and decant the liquid into a 125-mL (or larger) separatory funnel.

3.5 Repeats Steps 3 and 4, combining all liquids into the separatory funnel. If any solids remain, set aside the wet centrifuged solids for ultrasonic extraction (step 3.7).

3.6 Perform a set of three sequential separatory funnel shakeout extractions of liquid using 25-mL portions of methylene chloride. Collect and combine the three extracts in the 250-mL amber bottle labeled as designated below.

AN-102 CS AR PCB-x
Where, x = B for blank, S for sample, D for sample duplicate,
MS for matrix spike, MSD for matrix spike duplicate, and
LCS for lab control sample

3.7 To any solids remaining from Step 3.5, add 2-3 times amount of anhydrous sodium sulfate desiccant and stir with a glass or metal rod until a sandy texture is obtained.

3.8 Add 25 ml of methylene chloride/acetone mixture (1:1) and ultrasonicate (pulsed) for 2 minutes. Settle (or centrifuge if necessary) and decant the extract into the 250-mL amber bottle labeled in Step 3.6.

3.9 Repeat Step 3.8 two additional times and combine the extracts.

For further guidance and questions regarding execution of these steps for PCB extraction, contact Eric W. Hoppe, 376-2126.

Figure 1: AN-102 As-Received PCB Extraction Scheme

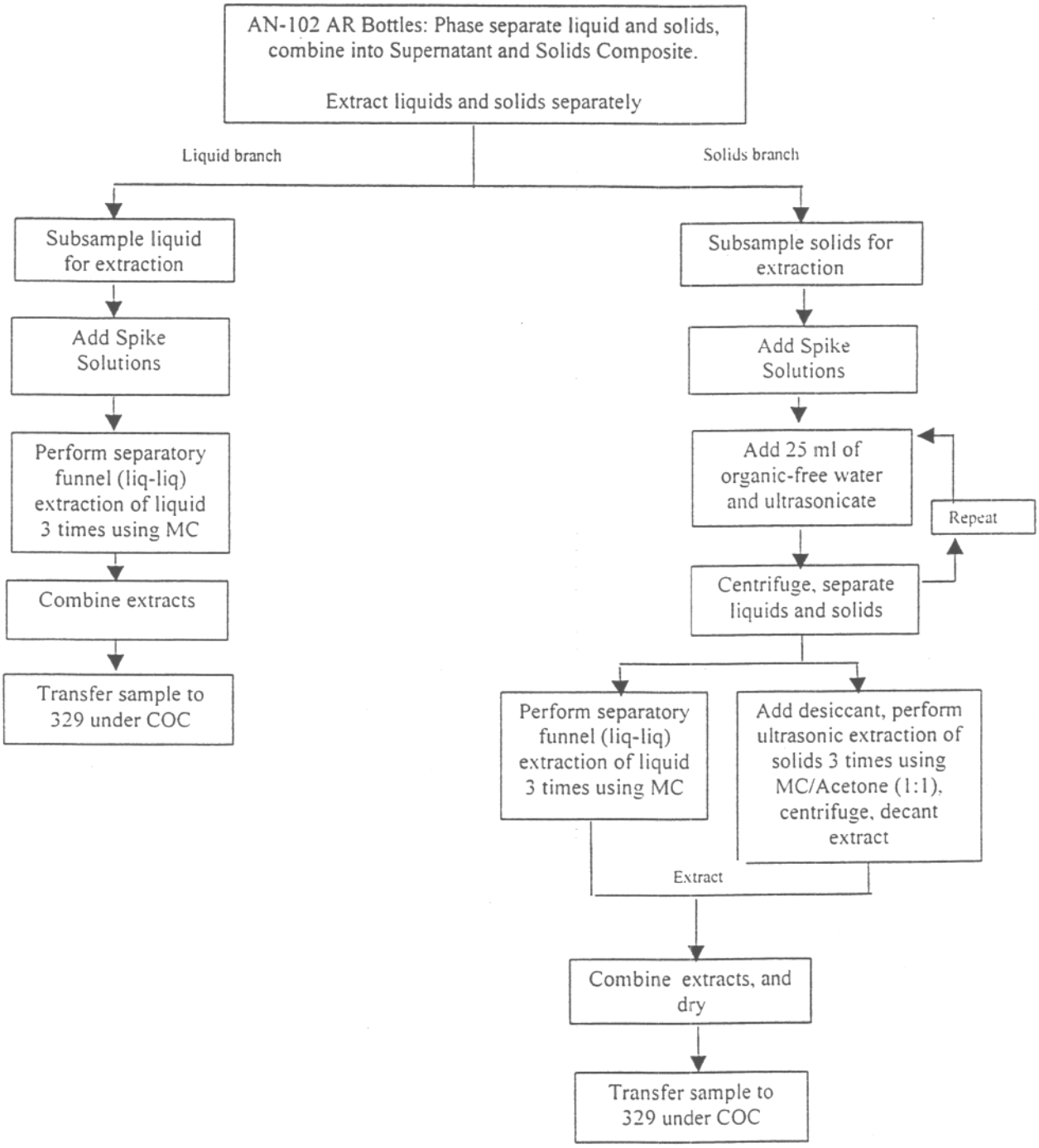


Table 1 Supernatant Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
	µg/ml	
Al	7.5E+01	ICP-AES (AA may be used for Na, K, Cs, Rb or ICP-MS for Cs and Rb)
Ba	2.3E+00	
Ca	1.5E+02	
Cd	7.5E+00	
Cr	1.5E+01	
Cs	1.5E+00	
Fe	1.5E+02	
K	7.5E+01	
La	3.5E+01	
Mg	3.0E+02	
Na	7.5E+01	
Ni	3.0E+01	
P	6.0E+02	
Pb	3.0E+02	
Rb	1.0E+00	
S	3.0E+01	
U	6.0E+02	
U	7.8E+02	Kin. Phosphorescence
TIC	1.5E+02	Silver catalyze persulfate oxidation method
TOC	1.5E+03	
TOC	1.5E+03	Furnace oxidation method
TIC	1.5E+02	
Hg^{1}	1.5E+00	Cold Vapor AA
Cl	3.0E+02	IC
F	1.5E+02	
NO_2	3.0E+03	
NO_3	3.0E+03	
PO_4	2.5E+03	
SO_4	2.3E+03	
	µCi/ml (except as noted)	
^{237}Np	2.7E-02	ICP-MS
^{239}Pu	3.0E-02	
^{240}Pu	1.0E-02	
^{241}Pu / ^{241}Am	5.1E-02	
^{99}Tc	1.5E-03	
^{99}Tc	1.5E-03	Separations / Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{90}Sr	1.5E-01	Separations / Liquid Scintillation
^{238}Pu	1.0E-02	Separations / AEA
$^{239/240}Pu$	3.0E-02	
^{241}Am	3.0E-02	
^{242}Cm	1.5E-01	
$^{243/244}Cm$	1.5E-02	
^{154}Eu	2.0E-03	Extended Counting Time GEA
^{155}Eu	9.0E-02	
^{60}Co	1.0E-02	
^{137}Cs	9.0E+00	
Total Alpha	2.3E-01	Alpha counting
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244
total and free OH	7.5E+04 µg/ml	Titration
Ammonia[1]	1.4E+02 µg/ml	ISE

Table 1		
Analyte	Minimum Reportable Quantity	Analysis Method
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Organic Analytes	µg/ml	
EDTA	1.5E+03	HPLC / LC-MS (if methods are available)
HEDTA	1.5E+03	
Oxalate	1.5E+03	
Citrate	1.5E+03	
Formate	1.5E+03	
Gluconate	1.5E+03	
Gylcolate	1.5E+03	
D2EHPA	1.5E+03	
NTA	1.5E+03	
IDA	1.5E+03	
[1]These analyses are only required for the as-received AN-102 composite sample.		

Table 2. HLW Solids Analyses		
Analyte	Minimum Reportable Quantity	Analysis Method
	µg/gm	
Ag	9.0E+02	ICP-AES
Al	3.3E+02	
Ba	6.0E+02	
Bi	6.0E+03	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Cu	1.8E+01	
Fe	1.4E+02	
La	6.0E+01	
Mg	5.4E+02	
Mn	3.0E+02	
Na	1.5E+02	
Ni	1.6E+02	
Nd	6.0E+02	
P	6.0E+02	
Pb	6.0E+02	
Pd	3.0E+02	
Rh	3.0E+02	
Ru	3.0E+02	
S	6.0E+02	
Si	3.0E+03	
Sr	3.0E+02	
Ti	1.5E+02	
Zr	6.0E+02	
Zn	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Hg^1	1.5E+00	Cold Vapor AA
Cl	2.3E+02	IC
F	7.5E+03	
NO_2	4.5E+02	
NO_3	4.5E+02	
PO_4	6.0E+02 (as P)	

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
CN^{1}	3.0E+00	CN analysis
NH_3[1]	6.0E+01	ISE
As	3.0E+00	ICP-MS
B	3.0E+00	
Be	3.0E+00	
Ce	6.0E+00	
Co	3.0E+00	
K	1.5E+03	
Li	3.0E+01	
Mo	3.0E+01	
Pr	6.0E+00	
Pt	3.0E+00	
Pu	6.0E+00	
Rb	6.0E+00	
Sb	1.2E+01	
Se	3.0E+02	
Ta	6.0E+00	
Te	6.0E+00	
Th	6.0E+02	
Tl	6.0E+02	
U	6.0E+02	
V	6.0E+00	
W	6.0E+00	
Y	6.0E+00	
$^{127}I^{1}$	1.5E+00	
$^{129}I^{1}$	3.0E+01	
^{233}U	6.0E+00	
^{234}U	6.0E+00	
^{235}U	6.0E+00	
^{236}U	6.0E+00	
^{238}U	6.0E+00	
^{237}Np	1.8E+00	
^{99}Tc	6.0E+00	
	µCi/gm	
$^{3}H^{1}$	1.5E-02	Separations / Liquid Scintillation
$^{14}C^{1}$	1.8E-03	Separations / Liquid Scintillation
^{90}Sr	7.0E+01	Separations / Beta Gas Flow Proportional Counter
^{238}Pu	6.0E-02	Separations / AEA
$^{239/240}Pu$	6.0E+00	
^{241}Am	1.8E-02	
^{242}Cm	1.2E-02	
$^{243/244}Cm$	1.2E-02	
^{60}Co	1.2E-02	Extended Counting Time GEA
$^{125}Sb^{1}$	6.0E+00	
$^{126}Sn^{1}$	6.0E-02	
$^{134}Cs^{1}$	9.0E-01	
$^{135}Cs^{1}$	5.3E-03	
^{137}Cs	6.0E-02	
^{152}Eu	6.0E-02	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{241}Am	6.0E+00	
Total Alpha	1.0E-03	Alpha Count
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243+244

Table 2. HLW Solids Analyses		
Analyte	**Minimum Reportable Quantity**	**Analysis Method**
Sum of Alpha	To be determined	Summation of Pu-238, Pu-239, Pu-240, Am-241, Cm-242, Cm-243/244
^{241}Pu[1]	1.2E+00 µCi/gm	Beta Liquid Scintillation Counting
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/ml	
Mineral types / structures		X-ray diffraction
[1]These analyses are only required for the as-received AN-102 composite sample.		

Table 3. Quality Control Parameters for Liquid Analysis

Liquid Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery[a]	Spike %Recovery[b]	Duplicate RSD[c]
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Na	ICP/AES	80 - 120%	75 - 125%	<15%
	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Ti, V, W, mass	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-, PO_4^{3-}	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation	80 - 120%	75 - 125%	<15%
$Cs^{[d]}$, $Eu^{[f]}$	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH^-	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO_3^{2-}	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. scintillation	80 - 120%	N/A[e]	<15%
^{14}C	Separation/liq. scintillation	80 - 120%	75 - 125%	<15%
$^{60}Co^{[f]}$	GEA	NP	N/A[g]	<15%
^{79}Se	Liq. scintillation	NP	N/A[e]	<15%
^{90}Sr	Isotopic specific separation/beta count	75 - 125%	N/A[e]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{99}Tc (pertechnetate)	Separation/beta count	80 - 120%	70 - 130%	<15%
^{125}Sb	GEA	to be obtained		
^{126}Sn	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or Separation/GEA	NP	N/A[g]	<15%
^{137}Cs	GEA	NP	N/A[e]	<15%
$^{152}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
$^{154}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
$^{155}Eu^{[f]}$	GEA	NP	N/A[g]	<15%
^{231}Pa	ICP/MS	Developed by Laboratory		
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
$^{237}Np^{[e]}$	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, $^{240}Pu^{[h]}$	Separation/AEA	NP	N/A[e]	<15%
$^{241}Pu/Am$, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[e]	<15%
^{242}Cm	Separation/AEA	NP	N/A[e]	<15%
$^{243}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[e]	<15%
Total Alpha[h]	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%

^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[c]	<15%
^{242}Cm	Separation/AEA	NP	N/A[c]	<15%
^{241}Am/Cm	ICP/MS	90 - 110%[d]	75 - 125%	<15%
$^{243+244}$Cm	Separation/AEA	NP	N/A[c]	<15%
Total Alpha[h]	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids	Gravimetric	80 - 120%	N/A	<21%

Acronyms:
AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

[a] LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

[b] For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

[c] RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

[d] ICP-MS mass unit 90 includes ^{90}Sr, ^{90}Y, and ^{91}Zr.

[e] Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.

[f] An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

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

[h] The sum of ^{238}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Am activities will be used as a measurement of alpha-emitting TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes that are defined as alpha-emitting TRU (e.g., ^{237}Np, ^{242}Pu, ^{242}Cm, ^{243}Am, and $^{243+244}$Cm) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm, is required specifically for class C waste determination.

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

Table 4. Quality Control Parameters for Solids Analysis

Solids Fraction	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery[a]	Spike % Recovery[b]	Duplicate RSD[c]
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Na	ICP/AES	80 - 120%	75 - 125%	<15%
	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th,	ICP/MS	80 - 120%	70 - 130%	<15%
Cl^-, F^-, NO_2^-, NO_3^-	IC	80 - 120%	75 - 125%	<15%
CN^-	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs[e]	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH_3/NH_4^+	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO_3^-	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC	silver-catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
^3H	Separation/liq. Scintillation	80 - 120%	N/A[g]	<15%
^{14}C	Separation/liq. Scintillation	80 - 120%	75 – 125%	<15%
^{59}Ni[h]	Separation/GEA	NP	N/A[g]	<15%
^{63}Ni[h]	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{60}Co[i]	GEA	NP	N/A[k]	<15%
^{90}Sr[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{90}Y[j]	Isotopic specific separation/beta count	75 - 125%	N/A[g]	<15%
^{93}Zr[h, l]	beta-liq scintillation	NP	N/A[g]	<15%
^{99}Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m}Sn[i]	Separation/GEA	NP	N/A[g]	<15%
^{125}Sb[m]	GEA	to be obtained		
^{126}Sb[i, n]	Separation/GEA	NP	N/A[g]	<15%
^{126m}Sb[i, n]	Separation/GEA	NP	N/A[g]	<15%
^{126}Sn[n]	ICP/MS	80 - 120%	70 - 130%	<15%
^{129}I	ICP/MS or	NP	N/A	<15%
^{135}Cs[m]	ICP/MS	80 - 120%	70 - 130%	<15%
^{137}Cs	GEA	NP	N/A	<15%
^{151}Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A[g]	<15%
^{152}Eu[i]	GEA	NP	N/A[k]	<15%
^{154}Eu[i]	GEA	NP	N/A[k]	<15%
^{155}Eu[i]	GEA	NP	N/A[k]	<15%
^{233}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{234}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{235}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{236}U	ICP/MS	90 - 110%	75 - 125%	<15%
^{238}U	ICP/MS	80 - 120%	70 - 130%	<15%
^{237}Np	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
^{238}Pu, ^{239}Pu, ^{240}Pu	Separation/AEA	NP	N/A[g]	<15%
^{241}Pu/Am, ^{242}Pu	ICP/MS	80 - 120%	70 - 130%	<15%
^{241}Am	Separation/AEA	NP	N/A[g]	<15%
^{243}Cm	Separation/AEA	NP	N/A[g]	<15%
$^{241}Am/Cm$	ICP/MS	90 - 110%	75 - 125%	<15%
$^{243+244}Cm$	Separation/AEA	NP	N/A[g]	<15%
Total Alpha	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids	Gravimetric	80 - 120%	N/A	<21%

Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids	Gravimetric	80 - 120%	N/A	<21%

Acronyms:

AEA – Alpha Energy Analysis
CVAA – Cold Vapor Atomic Absorption
GEA – Gamma Energy Analysis
IC – Ion Chromatography
ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – Inductively Coupled Plasma Mass Spectroscopy
LSC – Laboratory Control Standard
N/A – Not applicable
NP – Not performed
RSD – Relative Standard Deviation
Wt% – Weight percent

Footnotes:

[a] LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

[b] For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other analytes, the accuracy is determined based on use of serial dilutions.

[c] RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit. RSD = (standard deviation of the mean/mean) x 100

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

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

[f] Not used.

[g] Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.

[h] Radionuclide only required for WAPS justification.

[i] An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{152}Eu, ^{154}Eu, ^{155}Eu.

[j] Combined analysis of ^{90}Sr and ^{90}Y.

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

[l] Combined analysis with ^{93m}Nb.

[m] Combined analysis with ^{125m}Te.

[n] Combined analysis of ^{126}Sn, ^{126}Sb, and ^{126m}Sb.

File: ASR 6019

Urie, Michael W
From: Urie, Michael W
Sent: Thursday, January 25, 2001 11:20 AM
To: Wagner, Jerome J; Farmer, Orville T III; Soderquist, Chuck Z; Steele, Marilyn J; Thomas, L (May-Lin); Greenwood, Larry R; Hoppe, Eric W; Campbell, James A; Steele, Rick T; Hoopes, Francis V; Swoboda, Robert G
Subject: ASR 6019 and 6025

More thoughts/issues

In past work we have been somewhat lax in ensuring that all the QC required by the QA Plan has been prepared and analyzed. This is particularly true of the LCS and MS (required to be added prior to processing, except for radiochem).

The work conducted under these ASRs needs to have full QC. This includes LCS, MSs, blanks, and duplicates. For preparations in the hot cells, the hot cell staff will need to know what to use for an LCS and MS (and how much MS to use). The LCS needs to have all the analytes of interest or a blank spike needs to be used for evaluating the preparative process.

I will assume that for each hot cell preparative operation, each cognizant staff will work with the hot cell staff to establish the appropriate LCS and MS material for each activity. This is particularly crucial for the PCB, digestion-128 (1 mL of liquid diluted to 25 mL) for metals, digestion-129 (0.5 g solids diluted to 25 mL) for metals, water leaches of solids for IC and ammonia, and organic analyses. Where an appropriate LCS does not exist, a Blank Spike may be used. Hot cell staff should be able to contact the following for information:

PCB	E. Hoppe
Digestion – Radiochem	C. Soderquist
Digestion – ICP	J. Wagner
Digestion – ICP-MS	O. Farmer
Fusion – ICP-MS	O. Farmer
Organic sample dose reduction	J. Campbell

Also, for sub-samples, each cognizant scientist should provide the hot cell staff with the quantity of material needed for the specific analysis, and if there are any special handling issues associated with the sub-sampling. Conversely, prior to sub-sampling for specific analyses, the hot cell staff should check with the cognizant staff on sub-sampling requirements (e.g., preservation (ammonia, Hg), special containers, etc.) Contacts should be as follows:

IC	M. Steele
Tc-99 w/o oxidation	C. Soderquist
TOC/TIC (both)	M. Steele
Ammonia	C. Soderquist
Hg	J. Wagner
CN	M. Thomas
OH	R. Swoboda
Tritium	R. Swoboda

Thanks,

Mike

Michael W. Urie
Radiochemical Processing Group
RPL P7-22
Voice: (509) 376-9454
Fax: (509) 373-9675

Pacific Northwest National Laboratory
Battelle Boulevard
PO Box 999
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e-mail: mike.urie@pnl.gov

Urie, Michael W

From: Urie, Michael W
Sent: Wednesday, January 24, 2001 7:58 AM
To: Greenwood, Larry R; Soderquist, Chuck Z; Wagner, Jerome J; Steele, Rick T; Swoboda, Robert G; Campbell, James A; Hoppe, Eric W; Thomas, L (May-Lin); Farmer, Orville T III
Subject: ASR 6109 and 6025

ASR 6019.01 Characterization of As-Received AN-102 (supernatant and wet centrifuged solids)
ASR 6025 Characterization of Diluted Feed of AN-102+C-104 leachates/filtrates (supernatant and wet centrifuged solids)

Both of these samples are currently in the laboratory. Each sample is composed of a supernatant sample and a solid (wet centrifuged solids) sample. All of the analyses required by ASR 6025 are also required by ASR 6019. ASR 6019 has a few additional test required for the As-received material. This is an opportunity to "batch" samples to save some dollars. Please batch these samples whenever possible. If the preparations can not be batch, the analysis probably can be.

Mike

Michael W. Urie
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BLANK

SAL ADDENDUM TO ASR 6019

January 29, 2001

By: MW Thurman

The SAL is to maintain material balance on the two AN-102 As-Received containers **AN-102-SUP AR (01-00429) and AN-102-CS AR (01-00430)** by recording gross weight following each daily operation.

TO: ATS, FVH, File

BLANK

ADDENDUM
ASR 6019, ASR 6025, ASR 6031

The SAL is instructed to use the attached procedures (PNL-ALO- 501 and PNL-ALO-504) to perform the following:

Total Weight Percent Solids
Total Dissolved Solids
Density (supernatant)

January 30, 2000
By _____
MW Urie

Distributed to: RTS, FVH, ASR files

Use category Q procedures as Reference Use.
K. Smith 1/30/01

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-501 Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges.

APPLICABILITY

This procedure will be used to measure the following physical properties of solutions, slurries, and sludges:

- Density or Specific Gravity
- pH of solutions
- Physical Appearance
- Settling Rate
- Shear Strength
- Shear Stress versus Shear Rate
- Vol% Centrifuged Solids
- Vol% Settled Solids
- Wt% Dissolved Solids
- Wt% Centrifuged Solids
- Wt% Total Oxides
- Wt% Total Solids
- Yield Stress

This procedure is applicable to samples that are homogenous and representative of the bulk sample with the exception of the sample used for shear strength. If not homogenous, the sample will be mixed and the procedure documented in or on the appropriate Laboratory Record Book (LRB), data record, or test instruction.

DEFINITIONS

Apparent Viscosity - Viscosity of a fluid defined as a ratio of the shear stress (at a particular shear rate) and the shear rate.

Density or Specific Gravity - Density is the mass per unit volume and specific gravity is the ratio of the density of the sample and a reference material.

Interstitial Solution - The solution contained within the original sludge sample.

Author RD Scheele Date 3/9/92	Project Mgr. TE Jones Date 3/9/92	QAD Representative GK Gerke Date 3/9/92
Technical Reviewer Tingey Date	Line Mgr. GH Bryan Date 3/10/92	Other Date

Procedure No.	Revision No.	Effective Date	Page
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Newtonian - A fluid with no yield stress and a viscosity that is independent of shear rate.

Non-Newtonian - A fluid whose viscosity varies with shear rate.

pH - pH is -log $[H^+]$. pH is applicable to aqueous solutions or slurries only.

Physical Appearance - The physical state (solid, liquid, sludge, slurry), color, and consistency.

Rheogram/Flow Curve - A plot of a fluid's shear response as a function of the shear rate. A plot of shear stress versus shear rate.

Sludge - Wet solids having little or no standing liquid, i.e. mud-like.

Slurry - A thin mixture of solids and solution.

Solution - A liquid phase containing dissolved material.

Solids Settling Rate - The rate at which the solids in a homogenized sample settle.

Vol% Settled Solids - The percentage of the volume of the sample that the settled solids occupy.

Vol% Centrifuged Solids - The percentage of the volume of the sample that the solids occupy after being centrifuged for a specific time and force.

Wt% Total Oxides - The percentage of the mass of the sample that remains after converting all non-volatile elements to oxides. Some volatile elements such as cesium might be lost in this process.

Wt% Centrifuged Solids - The percentage of the mass of the sample that the solids occupy after being centrifuged.

Wt% Dissolved Solids - The percentage of the mass of sample that is dissolved in the aqueous portion of the sample.

Wt% Total Solids - The percentage of the mass of the sample that remains after removing volatiles including free water by drying at 105 ± 5°C for 24 h.

Yield Stress - The minimum stress required to initiate fluid movement.

RESPONSIBLE STAFF

Analyst
Cognizant Scientist

PROCEDURE

1.0 General Requirements

The COGNIZANT SCIENTIST is responsible for preparing the test instructions to implement each step of this procedure and supplying the analyst with the test instructions. Because of the nature and diversity of samples that will be analyzed using this procedure, the cognizant scientist is responsible for assuring that the test instructions satisfy the requirements of this procedure and provide a unique sample or container identifier to insure sample traceability and control.

All measurements will be made at the temperature specified (± 5°C) in the test instructions or at ambient temperature if unspecified. Temperature will be measured using a calibrated thermocouple or RTD and digital thermometer or a mercury thermometer. If the test instructions specify a temperature for any measurement except, vol% centrifuged solids, wt% solids, wt% dissolved solids, or wt% total oxides; a constant temperature bath/circulator will be used.

The results of all measurements will be recorded in the appropriate LRB or on the test instructions or data sheet. A copy of the test instructions sheet will be entered into the LRB or the project records. If used, the LRB will be the responsibility of the COGNIZANT SCIENTIST. The ANALYST or other authorized personnel may also record information in the LRB, test instruction, or data sheet.

The calibrated instrumentation used will be recorded in the LRB, on the test instruction, or data sheet.

2.0 Procedure for Physical Appearance Measurement

This procedure will be used to observe some general physical properties for solutions, sludges, and slurries.

2.1 Equipment and Materials

None

2.2 Determination of Physical Appearance

Describe the sample in terms of its physical state (solution, slurry, or sludge), its color, its ability to hold its shape, and any other observable physical characteristics.

The ANALYST'S ability to observe these properties may be limited by work location. For example if the sample is located in the 325A shielded facility, the actual color and the observed color may be different due to the yellow leaded glass used in the shielded cell observation windows. The properties observed in this procedure are qualitative not quantitative.

3.0 Procedure for Settling Rate and Vol% Settled Solids Measurement

3.1 Equipment and Materials

- 15 or 50 mL Graduated Plastic Centrifuge Tube with Cap
- Analytical Balance, 2-place (minimum sensitivity)
- Plastic Rod or Plastic Spatula
- Centrifuge

3.2 Determination of Solids Settling Rate and Vol% Settled Solids

Label and weigh the centrifuge tube to be used. Determine the cm/mL for the centrifuge tube by measuring the distance between the 4 and 14 mL graduations for the 15 mL tube or between the 10 and 20 mL graduations for the 50 mL tube. Calculate cm/mL.

Transfer the sample to the preweighed, labeled centrifuge tube and seal. Reweigh and determine the sample mass by difference. Measure the volume of the total sample using the graduations on the centrifuge tube. Begin the settling rate measurements on a Monday, Tuesday, or Wednesday and no later than 1 h after the start of the working day. Record the start time and the times that all succeeding measurements are taken. Allow the solids in the sample to settle and measure the total sample and the solids volumes. The time between settling measurements is dependent upon the settling rate of the sample. The cognizant scientist will determine the time period between measurements to obtain appropriate settling rate data. The solids and total sample volumes should be measured at time increments no longer than every two hours during a the working day. Also, measure the total sample and solids volumes at the end of the working day and at the beginning of the next working day. Measure every two hours until no change is observed between three successive measurements or until 3 days have transpired.

Volumes will normally be measured to ± 0.2 mL when using the 15 mL centrifuge cone and to ± 1 mL when using the 50 mL centrifuge cone. The recommended sample volumes are ≥10 mL for the 15 mL tube and ≥30 mL for the 50 mL tube.

Present the settling rate as the solids height or vol% solids versus time. The solids height will be calculated by multiplying the solids volume by the height of 1 mL. Calculate Vol% Settled Solids by dividing the solids volume by the total volume and multiplying by 100%.

Some samples may stick to the side of the centrifuge tube. Centrifuge these samples briefly to drive them to the bottom of the tube to measure the sample volume. If the centrifuging causes separation of the solids and liquid, shake or blend with a plastic rod or spatula or as directed by the COGNIZANT SCIENTIST before beginning the settling procedure. Reweigh the tube and sample and calculate the new sample weight by difference.

The upper solids level may be difficult to determine because of different solids settling rates. The solids volume will be defined as the uppermost volume in which solids are present or the solids-solution interface which is the point where the sample becomes opaque.

4.0 Procedure for Density or Specific Gravity Measurement

This procedure will be used to measure the density or specific gravity of sludges, solutions, and slurries. This procedure will refer only to the measurement of density. If the user desires specific gravity, divide the measured density by the density of the reference material.

4.1 Equipment and Materials

- Graduated Plastic Centrifuge with Cap
- Centrifuge
- Analytical Balance, 2-place (minimum sensitivity)
- Volumetric Flask (Option for Solutions)

4.2 Determination of Density

Transfer the sample to a preweighed, labeled centrifuge tube and seal. Reweigh and calculate the sample mass by difference. As an alternative to a new sample, the sample from 4.3.2 may be used. The sealed centrifuge tube will be centrifuged for 1 h at ~1000 gravities. Measure the total volume of sample using the graduations on the centrifuge tube.

Calculate the density by dividing the sample mass by the sample volume. The sample mass will normally be measured to ± 0.02 g and the sample volume will normally be measured to ± 0.2 mL for the 15 mL tube or ± 1 mL for the 50 mL tube. The recommended sample volumes are 10 and 30 mL for the 15 and 50 mL centrifuge tubes, respectively.

With sludge samples the solids will often slope because of the centrifuging, the volume of the sample will be the average of the high and low volumes observed for the sample.

An alternative method of determining a solution's density will be to transfer a sample into a preweighed, labeled volumetric flask. Determine the sample mass by difference. This alternative method is preferable to the use of a graduated centrifuge tube because of increased accuracy for the volume measurement. The volume of a volumetric flask will normally be known to ± 0.04 mL.

5.0 Procedure for Determination of Vol%, Wt% and Densities of Centrifuged Solids and Supernate

This procedure will be used for determining the vol%, wt%, and the densities of centrifuged solids and centrifuged supernates for sludges and slurries.

5.1 Equipment and Materials

- Graduated Plastic Centrifuge Tube with Cap
- Clinical Centrifuge
- Analytical Balance, 2-place (minimum sensitivity)
- graduated cylinder
- Transfer Pipet and tips

5.2 Determination of Vol%, Wt%, and Densities of Centrifuged Solids and Supernate.

Transfer sample to a preweighed, labeled centrifuge tube and reweigh. Calculate the mass of the sample by difference. The sample and tube from Part4.2 may be used as the centrifuged sample. Centrifuge the sample for 1 h at maximum speed on the centrifuge. Record gravities apply. Measure the total sample and solids volume using the centrifuge tube graduations. The recorded solids volume will be the average of the high and low solids volume measurements. The supernate volume is the total sample volume less the solids volume. Volume is normally measured to ± 0.2 or ± 1 mL for the 15 and 50 mL centrifuge tubes, respectively.

The vol% centrifuged solids is the solids volume divided by the total volume multiplied by 100%. The vol% centrifuged supernate is 100% - vol% centrifuged solids.

To determine the wt% centrifuged solids, decant or transfer with a pipet the centrifuged supernate into a labeled, preweighed graduated cylinder. Measure the mass of the centrifuge tube and solids. Calculate the centrifuged solids mass by difference. Measure the volume of the supernate in the graduated cylinder and

weigh the recovered centrifuged supernate andgraduated cylinder. Calculate the mass of the centrifuged supernate by difference.

The wt% centrifuged solids is the solids' mass divided by the total sample mass multiplied by 100%. The wt% centrifuged supernate is the mass of the supernate divided by the total sample mass multiplied by 100%. The centrifuged solids density is the mass of the solids divided by the solids volume and the centrifuged supernate density is the supernate's mass divided by its volume.

6.0 Procedure for Determination of Wt% Total Solids, Wt% dissolved Solids, and Wt% Total Oxides.

This procedure will be used for determining the wt% total solids, wt% dissolved solids, and wt% total oxides in solutions, sludges, and slurries.

6.1 Equipment and Materials

- Ceramic Crucibles
- Glass Vials
- Analytical Balance, 2-place
- Desiccator Containing Dry Indicating Desiccant
- Muffle Furnace (100 to >1000°C)
- Drying Oven

6.2 Determination of Wt% Total Solids, Wt% Dissolved Solids, and Wt% Total Oxides.

Transfer sample to a preweighed, labeled crucible or vial and weigh the sample in the vial or crucible. Use crucibles for samples for which total oxides will be determined and use vials for samples for which only wt% solids are to be determined. Calculate the sample mass by difference. Allow the sample to air dry at least overnight to remove free liquid and to prevent splattering in the oven.An alternate method for removing free liquid, such as using a heat lamp, may be designated by the COGNIZANT SCIENTIST. When the free liquid has evaporated, transfer the sample and container to a 105 ± 5°C drying oven or furnace. Hold at temperature for 24 h. Remove the dried samples and containers and place in the desiccator to cool to room temperature. It should take at least 1 h to cool the sample. Reweigh the dried sample and container. Determine the residual sample weight by difference. The mass of the sample will normally be measured to ± 0.02 g.

The wt% total solids is the residual mass divided by the initial sample mass multiplied by 100%. For solutions the wt% dissolved solids is the residual mass divided by the initial sample mass multiplied by 100%. For slurries or sludges with interstitial

solutions that are recoverable using centrifugation, the wt% dissolved solids is the weight fraction dissolved solids in the centrifuged supernate times the weight fraction centrifuged supernate in the sludge/slurry determined using the procedure found in Section5 times 100%.

Place the sample and crucible from the wt% total solids procedure into the furnace. Do not perform this procedure with the glass vials. Heat the furnace to >1000°C and hold for 30 min between 1000 and 1050°C. Turnoff the furnace and cool to 150°C. Transfer the crucibles using tongs to the desiccator and cool to room temperature. It should take 1 to 2 h to cool the sample. Once cool weigh the sample and crucible. Determine the residual oxide mass by difference. Consider that volatile elements may also be lost.

The wt% total oxide is the residual oxide mass divided by the initial sample mass multiplied by 100%.

7.0 Procedure for Determining pH

This procedure will be used to determine the pH of a solution or slurry.

7.1 Equipment and Materials

- pH Meter and Electrode
- pH 4, 7 and 10 Buffers
- Plastic beakers or vials

7.2 Determination of pH

Standard Laboratory procedure will be used to measure the pH of solutions or slurries. Prior to use daily calibration of the pH meter and electrode will be performed using a two or three standard method. The temperature of the standards and samples will be taken into account. The electrode calibration will be done using pH buffers that are traceable to the manufacturers lot number. The expiration date for the buffers used in the check must not have lapsed. The electrode will be prepared for use and stored in accordance with the manufacturer's directions.

Sufficient sample will be added to a beaker or vial to cover the sensing portion of the electrode. Remove the probe from the storage solution, rinse with deionized (DI) water, and blot dry with a tissue. Insert the probe into the sample. Once the reading has stabilized, record the pH value and rinse the electrode with DI water. After the pH for the last sample has been determined, rinse and blot dry the electrode, and return it to its storage fluid.

The pH meter must have an accuracy of ±0.02 pH units. Data will normally be reported to the nearest 0.1 pH unit.

8.0 Procedure for Determination of Shear Stress vs Shear Rate and Yield Stress

This procedure will be used for determining the shear stress versus shear rate, yield stress, and shear strength for solutions, sludges, and slurries.

8.1 Equipment and Materials

- Viscometer
- Certified Newtonian Viscosity Standards
- Constant Temperature Bath/Circulator

8.2 Performance Requirements

Performance checks of the viscometer will be performed monthly or within approximately 30 days of measurement using certified Newtonian viscosity standards traceable to the manufacturer's lot number. The viscometer used must have a demonstrated accuracy of ± 15% at apparent viscosity measurements less than 10 CP and ± 10% at apparent viscosity measurements greater than 10 CP.

The data will be generated following the instructions provided in the instrument's Instruction Manual. The data will be in the form of a rheogram or flow curve, which is a plot of shear stress as a function of shear rate.

8.3 Measurement of Shear Stress vs Shear Rate and Yield Stress

Select and install the Sensor. For measurements at elevated temperatures, adjust the temperature setting for the constant temperature circulator to the desired level. Install the temperature control shroud. Transfer the sample by weight into the viscometer beaker. If the MV I Rotor is used transfer 40 mL. If the MV II Rotor is used transfer 55 mL. The weight transferred equals the density times the volume. After the coolant in the shroud reaches the desired temperature, stir the sample, and install the sample and beaker in the viscometer. Allow the sample to remain in position for 5 min to bring the sample to temperature unless instructed otherwise by the cognizant scientist. Obtain a rheogram. Adjust the temperature controller to the next temperature. Once the temperature of the coolant in the shroud reaches the desired level, allow the sample to equilibrate for 5 min before obtaining a rheogram. Repeat as necessary to obtain rheograms at the temperatures of interest.

It is important that the time between installation of the sample and beaker in the viscometer and the acquisition of the rheogram be as short as possible to minimize the effects of any solids settling.

To calculate the apparent viscosity, measure the shear stress (from the y axis) of the sample at a selected shear rate (from the x axis) and divide the shear stress by shear rate.

The fluid yield stress is obtained from the rheogram. A rheogram for a material with a yield stress will have two portions to it. The first appears as a nearly vertical line beginning at the origin and running up the ordinate. This line forms an angle, ß, with the ordinate. This portion of the rheogram is recording the behavior of the material as it acts like a solid or gel. When sufficient force is transmitted to the material to break the gel or make it yield, the rheogram will angle sharply to the right, and from then on the behavior of the material as a fluid will be recorded. This sharp angle that is recorded as the material becomes fluid is the yield point. The height of this break measured in Pascals along the ordinate is the value of the material's yield stress.

Some samples exhibit a creeping transition from solid to liquid behavior. this creep is recorded as a rounded yield point that is more difficult to identify. In cases where creep is exhibited, the yield point is defined as the intersection of a tangent drawn along the solid portion of the rheogram with a tangent drawn along the fluid portion of the rheogram.

Yield stress data is normally reported with a degree of precision of ± 0.1 Pa.

9.0 Procedure for Determination of Shear Strength

This procedure will be used to measure the shear strength of sludge samples. Shear strength is a semi-quantitative/qualitative/relative measure of the force that is required to move the material. No calibration standards are currently available to calibrate the shear vane.

9.1 Equipment and Materials

- Viscometer
- Shear Vane

9.2 Measurement of Shear Strength

Install shear vane into the viscometer. Position sample beneath the shear vane. Raise the sample slowly using the lab jack, centering the shear vane in the sample. Submerge the vane with at least 3 mm of sample above and below the vane. Based on the appearance of the sample, the COGNIZANT SCIENTIST or ANALYST will select the stress scale and the shear rate. Obtain the rheogram. Note: Shear Strength is dependent on sample history. The sample should be disturbed as little as possible before measurement or should be allowed to age for at least 2 days before the shear strength is determined.

The size and geometry of the sample is important when measuring shear strength. Try to select a sample geometry with a diameter of at least 4 shear vane diameters and a depth such that there is at least 3 mm of sample above and below the shear vane. The shear vane should be located at least two shear vane diameters away from the container walls.

The rheogram should show a peak at the beginning, level off, and finally drop off. The shear strength is calculated by taking the peak torque at a fraction of the full-scale reading on the chart, multiplying by $\%\tau$, multiplying by the full scale τ, and dividing by the surface area of the shear vane fins. The shear strength is reported in dynes/cm^2.

10.0 Quality Assurance

Work performed using this technical procedure will meet Impact Level II requirements as described in PNL-MA-70. All data generated using this procedure will be recorded on test instructions or directly in a LRB. The completed test instructions will later be entered into the appropriate LRB.

Changes to the test instructions can be made only by Cognizant Scientist and will be documented by crossing out the original information with a single line, and recording, initialling, and dating the changes. Major changes will be made by revision of the test instruction.

PNL Operating Procedure

Org. Code: D9T41
Procedure No.: PNNL-ALO-504
Rev. No.: 2

Title: PERCENT SOLIDS DETERMINATION OF SOILS/SLUDGES/SOLIDS

Work Location: 325 Building -- General

Page 1 of 6

Author: RT Steele

Effective Date: 07/14/97
Supersedes Date: 09/26/90

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

Was a Procedure Worksheet Completed?
__ Yes X No

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

Signatures:

Author: Robert L. Steele — RT Steele (Signature) — 7/21/97 (Date)

Technical Reviewer: MW Urie — MW Urie (Signature) — 7/21/97 (Date)

Approval:

Line Manager: KJ Kuhl-Klinger — KJ Kuhl-Klinger (Signature) — 8/22/97 (Date)

Concurrences, as appropriate:

Building Manager: P. S. Date — (Signature) — 8-8-97 (Date)

Health & Safety: [illegible] — (Signature) — 7/28/97 (Date)

Quality Programs: TL Almeida — TL Almeida (Signature) — 8/18/97 (Date)

Radiological Control: [illegible] — (Signature) — 8/13/97 (Date)

(Other) — (Signature) — (Date)

PNL Operating Procedure	Org. Code: D9T41 Procedure No.: PNNL-ALO-504 Rev. No.: 2
Title: PERCENT SOLIDS DETERMINATION OF SOILS/SLUDGES/SOLIDS	

PURPOSE/SCOPE

This procedure provides a uniform method for determining the weight percent solids fraction of samples. The methodology is consistent with the Contract Laboratory Program (ILM02.1) percent solids determination method.

APPLICABILITY

This procedure applies to solid, soil, sludge, slurry, and mixed-phase aqueous samples that are stable after drying to 105 °C.

HAZARD ASSESSMENT/HAZARD MITIGATION SUMMARY

Typically, the sample materials analyzed are chemically hazardous (e.g., corrosive, carcinogenic, toxic, etc.) and/or radioactive. When handling radioactive materials, the principles of ALARA shall be applied to minimize exposure and precautions taken to mitigate the spread of radioactive contamination. When hazardous materials are handled, laboratory staff are alerted to the potential hazards via the PNNL Sample Information Check List. Personnel protective clothing shall be specified in the applicable Chemical Safety Protocol or Radiological Work Permit (RWP).

Extreme care must be taken when removing the hot sample containers from the drying oven to avoid burns or sample spills. The use of tongs is mandatory when removing containers from the drying oven.

EMERGENCY RESPONSE

Emergency response actions can be initiated by 1) the activation of an emergency signal such as a fire gong, evacuation siren, or radiation detection/monitoring device alarms, 2) loss of building electrical power, and 3) from an unforeseen accident during sample handling or analysis activities. In all cases, proceed in accordance with the Building Emergency Procedure. Emergency response actions shall be reported to the Single Point of Contact at 375-2400. Notify Line Management immediately of emergency actions taken.

PREREQUISITES

Laboratory staff shall be trained to this procedure, prior to conducting any of the activities described herein.

Based on the samples handled (e.g., radioactive or non-radioactive, hazardous or non-hazardous), one or more of the following prerequisite training classes may be required:

- Laboratory Standard Hazard Communication
- Radiological Worker I or Radiological Worker II
- Laboratory Fume Hood Safety (including Radiological Fume Hood Safety)
- Safe Operating Procedure (SOP) "Routine Management, Storage, and Disposal of Hazardous, Low-Level Radioactive, or Mixed Waste", GEN-325-WM1

PRECAUTIONS AND LIMITATIONS

This procedure is applicable to determining weight percent solids on drying, or percent weight loss on drying (i.e., 100 - weight percent solids). In general, this procedure is not applicable to the determination of water content, since any component volatile at 105 °C can not be differentiated from water and all water may not be released at a temperature of 105 °C. However, with Cognizant Scientist approval, this procedure may be used to evaluate the absorbed moisture content (moisture loss on drying) for applicable sample of known chemical composition.

QUALITY CONTROL INSTRUCTIONS

Sample weights stated in this procedure may be varied by the Cognizant Scientist. This option is especially useful when the quantity of sample is limited or ALARA principles apply. Use of a sample data sheet or Analytical Chemistry Laboratory Record Book (ACL RB) is required for recording sample data and analyst's comments. A weight percent solids determination on a sample duplicate shall be performed with each analytical batch of samples. An analytical batch may range in size from 1 to 20 analytical samples. Calibration data for all Measuring and Test Equipment used in this procedure (i.e., balance and oven thermometer/thermocouple) must documented and retrievable.

EQUIPMENT AND REAGENTS

- Analytical Balance capable of weighing to at least 0.001 g.
- Drying Oven capable of maintaining a temperature of 105 $\pm$ 2 °C.
- Weighing Containers (e.g., porcelain crucibles, glass beakers)
- Lids for Weighing Containers (optional)
- Desiccator

WORK INSTRUCTIONS

1.0 PERCENT SOLIDS DETERMINATION

A sample is weighed into tared containers, dried at 105 °C for 12 to 24 hours, and reweighed to determine the solids fraction of the original sample. Weight data is recorded on a sample data sheet (or ACL RB).

1.1 Thoroughly mix the sample to be analyzed; mixing can be either manual or mechanical.

1.2 Label and weigh the weighing container to be used for the determination. Record this container tare weight (CW_c) to nearest 0.001 gram on a sample data sheet (See Exhibit 1 for example). Note: Be sure to include one duplicate analysis per each analytical batch.

1.3 Add sample to be analyzed to the weighing container, and weigh. Record this wet gross weight (GW_w) to the nearest 0.001 grams on the data sheet.

> Typical sample size is 5 to 10 grams. However, sample availability may limit the quantity of sample to be analyzed to less than 5 grams. When less than 2 grams of sample are available, consult the Cognizant Scientist prior to proceeding with the analysis.

1.4 Repeat Steps 1.1 through 1.4 until all samples being processed in the analytical batch have been weighed, then continue with Step 1.5.

1.5 Place the samples (i.e., entire analytical batch) into a drying oven maintained at 105 ± 2°C. Record the date, time, and oven temperature at the time the samples are placed into the oven. Note: When used, weighing container lids must be tipped to allow evaporating moisture to escape.

1.6 Maintain the samples at 105 ± 2 °C for 12 to 24 hours.

1.7 Remove the dried samples from the oven, and record date, time, and temperature on the data sheet. Immediately transfer the samples to a desiccator, close the desiccator, and allow samples to cool.

1.8 Once cool, remove a sample from the desiccator (close desiccator) and weigh immediately. Record this dry gross weight (GW_d) to the nearest 0.001 gram on the data sheet. Repeat until all samples have been weighed.

When obtaining the dry gross weight, an unstable balance reading is indicative of:

- Attempting to obtain the weight before sample has reached ambient temperature. This condition results in a trendless, unstable balance reading. If this condition occurs, return the sample to the desiccator for 20 minutes, then repeat Step 1.8. Repeat this process until two consecutive weight readings are within 0.003 gram.

- The sample being hygroscopic. This condition results in a steadily increasing balance reading. If this condition occurs, return the sample to the drying oven, dry for 1 hour, cool, and reweigh immediately. Repeat this process until two consecutive weight readings are within 0.003 gram. If the sample absorbs moisture too rapidly to obtain a stable reading, consult Cognizant Scientist.

2.0 CALCULATIONS

$$\text{Wt\% Solids} = \frac{(GW_d - CW_t)}{(GW_w - CW_t)} * 100$$

Where,

Wt%	=	Weight Percent Solids
GW_d	=	Dry Gross Weight (Step 1.8)
GW_w	=	Wet Gross Weight (Step 1.3)
CW_t	=	Container Tare Weight (Step 1.2)

SOURCE REQUIREMENTS

Quality Assurance Plan for Activities Conducted by the Analytical Chemistry Laboratory (ACL), MCS-033, (Section 8)

Quality Assurance, PNNL-MA-70, 1997, (PAP-70-1201).

Hanford Analytical Services Quality Assurance Requirement Documents, Volume 4, (Section 4.0).

USEPA CLP SOW for Inorganic Analyses, Document Number ILM02.0 (1990), ILM02.1 (1991). Exhibit D, Part F.

EXHIBITS/ATTACHMENTS

Exhibit 1: "Wt% Solids Data Sheet"

Pacific Northwest National Laboratory
Analytical Chemistry Laboratory

WT% SOLIDS DATA SHEET

CLIENT: _____ WORK PACKAGE: _____ ASR/LOI/TI: _____

QA PLAN: _____ IMPACT LEVEL: _____ PROCEDURE NO: PNNL-ALO-504

SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	CONTAINER TARE WEIGHT (CW_t)	GROSS WET WEIGHT (GW_w) [Wet Sample + Container]	GROSS DRY WEIGHT (GW_d) [Dry Sample + Container]	WEIGHT % SOLIDS

EXAMPLE

Alternate forms may be used, provided that the content and method of processing is consistent with this procedure.

$$\text{WT\% SOLIDS} = \frac{(GW_d - CW_t) * 100}{(GW_w - CW_t)}$$

DATE/TIME IN: _____ OVEN TEMPERATURE: _____ °C

DATE/TIME OUT: _____ OVEN TEMPERATURE: _____ °C

THERMOCOUPLE ID: _____ M&TE ID: _____

Analyst: _____ Date: _____ Reviewer: _____ Date: _____

EXHIBIT 1 - WT% SOLIDS DATA SHEET

RECORD COPY

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor: MW Thie
Signature
Print Name
Phone _____ MSIN _____

PNNL Project #: 42040
Charge Code: F22616 + MULTIPLE
Date Required: 3-19-01

Matrix Type Information

- **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- **Solids:** ___ Soil X Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # _____
- **Disposition of Treated Samples:**
  Dispose X Return _____

QA/Special Requirements

- **QA Plan:**
  SBMS _____
  HASQARD (CAWSRP) X
- **Additional QA Requirements?** No _____
  or Reference Doc # YES TWRS 34 4 ONSR
- **Field COC?** No ✓ Yes _____
- **Lab COC Required?** No ✓ Yes _____
- **Hold Time:** None ✓
  or RCRA _____ CERCLA _____
  Other, Specify _____
  & Date Sampled _____
  Time Sampled _____
- **Special Storage Requirements:**
  None ✓ Refrigerate (4°C) _____
  or Other, specify _____
- **Data Quality Review Required?** No ✓ Yes _____

Waste Designation Information

- **Sample Information Check List Attached?** Yes _____
  or Reference Doc #
  or Previous ASR # 6011.00 AS
  or Previous RPL ID #

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

Additional or Special Instructions _____

same original

Send Report To MW Thie **Phone** 6-9454
Phone _____

Preliminary results requested, as available? No X Yes _____ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: 12/20/00
Delivered By (optional) ~~[illegible]~~ HLRF
Time Delivered (optional) _____
Group ID (optional) _____
CMC Waste Sample? No X Yes _____
Cost Estimate, if requested: $ _____

Received By: SAL

ASR Number: 6019.02
RPL Numbers: 01-00429 & 430

RPG/CMC Work Accepted By: MW Thie **Signature/Date:** MW Thie 2-9-01

RTS, OTF

Special Instructions: ASR 6019 Rev 0.02
As Received Analyses for AN-102 Supernatant Sample 01-00429 and Centrifuged Solids Sample 01-00430

AN-102 Supernatant Sample 01-00429 – LIMS Tests			
Density/Solution	SAL	-- Use F22618	
Wt% Solids/TDS	SAL	-- Use F22618	
Ext-Solvent (for PCBs)	SAL	-- Use F22618	
PCB/Pesticides	ORG	-- Use F22620	
Digestion-128	SAL	-- Use F22618	
ICP-211-CMC	LAB	-- Use CMC K88407	
ICP/MS	ADV INORG	-- Use F22619	
GEA-381/474-CMC	RAD	-- Use CMC K88414	
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416	
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412	
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411	
U/KPA-4014-CMC	RAD	-- Use CMC K88423	
Sr-90-476/408-CMC	RAD	-- Use CMC K88419	
Sub-Sample	SAL	-- Use F22618	
IC-212-CMC	LAB	-- Use CMC K88406	
Tc99-432/408-CMC (w/o oxid.)	RAD	-- Use CMC K88421	
TOC/TIC-381-CMC	LAB	-- Use CMC K88409	
TOC/TIC-380-CMC	LAB	-- Use CMC K88409	
NH3-ISE	LAB	-- Use F22619	
Hg-131/201-CMC	LAB	-- Use CMC K88408	
OH-/Titration-228-CMC	LAB	-- Use CMC K88426	
Chelators	ORG	-- Use F22620	
Acids/Organic	ORG	-- Use F22620	
Phosphates/Organic	ORG	-- Use F22620	
AN-102 Centrifuged Solids Sample 01-00430 – LIMS Tests			
Wt% Solids/Total (after phase separation)	SAL	-- Use F22618	
Ext-Solvent (for PCBs)	SAL	-- Use F22618	
PCB/Pesticides	ORG	-- Use F22620	
Digestion-129	SAL	-- Use F22618	
ICP-211-CMC	LAB	-- Use CMC K88407	
ICP/MS	ADV INORTG	-- Use F22619	
GEA-381/474-CMC	RAD	-- Use CMC K88414	
Alpha/Gross-4001/408-CMC	RAD	-- Use CMC K88416	
Am,Cm/AEA-417/422-CMC	RAD	-- Use CMC K88412	
Pu/AEA-417/422-CMC	RAD	-- Use CMC K88411	
Pu241-417/474-CMC	RAD	-- Use CMC K88417	
U/KPA-4014-CMC	RAD	-- Use CMC K88423	
Sr-90-476/408-CMC	RAD	-- Use CMC K88419	
Fusion-116	SAL	-- Use F22618	
ICP/MS (Pt,Pd,Rh,Ru only)	ADV INORG	-- Use F22619	
Fusion-114 (For Iodine)	SAL	-- Use F22618	ASR Rev .02
ICP/MS (I-127, I-129 only)	ADV INORG	-- Use F22619	ASR Rev .02
Leach/Water-103	SAL	-- Use F22618	
IC-212-CMC	LAB	-- Use CMC K88406	
NH3-ISE	LAB	-- Use F22619	
H3-418/474-CMC	RAD	-- Use CMC K88422	
Sub-Sample	SAL	-- Use F22618	
TOC/TIC-381-CMC	LAB	-- Use CMC K88409	
TOC/TIC-380-CMC	LAB	-- Use CMC K88409	
CN/Total	LAB	-- Use F22619	
C14-381/474-CMC	RAD	-- Use CMC K88410	
Hg-131/201-CMC	LAB	-- Use CMC K88408	

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature: MW Thrasher
Print Name: _____
Phone _____ MSIN _____

PNNL Project #: 42365
Charge Code: W5791Z
Date Required: 6-15-01

Matrix Type Information

- ♦ **Liquids:** ___ Aqueous ___ Organic ___ Multi-phase
- ♦ **Solids:** ___ Soil ___ Sludge ___ Sediment
  ___ Glass ___ Filter ___ Metal
  ___ Smear ___ Organic ___ Other
- ♦ **Other:** ___ Solid/Liquid Mixture, Slurry
  ___ Gas ___ Biological Specimen

If sample matrices vary, specify on Request Page

QA/Special Requirements

- ♦ **QA Plan:**
  SBMS _____
  HASQARD (CAWSRP) _____
- ♦ **Additional QA Requirements?** No _____
  or Reference Doc # _____
- ♦ **Field COC?** No _____ Yes _____
- ♦ **Lab COC Required?** No _____ Yes _____
- ♦ **Hold Time:** None _____
  or RCRA _____ CERCLA _____
  Other, Specify _____
  & Date Sampled _____
  Time Sampled _____
- ♦ **Special Storage Requirements:**
  None _____ Refrigerate (4°C) _____
  or Other, specify _____
- ♦ **Data Quality Review Required?** No _____ Yes _____

Disposal Information

- ♦ **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc # _____
- ♦ **Disposition of Treated Samples:**
  Dispose _____ Return _____

Waste Designation Information

- ♦ **Sample Information Check List Attached?** Yes _____
  or Reference Doc # _____
  or Previous ASR # _____
  or Previous RPL ID # _____

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

Additional or Special Instructions _____

Send Report To _____ **Phone** _____

_____ **Phone** _____

Preliminary results requested, as available? No ____ Yes _____ (requesting preliminary results may increase cost)

Receiving and Login Information *(to be completed by laboratory staff)*

Date Delivered: _____
Delivered By (optional) _____
Time Delivered (optional) _____
Group ID (optional) _____
CMC Waste Sample? No _____ Yes _____
Cost Estimate, if requested: $ _____

Received By: _____

ASR Number: 6019.03
RPL Numbers: 01-00429

RPG/CMC Work Accepted By: MW Thrasher **Signature/Date:** MW Thrasher 5-29-01

MW, JAC

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Lab Staff Use Only RPL ID #	Client Sample ID	Sample Description (& Matrix, If Varies)	Analysis Requested (1)	Lab Staff Use Only Test	Library
01-0429	AN-102 SUPAR		Add Succinic ~~Add~~ Acid and EDTA TO ORGANIC LIST Table 2 MRQs set at 1.5E3 mg/L		

(1) See "Analysis Requested" Instructions: Provide analytes of interest and required detection levels. [Information provided: Above ____ ; On Attachment ___]

ASR # 6019.03

Page _____ of _____

Addendum to ASRs 6019 and 6031

Special Instructions for Ammonia Spike Preparation, Ammonia Sampling, and Ammonia Sample Preservation

2/27/01

Spike Level

Previous analyses of Hanford tank waste samples for ammonia have come out around 10^{-5} to 10^{-6} molar in ammonia. The detection limit is around 10^{-6} molar for a 0.2-mL sample size (direct ISE measurement, sample aliquot diluted to 5 mL). A matrix spike must be at least ten times the detection limit and should at least double the sample ammonia concentration. The most convenient volume of spike for the hot cell technician to measure is 1 mL.

We'll make the spike solution around 10^{-4} molar. That way, a 1-mL spike added to a 0.2-mL sample will be 10 to 100 times the typical sample ammonia concentration, and comfortably above the detection limit. The calculated spike recovery will be accurate.

Spike Preparation

NH_4Cl weighs 53.491 g/mole

1. Weigh a 100-mL vol flask empty. Weigh out about 5.3491 g of NH_4Cl (weighed to the nearest 0.1 mg) and rinse it completely into the 100-mL vol flask with deionized water. Let the NH_4Cl dissolve completely, then fill the flask to the line. Weigh the flask full, and calculate the density. Calculate the actual molarity of the solution.

2. Make a 1:100 dilution of this standard, using deionized water, by weight and volume. (This makes 10^{-2} molar NH_3.)

3. Make another 1:100 dilution, but before adding water up to the line, add 1 mL of concentrated H_2SO_4. (This makes 10^{-4} molar NH_3, preserved to about pH 0.5.)

4. In the spike prep record, include the NH_4Cl manufacturer and lot number, balance number, pipet ID and checks, and date of preparation.

Provide to the hot cell staff about 15 mL of the 10^{-4} molar NH_3 solution in a 20-mL liquid scintillation vial. (The hot cell sample preparation doesn't need that much, but the ammonia standard must be stored with minimal headspace to avoid concentration by evaporation.)

Sample Preservation for Ammonia

Hanford tank supernatant samples are typically around 1 to 2 molar in $NaOH + Na_2CO_3$, or even higher. The samples must be preserved by acidification before spiking, or part of the spike will immediately be lost.

A 0.2-mL aliquot has 0.2 to 0.4 millimoles of base. This requires about 2 mL of 0.2M H_2SO_4. To assure that the samples are acidic, we'll use 5 mL of 0.2 M H_2SO_4 per 0.2 mL of raw supernatant.

The same amount of acid preservative will be adequate for tank solids, which normally have less base than the supernatant.

The following instructions replace the usual water leach procedure for ammonia. The usual water leach procedure has no provision for sample preservation, and ammonia would be lost during sample handling and the leaching process. The instructions below will avoid such loss of ammonia. After the samples are measured into dilute sulfuric acid as directed below, no further water leach is necessary, and the sample solutions will be immediately ready for ISE ammonia analysis.

Instructions for Subsampling for Ammonia

1. Outside the hot cells, label a vial for each sample, duplicate, blank, matrix spike, matrix spike duplicate, and reagent spike. Each batch consists of:

ASR 6019.03	ASR 6031.01
Process blank	Process blank
01-0429	01-0520
01-0429 Dup	01-0520 Dup
01-0430	01-0520 MS
01-0430 Dup	01-0520 MSD
01-0429 MS	Reagents-only spike
01-0429 MSD	
Reagents-only spike	

2. To each vial, add 5 mL of 0.2M H_2SO_4, accurately measured. On the bench sheet, record the actual volume of H_2SO_4 used.

   (The molarity of the H_2SO_4 is not critical, but should be within about 20% of 0.2M.)

3. Cap the vials and take them into the hot cell. Keep the vials upright, so that no liquid touches the cap.

4. Keep each vial capped until it is needed. Measure the homogenized sample into the vial, by weight or volume. Use about 0.2 mL of each supernatant sample, and 0.2 g of the solid sample. Record the weight or volume on the bench sheet as usual, according to procedure.

   Cap each vial as soon as the sample is added.

5. Swirl each vial gently to mix its contents, then let the vials stand for several minutes.

6. **To each spike vial** (but not to the samples, duplicates, or blanks), add 1 mL of ammonia spike. Re-cap the spike vials. Measure the spike with an accurate pipet and record the exact volume used on the bench sheet.

7. Survey the vials out of the hot cell. The samples in the vials are preserved, and can wait up to two weeks before analysis.

Battelle PNNL/RPG/Inorganic Anlaysis ... ICPAES Data Report

Project / WP#: ~~42040~~ 42365 / F22626
ASR#: 6019 & 6025
Client: M. Urie
Total Samples: 4

	From	To
RPL#:	01-00429, -00430	01-00441, -00442
Client ID:	AN-102-SUP-AR, -CS-AR	AN-102-SUP-DF, -CS-DF

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 02-15-01 (A0645)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

Jerry L. Dagnan 7-6-01
Reviewed by

M W Urie 7-12-01
Concur

Two samples, "AN-102-SUP-AR" and "AN-102-SUP-DF" (RPL ID# 01-00429 and 01-00441), were processed by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-128 acid digestion of as-received and diluted-feed supernatant liquid. Approximately 1 mL of material (density 1.437 g/mL (supernatant liquid, as-received) and 1.159 g/mL (supernatant liquid, diluted-feed), prepared in duplicate; duplicate digestion process blanks (reagents only) and a blank-spike were processed and analyzed by ICPAES. Each sample aliquot was transferred to digestion vessel using a 1 mL pipet and weighed. Sample volumes were estimated by dividing sample aliquot weight by sample density. (Sample density data for wet centrifuged solids and supernatant liquids were determined from earlier sample measurements.) Following acid digestion each sample was diluted to approximately 25 mL and weighed. Final volume was estimated by dividing the final weight of prepared solution by its density. Density of solution was determined by weighing a 1 mL aliquot. ICPAES data is reported as µg/mL.

Also, two samples, "AN-102-CS-AR" and "AN-102-CS-DF" (RPL ID# 01-00430 and 01-00442), were processed by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-129 acid digestion of as-received (wet solids) and diluted-feed (dry solids) centrifuged material. Approximately 0.5 to 0.8 g of wet solids material (AN-102-CS-AR) was prepared in duplicate, duplicate digestion process blanks (reagents only), matrix-spikes (each sample), and blank-spike were processed and analyzed by ICPAES. After acid digestion each sample was diluted to a final volume approximately 25 mL and weighed. Approximately 0.22 to 0.29 g of dry solids material (AN-102-CS-DF) was prepared but reported as wet sample (µg/g-wet) based upon 35.1 wt% solids of original wet material. Sample aliquot and final volumes were weighed and volumes determined from estimated density measurements as noted above.

ICPAES results have been corrected for process dilution resulting from acid digestion of the supernatant and centrifuged solids material and analytical dilution during ICPAES analysis session. Wet solids and supernatant sample material was transferred to digestion vessels using a 1 mL pipette and weighed. Final sample volume was determined by dividing weight of final sample volume by estimated density. Density of final liquid was estimated by weighing a 1 mL aliquot of final diluted sample.

Analytes of interest and MRQ's listed in Table 1. Supernatant Analyses of Special Instructions include the following: Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Na, Ni, P, Pb, and U. Analytes of interest and MRQ's listed in Table 2. HLW Solids Analysis include all of the analytes listed in Table 1, (except K and U) plus Ag, Bi, Cu, Mn, Nd, Pd, Rh, Ru, Si, Sr, Ti, Zn, and Zr. EQL limits, adjusted for sample preparation and analytical dilution, were generally below MRQ's except for potassium. MRQ in Table 1 for potassium is 75 µg/mL. However, potassium in the supernatant samples measured about 18,800 µg/mL, well above MRQ. The following analytes required for HLW solids (centrifuged solids) did not meet MRQ specifications: Ca, Cd, Cu, La, Na, Pd, Rh, and Ru. Data report lists EQL's for samples and MRQ specifications.

See attached ICPAES Data Report for final results.

Quality control check-standard results met tolerance requirements for all analytes of interest reported except where noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan.

Five fold serial dilution:

All analytes of interest were within tolerance limit of 10% after correcting for dilution except for the following: Calcium varied by about 25% to 50% in supernatant duplicate and matrix-spike samples (RPL# 01-00429) and by about 150% in the centrifuged solids sample (RPL# 01-00430). Barium differed by about 11% and sodium differed by about 20% between 10-fold and 2-fold dilution of centrifuged solids in matrix-spike sample (RPL# 01-00442-MS1).

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 15% RPD for all analytes of interest greater than EQL except for nickel (40%) and zirconium (51%) in centrifuge solids (DF) sample RPL#01-00442. The original and duplicate sample was not within specification limit of ≤ 3% RPD for sodium greater than EQL except in centrifuge solids sample RPL#01-00442 (1% RPD). Sodium RPD generally varied from 3.8% to 7.8%.

Post-Spiked Samples (Group A):

All analytes of interest were recovered within tolerance of 75% to 125% post-spike recovery.

Post-Spiked Samples (Group B):

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

All analytes of interest were recovered within tolerance of 80% to 120% except silver. Silver recovery was low (about 22%). Low recovery may be due to the amount of hydrochloric acid used to process the sample (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples. Potassium recovery was also low (72% to 75%) for both sample preparation procedures (PNL-ALO-128 and –129). No apparent reason for the discrepancy in known.

Matrix Spiked Sample:

All analytes of interest were recovered within tolerance of 75% to 125% except low recovery of silver (23%), and potassium (65%) in matrix-spiked supernatant sample RPL#01-00429. Recovery of silver (26% and 35%), calcium (49%), copper (129%), magnesium (131%), nickel (163%)

and zirconium (54%) exceeded tolerance limits in the matrix-spiked centrifuged solids. Low recovery for silver is likely due to the amount of hydrochloric acid used to process samples (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples. Low or over recovery of the other analytes may be due to non-homogeneity of the centrifuged solids (RPL#01-00442).

Quality Control Check Standards:

Concentration of all analytes of interest is within tolerance limit of ± 10% accuracy in the check standards QC_MCVA, QC_MCVB, and QC_SSTMCV with the following exceptions. Sodium (11% to 14%), silicon (10% to 12%), and potassium (11% to 17%) measurements of QC_MCVA exceed tolerance limit of 10%. Potassium (15%, 18%) and sodium (15% & 16%) was also high in check standard QC_SSTMCV. High recovery for these analytes may be due to sample carry-over and require longer clean-out time.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except for one of two measurements of potassium. One of the two potassium measurements was 13% high. All other analytes measured were within tolerance.

Process Blank:

Concentration of all analytes of interest is within tolerance limit of ≤EQL or less than ≤5% of the sample.

Laboratory Control Standard (LCS):

A blank-spike containing all analytes of interest was prepared and analyzed instead of an LCS (See "Blank Spike" above for details).

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	MRQ[1] (ug/g)	Multiplier= / RPL/LAB #= / Client ID= / Run Date= / (Analyte)	EQL (ug/g)	40.3 01-00430-PB1 Proc.Blank-1 (ALO-129) 2/15/2001 ug/g-wet	EQL (ug/g)	38.0 01-00430-PB2 Proc.Blank-2 (ALO-129) 2/15/2001 ug/g-wet	EQL (ug/g)	84.5 01-0430 @2 AN-102-CS-AR (ALO-129) 2/15/2001 ug/g-wet	EQL (ug/g)	100.2 01-0430-DUP @2 AN-102-CS-AR (dup) (ALO-129) 2/15/2001 ug/g-wet	EQL (ug/g)	63.6 01-00442 @2 AN-102-CS-DF (ALO-129) 2/15/2001 ug/g-wet	EQL (ug/g)	62.5 01-0442-DUP @2 AN-102-CS-DF (dup) (ALO-129) 2/15/2001 ug/g-wet
0.025	900	Ag	10.1	--	9.5	--	21.1	--	25.1	--	15.9	--	15.6	--
0.060	330	Al	24	--	23	[2.6]	51	28,000	60	29,000	38	58,100	37	53,100
0.250		As	101	--	95	--	211	--	251	--	159	[17]	156	--
0.050		B	20	50.1	19	60.2	42	89.8	50	97.1	32	59.8	31	54.3
0.010	600.0	Ba	4.0	--	3.8	--	8.5	26.9	10.0	28.0	6.4	78.6	6.2	78.6
0.010		Be	4.0	--	3.8	--	8.5	--	10.0	--	6.4	7.12	6.2	7.03
0.100	6000	Bi	40	--	38	--	85	--	100	--	64	[12]	62	[11]
0.250	180	Ca	101	--	95	--	211	434	251	407	159	441	156	405
0.015	11.0	Cd	6	--	6	--	13	37.4	15	38.5	10	20.0	9	19.6
0.200		Ce	81	--	76	--	169	[17]	200	--	127	[72]	125	[70]
0.050		Co	20	--	19	--	42	--	50	--	32	--	31	[3.1]
0.020	120	Cr	8	--	8	--	17	1,800	20	1,850	13	4,550	12	4,580
0.025	18	Cu	10	--	9	--	21	[19]	25	[19]	16	[11]	16	[13]
0.050		Dy	20	--	19	--	42	--	50	--	32	--	31	--
0.100		Eu	40	--	38	--	85	--	100	--	64	--	62	--
0.025	140	Fe	10	[4.9]	9	[1.2]	21	1,150	25	1,180	16	3,260	16	3,640
2.000		K	806	--	760	--	1690	[1,100]	2005	[1,100]	1271	[580]	1249	[550]
0.050	60	La	20	--	19	--	42	[32]	50	[32]	32	106	31	105
0.030		Li	12	--	11	--	25	--	30	--	19	[11]	19	[11]
0.100	450	Mg	40	--	38	--	85	[11]	100	--	64	[55]	62	[53]
0.050	300	Mn	20	--	19	--	42	254	50	261	32	764	31	765
0.050		Mo	20	--	19	--	42	[33]	50	[33]	32	[15]	31	[16]
0.150	150	Na	60	73.1	57	86.5	127	160,000	150	173,000	95	58,700	94	59,300
0.100	600	Nd	40	--	38	--	85	[65]	100	[64]	64	181	62	179
0.030	160	Ni	12	--	11	--	25	254	30	261	19	105	19	157
0.100	600	P	40	--	38	--	85	1,390	100	1,420	64	1,060	62	1,060
0.100	600	Pb	40	--	38	40.3	85	245	100	244	64	412	62	400
0.750	300	Pd	302	--	285	--	634	--	752	--	477	--	469	--
0.300	300	Rh	121	--	114	--	254	--	301	--	191	--	187	--
1.100	300	Ru	443	--	418	--	930	--	1103	--	699	--	687	--
0.500		Sb	201	--	190	--	423	--	501	--	318	--	312	--
0.250		Se	101	--	95	--	211	--	251	--	159	--	156	--
0.500	3000	Si	201	[120]	190	[130]	423	[190]	501	[210]	318	3,630	312	3,630
1.500		Sn	604	--	570	--	1268	--	1504	--	953	--	937	--
0.015	300	Sr	6	--	6	--	13	[6.1]	15	[6.2]	10	23.8	9	23.8
1.500		Te	604	--	570	--	1268	--	1504	--	953	--	937	--
1.000		Th	403	--	380	--	845	--	1002	--	636	[160]	625	[160]
0.025	150	Ti	10	--	9	--	21	--	25	--	16	[3.2]	16	[3.3]
0.500		Tl	201	--	190	--	423	--	501	--	318	--	312	--
2.000		U	806	--	760	--	1690	--	2005	--	1271	[330]	1249	[310]
0.050		V	20	--	19	--	42	--	50	--	32	--	31	--
2.000		W	806	--	760	--	1690	--	2005	--	1271	--	1249	--
0.050		Y	20	--	19	--	42	[9.4]	50	[9.5]	32	[26]	31	[26]
0.050	6	Zn	20	--	19	--	42	[21]	50	[23]	32	79.5	31	77.9
0.050	600	Zr	20	--	19	--	42	46.3	50	[47]	32	58.2	31	97.8

(1) MRQ's are for un-dissolved solids (Table 2 HLW Solids Analyses).
Measurement results are for un-dissolved solids plus interstitial liquids

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Det. Limit (ug/mL)	MRQ (ug/mL)	Multiplier= / RPL/LAB #= / Client ID= / Run Date= (Analyte)	EQL (ug/mL)	26.2 01-00429-PB1 Proc.Blank-1 (ALO-128) 2/15/2001 (ug/mL)	EQL (ug/mL)	26.3 01-00429-PB2 Proc.Blank-2 (ALO-128) 2/15/2001 (ug/mL)	EQL (ug/mL)	136.0 01-00429 @5 AN-102-SUP-AR (ALO-128) 2/15/2001 (ug/mL)	EQL (ug/mL)	52.3 01-00429-DUP @2 AN-102-SUP-AR-dup (ALO-128) 2/15/2001 (ug/mL)	EQL (ug/mL)	50.4 01-00441 @2 AN-102-SUP-DF (ALO-128) 2/15/2001 (ug/mL)	EQL (ug/mL)	49.9 01-00441-DUP @2 AN-102-SUP-DF-DUP (ALO-128) 2/15/2001 (ug/mL)
0.025		Ag	6.6	--	6.6	--	34.0	--	13.1	--	12.6	--	12.5	--
0.060	75	Al	16	[5.0]	16	[4.9]	82	12,100	31	12,400	30	5,390	30	5,280
0.250		As	66	--	66	--	340	--	131	--	126	--	125	--
0.050		B	13	42.9	13	43.8	68	77.8	26	81.8	25	51.5	25	48.0
0.010	2.3	Ba	2.6	--	2.6	--	13.6	--	5.2	--	5.0	--	5.0	--
0.010		Be	2.6	--	2.6	--	13.6	--	5.2	--	5.0	--	5.0	--
0.100		Bi	26	--	26	--	136	--	52	--	50	--	50	--
0.250	150	Ca	66	--	66	--	340	465	131	513	126	169	125	137
0.015	7.5	Cd	4	--	4	--	20	61.2	7.9	62.3	8	16.9	7	16.4
0.200		Ce	52	--	53	--	272	--	105	--	101	--	100	--
0.050		Co	13	--	13	--	68	--	26	[3.9]	25	--	25	--
0.020	15	Cr	5	--	5	--	27	216	10	213	10	69.9	10	68.2
0.025		Cu	7	--	7	--	34	[23]	13	23.9	13	[6.8]	12	[6.7]
0.050		Dy	13	--	13	--	68	--	26	--	25	--	25	--
0.100		Eu	26	--	26	--	136	--	52	--	50	--	50	--
0.025	150	Fe	7	[1.6]	7	--	34	37.6	13	37.3	13	[6.7]	12	[6.4]
2.000	75	K	525	--	525	--	2720	[2,000]	1047	1,960	1009	[540]	998	[550]
0.050	35	La	13	--	13	--	68	[16]	26	[15]	25	[3.2]	25	[3.3]
0.030		Li	8	--	8	--	41	--	16	--	15	--	15	[1.6]
0.100	300	Mg	26	--	26	--	136	--	52	--	50	--	50	--
0.050		Mn	13	--	13	--	68	[17]	26	[16]	25	--	25	--
0.050		Mo	13	--	13	--	68	[53]	26	52.0	25	[16]	25	[15]
0.150	75	Na	39	55.2	39	58.3	204	180,000	79	188,000	76	74,100	75	72,300
0.100		Nd	26	--	26	--	136	[33]	52	[30]	50	[6.2]	50	[6.7]
0.030	30	Ni	8	[0.96]	8	[0.89]	41	419	16	413	15	123	15	120
0.100	600	P	26	--	26	--	136	1,800	52	1,830	50	607	50	594
0.100	300	Pb	26	--	26	--	136	188	52	183	50	[49]	50	[49]
0.750		Pd	197	--	197	--	1020	--	393	--	378	--	374	--
0.300		Rh	79	--	79	--	408	--	157	--	151	--	150	--
1.100		Ru	289	--	289	--	1496	--	576	--	555	--	549	--
0.500		Sb	131	--	131	--	680	--	262	--	252	--	250	--
0.250		Se	66	--	66	--	340	--	131	--	126	--	125	--
0.500		Si	131	151	131	139	680	[290]	262	[180]	252	290	250	270
1.500		Sn	393	--	394	--	2040	--	785	--	757	--	749	--
0.015		Sr	4	--	4	--	20	[2.3]	8	[2.3]	8	--	7	--
1.500		Te	393	--	394	--	2040	--	785	--	757	--	749	--
1.000		Th	262	--	263	--	1360	--	523	--	504	--	499	--
0.025		Ti	7	--	7	--	34	--	13	--	13	--	12	--
0.500		Tl	131	--	131	--	680	--	262	--	252	--	250	--
2.000	600	U	525	--	525	--	2720	--	1047	--	1009	--	998	--
0.050		V	13	--	13	--	68	--	26	--	25	--	25	--
2.000		W	525	--	525	--	2720	--	1047	[130]	1009	--	998	--
0.050		Y	13	--	13	--	68	--	26	[2.8]	25	--	25	--
0.050		Zn	13	--	13	--	68	--	26	[6.2]	25	[3.3]	25	--
0.050		Zr	13	--	13	--	68	[8.4]	26	[7.9]	25	--	25	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

BLANK

Battelle, PNNL / AIAL
Inorganic Analysis / ICP-MS Data Report

Project / WP#: 42040 / W57911
ASR#: 6019
Client: Mike Urie
Total Samples: 2

RPL #	Client ID
01-00429	AN-102-SUP-AR
01-00430	AN-102-CS-AR

Procedure: PNL-ALO-280 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: WB36913 ICP/MS, VG Elemental
512-06-01-014 Mettler AJ100 Balance

Analyst: James P Bramson

Analysis		Analysis Date	Analysis Files		
			Experiment	Procedure	Element Menu
Supernatant					
Cs, Rb, Tc		3/27/01	27MAR01	010327a	CsRbTc
Actinides		4/4/01, 4/12/01	4APR01 12APR01	010404a 010412a	actinides
Wet Centrifuged Solids					
Acid Digestion	Mass 0-100	4/23/01, 4/25/01	23APR01 25APR01	010423a 0104255b	7-100
	Mass 100-200	4/19/01	19APR01	010419a	100-210
	I-127/129	4/27/01 4/30/01	27APR01 30APR01	010427a 010430a	iodine
	Actinides	4/3/01, 4/4/01 4/11/01	3APR01 4APR01 11APR01	010403a 010404a 010411a	UTh actinides
	U Isotopics	4/11/01	11APR01	010411a	100-210
Fusion	PMG Metals	3/28/01	28MAR01	010328a	NM
Fusion	I-127/129	4/27/01 4/30/01	27APR01 30APR01	010427a 010430a	iodine

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Thomas 8.23.01
Reviewed By

James Bramson 8/24/01
Concur

Two samples, As Received AN-102 Supernatant and Wet Centrifuged Solids, were analyzed on a radioactive material contained ICP/MS for the requested analytes.

1. Analysis

See attached ICP/MS data reports for final results and run order for the analytical batches. The final results have been corrected for all laboratory dilutions performed on the samples during analysis. Preparation blanks, blank spikes, duplicates, and matrix spikes were submitted with the samples. The analyses were separated into several analytical batches:

A. Supernatants
   - A.1. Cs, Rb, Tc
   - A.2. Actinides

B. Wet Centrifuged Solids
   - B.1. Acid Digestion
     - B.1.1. Mass 0-100
     - B.1.2. Mass 100-200
     - B.1.3. I-127/129
     - B.1.4. Actinides
     - B.1.5. Uranium Isotopics
   - B.2. Fusion - PMG Metals
   - B.3. Fusion - I-127/129

Lithium, beryllium, and boron are at the end of the detector range and the instrument response tends to have a wide variation in this region. Arsenic, selenium, and vanadium may also have a wide variation in the results due to the extensive chain of isobaric interference corrections. The calibration curves of four analytes had correlation coefficients < 0.995: lithium and I-127 (during the analysis of the preparation blanks), boron (during the analysis of the samples), and selenium (during the analysis of preparation blanks and samples).

2. Quality Control

Duplicates (DUP). Duplicates were submitted with the samples. In addition, replicate analyses were also performed. The QC acceptance criterion was an RPD of < 15%.

A. Supernatants
   - A.1. Cs, Rb, Tc. The duplicate (and replicate) analyses met the QC criterion.
   - A.2. Actinides. The duplicate (and replicate) analyses met the QC criterion.

B. Wet Centrifuged Solids
   - B.1. Acid Digestion
     - B.1.1. Mass 0-100. The duplicate QC criterion was not met for Li, Be, B, Co, and As. The replicate analysis did not meet the duplicate QC criterion for Li and B. Be and As are near the detection limit.
     - B.1.2. Mass 100-200. The QC criterion was not met for Sb in the duplicate and replicate analysis. This analyte was near the detection limit.

B.1.3. I-127/129. The QC criterion was met in both duplicate and replicate analysis.
B.1.4. Actinides. The QC criterion was met in both duplicate and replicate analysis.
B.1.5. Uranium Isotopics. The QC criterion was not met in the U-233 and U-236 duplicate analysis. The replicate analysis did not meet the duplicate QC criterion for U-233. For U-233, this may be due to the low count rate.
B.2. Fusion - PMG Metals. The QC criterion was met in both duplicate and replicate analysis.
B.3. Fusion - I-127/129. RPD values were not calculated since the concentrations were below detection level.

Matrix Spikes (MS). Matrix spikes were submitted with the samples. In addition post spike (PS) analyses were also performed.

A. Supernatants
A.1. Cs, Rb, Tc. The QC criterion of 70-130% was met in both MS and PS analysis.
A.2. Actinides. These analytes did not appear to have been spiked into the sample, however the PS met the MS QC criterion of 75-125%
B. Wet Centrifuged Solids
B.1. Acid Digestion
B.1.1. Mass 0-100. The MS did not meet the QC criterion met for Li and B. The PS did not meet the MS QC criterion for Li and Se.
B.1.2. Mass 100-200. The MS did not meet the QC criterion for W. The PS did not meet the MS QC criterion for Ce. The cause for the PS failure is unknown.
B.1.3. I-127/129. These analytes did not appear to be spiked into the samples. Although there is no QC acceptance criterion for these analytes, the PS was within 75-125%.
B.1.4. Actinides. The QC criterion was met.
B.1.5. Uranium Isotopics. These analytes did not appear to be spiked into the samples. No PS was performed.
B.2. Fusion - PMG Metals. The QC criterion was met.
B.3. Fusion - I-127/129. The MS and PS for I-129 was within 75-125%. However the PS for I-127 was 140%. The cause for the high recovery is unknown.

Preparation Blank (PB) and Blank Spike (BS). Two preparation blanks and a blank spike were submitted with the samples. In addition a post spike (PS) of one of the preparation blanks was performed.

A. Supernatants
A.1. Cs, Rb, Tc. The PBs were below detection level and the recovery for both BS and PS met the QC criterion of 70-130%.
A.2. Actinides. The PBs were below detection level. The analytes did not appear to be spiked into the BS, however the PS met the MS QC criterion of 75-125%.
B. Wet Centrifuged Solids
B.1. Acid Digestion

B.1.1. Mass 0-100. Most of the elements were below the detection level of the instrument except for B, V, As, and Y. Boron was well above the EQL and MRQ. Vanadium was above the EQL but below the MRQ. The arsenic was below the EQL. Yttrium was above detection level in PB2, but below the EQL. The BS did not meet the QC criterion for B. The PS did not meet the MS QC criterion for Li and Se.

B.1.2. Mass 100-200. All analytes were below the detection level except for Ta, which was slightly above the EQL but below the MRQ.

B.1.3. I-127/129. The PB was below detection level. These analytes did not appear to be spiked into the BS. Although there is no spike QC acceptance criterion, the PS was within 75-125%.

B.1.4. Actinides. The PBs were below detection level. The BS (and PS) met the spike QC criterion.

B.1.5. Uranium Isotopics. The PBs were below detection level. These analytes did not appear to be spiked into the BS. A PS was not performed.

B.2. Fusion - PMG Metals. The PBs were below detection level. The BS (and PS) met the spike QC criterion.

B.3. Fusion - I-127/129. The PBs were below detection level. The BS (and PS) were within 75-125%.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criterion of less than the estimated quantitation limit (< EQL) was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV).

A. Supernatants

A.1. Cs, Rb, Tc. The ICV/CCVs met the QC criterion except for the final CCV during the Tc analysis (89.7%), which was just outside the QC criterion of 90-110%.

A.2. Actinides. Some of the ICV/CCV QC criterion was not met for Pu-239 (87%), Pu-240 (81%, 81%) and AM-241 (89.8%, 89.8%, 81%) during the analysis of the PBs and for Pu-240 (115%, 83%) and Am-241 (112%) during the analysis of the samples. For Pu-240 and Am-241, the count rates are very small.

B. Wet Centrifuged Solids

B.1. Acid Digestion

B.1.1. Mass 0-100. Some of the ICV/CCV QC criterion were not met for B (113%, 112%), V (111%, 111%, 113%), As (115%), Se (119%-228%), Rb (114%), and Mo (110%-120%). For the selenium analysis, all of the samples including the spikes have selenium concentrations below the MRQ.

B.1.2. Mass 100-200. Some of the ICV/CCV QC criterion were not met for W (120%), Ta (89.5%, 80%), and Tl (113%).

B.1.3. I-127/129. The QC criterion was not met for the final CCV during the I-129 (87%) analysis of the samples.

B.1.4. Actinides. Some of the QC criterion were not met for Pu-239 (86%) and Pu-240 (82%, 81%) during the analysis of the PBs and Pu-239 (116%) during the analysis of the samples.

B.1.5. Uranium Isotopics. N/A

B.2. Fusion - PMG Metals. The CCVs did not meet the QC criterion for Pd (78%-88%).

B.3. Fusion - I-127/129. One of the CCVs did not meet the QC criterion during the analysis of the PBs for I-129 (89%). One of the CCVs did not meet the QC criterion during the analysis of the samples for I-127 (80%) and I-129 (87%).

Internal Standard (IS). The ISs met the QC criterion of 30% to 120%.

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019
Log-In: 01-00429
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [signature] 5/29/01

Analyst: [signature] 5/29/01

Unless otherwise specified, the results are reported in µCi analyte/ml of original sample for radioisotopes; µg analyte/ml of original sample for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/ml	Rb µg/ml ± 1SD	MDL µCi/ml	^{99}Tc µCi/ml ± 1SD	MDL µg/ml	^{133}Cs µg/ml ± 1SD
	1%HNO3		ICB		< 1.32E-06		< 6.91E-08		< 2.45E-06
	1%HNO3		CCB1		< 1.47E-06		< 7.70E-08		< 2.73E-06
	1%HNO3		CCB2		< 1.49E-06		< 7.78E-08		< 2.76E-06
	1%HNO3		CCB3		< 1.22E-06		< 6.40E-08		< 2.27E-06
	ICV		ICV1		2.01E-04 ± 2.11E-06		3.29E-06 ± 2.69E-08		1.94E-04 ± 1.29E-06
	CCV		CCV1a		2.01E-04 ± 3.10E-07		3.20E-06 ± 3.71E-08		1.87E-04 ± 2.57E-06
	CCV		CCV2a		2.10E-04 ± 5.36E-06		3.28E-06 ± 5.30E-08		1.93E-04 ± 2.12E-06
	CCV		CCV3a		2.13E-04 ± 2.74E-06		3.04E-06 ± 4.40E-08		1.85E-04 ± 6.43E-07
True Value					**2.00E-04**		**3.39E-06**		**2.00E-04**
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	1.20E-01	< 1.20E-01	6.29E-03	< 6.29E-03	2.23E-01	< 2.23E-01
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	1.09E-01	1.73E+01 ± 3.13E-01	5.73E-03	2.37E-01 ± 3.79E-03	2.03E-01	1.50E+01 ± 1.71E-01
Spike Concentration					2.04E-04		2.80E-06		1.77E-04
Spike Recovery					**102%**		**83%**		**88%**
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	1.28E-01	< 1.28E-01	6.71E-03	< 6.71E-03	2.38E-01	< 2.38E-01
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS	4.52E-01	2.18E+01 ± 1.90E-01	2.37E-02	< 2.37E-02	8.39E-01	3.00E+01 ± 4.65E-01
Spike Concentration					7.73E-05				1.06E-04
Spike Recovery					**103%**				**94%**
01-00429	01-00429	AN-102-SUP-AR	Sample4	9.86E-02	8.91E+00 ± 7.69E-02	5.16E-03	1.48E-01 ± 1.97E-03	1.83E-01	9.60E+00 ± 1.05E-01
01-00429-DUP	01-00429-DUP	AN-102-SUP-AR	Sample5	9.42E-02	8.56E+00 ± 3.50E-02	4.93E-03	1.48E-01 ± 1.87E-03	1.75E-01	9.61E+00 ± 6.47E-02
01-00429	01-00429 duplicate	AN-102-SUP-AR	Sample6	9.89E-02	8.92E+00 ± 7.10E-02	5.17E-03	1.56E-01 ± 2.10E-03	1.83E-01	9.76E+00 ± 1.09E-01
Duplicate %RPD					**0.1%**		**4.8%**		**1.7%**
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	9.91E-02	2.67E+01 ± 2.16E-01	5.18E-03	4.08E-01 ± 7.97E-03	1.84E-01	2.50E+01 ± 5.16E-02
Spike Concentration					2.17E-04		3.18E-06		1.88E-04
Spike Recovery					**109%**		**94%**		**94%**
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	3.42E-01	3.21E+01 ± 3.36E-01	1.79E-02	1.68E-01 ± 3.85E-03	6.35E-01	3.98E+01 ± 9.41E-01
					8.19E-05				1.07E-04
					117%				**102%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01 to 4/12/01

Unless otherwise specified, the results are reported in µCi analyte/ml of original

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019
Log-In: 01-00429
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [signature] 6-1-01

Analyst: [signature] 5/29/01

sample for radioisotopes; µg analyte/ml of original sample for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/ml	^{237}Np µCi/ml ± 1SD	MDL µCi/ml	^{239}Pu µCi/ml ± 1SD	MDL µCi/ml	^{240}Pu µCi/ml ± 1SD
	1%HNO3 (blanks)		ICB		< 1.58E-10		< 2.85E-08		< 4.37E-08
	1%HNO3 (blanks)		CCB1		< 1.95E-10		5.90E-08 ± 5.76E-09		< 5.40E-08
	1%HNO3 (blanks)		CCB2		< 1.37E-10		< 2.47E-08		< 3.79E-08
	1%HNO3 (samples)		ICB		< 6.21E-10		< 8.51E-08		< 1.56E-07
	1%HNO3 (samples)		CCB1		< 6.48E-10		< 8.88E-08		< 1.63E-07
	1%HNO3 (samples)		CCB2		< 6.46E-10		< 8.85E-08		< 1.63E-07
	ICV (blanks)		ICV		3.24E-08 ± 5.70E-10		3.22E-06 ± 1.94E-07		1.90E-07 ± 1.55E-08
	CCV (blanks)		CCV1		3.26E-08 ± 1.00E-09		3.04E-06 ± 4.15E-08		2.25E-07 ± 2.75E-08
	CCV (blanks)		CCV2		3.18E-08 ± 6.00E-10		2.64E-06 ± 6.37E-08		1.89E-07 ± 3.50E-09
	CCV (blanks)		CCV3		3.77E-08 ± 1.25E-09		3.17E-06 ± 5.08E-08		2.19E-07 ± 1.28E-08
True Value					**3.52E-08**		**3.04E-06**		**2.34E-07**
	ICV (samples)		ICV		3.70E-08 ± 3.93E-10		2.97E-06 ± 2.84E-08		2.34E-07 ± 2.92E-09
	CCV (samples)		CCV1		3.55E-08 ± 4.04E-10		3.19E-06 ± 6.69E-08		2.68E-07 ± 4.82E-08
	CCV (samples)		CCV2		3.86E-08 ± 2.46E-10		2.89E-06 ± 1.66E-07		1.94E-07 ± 5.76E-09
True Value					**3.52E-08**		**3.04E-06**		**2.34E-07**
	20ppb U030 uranium (blanks)		U030a		< 1.53E-10		< 2.76E-08		< 4.23E-08
	30ppb 4321B uranium (blanks)		4321b1		< 1.75E-10		7.82E-08 ± 2.87E-09		< 4.83E-08
	30ppb 4321B uranium (blanks)		4321b2		< 1.42E-10		3.46E-08 ± 1.52E-08		< 3.92E-08
	10ppb 4321B uranium (samples)		4321b		< 7.91E-10		< 1.08E-07		< 1.99E-07
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	1.12E-06	< 1.12E-06	2.01E-04	< 2.01E-04	3.09E-04	< 3.09E-04
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	1.18E-06	1.90E-04 ± 9.44E-06	2.13E-04	1.59E-02 ± 1.21E-03	3.27E-04	1.01E-03 ± 1.23E-04
Spike Concentration					3.36E-08		2.81E-06		1.79E-07
Spike Recovery					**95%**		**92%**		**77%**
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	1.10E-06	< 1.10E-06	1.99E-04	< 1.99E-04	3.05E-04	< 3.05E-04
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS1	7.89E-07	< 7.89E-07	1.42E-04	< 1.42E-04	2.18E-04	< 2.18E-04
01-00429	01-00429	AN-102-SUP-AR	Sample4	2.73E-06	1.21E-04 ± 3.76E-06	3.74E-04	6.75E-03 ± 1.38E-04	6.86E-04	2.07E-03 ± 1.13E-04
01-00429-DUP	01-00429-DUP	AN-102-SUP-AR	Sample5	2.42E-06	1.20E-04 ± 1.32E-06	3.32E-04	6.18E-03 ± 1.82E-04	6.10E-04	1.96E-03 ± 2.61E-04
01-00429	01-00429 duplicate	AN-102-SUP-AR	Sample6	2.51E-06	1.21E-04 ± 2.91E-06	3.44E-04	6.34E-03 ± 1.21E-04	6.32E-04	2.19E-03 ± 2.05E-04
Duplicate %RPD					**0.7%**		**6.3%**		**5.5%**
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	2.67E-06	2.97E-04 ± 6.02E-06	3.66E-04	2.72E-02 ± 2.01E-04	6.72E-04	3.35E-03 ± 2.82E-04
Spike Concentration					3.23E-08		3.75E-06		2.35E-07
Spike Recovery					**92%**		**124%**		**101%**
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	2.65E-06	1.24E-04 ± 3.51E-06	3.62E-04	6.15E-03 ± 6.16E-05	6.66E-04	2.03E-03 ± 1.40E-04

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01 to 4/12/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019
Log-In: 01-00429
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6-1-01

Analyst: JB Brown 5/29/01

sample for radioisotopes; µg analyte/ml of original sample for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/ml		^{241}Am µg/ml	±	1SD
	1%HNO3 (blanks)		ICB		<	2.46E-07		
	1%HNO3 (blanks)		CCB1		<	3.04E-07		
	1%HNO3 (blanks)		CCB2		<	2.13E-07		
	1%HNO3 (samples)		ICB		<	3.60E-07		
	1%HNO3 (samples)		CCB1		<	3.76E-07		
	1%HNO3 (samples)		CCB2		<	3.75E-07		
	ICV (blanks)		ICV			4.49E-05	±	1.01E-06
	CCV (blanks)		CCV1			4.49E-05	±	1.74E-06
	CCV (blanks)		CCV2			4.03E-05	±	1.12E-06
	CCV (blanks)		CCV3			4.78E-05	±	1.50E-06
True Value						**5.00E-05**		
	ICV (samples)		ICV			5.17E-05	±	9.44E-07
	CCV (samples)		CCV1			5.10E-05	±	9.97E-07
	CCV (samples)		CCV2			5.59E-05	±	1.12E-06
True Value						**5.00E-05**		
	20ppb U030 uranium (blanks)		U030a		<	2.38E-07		
	30ppb 4321B uranium (blanks)		4321b1		<	2.72E-07		
	30ppb 4321B uranium (blanks)		4321b2		<	2.21E-07		
	10ppb 4321B uranium (samples)		4321b		<	4.59E-07		
01-00429-PB1	01-00429-PB1	PROCESS BLANK	PB1	1.74E-03	<	1.74E-03		
01-00429-PB1	01-00429-PB1 + spike	PROCESS BLANK	PBS1	1.84E-03		2.60E-01	±	1.27E-02
Spike Concentration						4.59E-05		
Spike Recovery						**92%**		
01-00429-PB2	01-00429-PB2	PROCESS BLANK	PB2	1.72E-03	<	1.72E-03		
01-00429-BS2	01-00429-BS2	BLANK SPIKE	BS1	1.23E-03	<	1.23E-03		
01-00429	01-00429	AN-102-SUP-AR	Sample4	1.58E-03		4.86E-02	±	3.11E-05
01-00429-DUP	01-00429-DUP	AN-102-SUP-AR	Sample5	1.41E-03		4.93E-02	±	7.94E-04
01-00429	01-00429 duplicate	AN-102-SUP-AR	Sample6	1.46E-03		4.78E-02	±	7.83E-04
Duplicate %RPD						**1.7%**		
01-00429	01-00429 + spike	AN-102-SUP-AR	Sample8	1.55E-03		3.10E-01	±	3.90E-03
Spike Concentration						4.80E-05		
Spike Recovery						**96%**		
01-00429-MS2	01-00429-MS2	AN-102-SUP-AR	Sample7	1.54E-03		5.07E-02	±	4.48E-04

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01 to 4/12/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6-1-01

Analyst: [signature] 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Li µg/g ± 1SD	MDL µg/g	Be µg/g ± 1SD	MDL µg/g	B µg/g ± 1SD
	1%HNO3 (blanks)		ICB		< 5.63E-06		< 1.43E-06		< 5.63E-06
	1%HNO3 (blanks)		CCB1		< 7.46E-06		< 1.90E-06		< 7.46E-06
	1%HNO3 (blanks)		CCB2		< 7.89E-06		< 2.01E-06		< 7.90E-06
	1%HNO3 (samples)		ICB		< 4.09E-06		< 4.52E-06		< 3.00E-05
	1%HNO3 (samples)		CCB1		< 6.52E-06		< 7.20E-06		< 4.77E-05
	1%HNO3 (samples)		CCB2		< 7.39E-06		< 8.17E-06		< 5.41E-05
	ICV (blanks)		ICV		1.88E-04 ± 4.64E-06		2.12E-04 ± 9.29E-07		1.88E-04 ± 7.64E-06
	CCV (blanks)		CCV1		2.10E-04 ± 1.89E-05		2.03E-04 ± 6.54E-06		2.18E-04 ± 2.48E-05
	CCV (blanks)		CCV2		2.12E-04 ± 1.05E-05		1.99E-04 ± 1.96E-06		2.26E-04 ± 6.22E-06
True Value					**2.00E-04**		**2.00E-04**		**2.00E-04**
	ICV (samples)		ICV		4.91E-04 ± 2.41E-05		4.74E-04 ± 2.24E-05		5.14E-04 ± 3.45E-05
	CCV (samples)		CCV1		4.73E-04 ± 7.42E-06		4.85E-04 ± 3.43E-05		5.62E-04 ± 2.93E-05
	CCV (samples)		CCv2		5.10E-04 ± 6.52E-05		5.28E-04 ± 4.09E-05		5.03E-04 ± 6.90E-06
True Value					**5.00E-04**		**5.00E-04**		**5.00E-04**
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	3.94E-01	< 3.94E-01	1.00E-01	< 1.00E-01	3.94E-01	2.30E+01 ± 3.14E+01
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	6.97E-01	3.06E+01 ± 8.77E+00	1.77E-01	2.60E+01 ± 2.93E+00	6.98E-01	5.04E+01 ± 1.03E+01
Spike Concentration					2.89E-04		2.46E-04		2.59E-04
Spike Recovery					**145%**		**123%**		**129%**
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	4.06E-01	< 4.06E-01	1.03E-01	< 1.03E-01	4.06E-01	1.86E+01 ± 7.11E-01
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	7.23E+00	3.81E+02 ± 1.04E+02	5.53E-01	3.65E+01 ± 3.85E+00	2.18E+00	3.94E+01 ± 1.56E+00
Spike Concentration					3.60E-04		1.38E-04		6.19E-05
Spike Recovery					**122%**		**117%**		**52%**
01-00430	01-00430	AN-102-CS-AR	Sample4	2.94E-01	5.74E-01 ± 4.00E-02	3.25E-01	1.26E+00 ± 3.40E-01	2.15E+00	2.76E+01 ± 3.03E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	3.99E-01	3.56E+01 ± 6.99E-01	4.41E-01	9.83E-01 ± 5.36E-02	2.92E+00	5.13E+01 ± 9.16E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	3.18E-01	± 3.34E-01	3.51E-01	9.57E-01 ± 1.17E-01	2.33E+00	2.76E+01 ± 1.90E-01
Duplicate %RPD							**27.3%**		**0.01%**
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	3.35E-01	2.99E+01 ± 3.38E+00	3.70E-01	2.79E+01 ± 3.68E+00	2.45E+00	5.11E+01 ± 1.52E+00
Spike Concentration					6.95E-04		6.30E-04		5.55E-04
Spike Recovery					**139%**		**126%**		**111%**
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	3.97E+00	1.92E+03 ± 3.10E+01	2.31E+00	1.98E+02 ± 3.35E+00	1.53E+01	3.74E+02 ± 6.40E+00
Spike Concentration					3.63E-03		7.44E-04		1.31E-03
Spike Recovery					**241%**		**99%**		**35%**
01-00442	01-00442	AN-102-CS-DF	Sample1	2.49E-01	2.41E+01 ± 1.81E-01	2.75E-01	1.47E+01 ± 4.03E-01	1.82E+00	1.72E+01 ± 8.87E-02
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	3.10E-01	2.35E+01 ± 5.30E-01	3.43E-01	1.39E+01 ± 7.87E-01	2.27E+00	1.76E+01 ± 2.68E-01
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	2.81E-01	2.47E+01 ± 3.61E-01	3.10E-01	1.40E+01 ± 7.12E-01	2.06E+00	1.83E+01 ± 4.46E-01
Duplicate %RPD					**2.6%**		**4.9%**		**6.2%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6/1/01

Analyst: [signature] 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	V µg/g ± 1SD	MDL µg/g	Co µg/g ± 1SD	MDL µg/g	As µg/g ± 1SD
	1%HNO3 (blanks)		ICB		< 3.96E-07		< 5.64E-06		< 1.34E-05
	1%HNO3 (blanks)		CCB1		< 5.25E-07		< 7.47E-06		< 1.78E-05
	1%HNO3 (blanks)		CCB2		< 5.55E-07		< 7.90E-06		< 1.88E-05
	1%HNO3 (samples)		ICB		< 2.65E-06		< 1.08E-06		< 3.06E-05
	1%HNO3 (samples)		CCB1		< 4.21E-06		< 1.72E-06		< 5.87E-05
	1%HNO3 (samples)		CCB2		< 4.78E-06		< 1.95E-06		< 5.95E-05
	ICV (blanks)		ICV		2.22E-04 ± 2.55E-06		2.11E-04 ± 2.75E-06		2.19E-04 ± 4.35E-06
	CCV (blanks)		CCV1		1.98E-04 ± 5.79E-06		2.00E-04 ± 9.36E-06		2.08E-04 ± 9.53E-06
	CCV (blanks)		CCV2		2.20E-04 ± 4.89E-06		2.11E-04 ± 5.18E-06		2.12E-04 ± 1.16E-05
True Value					**2.00E-04**		**2.00E-04**		**2.00E-04**
	ICV (samples)		ICV		5.30E-04 ± 1.53E-05		5.25E-04 ± 9.62E-06		5.35E-04 ± 3.13E-05
	CCV (samples)		CCV1		5.55E-04 ± 1.98E-05		5.07E-04 ± 1.05E-05		5.77E-04 ± 1.70E-05
	CCV (samples)		CCv2		5.64E-04 ± 1.41E-05		5.48E-04 ± 2.19E-05		5.05E-04 ± 3.72E-05
True Value					**5.00E-04**		**5.00E-04**		**5.00E-04**
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	2.78E-02	5.14E+00 ± 9.18E-02	3.95E-01 <	3.95E-01	9.39E-01	3.52E+00 ± 3.46E-01
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	4.91E-02	3.23E+01 ± 2.21E+00	6.98E-01	2.32E+01 ± 7.37E-01	1.66E+00	2.67E+01 ± 1.90E+00
Spike Concentration					2.56E-04		2.19E-04		2.19E-04
Spike Recovery					**128%**		**109%**		**110%**
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	2.85E-02	4.01E+00 ± 3.25E-01	4.06E-01 <	4.06E-01	9.66E-01	2.29E+00 ± 1.10E-01
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	1.53E-01	7.05E+01 ± 3.13E+00	2.18E+00	3.40E+01 ± 5.65E-01	5.18E+00	3.28E+01 ± 1.06E+00
Spike Concentration					2.47E-04		1.28E-04		1.11E-04
Spike Recovery					**104%**		**109%**		**94%**
01-00430	01-00430	AN-102-CS-AR	Sample4	1.90E-01	6.49E+00 ± 9.90E-02	7.74E-02	2.57E+00 ± 8.81E-02	2.40E+00	5.88E+00 ± 6.05E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	2.58E-01	6.72E+00 ± 1.62E-01	1.05E-01	2.15E+00 ± 1.98E-01	3.31E+00	7.19E+00 ± 7.77E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	2.05E-01	6.42E+00 ± 5.40E-02	8.37E-02	2.77E+00 ± 1.83E-01	2.60E+00	6.37E+00 ± 8.27E-01
Duplicate %RPD					**1.1%**		**7.6%**		**8.1%**
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	2.17E-01	2.49E+01 ± 7.51E-01	8.82E-02	2.13E+01 ± 8.62E-01	2.77E+00	2.52E+01 ± 1.82E+00
Spike Concentration					4.36E-04		4.43E-04		4.56E-04
Spike Recovery					**87%**		**89%**		**91%**
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	1.35E+00	8.19E+01 ± 2.25E+00	5.50E-01	4.03E+01 ± 6.46E-01	1.86E+01	2.09E+02 ± 2.05E+01
Spike Concentration					2.85E-04		1.42E-04		7.68E-04
Spike Recovery					**126%**		**126%**		**102%**
01-00442	01-00442	AN-102-CS-DF	Sample1	1.61E-01	5.10E+00 ± 1.39E-01	6.56E-02	8.61E-01 ± 2.22E-02	1.60E+00	6.43E+00 ± 4.16E-01
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	2.01E-01	5.65E+00 ± 2.69E-01	8.17E-02	1.89E+00 ± 1.10E-01	2.01E+00	7.32E+00 ± 1.29E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	1.82E-01	5.60E+00 ± 1.28E-01	7.40E-02	8.68E-01 ± 2.47E-02	1.81E+00	6.88E+00 ± 9.46E-01
Duplicate %RPD					**9.4%**		**0.8%**		**6.7%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6/1/01

Analyst: [signature] 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Se µg/g ± 1SD	MDL µg/g	Rb µg/g ± 1SD	MDL µg/g	Y µg/g ± 1SD
	1%HNO3 (blanks)		ICB	<	1.78E-04	<	3.70E-06	<	1.75E-07
	1%HNO3 (blanks)		CCB1	<	2.36E-04	<	4.90E-06	<	2.32E-07
	1%HNO3 (blanks)		CCB2	<	2.50E-04	<	5.18E-06	<	2.46E-07
	1%HNO3 (samples)		ICB	<	1.45E-04	<	2.31E-06	<	2.47E-06
	1%HNO3 (samples)		CCB1	<	2.74E-04	<	4.44E-06	<	3.93E-06
	1%HNO3 (samples)		CCB2	<	2.90E-04	<	4.50E-06	<	4.46E-06
	ICV (blanks)		ICV		2.34E-04 ± 2.09E-05		2.20E-04 ± 7.09E-07		2.17E-04 ± 3.61E-06
	CCV (blanks)		CCV1		3.63E-04 ± 3.20E-04		2.05E-04 ± 9.54E-06		1.98E-04 ± 8.24E-06
	CCV (blanks)		CCV2	<	2.27E-04		2.13E-04 ± 8.01E-06		2.10E-04 ± 3.88E-06
True Value					**2.00E-04**		**2.00E-04**		**2.00E-04**
	ICV (samples)		ICV		5.97E-04 ± 2.31E-04		5.40E-04 ± 4.78E-06		5.17E-04 ± 1.87E-05
	CCV (samples)		CCV1		1.14E-03 ± 1.47E-04		5.50E-04 ± 7.03E-06		5.14E-04 ± 8.33E-06
	CCV (samples)		CCv2		6.98E-04 ± 3.34E-04		5.71E-04 ± 2.15E-05		5.19E-04 ± 5.00E-06
True Value					**5.00E-04**		**5.00E-04**		**5.00E-04**
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	1.25E+01 <	1.25E+01	2.59E-01 <	2.59E-01	1.23E-02 <	1.23E-02
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	2.20E+01	3.31E+01 ± 1.88E+01	4.58E-01	2.12E+01 ± 7.33E-01	2.17E-02	2.13E+01 ± 1.37E+00
Spike Concentration					3.13E-04		2.00E-04		2.01E-04
Spike Recovery					**156%**		**100%**		**100%**
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	1.28E+01 <	1.28E+01	2.66E-01 <	2.66E-01	1.26E-02	3.78E-02 ± 9.44E-03
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	2.29E+02	2.62E+02 ± 1.83E+02	1.43E+00	5.59E+01 ± 1.80E+00	6.77E-02	5.97E+01 ± 1.78E+00
Spike Concentration					9.89E-04		2.11E-04		2.25E-04
Spike Recovery					**84%**		**89%**		**95%**
01-00430	01-00430	AN-102-CS-AR	Sample4	1.20E+02 <	1.20E+02	1.81E-01	4.77E+00 ± 2.32E-01	1.77E-01	9.59E+00 ± 1.39E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	1.68E+01 <	1.68E+01	2.50E-01	4.64E+00 ± 4.22E-01	2.41E-01	9.02E+00 ± 2.39E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	1.31E+01 <	1.31E+01	1.96E-01	5.08E+00 ± 1.50E-01	1.92E-01	9.67E+00 ± 1.12E-01
Duplicate %RPD							**6.3%**		**0.8%**
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	9.29E+00	2.85E+01 ± 1.11E+01	2.09E-01	2.37E+01 ± 4.82E-01	2.02E-01	2.71E+01 ± 2.44E-01
Spike Concentration					6.74E-04		4.49E-04		4.14E-04
Spike Recovery					**135%**		**90%**		**83%**
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	8.74E+01 <	8.74E+01	1.41E+00	7.77E+01 ± 3.15E+00	1.26E+00	6.67E+01 ± 1.03E+00
Spike Concentration							2.75E-04		2.16E-04
Spike Recovery							**122%**		**95%**
01-00442	01-00442	AN-102-CS-DF	Sample1	1.10E+01 <	1.10E+01	1.21E-01	1.11E+00 ± 1.65E-01	1.50E-01	2.61E+01 ± 3.41E-01
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	1.40E+01 <	1.40E+01	1.52E-01	1.24E+00 ± 5.59E-02	1.87E-01	2.55E+01 ± 9.67E-01
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	1.22E+01 <	1.22E+01	1.37E-01	1.14E+00 ± 1.47E-01	1.69E-01	2.68E+01 ± 1.11E-01
Duplicate %RPD							**2.5%**		**2.6%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: KMP 6/1/01

Analyst: JC Bronson 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Mo µg/g ± 1SD	MDL µCi/g	99Tc µCi/g ± 1SD
	1%HNO3 (blanks)		ICB	<	1.50E-05	<	3.88E-08
	1%HNO3 (blanks)		CCB1	<	1.89E-05	<	4.89E-08
	1%HNO3 (blanks)		CCB2	<	2.14E-05	<	5.54E-08
	1%HNO3 (samples)		ICB	<	2.87E-05	<	2.87E-08
	1%HNO3 (samples)		CCB1	<	5.43E-05	<	5.42E-08
	1%HNO3 (samples)		CCB2	<	5.76E-05	<	5.76E-08
	ICV (blanks)		ICV		2.26E-04 ± 2.14E-05		3.63E-06 ± 8.06E-08
	CCV (blanks)		CCV1		2.18E-04 ± 1.85E-05		3.60E-06 ± 1.76E-07
	CCV (blanks)		CCV2		2.12E-04 ± 7.59E-05		3.71E-06 ± 2.74E-07
True Value					**2.00E-04**		**3.39E-06**
	ICV (samples)		ICV		5.52E-04 ± 2.91E-05		8.45E-06 ± 4.64E-07
	CCV (samples)		CCV1		6.05E-04 ± 4.16E-05		9.31E-06 ± 3.66E-07
	CCV (samples)		CCv2		5.61E-04 ± 2.23E-05		9.04E-06 ± 1.84E-07
True Value					**5.00E-04**		**8.48E-06**
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	2.47E+00 <	1.05E+00	1.84E-03 <	2.72E-03
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	4.36E+00	2.26E+01 ± 7.23E-01	3.68E-01	3.68E-01 ± 2.45E-02
Spike Concentration					2.13E-04		2.05E-04
Spike Recovery					**107%**		**103%**
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	2.54E+00 <	1.08E+00	7.72E-04 <	2.79E-03
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2&BS3	5.66E+00	3.17E+02 ± 7.60E+00	1.44E-02 <	5.26E-02
Spike Concentration					1.20E-03		
Spike Recovery					**101%**		
01-00430	01-00430	AN-102-CS-AR	Sample4	2.38E+00	3.34E+01 ± 1.28E+00	2.37E-03	9.50E-02 ± 5.11E-03
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	3.34E+00	3.26E+01 ± 2.66E+00	3.34E-03	1.03E-01 ± 3.13E-03
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample5	2.60E+00	3.41E+01 ± 2.47E+00	2.60E-03	1.04E-01 ± 1.67E-03
Duplicate %RPD					**2.1%**		**8.7%**
01-00430	01-00430 + spike	AN-102-CS-AR	Sample9	2.91E+00	4.91E+01 ± 1.65E+00	2.90E-03	4.53E-01 ± 5.80E-03
Spike Concentration					3.73E-04		4.99E-04
Spike Recovery					**75%**		**100%**
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample6&8	1.73E+01	3.58E+02 ± 1.34E+01	1.73E-02	8.75E-02 ± 1.66E-02
Spike Concentration					1.22E-03		
Spike Recovery					**108%**		
01-00442	01-00442	AN-102-CS-DF	Sample1	2.19E+00	1.19E+01 ± 4.35E-01	2.19E-03	3.39E-02 ± 2.37E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample3	2.78E+00	1.35E+01 ± 1.12E+00	2.78E-03	3.03E-02 ± 2.54E-03
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample2	2.42E+00	1.02E+01 ± 1.09E+00	2.42E-03	3.16E-02 ± 3.45E-04
Duplicate %RPD					**15.4%**		**6.8%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 6.1.01

Analyst: 6/1/01

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Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Sb µg/g ± 1SD	MDL µg/g	Te µg/g ± 1SD	MDL µg/g	Ta µg/g ± 1SD
	1%HNO3		ICB		< 9.89E-06		< 1.29E-04		3.59E-05 ± 5.49E-04
	1%HNO3		CCB1		< 9.78E-06		< 1.28E-04		3.74E-05 ± 3.98E-05
	1%HNO3		CCB2		< 9.78E-06		< 1.28E-04		3.64E-05 ± 5.43E-05
	1%HNO3		CCB3		< 9.82E-06		< 1.29E-04		3.87E-05 ± 1.56E-05
	ICV		ICV		2.16E-04 ± 1.72E-05		2.04E-04 ± 4.85E-06		1.79E-04 ± 1.11E-05
	CCV		CCV1		2.19E-04 ± 2.24E-05		1.93E-04 ± 4.97E-06		1.85E-04 ± 2.23E-06
	CCV		CCV2		2.03E-04 ± 8.92E-06		2.04E-04 ± 6.20E-06		1.59E-04 ± 9.43E-06
	CCV		CCV3		1.94E-04 ± 7.75E-06		2.06E-04 ± 2.66E-05		1.97E-04 ± 4.69E-06
True Value					**2.00E-04**		**2.00E-04**		**2.00E-04**
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	5.03E-02 <	5.03E-02	6.58E-01 <	6.58E-01	3.24E-02	1.99E-01 ± 9.14E-02
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	5.25E-01	1.09E+00 ± 7.77E-02	6.87E-01	1.07E+00 ± 1.41E-01	3.38E-02	9.80E-01 ± 4.22E-02
Spike Concentration					2.06E-04		2.02E-04		1.47E-04
Spike Recovery					**103%**		**101%**		**74%**
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.28E-02 <	5.28E-02	6.90E-01 <	6.90E-01	3.40E-02	2.00E-01 ± 5.72E-02
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	4.84E+00	1.23E+02 ± 2.21E+00	6.33E+01 <	6.33E+01	3.12E+00	4.73E+01 ± 2.90E+00
Spike Concentration					2.33E-04		1.20E-04		8.94E-05
Spike Recovery					**99%**		**101%**		**76%**
01-00430	01-00430	AN-102-CS-AR	Sample9	4.31E-02	1.16E-01 ± 3.36E-02	5.64E-01	1.48E+00 ± 1.77E-01	2.77E-02	2.45E-01 ± 1.18E-02
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample10	4.77E-02	1.99E-01 ± 3.05E-02	6.25E-01	1.66E+00 ± 5.40E-01	3.07E-02	2.61E-01 ± 5.97E-02
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample11	4.27E-02	1.81E-01 ± 4.85E-02	5.59E-01	1.37E+00 ± 1.30E-01	2.75E-02	2.22E-01 ± 2.75E-02
Duplicate %RPD					**44.0%**		**7.4%**		**10.0%**
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	7.51E-01	2.12E+01 ± 1.30E+00	9.83E+00	1.95E+01 ± 1.21E+00	4.84E-01	1.33E+01 ± 3.94E-01
Spike Concentration					2.50E-04		2.13E-04		1.55E-04
Spike Recovery					**125%**		**107%**		**77%**
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	4.63E+00	1.23E+02 ± 6.45E+00	6.05E+01	6.15E+01 ± 1.31E+00	2.98E+00	4.21E+01 ± 1.18E+00
Spike Concentration					2.32E-04		1.13E-04		7.91E-05
Spike Recovery					**103%**		**100%**		**70%**
01-00442	01-00442	AN-102-CS-DF	Sample12	2.96E-02	1.34E-01 ± 2.39E-02	3.87E-01	4.38E+00 ± 4.31E-01	1.90E-02	2.65E-01 ± 2.28E-02
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample13	3.03E-02	1.56E-01 ± 2.86E-02	3.97E-01	4.07E+00 ± 2.28E-01	1.95E-02	1.89E-01 ± 1.70E-02
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample14	3.05E-02	1.16E-01 ± 4.73E-03	4.00E-01	4.23E+00 ± 5.12E-02	1.97E-02	2.78E-01 ± 4.97E-03
Duplicate %RPD					**14.2%**		**3.3%**		**4.9%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: KMD 6/1/01

Analyst: JB 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Ce µg/g	±	1SD	MDL µg/g	Pr µg/g	±	1SD	MDL µg/g	W µg/g	±	1SD
	1%HNO3		ICB		< 8.17E-06				< 2.66E-06				< 1.18E-05		
	1%HNO3		CCB1		< 8.08E-06				< 2.63E-06				< 1.17E-05		
	1%HNO3		CCB2		< 8.08E-06				< 2.63E-06				< 1.17E-05		
	1%HNO3		CCB3		< 8.11E-06				< 2.64E-06				2.14E-05	±	3.60E-07
	ICV		ICV		2.17E-04	±	6.15E-06		1.94E-04	±	3.02E-06		1.92E-04	±	9.37E-06
	CCV		CCV1		2.20E-04	±	2.91E-06		2.03E-04	±	8.41E-06		2.03E-04	±	1.16E-05
	CCV		CCV2		1.91E-04	±	5.13E-06		1.97E-04	±	6.48E-06		1.92E-04	±	9.74E-06
	CCV		CCV3		2.07E-04	±	7.16E-06		2.16E-04	±	4.85E-06		2.39E-04	±	1.03E-05
True Value					**2.00E-04**				**2.00E-04**				**2.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	4.15E-01	< 4.15E-01			1.35E-01	< 1.35E-01			6.02E-01	< 6.02E-01		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	4.34E-01	1.05E+01	±	5.01E-01	1.41E-01	1.07E+01	±	3.62E-01	6.29E-01	1.13E+01	±	5.52E-01
Spike Concentration					1.98E-04				2.02E-04				2.13E-04		
Spike Recovery					**99%**				**101%**				**107%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	4.36E-01	< 4.36E-01			1.42E-01	< 1.42E-01			6.32E-01	< 6.32E-01		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	4.00E+00	5.34E+01	±	8.73E-01	1.30E+00	5.76E+01	±	6.81E-01	5.80E+00	4.30E+01	±	4.90E+00
Spike Concentration					1.01E-04				1.09E-04				8.11E-05		
Spike Recovery					**85%**				**92%**				**69%**		
01-00430	01-00430	AN-102-CS-AR	Sample1	3.78E-01	2.25E+01	±	8.87E-01	1.23E-01	1.75E+01	±	2.77E-01	5.48E-01	9.28E+01	±	4.56E+00
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample2	4.54E-01	2.35E+01	±	2.43E-01	1.48E-01	1.76E+01	±	2.20E-01	6.58E-01	1.01E+02	±	2.46E+00
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample3	3.64E-01	2.32E+01	±	4.51E-01	1.19E-01	1.79E+01	±	4.25E-01	5.28E-01	9.91E+01	±	5.09E+00
Duplicate %RPD					**2.9%**				**2.0%**				**6.6%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	6.20E-01	4.53E+01	±	6.45E-01	2.02E-01	3.45E+01	±	6.10E-01	8.99E-01	1.40E+02	±	3.56E+00
Spike Concentration					2.70E-04				2.01E-04				5.54E-04		
Spike Recovery					**135%**				**101%**				**111%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	3.82E+00	4.21E+02	±	4.01E+00	1.24E+00	7.53E+01	±	9.34E-01	5.54E+00	1.32E+02	±	5.40E+00
Spike Concentration					7.52E-04				1.09E-04				7.45E-05		
Spike Recovery					**100%**				**97%**				**66%**		
01-00442	01-00442	AN-102-CS-DF	Sample4	2.30E-01	7.14E+01	±	1.03E+00	7.50E-02	4.71E+01	±	2.11E-01	3.34E-01	4.26E+01	±	1.86E+00
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample5	2.20E-01	6.90E+01	±	8.26E-01	7.18E-02	4.67E+01	±	1.05E+00	3.19E-01	4.20E+01	±	2.48E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample6	2.51E-01	7.22E+01	±	5.19E-01	8.17E-02	4.78E+01	±	1.42E+00	3.64E-01	4.64E+01	±	1.03E+00
Duplicate %RPD					**1.1%**				**1.5%**				**8.7%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019
Log-In: 01-00430-Zr
Report Date: 5/29/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by [illegible] 6/1/01

Analyst: [signature] 6/1/01

Unless otherwise specified, the results are reported in µg analyte/g of wet centrifuged solids.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Ru µg/g ± 1SD	MDL µg/g	Rh µg/g ± 1SD	MDL µg/g	Pd µg/g ± 1SD	MDL µg/g	Pt µg/g ± 1SD
	1%HNO3		ICB		< 2.90E-06		< 1.37E-06		< 3.05E-06		< 1.47E-06
	1%HNO3		CCB1		< 3.42E-06		< 1.62E-06		< 3.59E-06		< 1.73E-06
	1%HNO3		CCB2		< 3.88E-06		< 1.84E-06		< 4.07E-06		< 1.96E-06
	1%HNO3		CCB3		< 5.42E-06		< 2.57E-06		< 5.69E-06		< 2.74E-06
	0.1ppb		ICV1		1.02E-04 ± 1.73E-06		9.73E-05 ± 2.80E-06		1.09E-04 ± 1.32E-05		1.02E-04 ± 2.47E-06
	0.1ppb		CCV1a		1.00E-04 ± 2.35E-06		9.64E-05 ± 1.47E-06		8.81E-05 ± 6.00E-06		1.03E-04 ± 1.09E-06
	0.1ppb		CCV2a		1.03E-04 ± 9.03E-07		9.96E-05 ± 2.46E-06		8.45E-05 ± 3.58E-06		9.89E-05 ± 6.74E-07
	0.1ppb		CCV3a		1.06E-04 ± 1.17E-06		9.97E-05 ± 4.29E-06		7.78E-05 ± 7.28E-06		1.06E-04 ± 2.32E-06
01-00430-PB1-Zr	01-00430-PB1-Zr	PROCESS BLANK	PB1	1.34E-01	< 1.34E-01	6.34E-02	< 6.34E-02	1.41E-01	< 1.41E-01	6.78E-02	< 6.78E-02
01-00430-PB1-Zr	01-00430-PB1-Zr+spike	PROCESS BLANK	PBS1	1.39E-01	2.92E+00 ± 9.65E-02	6.57E-02	3.91E+00	1.46E-01	4.30E+00 ± 1.62E-01	7.02E-02	3.91E+00 ± 1.03E-01
Spike Concentration					7.22E-05		9.66E-05		1.06E-04		9.68E-05
Spike Recovery					**107%**		**97%**		**106%**		**97%**
01-00430-PB2-Zr	01-00430-PB2-Zr	PROCESS BLANK	PB2	1.35E-01	< 1.35E-01	6.37E-02	< 6.37E-02	1.41E-01	< 1.41E-01	6.80E-02	< 6.80E-02
01-00430-BS-Zr	01-00430-BS-Zr	BLANK SPIKE	BS	1.34E+00	7.51E+01 ± 3.02E-01	6.34E-01	1.13E+02 ± 2.02E+00	1.40E+00	1.21E+02 ± 3.14E+00	6.77E-01	1.07E+02 ± 1.01E+00
Spike Concentration					2.76E-04		2.80E-04		3.00E-04		2.64E-04
Spike Recovery					**104%**		**105%**		**113%**		**99%**
01-00430-Zr	01-00430-Zr	AN-102-CS-AR	Sample1&4	1.42E-01	1.47E+01 ± 1.21E-02	6.74E-02	4.90E+00 ± 2.63E-02	1.49E-01	1.30E+01 ± 2.62E-01	7.20E-02	< 7.20E-02
01-00430-DUP-Zr	01-00430-DUP-Zr	AN-102-CS-AR	Sample2&5	1.57E-01	1.56E+01 ± 4.89E-01	7.45E-02	5.31E+00 ± 1.11E-01	1.65E-01	1.35E+01 ± 1.78E-01	7.96E-02	< 7.96E-02
01-00430-Zr	01-00430-Zr duplicate	AN-102-CS-AR	Sample3&6	1.62E-01	1.44E+01 ± 1.63E-01	7.68E-02	4.91E+00 ± 2.10E-01	1.70E-01	1.29E+01 ± 4.31E-01	8.21E-02	< 8.21E-02
Duplicate %RPD					**2.0%**		**0.2%**		**0.7%**		
01-00430-Zr	01-00430-Zr + spike	AN-102-CS-AR	Sample8	3.69E-01	1.94E+01 ± 3.02E-01	1.75E-01	1.26E+01 ± 2.39E-01	3.87E-01	2.03E+01 ± 8.87E-01	1.87E-01	8.10E+00 ± 2.74E-01
Spike Concentration					5.79E-05		9.60E-05		9.09E-05		1.00E-04
Spike Recovery					**86%**		**96%**		**91%**		**100%**
01-00430-MS-Zr	01-00430-MS-Zr	AN-102-CS-AR	Sample7	1.63E+00	7.99E+01 ± 2.07E+00	7.72E-01	1.04E+02 ± 2.91E+00	1.71E+00	1.03E+02 ± 1.73E+00	8.25E-01	1.01E+02 ± 2.03E+00
Spike Concentration					2.68E-04		2.74E-04		2.48E-04		2.80E-04
Spike Recovery					**101%**		**103%**		**93%**		**105%**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/28/01
Instrument Filenames: Experiment (28MAR01), Procedure(010328a), Element Menu (NM)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: SMP 6-01-01

Analyst: J P Bronson 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	Pt µg/g	±	1SD	MDL µg/g	Tl µg/g	±	1SD
	1%HNO3		ICB	<	1.11E-05			<	5.15E-06		
	1%HNO3		CCB1	<	1.06E-05			<	5.09E-06		
	1%HNO3		CCB2	<	1.10E-05			<	5.09E-06		
	1%HNO3		CCB3	<	9.58E-06			<	5.11E-06		
	ICV		ICV		2.19E-04	±	1.38E-05		1.92E-04	±	8.62E-06
	CCV		CCV1		2.13E-04	±	1.93E-05		2.06E-04	±	7.06E-06
	CCV		CCV2		1.92E-04	±	9.43E-06		1.80E-04	±	3.42E-06
	CCV		CCV3		1.93E-04	±	6.02E-06		2.26E-04	±	1.69E-06
True Value					**2.00E-04**				**2.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	5.48E-02 <	5.48E-02			2.62E-02 <	2.62E-02		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PB1S	5.64E-02	1.06E+00	±	2.31E-02	2.73E-02	1.02E+00	±	3.28E-02
Spike Concentration					2.00E-04				1.93E-04		
Spike Recovery					**100%**				**96%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.66E-02 <	5.66E-02			2.75E-02 <	2.75E-02		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS	5.54E+00 <	5.54E+00			2.52E+00	5.75E+02	±	1.33E+01
Spike Concentration									1.09E-03		
Spike Recovery									**92%**		
01-00430	01-00430	AN-102-CS-AR	Sample9	4.41E-02 <	4.41E-02			2.24E-02 <	2.24E-02		
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample10	5.02E-02 <	5.02E-02			2.48E-02 <	2.48E-02		
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample11	4.52E-02 <	4.52E-02			2.22E-02 <	2.22E-02		
Duplicate %RPD											
01-00430	01-00430 + spike	AN-102-CS-AR	Sample8	8.19E-01	1.73E+01	±	2.51E-01	3.91E-01	4.31E+01	±	1.17E+00
Spike Concentration					2.05E-04				5.10E-04		
Spike Recovery					**102%**				**102%**		
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample7	5.05E+00 <	5.05E+00			2.41E+00	5.79E+02	±	8.97E+00
Spike Concentration									1.09E-03		
Spike Recovery									**97%**		
01-00442	01-00442	AN-102-CS-DF	Sample12	3.20E-02 <	3.20E-02			1.54E-02	2.03E-02	±	5.88E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample13	3.17E-02 <	3.17E-02			1.58E-02	5.81E-02	±	5.08E-03
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample14	3.25E-02 <	3.25E-02			1.59E-02	4.80E-02	±	1.14E-02
Duplicate %RPD									**81.0%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: MPS 6-1-01

Analyst: JC Bowers 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g		Th µg/g	±	1SD	MDL µg/g		U µg/g	±	1SD
	1%HNO3		ICB		<	2.55E-06				<	1.92E-06		
	1%HNO3		CCB1		<	2.36E-06				<	1.78E-06		
	1%HNO3		CCB2		<	2.27E-06				<	1.71E-06		
	1%HNO3		CCB3		<	2.09E-06				<	1.58E-06		
	1%HNO3		CCB4		<	2.11E-06				<	1.59E-06		
	ICV		ICV			1.03E-04	±	5.35E-06			1.02E-04	±	1.97E-06
	CCV		CCV1			1.03E-04	±	4.47E-06			1.08E-04	±	5.71E-06
	CCV		CCV2			1.06E-04	±	1.98E-06			1.02E-04	±	2.16E-06
	CCV		CCV3			1.00E-04	±	1.89E-06			9.70E-05	±	1.81E-06
	CCV		CCV4			1.00E-04	±	3.72E-06			9.72E-05	±	1.94E-06
True Value						**1.00E-04**					**1.00E-04**		
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	6.75E-01	<	6.75E-01			5.09E-01	<	5.09E-01		
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS1	5.97E-01		3.02E+01	±	7.35E-01	4.51E-01		2.96E+01	±	4.03E-01
Spike Concentration						1.14E-04					1.12E-04		
Spike Recovery						**114%**					**112%**		
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	6.92E-01	<	6.92E-01			5.23E-01	<	5.23E-01		
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	5.73E-01		8.49E+01	±	4.71E+00	4.33E-01		7.43E+01	±	2.21E+00
Spike Concentration						3.21E-04					2.81E-04		
Spike Recovery						**81%**					**71%**		
01-00430	01-00430	AN-102-CS-AR	Sample6	4.30E-01		2.61E+01	±	1.28E+00	3.25E-01		6.70E+01	±	3.46E+00
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	5.56E-01		2.67E+01	±	1.27E+00	4.20E-01		6.39E+01	±	1.11E+00
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	4.45E-01		2.79E+01	±	5.90E-01	3.36E-01		6.47E+01	±	1.49E+00
Duplicate %RPD						**6.7%**					**3.5%**		
01-00430	01-00430 + spike	AN-102-CS-AR	Sample14	4.25E-01		4.47E+01	±	7.80E-01	3.21E-01		8.73E+01	±	1.43E+00
Spike Concentration						8.79E-05					9.61E-05		
Spike Recovery						**88%**					**96%**		
01-00430-MS	01-00430-MS	AN-102-CS-AR	Sample12	6.03E+00		5.34E+02	±	5.97E+00	4.55E+00		6.60E+02	±	1.62E+01
Spike Concentration						1.92E-04					2.24E-04		
Spike Recovery						**102%**					**119%**		
01-00442	01-00442	AN-102-CS-DF	Sample9	1.39E+00		6.40E+01	±	1.21E+00	1.05E+00		2.80E+02	±	2.93E+00
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.40E+00		6.18E+01	±	7.10E-01	1.06E+00		2.77E+02	±	2.34E+00
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	1.37E+00		6.89E+01	±	7.24E-01	1.03E+00		2.81E+02	±	7.43E+00
Duplicate %RPD						**7.4%**					**0.5%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [signature] 6-1-01

Analyst: [signature] 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	U233 µCi/g	± 1SD	MDL µCi/g	U234 µCi/g	± 1SD	MDL µCi/g	U235 µCi/g	± 1SD
	1%HNO3		ICB		< 3.23E-10			< 3.50E-10			< 1.55E-13	
	1%HNO3		CCB1		< 1.20E-10			< 1.58E-10			< 3.02E-14	
	1%HNO3		CCB2		< 1.13E-11			< 3.24E-11			< 4.29E-14	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	7.61E-06	< 7.61E-06		3.03E-05	< 3.03E-05			< 1.12E-08	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	5.67E-06	< 5.67E-06		6.75E-06	< 6.75E-06			< 1.59E-08	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	7.95E-06	< 7.95E-06		6.50E-06	< 6.50E-06		1.12E-08	4.32E-07	± 2.21E-08
01-00430	01-00430	AN-102-CS-AR	Sample6	7.61E-06	6.13E-05	± 1.21E-05	3.24E-05	< 3.24E-05		1.12E-08	9.89E-07	± 3.72E-08
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	7.61E-06	5.07E-05	± 8.95E-06	2.91E-05	< 2.91E-05		1.12E-08	9.84E-07	± 3.12E-08
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	7.61E-06	4.77E-05	± 7.17E-06	2.61E-05	< 2.61E-05		1.12E-08	1.01E-06	± 3.62E-08
Duplicate %RPD					**25.1%**						**1.6%**	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	7.61E-06	1.14E-04	± 1.81E-05	3.03E-05	8.60E-05	± 1.38E-05	1.12E-08	4.45E-06	± 1.90E-07
01-00442	01-00442	AN-102-CS-DF	Sample9	7.61E-06	1.09E-03	± 5.88E-05	3.03E-05	1.33E-04	± 2.06E-05	1.12E-08	4.47E-06	± 8.62E-08
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	7.61E-06	1.15E-03	± 6.85E-05	3.03E-05	1.20E-04	± 5.14E-06	1.12E-08	4.33E-06	± 1.55E-07
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	7.61E-06	1.06E-03	± 5.67E-05	3.03E-05	1.20E-04	± 1.85E-05	1.12E-08	4.44E-06	± 8.53E-08
Duplicate %RPD					**2.8%**			**10.9%**			**0.6%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: tmPS 6-1-01

Analyst: JP Bramson 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	U236 µCi/g	± 1SD	MDL µCi/g	U238 µCi/g	± 1SD
	1%HNO3		ICB		< 2.52E-12			< 7.02E-13	
	1%HNO3		CCB1		< 1.07E-12			< 6.00E-13	
	1%HNO3		CCB2		< 2.91E-14			< 5.66E-13	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1		< 1.75E-07			< 1.67E-07	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2		< 1.27E-07			< 1.72E-07	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.75E-07	< 1.75E-07		1.67E-07	2.49E-05 ±	1.34E-06
01-00430	01-00430	AN-102-CS-AR	Sample6	1.75E-07	1.34E-06 ±	8.29E-08	1.67E-07	2.24E-05 ±	8.87E-07
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	1.75E-07	1.12E-06 ±	4.56E-08	1.67E-07	2.13E-05 ±	5.75E-07
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	1.75E-07	1.33E-06 ±	2.46E-07	1.67E-07	2.16E-05 ±	9.45E-07
Duplicate %RPD					**0.7%**			**3.5%**	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	1.75E-07	1.23E-06 ±	1.47E-07	1.67E-07	2.21E-04 ±	1.26E-05
01-00442	01-00442	AN-102-CS-DF	Sample9	1.75E-07	6.62E-06 ±	1.59E-07	1.67E-07	9.34E-05 ±	6.57E-07
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.75E-07	6.02E-06 ±	2.45E-07	1.67E-07	9.24E-05 ±	1.44E-06
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	1.75E-07	6.08E-06 ±	4.70E-07	1.67E-07	9.37E-05 ±	2.17E-06
Duplicate %RPD					**8.4%**			**0.4%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: MPJ 6-1-01

Analyst: JBrown 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/g	237Np µCi/g	± 1SD	MDL µCi/g	239Pu µCi/g	± 1SD	MDL µCi/g	240Pu µCi/g	± 1SD
	1%HNO3 (blanks)		ICB		< 1.58E-10			< 1.78E-08			< 4.37E-08	
	1%HNO3 (blanks)		CCB1		< 1.95E-10			5.90E-08 ±	5.76E-09		< 5.40E-08	
	1%HNO3 (blanks)		CCB2		< 1.37E-10			< 1.54E-08			< 3.79E-08	
	1%HNO3 (samples)		ICB		< 5.00E-10			< 7.99E-08			< 5.15E-08	
	1%HNO3 (samples)		CCB1		< 5.21E-10			< 8.32E-08			< 5.36E-08	
	1%HNO3 (samples)		CCB2		< 6.43E-10			< 1.03E-07			< 6.62E-08	
	1%HNO3 (samples)		CCB3		< 6.25E-10			< 9.98E-08			< 6.43E-08	
	1%HNO3 (samples)		CCB4		< 6.29E-10			3.83E-07 ±	3.88E-08		< 6.48E-08	
	1%HNO3 (samples)		CCB5		< 1.03E-09			4.34E-07 ±	3.61E-08		< 1.06E-07	
	ICV (blanks)		ICV		3.24E-08 ±	5.70E-10		3.22E-06 ±	1.94E-07		1.90E-07 ±	1.55E-08
	CCV (blanks)		CCV1		3.26E-08 ±	1.00E-09		3.04E-06 ±	4.15E-08		2.25E-07 ±	2.75E-08
	CCV (blanks)		CCV2		3.18E-08 ±	6.00E-10		2.64E-06 ±	6.37E-08		1.89E-07 ±	3.50E-09
	CCV (blanks)		CCV3		3.77E-08 ±	1.25E-09		3.17E-06 ±	5.08E-08		2.19E-07 ±	1.28E-08
True Value					**3.52E-08**			**3.06E-06**			**2.33E-07**	
	ICV (samples)		ICV		3.79E-08 ±	2.19E-09		3.39E-06 ±	3.91E-08		2.08E-07 ±	1.09E-08
	CCV (samples)		CCV1		3.58E-08 ±	1.51E-09		3.54E-06 ±	5.71E-08		2.46E-07 ±	3.15E-08
	CCV (samples)		CCV2		3.72E-08 ±	1.07E-09		3.27E-06 ±	1.11E-07		2.35E-07 ±	5.70E-08
True Value					**3.52E-08**			**3.06E-06**			**2.33E-07**	
	30ppb 4321B uranium (blanks)		4321b1	1.75E-10 <	1.75E-10		1.97E-08	7.82E-08 ±	2.87E-09	4.83E-08 <	4.83E-08	
	30ppb 4321B uranium (blanks)		4321b2	1.42E-10 <	1.42E-10		1.60E-08	3.46E-08 ±	1.52E-08	3.92E-08 <	3.92E-08	
	10ppb U030 uranium (samples)		U030e		< 1.06E-09			< 1.70E-07			< 1.10E-07	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB3	1.94E-06 <	1.94E-06		2.19E-04 <	2.19E-04		5.38E-04 <	5.38E-04	
01-00430-PB1	01-00430-PB1 + spike	PROCESS BLANK	PBS2	2.20E-06	7.49E-04 ±	2.38E-05	2.48E-04	6.38E-02 ±	6.13E-04	6.09E-04	5.01E-03 ±	3.02E-05
Spike Concentration					7.07E-08			6.03E-06			4.73E-07	
Spike Recovery					**100%**			**99%**			**101%**	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB4	1.89E-06 <	1.89E-06		2.13E-04 <	2.13E-04		5.22E-04 <	5.22E-04	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.54E-06 <	1.54E-06		1.74E-04	2.23E-04 ±	1.91E-05	4.27E-04 <	4.27E-04	
01-00430	01-00430	AN-102-CS-AR	Sample11	5.64E-06	9.04E-04 ±	2.49E-05	9.01E-04	5.60E-02 ±	2.16E-03	5.81E-04	1.51E-02 ±	3.55E-04
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample12	8.10E-06	9.38E-04 ±	1.81E-05	1.29E-03	5.51E-02 ±	6.15E-04	8.34E-04	1.50E-02 ±	1.19E-03
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample13	6.13E-06	8.94E-04 ±	2.66E-05	9.78E-04	5.15E-02 ±	1.14E-03	6.31E-04	1.39E-02 ±	3.83E-04
Duplicate %RPD					**1.2%**			**8.4%**			**8.1%**	
01-00430	01-00430 + spike	AN-102-CS-AR	Sample15	6.51E-06	1.19E-03 ±	3.15E-05	1.04E-03	8.83E-02 ±	2.35E-03	6.70E-04	1.65E-02 ±	6.04E-04
Spike Concentration					3.38E-08			3.82E-06			1.61E-07	
Spike Recovery					**96%**			**126%**			**69%**	
01-00430-MS	01-00430-MS2	AN-102-CS-AR	Sample14	6.86E-06	7.33E-04 ±	1.04E-05	1.10E-03	2.93E-02 ±	8.93E-04	7.06E-04	7.55E-03 ±	5.50E-04
01-00442	01-00442	AN-102-CS-DF	Sample8	4.42E-05	6.30E-04 ±	3.08E-05	7.05E-03	1.43E-01 ±	2.89E-03	4.55E-03	3.88E-02 ±	4.01E-03
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample9	4.24E-05	5.71E-04 ±	2.77E-05	6.77E-03	1.34E-01 ±	4.00E-03	4.37E-03	3.66E-02 ±	3.64E-04
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample10	4.07E-05	5.70E-04 ±	3.15E-05	6.50E-03	1.39E-01 ±	5.00E-03	4.19E-03	3.51E-02 ±	4.35E-03
Duplicate %RPD					**10.1%**			**2.7%**			**10.0%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019
Log-In: 01-00430-I
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [signature] 6-1-01

Analyst: [signature] 6/1/01

Unless otherwise specified, the results are reported in µg analyte/g of wet centrifuged solids for ^{127}I, µCi analyte/g of wet centrifuged solids for ^{129}I.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	127I µg/g	±	1SD	MDL µCi/g	129I µCi/g	±	1SD
	1%HNO3 (blanks)		ICB	<	1.39E-05			<	1.81E-09		
	1%HNO3 (blanks)		CCB1	<	1.47E-05			<	1.96E-09		
	1%HNO3 (blanks)		CCB2	<	1.34E-05			<	1.78E-09		
	1%HNO3 (blanks)		CCB3	<	1.33E-05			<	1.78E-09		
	1%HNO3 (samples)		ICB	<	6.01E-06			<	2.46E-09		
	1%HNO3 (samples)		CCB1	<	7.11E-06			<	2.86E-09		
	1%HNO3 (samples)		CCB2	<	8.50E-06			<	3.42E-09		
	1%HNO3 (samples)		CCB3	<	7.17E-06			<	2.88E-09		
	ICV (blanks)		ICV1		5.59E-05	±	6.49E-06		8.35E-09	±	6.04E-10
	CCV (blanks)		CCV2		4.72E-05	±	7.76E-07		9.25E-09	±	4.67E-10
	CCV (blanks)		CCV4		5.22E-05	±	6.03E-06		7.76E-09	±	2.16E-10
	CCV (blanks)		CCV6		5.46E-05	±	1.70E-06		8.09E-09	±	8.30E-10
True Value					**5.00E-05**				**8.71E-09**		
	ICV (samples)		ICV1		4.79E-05	±	5.63E-06		8.22E-09	±	1.20E-09
	CCV (samples)		CCV1		4.93E-05	±	4.95E-06		8.40E-09	±	4.81E-10
	CCV (samples)		CCV3		4.76E-05	±	3.56E-06		7.61E-09	±	6.08E-10
	CCV (samples)		CCV5		4.02E-05	±	1.40E-05		7.98E-09	±	8.53E-10
True Value					**5.00E-05**				**8.71E-09**		
01-00430-PB1-I	01-00430-PB1-I	PROCESS BLANK	PB1	1.36E+00 <	1.36E+00			1.81E-04 <	1.81E-04		
01-00430-PB1-I	01-00430-PB1-I + spike	PROCESS BLANK	PBS3	1.41E+00	9.23E+00	±	6.44E-01	1.78E-04	6.72E-04	±	4.20E-05
Spike Concentration					1.03E-04				4.29E-05		
Spike Recovery					**101%**				**86%**		
01-00430-PB2-I	01-00430-PB2-I	PROCESS BLANK	PB2	1.45E+00 <	1.45E+00			1.94E-04 <	1.94E-04		
01-00430-BS2-I	01-00430-BS2-I	BLANK SPIKE	BS3	1.25E+00 <	1.25E+00			1.67E-04	6.81E-04	±	9.52E-05
Spike Concentration									4.35E-05		
Spike Recovery									**88%**		
01-00430-I	01-00430-I	AN-102-CS-AR	Sample11	5.49E-01 <	5.49E-01			2.21E-04 <	2.21E-04		
01-00430-DUP-I	01-00430-DUP-I	AN-102-CS-AR	Sample13	5.90E-01 <	5.90E-01			2.37E-04 <	2.37E-04		
01-00430-I	01-00430-I duplicate	AN-102-CS-AR	Sample12	4.91E-01 <	4.91E-01			1.97E-04 <	1.97E-04		
01-00430-I	01-00430-I + spike	AN-102-CS-AR	Sample15	5.96E-01	9.24E+00	±	4.45E-01	2.10E-04	9.74E-04	±	5.02E-05
Spike Concentration					1.43E-04				8.66E-05		
Spike Recovery					**140%**				**87%**		
01-00430-MS2-I	01-00430-MS2-I	AN-102-CS-AR	Sample14	1.10E+00 <	1.10E+00			4.40E-04	6.52E-04	±	8.81E-05
Spike Concentration									4.16E-05		
Spike Recovery									**84%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/27/01, 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: tmPJ 6.1.01

Analyst: JL Bruns 6/1/01

Unless otherwise specified, the results are reported in µCi analyte/g of wet centrifuged solids for radioisotopes; µg analyte/g of wet centrifuged solids for all other elements.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/g	127I µg/g	± 1SD	MDL µCi/g	129I µCi/g	± 1SD
	1%HNO3 (blanks)		ICB		< 1.05E-05			< 1.81E-09	
	1%HNO3 (blanks)		CCB1		< 4.62E-05			< 1.96E-09	
	1%HNO3 (blanks)		CCB2		< 1.01E-05			< 1.78E-09	
	1%HNO3 (blanks)		CCB3		< 1.01E-05			< 1.78E-09	
	1%HNO3 (samples)		ICB		< 1.33E-05			< 2.46E-09	
	1%HNO3 (samples)		CCB1		< 1.53E-05			< 2.86E-09	
	1%HNO3 (samples)		CCB2		< 1.84E-05			< 3.42E-09	
	ICV (blanks)		ICV1		5.48E-05 ±	6.36E-06		8.35E-09 ±	6.04E-10
True Value					**5.19E-05**			**8.71E-09**	
	CCV (blanks)		CCV1		4.62E-05 ±	7.61E-07		9.25E-09 ±	4.67E-10
	CCV (blanks)		CCV3		5.12E-05 ±	5.91E-06		7.76E-09 ±	2.16E-10
	CCV (blanks)		CCV5		5.35E-05 ±	1.66E-06		8.09E-09 ±	8.30E-10
True Value					**5.11E-05**			**8.71E-09**	
	ICV (samples)		ICV1&2		4.80E-05 ±	5.02E-06		8.22E-09 ±	1.20E-09
True Value					**5.19E-05**			**8.71E-09**	
	CCV (samples)		CCV1&2		4.93E-05 ±	4.42E-06		8.40E-09 ±	4.81E-10
	CCV (samples)		CCV3&4		4.78E-05 ±	3.17E-06		7.61E-09 ±	6.08E-10
True Value					**5.11E-05**			**8.71E-09**	
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB5	1.15E-01 <	1.15E-01		2.04E-05 <	2.04E-05	
01-00430-PB1	01-00430-PB1+ spike	PROCESS BLANK	PBS2&5	1.29E-01	1.15E+00 ±	7.18E-02	2.17E-05	7.03E-05 ±	1.43E-05
Spike Concentration					1.09E-04			6.64E-09	
Spike Recovery					**106%**			**76%**	
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB6	1.13E-01 <	1.13E-01		2.00E-05 <	2.00E-05	
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	1.06E-01 <	1.06E-01		1.87E-05 <	1.87E-05	
01-00430	01-00430	AN-102-CS-AR	Sample4	5.09E-01	2.43E+00 ±	1.17E-01	5.79E-05	1.53E-04 ±	8.35E-06
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample3	5.44E-01	2.46E+00 ±	4.59E-01	6.20E-05	1.70E-04 ±	2.76E-05
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample2	4.59E-01	2.35E+00 ±	4.40E-01	5.22E-05	1.46E-04 ±	1.76E-05
Duplicate %RPD					**3.3%**			**4.7%**	
01-00430	01-00430 + spike	AN-102-CS-AR	Sample6&7	8.35E-01	4.43E+00 ±	3.02E+00	4.94E-05	4.23E-04 ±	2.76E-05
Spike Concentration					5.91E-05			1.60E-08	
Spike Recovery					**116%**			**92%**	
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample5	6.37E-01	2.27E+00 ±	1.83E-01	7.25E-05	1.51E-04 ±	1.07E-05
01-00442	01-00442	AN-102-CS-DF	Sample8	1.87E-01	8.37E-01 ±	1.45E-01	2.13E-05	6.10E-05 ±	1.02E-05
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	1.82E-01	6.12E-01 ±	3.62E-02	2.07E-05	5.50E-05 ±	1.69E-06
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample9	1.80E-01	7.57E-01 ±	3.00E-02	2.05E-05	6.80E-05 ±	5.01E-06
Duplicate %RPD					**10.1%**			**10.8%**	

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/3/01 to 4/30/01

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/30/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6.1.01

Analyst: [signature] 6/1/01

Log-In Number	Sample ID	Client ID	ICP/MS ID	Total Uranium Analyzed µg/g ± 1SD	U233 Abundance	U234 Abundance	U235 Abundance	U236 Abundance	U238 Abundance
	1%HNO3		ICB	< 1.92E-06	-1.75E-02	-2.92E-02	-3.75E-02	-2.03E-02	1.09E+00
	1%HNO3		CCB1	< 1.78E-06	-7.01E-03	1.43E-02	-7.85E-03	-9.28E-03	1.00E+00
	1%HNO3		CCB2	< 1.71E-06	6.86E-04	3.04E-03	1.16E-02	2.63E-04	9.85E-01
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	< 5.09E-01	1.55E-03	9.56E-03	1.02E-02	5.30E-03	9.75E-01
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	< 5.23E-01	-1.12E-03	2.07E-03	1.41E-02	3.74E-03	9.80E-01
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	7.43E+01 ± 2.21E+00	1.11E-05	1.40E-05	2.69E-03	7.93E-06	9.97E-01
01-00430	01-00430	AN-102-CS-AR	Sample6	6.70E+01 ± 3.46E+00	9.50E-05	7.74E-05	6.83E-03	3.10E-04	9.93E-01
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	6.39E+01 ± 1.11E+00	8.23E-05	7.29E-05	7.13E-03	2.70E-04	9.93E-01
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	6.47E+01 ± 1.49E+00	7.64E-05	6.46E-05	7.19E-03	3.18E-04	9.92E-01
01-00442	01-00442	AN-102-CS-DF	Sample9	2.80E+02 ± 2.93E+00	4.04E-04	7.63E-05	7.39E-03	3.65E-04	9.92E-01
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	2.77E+02 ± 2.34E+00	4.30E-04	6.97E-05	7.23E-03	3.36E-04	9.92E-01
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	2.81E+02 ± 7.43E+00	3.91E-04	6.82E-05	7.32E-03	3.34E-04	9.92E-01
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	6.60E+02 ± 1.62E+01	1.80E-05	2.09E-05	3.12E-03	2.88E-05	9.97E-01

Log-In Number	Sample ID	Client ID	ICP/MS ID	Total Uranium Analyzed µg/g ± 1SD	U233 µCi/g ± 1SD	U234 µCi/g ± 1SD	U235 µCi/g ± 1SD	U236 µCi/g ± 1SD	U238 µCi/g ± 1SD
	1%HNO3		ICB	< 1.92E-06	-3.23E-10	-3.50E-10	-1.55E-13	-2.52E-12	7.02E-13
	1%HNO3		CCB1	< 1.78E-06	-1.20E-10	1.58E-10	-3.02E-14	-1.07E-12	6.00E-13
	1%HNO3		CCB2	< 1.71E-06	1.13E-11	3.24E-11	4.29E-14	2.91E-14	5.66E-13
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB1	< 5.09E-01	7.61E-06	3.03E-05	1.12E-08	1.75E-07	1.67E-07
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB2	< 5.23E-01	-5.67E-06	6.75E-06	1.59E-08	1.27E-07	1.72E-07
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	7.43E+01 ± 2.21E+00	7.95E-06 ± 1.17E-06	6.50E-06 ± 6.52E-07	4.32E-07 ± 2.21E-08	3.81E-08 ± 3.24E-08	2.49E-05 ± 1.34E-06
				%RSD	14.7%	10.0%	5.1%	85.0%	5.4%
01-00430	01-00430	AN-102-CS-AR	Sample6	6.70E+01 ± 3.46E+00	6.13E-05 ± 1.21E-05	3.24E-05 ± 6.04E-06	9.89E-07 ± 3.72E-08	1.34E-06 ± 8.29E-08	2.24E-05 ± 8.87E-07
				%RSD	19.77%	18.7%	3.8%	6.2%	4.0%
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample7	6.39E+01 ± 1.11E+00	5.07E-05 ± 8.95E-06	2.91E-05 ± 6.55E-06	9.84E-07 ± 3.12E-08	1.12E-06 ± 4.56E-08	2.13E-05 ± 5.75E-07
				%RSD	17.7%	22.5%	3.2%	4.1%	2.7%
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample8	6.47E+01 ± 1.49E+00	4.77E-05 ± 7.17E-06	2.61E-05 ± 5.46E-06	1.01E-06 ± 3.62E-08	1.33E-06 ± 2.46E-07	2.16E-05 ± 9.45E-07
				%RSD	15.04%	21.0%	3.6%	18.4%	4.4%
01-00442	01-00442	AN-102-CS-DF	Sample9	2.80E+02 ± 2.93E+00	1.09E-03 ± 5.88E-05	1.33E-04 ± 2.06E-05	4.47E-06 ± 8.62E-08	6.62E-06 ± 1.59E-07	9.34E-05 ± 6.57E-07
				%RSD	5.4%	15.5%	1.9%	2.4%	0.7%
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample10	2.77E+02 ± 2.34E+00	1.15E-03 ± 6.85E-05	1.20E-04 ± 5.14E-06	4.33E-06 ± 1.55E-07	6.02E-06 ± 2.45E-07	9.24E-05 ± 1.44E-06
				%RSD	6.0%	4.3%	3.6%	4.1%	1.6%
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample11	2.81E+02 ± 7.43E+00	1.06E-03 ± 5.67E-05	1.20E-04 ± 1.85E-05	4.44E-06 ± 8.53E-08	6.08E-06 ± 4.70E-07	9.37E-05 ± 2.17E-06
				%RSD	5.3%	15.5%	1.9%	7.7%	2.3%
01-00430-MS2	01-00430-MS2	AN-102-CS-AR	Sample12	6.60E+02 ± 1.62E+01	1.14E-04 ± 1.81E-05	8.60E-05 ± 1.38E-05	4.45E-06 ± 1.90E-07	1.23E-06 ± 1.47E-07	2.21E-04 ± 1.26E-05
				%RSD	15.9%	16.0%	4.3%	11.9%	5.7%
				1µg=µCi	0.00963744	0.0062356	2.16E-06	6.47E-05	3.36E-07

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/11/01
Instrument Filenames: Experiment (11APR01), Procedure(010411a), Element Menu (100-210)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/14/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [illegible] 6-1-01

Analyst: [signature] 6/1/01

Log-In Number	Sample ID	Client ID	ICP/MS ID		U-233 Abundance	U-234 Abundance	U-235 Abundance	U-236 Abundance	U-238 Abundance
	1%HNO3		ICB	Average	-2.41E-02	-1.92E-02	3.48E-02	-1.00E-02	9.94E-01
				stdev	8.45E+01	1.88E+02	2.07E+02	7.24E+01	2.98E+02
				%SD	-350176.3%	-982338.8%	593058.0%	-722644.2%	29991.0%
	U030 uranium ~2.5ppb		U030a	Average	1.00E-05	2.17E-04	2.99E-02	1.79E-04	9.70E-01
				stdev	7.26E-06	4.16E-05	1.63E-04	3.97E-05	1.52E-02
				%SD	72.6%	19.2%	0.5%	22.2%	1.6%
				True Value		**1.90E-04**	**3.05E-02**	**2.00E-04**	**9.69E-01**
	U030 uranium ~20ppb		U030c	Average	-2.24E-06	1.97E-04	3.10E-02	2.07E-04	9.69E-01
				stdev	4.01E-06	1.77E-05	2.01E-03	1.67E-05	6.24E-02
				%SD	-179.1%	9.0%	6.5%	8.1%	6.4%
				True Value		**1.90E-04**	**3.05E-02**	**2.00E-04**	**9.69E-01**
	U030 uranium ~10ppb		U030d	Average	5.78E-06	1.86E-04	3.04E-02	1.96E-04	9.69E-01
				stdev	2.52E-06	9.76E-06	2.37E-03	1.93E-05	4.97E-02
				%SD	43.6%	5.2%	7.8%	9.9%	5.1%
				True Value		**1.90E-04**	**3.05E-02**	**2.00E-04**	**9.69E-01**
	NIST 4321b uranium 10ppb		4321b1	Average	4.20E-07	4.54E-05	7.32E-03	-1.22E-06	9.93E-01
				stdev	1.17E-05	9.99E-06	9.94E-05	2.96E-06	1.84E-02
				%SD	2795.2%	22.0%	1.4%	-242.4%	1.9%
				True Value		**5.29E-05**	**7.20E-03**		**9.93E-01**
	NIST 4321b uranium 5ppb		4321b2	Average	8.76E-07	4.86E-05	7.35E-03	-3.31E-06	9.93E-01
				stdev	1.00E-05	2.16E-05	1.67E-04	8.51E-06	1.98E-02
				%SD	1144.9%	44.5%	2.3%	-257.3%	2.0%
				True Value		**5.29E-05**	**7.20E-03**		**9.93E-01**
	1%HNO3		CCB2	Average	9.31E-03	8.09E-03	-1.55E-02	-2.99E-04	1.01E+00
				stdev	1.75E-02	1.79E-02	7.35E-03	1.55E-02	6.48E-02
				%SD	187.7%	221.6%	-47.4%	-5181.7%	6.4%
01-00442	01-00442	AN-102-CS-DF	Sample1	Average	4.04E-04	7.63E-05	7.39E-03	3.65E-04	9.92E-01
				stdev	2.18E-05	1.18E-05	1.43E-04	8.78E-06	6.98E-03
				%SD	5.4%	15.5%	1.9%	2.4%	0.7%
01-00442-DUP	01-00442-DUP	AN-102-CS-DF	Sample 2	Average	4.30E-04	6.97E-05	7.23E-03	3.36E-04	9.92E-01
				stdev	2.56E-05	2.98E-06	2.59E-04	1.37E-05	1.55E-02
				%SD	6.0%	4.3%	3.6%	4.1%	1.6%
01-00442	01-00442 duplicate	AN-102-CS-DF	Sample 3	Average	3.91E-04	6.82E-05	7.32E-03	3.34E-04	9.92E-01
				stdev	2.09E-05	1.06E-05	1.41E-04	2.59E-05	2.29E-02
				%SD	5.3%	15.5%	1.9%	7.7%	2.3%
01-00430	01-00430	AN-102-CS-AR	Sample 4	Average	9.50E-05	7.74E-05	6.83E-03	3.10E-04	9.93E-01
				stdev	1.88E-05	1.44E-05	2.57E-04	1.91E-05	3.94E-02
				%SD	20%	18.7%	3.8%	6.2%	4.0%
01-00430-DUP	01-00430-DUP	AN-102-CS-AR	Sample 5	Average	8.23E-05	7.29E-05	7.13E-03	2.70E-04	9.93E-01
				stdev	1.45E-05	1.64E-05	2.26E-04	1.10E-05	2.68E-02
				%SD	17.7%	22.5%	3.2%	4.1%	2.7%
01-00430	01-00430 duplicate	AN-102-CS-AR	Sample 6	Average	7.64E-05	6.46E-05	7.19E-03	3.18E-04	9.92E-01
				stdev	1.15E-05	1.35E-05	2.59E-04	5.87E-05	4.34E-02
				%SD	15%	21.0%	3.6%	18.4%	4.4%
01-00430-MS	01-00430-MS	AN-102-CS-AR	Sample 7	Average	1.80E-05	2.09E-05	3.12E-03	2.88E-05	9.97E-01
				stdev	2.85E-06	3.35E-06	1.33E-04	3.44E-06	5.66E-02
				%SD	15.9%	16.0%	4.3%	11.9%	5.7%
	1%HNO3		CCB2	Average	-1.52E-02	1.32E-02	-6.76E-02	-6.87E-03	1.06E+00
				stdev	3.15E-02	4.68E-02	4.48E-02	3.66E-02	2.52E-01
				%SD	-207%	354.2%	-66.3%	-532.8%	23.7%
	U030 uranium ~10ppb		U030c	Average	-2.49E-07	1.70E-04	3.01E-02	1.93E-04	9.70E-01
				stdev	9.74E-06	3.35E-05	7.93E-04	1.46E-05	2.67E-02
				%SD	-3909.3%	19.7%	2.6%	7.6%	2.8%
				True Value		**1.90E-04**	**3.05E-02**	**2.00E-04**	**9.69E-01**

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/11/01
Instrument Filenames: Experiment (11APR01), Procedure(010411a), Element Menu (100-210)

Client: Mike Urie
WP/Project: F22619/42040
ASR: 6019; 6025
Log-In: 01-00430; 01-00442
Report Date: 5/14/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: [signature] 6/1/01

Analyst: [signature] 6/1/01

Log-In Number	Sample ID	Client ID	ICP/MS ID		U-233 Abundance	U-234 Abundance	U-235 Abundance	U-236 Abundance	U-238 Abundance
	1%HNO3		ICB	Average	-1.75E-02	-2.92E-02	-3.75E-02	-2.03E-02	1.09E+00
				stdev	3.48E+01	3.18E+01	4.39E+01	4.17E+01	1.33E+02
				%SD	-199143.8%	-108750.2%	-117288.4%	-205950.5%	12277.8%
	NIST 4321b uranium 10ppb		4321b	Average	-1.88E-07	6.78E-05	7.39E-03	1.46E-06	9.93E-01
				stdev	4.48E-06	6.49E-06	2.78E-04	1.72E-06	1.84E-02
				%SD	-2387.8%	9.6%	3.8%	118.1%	1.9%
				True Value		**5.29E-05**	**7.20E-03**		**9.93E-01**
	1%HNO3		CCB1	Average	-7.01E-03	1.43E-02	-7.85E-03	-9.28E-03	1.00E+00
				stdev	1.06E-02	2.21E-02	4.70E-02	2.86E-02	3.89E-01
				%SD	-151.5%	155.2%	-599.0%	-307.9%	38.8%
01-00430-PB1	01-00430-PB1	PROCESS BLANK	PB3	Average	1.55E-03	9.56E-03	1.02E-02	5.30E-03	9.75E-01
				stdev	1.29E-03	5.11E-03	1.12E-03	9.87E-04	9.03E-02
				%SD	82.9%	53.4%	11.0%	18.6%	9.3%
01-00430-PB2	01-00430-PB2	PROCESS BLANK	PB4	Average	-1.12E-03	2.07E-03	1.41E-02	3.74E-03	9.80E-01
				stdev	3.76E-03	5.00E-03	7.24E-03	8.79E-03	6.61E-02
				%SD	-334.4%	241.4%	51.5%	234.8%	6.7%
	U030 uranium ~20ppb		U030b	Average	-8.67E-07	1.88E-04	3.03E-02	1.63E-04	9.69E-01
				stdev	4.07E-06	1.00E-05	1.70E-03	7.95E-06	5.54E-02
				%SD	-470.1%	5.3%	5.6%	4.9%	5.7%
				True Value		**1.90E-04**	**3.05E-02**	**2.00E-04**	**9.69E-01**
	NIST4321b uranium 30ppb		4321b2	Average	-7.51E-07	5.18E-05	7.21E-03	-1.47E-06	9.93E-01
				stdev	9.03E-07	1.33E-07	1.32E-04	9.96E-07	2.19E-02
				%SD	-120.21%	0.3%	1.8%	-67.9%	2.2%
				True Value		**5.29E-05**	**7.20E-03**		**9.93E-01**
	1%HNO3		CCB2	Average	6.86E-04	3.04E-03	1.16E-02	2.63E-04	9.85E-01
				stdev	1.49E-03	1.65E-03	2.32E-03	2.16E-03	1.25E-01
				%SD	217.50%	54.2%	20.0%	823.4%	12.7%
01-00430-BS2	01-00430-BS2	BLANK SPIKE	BS2	Average	1.11E-05	1.40E-05	2.69E-03	7.93E-06	9.97E-01
				stdev	1.63E-06	1.41E-06	1.38E-04	6.74E-06	5.36E-02
				%SD	14.7%	10.0%	5.1%	85.0%	5.4%

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/11/01
Instrument Filenames: Experiment (11APR01), Procedure(010411a), Element Menu (100-210)

BLANK

Battelle – PNNL / AIAL
Inorganic Analysis / 320-ICP/MS Data Report

Project / WP#: 42040 / W57911
ASR#: 6019
Client: Mike Urie
Total Samples: 4

RPL #	Client ID
01-00429 PB1	AN-102-SUP-AR
01-00429 PB2	AN-102-SUP-AR
01-00429	AN-102-SUP-AR
01-00429 DUP	AN-102-SUP-AR

Procedure: PNL-SC-01 Rev. 1, *Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: WB36913 ICP/MS, VG Elemental
512-06-01-014 Mettler AJ100 Balance

Analyst: Orville Thomas Farmer III

Report Written by: Orville Thomas Farmer III

Analysis Date(s): 9/23/01

Analysis Files: Experiments – 21SEP01
Procedures – CS-ISO
Element Menus – CS-ISO

Laboratory Record Book (LRB): LRB BNW 56465: Sample login: p.59
LRB BNW 56465: Cs Isotopics assay: p.59

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

9-25-01
Reviewed By

Concur 9-25-01

1. Analysis

Four liquid samples for Cs isotopic analysis were received from the client and were analyzed by HPIC-ICP/MS. All samples were diluted by a factor of 100 as received and screened to determine an estimate of the Cs concentration. The screening results were used to determine optimum Cs response during the on-line separation into the ICP/MS.

A Dionex HPIC system was configured on-line with the ICP/MS for the separation of Ba and Cs isotopes. A CS3 cation column was used as the stationary phase and a gradient eluent of nitric acid was used as the mobile phase for this separation. Data was collected using peak-hopping data acquisition parameters.

ICP/MS results for all solutions are reported in **atomic abundance** and are instrument blank corrected. In Table 1, reports results are in atomic abundance. In Table 2, the precision of the analysis was determined by analyzing three separate aliquots of a single unknown tank sample. These three solutions were analyzed to measure the separation and analysis procedures accuracy and precision.

Table 1: Cs Atomic Abundance

File Name	Cs-133 +/- 1 sigma	Cs-135 +/- 1 sigma	Cs-137 +/- 1 sigma
01-00429 PB1	0.017	N.D.	N.D.
01-00429 PB2	0.017	N.D.	N.D.
01-00429	0.601 +/- 0.002	0.166 +/- 0.001	0.233 +/- 0.001
01-00429 DUP	0.602 +/- 0.001	0.166 +/- 0.0001	0.232 +/- 0.002

N.D. Blanks were analyzed in the same manner as the samples. The response of the blanks was below instrument detection for Cs-135 and Cs-137 and no Cs ratios were calculated. The PB solutions were used only to subtract Cs-133 due to the preparation process.

Table 2: Cs Atomic Abundance Check solution

File Name	Cs-133 +/- 1 sigma	Cs-135 +/- 1 sigma	Cs-137 +/- 1 sigma
Check 1	0.6033 +/- 0.0023	0.1646 +/- 0.0008	0.2321 +/- 0.0020
Check 2	0.6044 +/- 0.0018	0.1634 +/- 0.0003	0.2322 +/- 0.0021
Check 3	0.6022 +/- 0.0023	0.1643 +/- 0.0002	0.2335 +/- 0.0021
Avg ratio	0.6033	0.1641	0.2326
STDEV	0.00089	0.00051	0.00064
%RSD	0.15	0.31	0.28

Currently there does not exist a known total Cs check sample and therefore a triplicate is used to measure precision of the procedure.

QC Summary:

Duplicate (DUP): A duplicate was submitted and analyzed. The duplicate RPD was determined to be 0.12% for Cs-133, 0.14% for Cs-135 and 0.40% for Cs-137.

Matrix Spike (MS): No post matrix spike was analyzed for this work.

Post Spike (PS): Not a QC requirement.

Preparation Blank (PB) and Laboratory Control Standard (LCS/BS): A preparation blank was submitted and analyzed and meet the criteria of being < 5% of the measured concentration in the samples.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB): Not a QC requirement.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): Not a QC requirement.

Internal Standard (ISTD): Not a QC requirement.

BLANK

Battelle
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Project No. 42040

Internal Distribution
File/LB

Date June 29, 2001

To M. W. Urie

From L. R. Greenwood

Subject Radiochemical Analyses for AN-102 –ASR 6019, 25

Samples of the supernates and centrifuged solids from tank AN-102 were analyzed for gamma emitters, ^{90}Sr, total alpha, U, Pu, and Am/Cm according to ASRs 6019 and 6025. Sample AN-102-CS-AR was also analyzed for ^{3}H and ^{241}Pu. The samples were acid digested in the hot cells according to procedures PNL-ALO-128 and –129 and aliquots were delivered to the laboratory for analysis. The attached reports list measured analyte activities in the original sample material in units of μCi/ml for the supernates. The solids results are reported per gram of wet weight. Sample AN-102-CS was dried prior to analysis and the results were corrected for a wt% solids value of 35.1%. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

The hot cell sample preparations were performed in two separate batches, as indicated by the horizontal lines separating the results in the attached tables. However, all of the samples were initially run together in one batch in the laboratory. The hot cell process blanks prepared with each batch of samples showed significant alpha contamination, as discussed below.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. Initially, the samples were diluted prior to gamma analysis. However, in order to meet the requested detection limits, direct aliquots of the hot cell preparations were counted for periods of 4 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. The AN-102 samples also showed the presence of ^{60}Co, ^{154}Eu, ^{155}Eu, and ^{241}Am in most of the samples. The MRQ values for extended counting time GEA were met in all cases except for ^{155}Eu for sample AN-102-SUP, where the detection limit was only slightly above the MRQ value of 9.E-2 μCi/ml. Detection limits are listed in the tables. All of the hot cell process blanks showed the presence of ^{137}Cs and some of them also showed ^{134}Cs. However, in all cases, the activities in the blanks were negligible with respect to the samples. Sample duplicates showed excellent repeatability with RPD or RSD values less than 10% except for ^{155}Eu and ^{241}Am for sample AN-102-SUP-DF where the RPD values are in reasonable agreement considering the counting uncertainties.

Total Alpha

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. The sums of the individual alpha emitters, as discussed below, are in very good agreement with the total alpha data indicating minimal losses due to alpha self-absorption. As mentioned previously, all of the hot cell preparation batches showed significant alpha contamination. Although this effect is not so evident in the total alpha data, the data for individual alpha emitters is compromised in some cases, as discussed in the following section. The LCS and matrix spike recoveries with ^{239}Pu were 103% and 100%, respectively. No alpha contamination was detected in the laboratory blank. RPD and RSD values are less than 3% in all cases.

Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and the samples were counted by alpha spectrometry according to PNL-ALO-422. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. Both the plutonium and americium radiochemical yields were excellent, averaging about 95%. Neither Pu, Am nor Cm were detected in the laboratory preparation blank. However, all of the hot cell process blanks indicated significant contamination for some of the isotopes. For the first hot cell batch with samples AN-102-SUP-AR and AN-102-SUP-DF, the process blank contamination is 20% and 100% of the sample activities, respectively, for ^{238}Pu, and 10% and 37% of the sample activities, respectively, for $^{243/244}$Cm. For the second hot cell batch with samples AN-102-CS-AR and AN-102-CS-DF, the process blank contamination levels are 5% and 12% of the sample activities, respectively, for $^{243/244}$Cm. For both batches, the process blank contamination levels are not significant for the other alpha emitting isotopes.

The LCS and matrix spike recoveries were 96 to 107% for both Pu and Am. RPD and RSD values were generally quite good except for ^{243,244}Cm in sample AP-101-DF, where the counting uncertainties are quite high.

The ^{241}Pu beta activity was measured in sample AN-102-CS-AR by placing the precipitation plates from the Pu/AEA measurements in glass scintillation vials, adding scintillation cocktail, and liquid scintillation counting according to procedure PNL-ALO-474. A blank filter was used to determine the background counting rate and a blank filter was spiked with a ^{241}Pu standard to determine the beta counting efficiency. Radiochemical yields were taken from the Pu/AEA tracer measurements. Blank spike and sample spike yields were determined in the Pu/AEA measurements and were not separately measured for ^{241}Pu. No ^{241}Pu was detected in the sample or duplicate and the detection limit was well below the MRQ value of 1.2 μCi/g. Weak ^{241}Pu activity was seen in the hot cell process blanks, although the level was negligible with respect to the MRQ value. No activity was seen in the laboratory blank.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. However, this correction was negligible with respect to the activity in the samples. No ^{90}Sr was found in the laboratory preparation blank. All of the hot cell process blanks showed ^{90}Sr activities, although the levels are not significant. RPD values are less than 7% in all cases indicating good sample reproducibility. The LCS and matrix spike recoveries were 88% and 91%, respectively.

Total Uranium

Total uranium was measured according to procedure PNNL-ALO-4014 using Kinetic Phosphoresence Analysis (KPA). Sample solutions from the hot cell were evaporated dry with nitric acid, then re-dissolved in dilute nitric acid for uranium analysis. No uranium separation was done. Results are reported in μg/ml of supernate. The uranium content is well below the MRQ value of 780 μg/ml. The repeatability of duplicate samples is excellent with RPD values of 2%. Uranium was detected in the hot cell blanks, but at a concentration less than 1% of the sample uranium concentration.

The instrument blanks had no detectable uranium. All the instrument standards except the lowest one gave results within 3% of their known values. The lowest standard is at 2.5 times the detection limit, and has an inherently higher uncertainty.

Tritium

Sample AN-102-CS-AR was analyzed in duplicate for tritium using a water leach performed in the hot cells. The water in the sample was separated by distillation, using procedure PNL-ALO-418, and the tritium was measured by liquid scintillation counting according to procedure PNL-ALO-474. Due to the anticipated high level of ^{137}Cs in this sample, two successive distillations were performed. In spite of this precaution, the beta energy spectrum clearly showed the presence of ^{137}Cs contamination. However, using the counts in the tritium energy region of the beta spectrum as an upper limit on the tritium activity results in detection limits of 2 to 3E-3 μCi/g, a factor of 5 or more below the requested MRQ value of 1.5E-2 μCi/g. The hot cell process blank shows weak tritium contamination well below the MRQ value. However, the process blank did not show any contamination from high-energy beta emitters. No beta activities were detected in the laboratory blank and the detection limit was < 2.E-4 μCi/g. The LCS and matrix spike tritium recoveries were excellent at 99% and 100%, respectively.

Pertechnetate ^{99}Tc

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken

for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated fractions were then counted according to procedure RPG-CMC-408. The samples were also counted by liquid scintillation counting according to procedure PNL-ALO-474 to confirm that the beta energy spectra matched that of ^{99}Tc and that no other beta emitters were present. The LCS blank spike recovery of a ^{99}Tc standard was 92%. However, a matrix spike gave a standard recovery of 248%. The reason for this high recovery is not known. Sample duplicates and lab replicates gave good agreement with RPD values less than 1%. The ^{99}Tc activities in the hot cell and laboratory blanks were negligible or non-detectable and well below the requested MRQ values.

Review:
C. Sodergren 6-29-01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurement Center

01-0429

8/23/01

Client : Mike Urie

Cognizant Scientist: L R Greenwood

Date : 8/23/01

Concur : T Trang-Le

Date : 8/23/01

PNL-ALO-476 (Sr-90) PNL-ALO-450 (GEA)

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID / Client ID		Sr-90 Error %	Cs-134 Error %	Cs-137 Error %	Co-60 Error %	Sb-125	SnSb-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0429PB1		1.47E-2	4.58E-4	6.42E-3							
Process Blank		3%	16%	3%							
	MDA	<4.E-4	<2.E-4	<2.E-4	<3.E-4	<6.E-4	<2.E-4	<9.E-4	<6.E-4	<6.E-4	<6.E-4
01-0429PB2		4.47E-3		2.17E-3							
Process Blank		5%		7%							
	MDA	<4.E-4	<3.E-4	<3.E-4	<3.E-4	<5.E-4	<2.E-4	<9.E-4	<6.E-4	<6.E-4	<6.E-4
01-0429		5.58E+1		3.66E+2	8.58E-2				2.30E-1		
AN-102-SUP-AR		3%		2%	3%				3%		
	MDA	<7.E-1	<8.E-3	<5.E-2	<2.E-3	<2.E-1	<1.E-1	<9.E-3	<9.E-3	<1.E-1	<1.E-1
01-0429 DUP		5.86E+1		3.71E+2	8.39E-2				2.31E-1		1.65E-1
AN-102-SUP-AR		3%		2%	3%				3%		31%
	MDA	<7.E-1	<8.E-3	<5.E-2	<3.E-3	<2.E-1	<1.E-1	<8.E-3	<1.E-2	<1.E-1	<1.E-1
	RPD	5%		1%	2%				1%		
01-0441		1.41E+1		1.02E+2	2.55E-2				5.83E-2	4.00E-2	4.17E-2
AN-102-SUP-DF		3%		2%	2%				2%	10%	14%
	MDA	<4.E-1	<1.E-3	<7.E-3	<4.E-4	<3.E-2	<1.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
01-0441 DUP		1.34E+1		1.02E+2	2.53E-2				5.80E-2	3.53E-2	3.22E-2
AN-102-SUP-DF		3%		2%	2%				2%	10%	16%
	MDA	<4.E-1	<1.E-3	<7.E-3	<3.E-4	<3.E-2	<1.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
	RPD	5%		0%	1%				1%	12%	26%
01-0441 REP		1.33E+1									
AN-102-SUP-DF		3%									
	MDA	<4.E-1									
01-0430PB1		7.84E-2		7.32E-3							
Process Blank		3%		6%							
	MDA	<2.E-3	<5.E-4	<6.E-4	<4.E-3	<1.E-3	<5.E-4	<2.E-3	<2.E-3	<2.E-3	<1.E-3
01-0430PB2		4.59E-2	9.60E-4	2.48E-2							
Process Blank		3%	13%	3%							
	MDA	<2.E-3	<4.E-4	<5.E-4	<4.E-3	<1.E-3	<5.E-4	<2.E-3	<2.E-3	<2.E-3	<2.E-3
01-0430		1.40E+2		2.16E+2	5.76E-2				5.08E-1	3.14E-1	4.12E-1
AN-102-CS-AR		3%		2%	3%				2%	9%	12%
	MDA	<1.E+0	<6.E-3	<4.E-2	<3.E-3	<2.E-1	<7.E-2	<1.E-2	<9.E-3	<2.E-1	<2.E-1
01-0430 DUP		1.47E+2		2.16E+2	5.66E-2				5.15E-1	3.26E-1	4.29E-1
AN-102-CS-AR		3%		2%	4%				2%	10%	12%
	MDA	<2.E+0	<6.E-3	<4.E-2	<4.E-3	<2.E-1	<8.E-2	<1.E-2	<9.E-3	<2.E-1	<2.E-1
	RPD	5%		0%	2%				1%	4%	4%
01-0442**		4.31E+2		8.49E+1	2.07E-2			2.15E-2	1.22E+0	7.27E-1	1.16E+0
AN-102-CS-DF		3%		2%	2%			5%	2%	3%	4%
	MDA	<7.E+0	<2.E-3	<6.E-3	<6.E-4	<2.E-2	<1.E-2	<3.E-3	<3.E-3	<2.E-2	<2.E-2
01-0442 DUP**		4.02E+2		8.39E+1	2.11E-2			2.18E-2	1.21E+0	7.16E-1	1.15E+0
AN-102-CS-DF		3%		2%	2%			5%	2%	3%	4%
	MDA	<7.E+0	<2.E-3	<6.E-3	<7.E-4	<2.E-2	<1.E-2	<3.E-3	<3.E-3	<2.E-2	<2.E-2
	RPD	7%		1%	2%			2%	1%	1%	0%
Sample spike		88%									
Blank spike		91%									
Blank		<1.E-4									

*Supernates are reported per ml and solids are reported per gram.

**Sample AN-102-CS-DF is reported per gram of wet weight using a value of 35.1% wt% solids.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurement Center

01-0429

08/20/01

Client : Mike Urie

Cognizant Scientist: L P Greenwood Date : 8/20/01

Concur : T Trang-Le Date : 8/23/01

RPG-CMC-408 (Total Alpha)
PNL-ALO-417,496 (Pu, Am/Cm)

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID Client ID		Alpha Error %	Pu-238 Error %	Pu-239/240 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	Sum of Alpha Error %
01-0429PB1		1.10E-3	3.20E-4	4.07E-5	1.06E-4	5.71E-4		1.04E-3
Process Blank		5%	3%	6%	6%	3%		4%
	MDA	<6.E-5	<2.E-6	<2.E-6	<4.E-6	<4.E-6	<2E-6	
01-0429PB2		3.60E-4	9.49E-5	1.67E-5	4.45E-5	1.56E-4		3.12E-4
Process Blank		10%	3%	6%	4%	3%		4%
	MDA	<6.E-5	<6.E-7	<5.E-7	<1.E-6	<1.E-6	<5E-7	
01-0429		1.66E-1	1.66E-3	6.00E-3	1.49E-1	6.51E-3	6.53E-4	1.64E-1
AN-102-SUP-AR		4%	14%	6%	2%	6%	20%	3%
	MDA	<5.E-3	<3.E-4	<2.E-4	<2.E-4	<2.E-4	<2.E-4	
01-0429 DUP		1.66E-1	1.63E-3	5.80E-3	1.52E-1	6.91E-3	6.04E-4	1.67E-1
AN-102-SUP-AR		4%	11%	5%	2%	7%	23%	3%
	MDA	<6.E-3	<1.E-4	<1.E-4	<3.E-4	<2.E-4	<2.E-4	
	RPD	0%	2%	3%	2%	6%	8%	2%
01-0441		4.60E-2	6.75E-4	1.40E-3	3.83E-2	2.26E-3	1.79E-4	4.28E-2
AN-102-SUP-DF		7%	14%	9%	2%	6%	23%	3%
	MDA	<4.E-3	<1.E-4	<9.E-5	<2.E-4	<7.E-5	<7.E-5	
01-0441 DUP		4.64E-2	3.03E-4	1.58E-3	3.66E-2	1.57E-3	1.30E-4	4.02E-2
AN-102-SUP-DF		8%	26%	8%	2%	8%	28%	3%
	MDA	<6.E-3	<2.E-4	<9.E-5	<2.E-4	<7.E-5	<6.E-5	
	RPD	1%	76%	12%	5%	36%	32%	6%
01-0430PB1		3.92E-3	1.27E-3	1.52E-4	3.33E-4	1.97E-3		3.73E-3
Process Blank		4%	2%	3%	5%	3%		4%
	MDA	<2.E-4	<2.E-6	<2.E-6	<5.E-6	<5.E-6	<4E-6	
01-0430PB2		3.11E-3	8.59E-4	9.49E-5	2.15E-4	1.61E-3		2.78E-3
Process Blank		4%	2%	4%	6%	3%		4%
	MDA	<2.E-4	<2.E-6	<2.E-6	<6.E-6	<5.E-6	<4E-6	
01-0430		4.93E-1	1.09E-2	4.16E-2	4.44E-1	1.81E-2		5.15E-1
AN-102-CS-AR		3%	11%	5%	3%	10%		4%
	MDA	<8.E-3	<2.E-3	<2.E-3	<2.E-3	<2.E-3	<1E-3	
01-0430 DUP		5.06E-1	1.29E-2	4.17E-2	4.38E-1	1.62E-2		5.09E-1
AN-102-CS-AR		3%	11%	6%	3%	11%		4%
	MDA	<2.E-2	<2.E-3	<2.E-3	<3.E-3	<2.E-3	<2E-3	
	RPD	3%	17%	0%	1%	11%		1%

Measured Activities (uCi/ml or g) with 1-sigma error*

ALO ID Client ID		Alpha Error %	Pu-238 Error %	Pu-239/240 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	Sum of Alpha Error %
01-0442**		1.35E+0	2.61E-2	1.16E-1	1.08E+0	6.80E-2	3.52E-3	1.29E+0
AN-102-CS-DF		2%	6%	3%	2%	3%	14%	3%
	MDA	<6.E-3	<7.E-4	<8.E-4	<5.E-4	<4.E-4	<4.E-4	
01-0442 DUP**		1.33E+0	2.64E-2	1.08E-1	1.08E+0	4.32E-2	2.48E-3	1.26E+0
AN-102-CS-DF		2%	6%	3%	2%	5%	22%	3%
	MDA	<7.E-3	<6.E-4	<5.E-4	<1.E-3	<9.E-4	<7.E-4	
	RPD	2%	1%	7%	0%	45%	35%	3%
Sample spike		103%		105%	97%			
Blank spike		100%		107%	96%			
Blank		<4.E-3	<6.E-6	<6.E-6	<8.E-6	<6.E-6	<4.E-6	

*Supernates are reported per ml and solids are reported per gram.
**Sample AN-102-CS-DF is reported per gram of wet weight using a value of 35.1% wt% solids.

Battelle Pacific Northwest Laboratory 01-0429
Radiochemical Processing Group-325 Building
Chemical Measurement Center 08/20/01

Client : Mike Urie

Cognizant Scientist: L R Greenwood Date : 8/20/01

Concur : T Trang-Le Date : 8/26/01

PNL-ALO-432 (Tc-99)

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID		Pertechnetate Tc-99 Error %	 MDA
01-0429 CB Process Blank			<2.E-6
01-0429 AN-102-SUP-AR		5.55E-2 4%	<6.E-6
01-0441 AN-102-SUP-DF		2.20E-2 4%	<4.E-6
01-0441 Rep AN-102-SUP-DF		2.17E-2 4%	<4.E-6
	RPD	1%	
Sample spike		248%	
Blank spike		92%	
Blank		7.89E-06 23%	<6.E-6

Battelle Pacific Northwest Laboratory 01-0429
Radiochemical Processing Group-325 Building
Chemical Measurement Center 08/20/01

Client : Mike Urie

Cognizant Scientist: J R Greenwood Date : 8/20/01

Concur : T Traing-le Date : 8/23/01

PNL-ALO-418, 474 (H-3)
PNL-ALO-417, 474 (Pu-241)

Measured Activities (uCi/g) with 1-sigma error*

ALO ID Client ID	H-3* Error %	MDA	Pu-241 Error %	MDA
01-0430 PB Process Blank	7.46E-4 7%	<2.E-4	8.17E-3 6%	<2.E-4
01-0430 PB2 Process Blank			6.18E-3 6%	<2.E-4
01-0430 AN-102-CS-AR		<2.E-3		<1.E-1
01-0430 DUP AN-102-CS-AR		<3.E-3		<1.E-1
RPD				
Sample spike	100%			
Blank spike	99%			
Blank		<2.E-4		<2.E-4

*The tritium samples showed weak contamination with Cs-137 above the detection limit of <2.E-4 uCi/g.

Battelle Pacific Northwest Laboratory 01-0429
Radiochemical Processing Group-325 Building
Chemical Measurement Center 08/27/01

Client : Mike Urie

Cognizant Scientist: L K Greenwood Date : 8/27/01

Concur : C Jackgino Date : 8-27-01

RPG-CMC-4014 (Uranium KPA)

Measured Activities (ug/ml) with 1-sigma error

ALO ID Client ID	Uranium Error %	MDL
01-0429PB1 Process Blank	6.08E-2 2%	6.E-3
01-0429PB2 Process Blank	3.23E-2 2%	6.E-3
01-0429 AN-102-SUP-AR	1.15E+1 5%	6.E-3
01-0429 DUP AN-102-SUP-AR	1.23E+1 5%	6.E-3
RPD	7%	
01-0441 AN-102-SUP-DF	1.44E+1 4%	5.E-3
01-0441 Rep AN-102-SUP-DF	1.38E+1 4%	5.E-3
01-0441 DUP AN-102-SUP-DF	1.55E+1 4%	5.E-3
RSD	6%	
Lab Blank	<6.E-3	

Instrument Standards

	Observed	Expected	Spike Yield
Blank 1	5.05E-6	-	
Blank 2	0.00E+0	-	
Blank 3	1.30E-5	-	
IV 5.E-1	5.07E-1	5.05E-1	100%
IV 5.E-1	5.07E-1	5.05E-1	100%
*IV 5.E-5	6.20E-5	5.07E-5	122%

*This standard is near the detection limit, where uncertainty is higher.
Sample solutions ranged from 1.06E-4 to 6.22E-2 µg/mL

Battelle
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Project No.

Internal Distribution
File/LB

Date August 30, 2001

To Mike Urie

From Chuck Soderquist C Soderquist

Subject C-14 Analysis for ASR 6019

The carbon-14 concentration has been measured in the AN-102 centrifuged solids as-received sample (lab number 01-430). The average measured sample C-14 concentration is 6.3E-4 μCi/g ± 25% at 1s. The duplicates agree reasonably well, considering the high analytical uncertainty. The detection limit attained is 4E-4 μCi/g. (The MRQ is 1.8E-3 μCi/mL.)

Duplicate samples of AN-102 centrifuged solids were weighed out in a hot cell, then removed from the hot cell. Each sample size was around 0.13 grams. The samples were oxidized by the hot persulfate method (procedure PNL-ALO-381) and the carbon dioxide was caught in a trap. The trap solution was counted by liquid scintillation for C-14 activity (procedure PNL-ALO-474). All the liquid scintillation counts were prepared and counted in duplicate.

Six spikes were processed through the hot persulfate sample oxidation. One spike was lost when a flask cracked. The average spike recovery, using the remaining five spikes, was 70% with a standard deviation of about 5%. All the sample results were corrected for this spike recovery. Because the spikes were used for correcting the sample results for analyte recovery, they could not be reported as spikes.

The four laboratory blanks processed through the hot persulfate oxidation averaged 1.E-4 μCi/g. All were at or below the detection limit. The hot cell blank came out 2E-4 μCi/g, also below the detection limit.

Two matrix spikes were processed through the hot persulfate oxidation. The spike recovery of the first is 86%. The second matrix spike came up contaminated with another beta emitter (identity unknown, but with a higher energy than C-14) and was unusable and the data had to be thrown out. (This matrix spike was the only sample in the batch materially affected by such contamination. None of the other liquid scintillation beta counts had any significant contamination by higher energy beta emitters.)

Three direct C-14 spikes (not processed by hot sulfate oxidation) were counted by liquid scintillation to directly measure the counting efficiency. The one with the lowest C-14 concentration gave a high result (120% counting efficiency) and had to be thrown out. The reason for this is unknown. The other two direct spikes averaged 0.95 counts per decay, about what is expected for C-14.

Review:
LR Greenwood
8-30-01

Battelle, Pacific Northwest Laboratory 01-429
Radiochemical Processing Group-325 Building 8/30/2001
Chemical Measurement Center

Client : Mike Urie

Cognizant Scientist: E. Sodergren Date : 8-30-01

Concur : L R Greenwood Date : 8-30-01

Procedure PNL-ALO-381, Carbon analysis by hot persulfate oxidation
Procedure PNL-ALO-474, Liquid scintillation counting

Sample	Measured C-14 Concentration µCi/g ± 1s		MDC
01-0430 AN-102-CS-AR	5.44E-4	± 27%	3E-4
01-0430 DUP AN-102-CS-AR	7.19E-4	± 24%	4E-4
RPD	28%		
01-430 MS 1	86%		
Hot cell blank	< 4E-4		4E-4
Blank 1	< 4E-4		4E-4
Blank 2	< 4E-4		4E-4
Blank 3	< 4E-4		4E-4
Blank 4	< 4E-4		4E-4

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client: M Urie
ASR Number: 6019, 6025 Supernatants
Charge Code/Project: F22616-F22626/42040
Sample Receipt Date: 6019 - 00/29/2001
6025 – 01/23/2001
Sample Prep Date: N/A
Sample Analysis Date: 04/03-27/2001
(01-00429 Rerun) 05/10/2001
Analyst: MJ Steele

Preparation Procedure: N/A
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Raw data, standards information, preparation benchsheets filed with ASR 6019 Urie: Project 42365

Samples 01-00429, 01-00441 Final Results:

RPL Number	Sample ID	$F^{(1)}$ µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	0.25	0.25	0.50	0.25	0.500	0.500	0.500	0.500
01-00429 PB	Hot Cell Blank	< 0.25	< 0.25	< 0.50	< 0.25	< 0.50	< 0.50	< 0.50	< 0.50
	EQL	*2,500*	*2,500*	*5,000*	*2,500*	*5,000*	*5,000*	*5,000*	*5,000*
01-00429	*AN-102-SUP-AR*	*< 2500*	*8,560*	*19,100*	*< 2500*	*404,000*	*< 5000*	*32,800*	*< 5000*
01-00429 Dup	*AN-102-SUP-AR*	*< 2500*	*5,080*	*96,100*	*< 2500*	*233,000*	*< 5000*	*18,500*	*< 5000*
	RPD	*n/a*	*36*	*47*	*n/a*	*38*	*n/a*	*40*	*n/a*
01-00429 MS % Rec	*AN-102-SUP-AR MS*	*136*	*108*	*87*	*95*	*132*	*102*	*91*	*102*
	EQL	2,500	2,500	5,000	2,500	5,000	5,000	5,000	5,000
Rerun 01-00429	AN-102-SUP-AR	< 2500	4,780	85,900	< 2500	223,000	< 5000	16,800	< 5000
Rerun 01-00429 Dup	AN-102-SUP-AR	< 2500	4,860	84,800	< 2500	219,000	< 5000	16,900	< 5000
	RPD	n/a	2	1	n/a	2	n/a	1	n/a
Rerun 01-00429 MS %Rec	AN-102-SUP-AR MS	96	94	104	96	115	95	93	102
	EQL	200	200	500	250	2,000	200	400	400
01-00441	AN-102-SUP-DF	3,700	1,170	25,900	< 250	61,400	< 200	4,470	2,300
01-00441 Dup	AN-102-SUP-DF	3,700	1,130	25,800	< 250	60,600	< 200	4,390	2,270
	RPD	0	3	1	n/a	1	n/a	1	1
LCS %Rec	60/160 Spike Solution	107	105	105	106	101	104	102	107

n/a = not applicable since either or both the sample and duplicate results are not above the EQL.

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

The sample was prepared for ion chromatography anion analysis by dilution from 800-fold to 10,000-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample as dilutions less than 800-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the table. Poor reproducibility of the AN-102 supernatant analysis conducted April 3 to April 27 prompted the need for reanalysis. The AN-102 sample, duplicate, and matrix spike were reanalyzed on May 10, with the reanalysis providing good QC and reasonable comparison to the original AN-102 duplicate. The 'Rerun' results are considered the most reliable, and essentially meet all QC criteria.

Q.C. Comments:

Duplicates: The samples AN-102 Sup AR and AN-102 Sup DF were sub-sampled in the hot cell. The samples and duplicates were aliquotted at the IC workstation. The initial AN-102 Sup AR sample and duplicate analysis did not meet the RPD acceptance criteria of < 20%. The results appeared to differ by about a factor of two, suggesting the possibility of a dilution error. Additional sample and duplicate samples for AN-102 Sup AR were prepared and analyzed. The RPD for the reanalysis meets the acceptance criteria of <20%.

Matrix Spike (010207 MS High, 60/160): A matrix spike was prepared from each sample. The fluoride and nitrate matrix spikes for the initial AN-102 Sup AR sample produced very high recoveries (i.e., 36% and 36%, respectively). Like the sample and duplicate, the matrix spike for the AN-102 Sup AR sample was prepared again and reanalyzed. The AN-102 Sup DF and the reanalyzed AN-102 Sup AR matrix spiked had anion recoveries were within the 75% to 125% recovery acceptance criteria.

Laboratory Control Sample-LSC/BS (100207 MS High, 60/160): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared and measured at the same time as the Matrix Spike sample and demonstrated recoveries within the 80% to 120% acceptance criteria.

System Blank/Processing Blanks: Thirty-four system blanks were analyzed throughout the analysis runs (including the reruns). No anions were detected in the system blanks above the estimate quantitation level. Also, no anions were detected in the hot cell blank (01-00429).

Quality Control Calibration Verification Check Standards: Twenty-two mid-range verification standards were analyzed throughout the analysis runs (including the reruns). Except for one chloride measurement and seven oxalate measurement, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The chloride measurement that failed produced a recovery of 89% and the oxalate measurements produced recoveries of from 110% to 112%.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: MW Thurn Date 6-18-01

Review/Approval: MJ Steele Date 7-19-01

Archive Information:

Files: ASR 6019 6025 Sup Urie.doc	ASR 6014 6019 6025 6031 6057.xls

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	M Urie	Charge Code/Project:	F22626/42040
ASR Number:	6019 solids	Sample Receipt Date:	12/20/2000
Sample Prep Date:	N/A	Sample Analysis Date:	04/04/2001
Analyst:	MJ Steele		

Preparation Procedure: PNL-ALO-103, "Water Leach of Sludges, Soils, and other Solids Samples"
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Sample 01-00430 (wet centrifuged solids) Final Results:

RPL Number	Sample ID	F µg/g	Cl µg/g	NO_2 µg/g	Br µg/g	NO_3 µg/g	PO_4 µg/g	SO_4 µg/g	C_2O_4 µg/g
	EQL	12	12	24	12	24	24	24	24
01-00430 PB	Hot Cell Blank	< 12	44	< 24	< 12	114	< 24	< 24	< 24
	EQL	250	250	500	250	500	500	500	500
01-00430	AN-102-CS-AR	3,900	3,520	57,400	< 250	137,000	8,970	17,547	30,007
01-00430 Dup	AN-102-CS-AR	3,700	3,140	52,700	< 250	128,000	7,380	15,369	27,584
	RPD	4	8	6	n/a	5	14	9	6
01-00430 MS %Rec	AN-102-CS-AR MS	n/m	n/m	n/m	n/m	n/m	n/m	n/m	n/m
01-00430 Post MS %Rec	Post Spike with 60/160	106	102	104	104	110	98	99	106
01-00430 BS %Rec	Hot Cell Blank Spike	104	108	93	101	110	94	99	n.p.
LCS % Rec	60/160 Spike Solution	107	105	105	106	101	104	102	107

n/a = not applicable since either or both the sample and duplicate results are not above the EQL

n/m = not measured; MS concentration either below EQL or sample concentration exceeds MS by 5x.

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

The oxalate results are considered qualitative. The LCS processed from the hot cell shows demonstrates NO recovery for oxalate; the reason is unknown.

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%.

The sample was prepared for ion chromatography anion analysis by leaching at approximately a 100:1 ratio with distilled de-ionized water. Once the leachates were prepared they had to be diluted an additional 100-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample at dilutions less than 100-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the table.

Q.C. Comments:

Duplicates: A sample and duplicate leach were performed in the hot cell and these duplicates analyzed. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20%.

Matrix Spike (010207 MS High, 60/160): A matrix spike was prepared from the sample by diluting the 010207 MS solution by 10-fold during the leaching process. It was anticipated that the leachates from the samples 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 were performed to assess matrix interferences.

Post Spike (010207 MS High, 60/160): Since the MS was lost due to dilution, a post spike was prepared by adding the same spiking solution as used for the MS to the sample, but after diluting the sample by 100-fold. The post spike recoveries meet the acceptance criteria of 75% to 125% recovery for all anions.

Laboratory Control Sample-LSC/BS (010207 MS High, 60/160): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared, process like the sample (i.e., through the hot cell preparative process), and analyzed like the sample. The LCS/BS demonstrated recoveries within the 80% to 120% acceptance criteria, except for the oxalate. The oxalate demonstrated 0% recovery (i.e., there was no peak detected for oxalate). Due to the failure or the LCS, the oxalate results are considered for information only.

System Blank/Processing Blanks: Twelve system blanks were analyzed throughout the analysis runs. No anions were detected in the system blanks above the estimate quantitation level. However, a slight chloride and nitrate contamination were detected in the hot cell blank process blank (01-00430PB). This blank level, if actually present in the samples contributes about 500 µg/ml to the chloride results and about 1,300 µg/ml to the nitrate results. The PB chloride contribution to sample exceeds 5% and therefore the chloride results do not meet the PB acceptance criteria. The PB nitrate concentration is well within the acceptance criteria.

Quality Control Calibration Verification Check Standards: Six mid-range verification standards were analyzed throughout the analysis runs. Except for two oxalate measurement, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The oxalate measurements produced recoveries of 112%.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: MW Thurston Date 7-18-01

Review/Approval: MJ Steele Date 7-19-01

Archive Information:

Files: ASR 6019 solids Urie.doc	ASR 6014 6019 6025 6031 6057.xls

Battelle, PNNL / RPG / Inorganic Analysis ... Cyanide Data Report

Project / WP#: 42040 / W57911
ASR#: 6019
Client: Mike Urie
Total Samples: 1

RPL #	Client ID
01-00430	AN-102-CS-AR

Procedure: PNL-ALO-287 Rev. 0, *Midi and Micro Distillation of Cyanide in Liquid and Solid Samples*
PNL-ALO-298 Rev. 0, *Total Cyanide Determination by Spectrophotometry (Manual or Automated) or Argentometric Titration*

M&TE Number:

WC36517	Lachet QuikChem Analyzer
360-06-01-036	Mettler AT400 Balance
360-06-01-031	Mettler AT400 Balance

Analyst: LMP Thomas

Analysis Date: 3/28/01

Analysis Files: Calibration - 01032801
Sample Trays - 01032802.RS, 01032803.RS, 01032804.RS

For Calibration and Maintenance Records, see
Chemical Measurement Center 98620 file:

MO Thu 7/3/01
Reviewed By

LMP Thomas
Concur

One sample, AN-102-CS-AR, was submitted for cyanide analysis. The sample was aliquoted in the hot cell, prepared by micro-distillation in a fume hood, and analyzed by automated spectrophotometry. The analysis of this sample included one duplicate, matrix spike and preparation blank. Quality control check standards relative to the sample preparation and instrument performance were also analyzed.

1. Analysis

Results from the analysis of the AN-102-CS-AR sample, are provided in the table below.

RPD success criteria: <20%				
RPL #	**Sample ID**	**Det Limit mg/kg**	**Measured mg/kg**	**RPD %**
01-00430	AN-102-CS-AR	1.1	33.1	
01-00430	AN-102-CS-AR DUP	0.6	32.5	1.8
Average			32.8	

2. Quality Control

Duplicate (DUP). The RPD of the AN-102-CS-AR samples meet the QC success criteria.

Matrix Spike (MS). Typically, when the cyanide concentration in the sample is unknown, the samples are spiked such that the spiking level is in the mid-range of the calibration curve. For this sample, the spiking level is approximately an order of magnitude too low. The matrix spike does not meet the success criteria, most likely due to the low spiking level.

RPL #	**Sample ID**	**Det Limit mg/kg**	**MS success criteria: 75% to 125% recovery RPD success criteria: < 20%**			
			spike level mg/kg	**Sample mg/kg**	**MS/MSD mg/kg**	**Recovery %**
01-00430	AN-102-CS-AR MS	0.6	4.53	32.8	39.5	147

Preparation Blank (PB) and Laboratory Control Standard (LCS). The combination of all of the distillation reagents is used as the PB. The results of the PB are based on the average sample size of the sample aliquots. The PB meets the success criteria. The LCS-solid is not within ±10% of the certified value, but is within the 80.4 to 322 mg/kg vendor performance acceptance limits. In subsequent analysis of the LCS-solid it was found that the large mesh size of this material was the most likely cause of the wide variability seen in its cyanide recovery. An LCS liquid is prepared as an additional QC check and it meets the QC criteria.

Sample ID	PB success criteria: < EQL		LCS success criteria: vendor certification or 90% - 110% of expected value		
	Success Criteria mg/kg	Measured mg/kg	Expected mg/kg	Measured mg/kg	Recovery %
PB	< 3.7	< 3.7			
LCS-solid			201	94	47
LCS-liquid			0.12 (1)	0.12 (1)	98
(1) Units are in mg/L					

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 0.25 M NaOH, which is the matrix of the distillates, and are not distilled. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: <EQL		
Sample ID	Success Criteria mg/L (1)	Measured mg/L (1)
ICB	<0.010	<0.010
CCB 1	<0.010	<0.010
CCB 2	<0.010	<0.010
CCB 3	<0.010	<0.010
(1) Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 85% to 115% recovery Calibration Range: 0 - 0.30 mg/L				
Sample ID	ICV/CCV - low		ICV/CCV - high	
	Measured mg/L (1)	Recovery %	Measured mg/L (1)	Recovery %
Expected Value	0.030	n.a.	0.12	n.a.
ICV	0.029	97	0.12	98
CCV 1	0.032	104	0.13	104
CCV 2	0.032	105	0.13	105
CCV 3	0.032	104	0.13	105
n.a. = not applicable (1) Units are based on per liter of sample at the instrument.				

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.010 mg/L		
Expected mg/L (1)	Measured mg/L (1)	Recovery %
0.0047	0.0037	79%
(1) Units are based on per liter of sample at the instrument.		

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Project No. 42040

Internal Distribution
File/LB

Date June 27, 2001

To Mike Urie

From Chuck Soderquist C. Soderquist

Subject Ammonia Analysis of Samples AN-102-SUP As-Received and AN-102 CS As-Received

The attached report has the analytical results from ammonia measurement in the AN-102-SUP as-received and AN-102 CS as-received samples, measured by ion selective electrode. These are sample numbers 01-429 and 01-430, ASR 6019.

The samples were prepared in a hot cell by dissolving raw sample in water and adding dilute sulfuric acid as a preservative. Both samples were prepared in duplicate. A laboratory control sample (made from an ammonium chloride standard in water, since no tank waste standard exists), a matrix spike, and a blank were also prepared in cell.

The samples were analyzed for ammonia in an open laboratory hood by ion selective electrode (procedure RPG-CMC-226). The hot cell had diluted the samples about 25 fold, and the laboratory diluted the samples another 10 fold during analysis. Because the samples were acidified, the sample vials could be opened without loss of ammonia.

The samples had easily detectable ammonia. The hot cell blank had slightly detectable ammonia, but well below the linear range of the ammonia probe and below the reported EQL. The sample duplicates and the lab replicate agree well. The laboratory control sample gave a recovery of 120%, which is higher than expected, but within project QC limits. The two matrix spikes had too little ammonia spike for the ammonia already in the samples (6% of the sample ammonia concentration), and consequently the matrix spike results had poor precision (62% and 66% recovery).

The method detection limit is the lower end of the linear range of the ammonia electrode, about 1.5 x 10^{-5} molar ammonia. Corrected for the sample dilution, this comes out about 0.07 mg ammonia per mL or gram of sample. The sample ammonia concentrations are above this concentration.

The ammonia probe slope and linearity were initially measured with ammonium chloride standards ranging from 5 x 10^{-7} to 1 x 10^{-1} molar. The probe is linear from about 1.5 x 10^{-5} molar up to at least 1 x 10^{-1} molar (and possibly higher). We ran routine probe slope checks using four standards from 1 x 10^{-6} through 1 x 10^{-3} molar, and calculated the slope using at least three points within the linear range. The slope was reproducible to better than 1% on the day the AN-102 samples were run.

All sample measurements were made by the method of standard additions, except when the

ammonia concentration was below the linear range of the probe. The probe intercept drops out of the calculation with this method, improving the analytical precision. (The intercept is normally less reproducible than the slope.)

The initial calibration verification gave a recovery of 102%, and the continuing calibration verifications gave recoveries of 93% and 92%. No instrument blanks had detectable ammonia.

Review:
J R Freeman
6-28-01

Battelle Pacific Northwest Laboratory 01-0429 ammonia
Radiochemical Processing Group-325 Building
Chemical Measurement Center 6/27/2001

Client : Mike Urie

Cognizant Scientist: C. Soderquist Date : 6-28-01

Concur : L R Greenwood Date : 6-28-01

Procedure: **RPG-CMC-226**
Ammonia Analysis by Ion Selective Electrode

Sample	Lab ID		Ammonia Concentration ± 1s		EQL
Process Blank	01-429 PB		3E-3 mg/mL		0.07
AN-102-SUP-AR	01-429		1.53E-1 mg/mL	± 3%	0.07
	01-429 Dup		1.50E-1 mg/mL	± 3%	0.07
		RPD	2%		
AN-102-CS-AR	01-430		8.70E-2 mg/g	± 3%	0.07
	01-430 Rep		8.81E-2 mg/g	± 3%	0.07
	01-430 Dup		9.13E-2 mg/g	± 3%	0.07
		RSD	3%		
	LCS		120% recovery		
	01-429 MS		62% recovery	Matrix spikes were too small compared to the sample ammonia concentration. Spike recoveries are within expected uncertainty.	
	01-429 MSD		66% recovery		

Dilution in hot cell was about 25x
Further dilution in laboratory was about 10x

BLANK

Battelle, PNNL / RPG / Inorganic Analysis ... Mercury Data Report

Project / WP#: 42365 / K88408
ASR#: 6019
Client: Mike Urie
Total Samples: 2

RPL #	Client ID
00-00429	AN-102-SUP-AR
00-00430	AN-102-CS-AR

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number: WD30853 CETAC, Mercury Analyzer, Model M-6000A
360-06-01-029 Mettler AT400 Balance

Digestion Date: 8/1/01
Analysis Date: 8/1/01

Analysis File: 01080101.DB

Analyst: LMP Thomas

For Calibration and Maintenance Records, see Chemical Measurement Center 98620 RIDS

9-07-01
Reviewed By / Date

Two samples, As Received AN-102 supernatant and wet centrifuged solids, were submitted for mercury analysis. The samples were aliquoted in the hot cell, prepared by digestion in a fume hood, and analyzed by cold vapor atomic absorption spectroscopy (CVAA). The analyses of these samples included one duplicate and matrix spike for each sample type. Quality control check standards relative to the sample preparation and instrument performance were also analyzed.

1. Analysis

Results from the analysis of the As Received AN-102 supernatant are provided in the table below. The concentration is reported in µg of mercury per liter of supernatant.

RPD success criteria: < 20%				
RPL #	**Sample ID**	**Det Limit µg/L**	**Measured µg/L**	**RPD %**
01-00429	AN-102-SUP-AR	0.142	< 0.142	
01-00429	AN-102-SUP-AR DUP	0.140	< 0.140	
Average			< 0.141	
Preparation DF (v/v)			52.1	
Analysis DF (v/v)			1	
DF - dilution factor				

Results from the analysis of the As Received AN-102 wet centrifuged solids are provided in the table below. The concentration is reported in µg of mercury per gram of wet centrifuged solids solids.

RPD success criteria: < 20%				
RPL #	**Sample ID**	**Det Limit µg/g**	**Measured µg/g**	**RPD %**
01-00430	AN-102-CS-AR	6.4E-04	1.92E-.03	
01-00430	AN-102-CS-AR DUP	7.2E-04	1.80E-03	6.8
Average			1.86E-03	
Preparation DF (mLs/g)			88.2	
Analysis DF (v/v)			1	
DF - dilution factor				

2. Quality Control

Duplicate (DUP). Since the concentration of mercury in the supernatant was below the detection limit, no RPD was calculated. The RPD of the centrifuged solids meets the success criteria.

Matrix Spike (MS). The matrix spike results for both supernatant and centrifuged solids do not meet the success criteria. The cause is unknown.

MS success criteria: 75% to 125% of expected value					
RPL ID	**Sample ID**	**Spike**	**Sample**	**Measured**	**Recovery**
00-00429	AN-102-SUP-AR MS	50.3 µg/L	< 0.0695 µg/L	16.5 µg/L	33%
00-00430	AN-102-CS-AR MS	0.0994 µg/g	0.0019 µg/g	0.0219 µg/g	20%

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PB and LCS analysis are presented in the table below. The result of the PB is based on the average sample size. The PB and LCS meet the success criteria.

Sample ID	**PB success criteria: < EQL**		**LCS success criteria: 90% - 110% of expected value**		
	Success Criteria	**Measured**	**Expected**	**Measured**	**Recovery**
AN-102-SUP-AR	< 2.6 µg/L	0.14 µg/L	1601 µg/L	1531 µg/L	96%
AN-102-CS-AR	< 0.0044 µg/g	< 0.0007 µg/g	1.40 µg/g	1.35 µg/g	96%

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: < EQL		
Sample ID	**Criteria µg/L [1]**	**Measured µg/L [1]**
ICB	<0.05	<0.05
CCB 1	<0.05	<0.05
CCB 2	<0.05	<0.05
CCB 3	<0.05	<0.05
CCB 4	<0.05	<0.05
CCB 5	<0.05	<0.05
[1] Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 90% to 110% recovery Calibration Range: 0 - 5 µg/L		
Sample ID	**Measured µg/L[1]**	**Recovery %**
Expected	2.00	
ICV	2.01	101
CCV 1	2.07	104
CCV 2	2.07	103
CCV-3	2.07	104
CCV 4	2.09	104
CCV-5	2.04	102
[1] Units are based on per liter of sample at the instrument.		

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery **Lowest calibration std: 0.05 µg/L**		
Expected µg/L[(1)]	**Measured µg/L[(1)]**	**Recovery %**
0.050	0.039	78
[(1)] Units are based on per liter of sample at the instrument.		

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on the manufacturer's recommendations for non-complex, aqueous matrices. The estimated quantitation limit (EQL) is defined as the lowest calibration standard. Actual detection limits or quantitation limits for specific matrices may be determined, if requested.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

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Date: 03/20/01

Subject: Hydroxide Analyses for: AN-102 composite
ASR: 6019.01
ASR: 6025

To: Mike Urie

From: Bob Swoboda 3/22/01

As recieved composites from tank **AN-102 Supernatant (01-00429)** and from **AN-102 Diluted Feed Supernatant (01-00441)** were analyzed in duplicate or triplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. The duplicate results on 01-00429 gave an average OH molarity of 0.25, which equals 4.23E+3 ug/ml, with a standard deviation of 25%. The triplicate results on 01-00441 gave an average OH molarity of 0.28, which equals 4.56E+3 ug/ml, with a relative standard deviation of 9%. The RSD value for sample 01-00429 is higher than the QC acceptance criteria of 15%; however, the hydroxide level is rather low in these samples at only 6% of the MRQ value. The standard recovery averaged 98% and a sample spike recovered at 97%, thus satisfying the QC acceptance criteria. No hydroxide was detected in the cell blank or reagent blank. Second and third inflection points, which are frequently associated with carbonate and bicarbonate, were detected in sample 01-00429 at 1.92 moles with a standard deviation of 3% and 1.20 moles with a standard deviation of 10%. For sample 01-00441, these points were at 0.76 moles, with a standard deviation of 14%, and at 0.56 moles, with a standard deviation of 8%. The titration curves are included with the report.

Reviewed: L.A. Greenwood 3/22/01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6019.01

WP# k88426

Hydroxide and Alkalinity Determination
Procedure: **PNL-ALO-228** Equip # **WB76843**

Analyst: [signature] 3/22/01

Reviewer: [signature] 3-22-01

Report Summary

RPG #	Client ID		Concentration, moles First Point	Second Point	Third Point
01-0429-CB	Cell Blank		0	0	0
01-0429	AN-102-SUP-AR		0.28	1.89	1.26
01-0429	AN-102-SUP-AR	Rep	0.22	1.94	1.13
		RPD	25%	3%	10%
Average hydroxide concentration in ug/ml			4.23E+3		
01-0441	AN-102-SUP-DF		0.25	0.81	0.54
01-0441	AN-102-SUP-DF	Rep	0.30	0.70	0.58
01-0441	AN-102-SUP-DF	Trip.	0.26	0.76	0.55
		RSD	9%	7%	4%
Average hydroxide concentration in ug/ml			4.56E+3		
Reagent Blank			0		
Standard 1			98%		
Standard 2			97%		
MS 01-0441	Matrix spike		97%		

Note: Results are presented for the first, second, and third inflection points on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second and third points generally represent the carbonate and bicarbonate concentrations.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client:	M. Urie	**Charge Code/Project:**	W57911 / 42365
RPL Numbers:	01-0429/0430 & 01-0441	**ASR Number:**	6019 and 6025
Analyst:	MJ Steele	**Analysis Date:**	July 24, 2001

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

AN-102 As-Received and AN-102/C-104 Diluted Feed Supernatant Results

RPL Number	Sample ID	TIC MDL µg C/mL	TIC µg C/mL	TIC RPD (%)	TOC MDL µg C/mL	TOC µg C/mL	TOC RPD (%)	TC µg C/mL	TC RPD (%)
01-0429	AN102-SUP-AR	33	10,900		87	29,400		40,300	
01-0429 Dup	AN102-SUP-AR	65	11,000	1%	170	29,100	1%	40,000	1%
01-0429 MS %Rec	AN102-SUP-AR		101%			92%		95%	
01-0441	AN102-SUP-DF	33	6,000		87	9,300		15,300	
01-0441 Dup	AN102-SUP-DF	33	6,000	0%	87	9,700	4%	15,700	3%
01-0441 MS %Rec	AN102-SUP-DF		99%			98%		98%	
BS/LCS %Rec	Blank Spike		100.3%			104.4%			

AN-102 As-Received Wet Centrifuged Solids Results

RPL Number	Sample ID	TIC MDL µg C/g	TIC µg C/g	TIC RPD (%)	TOC MDL µg C/g	TOC µg C/g	TOC RPD (%)	TC µg C/g	TC RPD (%)
01-0430	102-AN-CS-AR	66	17,100		180	25,900		43,000	
01-0430 Dup	102-AN-CS-AR	76	17,600	3%	200	25,300	2%	42,800	0%
01-0430 MS %Rec	102-AN-CS-AR		44%			160%		95%	
01-0430 MSD %Rec	102-AN-CS-AR		75%			119%		94%	
BS/LCS %Rec	Blank Spike		100%			101%			

The TOC/TIC analyses of the samples submitted under ASRs 6019 and 6025 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The tables above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

TIC/TOC analysis was only performed on the AN-102 CS AR, since there was insufficient AN-102 CS DF available for TIC/TOC analysis. Per the client's request the AN-102 CS DF TIC/TOC analysis was cancelled.

Q.C. Comments:

The standards for TIC and TOC supernatant analysis are liquid carbon standards from Ricca Chemical and VWR, respectively. The lot numbers and expiration dates are included on the raw data benchsheets.

For TIC and TOC solids analysis pure chemical compounds are used as the calibration, as well as matrix spiking, standards. The TIC analysis uses calcium carbonate and the TOC uses α-Glucose (JT Baker and Aldrich lot numbers are provided on the raw data benchsheets).

The QC for the methods involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes.

Supernatant Analysis:

Laboratory Control Sample/Blank Spike: The LCS/BS was within acceptance criteria of 80% to 120% required by the client's Test Specification.

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

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is acceptable for the samples. All the TIC and TOC RPDs meet the acceptance criteria of <15% established by the client's Test Specification.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes (for both AN-102 SUP AR and AN-102 SUP DF) demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery.

Solids Analysis:

Laboratory Control Sample/Blank Spike: The LCS/BS was within acceptance criteria of 80% to 110% required by the client's Test Specification.

Calibration Blanks: The three calibration blanks run at the beginning, middle, and end of the analysis run averaged 11 µgC TIC and 69 µgC TOC. These calibration/system blanks are considered acceptable for the concentration of TIC and TOC measured in the samples. The standard deviation for the TIC and TOC blanks are within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: The TIC and TOC RPDs meet the acceptance criteria of <15% established by the client's Test Specification.

Matrix Spike: The matrix spike (MS) recovery was very low for TIC and very high for the TOC spiked sample. A matrix spike duplicate (MSD) recovered within the acceptance criteria of 75% to 125% recovery; albeit the TIC recovery was still lower than normal and the TOC higher than normal. The total carbon recovery for both the MS and MSD is calculated at about 95%, suggesting that the quantity of the sulfuric acid added to the sample for analysis of TIC was insufficient to fully

acidify the sample. Based on the MS and MSD recoveries, there is a possibility that the reported TIC for the solids is bias low and the TOC is bias high.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data. The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: _____ **Date** 8-21-01

Review/Approval by: _____ **Date** 8-23-01

Archive Information:

Files: ASR 6019 6025 Urie P.doc	ASR 6019L&S 6025L 6104L 6015L.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie
Project :	42040
Work Pkg:	CMC
Analyzed:	July 24, 2001
ASR:	6019.01, 6025

Analyzer M&TE:	WC01713 -- 701	
Balance M&TE:	360-06-01-023	
TOC STD: VWR CMS#161356	0.10% Carbon	<<[G]
TIC STD: VWR CMS# 161355	0.10% Carbon	<<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch) 7-24-01	13.0	68.0
	Calibration blank (start of batch)7-24-01	14.0	53.0
	Calibration blank	15.0	80.0
	Calibration blank	23.0	81.0
	Calibration blank (end of batch) 7-24-01	40.0	87.0

TIC	TOC	
21.0	73.8	<<< Blank Average (ug C)
11.3	13.5	<<< Blank Std Dev (ug C)
2.16	*5.8*	*<<< Pooled Std Dev (ug C)*
6.5	*17.3*	*<<< Method Det. Limit (ug C)*

Is Blank Std Dev < Method Det Limit?	
TIC	No
TOC	Yes

		Total Inorganic Carbon (TIC)					Total Organic Carbon (TOC)				
		[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec		[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch) 7-24-01	991	21	1.0000	97.0		1036	74	1.0000	96.2	
	Calibration Standard (start of batch) 7-24-01	995	21	1.0000	97.4		1035	74	1.0000	96.1	
	Calibration Standard CCV	1015	21	1.0000	99.4		1100	74	1.0000	102.6	
	Calibration Standard	999	21	1.0000	97.8		1032	74	1.0000	95.8	
	Calibration blank (end of batch) 7-24-01	1035	21	1.0000	101.4		1128	74	1.0000	105.4	
		[L] Average TIC % Rec >>>>			98.6	<<[L]	[P] Average TOC % Rec >>>>			99.2	<<[P]
QC	Blank Spike/LCS	1024	21	1.0000	100.3		1118	74	1.0000	104.4	

Formulas:	Standard TIC % Recovery = ((A-B)/((C/100)*D))*10^{-6}*100	Matrix Spike Recoveries:
	Standard TOC % Recovery = ((E-F)/((G/100)*H))*10^{-6}*100	TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U
	Sample TIC (ug C/ml or ug C/g) = (I-J)/(K*L/100)	TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U
	Sample TOC (ug C/ml or ug C/g)= (M-N)/(O*P/100)	TC % Recovery = (((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/$U^{TIC+TOC}$
Comments:	Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.	
	The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.	
	If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".	
	TIC and TOC are measured; TC is the sum of the TIC and TOC results.	

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Urie
Project :	42040
Work Pkg:	CMC
Analyzed:	July 24, 2001
ASR:	6019.01, 6025

Analyzer M&TE:	WC01713 -- 701	
Balance M&TE:	360-06-01-023	
TOC STD: VWR CMS#161356	0.10% Carbon	<<[G]
TIC STD: VWR CMS# 161355	0.10% Carbon	<<[C]

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

		[I] Raw	[J]	[K] Sam	TIC	TIC	[M] Raw	[N]	[O] Sam	TOC	TOC	TC	TC
ACL Number	Client Sample ID (Liquids)	TIC (ug C)	Blk (ug C)	Vol (ml)	(ug C/ml)	RPD (%)	TOC (ug C)	Blk (ug C)	Vol (ml)	(ug C/ml)	RPD (%)	(ug C/ml)	RPD (%)
01-00429	An102-AR	2175	21	0.20	10,923		5905	74	0.20	29,379		40,302	
01-00429 Dup	An102-AR	1102	21	0.10	10,963	0	2960	74	0.10	29,083	1	40,047	1
01-00429 MS	An102-AR	1060	21	0.05	see below		2430	74	0.05	see below		see below	
01-00441	An102-DF	1197	21	0.20	5,963		1925	74	0.20	9,327		15,290	
01-00441 Dup	An102-DF	1212	21	0.20	6,040	1	2000	74	0.20	9,705	4	15,744	3
01-00441 MS	An102-DF	1590	21	0.10	see below		2012	74	0.10	see below		see below	
	(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K])												

Matrix Spike Results

		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS	
ACL Number	Client Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol (ml)	(ug C)	wt (g)	(ug C)	% Recovery	
01-00429 MS	TIC Recovery	1060	21	10963	0.05	548	0.5000	500	101.1	TIC
	TOC Recovery	2430	74	29083	0.05	1454	1.0000	1000	92.0	TOC
	Total Carbon Recovery (TIC + TOC)							1500	95.0	TC
01-00441 MS	TIC Recovery	1590	21	6040	0.10	604	1.0000	1000	98.7	TIC
	TOC Recovery	2012	74	9705	0.10	970	1.0000	1000	98.3	TOC
	Total Carbon Recovery (TIC + TOC)							2000	98.5	TC

Reviewer/date: D L Baldwin 8-23-01



Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client: M. Urie
RPL Numbers: 01-0429/0430 & 01-0441
Analyst: MJ Steele

Charge Code/Project: W57911 / 42365
ASR Number: 6019 and 6025
Analysis Date: 7/24, 8/01, 8/22, & 8/23 2001

Procedure: PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"
M&TE: Carbon System (WD13071); Balance (360-06-01-023).

AN-102 As-Received and AN-102/C-104 Diluted Feed Supernatant Results

RPL Number	Sample ID	TOC MDL µgC/mL	TOC µgC/mL	TOC RPD %	TC MDL µgC/mL	TC µgC/mL	TC RPD %	TIC MDL (b) µgC/mL	TIC (c) µgC/mL
01-00429	AN102-SUP AR	140	18,200		140	41,400		140	23,200
01-00429 Dup	AN102-SUP AR	60	17,900	2%	120	40,800	1%	120	22,900
01-00429 MS	AN102-SUP AR		(a)			114%			
01-00441	AN102-SUP DF	140	15,800		140	16,000		140	200
01-00441 Dup	AN102-SUP DF	60	15,700	1%	60	16,100	1%	60	400
01-00441 MS	AN102-SUP DF		85%			95%			
BS/LCS	Blank Spike		97%			99%			

(a) 01-00441 MS is the analysis batch MS QC sample for the TOC determination
(b) TIC MDL set to TC MDL
(c) TIC results obtained by difference (TC – TOC); analysis performed on two independent sample aliquots

AN-102 As-Received Wet Centrifuged Solids Results

RPL Number	Sample ID	TOC MDL µgC/g	TOC µgC/g	TOC RPD %	TC MDL µgC/g	TC µgC/g	TC RPD %	TIC MDL (b) µgC/g	TIC (c) µgC/g
01-00430	AN102-CS AR	300	17,400		560	35,900		560	18,500
01-00430 Dup	AN102-CS AR	390	21,900	23%	200	36,000	0%	200	14,100
01-00430 MS	AN102-CS AR		100% (a)			82%			
BS/LCS	Blank Spike		99%			104%			

(a) Sample 01-01017MS is the analysis batch MS QC sample for the TOC Determination.
(b) TIC MDL set to TC MDL
(c) TIC results obtained by difference (TC – TOC); analysis performed on two independent sample aliquots

The TOC/TIC analyses of the samples submitted under ASRs 6019 and 6025 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 750 °C for 30 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 30 minutes. The total inorganic carbon is obtained by difference.

The tables above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

For the centrifuged solids, the TOC and TC analysis were only performed on the AN-102 CS AR, since there was insufficient AN-102 CS DF available for analysis. Per the client's request the AN-102 CS DF TOC/TC analysis was cancelled.

Q.C. Comments:

The calibration and QC standards for TC and TOC analysis are liquid or solid carbon standards or pure chemicals from Ricca, Aldrich, Sigma, Mallinckrodt and VWR. The identification of the standards and their Chemical Management System (CMS) numbers and expiration dates are included on the raw data benchsheets.

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

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

For each days analysis run, the QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes.

Blank Spike/Laboratory Control Sample: The BS/LCS was within acceptance criteria of 80% to 120% required by the client's Test Specification for both the TC and TOC analysis of the supernatant and solids.

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

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes (for AN-102 SUP AR, AN-102 SUP DF, and AN-102 CS AR) demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery. Matrix spikes were not prepared for every AN-102 sample for both the TC and TOC analyses; however, when a matrix spike was not prepared from the AN-102 samples, a matrix spike from other samples in the batch was analyzed per the QA plan requirements.

Furnace Results Compared to Hot Persulfate Results

		TIC		TOC		TC	
		Persul.	Furn. (a)	Persul.	Furn.	Persul. (b)	Furn.
RPL Number	Sample ID	µgC/mL	µgC/mL	µgC/mL	µgC/mL	µgC/mL	µgC/mL
01-0429	AN102-SUP-AR	10,900	23,200	29,400	18,200	40,300	41,400
01-0429 Dup	AN102-SUP-AR	11,000	22,900	29,100	17,900	40,000	40,800
01-0441	AN102-SUP-DF	6,000	200	9,300	15,800	15,300	16,000
01-0441 Dup	AN102-SUP-DF	6,000	400	9,700	15,700	15,700	16,100
		µgC/g	µgC/g	µgC/g	µgC/g	µgC/g	µgC/g
01-0430	AN-102-CS-AR	17,100	18,500	25,900	17,400	43,000	35,900
01-0430 Dup	AN-102-CS-AR	17,600	14,100	25,300	21,900	42,800	36,000

Persul. = Results from hot persulfate method

Furn. = Results from furnace combustion method – 750 °C for TOC and 1000 °C for TC

(a) Results are obtained by difference (TC – TOC); analysis performed on two independent sample aliquots

(b) Results are obtained by summation (TIC + TOC); analyses performed on same sample aliquot

The two methodsappear to produce comparable results for TC for the AN-102 supernatants; however, there is about a 15% difference between the TC results for the AN-102 centrifuged solids, with the furnace producing the lower results. However, there are significant differences between the TIC and TOC results reported by each method. Based on the AN-102 as-received results, the persulfate results appear to have a higher likelihood of being accurate; that is, it is entirely plausible that there are organic compounds in both the solids and the supernatant that are difficult to oxidize at 750 °C, leading to low TOC and high TIC for the furnace method. For the AN-102 diluted with washes and leaches from C-104, the reason for the discrepancy between the persulfate method and furnace method is unknown, but it appears that the inorganic carbon, perhaps in the form of easily oxidized metal carbonate, is being combusted at 750 °C with the furnace method.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- For both the TC and TOC, the analysis Method Detection Limit (MDL) is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's *t* values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.

Report Prepared by: _____ **Date** 9/14/01

Review/Approval by: _____ **Date** 9/14/01

Raw Data Calculation/Archive Information: File: ASR 6019L&S 6025L 6104L 6015L.xls

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie	Temp.	Balance M&TE: 360-06-01-023
Project :	42040	1000 Degree C	TOC Cal Std: VWR CMS#161356 0.10% Carbon <<[G]
Work Pkg:	CMC	Run time	TIC Cal Std: VWR CMS#161355 0.10% Carbon <<[G]
Analyzed:	July 24, 2001 MJ Steele 9/12/01	30 Minutes	LCS/BS/MS TOC Std: RICCA CMS#161730 0.10% Carbon <<[C]
ASR:	6019.01, 6025, 6031		LCS/BS/MS TIC Std: RICCA CMS#161734 0.10% Carbon <<[C]

		Raw TC (ug C)	
Blanks:	Calibration blank (start of batch)	4.6	
	Calibration blank (start of batch)	5.2	
	Calibration blank	3.7	
	Calibration blank (end of batch)	4.2	

4.4	<<< Blank Average (ug C)
0.6	<<< Blank Std Dev (ug C)
4	<<< # of Blanks analyzed
2.4	<<< Method Det. Limit (ug C) [M]

		Total Carbon (TC)				
		[A] Raw TC (ug)	[B] Blk (ug)	[D] Std Vol (ml)	TIC % Rec	
Standards:	Calibration Standard (start of batch)	447.5	4	0.50	88.6	0.25 ml TIC and TOC Cal Std
	Calibration Standard (start of batch)	467.3	4	0.50	92.6	0.25 ml TIC and TOC Cal Std
	Calibration Standard	237.4	4	0.25	93.2	0.25 ml TOC Cal Std
	Calibration Standard (end of batch)	468.3	4	0.50	92.8	0.25 ml TIC and TOC Cal Std
QC	Blank Spike/LCS	457.2	4	0.50	98.7	0.25 ml TIC and TOC LCS/BS/MS Std

91.8 <<< [L] Average TC % Rec

Formulas:	Standard TC % Recovery = ((A-B)/((G/100)*D))*E^{-6}*100
	QC % Recovery = (((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)
	Sample TC (ug C/ml or ug C/g) = (I-J)/(K*L/100)
	MS TC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U

Comments:	Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.
	The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).
	For any TC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].
	If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie	Temp.	Balance M&TE: 360-06-01-023
Project :	42040	1000 Degree C	TOC Cal Std: VWR CMS#161356 0.10% Carbon <<[G]
Work Pkg:	CMC	Run time	TIC Cal Std: VWR CMS#161355 0.10% Carbon <<[G]
Analyzed:	July 24, 2001	30 Minutes	LCS/BS/MS TOC Std: RICCA CMS#161730 0.10% Carbon <<[C]
ASR:	6019.01, 6025, 6031		LCS/BS/MS TIC Std: RICCA CMS#161734 0.10% Carbon <<[C]

Note: Sample weights are on "as received" basis; i.e., wet weight

Sample Results		[I] Raw	[J]	[K] Sam	TC	TC	
RPL Number	Sample ID	TC (ug C)	Blk (ug C)	Vol. (ml)	(ug C/ml)	RPD (%)	
01-00429	AN102-SUP AR	1903	4	0.050	41,368		
01-00429 Dup	AN102-SUP AR	940	4	0.025	40,771	1	
01-00429 MS	AN102-SUP AR	1465	4	0.025	see below		0.25 ml TOC and TIC Std
01-00441	AN102-SUP DF	738	4	0.050	15,984		
01-00441 Dup	AN102-SUP DF	742	4	0.050	16,071	1	
01-00441 MS	AN102-SUP DF	960	4	0.050	see below		0.25 ml TOC Std
01-00520PB	Hot Cell Blank	15	4	0.500	23		
01-00520PB Du	Hot Cell Blank	10	4	0.500	11	n/a	
01-00520A	AP101-DF-A	1353	4	0.200	7,346		
01-00520A Dup	AP101-DF-A	1355	4	0.200	7,357	0	
01-00520B	AP101-DF-B	689	4	0.100	7,458		
01-00520B Dup	AP101-DF-B	700	4	0.100	7,578	2	
01-00520B MS	AP101-DF-B	1165	4	0.100	see below		0.25 ml TOC and TIC Std

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol. (ml)	(ug C)	wt (g)	(ug C)	% Recovery
01-00429 MS	Total Carbon Recovery (TC)	1465	4	40771	0.03	1019	0.5000	500	114.4
01-00441 MS	Total Carbon Recovery (TC)	960	4	16071	0.05	804	0.2500	250	95.0
01-00520B MS	Total Carbon Recovery (TC)	1165	4	7578	0.10	758	0.5000	500	101.3

Reviewer/date: MW Urie 8-12-02

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FINAL Report
Organic Acids-AN-102 Supernate and Diluted Feed
GM Mong, R Niederer

Introduction

The desired analytes for WTP samples AN-102 supernate and diluted feed included a component (gluconate) which is not listed in standard separations for organic acids by ion chromatography (Dionex and Alltech catalog references). Gluconate is the first oxidation step in transformation of glucose α-carbon to a carboxylic acid from an aldehyde/hemiacetal functionality. This represents our first attempt to analyze tank waste materials for gluconate by ion exchange chromatography

Several separation mechanisms were attempted to define a separation of gluconate from the other desired analytes (glycolate, formate, oxalate, citrate). A useful separation was discovered for acetate from glycolate using the Dionex AS-14 column and 2mM sodium borate as the eluent. This affords a near separation of glycolate from acetate; unfortunately gluconate does not elute from this system. The reasons for gluconate's failure to elute from this column was not pursued. The best separation scheme for gluconate inclusive of the other analytes was to employ our established AS-11 (Dionex) separation using 0.08mM NaOH as the initial eluent. This affords gluconate as a peak just beyond the unretained volume of the column at a retention time that overlaps with fluoride. No other separations (AS-14, AS-9, AS-4) appeared to generate fruitful separations for gluconate from the other monovalent analytes (glycolate, formate). Due to the low ionic strength of the initial eluent, any samples require extensive dilution to prevent retention time variation, and long equilibrations (20 minutes) were a necessity.

Once these limitations were defined, a workable separation of gluconate, glycolate, formate, oxalate, and citrate was accomplished from the other tank ions (chiefly nitrite, nitrate, and sulfate). Surprisingly, a peak with the retention characteristics of gluconate was observed in the 441 samples. A test to distinguish this peak from fluoride was done by adding calcium oxide to sample 441S. The peak's observed area upon dilution was not diminished; therefore, this material does not have the characteristics of fluoride, and matches the retention time of gluconate.

Analytical efforts were undertaken to extend the retention time for gluconate and glycolate as far from the unretained volume as possible. The peak found in the samples very near the unretained volume had the same retention time as gluconate in the standards.

Instrumentation and sample preparation:

Dionex system (WD24293) was used with a sodium hydroxide gradient elution, AS-11 anion exchange column with AG-11 guard column. The elution ramp was 0.08mM

NaOH for 7.5 min, ramping to 0.3mM NaOH at 9.4 min, 5mM NaOH at 10.4 min, 28mM NaOH at 20 minutes, followed by rapid equilibration to 0.08 mM NaOH which was maintained to 40 min.

Samples obtained from the hot-cell represent an approximate 20:1 dilution upon receipt for analysis. The 429 samples were further diluted 1:1000 for analysis; the 441 samples were further diluted 1:500 for analysis. BS, MS and MSD samples, obtained as part of the sample batch, were prepared by spiking final dilutions with known amounts of a mixed standard of gluconate, glycolate, formate, oxalate, and citrate.

Results:

Results of analysis are summarized below. The calculations are corrected to the original sample concentration.

1. Analysis 01-429 AN-102-Sup and 01-441-AN-102-DF

Sample No.	Gluconate µg/mL	Glycolate µg/mL	Formate µg/mL	Oxalate µg/mL	Citrate µg/mL
429S	<MDL (<1000)	11,000	8000	510	4400
429D	<MDL (<1000)	10,000	8000	410	4400
441S	41,000	3200	2800	1800	1200
441D	40,000	3400	2800	1800	1200

Note: Gluconate values are represented from a material peak eluting near the void volume. This peak matches the retention time for gluconate, and does not respond chemically as fluoride (evidenced by addition of calcium oxide to the concentrated sample). Elution times near the void volume only indicate identity. An exact match of retention time near the void volume may not constitute firm identity of the material.

Method Detection Limits:

Estimated detection limits are based on the IDL multiplied by the approximate dilution factor subjected to each sample. This value represents the minimum detectability per sample prior to any handling in the hot cell. Use these values for any estimation of net detectability in the raw sample prior to dilutions in the hot cell or instrumental workup.

Gluconate µg/mL	Glycolate µg/mL	Formate µg/mL	Oxalate µg/mL	Citrate µg/mL
1000	100	100	200	200

2. Quality Control:

Matrix spikes were produced using the final dilution volumes; spikes were added which approximately doubled the size of the native material in each sample (determined by separate analysis of the MS and MSD samples). Spike added amounts are in **µg/mL**.The values reported DO NOT take into account any dilutions of sample materials and represent spikes on diluted samples.

Sample No.	Gluconate Spike added % recovery	Glycolate Spike added % recovery	Formate Spike added % recovery	Oxalate Spike added % recovery	Citrate Spike added % recovery
441MS	1.1 (121%)	0.49 (105%)	0.25 (123%)	0.33 (89%)	0.60 (112%)
441MSD	1.1 (107%)	0.49 (105%)	0.25 (109%)	0.33 (86%)	0.60 (116%)

Blank Sample Data: An analysis was done on sample 429 B. IDL values indicate a value estimated for a clean sample without dilution.

Sample No.	Gluconate	Glycolate	Formate	Oxalate	Citrate
429B	<IDL (1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.2 µg/mL)	<IDL (0.2 µg/mL)

Blank Spike Sample Data: An analysis was done on sample 429 BS, a small peak was observed for oxalate. The reported value is indicative of the amount present in the delivered sample for analysis. This sample was spiked with analytes at 2 times the LLS value and analyzed.

Sample No.	Gluconate (spk added, %)	Glycolate (spk added, %)	Formate (spk added, %)	Oxalate (spk added, %)	Citrate (spk added, %)
429BS initial analysis	<IDL (1 µg/mL)	<IDL (0.1 µg/mL)	<IDL (0.1 µg/mL)	0.3µg/mL	<IDL (0.2 µg/mL)
429BS, spiked	1.1µg/mL, 106%	0.49µg/mL, 100%	0.25µg/mL, 108%	0.33µg/mL, 78%	0.60µg/mL, 89%

ICB and CCB data: All collected data was less than IDL for all analytes

ICV, CCV data: Blank water spiked at 2 times the LLS value; recovery is percentage calculated from calibration curves. ICV was prepared from independent materials, with the exception of gluconate.

	Gluconate	Glycolate	Formate	Oxalate	Citrate
ICV1	102%	100%	97%	101%	96%
ICV2	106%	92%	102%	102%	99%
CCV1	110%	99%	104%	102%	101%
CCV2	106%	99%	104%	102%	101%

LCS data: sample prepared at 2 times the LLS level.

	Gluconate	Glycolate	Formate	Oxalate	Citrate
LCS	104%	103%	103%	100%	100%

LLS data:

	Gluconate 0.75µg/mL	Glycolate 0.33µg/mL	Formate 0.17µg/mL	Oxalate 0.18µg/mL	Citrate 0.4µg/mL
LLS	96%	102%	101%	98%	95%

Comments: Almost all of the quality control data meets the success criteria. Blank spike recovery for oxalate and MSD recovery for oxalate are out of the ±15% range. All other data are acceptable.

If Gluconate is a continued concern for analysis, a more rigorous search for an analytical method (ion chromatography or other means) should be undertaken to improve analytical confidence in its identity in tank materials.

Prepared by GM Mong Gary Mong 10/15/01

Reviewed by JA Campbell J A Campbell 10/17/01

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1.0 Polychlorinated Biphenyls (PCB) Analysis

1.1 Introduction

For the PCB analysis, the supernatants and centrifuged solids samples were prepared and extracted in the SAL by the procedure outlined in the Analytical Service Request 6019.01 and Test Plan TI-RPP-WTP-072: Analysis for PCBs and Pesticides. Following extraction, the resulting methylene chloride (supernatants) or methylene chloride/acetone (solids) residues were transferred from the SAL under Chain-of-Custody to the building 329 organic analysis laboratories. The residues were then exchanged into hexane and concentrated to 2 mL. Following residue cleanup, analysis was performed for PCBs by gas chromatography/electron capture detection (GC/ECD).

1.2 Sample Preparation

Approximately 100 mL of supernate sample was diluted with 120 mL of organic free water to reduce the supernatant density. Matrix spike samples were performed in duplicate on 50 mL sample portions diluted with 60 mL of organic free water. The same source of organic free water was used for the method blank and the laboratory control sample (LCS) matrix. All of these samples were prepared in the SAL. Each of the diluted samples was extracted with methylene chloride three times using a separatory funnel. The resulting three extracts were combined and each passed through a sodium sulfate drying column.

The extracts were transferred to the 329 organic analysis laboratories. They were then concentrated and exchanged into hexane and taken to a final volume of 2.0 mL. The extracts were split and 1.0 mL was refrigerated. The remaining 1.0 mL of the extract residue was subjected to sulfuric acid cleanup. The residues were agitated with concentrated sulfuric acid and the remaining hexane fraction was analyzed for PCBs.

For the solids extraction, approximately 2.5 grams of centrifuged solids were extracted with water. The liquid from this extraction was subjected to the process described above for the supernatant. 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. The solid matrix for the LCS and the blank consisted of granular sodium sulfate and both were also extracted in the SAL.

The extracts were transferred to the 329 organic analysis laboratories, concentrated and exchanged into hexane. They were brought to a final volume of 2.0 mL. The extracts were split and 1.0 mL was refrigerated. The remaining 1.0 mL of the extract residue was subjected to the sulfuric acid cleanup. The residues were then analyzed for PCBs.

1.3 Instrumentation

The instrumentation used for the analysis of pesticides and PCBs consisted of a gas chromatograph equipped with two electron capture detectors (ECD). The analytical instrumentation is identified in Table 1.1. Both of the detectors were operated at 320° C. Injections were made on-column onto a 10-m fused silica retention gap, which was split between two analytical columns: a) 0.32 mm x 30 m CLP I (0.50 µm phase, Restek Corp.) and b) 0.32 mm x 30 m CLP II (0.25 µm film thickness, Restek Corp.).

Table 1.1 PCB/Pesticides Analysis Instrumentation

System/Instrument	Manufacturer	Model Number	M&TE (1) Number
Gas Chromatograph	Agilent	6890A	WD 30911

(1) Measuring and Test Equipment

1.4 Analysis Results

PCB results are presented in Table 1.2. No PCBs were found in the supernate samples. The centrifuged solid sample AN-102-CS-AR duplicate exhibited patterns of Aroclors 1254 and 1260, although they were quantified at only two and three times above the MDL. These results were not duplicated in the AN-102-CS-AR sample as no PCBs were found above the MDLs in this sample.

Table 1.2 PCB Results

	Tank Material	AN-102 Supernatant				AN-102 Wet Centrifuged Solids			
	Sample ID	MDL	01-0429 Proc. Blk	01-0429 Sample	01-0429 Duplicate	MDL	01-0430 Proc. Blk	01-0430 Sample	01-0430 Duplicate
CAS #	*Units*	µg/L	µg/L	µg/L	µg/L	µg/Kg	µg/Kg	µg/Kg	µg/Kg
Polychlorinated Biphenyl's Analyte List									
12674-11-2 53469-21-9	Aroclor 1016/1242	0.200	U	U	U	8.00	U	U	U
11104-28-2	Aroclor 1221	0.200	U	U	U	8.00	U	U	U
11141-16-5	Aroclor 1232	0.200	U	U	U	8.00	U	U	U
12672-29-6	Aroclor 1248	0.200	U	U	U	8.00	U	U	U
11097-69-1	Aroclor 1254	0.200	U	U	U	8.00	U	U	17.0
11096-82-5	Aroclor 1260	0.200	U	U	U	8.00	U	U	29.5
	Total PCB*	1.20	U	U	U	48.0	U	U	79.4
			% Recov.	% Recov.	% Recov.		% Recov.	% Recov.	% Recov.
	TCX (surrogate)		66.4	89.3	78.3		50.4	34.9	61.8
	DCB (surrogate)		61.7	64.6	26.3		118.9	76.5	114.2

U = Not detected; results less than MDL

*Total PCB is determined by adding the MDL values and any detected PCB quantities

1.5 QC Evaluation

Surrogate Recoveries:
Surrogate compounds were spiked at approximately 0.4 ug/L in the supernatant samples. As shown in Table 1.2, the recoveries ranged from a high of 89.3% for TCX to a low of 26.3% for DCB. They were notably higher in the spike and spike duplicate samples (Table 1.3) ranging from 79.7 to 163.1%, indicating 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 16 ug/Kg in the centrifuged solid samples. 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 in the spiked solid samples indicating these may have been doubly spiked with the surrogates as well.

Several of the surrogate recoveries were lower than the initial target of 70-130% with the exception of the matrix spike sample mentioned previously. All of the results were verified by an analysis of the remaining 1.0 mL aliquot of sample residue which had been split during sample preparation. No further action was taken.

Spike Recoveries:
Samples of both the supernatant and solids were spiked in duplicate. The results are presented in Table 1.3. Because limited sample was available, the quantity of supernate sample used for the matrix spike was approximately half of that used for unspiked sample analysis. Laboratory control samples (LCS) consisted of PCB spiked blank water or drying agent (granulated sodium sulfate) for the solids extractions. The LCS results are also presented in Table 1.3.

Several of the matrix spike recoveries were lower than the initial target of 70-130%. All of the results were verified by an analysis of the remaining 1.0 mL aliquot of sample residue which had been split during sample preparation. No further action was taken.

Table 1.3 AN-102 – PCB Spike Recoveries

	Tank Material	AN-102 Supernatant			AN-102 Wet Centrifuged Solids		
	Sample ID	00-1360		LCS	00-1361		LCS
		MS	MSD		MS	MSD	
	Units	%Rec (µg/L)	%Rec (µg/L)	%Rec (µg/L)	%Rec (µg/Kg)	%Rec (µg/Kg)	%Rec (µg/Kg)
CAS #	*MDL*	0.4	0.4	0.2	8	8	10
12674-11-2	Aroclor 1016	69.2 (2.86)	50.4 (2.07)	85.2 (1.67)	85.0 (69.45)	54.0 (44.99)	57.2 (57.2)
11096-82-5	Aroclor 1260	73.5 (3.30)	58.8 (2.67)	89.1 (1.75)	80.4 (84.57)*	44.2 (55.66)*	53.2 (53.2)
		% Rec	% Rec	% Rec	% Rec	% Rec	% Rec
	TCX (surrogate)	73.5	79.7	85.9	104.2	64.8	40.5
	DCB (surrogate)	120.8	125.5	90.6	160.1	126.7	65.5

* Amount of Aroclor 1260 found in the sample was subtracted from the spike quantity. The quantity subtracted was an average of the MDL in the sample and the amount present in the sample duplicate.

PCB Results:

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 ICV and CCV demonstrated high recovery by approximately 5 percent. As specified by the Test Plan TP-RPP-WTP-030 and the associated quality assurance, the source of the material used for the ICV and CCV standards was different than for the initial calibration standards. The difference can be attributed to the use of a different source of standard Aroclor 1260 material. All of the other ICV and CCV criteria were met for the remaining 9 PCB congeners and the two surrogate compounds.

Patterns of Aroclors 1254 and 1260 were identified in the centrifuged solid samples. They were present above the MDL only in AN-102-CS-AR duplicate. The levels found were quite low (within three times the MDL) and the ratio of congeners which 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 for additional definitive confirmation was not performed but is recommended. However, the quantity of PCBs found is well below conventional electron impact mass spectrometer detection limits and below the levels of PCB confirmed by GC/MS in WTP-RPT-008 rev 1, *Organic Analysis of C-104 Tank Waste*.

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Eric Hoppe
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5-1-01
Date

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Organic Phosphate Analysis

Introduction

A derivatization gas chromatography (GC) method was developed to analyze bis-(2-ethylhexyl)phosphoric acid (D2EHP) under the auspices of the Organic Tanks Safety Program (Campbell 1996). Derivatization gas chromatography/mass spectrometry (GC/MS) and GC/flame ionization detection (FID) for the analysis of D2EHP has been performed only twice in the past. Samples associated with tank 241-C-106 were obtained during washing and centrifuging experiments and appeared to contain a waxy or oily substance. Initial direct GC analysis of methylene chloride extracts of the matrix did not reveal the reason for high total organic carbon (TOC) values associated with these samples; tributylphosphate (TBP) and a later eluting component identified as butyl bis-(2-ethylhexyl) phosphate were found. Treatment of an aliquot of the sample with ethereal hydrochloric acid followed by diazomethane revealed a large quantity of D2EHP to be present in the sample. Quantitation of these three components afforded a good match to the measured TOC values; these three components make up about 75% of the total mass of the sample (Mong and Campbell 1999). Samples from tank 241-C-103 were taken from a layer amounting to at least 16 m^3 floating on top of aqueous waste to resolve safety concerns. The organic liquid was found to consist primarily of paraffin hydrocarbons and TBP in an approximately 1:2 ratio. Direct analysis of the liquid revealed smaller quantities of dibutylbutyl phosphonate (DBBP) and butyl bis-(2-ethylhexyl) phosphate. Treatment of an acidified portion of this sample did not reveal the presence of D2EHP (Mong and Campbell 1999, Campbell 1996). The previously mentioned studies focused primarily on identification and semi-quantitation.

The current analysis of the tank waste samples AN-102 as-received and AN-102/C-104 blended feed represents the first attempt at using diphenylphosphate (DPP) as a surrogate standard. Diphenylphosphate was chosen as a surrogate compound because its structure and potential reactivity are similar to D2EHP.

The analysis of the AN-102 samples for D2EHP has been conducted on a best effort basis. The samples were analyzed according test plan TP-RPP-WTP-047, "Identification and Quantification of D2EHP in Tank Waste". D2EHP was not detected in any of the samples.

Instrumentation

The derivatized phosphate samples were analyzed using a GC 5890 Series II (Hewlett Packard) equipped with a flame ionization detector and a fused silica column (DB-5MS [J&W] 30 m X 0.25 mm X0.25 μm film thickness). The temperature program used was the following: 40° C for 1 min, 40° C-260°C at 8°C/min and 260° C for 7 min. The injector was at 265°C and the detector was at 260°C. A 1 μL splitless injection was performed for the analyses. The retention times and identities of DPP and D2EHP were confirmed using GC/MS.

Standard and Sample preparation

Standards were prepared for DPP (2.3 µg/mL to 116 µg/mL) and for D2EHP (2.6 µg/mL to 130 µg/mL) in methylene chloride and derivatized using diazomethane prepared as discussed below.. A five-point calibration curve was constructed for both DPP and D2EHP.

Each sample, including quality control samples, was prepared from 5-mL aliquots, which were placed in a beaker and weighed. A 5-ml aliquot of water was added, and the sample was transferred to a separatory funnel. The pH was adjusted to 2 (measured with pH paper) with 6 N nitric acid. Each sample was then extracted three times with 25 mL of methylene chloride. Each sample was then extracted with 25 mL of butanol. The methylene chloride and butanol extracts were stored separately. The methylene chloride extracts were concentrated to a volume of less than 1 mL. Diazomethane was prepared by stirring an ethereal slurry of N-methyl-N-nitrourea (Pfalz and Bauer Chemical Company) over an ice-cold 40% KOH solution. The yellow diazomethane/ether solution is simply decanted off for refrigerated storage. Diazomethane, though hazardous if handled incautiously, is a reagent for methylation.

Approximately 100 µL of the diazomethane/ether solution was added to each extract. Contact of an acid phosphate (e.g. D2EHP) with diazomethane solution results in instantaneous evolution of nitrogen gas; excess diazomethane is indicated by the characteristic yellow color of the solution. Additional diazomethane is added until the yellow color persists with slow evolution of bubbles. The reaction is left covered, in a hood, for an additional hour to ensure complete conversion of the acid phosphates to the methyl esters. The volume of the samples was then adjusted to 1 mL using nitrogen blow-down techniques and analyzed using GC/FID. (The highly acidic nature of alkyl phosphates (pKa ca. 1.0-1.5) and the ability of phosphoric acid to dehydrate alcohols makes phosphates intractable to esterification in the laboratory with acidic reagents, such as mineral acids or BF_3, in conjuction with alcohols.)

The laboratory control sample (LCS) extracted in the hot cell was inadvertently spilled during the concentration procedure. Another LCS was prepared outside the hot cell by adding D2EHP and DPP to 5 mL of Milli-Q water adjusted to a pH of 12 using 6M NaOH. The pH of the sample adjusted to 2 using 6N nitric acid. The pH was measured with pH paper. The sample was then extracted in a separatory funnel 3 times with 25 mL of methylene chloride with vigorous shaking. The methylene chloride extracts were combined and concentrated to approximately 1 mL with K-D apparatus. The sample was derivatized with diazomethane following the procedure above, and the final volume was brought to 1 mL prior to analysis. The sample was analyzed using GC/FID.

Analysis Results

The results of analyses for the samples and recoveries for DPP and D2EHP are listed below in Table 1. The estimated quantitation limit (EQL) for D2EHP in the sample, sample duplicate, and process blank are based on the concentration of the lowest

calibration standard converted to the appropriate volume of sample (ca 5 mL). D2EHP was not detected in any of the samples.

Table 1. Organic Phosphate Sample and QC Results

		DPP		D2EHP	
RPL Number	Sample ID	µg/mL	Recovery	µg/mL [1]	
01-00429	AN-102-SUP AR	31.9	144%	<0.54	
01-00429-D	AN-102-SUP AR Dup	39.4	187%	<0.51	
01-00441	AN-102-SUP DF	17.7	83%	<0.52	
01-00441-D	AN-102-SUP DF Dup	18.4	87%	<0.51	
01-00429-PB	Process Blank	0.62	3%	<0.51	
QC Samples	Sample ID	µg/mL	Recovery	µg/mL	Recovery
LCS	Lab Control Sample	1.04	5%	10.3	42%
01-00441-MS	AN-102-SUP DF Matrix Spike	15.4	73%	20.3	84%
01-00441-MSD	AN-102-SUP DF Matrix Spike Dup	19.4	93%	25.6	107%

[1] The 'less than results' for D2EHP are the EQL based on the concentration of the lowest standard used to generate the calibration curve adjusted to the sample volume and dilutions.

Neither the Test Specification nor the governing QA Plan provides QC acceptance criteria for the organic phosphate analysis. However, to assess method performance the results of the QC samples (i.e.,, LCS and matrix spikes) were compared to the QC acceptance criteria for anions by ion chromatography. Recoveries for both the DPP and D2EHP from water matrices (i.e., process blank and LCS) were very poor. The reason for the high DPP recoveries in the AN-102-SUP AR samples is not apparent, but it is possible that the DPP spike may have been added twice. The low DPP recoveries observed in the blank and LCS and low D2EHP recovery in the LCS can not be explained.

To indicate the variance in derivatization, three replicate samples (i.e. calibration verification check standards) containing D2EHP and DPP were derivatized and analyzed. The recoveries ranged from 62-121% for DPP and 73-84% for D2EHP. Each sample contained 58 µg/mL (DPP) and 65 µg/mL for D2EHP. The results are shown in Table 2.

Table 2. DPP and D2EHP Standard Mixture Results

	DPP		D2EHP	
Sample	µg/mL	Recovery	µg/mL	Recovery
1	36	62	47	73
2	57	98	53	82
3	70	121	54	84

Recommendations

The analysis of tank waste samples for D2EHP was performed on a best effort basis, since this technique has not been thoroughly validated and qualified. From this work the appears to hold promise for producing reliable results for D2EHP; however, further

methods development is required. The apparent inconsistency in recoveries must be addressed. One of the chromatographic problems encountered was tailing of the peaks which may have contributed to high recoveries with certain samples. As a result of peak tailing, manual integration of the peaks was required. The peak-tailing problem may be solved in additional analyses by simply adjusting the chromatographic parameters such as the column temperature program and injection port temperature or changing the column.

In addition, in order to make this technique reliable and robust, a systematic study must be undertaken to evaluate the extraction process and the derivatization process to determine the reason for low recoveries in the control sample. It may be that the choice of DPP as a surrogate compound was not a good one. Also, it would be advisable to add a known methylated compound to examine the extraction efficiency.

Since the analyses of these sample, additional work has been done to evaluate the poor recoveries demonstrated by the process blank and LCS. Sodium sulfate has been added to an aqueous sample to form a saturated solution, then spiked with known volumes of DPP and D2EHP. The recovery for DPP was 60% and for D2EHP was 91%. The increased recovery is most likely due to a salting out effect. In addition, stability of the derivatized standard over time has been examined. The responses (i.e., apparent concentration) tended to decrease.

A test plan is being written that proposes testing on various matrices (e.g., water, simulated tank waste, and tank waste) to identify the cause of the low recovery problems and to develop a more rugged, reliable, consistent technique for the analysis of D2EHP. Areas to be addressed include derivative stability, ionic strength, pH, and method of agitation. Once the method has been verified with a water matrix, the method will be extended to simulated wastes.

References

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Campbell JA. 1996. Organic Tanks Safety Program: Advanced Organic Analysis FY 1996 Progress Report. PNNL-11309, Pacific Northwest National Laboratory, Richland, Washington.

Archive Information

Sample data and calculations located in LRB 14059 pages 7-8 and 28-31.
Analysis data and supporting data and documentation located in ASR 6016/6025 for Project 42020 (or 43265).

Prepared By: J. A. Campbell Date: 5/2/02

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Derivatization GC/FID Analysis of Chelators and Degration Products Report

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Charge Code/Project: W57912
Sample Receipt Date:
Analysis Date: 8/15/01

Preparation Procedure: TPR-PNNL-WTP-049
Analysis Procedure: TP-RPP-WTP-048
M&TE: WD14807,WD 14805 –GC
WD 30902, WD 30903 GC/MSD

Final Results

See attached report

Summary of Deviations

Spiking solutions for the MS and MSD were performed with citric acid and EDTA only. There were no standards available for nitrosoiminodiacetic acid (NIDA) and ED3A. The concentrations were calculated on the basis of the response compared to EDTA. In addition, the concentration of HEDTA was based on the response of EDTA.

Quality Control Sample Comments

See attached report.

General Comments

See attached report.

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Review/Approval by: R.B. Lucke R.B. Lucke **Date** 6-28-02

Archive Information

Chelator and Degradtion Product Analysis Using Derivatization GC/FID of AN-102 Supernate and Diluted Feed

J.A. Campbell

Introduction

Chelators such as ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl) ethylenediaminetriacetic acid (HEDTA), nitrilotriacetic acid (NTA), iminodiacetic acid (IDA), succinic acid (SA), ethylenediaminetriacetic acid (ED3A), and citric acid (CA) are not part of the Environmental Protection Agency's (EPA) list of targeted, hazardous chemicals; nonetheless, they are receiving renewed environmental interest. For chelators and chelator fragments, there is no accepted SW-846 type accepted methodology that we are aware of. As a result, derivatization gas chromatography (GC) was developed to analyze chelators under the auspices of the Organic Tanks Safety Program and Flammable Gas Safety Program several years ago. Derivatization gas chromatography/mass spectrometry (GC/MS) and GC/flame ionization detection (FID) for the analysis of chelators has been performed several times in the past. Samples associated with tank 241-SY-101, C-104, and C-201 (Grant, Mong, Lucke, Campbell 1996; Campbell et al. 1998; Campbell et al. 1994) and others have been analyzed.

The chelators are a class of compounds whose low volatility and high polarity preclude analysis by GC/MS or GC/FID without prior derivatization. Derivatization techniques that have been applied to the analysis of chelators include 1) reaction with boron trifluoride (BF_3/methanol) to form the methyl esters, 2) reaction with diazomethane to form methyl esters, 3) reaction with butanol/HCl to form butyl esters, and 4) reaction with silylation derivatizing reagents to form silyl derivatives (Campbell et al. 1998). The most useful method appears to be the reaction with BF_3/methanol, with the other methods giving, at best, modest results (Grant, Mong, Lucke and Campbell 1996; Campbell et al. 1994).

The analysis of tank waste samples for chelators and chelator degradation products was on a best effort basis. The samples were analyzed according test plan TP-PNNL-WTP-048 entitled "Derivatization GC/FID Analysis of Chelators and Degradation Products". In addition, the test plan TPR-RPP-WTP-049 entitled "Ion Exchange for Activity Reduction" was also employed to reduce the radioactivity level of the samples.

Chelators and degradation products were detected in the samples and duplicates. These include EDTA, HEDTA, SA, NTA, ED3A, and CA. As a result of the variability in the concentrations obtained, additional work is required to make this a reliable, robust technique.

Instrumentation

The derivatized samples containing the methyl esters were analyzed using a GC 5890 Series II (Hewlett Packard) equipped with a flame ionization detector and a capillary fused silica column (30 m X 0.25 mm X 0.25 μm film thickness). The temperature program used was the following: 40° C for 1 min, 40° C-260°C at 8°C/min and 260° C for 7 min. The injector was at 265°C and the detector was at 260°C. A 1 μL splitless injection was performed for the analyses.

The retention times and identities of the chelators and degradation products were confirmed using gas chromatography/mass spectrometry (Agilent 5973).

Standard and Sample preparation

Standards of EDTA, HEDTA, adipic acid (AA), CA, SA, NTA, and IDA were prepared ranging in concentration from approximately 500 to 30 µg/mL and subsequently derivatized with BF_3/methanol as discussed below. Adipic acid is used as a monitor of derivatization efficiency. The standards were analyzed using GC/FID.

Approximately 5 mL of supernate was diluted with 5 mL of H_2O and then eluted through a bed of AG50W-X8 resin (Biorad), 50-100 mesh, prepared in the sodium form. The resin must not be used in the usual H^+, since the chelator analytes tend to precipitate from solution as insoluble free acids when the matrix is exposed to the acid form of the resin. The sodium form of the cation exchange resin is conveniently prepared by stirring the resin with sufficient 6N NaOH until the solution remains basic to pH paper. The resin is then washed with Milli-Q water. After elution through the cation exchange resin, the net reduction of fission products (chiefly ^{137}Cs and ^{90}Sr) was sufficient that the sample could be removed from the hot cell environment and safely worked within a fume hood (Mong, Orr, Hoopes, Campbell 1998).

A 2-mL aliquot of tank waste material obtained after elution through a cation exchange resin was placed in a Reactivial and concentrated to dryness using nitrogen blow down techniques. Adipic acid was added to each of the samples as a check on derivatization efficiency. After the sample was completely dry, 2 ml of a 12% (w/v) BF_3 in methanol (Aldrich) was added and the sample was capped tightly. The Reactivial was placed into the heat block and heated to a temperature of 100° C for 1 hour. The sample was agitated twice during the reaction period. After one hour, the sample was allowed to cool to room temperature. While the sample was cooling, 15 mL of a 0.4 M K_2HPO_4 was poured into a sample vial. After the reaction mixture was cooled to room temperature, 2 mL of chloroform was added to the Reactivial and shaken. The solution was then added to the vial containing the phosphate buffer, shaken, and the aqueous and chloroform layers were allowed to separate. The pH of the aqueous layer was checked with 0-14 range pH paper and was in the range of 7-8. The top aqueous layer was decanted to waste. The bottom layer contains the derivatized organics for analysis. The chloroform solution was added to a vial containing approximately 200 mg of anhydrous Na_2SO_4 to collect the entrained water. The chloroform solution was added to a GC autosampler vial and capped. Prior to removal of the sample from the contaminated area fume hood, it was surveyed by the RCT. The samples were then analyzed using GC/FID. Confirmation of the identification of the analytes (SA, CA, EDTA, HEDTA, NIDA, and NTA) was performed using GC/MS. Tentative identification of nitroso-ED3A and ED3A was based on mass spectral data.

Results of Analyses

Standards were prepared for EDTA, HEDTA, CA, SA, IDA, and NTA (approximately 500-30 µg/mL) and derivatized using the method described above. A five-point calibration curve was constructed for EDTA, HEDTA, CA, SA, IDA, and NTA. HEDTA is also very susceptible to the final pH during the addition of phosphate buffer. This may be the reason the calibration curve for HEDTA was not linear and the results were highly variable. IDA suffers from a

similar fate or is susceptible to hydrolysis and the calibration curve is not linear, and the results were highly variable as well.

The results of analyses for the samples are shown in Table 1. In view of the fact no standards exist for nitroso-IDA (NIDA), nitroso-ED3A, or ED3A, the concentrations of these components were based on the assumption of equivalent response to EDTA. Both of these compounds are not commercially available and require independent synthesis. The results for these compounds would be termed qualitative based on the fact that no standards are readily available.

In the reaction of chelators with BF_3/methanol, methylation occurs at the carboxylic acid sites. However, methylation does not occur at hydroxy sites in the reaction of BF_3/methanol. The mass spectrum of methylated HEDTA shows a parent ion at m/z 288. The hydoxyethyl group of HEDTA resists methylation by BF_3/methanol and, instead, forms a cyclic, or intramolecular lactone with one of the neighboring acetate ligands. The MW 288 lactone accounts for only approximately 30% of the species detected. Trimethylated HEDTA with an unreacted, or free, hydroxyethyl group accounts for the remaining 70% of the HEDTA. It is speculated that the species is simply too polar to migrate through the GC column (Lokken et al. 1986; Grant, Mong, Lucke, and Campbell 1996). Due to nonlinearity and variabiltity of the calibration curve for HEDTA, the concentration of HEDTA was based on the assumption the response is similar to EDTA.

ED3A, when derivatized with BF_3/methanol, forms a lactam. In addition, ED3A also forms a nitroso-ED3A under derivatizing conditions. IDA also forms a nitroso-IDA (NIDA) under these conditions. The nitroso compounds are formed under acidic conditions (BF_3, the derivatizing reagent, is a Lewis acid) by the reaction of an analyte with a free amine hydrogen, as in the case of IDA and ED3A, with the high concentration of nitrite present in the waste samples. The identity of these compounds was confirmed using GC/MSD. It should be emphasized that the nitroso compounds are not present in the tank waste; they are artifacts of the derivatization procedure (Grant, Mong, Lucke, and Campbell 1996).

Table 1. Results of the Analyses for the AN-102 Samples and AN-AN-102 Diluted Feed (Concentrations are expressed in µg/mL of sample)

sample	SA	IDA	CA	NTA	EDTA	HEDTA	ED3A(a)
01-00429-s	35.9	3464.6	429.6	255.6	602.8	<120.1	1520.1
01-00429-d	<34.3	1388.5	<126.2	113.6	237.2	<120.1	556.7
01-00441-s	<34.3	1562.2	574.4	196.0	772.1	<120.1	761.7
01-00441-d	<34.3	1118.1	284.6	133.2	463.7	<120.1	535.7
Blank	<34.3	<120.1	<126.2	<97.2	<120.1	<120.1	<120.1

* The analyte is detected at the required retention time but the calculated concentration is below the calculated EQL. This is based on the lowest concentration of standard used in calibration.

SA- succinic acid
IDA- iminodiacetic acid
CA- citric acid
NTA- nitrilotriacetic acid
EDTA – ethylenediaminetetraacetic acid
HEDTA – N-(2-hydroxyethyl)ethylenediaminetriacetic acid
ED3A – ethylenediaminetriacetic acid
(a) Nitroso-ED3A and ED3A were tentatively identified based on mass spectral interpretation. The values for the blank were calculated using the lowest concentration of the standards used in calibration converted to the sample.

The ED3A category in Table 1 includes a combination of both ED3A and nitroso-ED3A. The concentration of IDA is based on the formation of NIDA. The concentrations of both ED3A and IDA are based on the assumption of a similar response to EDTA. Nitroso-ED3A and ED3A were tentatively identified using mass spectral data. Actual standards would have to be synthesized and compared with the retention times to positively confirm their identification.

The recovery for AA in the blank was 86%. The percent recoveries for AA for 429-s (91 %), 429-d (80%), 441-s (98%), and 441-d (85 %). The data were not corrected based on recovery of AA. Due to the fact that the chelators are very similar in structure and reactivity to EDTA and the acids similar to CA, only EDTA and CA were added to both the matrix spike and duplicate. For the matrix spike and matrix spike duplicate, the following results were obtained. The recoveries for the matrix spike and duplicate are listed in Table 2. This represents the first time MS and MSD samples have been analyzed with this technique.

Table 2. Recoveries for the MS and MSD

	CA	EDTA
01-00441-MS	47%	66%
01-00441-MSD	50%	58%

The recovery for adipic acid in these two samples are 107% (441-MS) and 103% (441-MSD). Chelators were not found in the blank.

The laboratory control sample (LCS) was compromised during sample preparation in the contamination hood, and a LCS was prepared and the corresponding sample preparation was performed outside of the hot cell facilities. The sample was eluted through cation exchange resin column. An aliquot was concentrated, derivatized, and analyzed using GC/FID. The corresponding recoveries for AA were 106%, SA 69%, EDTA 78%, NTA 71%, CA 83%, and HEDTA 38%. The concentration of HEDTA was not corrected.

Neither the Test Specification nor the governing QA Plan provides QC acceptance criteria for the chelator analysis. In addition, the target values for recoveries listed in the Test Plan are target values. There is no historical QC data for this analysis technique.

Recommendations

The analysis of tank waste samples for chelators and chelator degradation products was performed on a best effort basis. This a very complex procedure due to moisture and safety considerations. In addition, the formation of compounds (e.g. methyl ester of HEDTA) appears to be pH dependent and the recoveries are variable. The variation in recoveries and reproducibility has also been evident in other studies as well (Campbell et al. 1994; Campbell et al. 1998; Grant, Mong, Lucke, Campbell 1996). A problem encountered with these tank samples involved the separation of the chloroform and aqueous layer during derivatization. The solution was very cloudy which resulted in a lack of phase distinction. This may be an explanation for the variability in the results. The degree of variability will have to be addressed. One of the ways of possibly improving the variability is using smaller sample sizes. As the sample dries it tends to form a concrete-like material which may be impervious to the derivatizing reagent. Using a smaller sample may provide a thiner crusty level, and the derivatization process may be better. Heating at lower temperatures for longer periods of time during the derivatization process may also provide less variability in results. Further work with simulants to perfect the technique would be advisable; additional spiking experiments would also be beneficial. The derivatization method for chelators and degradation products, in its present form, requires additional methods development. A direct analysis technique, with no derivatization, potentially would be a more robust and reliable technique. Preliminary results with capillary electrophoresis shows promise as a direct analysis technique for chelators and degradation products; however, additional methods development is required for this technique as well.

The results for HEDTA, IDA, and ED3A should be considered semiquantitative in view of the fact that no standards exist for the nitroso-IDA, ED3A, and nitroso-ED3A and the variability observed with the results of HEDTA. It should be emphasized that this technique has not been thoroughly verified in terms of QA and QC requirements. However, I do think the potential is there for this technique to be useful for the analysis of chelators. Prior to this technique becoming a reliable technique, additional work is required. Work with tank waste simulants would lessen the cost of methods development.

It is estimated the additional methods development time to do spiking studies and solve the variability problems would require approximately 4-5 weeks. Simulated waste studies would be suggested to lessen the cost. In addition, under the auspices of another project, methods development involving capillary electrophoresis is underway. With this technique, the samples would be analyzed directly; no derivatization would be required prior to analysis. However, capillary electrophoresis methods development is just underway. Within several months, a capillary electrophoresis instrument capable of analyzing radioactive samples will be available. In order to detect smaller concentrations of chelators, GC/MS may be a more sensitive technique. The problems associated with sample preparation would still be present.

The concentration of citric acid as determined by ion chromatography is approximately 10 times the concentration determined by derivatization GC/MS. The IC method is a direct analysis method; no derivatization is required prior to analysis. As a result, the result obtained by IC may be more reliable at this point.

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In view of the fact there is a discrepancy in the TOC accounted for, I would like to suggest that other aliquots be dried, derivatized, and analyzed. The problem associated with the drying process has been tentatively identified and corrected. As a result, the drying time should be much less. The samples are already available and would not have to go through the hot cell facilities.

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