PNWD-3029
BNFL-RPT-023 Rev. 0

Mixing of Process Heels, Process Solutions, and Recycle Streams: Results of the Small-Scale Radioactive Tests

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LEGAL NOTICE

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1.0 INTRODUCTION

Various recycle streams will be combined with the low-activity waste (LAW) or the high-level waste (HLW) feed solutions during the processing of the Hanford tank wastes by BNFL, Inc. In addition, the LAW and HLW feed solutions will also be mixed with heels present in the processing equipment. This report describes the results of a test conducted by Battelle to assess the effects of mixing specific process streams. Observations were made regarding adverse reactions (mainly precipitation) and effects on the Tc oxidation state (as indicated by K_d measurements with SuperLig® 639). The work was conducted according to test plan BNFL-TP-29953-023, Rev. 0, *Small Scale Mixing of Process Heels, Solutions, and Recycle Streams*. The test went according to plan, with only minor deviations from the test plan. The deviations from the test plan are discussed in the experimental section.

2.0 EXPERIMENTAL

Sample Description. Five Hanford waste samples were used in this work. These are listed in Table 2.1 along with a description of the samples' origins.

Table 2.1. Hanford Tank Waste Materials Used in the Mixing Tests

Label	Description
AN107	AN-107 solution after Sr/TRU precipitation and cross-flow filtration
AN107 WASH	Composite solution obtained during washing of the AN-107 entrained solids
AW101	AW-101 solution after cross-flow filtration
AW101 WASH	Composite solution obtained during washing of the AW-101 entrained solids
C104 LEACH/WASH	Composite of C-104 washing and caustic leaching solution

Procedure. A detailed description of the experimental procedure can be found in Appendix A. Table 2.2 summarizes the specific mixing tests performed. For tests 1 through 5, the solutions were mixed at ambient temperature (~23°C) and three 5-mL aliquots were taken within 4 h of mixing. One aliquot was filtered through a 0.45-µm nylon membrane and was set aside for analysis. The other two were used for Tc K_d measurements as described below. After standing for a period of 2 weeks, three more 5-mL aliquots were taken. Again, one was filtered and saved for analysis, while the other two were used for Tc K_d measurements.(a)

For tests 6 through 9, the solutions were mixed and observed for two weeks. No Tc K_d measurements were performed for these mixtures. The solids formed in tests 6 and 7 were collected by centrifuging the mixture and then separating the liquid phase. The solids were dried at 105°C and analyzed by ICP/AES, IC, TIC/TOC, GEA, ^{90}Sr, and total alpha. The mass of the dried solids from Test 6 was 0.17 g and that from Test 7 was 0.12 g. These weights include the weight of any dissolved solids present in the interstitial liquid prior to drying.

(a) In the case of Test 2, one aliquot was lost due to a leak in the syringe filter. Because of this, only a single K_d measurement could be done after 2 weeks.

Table 2.2. Summary of Mixing Tests

Test #	Solution 1	Vol., mL	Mass, g	Solution 2	Vol., mL	Mass, g	Observations
1	AN107	3	3.9720	AW101	30	38.2534	Solution cloudy after 1.5 h; small amount of precipitate on the bottom of vial after 4 days. There was no further change after standing for another 10 days.
2	AN107	3	3.8118	C104 LEACH/WASH	30	31.9973	Same as Test 1
3	AN107	20	24.9540	C104 LEACH/WASH	20	20.9489	Solution was cloudy immediately after mixing; a dark brown solid had settled to the bottom of the vial after 1 day. There was no further change after standing for 2 weeks.
4	AW101	3	3.6916	C104 LEACH/WASH	30	32.1748	Clear solution after 2 weeks
5	AW101	20	24.8593	C104 LEACH/WASH	20	21.8070	Clear solution after 2 weeks
6	AN107	~15[(a)]	18.7667	AN107 WASH	2	2.0545	Solution was cloudy immediately after mixing; a dark solid had settled to the bottom of the vial after 3 days. There was no further change after standing for another 2 weeks.
7	AN107	~7[(b)]	7.7457	AN107 WASH	10	10.6165	Same as Test 6
8	AW101	20	24.9273	AW101 WASH	2	1.9836	Clear solution after 3 weeks
9	AW101	10	12.5535	AW101 WASH	10	10.1816	Clear solution after 3 weeks

(a) The test plan called for 20 mL, but only ~15 mL used because insufficient sample was available.
(b) The test plan called for 10 mL, but only ~7 mL used because insufficient sample was available.

Technetium K_d Measurements. The Tc K_ds were determined by stirring 5 mL of solution with 0.05 g of SuperLig® 639 (SL-639). The SL-639 was used as-received from IBC Advanced Technologies, Inc. (American Fork, Utah). For tests 4 and 5, the solution/SL-639 mixtures were stirred for 24 hours; for all other measurements, the mixtures were stirred for 1 week.(a) After stirring, the solutions were filtered through 0.45-μm nylon syringe filters. The filtered solutions were analyzed for ^{99}Tc by ICP-MS, as were aliquots of the solutions taken before contact with the SL-639. The untreated solutions were also analyzed for anions by ion chromatography and for hydroxide ion by titrimetry with standard HCl. The batch K_d was determined according to the following formula:

$$K_d = \frac{(C_o - C)W_s}{W_r C' F}$$

where C_o is the initial ^{99}Tc concentration (in ng/g), C is the final ^{99}Tc concentration (in ng/g), C' is the final ^{99}Tc concentration (in ng/mL), W_s is the mass of the solution, W_r is the mass of the resin, and F is mass of the dried resin divided by the mass of the as-received resin; F = 0.956, dried at 95°C.(a)

(a) The contact time was extended to 1 week to ensure equilibrium was attained.

(a) Blanchard, D.L. Jr, D.E. Kurath, and B.M. Rapko., 2000, *Small Column Testing of Superlig 639 for Removal of ^{99}Tc from Hanford Tank Waste Envelope C (Tank 241-AN-107)*, BNFL-RPT-022, Rev 0, (DRAFT), Pacific Northwest National Laboratory, Richland, Washington.

3.0 RESULTS

Table 3.1 presents the measured K_d values. Table 3.2 presents the anion concentrations in the various mixtures investigated. The K_d data are presented in terms of total Tc and in terms of TcO_4^-. The estimated TcO_4^- concentrations were determined by first estimating the fraction of the total Tc contributed by each solution mixed. This estimate was made based on the total Tc concentration in each solution and the amount of each solution mixed. The initial TcO_4^- concentration was then estimated by determining the fraction of TcO_4^- contributed by each solution using previous estimates of the TcO_4^- content in each solution (Kurath, Blanchard, and Bontha 1999).(b) All the Tc sorbed on the resin was assumed to be TcO_4^-. The final TcO_4^- concentration in solution was determined by difference between that estimated to be initially present and that sorbed on the resin.

The total Tc K_d values ranged from about 300 mL/g to 600 mL/g, except for tests 2 and 3 for which the K_d values were an order of magnitude lower. The low K_d values for tests 2 and 3 can be attributed to the fact that the origin of most of the Tc present in these mixtures was the AN-107 feed material. A significant fraction (75%) of the Tc in the AN-107 waste is not pertechnetate ion. Since SL-639 only removes pertechnetate, the K_d values are low for AN-107 (Kurath, Blanchard, and Bontha 1999).

The TcO_4^- K_d data are perhaps more informative. In all cases, the TcO_4^- K_d values were greater than 100 mL/g, but the values tended to be low for solutions where the origin of most of the Tc was AN-107 (Tests 2 and 3). The reason for this has not been definitively determined, but it is perhaps due to competition between sorption of TcO_4^- and the sorption of competing organic anions present in the AN-107 solution.

Information provided by IBC Advanced Technologies, Inc. indicates that NO_3^- is likely to be the most important competing anion present in the waste. Thus, it is of interest to analyze the K_d data as a function of NO_3^- concentration.(c) Figure 3.1 presents the TcO_4^- K_d values as a function of NO_3^-/TcO_4^- ratio. The values obtained are consistent with those reported for the pure AW-101 and AN-107 solutions (Kurath, Blanchard, and Bontha 1999). Thus, it appears that mixing of the solutions has no significant overall effect on the behavior of Tc. That is, the Tc K_d values are determined largely based on the amount of TcO_4^- present in the solutions to begin with.

(b) The TcO_4^- fraction in C104 LEACH/WASH was unknown; it was assumed to be 100% TcO_4^-.

(c) The NO_3^-/TcO_4^- ratio may not be an appropriate indicator of the relative pertechnetate K_d for the AN-107 waste or other similar wastes that contain relatively large concentrations of organic compounds that are complexing agents. This is because the pertechnetate comprised only about 25% of the total technetium present in the AN-107 waste. The non-pertechnetate fraction cannot be absorbed by SL-639 resin or other similar anion exchange materials.

The precipitates that formed when the AN-107 solution was mixed with the AN-107 entrained solids wash solution (at 1:1 and 10:1 ratios) were analyzed by ICP-AES. The solution remaining after the precipitation was also analyzed. Table 3.3 presents the results. The data are presented in terms of the absolute concentrations in the liquids and solids (μg/mL and μg/g, respectively). Data are also presented on the amount of each component in the analyzed solids (which includes material contributed by the interstitial liquid). These values were adjusted to account for the material contributed by the interstitial liquid. To make this adjustment, it was assumed that all the Na in the analyzed solids originated in the interstitial liquid.(b) Finally, the moles of each component in the precipitated solids were determined relative to Al. The results indicated that the precipitated solids contained primarily Al, B, Fe, Mn, and Si.

The solids were taken up in deionized water and were analyzed for ions by ion chromatography. Table 3.3 presents the IC results. The chloride, nitrite, nitrate, and sulfate in the analyzed solids were largely attributable to the interstitial liquid. The primary anions in the precipitate (as determined by IC) were fluoride and oxalate. It should be noted that the hydroxide content of the solids was not determined. It is likely that hydroxide is a major counter anion present.

The TOC analysis of the solids did not corroborate the oxalate results indicated by IC. Indeed the TOC analysis suggested that the organic carbon present in the dried solids was largely attributable to that present in the interstitial liquid. On the other hand, there appeared to be considerable TIC present in the precipitated solids. Because of the discrepancies between the TOC and IC data, caution should be exercised in using these data. A larger scale test would be useful so that enough solids can be collected for thorough characterization.

The radionuclide data are somewhat difficult to interpret, because there was no straight-forward way to correct the data for the contribution of the interstitial liquid. Estimated radionuclide concentrations in the precipitated solids are presented in Table 3.3. These concentrations should be viewed as qualitative because of the number of assumptions made in their determination [see footnote (d) in the table]. The estimated radionuclide concentrations indicate the precipitated solids should be handled as high-activity waste. The precipitated solids contained approximately 500 to 1100 μCi/g ^{137}Cs/g, 100 to 300 μCi ^{90}Sr/g, 1 μCi ^{99}Tc/g and 4 to 10 μCi TRU/g.

(b) There is no rigorous justification for this assumption, but qualitative observation of the amount of liquid carried over with the solids (0.5 to 1 mL) is consistent with the assumption.

Table 3.1. Technetium Distribution Coefficients

			Fraction of ^{99}Tc[a], %		Fraction as			NO_3^-/Tc,		NO_3^-/TcO_4^-
Test #	Solution 1	Solution 2	Solution 1	Solution 2	TcO_4^-, %[b]	Time[c]	Tc K_d mL/g	mole/mole	TcO_4^- K_d mL/g[d]	mole/mole
1	AN107	AW101	5	95	93.4	1 day	298	173424	383	236418
						1 day	297	173424	382	236305
						2 weeks	290	155268	368	209587
						2 weeks	280	148530	348	197281
2	AN107	C104 LEACH/WASH	86	14	35.5	1 day	36	86167	292	713709
						1 day	25	78781	121	400860
						2 weeks	31	77031	129	477828
3	AN107	C104 LEACH/WASH	98	2	26.5	1 day	28	77659	508	1447988
						1 day	22	74396	231	809963
						2 weeks	29	77746	561	1566350
						2 weeks	25	77052	348	1100738
4	AW101	C104 LEACH/WASH	92	8	97.2	1 day	394	159516	450	185289
						1 day	450	182801	527	217651
						2 weeks	488	201070	578	241646
						2 weeks	564	212687	677	258905
5	AW101	C104 LEACH/WASH	99	1	97.0	1 day	457	200998	532	241108
						1 day	430	197816	499	236284
						2 weeks	464	220261	544	265786
						2 weeks	435	211648	504	252557

(a) Fraction of ^{99}Tc in the mixture attributed to the individual solutions mixed.
(b) Estimated as described in the text.
(c) Time at which K_d measurement was initiated.
(d) Based on the estimated TcO_4^- fractions.

Table 3.2. Anion Concentrations in the Various Mixtures

Test #[(a)]	F^-	Cl^-	NO_2^-	Br^-	NO_3^-	PO_4^{3-}	SO_4^{2-}	$C_2O_4^{2-}$	OH^- (b)
1 - Day 1	830	< 250	46000	< 250	101000	< 500	1100	< 500	1.92
1 - 2 Weeks	970	< 250	45300	< 250	98700	670	1200	< 500	1.91
2 - Day 1	3000	< 25	4800	< 25	12100	430	520	600	0.37
2 - 2 Weeks	3700	< 25	4500	26	11300	420	520	600	0.37
3 - Day 1	1540(c)	430	13900	< 250	55500	< 500	1860	980	0.54
3 - 2 Weeks	2880(c)	430	13700	< 250	54100	< 500	1840	1000	0.54
4 - Day 1	2600	200	4900	< 125	8400	430	360	510	0.48
4 - Day 1 Replicate	2600	190	4900	< 125	8400	420	330	500	
4 - 2 Weeks	2700	210	5300	< 125	9000	450	< 250	540	0.45
5 - Day 1	1800	1100	22400	< 125	47000	< 500	< 500	< 500	0.86
5 - 2 Weeks	1800	1200	22700	< 125	47500	< 500	< 500	< 500	0.82

(a) Refer to Table 2.2.

(b) Determined by titration with standard HCl. Values presented are the average of duplicate runs.

(c) Value should be viewed as qualitative because of matrix interferences.

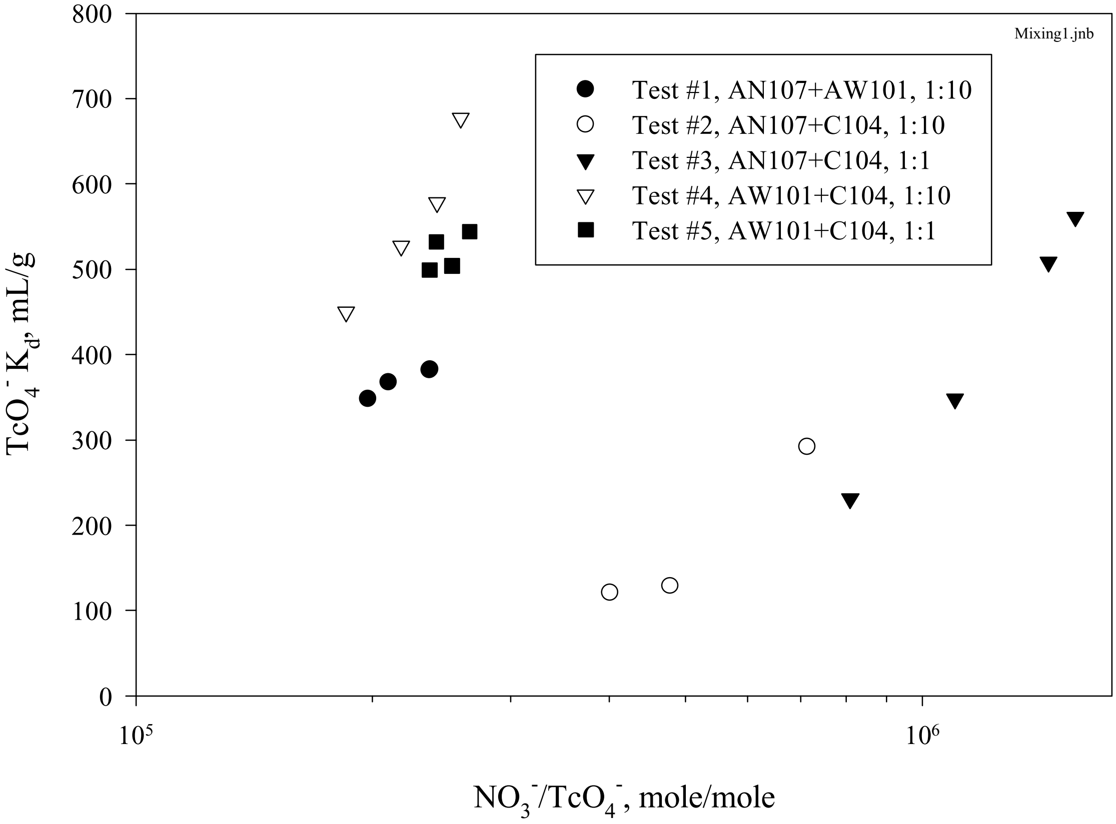

Figure 3.1. Pertechnetate K_ds Versus The Nitrate/Pertechnetate Ratio

Table 3.3. Composition of the Precipitate from the AN-107 Mixing Tests

	1:1 AN-107 Mix					10:1 AN-107 Mix				
	Liquid			Solid		Liquid			Solid	
Analyte	µg/mL	µg/g	µg(a)	Adjusted µg(b)	Relative Moles	µg/mL	µg/g	µg(b)	Adjusted µg(b)	Relative Moles
Ag	< 0.5	145	17.4	17.4	0.03	< 2.5	185	31	31	0.03
Al	1045	7320	878	128	1.00	2260	8150	1376	292	1.00
B	13	277	33.2	23.9	0.46	(24)	354	59.8	48.2	0.41
Ca	78	500	60.0	4.0	0.02	(180)	474	80.0	-6.3	-0.01
Cd	12.2	74.1	8.9	0.1	0.00	27.1	73.5	12.4	-0.6	0.00
Cr	18.9	141	16.9	3.4	0.01	44.8	135	22.8	1.3	0.00
Cu	9.2	65.2	7.8	1.2	0.00	(19)	59.5	10.0	0.9	0.00
Fe	(3.9)	1460	175	172	0.65	(9.5)	1070	181	176	0.29
K	(325)	(2000)	(240)	(7)	0.04	(750)	(2000)	(338)	-(22)	-0.05
Mn	< 1	3810	457	456	1.75	< 5.0	3150	532	529	0.89
Na	53000	317000	38040	0	0.00	107000	304000	51315	0	0.00
Ni	103	662	79.4	5.5	0.02	225	626	106	-2	0.00
P	128	766	91.9	0.0	0.00	228	787	133	24	0.07
Pb	34	257	30.8	6.4	0.01	(67)	224	37.8	5.7	0.00
Si	(38)	2460	295	268	2.01	(56)	4250	717	691	2.27
Sr	66	426	51.1	3.7	0.01	132	391	66.0	2.7	0.00
Zn	(3.7)	152	18.2	15.6	0.05	(6.9)	447	75.5	72.1	0.10
TOC	8330	39850	4782	-1197	-20.94	15900	44750	7554	-72	-0.55
TIC	4330	38850	4662	1554	27.20	8420	39250	6625	2587	19.91
Cl^-	380	2130	256	-17.1	-0.10	700	1940	327	-8	-0.02
F^-	< 250	2290	275	95.4	1.06	< 250	2270	383	263	1.28
NO_2^-	11600	70200	8424	98.3	0.45	24200	68200	11512	-94	-0.19
NO_3^-	47200	275000	33000	-877.1	-2.97	105000	274000	46251	-4105	-6.12
SO_4^{2-}	1620	9040	1085	-77.9	-0.17	3000	9260	1563	124	0.12
PO_4^{3-}	< 500	< 2000	< 240	< -151	-0.34	< 500	< 1800	< 304	< 64	0.06
$C_2O_4^{2-}$	2500	16900	2028	233.7	0.56	< 500	7370	1244	1004	1.05
	Liquid			Solid(c)		Liquid			Solid(c)	
	µCi/g	µCi/g	µCi(a)	Adjusted µCi(b)	Estimated µCi/g(d)	µCi/g	µCi/g	µCi(a)	Adjusted µCi(b)	Estimated µCi/g(d)
Cs-137	62.9	420	50.4	5.3	2204	117	430	72.6	16.5	3432
Sr-90	0.509	8.55	1.0	0.7	275	0.723	5.18	0.9	0.5	110
Tc-99(e)	0.0210	0.152	0.018	0.003	1.3	0.0425	0.152	0.026	0.005	1.1
Am-241	< 0.07	0.23	0.028	-0.023		< 0.2	< 0.5	0.084	-0.012	
Eu-154	0.014	0.269	0.032	0.023	9	0.0301	0.237	0.040	0.026	5
Eu-155	< 0.07	0.253	0.030	-0.020		< 0.2	< 0.5	0.084	-0.012	
Co-60	0.025	0.167	0.020	0.002	1.0	0.0483	0.163	0.028	0.004	
Total "	0.00591	0.245	0.029	0.025	10	0.0111	0.151	0.025	0.020	4

(a) Mass (or activity) of each component determined to be in the dried solids. This was determined by multiplying the concentration (in µg/g) by the mass of the dried solids (0.12 g for the 1:1 mix and 0.1688 g for the 10:1 mix).

(b) Mass (or activity) of each component calculated to be in the dried solids, subtracting out the contribution from the interstitial liquid. For this calculation, it was assumed that all Na in the dried solids was attributed to that dissolved in the interstitial liquid.

(c) The analytical process blank for the solids analysis indicated relatively high radionuclide concentrations. The concentrations in the blank were as follows (in µCi/g): Cs-137 3.38, Eu-154 0.0137, Eu-155 0.0178, Am-241 0.0328, and total alpha 0.0241.

(d) Determined by dividing the adjusted µCi values by the estimated mass of the precipitated solids. The mass of the precipitated solids was estimated to be 2400 µg and 4800 µg for the 1:1 AN107 Mix and the 1 0:1 AN107 Mix, respectively. The following assumptions were made in determining these estimates: 1) oxalate is present as $Al_2(C_2O_4)_3 4H_2O$, 2) the remaining aluminum is present as $Al(OH)_3$, 3) fluoride is present as $FeF_3 4.5H_2O$, 4) the remaining iron is present as $Fe(OH)_3$, 5) manganese is present as MnO_2, and 6) silicon is present as SiO_2.

4.0 CONCLUSIONS

A series of tests has been conducted in which different Hanford tank waste solutions (AN-107, AW-101, and C-104 sludge washing/leaching solutions) were mixed in varying proportions. The effects of mixing these solutions on Tc sorption on SuperLig® 639 were evaluated. The results indicate little or no adverse effect of mixing on the Tc K_d. The K_d values are largely dictated by the amount of TcO_4^- originally present in each solution before mixing. There does not appear to be appreciable reduction (or oxidation) of Tc(VII) in any of the cases examined.

A precipitate formed when the AN-107 LAW sample was mixed with the solution generated by washing the AN-107 entrained solids. This precipitate was rich in Al, B, Fe, Mn, and Si. Solids formation was also observed upon mixing the AN-107 sample with the AW-101 sample and upon mixing the AN-107 sample with the C-104 leach/wash solution. During plant operations, mixing of these solutions should be avoided to prevent formation of solids.

Future testing should focus on preventing solids formation and methods to dissolve solids that form upon mixing process solutions. Work should also be conducted to understand the mechanisms for the stabilization of the non-pertechnetate species in the AN-107 waste, with the intent to convert this to the pertechnetate form.

5.0 REFERENCES

Kurath, D.E., D.L. Blanchard, and J.R. Bontha. 1999. Ion Exchange Distribution Coefficients for ^{137}Cs and ^{99}Tc Removal from Hanford Tank Supernates AW-101 (Envelope A) and AN-107 (Envelope C), PNWD-2467, Pacific Northwest National Laboratory, Richland, Washington.

Appendix B. Analytical Reports

Appendix C. Calculations

Appendix A. Test Plan

Workplace Copy

PNNL Test Plan

Document No.: BNFL-TP-29953-023
Rev. No.: 0

Title: Small Scale Mixing of Process Heels, Solutions, and Recycle Streams

Work Location: RPL/SAL/511/516

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Author: GJ Lumetta

Effective Date:
Supersedes Date: New

Use Category Identification: Mandatory

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

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

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

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

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

FOR REVISIONS:

Is retraining to this procedure required? ____ Yes x No
Does the OJT package associated with this procedure require revision to reflect procedure changes? __ Yes__ No__ N/A

Approval:

	Signature	Date
Author	Gregg J. Lumetta	3/5/99
Technical Reviewer	Brian M. Rapko	3-5-99
RPL Manager		3/11/99
Project Manager	DE Kurath	3/9/99
RPG QE		3/9/99
BNFL	Michael E Johnson	3/11/99

Applicability

This test plan is to be used to fulfill the requirements of BNFL Test Specification No. 001580. The work will be conducted in the SAL hot cells and, if the radiological properties of the samples allow, in labs 511 and 516 in the RPL. 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: BNFL Inc. process engineering will use the information derived from this task to validate or modify the BNFL Inc. process flowsheet.

Objective: Battelle personnel will conduct small-scale tests using radioactive samples from candidate low-activity waste (241-AW-101 and 241-AN-107 liquid fractions) and high-level waste (241-C-104) tanks. The test objectives are to mimic planned operating conditions to determine, through qualitative observations, if chemical reactions (e.g., color change, foaming, gassing) and/or precipitation occur from mixing selected radioactive solutions. An additional test objective is to determine the Tc batch distribution value for solution mixtures, which will quantify the conversion of pertechnetate to the non-pertechnetate species.

Acronyms

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

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 indicated in Table 1 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. The calibration ID, date of calibration, and calibration expiration date must be recorded on the work place copy for each balance used.

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

No special equipment is required for this work.

Prerequisites

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

All work in labs 511 or 516 will be conducted according to PNNL Operating Procedure RPG-OP-511, *Routine Research Operations.*

The following radioactive tank samples are required:

		Label[a]
AN-107 solution after Sr/TRU precipitation	50 mL	AN107
AW-101 solution after cross-flow filtration	75 mL	AW101
C-104 permeate	100 mL	C104L
AN-107 entrained solids wash solution	3 mL	AN107Wash
AW-101 entrained solids wash solution	3 mL	AW101Wash

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

- 20-mL HDPE vial (50)
- 25-mL glass vials (2)
- 40-mL glass vials (4)
- Hot plate/stirrer
- 0.45-µm nylon syringe filters (30)
- 5-mL syringes (30)
- 0.45-µm nylon disposable filter units (4)
- 5-mL pipette
- SuperLig 639 (1 g)

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

	Rm 511 #3	Cell 2		Rm. 511 #1	Rm 511 #2
Calibration ID:	384-06-01-008	360-06-01-016		362-06-01-043	384-06-01-~~003~~ -005 m.l.l. 9/21/99
Calibration Date:	8/27/99	3/2/99	8-12-99	8/17/99	8/18/99
Expiration Date:	8/2000	8/99	2/2000	8/00	8/00

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

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[a] These labels will be used throughout this test plan to refer to these samples.

Also before beginning work, a routine performance check should be performed the 5-mL pipettor to be used to transfer the solutions for the K_d measurements. The performance check should include pipetting and weighing at least three 5-mL aliquots of deionized water. The results of the performance check shall be documented in the space below. The temperature shall also be recorded.

Work Instructions

> **Note**
>
> Where practical, catch pans should be used when working with the tank waste samples, so that they can be recovered if spilled.

Part 1. Mixing AN-107 With AW-101

1.1. Prepare the sample vials according to the following table. All vials should be 20 mL HDPE.

Sample ID	Vial Tare wt
AN107AW101-1A	8.0235 g.
AN107AW101-1B	8.0791 g.
AN107AW101-1C	8.1000 g.
AN107AW101-2A	8.1173 g.
AN107AW101-2B	8.0313 g.
AN107AW101-2C	8.1156 g.

10-29-99

1.2. Label two 20-mL HDPE vial as "AN107AW101-Kd1A" and "AN107AW101-Kd1B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

Wt. AN107AW101-Kd1A = 8.5983 g 10-29-99 (1.2a)

Wt. AN107AW101-Kd1B = 8.5879 g (1.2b) LPD 10-29-99

1.3. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107AW101-Kd1A and AN107AW101-Kd1B. Weigh each vial.

→ 8.5583 + 0.0541 = 8.6524

Note: vials tared to 0.0000, then resin added.

Wt. AN107AW101-Kd1A = ______ g (1.3a)

0.0541 – Wt. SuperLig 639 = 1.3a-1.2a = 0.0541 g (1.3b) LPD 10-29-99

Wt. AN107AW101-Kd1B = ______ g (1.3c)

0.0536 Wt. SuperLig 639 = 1.3c-1.2b = 0.0536 g (1.3d) LPD-10-29-99

→ 8.5879 + 0.0536 = 8.6415

1.4. Take vials AN107AW101-Kd1A and AN107AW101-Kd1B to the SAL and stage for subsequent steps.

1.5. Label a 40-mL glass vial as "AN107AW101-TEST" and place a magnetic stir bar in this vial. Weigh the vial with cap and stir bar.

Wt. AN107AW101-TEST = 24.5668 g ok (b) n.l.l. 11/1/99 (1.5a) LPD 10-29-99

AN107 Cs IX Batch Contact

11/1/99 n.l.l. w/ mike mann 1.6. Transfer 3 mL of sample AN107 to AN107AW101-TEST, then weigh the vial.

Wt. AN107AW101-TEST = 28.5388 g (a) (1.6a)

Wt. AN107 Added = 1.6a-1.5a = 3.9720 g (1.6b)

→ GL-060499B

1.7. Transfer 30 mL of sample AW101 to AN107AW101-TEST, then weigh the vial.

6 x 5mL
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Wt. AN107AW101-TEST = 66.7922 g (a) (1.7a)

Wt. AW101 Added = 1.7a-1.6a = 38.2534 g (1.7b)

Record the date and time at which the AW101 was mixed with the AN107.

Date/Time: 11/1/99 13:40 (1.7c)

Also record the cell temperature.

Temperature = 23 °C (1.7d)

(a) Balance 360-06-01-016

(b) Balance 362-06-01-043

1.8. Stir the contents of AN107AW101-TEST. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/1/99 No immediate reaction. At 15 sec the solution looked cloudy.

11/5/99 A slight amount of precipitate dusting the bottom of the vial.

1.8A. After 24 h, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

Notes

The first Tc K_d measurement must be started within 4 hours of mixing the AW101 and AN107 solutions.

5 mL portions of the AN107/AW101 mixture are to be transferred to vials AN107AW101-1A, AN107AW101-Kd1A, and AN107AW101-Kd1B as indicated below. These transfers are to be done in such a manner that the exterior of the vials will be free of smearable contamination. Appropriate steps should be taken by the SAL personnel to ensure this.

11/1/99 1.9. Filter 5.0 mL of the AN107/AW101 mixture through a 0.45-µm nylon membrane and place the filtered solution in AN107AW101-1A and remove the vial from the hot cell.

~~Tare wt AN107 AW101-1A = 8.0315~~

→ wt = 14.292

~~Note: The first 5 mL we tried to filter was lost (syringe filter leaked).~~ Note in wrong place. n.j.l. 11/1/99

1.10. Have an RCT determine the dose characteristics of sample AN107AW101-1A

Radiological Hold Point

RCT perform radiological dose survey of sample AN107AW101-1A. Corrected doses at contact, 15 cm, and 30 cm are required.

Note

Based on radiological dose of sample AN107AW101-1A, a determination will be made as to whether to perform the K_d measurements in the lab or hot cell.

1.11. Contact CSM (G.J. Lumetta) to determine whether samples are to be transferred to lab 511 or 516.

1.12. Transfer 5.0 mL of the AN107/AW101 mixture to AN107AW101-Kd1A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

1.13. Transfer 5.0 mL of the AN107/AW101 mixture to AN107AW101-Kd1B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

1.14. If appropriate, transfer AN107AW101-1A, AN107AW101-Kd1A, and AN107AW101-Kd1B to lab 511 or 516 (as directed by the cognizant scientist).

1.15. Weigh AN107AW101-Kd1A and AN107AW101-Kd1B.

Balance 384-06-01-008

Wt. AN107AW101-Kd1A = 14.904 g (1.15a)

Wt. Solution = 1.15a-1.3a = 6.252 g (1.15b)

Wt. AN107AW101-Kd1B = 14.823 g (1.15c)

Wt. Solution = 1.15c-1.3c = 6.182 g (1.15d)

1.16. Stir AN107AW101-Kd1A and AN107AW101-Kd1B for a minimum of 24 h

Start date/time: 11/1/99 16:30

Stop date/time: 11/8/99 14:45

11/8/99

1.17. Stop the stirrer and allow the resin to settle to the bottom of the vials.

1.18. Withdraw as much of the liquid as practical from AN107AW101-Kd1A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107AW101-1B. → wt = 13.812

1.19. Withdraw as much of the liquid as practical from AN107AW101-Kd1B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107AW101-1C. → wt = 13.745

1.20. Allow the mixture in AN107AW101-TEST to stand for two weeks

1.21. After 2 weeks, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/15/99 No significant ~~change of~~ change from ~~H~~ 11/5.

1.22. Label two 20-mL HDPE vial as "AN107AW101-Kd2A" and "AN107AW101-Kd2B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

Wt. AN107AW101-Kd2A = 8.5704 g (1.22a) HP 10-29-99

Wt. AN107AW101-Kd2B = 8.4876 g (1.22b) ↓

1.23. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107AW101-Kd2A and AN107AW101-Kd2B. Weigh each vial.

Wt. AN107AW101-Kd2A = 8.6238 g (1.23a)

Wt. SuperLig 639 = 1.23a-1.22a = 0.0534 g (1.23b)

Wt. AN107AW101-Kd2B = 8.5401 g (1.23c)

Wt. SuperLig 639 = 1.23c-1.22b = 0.0525 g (1.23d)

1.24. Filter 5.0 mL of the AN107/AW101 mixture through a 0.45-µm nylon membrane and place the filtered solution in AN107AW101-2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab. → wt = 14.239

1.25. Transfer 5.0 mL of the AN107/AW101 mixture to AN107AW101-Kd2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

1.26. Transfer 5.0 mL of the AN107/AW101 mixture to AN107AW101-Kd2B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

1.27. If appropriate, transfer AN107AW101-2A, AN107AW101-Kd2A, and AN107AW101-Kd2B to lab 511 or 516 (as directed by the cognizant scientist).

1.28. Weigh AN107AW101-Kd2A and AN107AW101-Kd2B.

Wt. AN107AW101-Kd2A = 14.872 g (1.28a)
Wt. Solution = 1.28a-1.23a = 6.248 g (1.28b)
Wt. AN107AW101-Kd2B = 14.846 g (1.28c)
Wt. Solution = 1.28c-1.23c = 6.306 g (1.28d)

1.29. Stir AN107AW101-Kd2A and AN107AW101-Kd2B for a minimum of 24 h

Start date/time: 11/15/99 15:20
Stop date/time: 11/22/99 12:30

1.30. Stop the stirrer and allow the resin to settle to the bottom of the vials.

11/22/99

1.31. Withdraw as much of the liquid as practical from AN107AW101-Kd2A and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AN107AW101-2B. → wt = 13.818

1.32. Withdraw as much of the liquid as practical from AN107AW101-Kd2B and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AN107AW101-2C. → wt = 13.792

1.33. Samples AN107AW101-1A, AN107AW101-1B, AN107AW101-1C, AN107AW101-2A, AN107AW101-2B, and AN107AW101-2C will be submitted for the analyses outlined in Table 1.

Part 2. Mixing C-104 Leachate With AN-107 At 10:1 Ratio

2.1. Prepare the sample vials according to the following table. All vials should be 20-mL HDPE.

Sample ID	Tare wt.
AN107C104-1A	8.0999 g.
AN107C104-1B	8.1214 g.
AN107C104-1C	8.1455 g.
AN107C104-2A	8.1049 g.
AN107C104-2B	8.1018 g.
AN107C104-2C	8.0205 g.

10-29-99

2.2. Label two 20-mL HDPE vial as "AN107C104-Kd1A" and "AN107C104-Kd1B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

Wt. AN107C104-Kd1A = 8.5639 g (2.2a) LPB 10-29-99

Wt. AN107C104-Kd1B = 8.5379 g (2.2b)

2.3. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107C104-Kd1A and AN107C104-Kd1B. Weigh each vial.

Vials tared to 0.0000, then resin added.

Balance 360-06-01-040
Cal. Date 8/17/99
Due 2/2000

Wt. AN107C104-Kd1A = ________ g (2.3a) 8.5639 + 0.0534 = 8.6173

Wt. SuperLig 639 = 2.3a-2.2a = 0.0534 g (2.3b)

Wt. AN107C104-Kd1B = ________ g (2.3c)

Wt. SuperLig 639 = 2.3c-2.2b = 0.0525 g (2.3d)

8.5379 + 0.0525 = 8.5904

2.4. Take vials AN107C104-Kd1A and AN107C104-Kd1B to the SAL and stage for subsequent steps.

2.5. Label a 40-mL glass vial as "AN107C104-TEST" and place a magnetic stir bar in this vial. Weigh the vial with cap and stir bar.

Wt. AN107C104-TEST = 24.4266 g (6) (2.5a) LPB 10-29-99

11/1/99 2.6. Transfer 3 mL of sample AN107 to AN107C104-TEST, then weigh the vial.

n.j.t. w/ Mike Mann

Wt. AN107C104-TEST = 28.2384 g (6) (2.6a)

Wt. AN107 Added = 2.6a-2.5a = 3.8118 g (2.6b)

"C104 Composite Leachate"

2.7. Transfer 30 mL of sample C104L to AN107C104-TEST, then weigh the vial.

6 × 5 mL
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Wt. AN107C104-TEST = 60.2357 g (6) (2.7a)

Wt. C104 Added = 2.7a-2.6a = 31.9973 g (2.7b)

Record the date and time at which the C104 was mixed with the AN107.

Date/Time: 11/1/99 14:00 (2.7c)

Also record the cell temperature.

Temperature = 23 °C (2.7d)

(6) Balance 360-06-01-016

2.8. Stir the contents of AN107C104-TEST. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/1/99 No immediate reaction. At 15:00, the solution looked cloudy.

2.8A. After 24 h, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/5/99 A small amount of precipitate on the bottom of the vial.

Notes

The first Tc K_d measurement must be started within 4 hours of mixing the C104 and AN107 solutions.

5 mL portions of the AN107/C104 mixture are to be transferred to vials AN107C104-1A, AN107C104-Kd1A, and AN107C104-Kd1B as indicated below. These transfers are to be done in such a manner that the exterior of the vials will be free of smearable contamination. Appropriate steps should be taken by the SAL personnel to ensure this.

2.9. Filter 5.0 mL of the AN107/C104 mixture through a 0.45-μm nylon membrane and place the filtered solution in AN107C104-1A and remove the vial from the hot cell.

Tare wt. AN107AW101-1A* = 8.0315 (Relabel as AN107C104-1A*)

Note: the first 5 mL we tried to filter was lost (syringe filter leaked).

2.10. Have an RCT determine the dose characteristics of sample AN107C104-1A

wt = 14.043

Radiological Hold Point

RCT perform radiological dose survey of sample AN107C104-1A. Corrected doses at contact, 15 cm, and 30 cm are required.

Note

Based on radiological dose of sample AN107C104-1A, a determination will be made as to whether to perform the K_d measurements in the lab or hot cell.

2.11. Contact CSM (G.J. Lumetta) to determine whether samples are to be transferred to lab 511 or 516.

2.12. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd1A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

2.13. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd1B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

2.14. If appropriate, transfer AN107C104-1A, AN107C104-Kd1A, and AN107C104-Kd1B to lab 511 or 516 (as directed by the cognizant scientist).

2.15. Weigh AN107C104-Kd1A and AN107C104-Kd1B.

Balance 384-06-01-008

Wt. AN107C104-Kd1A = 13.960 g (2.15a)

Wt. Solution = 2.15a-2.3a = 5.343 g (2.15b)

Wt. AN107C104-Kd1B = 13.967 g (2.15c)

Wt. Solution = 2.15c-2.3c = 5.377 g (2.15d)

2.16. Stir AN107C104-Kd1A and AN107C104-Kd1B for a minimum of 24 h

Start date/time: 11/1/99 16:30

Stop date/time: 11/2/99 14:45

2.17. Stop the stirrer and allow the resin to settle to the bottom of the vials.

2.18. Withdraw as much of the liquid as practical from AN107C104-Kd1A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-1B. wt = 13.027

2.19. Withdraw as much of the liquid as practical from AN107C104-Kd1B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-1C. wt = 13.051

2.20. Allow the mixture in AN107C104-TEST to stand for two weeks

2.21. After 2 weeks, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/15/99 No significant change from 11/5/99.

2.22. Label two 20-mL HDPE vial as "AN107C104-Kd2A" and "AN107C104-Kd2B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

8 10-29-99

Wt. AN107C104-Kd2A = 8.5628 g (2.22a)

Wt. AN107C104-Kd2B = 8.5289 g (2.22b)

2.23. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107C104-Kd2A and AN107C104-Kd2B. Weigh each vial.

8.5628 + 0.0511 = 8.6139

Wt. AN107C104-Kd2A = ________ g (2.23a)

0.0511g Wt. SuperLig 639 = 2.23a-2.22a = 0.0511 g (2.23b) 10-29-99

Wt. AN107C104-Kd2B = ________ g (2.23c)

0.0500g Wt. SuperLig 639 = 2.23c-2.22b = 0.0500g (2.23d) 10-29-99

2.24. Filter 5.0 mL of the AN107/C104 mixture through a 0.45-µm nylon membrane and place the filtered solution in AN107C104-2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab. → wt = 13.352

2.25. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

2.26. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd2B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

Note: Because of earlier mishap (step 2.9) we did not have enough material for this contact. It was therefore not done.

2.27. If appropriate, transfer AN107C104-2A, AN107C104-Kd2A, and AN107C104-Kd2B to lab 511 or 516 (as directed by the cognizant scientist).

2.28. Weigh AN107C104-Kd2A and AN107C104-Kd2B.

Wt. AN107C104-Kd2A = 13.933 g (2.28a)
Wt. Solution = 2.28a-2.23a = 5.319 g (2.28b)
~~Wt. AN107C104-Kd1B = ______ g~~ (2.28c)
~~Wt. Solution = 2.28c-2.23c = ______ g~~ (2.28d)

see bottom of p. 13. M.J.L. 11/15/99

2.29. Stir AN107C104-Kd2A and AN107C104-Kd2B for a minimum of 24 h

Start date/time: 11/15/99 15:20
Stop date/time: 11/22/99 12:30

2.30. Stop the stirrer and allow the resin to settle to the bottom of the vials.

2.31. Withdraw as much of the liquid as practical from AN107C104-Kd2A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-2B. → wt = 13.003

11/22/99 M.J.L.

2.32. ~~Withdraw as much of the liquid as practical from AN107C104-Kd2B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-2C.~~

M.J.L. 11/22/99

2.33. Samples AN107C104-1A, AN107C104-1B, AN107C104-1C, AN107C104-2A, AN107C104-2B, and ~~AN107C104-2C~~ will be submitted for the analyses outlined in Table 1. M.J.L. 11/22/99

Part 3. Mixing C-104 Leachate With AN-107 At 1:1 Ratio

3.1. Prepare the sample vials according to the following table. All vials should be 20-mL HDPE.

Sample ID	Tare wt.
AN107C104-3A	8.0570 g.
AN107C104-3B	7.8562 g.
AN107C104-3C	8.0884 g.
AN107C104-4A	8.0161 g.
AN107C104-4B	8.1188 g.
AN107C104-4C	8.1273 g.

HD 10-29-99 ↓

3.2. Label two 20-mL HDPE vial as "AN107C104-Kd3A" and "AN107C104-Kd3B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

Wt. AN107C104-Kd3A = 8.5905 g (3.2a) APD 10-29-99 ↓
Wt. AN107C104-Kd3B = 8.5662 g (3.2b)

3.3. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107C104-Kd3A and AN107C104-Kd3B. Weigh each vial.

8.5905 + 0.0537 = 8.6442

Wt. AN107C104-Kd3A = __________ g (3.3a)
0.0537 g Wt. SuperLig 639 = 3.3a-3.2a = 0.0537 g (3.3b) APD 10-29-99
Wt. AN107C104-Kd3B = __________ g → 8.5662 + 0.0553 = 8.6215 (3.3c)
0.0553 g Wt. SuperLig 639 = 3.3c-3.2b = 0.0553 g (3.3d) APD - 10-29-99

3.4. Take vials AN107C104-Kd3A and AN107C104-Kd3B to the SAL and stage for subsequent steps.

3.5. Label a 40-mL glass vial as "AN107C104-TEST2" and place a magnetic stir bar in this vial. Weigh the vial with cap and stir bar.

Wt. AN107C104-TEST2 = 24.7054 g (3.5a) APD 10-29-99

→ AN107 Cs IX Batch Contact

3.6. Transfer 20 mL of sample AN107 to AN107C104-TEST2, then weigh the vial.

→ Reweigh in cell = 24.7058
(Balance 360-06-01-016)

Wt. AN107C104-TEST2 = __________ g (3.6a)
Wt. AN107 Added = 3.6a-3.5a = 49.6598 g (3.6b)
→ 49.6598 - 24.7058 = 24.9540

3.7. Transfer 20 mL of sample C104L to AN107C104-TEST2, then weigh the vial.

Wt. AN107C104-TEST2 = 70.6087 g (3.7a)
Wt. C104 Added = 3.7a-3.6a = 20.9489 g (3.7b)

Record the date and time at which the C104 was mixed with the AN107.

Date/Time: 11/29/99 11:00 (3.7c)

Also record the cell temperature.

Temperature = 23.5 °C (3.7d)

3.8. Stir the contents of AN107C104-TEST2. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/29/99 11:00 Solution cloudy

3.8A. After 24 h, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

11/30/99 14:30 Dark brown solid had settled to the bottom of the vial.

12/7/99 14:00 No change.

Notes

The first Tc K_d measurement must be started within 4 hours of mixing the C104 and AN107 solutions.

5 mL portions of the AN107/C104 mixture are to be transferred to vials AN107C104-3A, AN107C104-Kd3A, and AN107C104-Kd3B as indicated below. These transfers are to be done in such a manner that the exterior of the vials will be free of smearable contamination. Appropriate steps should be taken by the SAL personnel to ensure this.

11/29/99
n.l.l. 3.9. Filter 5.0 mL of the AN107/C104 mixture through a 0.45-μm nylon membrane and place the filtered solution in AN107C104-3A and remove the vial from the hot cell.

→ wt. = 13.929 (a)

3.10. Have an RCT determine the dose characteristics of sample AN107C104-3A

Radiological Hold Point

RCT perform radiological dose survey of sample AN107C104-3A. Corrected doses at contact, 15 cm, and 30 cm are required.

Note

Based on radiological dose of sample AN107C104-3A, a determination will be made as to whether to perform the K_d measurements in the lab or hot cell.

3.11. Contact CSM (G.J. Lumetta) to determine whether samples are to be transferred to lab 511 or 516.

3.12. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd3A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

3.13. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd3B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

3.14. If appropriate, transfer AN107C104-3A, AN107C104-Kd3A, and AN107C104-Kd3B to lab 511 or 516 (as directed by the cognizant scientist).

3.15. Weigh AN107C104-Kd3A and AN107C104-Kd3B.

Wt. AN107C104-Kd3A = 14.348 g (a) (3.15a)
Wt. Solution = 3.15a-3.3a = 5.7038 g (3.15b)
Wt. AN107C104-Kd3B = 14.390 g (a) (3.15c)
Wt. Solution = 3.15c-3.3c = 5.7485 g (3.15d)

3.16. Stir AN107C104-Kd3A and AN107C104-Kd3B for a minimum of 24 h

Start date/time: 11/29/99 14:00
Stop date/time: 12/6/99 14:15

(a) Balance 384-06-01-008

12/6/99 n.g.l.

3.17. Stop the stirrer and allow the resin to settle to the bottom of the vials.

3.18. Withdraw as much of the liquid as practical from AN107C104-Kd3A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-3B. wt ≐ 13.166 ⇐ See tot wt (g) on p. 19.

3.19. Withdraw as much of the liquid as practical from AN107C104-Kd3B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-3C. wt = 13.393

3.20. Allow the mixture in AN107C104-TEST2 to stand for two weeks

3.21. After 2 weeks, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

3.22. Label two 20-mL HDPE vial as "AN107C104-Kd4A" and "AN107C104-Kd4B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

Wt. AN107C104-Kd4A = 8.6061 g (3.22a) YPD 10-29-99

Wt. AN107C104-Kd4B = 8.5292 g (3.22b)

3.23. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AN107C104-Kd4A and AN107C104-Kd4B. Weigh each vial.

→ 8.6061 + 0.0550 = 8.6611

Wt. AN107C104-Kd4A = __________ g (3.23a)

0.0550 g. Wt. SuperLig 639 = 3.23a-3.22a = 0.0550 g (3.23b) YPD 10-29-99

→ 8.5292 + 0.0551 = 8.5843

Wt. AN107C104-Kd4B = __________ g (3.23c)

0.0551 g Wt. SuperLig 639 = 3.23c-3.22b = 0.0551 g (3.23d) YPD 10-29-99

3.24. Filter 5.0 mL of the AN107/C104 mixture through a 0.45-µm nylon membrane and place the filtered solution in AN107C104-4A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab. → wt = 13.487 Balance 384-06-01-029 12/14/99

3.25. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd4A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

3.26. Transfer 5.0 mL of the AN107/C104 mixture to AN107C104-Kd4B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

3.27. If appropriate, transfer AN107C104-4A, AN107C104-Kd4A, and AN107C104-Kd4B to lab 511 or 516 (as directed by the cognizant scientist).

3.28. Weigh AN107C104-Kd4A and AN107C104-Kd4B.

Wt. AN107C104-Kd4A = 14.607 g (3.28a)

Wt. Solution = 3.28a-3.23a = 5.746 g (3.28b)

Wt. AN107C104-Kd4B = 14.323 g (3.28c)

Wt. Solution = 3.28c-3.23c = 5.739 g (3.28d)

3.29. Stir AN107C104-Kd4A and AN107C104-Kd4B for a minimum of 24 h

Start date/time: 12/14/99 10:30

Stop date/time: 12/21/99 10:30

3.30. Stop the stirrer and allow the resin to settle to the bottom of the vials.

3.31. Withdraw as much of the liquid as practical from AN107C104-Kd4A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-4B. → wt = 13,524 (a)

3.32. Withdraw as much of the liquid as practical from AN107C104-Kd4B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AN107C104-4C. → wt = 13.366 (a)

3.33. Samples AN107C104-3A, AN107C104-3B, AN107C104-3C, AN107C104-4A, AN107C104-4B, and AN107C104-4C will be submitted for the analyses outlined in Table 1.

Part 4. Mixing C-104 Leachate With AW-101 At 10:1 Ratio

4.1. Prepare the sample vials according to the following table. All vials should be HDPE.

Sample ID
AW101C104-1A
AW101C104-1B
AW101C104-1C
AW101C104-2A
AW101C104-2B
AW101C104-2C

(a) Balance 384-06-01-008

4.2. Label two 20-mL HDPE vial as "AW101C104-Kd1A" and "AW101C104-Kd1B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

A.I.L.
9/17/99

Wt. AW101C104-Kd1A = 7.1795 g (a) (4.2a)

Wt. AW101C104-Kd1B = 7.1981 g (a) (4.2b)

4.3. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AW101C104-Kd1A and AW101C104-Kd1B. Weigh each vial.

A.I.L.
9/17/99

Wt. AW101C104-Kd1A = 7.2293 g (a) (4.3a)

Wt. SuperLig 639 = 4.3a-4.2a = 0.0498 g (4.3b)

Wt. AW101C104-Kd1B = 7.2504 g (a) (4.3c)

Wt. SuperLig 639 = 4.3c-4.2b = 0.05230 g (4.3d)

9/21/99
A.I.L.

4.4. Take vials AW101C104-Kd1A and AW101C104-Kd1B to the SAL and stage for subsequent steps.

4.5. Label a 40-mL glass vial as "AW101C104-TEST" and place a magnetic stir bar in this vial. Weigh the vial with cap and stir bar.

(Balance in SAL)

Wt. AW101C104-TEST = 24.8567 g (4.5a)

→ GL-060499B

4.6. Transfer 3 mL of sample AW101 to AW101C104-TEST, then weigh the vial.

Wt. AW101C104-TEST = 28.5483 g (4.6a)

Wt. AW101 Added = 4.6a-4.5a = 3.6916 g (4.6b)

→ label → "C104 Composite Lumetta"

4.7. Transfer 30 mL of sample C104L to AW101C104-TEST, then weigh the vial.

Wt. AW101C104-TEST = 60.7231 g (4.7a)

Wt. C104 Added = 4.7a-4.6a = 32.1748 g (4.7b)

Record the date and time at which the C104 was mixed with the AW101.

Date/Time: 9/21/99 10:15 (4.7c)

Also record the cell temperature.

Temperature = 23 °C (4.7d)

(a) Balance 362-06-01-043

4.8. Stir the contents of AW101C104-TEST. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

9/21/99
s.j.l.

No immediate reaction.

4.8A. After 24 h, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

9/22/99 12:50 → No change in the solution.
s.j.l.

Notes

The first Tc K_d measurement must be started within 4 hours of mixing the C104 and AW101 solutions.

5 mL portions of the AW101/C104 mixture are to be transferred to vials AW101C104-1A, AW101C104-Kd1A, and AW101C104-Kd1B as indicated below. These transfers are to be done in such a manner that the exterior of the vials will be free of smearable contamination. Appropriate steps should be taken by the SAL personnel to ensure this.

9/21/99
n.l.r. 4.9. Filter 5.0 mL of the AW101/C104 mixture through a 0.45-µm nylon membrane and place the filtered solution in AW101C104-1A and remove the vial from the hot cell.

Note: cap to -1A ~~[illegible]~~ had to be changed because 1st one was contaminated.

4.10. Have an RCT determine the dose characteristics of sample AW101C104-1A

Radiological Hold Point

RCT perform radiological dose survey of sample AW101C104-1A. Corrected doses at contact, 15 cm, and 30 cm are required.

Note

Based on radiological dose of sample AW101C104-1A, a determination will be made as to whether to perform the K_d measurements in the lab or hot cell.

4.11. Contact CSM (G.J. Lumetta) to determine whether samples are to be transferred to lab 511 or 516.

4.12. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd1A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

4.13. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd1B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

4.14. If appropriate, transfer AW101C104-1A, AW101C104-Kd1A, and AW101C104-Kd1B to lab 511 or 516 (as directed by the cognizant scientist).

4.15. Weigh AW101C104-Kd1A and AW101C104-Kd1B.

Balance: 384-06-01-005

Wt. AW101C104-Kd1A = 12.342 g (4.15a)

Wt. Solution = 4.15a-4.3a = 5.113 g (4.15b)

Wt. AW101C104-Kd1B = 12.426 g (4.15c)

Wt. Solution = 4.15c-4.3c = 5.176 g (4.15d)

wt. AW101C104-1A = 11.886

4.16. Stir AW101C104-Kd1A and AW101C104-Kd1B for a minimum of 24 h

stirrer set at 1000 rpm

Start date/time: 9/21/99 2:45

Stop date/time: 9/22/99 2:45

9/22/99

4.17. Stop the stirrer and allow the resin to settle to the bottom of the vials.

4.18. Withdraw as much of the liquid as practical from AW101C104-Kd1A and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-1B. → wt = 11.560

4.19. Withdraw as much of the liquid as practical from AW101C104-Kd1B and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-1C. → wt = 11.539

4.20. Allow the mixture in AW101C104-TEST to stand for two weeks

4.21. After 2 weeks, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

10/6/99 No change - Solution Clear

4.22. Label two 20-mL HDPE vial as "AW101C104-Kd2A" and "AW101C104-Kd2B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

M.A.A.
9/17/99

Wt. AW101C104-Kd2A = 7.2422 g(9) (4.22a)

Wt. AW101C104-Kd2B = 7.1915 g(9) (4.22b)

4.23. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AW101C104-Kd2A and AW101C104-Kd2B. Weigh each vial.

M.A.A.
9/17/99

Wt. AW101C104-Kd2A = 7.2935 g(9) (4.23a)

Wt. SuperLig 639 = 4.23a-4.22a = 0.0513 g (4.23b)

Wt. AW101C104-Kd2B = 7.2414 g(9) (4.23c)

Wt. SuperLig 639 = 4.23c-4.22b = 0.0499 g (4.23d)

Filter AW101C104-TEST through a 0.45-μm nylon membrane and place filtered solution in

4.24. ~~Transfer~~ 5.0 mL of the AW101/C104 mixture ~~to~~ AW101C104-2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

Transfer

4.25. ~~Filter~~ 5.0 mL of the AW101/C104 mixture ~~through a 0.45-μm nylon membrane and place the filtered solution~~ in AW101C104-Kd2A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

M.A.A.
10/6/99

4.26. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd2B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

(9) Balance 362-06-01-043

4.27. If appropriate, transfer AW101C104-2A, AW101C104-Kd2A, and AW101C104-Kd2B to lab 511 or 516 (as directed by the cognizant scientist).

4.28. Weigh AW101C104-Kd2A and AW101C104-Kd2B.

Wt. AW101C104-Kd2A = 12.501 g $^{(1)}$ (4.28a)
Wt. Solution = 4.28a-4.23a = 5.208 g (4.28b)
Wt. AW101C104-Kd~~Y~~2B = 12.706 g $^{(1)}$ (4.28c)
Wt. Solution = 4.28c-4.23c = 5.465 g (4.28d)
wt. AW101C104-2A = 11.772$^{(1)}$

4.29. Stir AW101C104-Kd2A and AW101C104-Kd2B for a minimum of 24 h

Start date/time: 10/6/99 12:35
Stop date/time: 10/7/99 12:35

Note: 10/7/99 @ 8:20 it was discovered that AW101C104-Kd2A had "walked" off the stirrer. Placed back on stirrer and stirred until 10/8/99 8:10

4.30. Stop the stirrer and allow the resin to settle to the bottom of the vials.

4.31. Withdraw as much of the liquid as practical from AW101C104-Kd2A and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-2B. → Wt. = ~~11.765~~ Wrong place N.A.L. 10/7/99 Wt = 11.578

4.32. Withdraw as much of the liquid as practical from AW101C104-Kd2B and filter through a 0.45-μm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-2C. → wt = 11.763$^{(1)}$

4.33. Samples AW101C104-1A, AW101C104-1B, AW101C104-1C, AW101C104-2A, AW101C104-2B, and AW101C104-2C will be submitted for the analyses outlined in Table 1.

Part 5. Mixing C-104 Leachate With AW-101 At 1:1 Ratio

5.1. Prepare the sample vials according to the following table. All vials should be HDPE.

Sample ID
AW101C104-3A
AW101C104-3B
AW101C104-3C
AW101C104-4A
AW101C104-4B
AW101C104-4C

(1) Balance 384-06-01-008

5.2. Label two 20-mL HDPE vial as "AW101C104-Kd3A" and "AW101C104-Kd3B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.

m.l.l.
9/17/99

Wt. AW101C104-Kd3A = 7.2155 g(a) (5.2a)

Wt. AW101C104-Kd3B = 7.2650 g(a) (5.2b)

5.3. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AW101C104-Kd3A and AW101C104-Kd3B. Weigh each vial.

m.l.l.
9/17/99

Wt. AW101C104-Kd3A = 7.2655 g(a) (5.3a)

Wt. SuperLig 639 = 5.3a-5.2a = 0.0500 g (5.3b)

Wt. AW101C104-Kd3B = 7.3174 g(a) (5.3c)

Wt. SuperLig 639 = 5.3c-5.2b = 0.0524 g (5.3d)

9/21/99
m.l.l.

5.4. Take vials AW101C104-Kd3A and AW101C104-Kd3B to the SAL and stage for subsequent steps.

5.5. Label a 40-mL glass vial as "AW101C104-TEST2" and place a magnetic stir bar in this vial. Weigh the vial with cap and stir bar.

(Balance in SAL)

Wt. AW101C104-TEST2 = 25.2172 g (5.5a)

5.6. Transfer 20 mL of sample AW101 to AW101C104-TEST2, then weigh the vial.

Wt. AW101C104-TEST2 = 50.0765 g (5.6a)

Wt. AW101 Added = 5:6a-5.5a = 24.8593 g (5.6b)

5.7. Transfer 20 mL of sample C104L to AW101C104-TEST2, then weigh the vial.

Wt. AW101C104-TEST2 = 71.8835 g (5.7a)

Wt. C104 Added = 5.7a-5.6a = 21.8070 g (5.7b)

Record the date and time at which the C104 was mixed with the AW101.

Date/Time: 9/21/99 10:20 (5.7c)

Also record the cell temperature.

Temperature = 23 °C (5.7d)

(a) Balance 362-06-01-043

5.8. Stir the contents of AW101C104-TEST2. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

9/21/99
M.J.L.

No immediate reaction

5.8A. After 24 h, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

9/22/99 12:50
M.J.L.

No change in the solution.

Notes

The first Tc K_d measurement must be started within 4 hours of mixing the C104 and AW101 solutions.

5 mL portions of the AW101/C104 mixture are to be transferred to vials AW101C104-3A, AW101C104-Kd3A, and AW101C104-Kd3B as indicated below. These transfers are to be done in such a manner that the exterior of the vials will be free of smearable contamination. Appropriate steps should be taken by the SAL personnel to ensure this.

9/21/99

5.9. Filter 5.0 mL of the AW101/C104 mixture through a 0.45-μm nylon membrane and place the filtered solution in AW101C104-3A and remove the vial from the hot cell.

Note: Had to change cap on -3A because 1st one contaminated.

5.10. Have an RCT determine the dose characteristics of sample AW101C104-3A

Radiological Hold Point

RCT perform radiological dose survey of sample AW101C104-3A. Corrected doses at contact, 15 cm, and 30 cm are required.

Note

Based on radiological dose of sample AW101C104-3A, a determination will be made as to whether to perform the K_d measurements in the lab or hot cell.

5.11. Contact CSM (G.J. Lumetta) to determine whether samples are to be transferred to lab 511 or 516.

5.12. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd3A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

5.13. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd3B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

5.14. If appropriate, transfer AW101C104-3A, AW101C104-Kd3A, and AW101C104-Kd3B to lab 511 or 516 (as directed by the cognizant scientist).

5.15. Weigh AW101C104-Kd3A and AW101C104-Kd3B.

Balance: 384.06.01-005

Wt. AW101C104-Kd3A = 12.85~~7~~4 g (5.15a)

Wt. Solution = 5.15a-5.3a = 5.588 g (5.15b)

Wt. AW101C104-Kd3B = 12.944 g (5.15c)

Wt. Solution = 5.15c-5.3c = 5.627 g (5.15d)

wt. AW101C104 -3A = 12.294

5.16. Stir AW101C104-Kd3A and AW101C104-Kd3B for a minimum of 24 h

↓ stirrer set at 1000rpm

Start date/time: 9/21/99 2:45

Stop date/time: 9/22/99 2:45

9/22/99

5.17. Stop the stirrer and allow the resin to settle to the bottom of the vials.

5.18. Withdraw as much of the liquid as practical from AW101C104-Kd3A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-3B. → wt = 12.001

5.19. Withdraw as much of the liquid as practical from AW101C104-Kd3B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-3C. → wt = 11.958

5.20. Allow the mixture in AW101C104-TEST2 to stand for two weeks

5.21. After 2 weeks, record any observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation.

10/6/99 No change - solution clear

5.22. Label two 20-mL HDPE vial as "AW101C104-Kd4A" and "AW101C104-Kd4B." Place a Teflon-coated magnetic stir bar in each of these vials. Weigh the vials with cap and stir bar.
n.j.l.
9/17/99

Wt. AW101C104-Kd4A = 7.2163 g (a) (5.22a)

Wt. AW101C104-Kd4B = 7.2179 g (a) (5.22b)

5.23. Weigh out 0.050 ± 0.005 g of SuperLig 639 resin into each AW101C104-Kd4A and AW101C104-Kd4B. Weigh each vial.
n.j.l.
9/17/99

Wt. AW101C104-Kd4A = 7.2673 g (a) (5.23a)

Wt. SuperLig 639 = 5.23a-5.22a = 0.0510 g (5.23b)

Wt. AW101C104-Kd4B = 7.2717 g (a) (5.23c)

Wt. SuperLig 639 = 5.23c-5.22b = 0.0538 g (5.23d)

AW101C104-TEST2

5.24. Filter 5.0 mL of the AW101/C104 mixture through a 0.45-µm nylon membrane and place the filtered solution in AW101C104-4A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

5.25. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd4A. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

5.26. Transfer 5.0 mL of the AW101/C104 mixture to AW101C104-Kd4B. Remove the vial from the hot cell, if the K_d measurement is to be done in the lab.

5.27. If appropriate, transfer AW101C104-4A, AW101C104-Kd4A, and AW101C104-Kd4B to lab 511 or 516 (as directed by the cognizant scientist).

(a) Balance 362-06-01-043

5.28. Weigh AW101C104-Kd4A and AW101C104-Kd4B.

Wt. AW101C104-Kd4A = 12.854 g (b) (5.28a)

Wt. Solution = 5.28a-5.23a = 5.587 g (5.28b)

Wt. AW101C104-Kd4B = 13.083 g (b) (5.28c)

Wt. Solution = 5.28c-5.23c = 5.811 g (5.28d)

wt. AW101C104-4A = 12.247 (b)

5.29. Stir AW101C104-Kd4A and AW101C104-Kd4B for a minimum of 24 h

Start date/time: 10/6/99 12:35

Stop date/time: 10/7/99 12:35

5.30. Stop the stirrer and allow the resin to settle to the bottom of the vials.

5.31. Withdraw as much of the liquid as practical from AW101C104-Kd4A and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-4B. → wt. = 12.277 (b)

5.32. Withdraw as much of the liquid as practical from AW101C104-Kd4B and filter through a 0.45-µm nylon membrane. The filtered solution is to be placed in sample vial AW101C104-4C. → wt.: 12.147 (b)

5.33. Samples AW101C104-3A, AW101C104-3B, AW101C104-3C, AW101C104-4A, AW101C104-4B, and AW101C104-4C will be submitted for the analyses outlined in Table 1.

Part 6. Mixing Entrained Solids Wash Liquors With AN-107 and AW-101

6.1. Label four 25-mL glass vials as indicated below and place a magnetic stir bar in each vial. Weigh the vials with caps and stir bars.

Wt. AN107Mix@1:10 = 17.0885 g (6.1a)

Wt. AN107Mix@1:1 = 17.0463 g (6.1b)

10-29-99 ↓

Wt. AW101Mix@1:10 = 22.1108 g (6.1c)

Wt. AW101Mix@1:1 = 22.2261 g (6.1d)

6/22/99 D.J.R. w/ GVH

→ 00-0074 AN107 CUF WASH COMP

6.2. Transfer 2 mL of sample AN107WASH to vial AN107Mix@1:10 then weigh.

Wt. AN107Mix@1:10 = 19.1430 g (6.2a)

Wt. Added = 6.2a-6.1a = 2.0545 g (6.2b)

(b) Balance 384-06-01-008

11/29/99

6.3 Transfer 10 mL of sample AN107WASH to vial AN107Mix@1:1 then weigh.

Wt. AN107Mix@1:1 = 27.6628 g (6.3a)

Wt. Added = 6.3a-6.1b = 10.6165 g (6.3b)

AN107 Cs IX Batch Contact

6.4. Transfer 20 mL of sample AN107 to vial AN107Mix@1:10 then weigh.

Note: Due to lack of material, only 15 mL could be used.

Wt. AN107Mix@1:10 = 37.9097 g (6.4a)

Wt. Added = 6.4a-6.2a = 18.7667 g (6.4b)

n.l.l. 11/29/99

6.5 Transfer 10 mL of sample AN107 to vial AN107Mix@1:1 then weigh.

Note: Due to lack of material, only ~7 mL used.

Wt. AN107Mix@1:1 = 35.4085 g (6.5a)

Wt. Added = 6.5a-6.3a = 7.7457 g (6.5b)

6.6. Transfer 2 mL of sample AW101WASH to vial AW101Mix@1:10 then weigh.
(GL-060499C)

Wt. AW101Mix@1:10 = 24.0944 g 6/22/99 n.l.l. w/ FVH (6.6a)

Wt. Added = 6.6a-6.1c = 1.9836 g (6.6b)

6.7 Transfer 10 mL of sample AW101WASH to vial AW101Mix@1:1 then weigh.
(GL-060499C)

(Note: step 6.9 done first)

Wt. AW101Mix@1:1 = 44.9612 g 6/22/99 (6.7a)

Wt. Added = 6.7a-6.1d = 10.1816 g (6.7b)

44.9612 - 34.7796 → 6.9a

6.8. Transfer 20 mL of sample AW101 to vial AW101Mix@1:10 then weigh.
(GL-060499B)

Wt. AW101Mix@1:10 = 49.0217 g 6/22/99 @ 8:20 (6.8a)

Wt. Added = 6.8a-6.6a = 24.9273 g (6.8b)

6.9 Transfer 10 mL of sample AW101 to vial AW101Mix@1:1 then weigh.
(GL-060499B)

Wt. AW101Mix@1:1 = 34.7796 g 6/22/99 (6.9a)

Wt. Added = 6.9a-6.7a = 12.5535 g (6.9b)

34.7796 - 22.2261

6.10. Stir the contents of AN107Mix@1:10, AN107Mix@1:1, AW101Mix@1:10, and AW101Mix@1:1. Record any immediate observations concerning chemical reactions (e.g., color change, foaming, gassing) and/or precipitation. Then monitor these solutions once every 24-h period (excluding weekends and holidays) for two weeks. Record observations below.

	Date/Time	Observations
M.J.L.	6/22/99 8:20	AW101Mix@1:10 Nothing unusual – Clear solution
	8:20	AW101mix@1:1 " " " "
M.J.L.	6/23/99 8:00	No changes in AW101Mix@1:10 or AW101mix@1:1
M.J.L.	6/24/99 8:40	"
M.J.L.	6/25/99 11:00	"
M.J.L.	6/28/99 3:30PM	"
M.J.L.	7/2/99 8:30	"
M.J.L.	7/16/99 2:15PM	"

M.J.L. 11/29/99 13:30 → AN107Mix@1:1 cloudy. A lighter colored layer floated on top at first. AN107Mix@1:10 Cloudy - - - - - - ⇒ Note: The AN107 solution appeared to be cloudy to begin with.

M.J.L. 11/30/99 14:30 cloudy, two "layers" had mixed. cloudy

Stirring stopped.

M.J.L. 12/2/99 15:00. Solutions no longer cloudy, solids had settled to the bottom of the vials.

M.J.L. 12/7/99 14:00 AN107Mix@1:1 ~~appears~~ appears the same, but the deposits in AN107Mix@1:10 appear to have dissolved. →

No. Solid still present on 12/17/99.

6.11. Prepare the sample vials according to the following table. Vials with the "-L" suffix should be HDPE, while those with the "-S" suffix should be glass.

Sample ID	Sample ID
AN107Mix@1:10-L	AN107Mix@1:10-S
AN107Mix@1:1-L	AN107Mix@1:1-S
AW101Mix@1:10-L	AW101Mix@1:10-S
AW101Mix@1:1-L	AW101Mix@1:1-S

6.12. Filter each mixture through a vacuum filtration unit with a 0.45-μm nylon membrane.

6.13. Place the filtered liquid fractions in the appropriate "-L" vials.

6.14. If solids are present on the filter, transfer these to the appropriate "-S" vial. Deionized water can be used to slurry the solids for transfer

6.15. Dry the solids to a constant weight at 105°C.

6.16. The samples are to be submitted for the analyses listed in Table 1.

See p. 33A

wt. immediately before submitting for analysis.

TABLE 1. Sample Matrix

	Sample ID	Acid Digestion	KOH Fusion	Na_2O_2 Fusion	ICP/AES	IC (anions)	TOC	TIC	ICP-MS (^{99}Tc)	GEA	^{90}Sr	Total Alpha	OH/pH
n.q.l. 11/23/99 (a)	AN107AW101-1A 14.288	X				X			X				X
	AN107AW101-1B 13,829	X							X				
	AN107AW101-1C 13.739	X							X				
	AN107AW101-2A 14.232	X				X			X				X
~~12.813~~	AN107AW101-2B 13.813	X							X				
	AN107AW101-2C 13.784	X							X				
	AN107C104-1A* 14.042	X				X			X				X
	AN107C104-1B 13.025	X							X				
	AN107C104-1C 13.048	X							X				
	AN107C104-2A 13.347	X				X			X				X
	AN107C104-2B 12.999	X n.q.l. 11/23/99							X				
	AN107C104-2C ~~12.999~~	X							X				
n.q.l. 12/21/99 (a)	AN107C104-3A 13.923	X				X			X				X
	AN107C104-3B 13.161	X							X				
	AN107C104-3C 13.384	X							X				
	AN107C104-4A 13.484	X				X			X				X
	AN107C104-4B 13.518	X							X				
	AN107C104-4C 13.359	X							X				
10/8/99 n.q.l. (a)	AW101C104-1A 11.818	X				X			X				X
	AW101C104-1B 11,470	X							X				
	AW101C104-1C 11,488	X							X				
	AW101C104-2A 11.761	X				X			X				X
	AW101C104-2B 11,578	X							X				
	AW101C104-2C 11.760	X							X				
	AW101C104-3A 12.21	X				X			X				X
	AW101C104-3B 11.934	X							X				
	AW101C104-3C 11.904	X							X				
	AW101C104-4A 12.343	X				X			X				X
	AW101C104-4B 12.221	X							X				
	AW101C104-4C 12.140	X							X				
	AN107Mix@1:10-L	X			X	X	X	X	X	X	X	X	
	AN107Mix@1:1-L	X			X	X	X	X	X	X	X	X	
	AW101Mix@1:10-L	X			X	X	X	X	X	X	X	X	
	AW101Mix@1:1-L	X			X	X	X	X	X	X	X	X	
	AN107Mix@1:10-S		X	X	X	X	X	X		X	X	X	
	AN107Mix@1:1-S		X	X	X	X	X	X		X	X	X	
	AW101Mix@1:10-S		X	X	X	X	X	X		X	X	X	
	AW101Mix@1:1-S		X	X	X	X	X	X		X	X	X	

(a) Balance 384-06-01-008

12/17/99
M.J.L.

AN107 Mix@1:1 and AN107 Mix@1:10 both had dark solids on the bottom of the vial.

Because there was not a lot of solids, we decided a ~~ce~~ centrifuge/decant would be a better approach than filtration.

		Tare	Wt. of Liquid
Two vials labeled as	"AN107 Mix@1:1 Liquid"	17.3338	34.8176
	"AN107 mix@1:10 Liquid"	17.2936	37.4460

Centrifuged AN107 Mix@1:1 and AN107 Mix@1:10.
Using pipette, liquid decanted to AN107 Mix@1:1 Liquid and AN107 Mix@1:10 Liquid, respectively.

Wts after transfer

AN107 Mix@1:1 = 17.8240
@1:10 = 17.6865

(Probably a mL or so liquid carried over with the solids.)

* Note: When liquids submitted for analysis, indicate on ASR that they should be filtered with 0.45-µm prior to analysis.

Solids in AN107 mix@1:1 and AN107 mix@1:10 dried at 105°C

Wt. after drying: AN107 mix@1:1 = 17.1663
AN107 mix@1:10 = 17.2573

wt. solids: AN107 mix@1:1 = 17.1663 − 17.0463 = 0.1200 g
AN107 mix@1:10 = 17.2573 − 17.0885 = 0.1688 g

Appendix B. Analytical Reports

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File
Mike Urie

Date November 23, 1999

To Gregg Lumetta

From Tom Farmer Thomas Farmer

Subject <u>ICP/MS Analysis of Submitted Samples</u>
(ACL #00-0043 through 00-0054)

Pursuant to your request, the 13 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 10x to 50x from the dilutions received. The results include your dilutions and are reported in ng analyte/ g (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.

JP Brown
11/23/99

Gregg Lumetta Tc-99 Analysis

November 23, 1999

Results are reported in ng analyte/ g (ppb) of original sample.
Uncertainty of the results is estimated at ±10%.

Sample ID	Client ID	ICP/MS Number	Tc-99 ng/g	*Ru-101 ng/g
1%HNO3		9b22b1	<2	
1%HNO3		9b22b22	<2	
00-0054PB	Process Blank	9b22b7	2.7±0.8	0.7
00-0043	AW101C104-1A	9b22b17	386	180
00-0044	AW101C104-1B	9b22b8	77.7	210
00-0045	AW101C104-1C	9b22b9	67.5	190
00-0046	AW101C104-2A	9b22b16	394	180
00-0047	AW101C104-2B	9b22b10	65.7	190
00-0047 + spike	AW101C104-2B	9b22b20	269	190
Spike Recovery			**111%**	
00-0048	AW101C104-2C	9b22b11	61.8	190
00-0049	AW101C104-3A	9b22b18	1810	530
00-0049 Dup.	AW101C104-3A	9b22b21	1750	510
00-0050	AW101C104-3B	9b22b13	321	550
00-0051	AW101C104-3C	9b22b14	328	550
00-0052	AW101C104-4A	9b22b19	1690	490
00-0053	AW101C104-4B	9b22b12	296	560
00-0054	AW101C104-4C	9b22b15	313	530
CCV results are reported in ng/ml (ppb)				
2.5ppb Tc-99 CCV		9b22b4	2.60	
2.5ppb Tc-99 CCV		9b22b23	2.60	
5ppb Co		9b22b24	<2	

*Calculated using response from indium. For information only.

DATA REVIEW

Reviewed by

Date: 23Nov99 Pages: 1 of 1

Gregg Lumetta Tc-99 Analysis

November 23, 1999(Revised 11/30/99)

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

Sample ID	Client ID	ICP/MS Number	Tc-99 ng/ml	*Ru-101 ng/ml
1%HNO3		9b22b1	<2	
1%HNO3		9b22b22	<2	
00-0054PB	Process Blank	9b22b7	2.8±0.8	0.7
00-0043	AW101C104-1A	9b22b17	418	190
00-0044	AW101C104-1B	9b22b8	84.0	230
00-0045	AW101C104-1C	9b22b9	73.3	210
00-0046	AW101C104-2A	9b22b16	430	200
00-0047	AW101C104-2B	9b22b10	71.4	210
00-0047 +	AW101C104-2B	9b22b20	292	210
Spike Recovery			110%	
00-0048	AW101C104-2C	9b22b11	67.5	210
00-0049	AW101C104-3A	9b22b18	2100	610
00-0049 Du	AW101C104-3A	9b22b21	2030	590
00-0050	AW101C104-3B	9b22b13	373	640
00-0051	AW101C104-3C	9b22b14	379	640
00-0052	AW101C104-4A	9b22b19	2010	580
00-0053	AW101C104-4B	9b22b12	344	650
00-0054	AW101C104-4C	9b22b15	358	610
CCV results are reported in ng/ml (ppb)				
2.5ppb Tc-99 CCV		9b22b4	2.60	
2.5ppb Tc-99 CCV		9b22b23	2.60	
5ppb Co		9b22b24	<2	

*Calculated using response from indium. For information only.

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File
Mike Urie

Date January 24, 2000

To Gregg Lumetta

From Tom Farmer Orville Thomas Farmer III
25Jan00

Subject <u>ICP/MS Analysis of Submitted Samples</u>
(ACL #00-00770 through 00-00775, 00-00778 through 00-00779)

Pursuant to your request, the 12 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 to 10x from the dilutions received. The results include your dilutions and are reported in both ng analyte/ ml (ppb) and ng analyte/ g (ppb) of the original sample ± one standard deviation.

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 372-0700 or James Bramson at 372-0624.

Lumetta Tc-99 Analysis

January 24, 2000

1/25/00

Results are reported in ng/ml (ppb) and ng/g (ppb) of original sample.

Sample ID	Client ID	ICP/MS Number	Tc-99 ng/ml	±	1SD	Tc-99 ng/g	±	1SD	^{101}Ru/^{102}Ru (*.541)	†^{101}Ru ng/ml
1%HNO3		00121a1	<0.1			<0.1				
1%HNO3		00121a21	<0.1			<0.1				
Blank		00121a23	<2.2			<2.2				
Blank Filter		00121a24	<1.5			<1.5				
00-00770	AN107C104-3A	00121a10	1490	±	5	1310	±	5	1.120	1700
00-00770 DUP	AN107C104-3A	00121a11	1430	±	50	1260	±	44	1.091	1600
00-00771	AN107C104-3B	00121a12	1140	±	81	998	±	71	1.097	1600
00-00772	AN107C104-3C	00121a13	1190	±	57	1040	±	50	1.114	1700
00-00773	AN107C104-4A	00121a14	1430	±	44	1240	±	38	1.120	1700
00-00774	AN107C104-4B	00121a15	1110	±	82	959	±	71	1.091	1600
00-00775	AN107C104-4C	00121a16	1120	±	13	980	±	11	1.088	1700
00-00775 + spike	AN107C104-4C	00121a20	2220	±	109	1950	±	96		
Spike Recovery			110%			111%				
00-00778	AN107Mix@1:1 Liquid	00121a17	1230	±	43	1100	±	38	1.124	1500
00-00778 DUP	AN107Mix@1:1 Liquid	00121a18	1240	±	44	1110	±	40	1.103	1400
00-00779	AN107Mix@1:10 Liquid	00121a19	2500	±	49	2040	±	40	1.098	2900
CCV results are reported in ng/ml (ppb)										
5ppb Tc-99 CCV		00121a5	4.79	±	0.1	4.79	±	0.1		
5ppb Tc-99 CCV		00121a25	5.00	±	0.1	5.00	±	0.1		
50ppb Co		00121a22	<0.1			<0.1				

* Natural ^{101}Ru/^{102}Ru ratio.

†Based on response from indium

DATA REVIEW

Reviewed by:

Date: 25Jan00 Pages: 1 of 1

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

ASR # 5533
WP# W45526

File: L:\radchem\hydroxide\asr5533
Analysis Date: 11/17/1999
Print Date: 11/24/99

Hydroxide and Alkalinity Determination
Governing Procedures: **PNL-ALO-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843**
Lab Loc. 525

Analyst: 11/24/99
Reviewer: 11/29/99

Titrant	Molarity
HCl	**0.2034**

Std. & Spike	Molarity
NaOH	**0.1018**

								OH				
								1st Equivalence				
RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0043	AW101C104-1A		0.100	0.1066	1.066	4	10.837	0.242	9.473	0.049	0.49	
00-0043	AW101C104-1A	Replicate	0.200	0.2105	1.053	6	11.205	0.459	9.695	0.093	0.47	
00-0043	AW101C104-1A	Replicate	0.300	0.3149	1.050	7	11.417	0.633	10.162	0.129	0.43	13.69%
00-0046	AW101C104-2A		0.300	0.3171	1.057	8	11.531	0.663	10.242	0.135	0.45	
00-0046	AW101C104-2A	Replicate	0.300	0.3182	1.061	9	11.619	0.683	9.889	0.139	0.46	2.97%
00-0049	AW101C104-3A		0.300	0.3399	1.133	10	11.763	1.292	10.436	0.263	0.88	
00-0049	AW101C104-3A	Replicate	0.300	0.3422	1.141	11	11.753	1.254	10.417	0.255	0.85	2.99%
00-0052	AW101C104-4A		0.300	0.3403	1.134	12	11.587	1.241	10.307	0.252	0.84	
00-0052	AW101C104-4A	Replicate	0.300	0.3415	1.138	13	11.733	1.162	10.415	0.236	0.79	6.58%
QC Data:												
Reag. Blk.			5.00			1					**OH % Recovery**	
Standard 1	0.1018 N NaOH		5.000	5.0281	1.006	2	11.663	2.208	10.358	0.4491	**88.2%**	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0178	1.004	3	11.89	2.206	10.366	0.4487	**88.2%**	Std 2
00-0043MS	C104-1A + 2mL 0.1N NaOH		0.200	0.2109	1.055	6	11.403	1.09	10.354	0.222	**63.4%**	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Performance checks

Balance #	360--01-06-037	Vol.	Wt.
Pipet #	H30762	5.00	4.944
Pipet #	2734494	0.500	0.496
Pipet #	120737	0.100	0.1013
Pipet #	120737	0.200	0.1997

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

ASR # 5533
WP# W45526

File: L:\radchem\hydroxide\asr5533
Analysis Date: **11/17/1999**
Print Date: 11/24/99

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Lo 525

Analyst: 11/24/99
Reviewer: 11/24/99

Titrant	Molarity
HCl	0.2034

			CO3					HCO3				
			2nd Equivalence					3rd Equivalence				
RPG #		Sample Vol. (mL	Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0043	0	0.100	0.422	6.816	0.037	0.366		0.501	4.891	0.016	0.16	
00-0043	Replicat	0.200	0.807	7.000	0.071	0.354		0.899	5.685	0.019	0.09	
00-0043	Replicat	0.300	1.145	7.803	0.104	0.347	5.32%	1.356	5.756	0.043	0.14	41.8%
00-0046	0	0.300	1.211	7.325	0.111	0.372		1.350	5.868	0.028	0.09	
00-0046	Replicat	0.300	1.192	7.393	0.104	0.345	7.38%	1.394	5.521	0.041	0.14	37.0%
00-0049	0	0.300	2.253	7.778	0.195	0.652		2.692	4.719	0.089	0.30	
00-0049	Replicat	0.300	2.220	7.937	0.196	0.655	0.52%	2.649	5.050	0.087	0.29	2.30%
00-0052	0	0.300	2.221	7.727	0.199	0.664		2.678	4.761	0.093	0.31	
00-0052	Replicat	0.300	2.187	7.876	0.208	0.695	4.49%	2.695	4.741	0.103	0.34	10.57%
						CO3 % Recovered				**HCO3 % recovered**		
Standard 1		5.000	2.439	7.825	0.04699	**9.2%**	sample	2.652	3.74	0.0433	**8.5%**	
Standard 2		5.000	2.436	7.836	0.04678	**9.2%**	sample	2.648	3.579	0.0431	**8.5%**	
00-0043MS		0.200	1.648	7.683	0.1135	159.5%	sample	1.997	5.226	0.0710	279.2%	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 5606
Client: G. Lumetta
WP# W45526

File: L:\radchem\hydroxide\asr5606
Analysis Date: 1/4/2000
Print Date: 1/7/00

Governing Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843** Lab Loc. 525

Analyst: 1-7-00
Reviewer: 1-7-00

Titrant	Molarity
HCl	**0.2034**
pH 7.0 reading =	6.92

Std. & Spik	Molarity
NaOH	**0.1018**

								OH				
								1st Equivalence				
RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0499	**AN107AW101-1A**		0.300	0.3702	1.234	4	12.330	2.827	10.491	0.575	1.92	
00-0499	**AN107AW101-1A**	Replicate	0.300	0.3727	1.242	5	12.267	2.82	10.535	0.574	1.91	0.25%
00-0502	**AN107AW101-2A**		0.300	0.3692	1.231	6	12.361	2.809	10.495	0.571	1.90	
00-0502	**AN107AW101-2A**	Replicate	0.200	0.2435	1.218	7	12.164	1.874	10.576	0.381	1.91	0.07%
00-0505	**AN107C104-1A**		0.300	0.3181	1.060	8	11.803	0.547	10.452	0.111	0.37	
00-0505	**AN107C104-1A**	Replicate	0.500	0.5327	1.065	9	11.902	0.906	10.586	0.184	0.37	0.62%
00-0508	**AN107C104-2A**		0.500	0.5352	1.070	10	11.903	0.916	10.493	0.186	0.37	
00-0508	**AN107C104-2A**	Replicate	0.500	0.5361	1.072	11	11.889	0.874	10.553	0.178	0.36	4.69%
QC Data:												
Reag. Blk.1			5.00			1	2.697					
Reag. Blk.2			5.00			14	4.328				**OH % Recovery**	
Standard 1	0.1018 N NaOH		5.000	5.034	1.007	2	11.426	2.428	10.457	0.4939	**97.0%**	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0464	1.009	3	12.088	2.391	10.601	0.4863	**95.5%**	Std 2
00-0499MS	00-0499 + 2mL 0.1N NaOH		0.100	0.1250	1.250	15	11.650	1.884	10.142	0.383	**94.2%**	MS
00-0508MS	00-0508 + 2mL 0.1N NaOH		0.500	0.5337	1.067	16	11.818	1.847	10.462	0.376	**95.1%**	MS

Performance checks using Balance # 360--01-06-037

Pipet #	Vol.	Wt.
H30762	5.00	4.9796
288618	0.300	0.2971
120737	0.100	0.0992
120737	0.500	0.5009

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
0

ASR # 5606

WP# W45526

File: L:\radchem\hydroxide\asr5606
Analysis Date: 01/04/2000
Print Date: 1/6/00

Governing Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: 1-6-2000

Reviewer: 1-6-00

Titrant	Molarity
HCl	0.2034

			CO3					HCO3				
RPG #		Sample Vol. (mL	2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0499	0	0.300	3.899	7.610	0.218	0.727		4.187	5.071	0.059	0.20	
00-0499	Replica	0.300	3.902	7.615	0.220	0.734	0.93%	4.195	5.055	0.060	0.20	1.7%
00-0502	0	0.300	3.866	7.616	0.215	0.717		4.153	5.049	0.058	0.19	
00-0502	Replica	0.200	2.613	7.557	0.150	0.752	4.76%	2.805	5.055	0.039	0.20	0.3%
00-0505	0	0.300	1.076	7.513	0.108	0.359		1.306	5.720	0.047	0.16	
00-0505	Replica	0.500	1.723	8.188	0.166	0.332	7.61%	2.236	5.389	0.104	0.21	28.93%
00-0508	0	0.500	1.756	7.878	0.171	0.342		2.234	5.366	0.097	0.19	
00-0508	Replica	0.500	1.704	8.172	0.169	0.338	1.20%	2.683	3.792	0.199	0.40	68.77%
						CO3 % Recovered				**HCO3 % recovered**		
Standard 1		5.000	2.598	7.926	0.03458	**6.8%**	sample	2.735	3.909	0.0279	**5.5%**	
Standard 2		5.000	2.588	7.826	0.04007	**7.9%**	sample	2.738	4.144	0.0305	**6.0%**	
00-0499MS		0.100	2.269	6.899	0.07831	**107%**	sample	2.375	4.713	0.0216	**109%**	sample
00-0508MS		0.500	2.686	8.141	0.17065	**100%**	sample	3.358	4.778	0.1367	**92%**	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

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

ASR # **5644**

WP# **W45526**

File: L:\radchem\hydroxide\asr# 5644
Analysis Date:
Print Date: 2/10/00

Hydroxide and Alkalinity Determination
Governing Procedures: **PNL-ALO-228:** Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843**
Lab Loc. 525

Analyst: 2/10/00
Reviewer: 2/10/00

Titrant	Molarity
HCl	**0.2034**

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	OH 1st Equivalence Point pH	Found millimoles base	Molarity base	millimole RPD
00-00770	**AN107-C104-3A**		0.200	0.2339	1.170	4	10.680	0.538	9.808	0.109	0.55	
00-00770	**AN107-C104-3A**	Replicate	0.500	0.5824	1.165	5	11.194	1.276	10.128	0.260	0.52	5.27%
00-00773	**AN107-C104-4A**		0.500	0.5779	1.156	6	11.501	1.217	10.659	0.248	0.50	
00-00773	**AN107-C104-4A**	Replicate	0.300	0.3478	1.159	7	11.491	0.845	9.849	0.172	0.57	14.58%
QC Data:												
Reag. Blk.			5.00			1	2.309				**OH % Recovery**	
Standard 1	0.1018 N NaOH		5.000	4.9959	0.999	2	10.217	2.425	7.877	0.4932	**96.9%**	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0017	1.000	3	11.376	2.406	7.396	0.4894	**96.1%**	Std 2
00-0773MS	An-107 + 2mL 0.1N NaOH		0.200	0.2311	1.156	8	11.338	1.429	10.083	0.291	**90.4%**	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Performance checks
Balance # 360--01-06-037

		Vol.	Wt.
Pipet #	H30762	5.00	5.008
Pipet #	2734494	0.500	0.4953
Pipet #	120737	0.100	0.2018
Pipet #	120737	0.200	

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

ASR # 5644

WP# W45526

File: L:\radchem\hydroxide\asr#
Analysis Date: 01/00/1900
Print Date: 02/10/2000

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: 2-10-00
Reviewer: 2/10/00

Titrant	Molarity
HCl	**0.2034**

				CO3					**HCO3**				
RPG #	Sample ID		Sample Vol. (mL)	**2nd Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	**3rd Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-00770	AN107-C104-3A	0	0.200	1.141	7.008	0.123	0.613		1.619	4.474	0.097	0.49	
00-00770	AN107-C104-3A	Replicate	0.500	2.797	7.381	0.309	0.619	0.89%	3.991	4.608	0.243	0.49	0.1%
00-00773	AN107-C104-4A	0	0.500	2.776	7.474	0.317	0.634		3.978	4.617	0.244	0.49	
00-00773	AN107-C104-4A	Replicate	0.300	1.657	7.401	0.165	0.551	14.12%	2.422	4.438	0.156	0.52	5.9%
							CO3 % Recovered						
Standard 1	0.1018 N NaOH		5.000	2.481	4.32	0.01139	0.002	sample					
Standard 2	0.1018 N NaOH		5.000	2.471	4.136	0.01322	0.003	sample			**HCO3 % recovered**		
00-0773MS	An-107 + 2mL 0.1N NaOH		0.200	2.083	7.246	0.13302	**108.0%**	sample	2.607	4.49	0.1066	**109.7%**	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0..100-mL of sample for each matrix spilke.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	G. Lumetta	**Charge Code/Project:**	W45526 / 29953
ACL Numbers:	00-0043, -0046, -0049, -0052	**ASR Number:**	5533
Analyst:	MJ Steele	**Analysis Date:**	November 01-03, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

		F	**Cl**	**NO_2**	**Br**	**NO_3**	**PO_4**	**SO_4**	**C_2O_4**
Lab ID	**Sample ID**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**
00-0043	**AW101C104-1A**	2,600	200	4,900	< 125	8,400	430	360	510
00-0043 Rep	**AW101C104-1A Rep**	2,600	190	4,900	< 125	8,400	420	330	500
	RPD	0%	5%	0%	n/a	0%	2%	7%	0%
00-0046	**AW101C104-2A**	2,700	210	5,300	< 125	9,000	450	< 250	540
00-0049	**AW101C104-3A**	1,800	1,100	22,400	< 125	47,000	< 500	< 500	< 500
00-0052	**AW101C104-4A**	1,800	1,200	22,700	< 125	47,500	< 500	< 500	< 500
	00-0052 MS Rec	94%	98%	104%	101%	112%	104%	107%	105%
	Blank Spike Rec	101%	98%	104%	106%	106%	106%	108%	106%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS Rec = Matrix Spike Standard % recovery
Blank Spike Rec = Blank Spike Standard % recovery

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation up to 2,000-fold to ensure that all anions were within the calibration range.

Q.C. Comments:

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the relative percent differences (RPD) between replicates are well within the acceptance criteria of 20% for all anions measured above the EQL.

Matrix Spike: A matrix spike was prepared and measured for sample AW101C104-4A. The spike recoveries for all anions are within the 75% to 125% recovery acceptance criteria.

Blank Spike: The blank spike is used as the laboratory control sample and recovered within the acceptance criteria of 80% to 120%.

System Blank/Processing Blanks: Approximately ten system blanks were process during the analysis of the samples. With the exception of only single nitrate value, no anions were detected above reportable concentrations in the system blanks. Since the nitrate results are high, this single QC failure does not affect the reported nitrate results.

Quality Control Calibration Verification Check Standards: Approximately ten mid-range verification standards were analyzed throughout the analysis runs. Except for a single phosphate value, the reported results for all analytes of interest were recovered within the acceptance criteria of ±10% for the verification standard. The one phosphate result recovered at +11% above the true value. This single phosphate failure has no impact on the reported results.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst: MJ Steele

Approval: ________ **Date** 11-12-99

Archive Information:

Files: ASR 5533 Lumetta.doc ASR 5463 5533 -36 -68 -71.xls

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File
Mike Urie

Date November 23, 1999

To Gregg Lumetta

From Tom Farmer Orvill Thomas Farmer III 13Dec99

Subject <u>ICP/MS Analysis of Submitted Samples</u>
(ACL #00-0499 through 00-0509)

Pursuant to your request, the 13 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 20x from the dilutions received (10x). The results include your dilutions and are reported in ng analyte/ 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. Semiquantitative Ru concentrations, corrected for sample dilution, 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.

12/13/99

Lumetta Tc-99 Analysis

December 9, 1999

Results are reported in ng analyte/ ml (ppb) of original sample.
Unless otherwise specified, the uncertainty of the results is estimated at ±10%.

Sample ID	ICP/MS Number	Tc-99 ng/ml	^{101}Ru/^{102}Ru (*0.541)	†^{101}Ru ng/ml
1%HNO3	9c08a1	2.6±0.9		
1%HNO3	9c08a7	6.2±3.9		
1%HNO3	9c08a23	8.1±2.9		
00-00499 PB1	9c08a8	4.4±2.2	2.318	2
00-00499 PB2	9c08a9	6.24	0.659	1
00-00499	9c08a21	3760	1.169	1100
00-00500	9c08a15	929	1.186	1200
00-00501	9c08a16	929	1.178	1200
00-00502	9c08a22	3970	1.167	1200
00-00503	9c08a17	998	1.187	1200
00-00503 DUP	9c08a19	1030	1.189	1200
00-00504	9c08a18	1060	1.176	1200
00-00505	9c08a13	308	1.152	530
00-00505 + spike	9c08a20	1370	1.185	520
Spike Recovery		**106%**		
00-00506	9c08a11	224	1.148	560
00-00507	9c08a12	245	1.104	560
00-00508	9c08a14	310±50	1.166	540
00-00509	9c08a10	234	1.110	560
4.5ppb Tc-99 CCV	9c08a24	4.34		
4.5ppb Tc-99 CCV	9c08a5	4.29		
30ppb Co	9c08a26	4.6±1.2		

* Natural ^{101}Ru/^{102}Ru ratio.

†Based on response from indium

DATA REVIEW

Reviewed by: O.J. Farmer III

Date: 13Dec99 Page: 1 of 1

12/13/99

Lumetta Tc-99 Analysis

December 9, 1999

Results are reported in ng analyte/ ml (ppb) of original sample.
Unless otherwise specified, the uncertainty of the results is estimated at ±10%.

Sample ID	ICP/MS Number	Tc-99 ng/ml	^{101}Ru/^{102}Ru (*0.541)	†^{101}Ru ng/ml
1%HNO3	9c08a1	2.6±0.9		
1%HNO3	9c08a7	6.2±3.9		
1%HNO3	9c08a23	8.1±2.9		
1W107AW101-1A 00-00499 PB1	9c08a8	4.4±2.2	2.318	2
-1A 00-00499 PB2	9c08a9	6.24	0.659	1
-1A 00-00499	9c08a21	3760	1.169	1100
-1B 00-00500	9c08a15	929	1.186	1200
-1C 00-00501	9c08a16	929	1.178	1200
-2A 00-00502	9c08a22	3970	1.167	1200
-2B 00-00503	9c08a17	998	1.187	1200
-2B 00-00503 DUP	9c08a19	1030	1.189	1200
-2C 00-00504	9c08a18	1060	1.176	1200
N107C104-1A* 00-00505	9c08a13	308	1.152	530
-1A* 00-00505 + spike	9c08a20	1370	1.185	520
Spike Recovery		106%		
-1B 00-00506	9c08a11	224	1.148	560
-1C 00-00507	9c08a12	245	1.104	560
-2A 00-00508	9c08a14	310±50	1.166	540
-2B 00-00509	9c08a10	234	1.110	560
4.5ppb Tc-99 CCV	9c08a24	4.34		
4.5ppb Tc-99 CCV	9c08a5	4.29		
30ppb Co	9c08a26	4.6±1.2		

* Natural ^{101}Ru/^{102}Ru ratio.

†Based on response from indium

DATA REVIEW

Reviewed by: O. J. Farmer III

Date: 13Dec99 Pages: 1 of 1

Lumetta Tc-99 Analysis

December 9, 1999 (revised 12/16/99)

Results are reported in ng analyte/ ml (ppb) of original sample.
Unless otherwise specified, the uncertainty of the results is estimated at ±10%.

Sample ID	ICP/MS Number	Density (g/ml)	Tc-99 ng/g	^{101}Ru/^{102}Ru (*0.541)	†^{101}Ru ng/g
1%HNO3	9c08a1		2.6±0.9		
1%HNO3	9c08a7		6.2±3.9		
1%HNO3	9c08a23		8.1±2.9		
00-00499 PB1	9c08a8	1	4.4±2.2	2.318	2
00-00499 PB2	9c08a9	1	6.24	0.659	1
00-00499	9c08a21	1.2354	3040	1.169	890
00-00500	9c08a15	1.2346	753	1.186	970
00-00501	9c08a16	1.2325	754	1.178	970
00-00502	9c08a22	1.2341	3220	1.167	970
00-00503	9c08a17	1.2366	807	1.187	970
00-00503 DUP	9c08a19	1.2366	833	1.189	970
00-00504	9c08a18	1.2327	860	1.176	970
00-00505	9c08a13	1.0642	289	1.152	500
00-00505 + spike	9c08a20	1.0642	1290	1.185	490
Spike Recovery			107%		
00-00506	9c08a11	1.0547	212	1.148	530
00-00507	9c08a12	1.0573	232	1.104	530
00-00508	9c08a14	1.0631	290±50	1.166	510
00-00509	9c08a10	1.0498	223	1.110	530
CCV results are reported in ng/ml (ppb)					
4.5ppb Tc-99 CCV	9c08a24		4.34		
4.5ppb Tc-99 CCV	9c08a5		4.29		
30ppb Co	9c08a26		4.6±1.2		

* Natural ^{101}Ru/^{102}Ru ratio.

†Based on response from indium

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	G. Lumetta	**Charge Code/Project:**	W45526 / 29953
ACL Numbers:	00-0499, -0502, -0505, -0508	**ASR Number:**	5606
Analyst:	MJ Steele	**Analysis Date:**	December 18-20, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

		F	**Cl**	**NO2**	**Br**	**NO3**	**PO4**	**SO4**	**C2O4**
Lab ID	**Sample ID**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**	**µg/ml**
00-00499	**AN107-AW101-1A**	830	< 250	46,000	< 250	101,000	< 500	1,100	< 500
00-00502	**AN107-AW101-2A**	970	< 250	45,300	< 250	98,700	670	1,200	< 500
00-00505	**AN107-C104-1A**	3,000	< 25	4,800	< 25	12,100	430	520	600
00-00508	**AN107-C104-2A**	3,700	< 25	4,500	26	11,300	420	520	600
Analytical Run Matrix Spike Results									
00-00615 MS	*N7-Tc-Elu-Comp MS Rec*	*94%*	*OvrRng*	*101%*	*103%*	*103%*	*103%*	*103%*	*101%*
00-00626 MS	*N7-Tc-0 MS Rec*	*112%*	*105%*	*122%*	*108%*	*OvrRng*	*111%*	*114%*	*111%*
00-00741 MS	*1-1 Rad MS Rec*	*OvrRng*	*113%*	*110%*	*111%*	*OvrRng*	*109%*	*114%*	*110%*

MS Rec = Matrix Spike Standard % recovery

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation up to 4,000-fold to ensure that all anions were within the calibration range.

Q.C. Comments:

Duplicates: No actual sample duplicates were provided to the laboratory for analysis.

Matrix Spike: No matrix spikes were prepared from the samples submitted under this ASR. However, samples from other ASR analyzed within the analytical run were within the 75% to 125% recovery acceptance criteria for those anions measured within the calibration range.

Blank Spike: No blank spikes were analyzed within the analytical runs.

System Blank/Processing Blanks: Twelve system blanks were process during the analysis of the samples. No anions were detected in the system blanks above the estimate quantitation level (i.e., the lowest calibration standard).

Quality Control Calibration Verification Check Standards: Seven mid-range verification standards were analyzed throughout the analysis runs. Except for only two oxalate values, the reported results for all anions of interest were recovered within the acceptance criteria of ±10% for the verification standard.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst: MJ Steele Date 1/13/00

Approval: MW Urie Date 1/19/00

Archive Information:

Files: ASR 5606 Lumetta.doc	ASR 5606 5626 5642.xls

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

Project: 29953
Client: G. Lumetta

RPL ID#: 00-00776 through 00-00779

Client ID: "AN107 MIX @ 1:1" through "AN107 MIX @ 1:10 LIQUID"

ASR Number: 5644

Total Samples: 4

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

Analyst: D.R. Sanders

Analysis Date (Filename): 01-27-00 (A0577)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry L Wagner 2-21-00
Reviewed by

2-22-00
Concur

2/17/00

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

Two radioactive solid samples, **AN107 MIX @ 1:1** and **AN107 MIX @ 1:10** (RPL ID# 00-00776 and 00-00777), were analyzed by ICPAES after preparation by the 325 Shielded Analytical Laboratory (SAL). Samples were prepared using PNL-ALO-129 acid digestion of solids procedure. Approximately 0.1 grams of sample were processed and diluted to a final volume of about 16ml. Concentrations reported for solids in µg/g have been corrected for process and analytical dilution. See attached bench sheet included with this report for actual weight and volumes used. The process blank for solids was calculated using the average weight of the two solid samples (0.1092 g) and a final volume of 17.48 ml. Solid samples contained mostly high concentrations of sodium and moderately high aluminum, manganese and silicon. Other analytes measured were generally lower in concentration.

Two radioactive liquid samples and a duplicate, **AN107 MIX @ 1:1 LIQUID** and **AN107 MIX @ 1:10 LIQUID** (RPL ID# 00-00778 and 00-00779), were analyzed by ICPAES after preparation by the Sample Receiving and Processing Laboratory (SRPL). Samples were prepared using PNL-ALO-128 acid digestion of solids procedure. Approximately 1.1 grams (1 ml) of sample was processed and diluted to a final volume of about 20ml and weighed. Concentration reported for liquids is in µg/ml (volume of sample/ final volume after processing) and µg/g (weight of sample/ final volume after processing) as requested by client for liquid samples only. Concentration reported is corrected for process and analytical dilution. See bench sheets included with this report for actual weight and volumes used. The process blank for liquids include reagents and water used to prepare samples. A 0.45µm filter was also prepared using PNL-ALO-128 procedure and analyzed. Liquid samples contained high concentration of sodium and a moderate amount of aluminum. Other analytes measured were much lower in concentration.

Specific analytes of interest requested are listed in table 2 "Analytical Requirements for Solids and Liquids. Analytes include: Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Ti, U, and Zn.

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

Five fold serial dilution:

Analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):

All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

None.

Matrix Spiked Sample:

None.

Quality Control Check Standards:

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

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes of interest is within tolerance of ± 5% accuracy.

Process Blank:

All analytes of interest is within tolerance limit of ≤ EQL or < 5% of sample concentration except as follows. All analytes of interest in the liquid samples processed using ALO-128 acid digestion were within tolerance limit. Only a small amount of nickel and zinc were detected in the liquid process blank (about 0.1 µg/ml each all below EQL). Iron, sodium, nickel, and silicon were above EQL in the ALO-129 acid digested solid samples. Iron concentration in the process blank is less than about 27% of any iron found in the solid samples. Sodium in the blank is much less than 5% of the sodium in any of the solid samples. Nickel concentration in the process blank is less than about 13% of any nickel found in the solid samples. Silicon concentration in the process blank is less than about 45% of that found in the solid samples.

Laboratory Control Standard (LCS):

None.

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

Comments:

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

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

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

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

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

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

	Multiplier=	160.1	194.1	122.2		
	ALO#=	00-0776-PB	00-0776	00-0777		
	Client ID=	*Process Blank / ALO-129*	*AN107 Mix @ 1:1*	*AN107 Mix @ 1:10*		
Det. Limit	Run Date=	1/27/00	1/27/00	1/27/00		
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g		
0.025	Ag	--	145	185	--	--
0.060	Al	[56]	7,320	8,150	--	--
0.250	As	--	--	--	--	--
0.050	B	498	277	354	--	--
0.010	Ba	[3.1]	[12]	13.9	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	500	474	--	--
0.015	Cd	[5.5]	74.1	73.5	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	[7.1]	--	--
0.020	Cr	[11]	141	135	--	--
0.025	Cu	[12]	65.2	59.5	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	287	1,460	1,070	--	--
2.000	K	--	[2,000]	[2,000]	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	[20]	--	--
0.050	Mn	--	3,810	3,150	--	--
0.050	Mo	--	[43]	[42]	--	--
0.150	Na	706	317,000	304,000	--	--
0.100	Nd	--	[20]	[19]	--	--
0.030	Ni	79.0	662	626	--	--
0.100	P	--	766	787	--	--
0.100	Pb	--	257	224	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	1,110	2,460	4,250	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	426	391	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[42]	152	447	--	--
0.050	Zr	--	[56]	[39]	--	--

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

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

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

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

	Multiplier=	17.4	17.4	17.8	18.0	81.5
	ALO#=	BLANK	BLK Filter	00-0778	00-0778-DUP	00-0779 @5
	Client ID=	*DDI:* *ALO-128* *LIQUIDS*	*DDI/0.45u filter* *ALO-128* *LIQUIDS*	*AN107 MIX* *1:1 LIQUID*	*AN107 MIX* *1:1 LIQUID*	*AN107 MIX* *1:10 LIQUID*
Det. Limit	*Run Date=*	1/27/00	1/27/00	1/27/00	1/27/00	1/27/00
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	--	--	940	939	1,840
0.250	As	--	--	--	--	--
0.050	B	--	--	11.7	11.6	[19]
0.010	Ba	--	--	--	[0.18]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	70.4	70.3	[150]
0.015	Cd	--	--	11.0	10.9	22.1
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	[1.0]	[1.0]	--
0.020	Cr	--	--	17.0	17.0	36.5
0.025	Cu	--	--	8.32	8.27	[15]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	[3.5]	[3.5]	[7.7]
2.000	K	--	--	[290]	[290]	[610]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	[6.3]	[6.3]	[13]
0.150	Na	--	--	47,800	47,300	86,900
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.95]	[0.94]	92.3	92.2	184
0.100	P	--	--	115	116	235
0.100	Pb	--	--	30.5	30.5	[55]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	[34]	[34]	[45]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	58.7	58.8	107
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[0.97]	--	[3.3]	[3.4]	[5.7]
0.050	Zr	--	--	--	--	--

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

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

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

	Multiplier=	20.0	20.0	20.0		20.0	100.0
	ALO#=	BLANK	BLK Filter	00-0778		00-0778-DUP	00-0779 @5
	Client ID=	DDI: ALO-128 LIQUIDS	DDI/0.45u filter ALO-128 LIQUIDS	AN107 MIX 1:1 LIQUID		AN107 MIX 1:1 LIQUID	AN107 MIX 1:10 LIQUID
Det. Limit	Run Date=	1/27/00	1/27/00	1/27/00		1/27/00	1/27/00
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	Mean	ug/mL	ug/mL
0.025	Ag	--	--	--		--	--
0.060	Al	--	--	1,050	1045	1,040	2,260
0.250	As	--	--	--		--	--
0.050	B	--	--	13.1	13.0	12.9	[24]
0.010	Ba	--	--	--		[0.20]	--
0.010	Be	--	--	--		--	--
0.100	Bi	--	--	--		--	--
0.250	Ca	--	--	78.9	78.45	78.0	[180]
0.015	Cd	--	--	12.3	12.2	12.1	27.1
0.200	Ce	--	--	--		--	--
0.050	Co	--	--	[1.1]		[1.2]	--
0.020	Cr	--	--	19.1	18.95	18.8	44.8
0.025	Cu	--	--	9.33	9.25	9.17	[19]
0.050	Dy	--	--	--		--	--
0.100	Eu	--	--	--		--	--
0.025	Fe	--	--	[3.9]	3.9	[3.9]	[9.5]
2.000	K	--	--	[330]	325	[320]	[750]
0.050	La	--	--	--		--	--
0.030	Li	--	--	--		--	--
0.100	Mg	--	--	--		--	--
0.050	Mn	--	--	--		--	--
0.050	Mo	--	--	[7.1]	7.05	[7.0]	[15]
0.150	Na	--	--	53,600	53000	52,400	107,000
0.100	Nd	--	--	--		--	--
0.030	Ni	[1.1]	[1.1]	104	103	102	225
0.100	P	--	--	129	128.5	128	288
0.100	Pb	--	--	34.2	34.0	33.8	[67]
0.750	Pd	--	--	--		--	--
0.300	Rh	--	--	--		--	--
1.100	Ru	--	--	--		--	--
0.500	Sb	--	--	--		--	--
0.250	Se	--	--	--		--	--
0.500	Si	--	--	[38]	38	[38]	[56]
1.500	Sn	--	--	--		--	--
0.015	Sr	--	--	65.9	65.55	65.2	132
1.500	Te	--	--	--		--	--
1.000	Th	--	--	--		--	--
0.025	Ti	--	--	--		--	--
0.500	Tl	--	--	--		--	--
2.000	U	--	--	--		--	--
0.050	V	--	--	--		--	--
2.000	W	--	--	--		--	--
0.050	Y	--	--	--		--	--
0.050	Zn	[1.1]	--	[3.7]	3.75	[3.8]	[6.9]
0.050	Zr	--	--	--		--	--

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

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

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

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-0776

2/16/2000

Client : Lumetta

Cognizant Scientist: LRGreenwood Date : 2-16-00

Concur : T Trang-le Date : 2/16/00

Procedure: PNL-ALO-450
Equipment: Gamma detectors G

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0776PB	<9.E-4	1.60E-2	4.86E-3	3.38E+0	1.37E-2	1.78E-2	3.28E-2
Process Blank		18%	8%	2%	6%	10%	10%
00-0776	1.67E-1	<4.E-1	1.90E-2	4.20E+2	2.69E-1	2.53E-1	2.30E-1
AN107Mix @1:1	3%		20%	2%	4%	20%	30%
00-0777	1.63E-1	<7.E-1	<2.E-2	4.30E+2	2.37E-1	<5.E-1	<5.E-1
An107Mix@1:10	5%			2%	7%		
00-0778	2.57E-2	<2.E-1	<3.E-3	6.32E+1	1.50E-2	<7.E-2	<7.E-2
An107-C104(1:1) Liquid	4%			2%	14%		
00-0778Dup	2.34E-2	<2.E-1	<3.E-3	6.25E+1	1.20E-2	<7.E-2	<7.E-2
An107-C104(1:1) Liquid	5%			2%	14%		
RPD	9%			1%	22%		
00-0779	4.83E-2	<3.E-1	<7.E-3	1.17E+2	3.01E-2	<2.E-1	<2.E-1
An107-C104(1:10) Liquid	5%			2%	14%		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-0776

3/10/00

Client : Lumetta

Cognizant Scientist: L. R. Greenwood Date : 3/10/00

Concur : T Trang-le Date : 3/10/00

Procedure: PNL-ALO-420

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Alpha Error +/-
00-0776PB Process Blank	2.41E-2 3%
00-0776 AN107Mix @1:1	2.50E-1 4%
00-0776 REP AN107Mix @1:1	2.39E-1 4%
RPD	4%
00-0777 An107Mix@1:10	1.51E-1 4%
00-0778PB Process Blank	<3.E-5
00-0778 An107-C104(1:1) Liquid	5.64E-3 8%
00-0778Dup An107-C104(1:1) Liquid	6.17E-3 8%
RPD	9%
00-0779 An107-C104(1:10) Liquid	1.11E-2 5%
Matrix Spike	98%
Blank Spike	102%
Blank	<3.62E-4

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

00-0776

3/23/00

Client : Lumetta

Cognizant Scientist: LRGreenwood Date : 3/23/00

Concur : T Trang-le Date : 3/23/00

Procedure: PNL-ALO-476

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Sr-90 Error +/-
00-0776 AN107Mix @1:1	8.55E+0 3%
00-0777 An107Mix@1:10	5.18E+0 3%
00-0778PB Process Blank	<1.E-4
00-0778 An107-C104(1:1) Liquid	5.03E-1 3%
00-0778Dup An107-C104(1:1) Liquid	5.14E-1 3%
RPD	2%
00-0779 An107-C104(1:10) Liquid	7.23E-1 3%
Matrix Spike	104%
Blank Spike	104%
Blank	<5.E-5

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client:	G. Lumetta	**Charge Code/Project:**	W45526/29953
ACL Numbers:	00-0770 to 00-0779	**ASR Number:**	5644
Analyst:	MJ Steele	**Analysis Date:**	March 15, 2000

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

The submitted AN107/C104 samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquids samples (reported in μg/ml) were analyzed following appropriate analytical dilutions to ensure that all reported anions were within the IC calibration range. The two solids samples were processed in the hot cells by leaching with a 100:1 ratio of water. The leachates were filtered prior to IC analysis and the results are reported in μg/g of as received solid.

		F	Cl	NO_2	Br	NO_3	PO_4	SO_4	C_2O_4
Lab ID	**Liquid Sample ID**	**ug/ml**	**ug/ml**	**ug/ml**	**ug/ml**	**ug/ml**	**ug/ml**	**ug/ml**	**ug/ml**
00-00770	AN107 C104 -3A	1,540	430	13,900	< 250	55,500	< 500	1,860	980
00-0770 MS	Matrix Spike Recovery	129%	113%	101%	103%	107%	110%	117%	110%
Working Spk	Recovery	109%	109%	105%	107%	106%	106%	106%	105%
00-00773	AN107 C104 -4A	2,880	430	13,700	< 250	54,100	< 500	1,840	1,000
00-00778	AN107 Mix @1:1 Liquid	< 250	380	11,600	< 250	47,200	< 500	1,620	2,500
00-00779	AN107 Mix @1:10 Liquid	< 250	700	24,200	< 250	105,000	< 500	3,000	< 500

		Solids	F	Cl	NO_2	Br	NO_3	PO_4	SO_4	C_2O_4
Lab ID	**Solids Sample ID**	**Dil Fctr**	**ug/g**	**ug/g**	**ug/g**	**ug/g**	**ug/g**	**ug/g**	**ug/g**	**ug/g**
00-00776 PB	Leach Process Blank	92.9	< 25	< 25	< 50	< 25	< 50	< 50	< 50	< 50
00-00776	AN107 Mix @1:1	98.9	2,290	2,130	70,200	< 1000	275,000	< 2000	9,040	16,900
00-00777	AN107 Mix @1:10	86.9	2,270	1,940	68,200	< 900	274,000	< 1800	9,260	7,370

Note: Fluoride is considered qualitative due to significant peak interferences making fluoride quantitation difficult.

Q.C. Comments:

Duplicates: No duplicate were provided.

Matrix Spike: A matrix spike was prepared from the AN107 C104-3A sample (00-0770) after the sample was delivered to the IC Workstation. No attempt to spike the supernatant in the hot cells was made. All anion recoveries, except fluoride, were within the 75% to 125% recovery acceptance criteria. The 129% fluoride MS recovery is just outside the acceptance criteria and is most likely due the difficulties in quantifying the fluoride peak (or establishing a good baseline) due to significant interferences (most probably from organic anion, such as formate or acetate). Based on these interferences, the fluoride results are considered qualitative.

Blank Spike: No blank spikes were prepared in the hot cells for either the liquid or the solid leached/dissolved solids. The working spike (i.e., the spike solution used to prepare the matrix spike samples) was measured at the same time as the Matrix Spike samples and demonstrated recoveries within the 90% to 110% acceptance criteria.

System Blank/Processing Blanks: Eleven system blanks were processed during the analysis of the liquid samples. Other than one oxalate system blanks, no anions were detected in the system blanks above the estimate quantitation level. The low level of oxalate detected in the system blank has not affect on the reported oxalate values.

Quality Control Calibration Verification Check Standards: Nine mid-range verification standards were analyzed throughout the analysis runs. For most of the anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. Two nitrite results recovered below the 90% lower limit; however these recoveries were all greater than 85%.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst: MJ Steele **Date** 3/21/00

Approval: **Date** 3/22/00

Archive Information:

Files: ASR 5644 Lumetta.doc ASR 5644 5667-8 5744.xls

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client:	G. Lumetta	**Charge Code/Project:**	W45536 / 29953
ACL Numbers:	00-0776 to 00-0779	**ASR Number:**	5644
Analyst:	MJ Steele	**Analysis Date:**	April 5, 2000

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

Final Results:

Results in ug Carbon / ml of Sample

Lab Number	Sample ID	Vol ml	TIC µg C/ml	TIC RPD %	TOC µg C/ml	TOC RPD %	TC µg C/ml	TC RPD %
00-0776 PB	Process Blank	0.50	<14		<36		<36	
00-0776	AN107 Mix @ 1:1	0.10	410		380		790	
00-0776 Dup	AN107 Mix @ 1:1 Dup	0.50	380	8	430	n/a	810	3
00-0777	AN107 Mix @ 1:10	0.50	430		480		910	
00-0777 Dup	AN107 Mix @ 1:10 Dup	0.50	490	13	560	15	1,050	14
00-0778	AN107 Mix @ 1:1 Liquid	0.10	4,440		8,500		12,900	
00-0778 Dup	AN107 Mix @ 1:1 Liquid Dup	0.10	4,220	5	8,160	4	12,400	5
00-0778 MS	AN107 Mix @ 1:1 Liquid MS	0.10	87.4%		96.5%		91.1%	
00-0779	AN107 Mix @ 1:10 Liquid	0.10	8,600		16,200		24,800	
00-0779 Dup	AN107 Mix @ 1:10 Liquid Dup	0.10	8,240	4	15,600	4	23,800	4

Results in ug Carbon / g of Sample (wet weight)

Lab Number	Sample ID	Wt g	TIC µg C/g	TIC RPD %	TOC µg C/g	TOC RPD %	TC µg C/g	TC RPD %
00-0776 PB	Process Blank	0.4986	<14		<37		<37	
00-0776	AN107 Mix @ 1:1	0.1009	410		380		790	
00-0776 Dup	AN107 Mix @ 1:1 Dup	0.5034	380	8	430	n/a	810	3
00-0777	AN107 Mix @ 1:10	0.5039	420		480		900	
00-0777 Dup	AN107 Mix @ 1:10 Dup	0.5061	480	13	550	15	1,000	14
00-0778	AN107 Mix @ 1:1 Liquid	0.1129	3,930		7,530		11,500	
00-0778 Dup	AN107 Mix @ 1:1 Liquid Dup	0.1109	3,800	3	7,350	2	11,200	3
00-0778 MS	AN107 Mix @ 1:1 Liquid MS	0.1112	88.1%		98.5%		92.3%	
00-0779	AN107 Mix @ 1:10 Liquid	0.1233	6,980		13,200		20,200	
00-0779 Dup	AN107 Mix @ 1:10 Liquid Dup	0.1217	6,770	3	12,800	3	19,600	3

RPD = Relative Percent Difference (between sample and duplicate/replicate); calculated from unrounded data.

The analyses of the samples submitted under ASR 5644 were performed by the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank. As requested by the ASR the TIC and TOC results are reported in both μg/g and μg/ml.

Q.C. Comments:

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) in either solid or liquid form.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards were all within acceptance criteria of 90% to 110%, with the average recovery being about 95% for TIC and 95% for TOC.

Calibration Blanks: The four calibration blanks run at the beginning, middle, and end of the analysis run were acceptable, averaging about 11 μgC TIC and 33 μgC TOC. These calibration/system blanks are considered good and the standard deviations for the TIC and TOC blanks is within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: No actual sample duplicates were identified on the ASR. However, the precision between the replicates, as demonstrated by the Relative Percent Difference (RPD), is good. All RPDs are within the acceptance criteria of 20%, with most RPDs being less than 10%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample 00-0778. The matrix spike demonstrated recoveries of about 87% for TIC and 97% for TOC; well within the acceptance criteria of 75% to 125% recovery.

ıts:

Final Results" have been corrected for all dilution performed on the sample
ing or analysis.

ion and bias are typically ±15% or better for non-complex samples that are free of

quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times
higher uncertainties, and RPDs are not calculated for any results less than 5 times

nay be reported as less than ("<") values. These less than values represent the
method detection limit), which is the system MDL adjusted for the volume of
ır the analysis. The system MDL is based on the attached pooled historical blank
uation and calculation of the system MDL is included in the data package.

by: MW Urie 4/11/00

l by: **Date** 4-12-00

tion:

Lumetta.doc ASR 5644 5667 5668 5744 Liq+Solids.xls

L SAMPLE

45536 / 29953
5644
April 5, 2000

ıdioactive Sludges

TC (ug C/g)	TC RPD (%)
<37	
790	
810	3
900	
1,000	14

TC (ug C/g)	TC RPD (%)
<3,500	
77,600	
79,800	3
78,200	
89,800	14

ounded data.

aching processes
he same as

4/17/00

'44 Liq+Solids.xls

Appendix C. Calculations

ENGINEERING WORKSHEET

Prepared By: M.J. Jumette	Date: 12/9/99	Project: 29953

Title/Subject: Mixing of Process Heels: Data Work Up

Example Calculation of Tc Kd

Consider contact AW101C104-Kd1

Sample AW101C104-1A → 386 ng/g 418 ng/mL

Sample AW101C104-1B → 77.7 ng/g 84.0 ng/mL

wt. solution = 5.113 g (4.15b)

wt. resin = 0.0498 g (4.3b)

$$K_d = \cancel{418\ ng/mL}\ \frac{(386-77.7\ ng/g)(5.113\ \overset{solution}{g})}{0.0498\ g\ resin} \cdot \frac{1}{84.0\ ng/mL} = \overset{394}{\cancel{377.2}}\ \frac{mL}{g}$$

M.J.L. 12/9/99

M.J.L. 12/9/99

M.J.L. 1/24/2000

$\uparrow$ $\frac{1}{0.957}$ (as per Lumetta PNWD-2467)

What is Kd in terms of TcO_4^-?

Assume all sorbed Tc is TcO_4^-.

Initial: (386 ng/g)(5.113g) = 1974 ng $\xrightarrow{0.972}$ 1919 ng as TcO_4^-

0.972 → fraction as TcO_4^-

Final: (77.7ng/g)(5.113g) = 397 ng

1974-397 = 1577 ng in resin

$$\begin{array}{r} 1919 \\ -1577 \\ \hline 342 \end{array}$$

342 ng as TcO_4^- in final solution.

÷ 5.113 g solution

66.9 ng/g

↓ 1.08 g/mL

72.3 ng/mL

$$Kd\ TcO_4^- = \frac{\overbrace{\cancel{(386-77.7\ ng/g)}}^{[(386 * 0.972)-72.3]}(5.113\ g\ soln.)}{0.0498\ g\ resin} \cdot \frac{1}{72.3\ ng/mL} \cdot \frac{1}{0.956} = \overset{}{\cancel{458}}\ 450\ mL/g$$

M.J.L. 3/30/00

M.J.L. 3/30/00

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ENGINEERING WORKSHEET

Page 1 of 2

Prepared By: S.A. Fiscaletta	Date: 12/22/99	Project:

Title/Subject: Mixing of Process Heels: Fraction of Tc Contributed By the Individual Solutions Mixed.

Soln. ID		Tc, ng/g
~~A~~ C	AW101 Permeate	5.1
~~B~~ D	AW101 Wash Solution	0.24
~~C~~ B	"00-0074 AN107 CUF Wash Comp"	0.29
~~D~~ A	"AN107 Cs IX Batch Contact"	2.67
~~E~~ E	"C104 Composite Lunetta"	0.0505

} Information from Kriston Brooks and Rich Hallen.

↳ See table 2.1 in report (changed to Roman numerals in report to avoid confusion S.A.F. 1/25/2000)

Test 1

3.9720 g A + 38.2534 g C

(38.2534 g)(5.1 ng/g) = 195.09 ng — 95% from C

(3.9720 g)(2.67 ng/g) = 10.61 ng — 5% from A

205.70

Test 2

3.8118 g A + 31.9923 g E

(31.9973 g)(0.0505 ng/g) = 1.616 ng — 14% from E

(3.8118 g)(2.67 ng/g) = 10.177 ng — 86% from A

11.793 ng

Test 3

24.9540 g A + 20.9484 g E

(20.9484 g)(0.0505 ng/g) = 1.058 ng — 2% from E

(24.9540 g)(2.67 ng/g) = 66.627 ng — 98% from A

67.685

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ENGINEERING WORKSHEET

Prepared By: S.J. Fumetta	Date: 12/22/99	Project:

Title/Subject: Mixing of Process Heels: Tc Fractions (contd.)

Test 4

3.6916 g C + 32.1748 g E

(3.6916 g)(5.1 ng/g) = 18.827 ng   92% from C
(32.1748 g)(0.0505 ng/g) = 1.625 ng   8% from E
20.452 ng

Test 5

24.8593 g C + 21.8070 g E

(24.8593 g)(5.1 ng/g) = 126.782 ng   99.1% from C
(21.8070 g)(0.0505 ng/g) = 1.101 ng   0.9% from E
127.883

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ENGINEERING WORKSHEET

Prepared By: D.J. [illegible]	Date: 1/24/2000	Project: BNFL 2995-3

Title/Subject: Mixing of Process Heels : Calculation of Pertechnetate Fraction in Mixtures

Test #1

5% of Tc from AW107 Solution
95% of Tc from AW101 Solution

AW107 Solution → 25% TcO_4^-
AW101 Solution → 97% TcO_4^-
} Kurath et al. PNWD-2467 Sept. 1999

$$\frac{(5 \times 0.25) + (95 \times 0.97)}{100} \times 100 = 93.4\%\ TcO_4^-$$

Test #2

86% Tc from AW107
14% Tc from C104 → Assume all TcO_4^-

$$(86 \times 0.25) + (14) = 35.5\%\ TcO_4^-$$

Test #3

98% Tc from AW107
2% Tc from C104

$$(98 \times 0.25) + 2 = 26.5\%\ TcO_4^-$$

Test #4

92% from AW101
8% from C104

$$(92 \times 0.97) + 8 = 97.2\%\ TcO_4^-$$

Test #5

99% from AW101
1% from C104

$$(99 \times 0.97) + 1 = 97.0\%\ TcO_4^-$$

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ENGINEERING WORKSHEET

Prepared By: M. J. Minette
Date: 3/30/2000
Project:
Title/Subject: Mixing of Process Heels : Estimate of Radionuclide Content of Precipitated Solids

(refer to Table 3.3 of the report BNFL-RPT-023)

Assumptions :

1\. Oxalate present as $Al_2(C_2O_4)_3 \cdot 4H_2O$ 390.08 g/mole

For 1:1 AN107 Mix : 73.2 mg C_2O_4 $\xrightarrow{\div 88.018 \text{ g/mole}}$ 0.8316 mmole

$\downarrow \div 3$

0.2772 mmole $Al_2(C_2O_4)_3 \cdot 4H_2O$

$\downarrow$ 390.08 g/mole

$\boxed{324 \text{ mg } Al_2(C_2O_4)_3 \cdot 4H_2O}$

~~÷ 2~~

$\downarrow \times 2$

0.5544 mmole Al

$\downarrow$ 26.982 g/mole

15.0 mg Al

$\downarrow$

127 mg Al in solids − 15 mg = 112 mg Al

2\. Remaining Al is $Al(OH)_3$ → 78.003 g/mole

$$(112 \text{ mg Al}) \frac{78.003 \text{ g/mole}}{26.982 \text{ g Al/mole}} = \boxed{324 \text{ mg } Al(OH)_3}$$

3\. Fluoride present as $FeF_3 \cdot 4\tfrac{1}{2} H_2O$ → 193.91 g/mole

79.3 mg F $\xrightarrow{\div 18.998 \text{ g/mole}}$ 4.1741 mmole F

$\downarrow \div 3$

1.3914 mmole $FeF_3 \cdot 4\tfrac{1}{2} H_2O$

$\downarrow$ 193.91 g/mole

~~26~~ $\boxed{270 \text{ mg } FeF_3 \cdot 4\tfrac{1}{2} H_2O}$

1.3914 mmole Fe

$\downarrow$ 55.847 g/mole

77.7 mg Fe

→ 172 − 78 = 94 mg Fe

4\. Remaining Fe is $Fe(OH)_3$

$$(94 \text{ mg Fe}) \left(\frac{106.868 \text{ g/mole}}{55.847 \text{ g/mole Fe}} \right) = \boxed{180 \text{ mg } Fe(OH)_3}$$

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ENGINEERING WORKSHEET

Prepared By: S. J. [illegible]	Date: 3/20/2000	Project:

Title/Subject: Mixing of Process Heels.

5\. Mn is ~~$Mn(OH)_2$~~ MnO_2

$$(456\ mg)\left(\frac{86.936\ g/mol}{54.938\ g\ Mn/mol}\right) = 722\ mg\ MnO_2$$

6\. Si is SiO_2

$$(269\ mg)\left(\frac{60.084\ g/mol}{28.086\ g\ Si/mol}\right) = 575\ mg\ SiO_2$$

∴

Total estimated mass = 324 + 324 + 270 + 180 + 722 + 575 = 2395 mg (0.0024 g)

So radionuclide content

Cs-137 1.3 µCi / 0.0024 g = 540 µCi/g
Sr-90 0.6 µCi / 0.0024 g = ~~8~~ 250
Eu-154 0.022 / = .9
Co-60 0.001 / = 0.4
TRU 0.025 / = 10.4

Repeating the above for the 10:1 mix material:

1\. $456\ mg\ C_2O_4 / 88.018 \longrightarrow 10.8614\ mmol \xrightarrow{\div 3} 3.6205\ mmol\ Al_2(C_2O_4)_3 \xrightarrow{390.08\ g/mol} \boxed{1412\ mg\ Al_2(C_2O_4)_3 \cdot 4H_2O}$

$\downarrow \times 2$

$7.2409\ mmol\ Al$

$\downarrow 26.982\ g/mol$

$195\ mg\ Al \Rightarrow 286 - 195 = 91\ mg\ Al$

2\. $(91\ mg\ Al)\left(\frac{78.033}{26.982}\right) = \boxed{263\ mg\ Al(OH)_3}$

3\. $239\ mg\ F / 18.998\ g/mol = 12.5803\ mmol\ F \xrightarrow{\div 3} 4.1934\ mmol\ FeF_3 \xrightarrow{193.91} \boxed{813\ mg\ FeF_3 \cdot 4\tfrac{1}{2}\ H_2O}$

$(4.1934\ mmol\ Fe)(55.847) = 234\ mg\ Fe$

4\. $176 - 234 = \sim 0\ Fe(OH)_3$

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ENGINEERING WORKSHEET

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Prepared By:
Date: 3/30/2000
Project:
Title/Subject: Mixing of Process Heels

5. $(529 \text{ mg Mn})\left(\frac{86.936}{54.938}\right) = \boxed{837 \text{ mg } MnO_2}$

6. $(690 \text{ mg Si})\left(\frac{60.084}{28.086}\right) = \boxed{1476 \text{ mg } SiO_2}$

Total Estimated Mass = 1412 + 263 + 813 + 0 + 837 + 1476 = 4801 mg → 0.0048 g

Cs-137	5.1 / 0.0048	=	1065 mg μCi/g
Sr-90	0.5 /	=	105
Eu-154	0.023 /	=	5
Co-60	0 /	=	0
TRU	0.019 /	=	4