PNWD-2465
BNFL-RPT-006 Rev. 0

Pacific Northwest
National Laboratory
Operated by Battelle for the
U.S. Department of Energy

LETTER REPORT

Washing of the AW-101 Entrained Solids

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August 1999

Prepared for BNFL, Inc. under
Project 29952/29953
Battelle, Richland, Washington, 99352

PNWD-2465

DISCLAIMER

Contents

1.0 Introduction

BNFL Inc. (BNFL) is under contract with the U.S. Department of Energy, River Protection Project (DOE-RPP) to design, construct, and operate facilities for treating wastes stored in the single-shell and double-shell tanks at the Hanford Site, Richland Washington. The DOE-BNFL RPP contract identifies two feeds to the waste treatment plant: 1) primarily liquid low-activity waste (LAW) consisting of less than 2 wt% entrained solids and 2) high-level waste (HLW) consisting of 10 to 200 g/L solids slurry.

The RPP contract includes three options for disposition of the entrained solids contained in low-activity waste feed solutions: 1) washing to remove sodium, cesium, and technetium then returning *via* pipeline to DOE-RPP, 2) vitrification along with pretreated LAW solutions, or 3) vitrification along with pretreated high-level waste (HLW).

BNFL requested Battelle test inhibited water (0.01 M NaOH) and caustic leaching (3 M NaOH) as methods for pretreating the solids entrained in the AW-101 sample. These methods are meant to remove certain nonradioactive components (e.g., Na, Al, Cr, P, and S) from the HLW fraction so as to reduce the volume of immobilized HLW.

This report describes the results of a test conducted by Battelle to assess the effects of inhibited water washing on the composition of the entrained solids in the diluted AW-101 low-activity waste (LAW) sample. The objective of this work was to gather data on the solubility of the AW-101 entrained solids in 0.01 M NaOH, so that BNFL can evaluate whether these solids require caustic leaching. The work was conducted according to test plan BNFL-TP-29953-9, Rev. 0, *LAW Entrained Solids Water Wash and Caustic Leach Testing*. The test went according to plan, with no deviations from the test plan. Based on the results of the 0.01 M NaOH washing, a decision was made by BNFL to not proceed with the caustic leaching test. The composition of the washed solids was such that caustic leaching would not result in significant reduction in the immobilized HLW volume.

2.0 Personnel

The Battelle personnel and their responsibilities in performing this test are given below.

Staff Member	Responsibilities
G.J. Lumetta	Cognizant scientist. Prepared test plan and designed experiment. Supervised performance of the test. Prepared analytical service request. Interpreted data and reported results.
R.C. Lettau	Hot cell technician. Performed test.
M.W. Urie	Managed chemical and radiochemical analytical work.
B.M. Rapko	Technical reviewer.
K.P. Brooks	Task Leader.

3.0 Experimental

Sample Description. The sample used in this test was labeled as AW-101 CL-1. The homogenization, dilution, caustic adjustment, and representative subsampling were performed as described in test plan BNFL-29953-6, *Sub-Sampling and Characterization of AN-107 and AW-101 Diluted Feed Samples* (Urie et al. 1999). The total volume of sample AW-101 CL-1 was 85 mL and it contained approximately 5 mL of settled solids. This sample was half of the material indicated as the "Caustic Leach" sample in Figure 1.1 of Urie et al. (1999).

Apparatus. The apparatus used consisted of an aluminum heating block placed on a hot plate/stirrer. The hot plate/stirrer was modified so that separate power could be applied to the heating and stirring functions. This allowed for continuous stirring, while the hot plate was powered by a temperature controller. The temperature controller used was a J-KEM Model 270 (J-KEM Electronics, Inc., St. Louis, MO). This temperature controller consists of two separate circuits. One is the temperature control circuit, while the other serves as an over-temperature device, which shuts down the system if a preset temperature is exceeded. The set point for the over-temperature circuit was set at 100°C for this test. A dual K-type thermocouple (model number CASS-116G-12-DUAL, Omega Engineering, Stamford, CT) was used to provide inputs to the temperature controller and over-temperature circuits. Both the J-KEM Model 270 and the dual thermocouple were calibrated before use. The aluminum heating block contained two wells. A vial containing water was placed in one of the wells, with the thermocouple wedged between this vial and the aluminum block. The vial containing the sample was placed in the other well.

Procedure.[a] The sample in AW-101 CL was mixed by swirling. The homogenized slurry was then filtered through a pre-weighed 0.45-μm nylon filtration unit (Nalgene no. 150-0045, Nalge Nunc International, Rochester, New York). The weights of the filtrate and filtered solids were determined to be 108.127 g and 2.075 g, respectively. Five 4-mL aliquots of 0.01 M NaOH were used to transfer the filtered solids to a 30-mL high-density polyethylene (HDPE) vial (this vial also contained a Teflon®-coated magnetic stir bar). The weight of the washing slurry was 18.769 g. This value is ~15% less than expected based on the weight of the filtered solids and the 0.01 M NaOH (washing soluiton) added; this was perhaps due to loss of liquid through the membrane during transferring process. The vial was equipped with a condenser tube, which allowed the system to vent during heating, but minimized evaporation. The mixture was heated and stirred at 85 ± 2 °C for 17 h. After cooling to room temperature, the mixture was weighed. The weight was 18.242 g, indicating 0.527 g lost to evaporation. The washing slurry was filtered through a pre-weighed 0.45-μm nylon filtration unit. During the transfer to the filter funnel, the stir bar also fell into the filter funnel. This was lifted out and the solids stuck to it were rinsed into the filter with a small amount of 0.01 M NaOH. The weights of the filtrate and filtered solids were determined to be 17.461 g and 1.317 g, respectively. Two aliquots (~10-mL each) of the filtrate were taken for analysis. There were no solids in this solution after 21.5 h, indicating no precipitation following filtration.

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[a] See Appendix A for a copy of the test plan and procedural notes.

The washing procedure described above was repeated three times for a total of four washes. The heating and mixing times for the second, third, and fourth washing steps were 16, 20, and 21 h, respectively. There was no evidence of precipitation in the wash solutions after standing overnight. The weight of the wet filtered solids were 1.210, 1.314, and 1.128 g after the second, third, and fourth washing steps, respectively. These weights can be viewed as nearly constant given the potential for variable water content in the wet solids. After the fourth washing step, the solids were transferred to a pre-weighed glass vial using deionized water. Excess water was evaporated at 80°C, then the solids were dried overnight at 105°C. The final weight of the dried washed solids was 0.058 g. This low weight was surprising given the wet weight of ~1 g. The solids apparently have a strong propensity to retain water within the filter unit.

The wash solutions were subjected to the following analytical procedures: IC(anions), TOC/TIC, acid digestion, ICP/AES, ICP-MS(Tc-99), Sr-90, total alpha, total uranium, and GEA.

Because of the small quantity of washed solids, it was not possible to perform all the analyses originally stated in the test specification. To dissolve the solids for analysis, 5 mL of 12 M HCl was added to the glass vial containing the dried washed solids. After heating at 90°C for ~1.5 h, most of the solids had dissolved, but some remained. In an attempt to dissolve the remaining solids, 1 mL of 16 M HNO_3 was added. Again, the mixture was heated at 90°C. After 1.75 h, a white solid had collected around the threads of the vial cap. The sample was evaporated to dryness at 90°C, then five 5-mL aliquots of 0.1 M HCl was used to quantitatively transfer the material to a clean HDPE vial. One-mL of 10 M HF was added and the mixture was evaporated at ~80°C until only about 2 mL remained. Another 5 mL 0.1 M HCl was added and the solution was filtered through a 0.2-μm nylon membrane. The filtered solution was diluted to 25 mL with 0.1 M HCl. This solution was subjected to the following analytical procedures: ICP/AES, ICP-MS(Tc-99), Sr-90, total alpha, total uranium, and GEA. The small amount of gray filtered solid was saved, but was not further analyzed.

4.0 Results

Table 1 presents the concentration of the analyzed AW-101 components in each washing solution and in the washed solids. A caveat must be placed on the results for the washed solids: the concentrations listed in Table 1 assume that the washed solids were dissolved completely for analysis. As indicated in the experimental section, a small amount of material did not dissolve on treatment with acid. Table 2 lists the mass (or activity) of each analyzed component present in each wash solution and the washed solids and Table 3 gives the percentage of each component found in each solution and the washed solids. These values were obtained by dividing the amount of the given component found in a particular solution or the washed solids (i.e., the value in Table 2) by the total amount of that component found in all the wash solutions and the washed solids; the resulting fraction was multiplied by 100 to give the percentage values.

Aluminum, K, and Na were removed reasonably well from the AW-101 entrained solids. The Na concentration in the final wash solution (243 μg/mL = 0.0106 M) was essentially the same as that in the wash solution added (0.010 M NaOH) indicating that essentially all soluble Na-containing components were removed. Only about 40% of the Cr was removed by dilute

hydroxide washing. The washed solids contained 3.5 wt% Cr. The main elements in the residual solids were U (18.5 wt%), Si (17.2 wt%), Na (5.7 wt%), Fe (4.9 wt%), Mn (4.5 wt%), Cr (3.5 wt%), Al (2.9 wt%), and Ca (2.3 wt%).

The radiochemical data indicated nearly quantitative removal of ^{137}Cs from the AW-101 entrained solids. Approximately 70% of the ^{99}Tc was also washed from the solids. The wash solution could be processed along with the liquid fraction of the AW-101 LAW to remove these two radioisotopes. Small fractions of the ^{90}Sr and TRU might also be present in the wash solution.

Much of the material found in the first wash solution can be attributed to dilution of the interstitial liquid rather than actual dissolution of entrained solids. Table 4 illustrates this. The volume of interstitial liquid in the filtered solids was estimated in the following manner. First, it was assumed that the Na present in the first wash solution was due only to dilution of the diluted AW-101 supernate and the 0.01 M NaOH (230 μg/mL Na) used as the washing medium. The Na concentration in the first wash solution was 12150 μg/mL, of which 12150 – 230 = 11,920 μg/mL is attributed to dilution of the interstitial supernate. Given the wash solution volume of 16.9 mL and the Na concentration in the diluted AW-101 supernate was 148,500 μg/mL (Urie 1999), the volume of the interstitial liquid was estimated as

$$V = (16.9\ mL)(11{,}920\ \mu g/mL)/(148{,}500\ \mu g/mL) = 1.36\ mL$$

This value was then used to determine the concentration expected for each AW-101 component expected in the first wash solution based on dilution (Table 4). In many cases, the difference between what was expected from dilution and what was actually measured was within 20%, indicating dilution was primarily responsible. Notable exceptions were ^{99}Tc, Cr, Si, U, TOC, and TIC. Thus, the washing procedure appeared to actually remove fractions of these latter components.

Table 5 presents the mass recoveries for the major waste components. These mass recoveries were calculated using the composition of the diluted AW-101 feed material reported by Urie et al. (1999). In that work, the AW-101 solids were dissolved for analysis using a KOH fusion method. The mass recoveries were generally low. This is probably due to a combination of loss of material during the various transfers made during the test (e.g., the transfer of solids from the filter membrane back into the washing bottle) and the incomplete dissolution of the washed solids for analysis. If the latter reason is the dominant cause, we can adjust the concentrations in the washed solids for the material not accounted for.

For example, based on the data in Urie et al. (1999) and the mass of the sample used, the amount of Al in the sample was calculated to be 31,542 μg. Yet, only 23,454 μg were determined in the wash solutions and the washed solids (a 74% recovery). Thus, 8,088 μg of Al was unaccounted for. Assuming this was in the undissolved portion of the washed solids, the adjusted Al concentration in the washed solids is given by (1682 + 8088 μg)/(0.0577 g solids) = 169,000 μg/g (Note: The 1682 μg is the amount measured in the washed solids and 0.0577 g was the weight of the washed solids). In this manner, the following adjusted values for the washed solids were determined: Al (16.9 wt%), Cr (5.5 wt%), Fe (6.6 wt%), Mn (6.7 wt%), and U (25.6 wt%).

Table 6 presents further comparisons to the data for the entrained solids reported in Urie et al. (1999). The concentrations could not be compared directly because the composition for the untreated entrained solids were reported on a wet-weight basis, whereas the washed solids were analyzed on a dry-weight basis. For this reason, the data were normalized to the Fe content. The percent of each component was determined based on the differences in the component concentrations relative to Fe before and after washing. For certain components (e.g., Cs, Tc, Al, Cr, K, and Na), the percent removals obtained in this manner agreed well with those reported in Table 3. However in most other cases, the removals indicated in Table 6 appear unreasonably high.

5.0 Conclusions and Recommendations

The results of this test suggest that caustic leaching would not provide much benefit for processing the AW-101 entrained solids. Washing with 0.01 M NaOH appeared to remove > 90% of the Al from the AW-101 solids. There is some uncertainty in this conclusion because of the low mass recovery for Al. Taking this uncertainty into account, the Al concentration in the washed solids was 2.9 to 16.9 wt%. Uranium is a major component of the washed AW-101 solids. Caustic leaching test with other tank sludges indicated that U in not generally soluble in the caustic media use (but there are some exceptions) (see Lumetta et al. 1996 and 1997; Rapko et al. 1995).

The Cr concentration (3.5 to 5.5 wt%) might present some problems in immobilizing the washed AW-101 solids. Previous studies we have done with other sludges suggest that caustic leaching might remove additional Cr, but a better strategy would be to add an oxidant during the washing process. Permanganate works very well, but sparging with air or ozone has also shown some promise (Rapko et al. 1996 and 1998). If the HLW volume is dictated by the Cr content, then an oxidative leaching process is recommended.

The concentrations of the major radionuclides contained in the washed solids were 4.51 μCi TRU/g (as indicated by the total alpha concentration), 2.43 μCi ^{241}Am/g, 1,950 μCi ^{90}Sr/g, and 35 μCi ^{137}Cs/g, indicating the solids should be treated as HLW. The washed solids represented only 0.05 wt% of the diluted AW-101 feed material. The blending of this material with the HLW sludge to be processed in Phase 1 Privatization should be considered. The impact to the overall flowsheet assuming the worst-case 16.9 wt% Al and 5.5 wt% Cr values, should be evaluated. Perhaps even with these assumed high Al and Cr concentrations in the washed LAW entrained solids, the overall impact of these solids on the flowsheet would be minimal if they were blended with the bulk HLW feed.

6.0 References

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Table 1. AW-101 Component Concentrations in the Wash Solutions and the Washed Solids.[a]

Analyte	First Wash AW101-AQ-30A[b]	Second Wash AW101-AQ-50A	Third Wash AW101-AQ-70A	Fourth Wash AW101-AQ-90A	Washed Solids AW101-AQ-100
Cesium-137	1.70E+01	1.14E+00	3.15E-01	1.88E-01	3.50E+01
Strontium-90	3.80E-02	1.75E-02	7.19E-02	6.85E-02	1.95E+03
Technetium-99	2.35E-02	2.53E-03	8.06E-04	3.93E-04	3.22E+00
Americium-241	<3E-04	<5E-05	<7E-04	<1E-03	2.43E+00
Europium-154	<2E-03	<2E-04	<8E-05	<2E-04	6.33E-01
Europium-155	<1E-02	<2E-03	<5E-04	<8E-04	7.80E-01
Total Alpha	<3E-04	<5E-05	2.70E-04	2.52E-04	4.51E+00
Ag	<1.9	<0.8	<0.8	<0.8	382
Al	1090	89.6	49.6	38.5	29159
Ba	<1.3	(0.1)	(0.1)	(0.2)	2803
Ca	<12.5	<5.0	<5.0	<5.0	23050
Cd	<1.9	<0.8	<0.8	<0.8	1135
Co	<3.1	<1.3	<1.3	<1.3	159
Cr	65.0	5.36	1.72	(0.47)	34965
Cu	<1.9	<0.8	<0.8	<0.8	563
Fe[c]	<3.1	(0.38)	(0.65)	(0.67)	48960
K	1615	(58)	<100	<100	(2166)
La	<3.1	<1.3	<1.3	<1.3	111
Mg	<12.5	<5.0	<5.0	<5.0	2080
Mn	<0.6	(0.1)	0.428	0.382	45494
Mo	<3.8	<1.5	<1.5	<1.5	<13
Na	12150	783	283	243	56759
Ni	(1.4)	<1.5	<1.5	<1.5	7149
P	(12)	(1.2)	(0.57)	<5.0	2045
Pb	<7.5	<3.0	<3.0	<3.0	3726
Si[d]	74.2	62.9	72.4	61.2	172444
Ti	<0.6	<0.3	(0.03)	(0.03)	332
U	3.03	1.19	4.45	4.16	175325
Zn[e]	<2.5	<1.0	<1.0	(0.25)	6716
Zr	<3.1	<1.3	(0.27)	(0.25)	7496
TOC	1900	<170	<80	<80	(f)
TIC	410	190	120	120	(f)
Cl^-	250	11	3	2.5	(f)
F^-	100	6.0	<1.4	<1.4	(f)
NO_3^-	7300	360	34	2.7	(f)
SO_4^{2-}	120	6.5	<2.8	<2.8	(f)
PO_4^{3-}	<50	<2.8	<2.8	<2.8	(f)

(a) For the liquids, concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. For the washed solids, concentrations for radionuclides are in units of µCi/g dry solids; all other components are in units of µg/g dry solids. Values in parentheses are within 10 times the analytical detection limit.
(b) The reported values for the metals are the average of two duplicate ICP/AES analyses.
(c) The process blank had a relatively high Fe content of 0.4 µg/mL.
(d) The process blank had a relatively high Si content of 119 µg/mL.
(e) The process blank had a relatively high Zn content of 0.3 µg/mL.
(f) Not determined because of acid dissolution method used to prepare analyte solution.

Table 2. Quantities in Each Wash Solution and in the Washed Solids[a]

Analyte	First Wash	Second Wash	Third Wash	Fourth Wash	Washed Solids	
Cesium-137	2.87E+02	2.21E+01	5.96E+00	3.35E+00	2.02E+00	
Strontium-90	6.42E-01	3.39E-01	1.36E+00	1.22E+00	1.13E+02	
Technetium-99	3.98E-01	4.92E-02	1.53E-02	6.99E-03	1.86E-01	
Americium-241	< 5E-03	< 1E-03	< 1E-02	< 2E-02	1.40E-01	
Europium-154	< 3E-02	< 4E-03	< 2E-03	< 4E-03	3.65E-02	
Europium-155	< 2E-01	< 4E-02	< 9E-03	< 1E-02	4.50E-02	
Total Alpha	< 5E-03	< 1E-03	5.11E-03	4.48E-03	2.60E-01	
Ag	< 32	< 16	< 15	< 14	22	
Al	18407	1741	939	685	1683	
Ba	< 22	(1.8)	(1.2)	(2.7)	162	
Ca	< 211	< 97	< 95	< 89	1330	
Cd	< 32	< 16	< 15	< 14	66	
Co	< 52	< 25	< 25	< 23	9	
Cr	1098	104	33	(8.4)	2018	
Cu	< 32	< 16	< 15	< 14	33	
Fe	< 52	(7.4)	(12.3)	(11.9)	2825	
K	27273	(1127)	< 1893	< 1779	(125)	
La	< 52	< 25	< 25	< 23	6	
Mg	< 211	< 97	< 95	< 89	120	
Mn	< 10	(1.8)	8.1	6.8	2625	
Mo	< 64	< 29	< 28	< 27	< 1	
Na	205180	15211	5358	4324	3275	
Ni	(24)	< 29	< 28	< 27	413	
P	(203)	(23)	(11)	< 89	118	
Pb	< 127	< 58	< 57	< 53	215	
Si	1252	1222	1371	1089	9950	
Ti	< 10	< 6	(0.59)	(0.57)	19	
U	51.1	23.1	84.3	74.0	10116	
Zn	< 42	< 19	< 19	(4.4)	388	
Zr	< 52	< 25	(5.1)	(4.4)	433	
TOC	32086	< 3303	< 1515	< 1423		(b)
TIC	6924	3691	2272	2135		(b)
Cl^-	4222	214	57	44		(b)
F^-	1689	117	< 27	< 25		(b)
NO_3^-	123277	6994	644	48		(b)
SO_4^{2-}	2026	126	< 53	< 50		(b)
PO_4^{3-}	< 844	< 54	< 53	< 50		(b)

(a) Radionuclides are given in µCi; other compoents are in µg. Values in parentheses are for components that were within 10 times the analytical detection limit.

(b) Not determined because of the acid dissolution method used to prepare the analyte solution.

Table 3. Percentage of Each AW-101 Component in the Wash Solutions and in the Washed Solids[a]

Analyte	First Wash	Second Wash	Third Wash	Fourth Wash	Washed Solids
Cesium-137	90	7	2	1	1
Strontium-90	1	0	1	1	97
Technetium-99	61	8	2	1	28
Americium-241	< 3	< 1	< 7	< 10	> 79
Europium-154	< 43	< 5	< 2	< 4	> 46
Europium-155	< 61	< 14	< 3	< 5	> 16
Total Alpha	< 2	0	2	2	96 > x > 94
Ag	< 32	< 16	< 15	< 14	> 22
Al	78	7	4	3	7
Ba	< 12	(1)	(1)	(1)	88 > x > 85
Ca	< 12	< 5	< 5	< 5	> 73
Cd	< 23	< 11	< 11	< 10	> 46
Co	< 39	< 19	< 18	< 17	> 7
Cr	34	3	1	(0.3)	62
Cu	< 29	< 14	< 14	< 13	> 30
Fe	< 2	(0.3)	(0.4)	(0.4)	97
K	85	(3)	< 6	< 6	< 15
La	< 40	< 19	< 19	< 18	> 5
Mg	< 34	< 16	< 15	< 15	> 20
Mn	< 0	(0.1)	0.3	0.3	99
Mo	< 43	< 20	< 19	< 18	> 1
Na	88	7	2	2	1
Ni	(5)	< 6	< 5	< 5	95 > x > 79
P	(46)	(5)	(2)	< 20	47 > x > 27
Pb	< 25	< 11	< 11	< 10	> 42
Si	8	8	9	7	67
Ti	< 28	< 16	(2)	(2)	> 53
U	0.5	0.2	0.8	0.7	98
Zn	< 9	< 4	< 4	(0.9)	> 82
Zr	< 10	< 5	(1.0)	(0.9)	> 83

(a) Parentheses indicate that component was within 10 times the analytical detection limit.

Table 4. Expected Concentrations in the First Wash Solution Based on Dilution of the Interstitial Liquid[a]

Analyte	Diluted Supernate[b]	Concentration in First Wash		
		Based on Dilution[c]	Found	Difference, %
Cesium-137	230	18.5	17.0	-8
Strontium-90	< 0.5	< 0.04	0.038	-6
Technetium-99	0.094	0.0075	0.024	213
Al	16350	1317	1090	-17
Cr	56.1	4.52	65.0	1339
K	23000	1852	1615	-13
Na	148500	11959	12150	2
Ni	(4.8)	(0.39)	(1.4)	262
P	323	26.0	(12)	-54
Si	(130)	(10.5)	74.2	608
U	3.22	0.259	3.03	1067
TOC	1560	126	1900	1412
TIC	2155	174	410	136
Cl^-	3300	266	250	-6
F^-	830	67	100	50
NO_3^-	123000	9906	7300	-26
SO_4^{2-}	1850	149	120	-19

(a) Radionuclides are reported in units of µCi/mL; all other components are in units of µg/mL.
(b) Values taked from Urie 1999. Each value is an average of duplicate measurements.
(c) It was assumed that there were 1.36 mL of interstitial liquid. This value was determined assuming the Na concentration in the wash solution was strictly due to dilution of the interstitial liquid plus the 0.01 M NaOH used as the wash medium.

Table 5. Mass Recoveries for Key AW-101 Waste Components

Component	Recovery, %
Cesium-137	78
Strontium-90	28
Technetium-99	71
Americium-241	26
Total Alpha	20
Al	74
Cr	74
Fe	76
K	91
Mn	66
P	50
U	69

Table 6. Comparison of the Compositions of the Washed AW-101 Solids to the Wet Untreated Solids

	Wet Entrained Solids[a]		Dry Washed Solids		
Analyte	µCi/g or µg/g	Ci/g Fe or g/g Fe	µCi/g or µg/g	Ci/g Fe or g/g Fe	Removed, %
Cesium-137	192	0.138	35.0	7.14E-04	99
Strontium-90	151	0.108	1954	3.99E-02	63
Technetium-99	0.350	0.00025	3.22	6.57E-05	74
Americium-241	0.25	0.00018	2.43	4.96E-05	72
Europium-154	< 0.2	< 0.0001	0.63	1.29E-05	91
Europium-155	< 0.5	< 0.0004	0.78	1.59E-05	96
Total Alpha	0.511	0.00037	4.51	9.20E-05	75
Ag	(90)	(0.064)	382	0.0078	88
Al	14500	10.4	29159	0.596	94
Ba	(25)	(0.018)	2803	0.057	-218
Ca	(1700)	(1.22)	23050	0.471	62
Cd	(35)	(0.025)	1135	0.023	7
Co	< 44	< 0.03	159	0.0032	90
Cr	1620	1.17	34965	0.714	39
Cu	< 22	< 0.02	563	0.012	27
Fe	1390	1.00	48960	1.00	--
K	17200	12.4	(2166)	(0.044)	100
La	< 44	< 0.03	111	0.0023	93
Mg	(255)	(0.183)	2080	0.042	77
Mn	1415	1.02	45494	0.929	9
Mo	< 44	< 0.03	< 13	< 0.0003	99
Na	127500	91.7	56759	1.16	99
Ni	215	0.155	7149	0.146	6
P	(385)	(0.277)	2045	0.042	85
Pb	(120)	(0.086)	3726	0.076	12
Si	(2200)	(1.58)	172444	3.52	-123
Ti	< 22	< 0.02	332	0.0068	57
U	5440	3.91	175325	3.58	9
Zn	< 44	< 0.03	6716	0.137	-333
Zr	(220)	(0.158)	7496	0.153	3

(a) Urie et al. 1999.

(b) Percent removed = $100*(C_o-C)/C_o$ where C_o is the Fe-normalized concentration in the wet centrifuged and C is the Fe-normalized concentration in the washed solids.

Appendix A. Test Plan

Work place Copy

PNNL Test Plan	Document No.: BNFL-TP-29953-9 Rev. No.: 0
Title: LAW Entrained Solids Water Wash and Caustic Leach Testing	
Work Location: RPL/SAL	Page 1 of 19
Author: GJ Lumetta	Effective Date: December 14, 1998 Supersedes Date: New
Use Category Identification: Mandatory	
Identified Hazards: __ Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	Required Reviewers: X Technical Reviewer X Other: Client __Building Manage X Other: Project Manager __Radiological Control X Other: RPL Manager __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 ___ No
Does the OJT package associated with this procedure require revision to reflect procedure changes? __ Yes__ No__ N/A

Approval:

	Signature	Date
Author	Gregory J. Lumetta	1/5/99
Technical Reviewer	Brian Rapko	1-11-99
RPL Manager	[signature]	1-12-99
Project Manager	D. E. Kurath	1/11/99
RPG QE	[signature]	1/12/99
BNFL	P. Swanson	1/19/99

Applicability

This test plan is to be used to determine 1) the aqueous-insoluble fraction of the entrained solids from BNFL LAW samples and 2) the caustic-insoluble fraction of the entrained solids from BNFL LAW samples. The work will be conducted in the SAL hot cells. The work will be conducted by Radiochemical Processing Group staff. This work is being done as part of the Technical Support to BNFL for Phase 1B project.

Test Objectives

Justification: This activity supports confirmation of the process sequence, equipment performance, and design parameters for caustic leaching of solids separated from the low-activity waste (LAW) solutions.

Objective: This task will gather data on the inhibited water solubility of solids entrained in the LAW solutions. Caustic leaching experiments will estimate the removal efficiency for caustic soluble components and aid in determining the disposition of these solids.

Definitions

BNFL	British Nuclear Fuels Ltd.
HDPE	High-density polyethylene
HLW	High-level waste
RPL	Radiochemical Processing Laboratory

Emergency Response

In the event of building audible alarms (e.g., fire or criticality) personnel should proceed in accordance with the RPL Building Emergency Procedure. If time permits, ensure that test materials are secured from spilling prior to exiting the area.

Quality Control

Quality assurance for work conducted under this Test Plan is governed by the Standards-Based Management System (SBMS). The quality control for each analysis will be established per Quality Assurance Plan MCS-033. MCS-033 specifies the minimum calibration and verification requirements for analytical systems, as well as batch processing quality control samples to monitor preparations (i.e., blanks, duplicates, matrix spikes, and laboratory control standards).

A work place copy of this document shall be present at the work location. Specific information regarding each test (e.g., sample numbers) will be recorded on the work place copy and kept as project records.

As discussed in the Prerequisites section, calibrated balances must be used in performing this test. Likewise, a calibrated temperature controller is required. The calibration ID, date of calibration, and calibration expiration date must be recorded on the work place copy for each balance used and for the temperature controller.

Measured weights will be recorded on the work place copy at the indicated spot in the work instructions.

Hand written changes or corrections made to the work place copy will be made by means of a single line-out. Such changes or corrections shall be initialed and dated by the staff member making the change and by the cognizant scientist.

Equipment Description

A standard laboratory hot plate/magnetic stirrer will be used for this test. An aluminum heating block will be placed on the hot plate/stirrer to heat the sample. The apparatus will be equipped with two thermocouples. One of the thermocouples will be connected to a temperature controller, while the other will be connected to an over-temperature shut-off device. The latter will be used to ensure the sample is not over heated, which could result in lose of sample.

Prerequisites

Staff performing the work must read and understand the entire test plan prior to beginning work.

The following are items that should be staged prior to start of the test.

- Wide-mouth HDPE bottle; size to be determined (2)
- 20-mL HDPE vial (14)
- 30- to 40-mL glass vials (2)
- Hot plate/stirrer
- Aluminum heating block
- Temperature controller with temperature read-out
- Over-temperature shut-off device
- 0.45-µm nylon syringe filters (2)
- 5-mL syringes (2)
- 0.45-µm nylon disposable filter units (9)
- Adjustable 5-mL pipette
- 0.01 M NaOH
- 3 M NaOH

Preparation of 0.01 M NaOH/L

10 mL 0.1014 ± 0.0001 M NaOH (9/10/98 rgs Chem Rec 43) was diluted to 100mL with deionized water.
M.J. Turcotte
2/19/99

Another batch was prepared in the same manner.
2/17/99 M.J.T.

The temperature controller shall be calibrated by maintenance services. Record the following information regarding the temperature controller used.

		Dual Thermocouple	
Calibration ID:	02093	02899	02900
Calibration Date:	1/12/99	1/99	1/99
Expiration Date:	1/2000	1/2001	1/2001

A calibrated balance is required for this test. Record the following information regarding the balance(s) used.

	Cell-2		
Calibration ID:	360-06-01-016	Calibration ID:	
Calibration Date:	9-2-98	Calibration Date:	
Expiration Date:	2-99	Expiration Date:	

Before beginning work, a routine performance check should be performed and documented in the space below.

SN # HN2111 Weights due 4/99
SAL Cell-2.

5g = 5.0000
10g = 10.0000
20g = 20.00021 Rec
50g = 50.0002

Dom m 2/9/99 n.l.t.

Work Instructions

Notes
Where practical, catch pans should be used when working with the tank waste samples, so that they can be recovered if spilled. Throughout this test plan bottle, vials, etc. are labeled as "_____-XX-YY." The labels XX and YY are defined in the text. The tank number should be filled in the blank, e.g., "AW101."

Part 1. Determination of Aqueous-Insoluble Fraction

1.1. Obtain a LAW sample (AW-101 CL1) containing ~5 mL of settled solids, as directed by the cognizant scientist. Stir to homogenize the sample.

1.2 Label a disposable filter unit (0.45-μm nylon) as AW101-AQ-10

2/9/99 n.l.t. 1.3 Weigh AW101-AQ-10

Wt. AW101 AQ-10 = 65.7748 g (1.3A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 42.2160 g (1.3B)

2/10/99 n.l.t. 1.4 Connect AW101-AQ-10 to the vacuum line

2/10/99 14:00 M.M.-

1.5 Filter the homogenized sample through AW101-AQ-10

1.6 Disconnect from the vacuum once the liquid has filtered

1.7 Place the cap on the top of the filter unit and weigh AW101-AQ-10

Weighed in two pieces.
Bottom w/o cap = 136.1330
Top = 39.8434

Wt. AW101-AQ-10 = 175.9764 g (1.7A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = 150.3426 g (1.7B)

Save the filtered solution.

1.8 Determine the total weight of the sample

Wt. Sample = 1.7A-1.3A = 110.2016 g (1.8A)

Determine the weight of the filtered liquid

Wt. Liquid = 1.7B-1.3B = 108.1266 g (1.8B)

Determine the weight of the filtered solids

Wt. Solids = 1.8A-1.8B = 2.0750 g (1.8C)

1.9 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 20 mL (1.9A)

1.10 Label an appropriately sized wide-mouthed HDPE bottle as AW101-AQ-20. Place a stir bar in this bottle.

1.11 Weigh AW101-AQ-20 including cap and stir bar

Wt. AW101-AQ-20 = 12.4780 g (1.11A)

→ 4 mL each ++++

1.12 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 1.9A ÷ 5); transfer this slurry to AW101-AQ-20 Solids stuck fairly well to the filter membrane — difficult to slurry, but was OK once wetted down good.

1.13 Repeat step 1.12 four times to ensure complete transfer of the solids to AW101-AQ-20 *

Note: On third rinsing of filter, a few drops missed the bottle. → Used pipet to suck up as much as possible from secondary containment. ↓ into AW101-AQ-20

1.14 Place the cap back on AW101-AQ-20 and weigh

Wt. AW101-AQ-20 = 31.2475 g (1.14A)

Also, this appeared as though a small amount of the 0.01 M NaOH passed through the filter. } This was also caught in secondary containment and transferred to AW101-AQ-20.

* A small amount of solid residue remained on the filter - this was discarded.

M.M.- 2/10/99

Determine the weight of the slurry

Wt. Slurry = 1.14A-1.11A = 18.7685 g (1.14B)

1.15 Equip AW101 -AQ-20 with a condenser, then place in an aluminum heating block at 85°C

1.16 Stir the sample in AW101 -AQ-20 at 85°C for a minimum of 8 hours

Start date/time: 2/10/99 15:00
Stop date/time: 2/11/99 0751

1.17 Allow to cool to ambient temperature (1 hour)

1.18 Remove the condenser and replace the original cap on AW101 -AQ-20.
Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 30.7198 g (1.18A)

Determine mass loss due to evaporation

Wt. Lost = 1.18A-1.14A = 0.5277 g (1.18B)

1.19 Label a disposable filter unit (0.45-µm nylon) as AW101 -AQ-30

1.20 Weigh AW101 -AQ-30

Wt. AW101 AQ-30 = 65.4060 g (1.20A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.8178 g (1.20B)

1.21 Connect AW101 -AQ-30 to the vacuum line

Note
Stir bar fell into filter funnel. This was lifted out and rinsed with a small amount of 0.01 M NaOH. The rinse was allowed to collect in the filter funnel and was pulled through with vacuum. Then proceeded with step 1.23.

2/11/99 15:15 1.22 Filter the wash slurry

1.23 Disconnect from the vacuum once the liquid has filtered

1.24 Place the cap on the top of the filter unit and weigh AW101 -AQ-30

Wt. AW101 AQ-30 = 84.2648 g (1.24A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = 59.2792 g (1.24B)

Transfer two 10-mL aliquots of the filtered solution to clean 20-mL HDPE sample vials labeled as AW101 -AQ-30A and AW101 -AQ-30B.

2/11/99

Checked at 12:45 on 2/12/99
No evidence of solids.
M.A.L.

Note: Monitor the solution after ~24 h to determine if any solids form.

1.25 Determine the total weight of the slurry

Wt. Slurry = 1.24A-1.20A = 18.7788 g (1.25A)

Determine the weight of the filtered liquid

Wt. Liquid = 1.24B-1.20B = 17.4614 g (1.25B)

Determine the weight of the filtered solids

Wt. Solids = 1.25A-1.25B = 1.3174 g (1.25C)

1.26 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 20 mL (1.26A)

1.28 Weigh AW101 -AQ-20

Forgot to weigh. M.A.L. 2/11/99

Wt. AW101 -AQ-20 = — g (1.28A)

1.29 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 1.26A ÷ 5); transfer this slurry to AW101 -AQ-20 (→ 4 mL each) Note: solids were much easier to slurry than before (see step 1.12).

1.30 Repeat step 1.29 four times to ensure complete transfer of the solids to AW101 -AQ-20 ////

1.31 Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.7050 g (1.31A)

Determine the weight of the slurry

Wt. Slurry = 1.31A-~~1.28~~(1.11A) A = 19.2270 g (1.31B)

1.32 Equip AW101 -AQ-20 with a condenser, then place in an aluminum heating block at 85°C

1.33 Stir the sample in AW101 -AQ-20 at 85°C for a minimum of 8 hours

Start date/time: 2/11/99 @ 16:00
Stop date/time: 2-12-99 07:42

1.34 Allow to cool to ambient temperature 10 mins

1.35 Remove the condenser and replace the original cap on AW101 -AQ-20. Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.5136 g (1.35A)

M.A.L.
2/12/99

Determine mass loss due to evaporation

Wt. Lost = 1.35A-1.31A = 0.1914 g (1.36B)

1.36 Label a disposable filter unit (0.45-µm nylon) as AW101 -AQ-50

1.37 Weigh AW101 -AQ-50

Wt. AW101 AQ-50 = 65.1844 g (1.37A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.9823 g (1.37B)

1.38 Connect AW101 -AQ-50 to the vacuum line

2/12/99 13:00 1.39 Filter the wash slurry Again washed stir bar with 0.01 M NaOH as at step 1.27. - Some solids stuck to stir bar even after rinsing.

1.40 Disconnect from the vacuum once the liquid has filtered

1.41 Place the cap on the top of the filter unit and weigh AW101 -AQ-50

Wt. AW101 AQ-50 = 85.9188 g (1.41A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = 61.3064 g (1.41B)

Transfer two 10-mL aliquots of the filtered solution to clean 20-mL HDPE sample vials labeled as AW101 -AQ-50A and AW101-AQ-50B.

Pulled sample -50A at 13:40 on 2/12/99, but left remaining solution in AQ-50, so that it could be monitored for solids. (this bottle is clean).

Note: Monitor the solution after ~24 h to determine if any solids form.

2/17/99 10:00 Checked Soln. in AW101-AQ-50

1.42 Determine the total weight of the slurry

Wt. Slurry = 1.41A-1.37A = 20.7344 g (1.42A) Solution clear, no precipitate.

Determine the weight of the filtered liquid

Wt. Liquid = 1.41B-1.37B = 19.5241 g (1.42B) The remainder of this solution was transferred to AW101-AQ-50B.

Determine the weight of the filtered solids

M.J.L. 2/17/99

Wt. Solids = 1.42A-1.42B = 1.2103 g (1.42C)

1.43 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 20 mL (1.43A)

M.J.L. 2/12/99

1.45 Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 12.6000 g (1.45A)

1.46 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 1.43A + 5); transfer this slurry to AW101 -AQ-20 → 4 mL each +++

1.47 Repeat step 1.46 four times to ensure complete transfer of the solids to AW101 -AQ-20

1.48 Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.8450 g (1.48A)

Determine the weight of the slurry

Wt. Slurry = 1.48A-1.45A = 19.2450 g (1.48B)

→ Stopped here on 2/12/99; will resume next week.

✓ 1.49 Equip AW101 -AQ-20 with a condenser, then place in an aluminum heating block at 85°C

1.50 Stir the sample in AW101 -AQ-20 at 85°C for a minimum of 8 hours

Start date/time: 2-16-99 11:13 am
Stop date/time: 2-17-99 07:42 am

1.51 Allow to cool to ambient temperature 2 hrs

1.52 Remove the condenser and replace the original cap on AW101 -AQ-20. Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.6388 g (1.52A)

Determine mass loss due to evaporation

Wt. Lost = 1.52A-1.48A = 0.2062 g (1.52B)

1.53 Label a disposable filter unit (0.45-μm nylon) as AW101 -AQ-70

1.54 Weigh AW101 -AQ-70

Wt. AW101-AQ-70 = 65.2459 g (1.54A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 42.1154 g (1.54B)

1.55 Connect AW101 -AQ-70 to the vacuum line

2/17/99 10:15 1.56 Filter the wash slurry ~~Purge again~~, stir bar rinsed with a few mL 0.01 M NaOH.

1.57 Disconnect from the vacuum once the liquid has filtered

N.A.A. 2/17/99

1.58 Place the cap on the top of the filter unit and weigh AW101 -AQ-70

Wt. AW101 AQ-70 = 85.5126 g (1.58A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = 61.0680 g (1.58B)

Transfer two 10-mL aliquots of the filtered solution to clean 20-mL HDPE sample vials labeled as AW101 -AQ-70A and AW101 -AQ-70B. (Again, left ~4 mL behind in AQ-70 for observation.)

Note: Monitor the solution after ~24 h to determine if any solids form. 2/19/99 8:40 No solids in AW101-AQ-70 → Transferred solution to AW101-AQ-70B N.H.

1.59 Determine the total weight of the slurry

Wt. Slurry = 1.58A-1.54A = 20.2667 g (1.59A)

Determine the weight of the filtered liquid

Wt. Liquid = 1.58B-1.54B = 18.9526 g (1.59B)

Determine the weight of the filtered solids

Wt. Solids = 1.59A-1.59B = 1.3141 g. (1.59C)

1.60 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 20 mL (1.60A)

1.62 Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 12.5915 g (1.62A)

1.63 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 1.60A ÷ 5 = 5 x 4 mL); transfer this slurry to AW101 -AQ-20 ###

1.64 Repeat step 1.63 four times to ensure complete transfer of the solids to AW101 -AQ-20

1.65 Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.9706 g (1.65A)

Determine the weight of the slurry

Wt. Slurry = 1.65A-1.62A = 19.3791 g (1.65B)

1.66 Equip AW101 -AQ-20 with a condenser, then place in an aluminum heating block at 85°C

N.H. 2/17/99

1.67 Stir the sample in AW101 -AQ-20 at 85°C for a minimum of 8 hours

Start date/time: 2/17/99 11:05
Stop date/time: 2-18-99 07:42

1.68 Allow to cool to ambient temperature 1 hr.

1.69 Remove the condenser and replace the original cap on AW101 -AQ-20.
Weigh AW101 -AQ-20

Wt. AW101 -AQ-20 = 31.7471 g (1.69A)

Determine mass loss due to evaporation

Wt. Lost = 1.65A-1.69A = 0.2235 g (1.69B)

1.70 Label a disposable filter unit (0.45-μm nylon) as AW101 -AQ-90

1.71 Weigh AW101 -AQ-90.

Wt. AW101-AQ-90 = 65.2234 g (1.71A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 42.0873 g (1.71B)

1.72 Connect AW101 -AQ-90 to the vacuum line

2/19/99 8:50 1.73 Filter the wash slurry stir bar rinsed with small amount of 0.01 M NaOH, with rinse being passed through the filter into AW101-AQ-90

1.74 Disconnect from the vacuum once the liquid has filtered

1.75 Place the cap on the top of the filter unit and weigh AW101 -AQ-90

Wt. AW101 - AQ-90 = 84.2158 g (1.75A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = 59.9514 g (1.75B)

Transfer two 10-mL aliquots of the filtered solution to clean 20-mL HDPE sample vials labeled as AW101 -AQ-90A and AW101 -AQ-90B. 10 mL into -AQ-90A, left rest in -AQ-90 for observation.

Note: Monitor the solution after ~24 h to determine if any solids form.

2/22/99 9:30 Soln. was clear & transferred to remaining AW101-AQ-90B

1.76 Determine the total weight of the slurry

Wt. Slurry = 1.75A-1.71A = 18.9924 g (1.76A)

M.J.L. 2/19/99

2/19/99 Determine the weight of the filtered liquid

Wt. Liquid = 1.75B-1.71B = 17.8641 g (1.76B)

Determine the weight of the filtered solids

Wt. Solids = 1.76A-1.76B = 1.1283 g (1.76C)

1.77 Label a glass vial as Aw101-AQ-100

1.78 Dry Aw101-AQ-100 at 105°C for a minimum of 1 h 08:45 → 10:48

1.79 Cool Aw101-AQ-100 to ambient temperature in a desiccator

1.80 Weigh Aw101-AQ-100

Wt. Aw101-AQ-100 = 16.8270 g (1.80A)

5 x 4 mL

1.81 Using several portions of deionized water, quantitatively transfer the washed solids from the filter membrane to Aw101-AQ-100 Note: First two aliquots of water were used to rinse vial Aw101-AQ-20, so that all solids could be easily recovered. Note: There were a few particles that seemed to float on the water; these were clearly observed.

1.82 Weigh Aw101-AQ-100

Wt. Aw101-AQ-100 = 35.7611 g (1.82A)

1.82 Heat Aw101-AQ-100 at 80°C to evaporate excess water

1.83 Heat Aw101-AQ-100 at 105°C overnight 2-23-99 removed from overnight heat.

1.84 Cool Aw101-AQ-100 to ambient temperature in a desiccator

1.85 Weigh Aw101-AQ-100

Wt. Aw101-AQ-100 = 16.8847 g[a] (1.85A)

1.86 Determine the dry weight of the washed solids

Wt. Dry Solids = 1.85A-1.82A = 0.05770 g (1.86A)

1.87 The washed solids are to be submitted for analysis. The cognizant scientist will prepare the required ASR.

M.L.H. 2/23/99

[a] A reweigh on this gave 16.8841 g

Note: Pages 13 through 19 deal with the caustic leaching test. Per the direction of BNFL, the caustic leaching was not performed. M.L. Hewitt 8/16/99

Appendix B. Raw Data

Analytical Chemistry Laboratory (ACL) Analytical Services Request (ASR)

(Cover Page ... information applicable to all samples in series)

Requested By: Gregg J. Lumetta (Print Name) | Gregg J. Lumetta 2/18/99 (Signature/Date) | 376-6911 (Phone) | P7-25 (MSIN)

Requester - Please Complete All Fields In This Section, Unless Specified "Optional" or ASR is a Revision

Request ID (optional): _____

PNL Project Number (if known): 29953

Work Order/Pkg.: (a)

Cost Estimate ($): _____

Protocol Requirement: X None __RCRA __CERCLA, or

Other (specify): _____

Hold Time Requirement: X None __RCRA __CERCLA, or

Other (specify): _____

TPA Support: X No, or

Milestone No.: _____

QA Plan: X MCS-033, or

Other ACL QA Plan (specify): _____

Additional QA Requirements: __No, or

Reference Doc.: _____

ACL COC Req'd (PNL-ALO-010): __No X Yes

Sample Storage Requirements: X No __Refrigerate, or

Other (specify): _____

Date Sampled (optional): _____

Time Sampled (optional): _____

Matrix: X Samples vary (specify on Request Page), or

Liquid: __Aqueous __Organic __Multi-phasic

Solid: __Soil __Sludge __Sediment __Glass

__Filter __Smear __Metal __Organic __Other Solids

Solid/Liquid Mixture: __ Gas: __

Biological: __Tissue __Urine __Feces

Process Knowledge: ✓ Sample Information Check List, or

Reference Doc.: _____

PCBs Present: __No __Yes

Sample Disposition ...

Untreated Sample(s): __Return __Dispose X Store, or

Reference Doc.: _____

Prep'd Sample(s): __Dispose __Return X Store, or

Reference Doc.: _____

Additional Instructions: X No, or

Reference Doc.: _____

Date Report Req'd: (b)

Send Report to: G.J. Lumetta

MSIN: P7-25 Phone: _____

Fax (optional): _____

For ACL Use Only ... Do Not Complete This Section

Date Delivered: _____

Time Delivered (optional): _____

Deliv. By (if known): _____

Received By: _____

Resp. ACL Mgr.: _____

Signature/Date: _____

Job Group (optional): _____

Sample Group (optional): _____

PNL Impact Level: __1 __2 __3

DQ Review Req'd: __No __Yes ACL Waste: __No __Yes

ASR Number: _____ Revision: __Yes

ACL Numbers: _____

(a) W48482 to samples AW101-50L-30A1 → AW101-50L-50A2
W48481 for all others

(b) ICP/AES data needed by 3/5/99
All others 4/2/99.

AW101 Samples

Sample ID	Description	Acid Digestion	KOH Fusion	Na_2O_2 Fusion	ICP/AES	IC (anions)	TOC	TIC	ICP-MS (^{99}Tc)	GEA	^{90}Sr	Total Alpha	Laser Fluorimetry (U)	
AW101-SOL-30A1	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-SOL-30A2	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-SOL-40A1	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-SOL-40A2	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-SOL-50A1	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-SOL-50A2	AW101 Liquid	X			X	X	X	X	X	X	X	X	X	
AW101-AQ-30A	AW101 Wash Solution	X			X	X	X	X	X	X	X	X	X	
~~AW101-AQ-30B~~	~~AW101 Wash Solution~~	~~X~~			~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	(a)
AW101-AQ-50A	AW101 Wash Solution	X			X	X	X	X	X	X	X	X	X	
~~AW101-AQ-50B~~	~~AW101 Wash Solution~~	~~X~~			~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	(a)
AW101-AQ-70A	AW101 Wash Solution	X			X	X	X	X	X	X	X	X	X	
~~AW101-AQ-70B~~	~~AW101 Wash Solution~~	~~X~~			~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	(a)
AW101-AQ-90A	AW101 Wash Solution	X			X	X	X	X	X	X	X	X	X	
~~AW101-AQ-90B~~	~~AW101 Wash Solution~~	~~X~~			~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	~~X~~	(a)
AW101-AQ-100	Washed AW101 Solids		X	X	X	X	X	X	X	X	X	X	X	

(a) Archive these samples for now, per instructions from Paul Townson on 2/18/99

M.J.L. 2/19/99

ASR 5275 Addendum

Gregg Lumetta
March 16, 1999

Sample AW101-AQ-100 has been dissolved in acid for analysis. The solution with the dissolved solids is labeled as **AW101-AQ-100D**. The sample matrix is 0.1M HCl.

AW101-AQ-100D needs to be subjected to the following analyses:

ICP
ICP-MS (^{99}Tc)
GEA
^{90}Sr
Total alpha
Laser fluorimetry (U)

Lumetta, Gregg J
From: MJohnson@bnflinc.com
Sent: Monday, March 01, 1999 7:45 AM
To: Lumetta, Gregg J; PTownson@bnflinc.com
Subject: Fwd[2]:AW-101 Testing

Gregg - I concur with your recommendation for analyzing the ~0.058 grams of AW-101 solids remaining after washing.
Please proceed with dissolution of the solids in acid and conducting analyses for metals and radionuclides per Table 6-1 from BNFL letter 000749 (Revision to Ultrafiltration / Solids Dissolution Test Specification. I understand there is insufficient solids to accomplish these analyses in duplicate. I also understand there is insufficient solids conduct the requested anion analyses.

Michael Johnson

_____Forward Header_____
Subject: AW-101 Testing
Author: "Lumetta Gregg J" <gregg.lumetta@pnl.gov>
Date: 2/23/99 2:23 PM

Paul:
This message is to update you on progress made and to seek your counsel on a technical issue we've encountered.
First, the solubility versus temperature test with the AW-101 LAW sample was completed. This test proceeded according to plan (test plan BNFL-TP-29953-7) with no problems encountered. The samples from this test have been submitted for analysis.
Second, the washing test with the AW-101 LAW entrained solids was also completed. Again, the test when according to plan (test plan BNFL-TP-29953-9). Samples of the washing solutions have been submitted for analysis.
However, here's where the problem comes in. There is only a small amount (0.058 g) of residual solids remaining. With this small amount of material, we cannot perform all the analyses originally planned, much less do them in duplicate. So the question is: How should we proceed with analyzing the solids?
My recommendation would be to dissolve the solids in acid (HCl with perhaps HNO3 or HF added, as needed). The resulting solution could then be analyzed, but we could not analyze for the following constituents: TOC, TIC, Cl, F, NO3, SO4, PO4 (although we would get the total P concentration by ICP).
I'll await your advice on this matter.

Gregg

RFC822.TXT

Protocol for Dissolving Sample AW101-AQ-100

Purpose

The quantity of residual solids from the AW-101 LAW entrained solids washing test was such that the full suite of analyses requested by BNFL could not be completed. There was not enough material to do the KOH and Na_2O_2 fusions. The purpose of this protocol is to dissolve the residual AW-101 solids for ICP/AES and radiochemical analysis.

3/4/99

Instructions

3/5/99

1. Heat AW101-AQ-100 at 105°C for 1 h start 11:19 am - 12:45 pm
2. Cool AW101-AQ-100 in a desiccator, then weigh

   Wt. AW101-AQ-100 = 16.8796 g (2a)

3. Add 5 mL of concentrated (12 M) HCl (Ultrex-grade) to AW101-AQ-100
4. Place the cap loosely on AW101-AQ-100 and heat in the aluminum heating block at 90°C. start 0938 1100 1045

   Occasionally, tighten the cap and swirl the vial to contact the acid with the solids on the wall of the vial. Loosen the cap and continue to heat.

5. Continue to heat until all solids dissolved. If solids are not dissolved after 1 hour, consult with GJ Lumetta.

   After ~1.5 h most of the solids had dissolved, but there was still some. Added 1 mL of conc. HNO_3 (at 11:00). Repeat step 4. After 1.75 h, a white solid had formed. This solid tended to collect around the threads of the cap. At this point, we proceeded with step 6 to evaporate the acid.

6. Once all solids dissolved, remove the cap from AW101-AQ-100 and evaporate to dryness at 90°C. 1604 3-5-99 done.
7. Add 4 mL of 0.1 M HCl to AW101-AQ-100 to dissolve the sample.
8. Transfer the solution to a 25-mL volumetric flask.
9. Add another 4 mL of 0.1 M HCl to AW101-AQ-100 to rinse the vial; transfer this rinse liquid to the volumetric flask.
10. Repeat step 8 three times for a total of 4 rinses of AW101-AQ-100. Note: Do not discard vial AW101-AQ-100.

Steps 7 - 10 revised as on next page. M.A. Lumetta 3/12/99

Note: Take measures to ensure bottle AW101-AQ-100C is free of external removable contamination when removed from the hot cell.

✓ 7. Use five 5-mL aliquots of 0.1 M HCl to quantitatively transfer the material from AW101-AQ-100 to AW101-AQ-100C (a 30-mL HDPE vial). HH 3-12-99

✓ 8. Remove AW101-AQ-100C from the hot cell.

✓ 9. Provided the dose rate (at 6") is less than 3000 mrem/h, transfer AW101-AQ-100C to lab 511. → 1725 mr

✓ 10. Heat AW101-AQ-100 at 105°C for 1 h start 1:30 pm 3-12-99 stop 3:30 pm "

✓ 11. Cool AW101-AQ-100 in a desiccator, then weigh

Wt. AW101-AQ-100 = 16,7787 g (15a)

✓ 12. Determine the weight of the solids

Wt. solids = 2a – 15a = 0.10090 g (15b)

3/12/99 M.J.L. 13. 1.0 mL of 10 M HF was added to AW101-AQ-100C (15:00). Mixture was stirred at ambient temperature.

3/15/99 M.J.L. 14. Solids were still present. Heated at ~80°C with lid off to evaporate down. 8:00 → 16:00 Had evaporated to ~2 mL Stopped heating.

3/16/99 M.J.L. 15. Added 5 mL 0.1 M HCl at 8:50. Heated at ~70°C until 12:15. Allowed to cool.

16. Filtered through a 0.45-µm nylon membrane. Flask AW101-AQ-100C was rinsed with several 2 mL aliquots of 0.1 M HCl with the rinse liquid being used to ring rinse the filter.

17. Filtered solution was diluted to 25 mL in a vol. flask → Sample (AW101-AQ-100D)

18. The nylon filter membrane with the filtered solids was placed in a glass vial labeled at (AW101-AQ-100E) and was allowed to air dry.

M.J. Lumetta 3/16/99

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

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	ICP/EQL @5 (ug/mL)	ICP/EQL @12.5 (ug/mL)	ICP/EQL @50 (ug/mL)	Filtrate, Wash Solutions MRQ (ug/mL)	12.5 99-1151-PB @1 Process Blank 3/15/99 (ug/mL)	50.0 99-1151 @2 AW101-SOL-30A1 3/15/99 (ug/mL)	50.0 99-1152 @2 AW101-SOL-30A2 3/15/99 (ug/mL)
0.015	Ag	0.8	1.9	7.5		--	[0.81]	[0.77]
0.060	Al	3.0	7.5	30.0	75.0	[7.3]	17,600	18,300
0.080	As	4.0	10.0	40.0		--	[12]	[12]
0.050	B	2.5	6.3	25.0		112	95.9	71.2
0.010	Ba	0.5	1.3	5.0	78.0	--	--	--
0.010	Be	0.5	1.3	5.0		--	[1.5]	[1.6]
0.100	Bi	5.0	12.5	50.0		--	--	--
0.100	Ca	5.0	12.5	50.0	150.0	--	[9.3]	[11]
0.015	Cd	0.8	1.9	7.5	75.0	--	[2.1]	[2.0]
0.100	Ce	5.0	12.5	50.0		--	--	--
0.025	Co	1.3	3.1	12.5	30.0	--	--	--
0.020	Cr	1.0	2.5	10.0	15.0	--	62.9	65.0
0.015	Cu	0.8	1.9	7.5	17.0	--	[1.6]	[1.6]
0.050	Dy	2.5	6.3	25.0		--	--	--
0.100	Eu	5.0	12.5	50.0		--	--	--
0.025	Fe	1.3	3.1	12.5	150.0	[0.39]	[3.5]	[3.5]
2.000	K	100.0	250.0	1000.0	75.0	--	24,400	25,600
0.025	La	1.3	3.1	12.5	35.0	--	--	--
0.005	Li	0.3	0.6	2.5		--	[0.57]	[0.44]
0.100	Mg	5.0	12.5	50.0	150.0	--	--	--
0.005	Mn	0.3	0.6	2.5	150.0	--	--	--
0.030	Mo	1.5	3.8	15.0	90.0	--	--	--
0.100	Na	5.0	12.5	50.0	75.0	117	143,000	145,000
0.100	Nd	5.0	12.5	50.0		--	--	--
0.030	Ni	1.5	3.8	15.0	30.0	--	[5.1]	[5.3]
0.100	P	5.0	12.5	50.0		--	344	358
0.060	Pb	3.0	7.5	30.0	300.0	--	38.9	42.7
0.300	Pd	15.0	37.5	150.0		--	--	--
0.300	Rh	15.0	37.5	150.0		--	--	--
0.075	Ru	3.8	9.4	37.5		--	[5.2]	[5.1]
0.050	Sb	2.5	6.3	25.0		--	--	--
0.050	Se	2.5	6.3	25.0		--	[5.7]	[5.3]
0.100	Si	5.0	12.5	50.0	170.0	119	264	202
1.000	Sn	50.0	125.0	500.0		--	[84]	[86]
0.005	Sr	0.3	0.6	2.5		--	--	--
0.500	Te	25.0	62.5	250.0		--	--	--
0.800	Th	40.0	100.0	400.0		--	--	--
0.005	Ti	0.3	0.6	2.5	17.0	[0.092]	--	--
0.250	Tl	12.5	31.3	125.0		--	--	--
2.000	U	100.0	250.0	1000.0	600.0	--	--	--
0.015	V	0.8	1.9	7.5		--	--	--
0.500	W	25.0	62.5	250.0		--	[74]	[76]
0.010	Y	0.5	1.3	5.0		--	--	--
0.020	Zn	1.0	2.5	10.0	16.5	[0.32]	[6.3]	[6.7]
0.025	Zr	1.3	3.1	12.5		--	[6.7]	[6.8]

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

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Analyte	Multiplier=	50.0	50.0	50.0	50.0	12.5
		ALO#=	99-1153 @2	99-1154 @2	99-1155 @2	99-1156 @2	99-1157 @1
		Client ID=	AW101-SOL-40A1	AW101-SOL-40A2	AW101-SOL-50A1	AW101-SOL-50A2	AW101-AQ-30A
		Run Date=	3/15/99	3/15/99	3/15/99	3/15/99	3/15/99
		(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.015	Ag		[0.84]	[0.81]	[0.84]	[0.82]	--
0.060	Al		18,600	18,600	19,200	18,700	1,080
0.080	As		[12]	[11]	[12]	[11]	--
0.050	B		98.0	89.8	89.2	94.7	31.3
0.010	Ba		--	--	--	--	--
0.010	Be		[1.6]	[1.6]	[1.7]	[1.6]	--
0.100	Bi		--	--	--	--	--
0.100	Ca		[11]	[11]	[12]	[10]	--
0.015	Cd		[2.1]	[2.0]	[2.1]	[2.0]	--
0.100	Ce		--	--	--	--	--
0.025	Co		--	--	--	--	--
0.020	Cr		67.5	67.4	70.6	68.8	64.4
0.015	Cu		[1.6]	[1.5]	[1.2]	[1.5]	--
0.050	Dy		--	--	--	--	--
0.100	Eu		--	--	--	--	--
0.025	Fe		[4.4]	[4.0]	[4.4]	[4.2]	--
2.000	K		26,000	26,000	26,700	26,000	1,590
0.025	La		--	--	--	--	--
0.005	Li		[0.51]	[0.39]	[0.48]	[0.40]	--
0.100	Mg		--	--	--	--	--
0.005	Mn		--	--	--	--	--
0.030	Mo		--	--	--	--	--
0.100	Na		146,000	146,000	147,000	146,000	12,000
0.100	Nd		--	--	--	--	--
0.030	Ni		[5.3]	[5.2]	[5.5]	[5.5]	[1.4]
0.100	P		361	357	372	364	[12]
0.060	Pb		48.6	40.0	40.7	42.0	[3.3]
0.300	Pd		--	--	--	--	--
0.300	Rh		--	--	--	--	--
0.075	Ru		[5.1]	[4.9]	[5.0]	[4.9]	--
0.050	Sb		--	--	--	--	--
0.050	Se		[5.5]	[5.5]	[4.7]	[5.0]	--
0.100	Si		269	274	248	269	79.8
1.000	Sn		[87]	[86]	[87]	[87]	--
0.005	Sr		--	--	--	--	--
0.500	Te		--	--	--	--	--
0.800	Th		--	--	--	--	--
0.005	Ti		--	--	--	--	--
0.250	Tl		--	--	--	--	--
2.000	U		--	--	--	--	--
0.015	V		--	--	--	--	--
0.500	W		[77]	[77]	[79]	[77]	--
0.010	Y		--	--	--	--	--
0.020	Zn		[6.6]	[6.6]	[7.0]	[6.7]	--
0.025	Zr		[7.0]	[7.0]	[7.2]	[7.1]	--

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

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= (Analyte)	12.5 / 99-1157-DUP @1 / AW101-AQ-30A-DUP / 3/15/99 (ug/mL)	5.0 / 99-1158 @1 / AW101-AQ-50A / 3/15/99 (ug/mL)	5.0 / 99-1159 @1 / AW101-AQ-70A / 3/15/99 (ug/mL)	5.0 / 99-1160 @1 / AW101-AQ-90A / 3/15/99 (ug/mL)
0.015	Ag	--	--	--	--
0.060	Al	1,100	89.6	49.6	38.5
0.080	As	--	--	--	--
0.050	B	29.8	27.5	27.4	27.5
0.010	Ba	--	[0.094]	[0.066]	[0.15]
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.100	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.100	Ce	--	--	--	--
0.025	Co	--	--	--	--
0.020	Cr	65.6	5.36	1.72	[0.47]
0.015	Cu	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	--	[0.38]	[0.65]	[0.67]
2.000	K	1,640	[58]	--	--
0.025	La	--	--	--	--
0.005	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.005	Mn	--	[0.093]	0.428	0.382
0.030	Mo	--	--	--	--
0.100	Na	12,300	783	283	243
0.100	Nd	--	--	--	--
0.030	Ni	[1.4]	--	--	--
0.100	P	[12]	[1.2]	[0.57]	--
0.060	Pb	--	[0.36]	--	--
0.300	Pd	--	--	--	--
0.300	Rh	--	--	--	--
0.075	Ru	--	--	--	--
0.050	Sb	--	--	--	--
0.050	Se	--	--	--	--
0.100	Si	68.5	62.9	72.4	61.2
1.000	Sn	--	--	--	--
0.005	Sr	--	--	--	--
0.500	Te	--	--	--	--
0.800	Th	--	--	--	--
0.005	Ti	--	--	[0.031]	[0.032]
0.250	Tl	--	--	--	--
2.000	U	--	--	--	--
0.015	V	--	--	--	--
0.500	W	--	--	--	--
0.010	Y	--	--	--	--
0.020	Zn	--	--	--	[0.25]
0.025	Zr	--	--	[0.27]	[0.25]

TCF-NE SANDERS 3-15-99

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

EXHIBIT I
PAGE 1 of 1

PNL-ALO-128 Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: Lumetta
Work Auth. Doc (WAD): ASR # 5275
Tank/Core/Project:
Special Instructions: for ASR 5275 states "No matrix spikes, LCS or blank spikes"

Work package number: Multiple
Project number: 29953
PNL QA plan:
PNL Impact level:
Prep. lab (SAL/SRPL/other): SRPL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Vial Identifier	Sample Volume (ml)	Spike added Volume (ml)	Spike added Weight (g)	Final solution Volume (ml)	Process Factor (1)	
1	PB-1151	—		2mL H2O	NA	NA	25mL		
2	99-1151	AW101-SOL-30A1		1mL					
3	99-1152	AW101-SOL-30A2							
4	99-1153	AW101-SOL-40A1							
5	99-1154	AW101-SOL-40A2							
6	99-1155	AW101-SOL-50A1							
7	99-1156	AW101-SOL-50A2							
8	99-1157	AW101-AQ-30A		2mL					
9	99-1157Dup	AW101-AQ-30A							
10	99-1158	AW101-AQ-50A		5mL					
11	99-1159	AW101-AQ-70A							
12	99-1160	AW101-AQ-90A							
13									
14									

Analyst's sample preparation comments: Pipet checks: #112498 #1.057DN @ 5mL
Balance # 360-66-01-026

	1.0013g	4.9833g
	1.0016g	4.9782g
	1.0017g	5.0012g
	1.0001g	4.9951g
	0.9971g	5.0020g

Spike source:
PNL spike ID number:
Anal. balance M&TE:
Sample filtered (yes/no):

(1) Process factor = Final volume (ml) /Sample volume (ml)

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: R.K. Berry 5/4/99 Reviewer/Date:

Rev. 2.0 7-28-95 JMR

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: G. J. Lumetta

ACL Number(s): 99-1161, 99-1458-Zr & 99-1458-Ni

Client ID: "AW101-AQ-100d", "AN107-AQ-100"(Zr) & "AN107-AQ-100"(Ni)

ASR Number: 5275 & 5319

Total Samples: 3

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

Analyst: J. J. Wagner

Analysis Date (Filename): 4-22-99 (A0524) and 4-27-99 (A0525)

See system file: "ICP-325-405-1" for traceability to Calibration, Quality Control, Verification, and Raw Data.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

J. J. Wagner 5-4-99
Reviewed by

M. D. Thomas 5-4-99
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

One radioactive solid sample AN107-AQ-100 (ASR 5319) was analyzed by ICPAES after preparation by the 325 Shielded Analytical Laboratory (SAL) using two fusion preparation procedures: PNNL-ALO-114 Na2O2-NaOH/Zr and PNNL-ALO-115 KOH/Ni. Approximately 0.05 to 0.06-gram aliquots were used for each procedure. After samples were fused they were diluted to a final volume of 50 ml. Additional dilution, up to 10 fold, was performed during ICPAES analysis. All measurement results reported have been corrected for preparation and analytical dilution. Because of limited sample material, duplicates were not prepared. Analytes of interest include Ag, Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Ti, U, Zn and Zr.

Sample AW101-AQ-100d (ASR 5275) was prepared by the client and analyzed by ICPAES without further processing other than necessary analytical dilution up to 5-fold. Analytes of interest are the same as those listed above. Measurement results have been corrected for analytical dilution only. Results are reported as µg/ml as agreed upon by the client.

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Solid samples)	All results are within tolerance limit of ≤ 10% after correcting for dilution.
(Aqueous samples)	All results were within tolerance limit of ≤ 10% after correcting for dilution except Mg in sample AW101-AQ-100d @5 and AW101-AQ-100d @1. Mg concentration was recovered within 13% after dilution correction. All other analytes of interest in the above sample were within 4% after dilution correction.

Duplicate RPD (Relative Percent Difference):

(Solid samples)	No duplicates were prepared because of limited sample material.
(Aqueous samples)	No duplicates were provided.

Post-Spiked Samples (Group A):

(Solid samples)	All analytes of interest were recovered within tolerance of 75 to 125%.
(Aqueous samples)	All analytes of interest were recovered within tolerance of 75 to 125%.

5/4/99

Post-Spiked Samples (Group B):

(Solid samples) All analytes of interest were recovered within tolerance of 75 to 125%.

(Aqueous samples) All analytes of interest were recovered within tolerance of 75 to 125%.

Blank Spike:

(Solid samples) A blank spike was not prepared.

(Aqueous samples) A blank spike was not provided.

Matrix Spiked Sample:

(Solid samples) A matrix spike was not prepared.

(Aqueous samples) A matrix spike was not provided.

Quality Control Check Standards:

Concentration of all analytes of interest, except for Si, was recovered within tolerance of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV. Calibration Blank (ICP98.0) concentration was less than two times IDL. Silicon was slightly high (about 14%) in one determination of QC_SSTMCV. Silicon in QC_MCVA check standard was within 5% of the true value of 20 µg/ml, which was run several times during the analysis, thus, measurement results for Silicon in the samples are not likely to be affected.

High Calibration Standard Check:

Verification of the high-end calibration concentration for all analytes of interest was within tolerance of ± 5% accuracy except for Ca, Fe, and U. These three analytes were slightly high, between 6% and 7%, in the high-end cal. check standard. Measurement results for these analytes in the samples were closer to mid-range concentrations like those found in QC_MCVA. Therefore, sample measurement results are not likely to be affected by the slightly high recovery for Ca, Fe, and U.

Process Blank:

(Solid samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except Ca in ALO-114 prepared samples and Na in ALO-115 prepared samples. Concentration of Ca in the process blank for sample AN107-AQ-100 (Zr) was about 52% of that in the sample. Concentration of Na in the process blank for sample AN107-AQ-100 (Ni) was about 12% of that in the sample.

(Aqueous samples) No preparation blank provided.

Laboratory Control Standard:

(Solid samples) All analytes of interest at a concentration equal to or greater than EQL were recovered within tolerance of 75% to 125% in both fusion prepared LCS standards. SRM-2710 Montana Soil was used for the LCS in both ALO-114 and ALO-115 fusion preparations.

(Aqueous samples) No LCS provided.

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

Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	814.3 / 99-1458-Zr-PB / AN107-AQ-100 / 4/22/99 / ug/g	1900.1 / 99-1458-Zr @ 2.3333 / AN107-AQ-100 / 4/22/99 / ug/g		
0.015	Ag	[12]	[52]	--	--
0.060	Al	[200]	18,800	--	--
0.080	As	--	[200]	--	--
0.050	B	[49]	--	--	--
0.010	Ba	[9.0]	1,410	--	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.100	Ca	3,540	6,820	--	--
0.015	Cd	--	[69]	--	--
0.100	Ce	[150]	5,820	--	--
0.025	Co	--	--	--	--
0.020	Cr	[35]	9,160	--	--
0.015	Cu	[28]	[94]	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	366	245,000	--	--
2.000	K	[2,000]	--	--	--
0.025	La	[30]	1,940	--	--
0.005	Li	[29]	[57]	--	--
0.100	Mg	--	[1,200]	--	--
0.005	Mn	[7.3]	143,000	--	--
0.030	Mo	--	--	--	--
0.100	Nd	[100]	6,710	--	--
0.030	Ni	439	749	--	--
0.100	P	[96]	[1,300]	--	--
0.060	Pb	[85]	18,700	--	--
0.300	Pd	--	[3,300]	--	--
0.300	Rh	--	--	--	--
0.075	Ru	--	[450]	--	--
0.050	Sb	[96]	[210]	--	--
0.050	Se	[76]	[350]	--	--
0.100	Si	[380]	6,930	--	--
1.000	Sn	--	[3,500]	--	--
0.005	Sr	56.0	220	--	--
0.500	Te	--	--	--	--
0.800	Th	--	--	--	--
0.005	Tl	[10]	185	--	--
0.250	Ti	--	--	--	--
2.000	U	--	[4,000]	--	--
0.015	V	[13]	[45]	--	--
0.500	W	--	--	--	--
0.010	Y	--	811	--	--
0.020	Zn	--	1,670	--	--

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=	994.0	4970.2					
	ALO#=	99-1458-NI-PB	99-1458-Ni @5					
	Client ID=	AN107-AQ-100	AN107-AQ-100					
Det. Limit	Run Date=	4/22/99	4/22/99					
(ug/mL)	(Analyte)	ug/g	ug/g					
0.015	Ag	[15]	[90]		--		--	
0.060	Al	[320]	20,000		--		--	
0.080	As	[100]	--		--		--	
0.050	B	[200]	[400]		--		--	
0.010	Ba	--	1,470		--		--	
0.010	Be	--	--		--		--	
0.100	Bi	--	--		--		--	
0.100	Ca	[150]	[2,200]		--		--	
0.015	Cd	--	[84]		--		--	
0.100	Ce	--	[4,300]		--		--	
0.025	Co	[30]	--		--		--	
0.020	Cr	[22]	10,100		--		--	
0.015	Cu	[24]	[100]		--		--	
0.050	Dy	--	--		--		--	
0.100	Eu	--	--		--		--	
0.025	Fe	388	273,000		--		--	
0.025	La	[25]	1,990		--		--	
0.005	Li	[15]	[68]		--		--	
0.100	Mg	[100]	[740]		--		--	
0.005	Mn	191	162,000		--		--	
0.030	Mo	--	--		--		--	
0.100	Na	3,820	26,500		--		--	
0.100	Nd	[100]	6,540		--		--	
0.100	P	[120]	[2,000]		--		--	
0.060	Pb	[110]	15,900		--		--	
0.300	Pd	--	[3,300]		--		--	
0.300	Rh	--	--		--		--	
0.075	Ru	--	[530]		--		--	
0.050	Sb	[82]	[330]		--		--	
0.050	Se	[110]	[570]		--		--	
0.100	Si	1,640	7,470		--		--	
1.000	Sn	--	--		--		--	
0.005	Sr	--	[170]		--		--	
0.500	Te	--	--		--		--	
0.800	Th	--	--		--		--	
0.005	Ti	[14]	[210]		--		--	
0.250	Tl	--	--		--		--	
2.000	U	--	--		--		--	
0.015	V	--	--		--		--	
0.500	W	--	--		--		--	
0.010	Y	--	652		--		--	
0.020	Zn	[39]	1,770		--		--	
0.025	Zr	--	3,190		--		--	

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

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

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

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

Det. Limit (ug/mL)	Analyte	Multiplier= 1.0 ALO#= 99-1161 @1 Client ID= AW101-AQ-100d Run Date= 4/22/99 (ug/mL)			
0.015	Ag	0.881	--	--	--
0.060	Al	67.3	--	--	--
0.080	As	[0.12]	--	--	--
0.050	B	34.3	--	--	--
0.010	Ba	6.47	--	--	--
0.010	Be	[0.057]	--	--	--
0.100	Bi	2.61	--	--	--
0.100	Ca	53.2	--	--	--
0.015	Cd	2.62	--	--	--
0.100	Ce	[0.46]	--	--	--
0.025	Co	0.366	--	--	--
0.020	Cr	80.7	--	--	--
0.015	Cu	1.30	--	--	--
0.050	Dy	[0.11]	--	--	--
0.100	Eu	[0.15]	--	--	--
0.025	Fe	113	--	--	--
2.000	K	[5.0]	--	--	--
0.025	La	0.257	--	--	--
0.005	Li	0.098	--	--	--
0.100	Mg	4.80	--	--	--
0.005	Mn	105	--	--	--
0.030	Mo	--	--	--	--
0.100	Na	131	--	--	--
0.100	Nd	[0.46]	--	--	--
0.030	Ni	16.5	--	--	--
0.100	P	4.72	--	--	--
0.060	Pb	8.60	--	--	--
0.300	Pd	--	--	--	--
0.300	Rh	--	--	--	--
0.075	Ru	[0.34]	--	--	--
0.050	Sb	[0.090]	--	--	--
0.050	Se	[0.23]	--	--	--
0.100	Si	398	--	--	--
1.000	Sn	[3.5]	--	--	--
0.005	Sr	0.734	--	--	--
0.500	Te	--	--	--	--
0.800	Th	[4.5]	--	--	--
0.005	Tl	0.766	--	--	--
0.250	Tl	--	--	--	--
2.000	U	428	--	--	--
0.015	V	[0.058]	--	--	--
0.500	W	--	--	--	--
0.010	Y	[0.021]	--	--	--
0.020	Zn	15.5	--	--	--
0.025	Zr	17.3	--	--	--

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 --- IC Report

WO/Project: W48481&W48482/29953
Client: G. Lumetta

ACL Nmbr(s): 99-1151 through 99-1160

Client ID: AW101 SOL and AW101 AQ series

ASR Nmbr 5275

Total Samples: 10 liquids

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography" (IC).

Analyst: MJ Steele

Analysis Date: March 30-31, 1999 and Reruns April 12-13, 1999

See Chemical Measurement Center 98620: IC File for Calibration and Maintenance Records.

M&TE Number: IC instrument -- WD25214
Mettler AT400 Balance – Cal. No. 360-06-01-031

Analyst: MJ Steele 4/27/99

Approval: [signature] 4/27/99

Final Results:

Ten liquid samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5275. The liquid samples were diluted 5-fold to 12.25-fold during the preparation of the samples prior to distribution to the IC workstation, and were diluted at the IC workstation up to 200-fold to ensure that all anions were within the calibration range. The samples were initially analyzed on March 30-31, 1999. From this run, the verification standards for many analytes were below the 90% recovery acceptance criteria. Therefore the samples were reanalyzed on April 12-13, 1999. Only results from the final analysis run are provided in this report. The results from the initial analysis run are included in the data package for information only.

Based on client communications the nitrate result for AW101-SOL-40A2 appears to be about a factor of two higher than expected. The only other analyte in this sample at a high enough concentration to provide reliable results nitrite, and the nitrite for sample AW101-SOL-40A2 is only slightly higher than sample AW101-SOL-40A1. To provide sufficient sample for all analyses requested, AW101-SOL samples had to be diluted 10-12 fold; it is possible that the small volume sample was contaminated during the initial dilution. Both the initial run (which failed QC) and the final run measured nitrate above 200,000 µg/ml.

The results for the samples from the April 12-13, 1999 run are presented in the table below.

SAMPLE ID	Client ID	Dil Factor	F µg/ml	Cl µg/ml	NO2 µg/ml	Br µg/ml	NO3 µg/ml	PO4 µg/ml	SO4 µg/ml	C204 µg/ml
99-1151 PB	PROCESS BLANK	5	<1.4	<1.4	<2.8	<1.4	<2.8	<2.8	<2.8	<2.8
	% RECOVERY		91	97	100	97	96	94	95	98
99-1151	AW101-SOL-30A1	12.25	1,300	3,600	69,400	<600	118,000	<1200	<1200	<1200
99-1152	AW101-SOL-30A2	10	1,300	4,100	62,300	<500	120,000	<1000	<1000	<1000
	% RECOVERY		91	115	110	105	111	100	103	107
99-1153 #1	AW101-SOL-40A1	12.25	1,600	4,400	75,300	<600	122,000	<1200	<1200	<1200
99-1153 #1 REPLICATE	AW101-SOL-40A1	12.25	1,400	5,000	80,000	<300	154,000	<613	1,752	<613
	RPD (%)		15	14	6	n/a	24	n/a	n/a	n/a
99-1153 #2	AW101-SOL-40A1	12.25	1,100	3,000	65,400	<300	124,000	1,400	1,400	<600
99-1153 #2 REPLICATE	AW101-SOL-40A1	12.25	1,200	3,800	64,600	<300	125,000	1,400	1,600	<600
	RPD (%)		6	22	1	n/a	0	4	11	n/a
99-1154	AW101-SOL-40A2	12.25	1,600	4,200	72,700	<600	227,000	<1200	<1200	<1200
99-1155	AW101-SOL-50A1	10	1,600	4,100	65,200	<500	126,000	<1000	<1000	<1000
99-1156	AW101-SOL-50A2	10	1,500	4,100	63,500	<500	122,000	<1000	<1000	<1000
99-1157	AW101-AQ-30A	5	100	250	3,800	<25	7,300	<50	120	6,400
99-1158	AW101-AQ-50A	5	6.0	11	170	<1.4	360	<2.8	6.5	210
99-1159	AW101-AQ-70A	5	<1.4	3.0	8.0	<1.4	34	<2.8	<2.8	<2.8
99-1160	AW101-AQ-90A	5	<1.4	2.5	<2.8	<1.4	2.7	<2.8	<2.8	<2.8
	% RECOVERY		98	110	108	107	103	101	104	86

[1] RPD = Relative Percent Difference (between sample and duplicate)

Q.C. Comments:

Following are results of quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan, MCS-033.

Working Blank Spike/Process Blank Spike: Process Blank Spike recoveries ranged from 91% to 100%, well within the acceptance criteria of 75% to 125%.

Matrix Spiked Sample: The matrix spike recovery for samples AW101-SOL-30A2 and AW101-AQ-90A ranged from 86% to 115%. Again, this is well within the acceptance criteria of 75% to 125%.

Duplicate: No duplicates were provided. However, the laboratory-dilution of sample AW101-SOL-40A1 was analyzed in replicate (i.e., two different analysis injections) at the IC workstation from two different IC workstation dilutions. Two replicate analyses failed the acceptance criteria of a Relative Percent Difference less than 20%; nitrate on IC dilution #1 and chloride on IC dilution #2. Based on QC performance of matrix spikes and verification standards, no explanation can be offered for the poor precision on the one nitrate from IC dilution #1. However, there are significant interference peaks between the fluoride and nitrite retention times than can account for the poor precision of the chloride results, since chloride peak baselines are difficult to establish.

System Blank/Processing Blanks: No anions were detected above reportable concentrations in the system blanks or in the processing/dilution blank.

Quality Control Calibration Verification Check Standards: Five mid-range verification standards were analyzed throughout the analysis run. For all reported results, the concentrations of all analytes of interest were recovered within the governing QA Plan acceptance criteria of ± 10% for the verification standard.

Notes:

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

2) 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.

3) Routine precision and bias is typically ± 15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions. Sample-specific precision and bias may be determined on each sample if required.

Battelle
Putting Technology To Work

Project No. 29953

Internal Distribution

Date April 6, 1999 File/LB

To G. Lumetta

From M. Urie

Subject Carbon Analysis Results for AW-101 SOL and AW-101 AQ Samples

The analysis of the AW-101-SOL and AW-101-AQ samples submitted under ASR 5275 was done by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same weighed sample, with TC being the sum of the TIC and TOC.

The samples were analyzed on April 1, 1999 and Table 1 below shows the results, rounded 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.

Due to the limited quantity of original sample available and the number of different analyses requested, the sample were diluted to provide enough volume for each of the analyses. All results are corrected for preparative dilutions and analysis dilutions, and are reported in microgram of carbon per milliliter of original sample.

QC Narrative

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC).

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type. The QC system calibration standards were all within acceptance criteria, with the average recovery being 93.9% for TIC and 97.1% for TOC. The calibration blanks were acceptable, averaging 16.7 μgC for TIC and 33.7 μgC for TOC.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike recovery from sample 99-1160 106% for TIC and 103% for TOC, well within the acceptance criteria of 75% to 125%. The precision, estimated by the RPD (Relative Percent Difference) between duplicates, could not be measured since the duplicate contained carbon less than 5 times the estimated quantitation limit.

Some results are 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.

Table 1: TIC, TOC, and TC Results

ALO Number	Sample ID	Vol ml	Prep Dilution Factor	TIC* µgC/ml	TIC RPD	TOC* µgC/ml	TOC RPD	TC* µgC/ml	TC RPD
99-1151 PB	PROCESS BLANK	2.00	5.00	<25		<40		< 65**	
99-1151	AW101-SOL-30A1	1.00	12.25	2760		1900		4660	
99-1152	AW101-SOL-30A2	1.00	10.00	2960		1940		4900	
99-1153	AW101-SOL-40A1	1.00	12.25	3040		2010		5050	
99-1154	AW101-SOL-40A2	1.00	12.25	2940		1960		4900	
99-1155	AW101-SOL-50A1	1.00	10.00	3170		2010		5180	
99-1156	AW101-SOL-50A2	1.00	10.00	2730		2030		4760	
99-1157	AW101-AQ-30A	0.50	5.00	410		1900		2310	
99-1158	AW101-AQ-50A	0.50	5.00	190		<170		360**	
99-1159	AW101-AQ-70A	1.00	5.00	120		<80		200**	
99-1160	AW101-AQ-90A	1.00	5.00	120		<80		200**	
99-1160 Dup	AW101-AQ-90A Dup	1.00	5.00	120	n/a	<80	n/a	200**	n/a
99-1160 Spike	AW101-AQ-90A Spike (%rec)	1.00	5.00	106%		103%		105%	

* Corrected for laboratory dilution performed prior to analysis
** Maximum TC (i.e., results calculated as if "< values" present in the sample)
RPD = Relative Percent Difference between sample and duplicate (n/a = not calculated since results <5xMDL)

Approve: D. R. Baldwin 4/6-99

Archive Information:

Files: C124-P-701.doc, C124-701.xls ASR: 5275

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File

Date March 10, 1999

To Mike Urie

From James Bramson

Subject ICP/MS Analysis of Submitted Samples
(ACL #99-1151 through 99-1160)

Pursuant to your request, the 11 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 5x (99-1159, 99-1160) and 20x (all others) from the dilutions provided. The results include your dilutions and are reported in ng/ml (ppb) of the original sample. Unless otherwise specified, the overall uncertainty of the values is conservatively estimated at ±10%, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Ru counts, corrected for sample dilution, are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0624 or Tom Farmer at 372-0700.

J Bowman 3/10/99

Lumetta Tc-99 Samples

March 10, 1999

The results are reported in ng/ml (ppb) of original sample.
The uncertainty of the results is estimated at ±10%.

Sample Number	Client Number	ICP/MS Number	Tc-99 ng/ml	Ru ratio 101/102 (*0.541)	†Ru-101 ng/ml
1%HNO3		9309a1	<0.05		
1%HNO3		9309a10	<0.05		
1%HNO3		9309a25	<0.05		
PB-1151		9309a11	<0.05	1.5217	3
99-1151	AW101-SOL-30A1	9309a21	6030	1.1917	1700
99-1151 + spike	AW101-SOL-30A1	9309a24	8740		
Spike Recovery			108%		
99-1152	AW101-SOL-30A2	9309a19	6230	1.1788	1800
99-1152 Dup.	AW101-SOL-30A2	9309a23	6210	1.2040	1700
99-1153	AW101-SOL-40A1	9309a20	6320	1.1136	1700
99-1154	AW101-SOL-40A2	9309a22	6230	1.1630	1700
99-1155	AW101-SOL-50A1	9309a17	6520	1.1806	1800
99-1156	AW101-SOL-50A2	9309a18	6400	1.1421	
99-1157	AW101-AQ-30A	9309a15	1380	1.1868	150
99-1157 Dupe	AW101-AQ-30A	9309a16	1390	1.0951	150
99-1158	AW101-AQ-50A	9309a14	149	1.2497	15
99-1159	AW101-AQ-70A	9309a13	47.4	1.0146	0.8
99-1160	AW101-AQ-90A	9309a12	23.1	1.6620	0.5
1ppb Tc-99		9309a7	0.977		
10ppb Tc-99		9309a26	10.3		
10ppm Co		9309a5	<0.05		

* Natural $^{101}Ru/^{102}Ru$ ratio.
†Based on response from yttrium.

Tc-99 0.017 Ci/g

$$\frac{ng}{mL} \cdot \frac{g}{10^9\,ng} \cdot \frac{0.017\,Ci}{g} \cdot \frac{10^6\,\mu Ci}{Ci}$$

DATA REVIEW

Reviewed by: [signature]

Date: 11 MAR 99 Pages: ____

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File

Date April 29, 1999

To Greg Lumetta

From Tom Farmer

Subject ICP/MS Analysis of Submitted Samples

Pursuant to your request, the sample that you submitted for analysis was analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc was used to generate the calibration curve. An independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. Unless otherwise specified, the overall uncertainty of the values is conservatively estimated at ±10%, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Approximate ^{101}Ru concentrations are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624

Lumetta Tc-99 Analysis

April 28, 1999

J Brown 4/30/99

Results are reported in ng analyte/ml solution submitted.
The uncertainty of the results is estimated at ±10%.

Sample Number	ICP/MS Number	Tc-99 ng/ml	$^{101}Ru/^{102}Ru$ (*.541)	†^{101}Ru ng/ml
1%HNO3	9428a1	<1		
1%HNO3	9428a4	<1		
99-1161 AW101-AQ-100b	9428a5	431	0.854	17
99-1161 Dup.	9428a7	443	1.166	18
99-1161 + spike	9428a8	629		
Spike Recovery		99%		
5ppb Tc-99 CCV	9428a6	4.78		
10ppb Co	9428a9	<1		

DATA REVIEW
Reviewed by: _____
Date: _____ Pages: _____

DATA REVIEW
Reviewed by: D. Blaine Tavener III
Date: 30Apr99 Pages: 1 of 1

AW101 Tank Liquids and Wash Solutions (ASR 5275)
Radiochemistry Analytical Results

Sample Preparation

Tank liquid and wash solution samples were analyzed from tank AW101. The samples were acid digested according to procedure PNL-ALO-128 in the laboratory prior to analysis. Radiochemistry results are shown on the attached table along with 1-sigma total uncertainties. All results are reported on a uCi per ml of liquid. Samples labeled "duplicate" are independent analyses from separate aliquots of starting material in the hot cell; those labeled "replicate" are separate aliquots analyzed in the laboratory.

Gamma Energy Analysis

The acid digested samples were directly gamma counted following procedure PNL-ALO-450. Most of the gamma emission from these samples is from Cs-137. The only other detectable gamma emitters were Co-60 and Cs-134. The prep blank had a negligible amount of Cs-137.. All of the duplicate results agree within the expected uncertainties. Since gamma analyses do not involve chemical separations, no sample spiking is performed. Due to the high level of Cs-137 in these samples, it was not possible to detect all of the other analytes at the requested Minimum Reportable Quantity values. Detection limits are thus reported for Eu-154, Eu-155, and Am-241.

Gross Alpha

For gross alpha measurements, aliquots of the digested samples were evaporated on planchets for counting following procedures PNL-ALO-420 and 421. Weak alpha activity was only detectable in two of the wash solutions. All of the other samples had detection limits well below the requested MRQ values. Sample and blank spike recoveries were acceptable. No alpha activity was found in either the prep blank or the lab blank.

Strontium-90

The Sr-90 analyses were conducted according to procedures PNL-ALO-476, 484, and 450 using a Sr-85 tracer to monitor the chemical yields. All of the samples had detectable levels of Sr-90. Sr-90 was not detected in the hot cell blanks. The blank and sample spike recoveries were acceptable. Duplicate results were in acceptable agreement considering the uncertainties on the measurements.

Uranium

Uranium was measured directly in the digested samples by kinetic phosphorescence following procedure PNL-ALO-4014. Uranium was detectable in all of the samples with concentrations ranging from 1-4 ug/ml. A negligible amount of uranium was seen in the prep blank; no uranium was detected in the lab blank. No uranium was detectable in the instrument blanks. The duplicate samples were in good agreement. All of the instrument check standards came out between 99% and 102%.

J R Greenwood
3-30-99

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-1151 Rev. 1
3/30/99

Client : Lumetta

Cognizant Scientist: L R Greenwood Date : 3/30/99

Concur : T Trang-Le Date : 3/30/99

Measured Activities (uCi/ml)

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Uranium ug/ml Error %	Co-60 Error %	Cs-134 Error %	Cs-137 Error %	Am-241 Error %	Eu-154 Error %	Eu-155 Error %
99-1151PB AW101-SOL-30A1	<4.E-5	<1.E-4	6.96E-5 3%	<9.E-6	<8.E-6	1.55E-5 29%	<5.E-5	<3.E-5	<3.E-5
99-1151 AW101-SOL-30A1	<6.E-3	9.49E-1 14%	2.73E+0 2%	<4.E-3	5.48E-2 10%	2.55E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1151 Rep AW101-SOL-30A1	<6.E-3								
99-1152 AW101-SOL-30A2	<6.E-3	4.00E-1 30%	2.80E+0 2%	<4.E-3	5.94E-2 9%	2.64E+2 2%	<2.E-1	<9.E-3	<2.E-1
99-1153 AW101-SOL-40A1	<7.E-3	5.19E-1 24%	3.00E+0 2%	<3.E-3	5.68E-2 10%	2.67E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1154 AW101-SOL-40A2	<6.E-3	6.96E-1 18%	2.98E+0 2%	<4.E-3	5.73E-2 9%	2.64E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1155 AW101-SOL-50A1	<6.E-3	5.34E-1 23%	3.15E+0 2%	<2.E-3	5.77E-2 7%	2.76E+2 2%	<7.E-2	<1.E-2	<7.E-2
99-1156 AW101-SOL-50A2	<8.E-3	3.52E-1 34%	3.08E+0 2%	<2.E-3	6.09E-2 7%	2.72E+2 2%	<7.E-2	<9.E-3	<7.E-2
99-1157 AW101-AQ-30A	<3.E-4	3.44E-2 18%	3.00E+0 4%	<4.E-4	3.03E-3 16%	1.69E+1 2%	<2.E-2	<2.E-3	<1.E-2
99-1157 DUP AW101-AQ-30A	<3.E-4	4.16E-2 15%	3.05E+0 4%	<4.E-4	3.18E-3 18%	1.71E+1 2%	<2.E-2	<2.E-3	<1.E-2
RPD		19%	2%		5%	1%			
99-1158 AW101-AQ-50A	<5.E-5	1.89E-2 8%	1.19E+0 4%	<7.E-5	2.38E-4 20%	1.14E+0 2%	<3.E-3	<2.E-4	<2.E-3
99-1158 Rep AW101-AQ-50A		1.60E-2 9%							
99-1159 AW101-AQ-70A	2.70E-4 10%	7.19E-2 3%	4.45E+0 4%	4.60E-5 17%	6.67E-5 19%	3.15E-1 2%	<7.E-4	<8.E-5	<5.E-4
99-1160 AW101-AQ-90A	2.52E-4 11%	6.85E-2 3%	4.16E+0 4%	<6.E-5	<7.E-5	1.88E-1 2%	<1.E-3	<2.E-4	<8.E-4
Matrix Spike	93%	117%							
Reagent Spike	88%	104%							
Blank	≤3.E-6	<1.E-4	<-1.78E-5						
Before Run UL 283-e			100%						
R-283-c			99%						
Mid Run UL -283-f			101%						
R-283-d			102%						
Post Run UL 283-e			99%						
R-283-d			102%						

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-1161
5/5/99

Client : Lumetta

Cognizant Scientist: J R Greenwood Date : 5/5/99

Concur : T Trang-Le Date : 5/5/99

Measured Activities (uCi/ml)

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Uranium ug/ml Error %	Co-60 Error %	Sb-125 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-1161 AW101-AQ-100	1.04E-2 3%	4.51E+0 3%	4.05E+2 3%	3.64E-3 2%	4.03E-3 3%	8.07E-2 2%	1.46E-3 3%	1.80E-3 6%	5.60E-3 5%
Matrix Spike	102%	127%							
Blank Spike	103%	96%	96% 102%						
Blank	<5.E-5	<8.E-6	<2.E-5						

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Pacific Northwest Laboratories

Page _____ of _____

ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
S.L. Lumetta	4/13/99	29953

Title/Subject: Measurement of AW101 and AN107 Solution Densities

Reviewed by KP Brooks 3/8/99

Pipet used: 1141166 (see attached calibration information)

1.00-mL aliquots were weighed on balance # _____ (Cal. expires 8/99)

Sample ID	#1	#2	#3	#4		Mean	Std Dev
AW101-AQ-30B	1.036	1.030	1.035	~~1.036~~ 1.036	S.L.L. 4/13/99	1.034	0.003
-50B	1.005	1.006	1.003	1.006		1.005	0.001
-70B	1.001	0.996	1.002	1.006		1.001	0.004
-90B	1.006	1.003	1.005	1.003		1.004	0.002
AN107-AQ-30A	1.070	1.076	1.077	1.073		1.074	0.003
-50A	1.011	1.000	1.008	1.003		1.006	0.005
-70A	1.0065	1.008	1.001	1.004		1.005	0.003
-90A	1.005	1.002	1.003	1.008		1.005	0.003

Dose rate on AN107-AQ-30A (hottest sample): Contact: 195 β 23 γ Wide Sheets RCT
6": 45 β 5 γ

Shielded Analytical Laboratory
Bench Sheet

Client: Lumetta, Gregg

WP Number: WR8481

TI#/ASR: E-mail dated 4-9-99

Procedure: SFO SOP 325-SAL-EDS-1

AW101 Sludge Wash Samples

SAMPLE IDENTIFICATION

- transfer the following to clean vials, then to rm 511:
- removed from archieve rack 4 slots 1, 2, 3, and 4, AW101-AQ-30B/50B/70B/90B
- AN107-AQ-30A/50A/70A/90A.

(placed into like labeled 20 ml vials.)

M&TE: — Cell 2 (360-06-01-016) Mettler AE160 Balance Other —

— Cell 5 (360-06-01-039) Mettler AT400 Balance

— Bench (360-06-01-024) Sartorius Balance

— Cell 5 (360-06-01-045) Toledo 3026 Balance

Analyst: R Lettau Date: 4-13-99 Reviewer: Rick Steele Date: 4/30/99

Lettau, Ralph C

From: Kelly, Elizabeth F
Sent: Friday, April 09, 1999 1:41 PM
To: Lettau, Ralph C
Subject: Transfer of Samples (Lumetta)

Ralph,

Welcome back!

Per Gregg Lumetta, he needs the following samples put into clean vials and transferred to lab 511. You have been scheduled to do this work on Monday, April 12th.

The work should be charged to W48481.

The samples are:

AW101-AQ-30B
AW101-AQ-50B
AW101-AQ-70B
AW101-AQ-90B

} rack 4 slot 1-4

AN107-AQ-30A
AN107-AQ-50A
AN107-AQ-70A
AN107-AQ-90A

****

Elizabeth Kelly
509/373-4146 (-9675 fax)
elizabeth.kelly@pnl.gov

DATA SHEET FOR PIPETTOR CALIBRATION

Procedure Number: PCS-TP-511-2 Revision Number: 1

1. Date Performed: 4/9/99
2. Pipettor ID: 1141166 (1-ml capcity)
3. Balance Calibration Code: 384-06-01-005
4. Balance Calibration Due Date: 8/99
5. Thermometer Control Number: 384-79-04-005
6. Volume 1 = 0.20
7. Volume 2 = 0.50
8. Volume 3 = 1.00
9. Ambient Temperature at Start of Procedure: 23 °C

Aliquot No.=	1	2	3	4	5	6
10. Mass Volume 1, g	0.202	0.196	0.199	0.197	—	—
11. Mass Volume 2, g	0.498	0.499	0.498	0.494	0.489	0.492
12. Mass Volume 3, g	1.002	1.002	0.996	0.998	1.004	—

13. Ambient Temperature at End of Procedure: 23 °C

Performed by: [signature] Date: 4/9/99
Reviewed by: Brian Lyke Date: 8-10-99

CALCULATION SHEET FOR PIPETTOR CALIBRATION

Procedure Number: PCS-TP-511-2

1. Mean Temperature: 23 °C

2. Density of Water: 0.9976 g/mL

		Mean Mass, g	Std. Dev., g	Accuracy, %	Precision, %
3.	Volume 1	0.199	0.003	± 0.26	± 1.51
4.	Volume 2	0.495	0.004	± 0.76	± 0.81
5.	Volume 3	1.000	0.003	± ~~0.21~~ 0.24 n.i.t.	~~± 0.10~~ ± 0.30 n.i.t.

Performed by: B.J. Smith Date: 4/9/99

Reviewed by: Brian Ryder Date: 8-10-99

Appendix C. Calculations

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ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
N.A. Turnette	4/26/99	29953

Title/Subject: AW101 Wash/Leach

Reviewed by KP Brooks

Excel spreadsheets were used to perform the required calculations. As an example of these calculations, we consider the case of Aluminum.

Sample AW101-AQ-30A → ICP was performed in duplicate. The average value from these duplicates was used.

$$\frac{1080 + 1100}{2} = 1090 \text{ ug/mL}$$

Sample AW101-AQ-30A → 89.6 ug/mL
-70A → 49.6
-90A → 38.5

To determine the amount of Al in each wash solution, the measured concentration is multiplied by the volume of the solution. The solution volumes were determined from the solution densities and the weights:

AW101-AQ-30	~~(1.034 g/mL)~~	(17.4614 g)/(1.034 g/mL) = 16.887 mL
-50	~~(1.005 g/mL~~	(19.5241 g)/(1.005 g/mL) = 19.427 mL
-70	~~1.001 g/mL~~	(18.9526 g)/(1.001 g/mL) = 18.934 mL
-90	~~1.004 g/mL~~	(17.8641 g)/(1.004 g/mL) = 17.793 mL

Thus,

AW101-AQ-30	(16.887 mL)(1090 ug/mL) =	18,407 ug Al
-50	(19.427 mL)(89.6 ug/mL) =	1,741 ug Al
-70	(18.934 mL)(49.6 ug/mL) =	939 ug Al
-90	(17.793 mL)(38.5 ug/mL) =	685 ug Al

The washed solids were dissolved in acid and diluted to a volume of 25 mL (sample AW101-AQ-100D). The resulting solution was found to contain 67.3 ug/mL Al.

(67.3 ug/mL)(25 mL) = 1682.5 ug Al in the washed solids

N.A.T. 5/4/99

$$\frac{1682.5 \text{ ug}}{0.0577 \text{ g solids}} = 29,159 \text{ ug Al/g dry solids}$$

ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
S.I. Sundberg	4/26/99	29953

Title/Subject: AW101 wash/Leach

Reviewed by KP Breaks

Percentage of Al in each solution and the washed solids:

Total Al = 18,407 + 1,741 + 939 + 685 + 1682 = ~~24,772~~ 23,454 mg

1st Wash	100 (18407 / ~~24772~~ 23,454)	=	78%	→ 78.5
2nd Wash	100 (1741 / ~~24772~~ 23,454)	=	7%	7.4
3rd Wash	100 (939 / ~~24772~~ 23,454)	=	4%	4.0
4th Wash	100 (685 / ~~24772~~ 23,454)	=	3%	2.9
Washed Solids	100 (1682 / ~~24772~~ 23,454)	=	7%	7.2
			99%	round down → 100.0

S.I.S. 4/26/99

OK

Caveat: A certain % amount of the washed solids did not dissolve in acid. The above values assume that all the solids were dissolved for analysis.

See the attached 2 Printouts for the results of the other calculations done within Excel.

Tables 1, 2, 4 3

ENGINEERING WORKSHEET

Prepared By: S.J. [illegible]	Date: 5/4/99	Project: 29953

Title/Subject: AW101 Wash/Leach : Mass Balance Considerations

Reviewed by KP Brooks

From BNFL-RPT-003, rev. 0 : $\rho = 1.32$ g/mL for centrifuged supernate

Sample was 97.5 wt% centrifuged supernate
2.5 wt% centrifuged solids.

Let's consider Aluminum

supernate → (17800 + 14900)/2 = 16350 µg/mL ÷ 1.32 g/mL → 12386 µg/g
solids → (14700 + 14300)/2 = 14500 µg/g

$$\frac{(97.5)(12386\ \mu g/g) + (2.5)(14500\ \mu g/g)}{100} = 12439\ \mu g/g$$ in the whole sample (supernate + solids)

Total wt. sample used in test = 110.2016 g (1.8A)

Hence,
(110.2016 g)(12439 µg/g) = 1370798 µg Al in total sample used.

But 108.1266 g. of supernate separate (1.8B).
↳ (108.1266 g)(12386 µg/g) = 1339256 µg taken out

So,
1370798 − 1339256 = 31542 µg Al into experiment

Total Al found in experiment (wash solutions + acid-dissolved solids) = 23454 µg (see p.2)

See Table 4 for mass balance on other key components.

∴ $100 \frac{23454}{31542} = 74.4\%$ recovery

Low recovery of Al ~~probably~~ possibly due to material undissolved in acid.

If this were the case, the Al content in the washed solids would be:

see p1 $\frac{\sout{23459}\ 1682 + 8070}{\sout{0.0???}\ 0.0577} = \sout{645216}\ 169012$ µg/g S.J.L. 5/4/99

ENGINEERING WORKSHEET

Prepared By: N.G. Fremette	Date: 5/17/99	Project:

Title/Subject: AW101 Wash/Leach: Mass Balance Considerations

Reviewed by KP Brooks

The adjusted Al concentration, assuming "lost" Al is in the undissolved solids is given on the previous page (16.9 wt%).

This calculation is repeated for other key components below.

Chromium

Supernate $(61.1 + 51.1)/2 = 56.1$ mg/mL $\xrightarrow{\div 1.32}$ 42.5 mg/g

Solids $(1640 + 1600)/2 = 1620$ mg/g

$$\frac{(97.5)(42.5\ \text{mg/g}) + (2.5)(1620\ \text{mg/g})}{100} = 81.94\ \text{mg/g}$$ in the whole sample

$(81.94\ \text{mg/g})(110.2016\ \text{g}) = 9030$ mg Cr

$(42.5\ \text{mg/g})(108.1266\ \text{g}) = 4595$ mg Cr (removed in supernate)

4435 mg Cr into expt.

total Cr found = $1098 + 104 + 33 + 8 + 2018 = 3261$ mg (from Table 2)

$100\ (3261)/4435 = 74\%$ recovery

4435 − 3261 = 1174 mg Cr unaccounted for.

Adjusted Cr conc. in washed solids: $(2018 + 1174)/0.0577 = 55320$ mg/g

↓

5.5 wt%

Iron

Supernate $(5.1 + 4.3)/2 = 4.7 \xrightarrow{\div 1.32} 3.6$ mg/g

Solids $(1350 + 1430)/2 = 1390$ mg/g

$((97.5)(3.6) + (2.5)(1390))/100 = 38.26$ mg/g in sample

$(38.26)(110.2016) = 4216$

$(3.6)(108.1266) = 389$

3827 mg Fe in.

Total Fe found: $7.4 + 12.3 + 11.9 + 2825 = 2857$ mg

3827 − 2857 = 970 mg unaccounted for.

$100\ (2857)/3827 = 75\%$ recovery

$(2825 + 970)/0.0577 = 65,771$ mg/g ⟹ 6.6 wt%

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ENGINEERING WORKSHEET

Prepared By: M.J. Lumetta	Date: 5/17/99	Project:

Title/Subject: AW101 Wash/Leach: Mass Balance Considerations

Reviewed by KP Brooks

Manganese

Supernate ⟶ 0

Solids (1390 + 1440)/2 = 1415 mg/g

$$\frac{(2.5)(1415)}{1000} = 35.4 \text{ mg/g}$$

(35.4 mg/g)(110.2016) = 3901 mg Mn in

Total Mn found = 1.5 + 8.1 + 6.8 + 2625 = 2642 mg Mn → 1259 mg unaccounted for

100(2642)/3901 = 68% recovery

(2625 + 1259)/0.0577 = 67,310 mg/g ⇒ ~~67.3~~ 6.7 wt%

Uranium

Supernate (3.32 + 3.12)/2 = 3.22 ÷1.32 → 2.4 mg/g

Solids (5420 + 5460)/2 = 5440 mg/g

((9765)(2.4) + (2.5)(5440))/100 = 138.34 mg/g

(138.34 mg/g)(110.2016 g) = 15245 mg

(2.4 mg/g)(108.1266 g) = 260

14985 mg U in → 4637 mg unaccounted for

Total U found: 51.1 + 23.1 + 8.43 + 74.0 + ~~10116~~ 10116 = 10348 mg

100(10348)/14985 = 69% recovery

$$\frac{10116 + 4637}{0.0577} = 255\,685 \text{ mg/g} \Rightarrow 25.6 \text{ wt\%}$$

ENGINEERING WORKSHEET

Prepared By: M.J. Truitt | Date: 8/6/99 | Project:

Title/Subject: AW101 Wash/Leach

Reviewed by KP Brooks

The filtered solids apparently retained considerable liquid. How much of the components in solution result from simply a dilution of this liquid (as opposed to actual dissolution of solids)?

Consider the first wash step.

2.0750 g of filtered solids (1.8C)

In the end we ended up with only 0.0177 g of washed solids

So assume we had ~2 g of interstitial liquid.
(Let's say: 2 mL)

Let's consider ^{137}Cs.

From PNWD-2463 (Urie et al. 1999): The diluted AW-101 supernatant liquid contained

(250 + 210)/2 = 230 uCi/mL

The volume of the first wash solution was 16.9 mL (see p.1)

So,

(230 uCi/mL)(2 mL) / 16.9 mL = 27.2 uCi/mL expected for dilution.

Measured in the first was → 17.0 uCi/mL

Similarly for Al:

PNWD-2463 → (17800 + 14900)/2 = 16350 mg/mL

(16350 mg/mL)(2 mL)/16.9 mL = 1935 mg/mL expected for dilution

Found 1090 mg/mL

Let's try to get a better assumption here. Assume that ~~aluminum~~ Na concentration in first wash solution is due to dilution of interstitial liquid + 0.01 M NaOH added
↳ (230 mg/mL)

12150 mg Na/mL found in first leach solution.

12150 − 230 = 11920 mg/mL due to dilution

ENGINEERING WORKSHEET

Prepared By: M. J. Hunatta	Date: 8/6/99	Project:

Title/Subject: AW101 wash/leach

Reviewed by
MP Brooks 8/9/99

From PNWD-2463 → (163000 + 134000)/2 = 148500 mg/mL Al in dil. AW 101

V (148500 mg/mL) = (16.9 mL)(11920 mg/mL)

V = 1.36 mL of interstitial liquid → Assume this

^{137}Cs : (230 mCi/mL)(1.36 mL)/16.9 mL = 18.5 mCi/mL (17.0 mCi/mL found)

Al : (16350 mCi/mL)(1.36 mL)/16.9 mL = 1316 mg/mL (1090 mg/mL found)