PNWD-3053
BNFL-RPT-036, Rev. 0

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Prepared for BNFL, Inc.
under Contract W375-LC-98-4168

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Battelle, Pacific Northwest Division
Richland, Washington 99352

Summary

BNFL Inc. is evaluating various pretreatment technologies to mitigate the impacts of sulfate on the LAW vitrification system. One pretreatment technology for separating sulfate from LAW solutions involves the use of SuperLig® 655 (SL-655), a proprietary ion exchange material developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. This report describes testing of SL-655 with diluted ([Na] 5 M) waste from Hanford Tank 241 -AN-107 at Battelle, Pacific Northwest Division.

Batch contact studies were conducted from 4 to 96 hours to determine the sulfate distribution coefficient and reaction kinetics. A small-scale ion exchange column test was conducted to evaluate sulfate removal, loading, breakthrough, and elution from the SL- 655. In all of these tests, an archived 241-AN-107 tank waste sample (pretreated to remove Cs, Sr, and transuranics elements) was used. The experimental details and results are described in this report.

Under the test conditions, SL- 655 was found to have no significant ion exchange affinity for sulfate in this matrix. The batch contact study resulted in no measurable difference in the aqueous sulfate concentration following resin contact (K_d 0). The column test also demonstrated SL - 655 had no practical affinity for sulfate in the tested matrix. Within experimental error, the sulfate concentration in the column effluent was equal to the concentration in the feed after passing 3 bed volumes of sample through the columns. Furthermore, some, if not all, of the decreased sulfate concentration in these first three column volumes of effluent can be ascribed to mixing and dilution of the 241-AN-107 feed with the interstitial liquid present in the column at the start of the loading cycle. Finally, ICP-AES measurements on the eluate solutions showed the presence of barium as soon as contact with the feed solution is completed. Barium is a metal not detected in the feed solution. Should the loss of barium be correlated with the resin's ability to selectively complex sulfate, then maintaining even the current limited resin characteristics for sulfate complexation over multiple cycles becomes questionable.

Terms, Symbols and Abbreviations

BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd
BV	bed volume
C	concentration
C_o	initial concentration
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively-coupled plasma optical emission spectrometry
K_d	distribution coefficient
LAW	low-activity waste
L/D	length:diameter ratio
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
RPL	Radiochemical Processing Laboratory
SAL	Shielded Analytical Laboratory
SL-655	SuperLig® 655 resin
TOC	total organic carbon
TIC	total inorganic carbon
TRU	transuranic

Units

°C	degrees celsius or centigrade
cm	centimeter
M	molarity, moles per liter
meq	milliequivalent
µCi	microCuries
µg	micrograms
µm	micrometer

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1.0 INTRODUCTION

The presence of sulfate ion in the Hanford low-activity waste (LAW) solutions generates several potential processing difficulties. Preliminary testing of the LAW vitrification system at the Vitreous States Laboratory (VSL) indicates a separate molten sulfur layer will form in the melter (nominally at 1150°C) at sufficiently high sulfate concentrations. A molten sulfur layer in the LAW melter can lead to accelerated corrosion of the melter and unacceptable operating conditions (e.g., steam explosion).

BNFL Inc. (BNFL) has been evaluating several methods to mitigate the impacts of sulfate on the LAW vitrification system, including pretreatment technologies, blending of high and low sulfate LAW solutions, modification to the LAW glass formulations, and volatilization of sulfur in the LAW melter as SO_2 or SO_3. BNFL is evaluating four pretreatment technologies for separating sulfate from LAW solutions:

- Ion Exchange (SuperLig® 655)
- Evaporation
- Precipitation
- Low-temperature crystallization

This report presents the results of a series of experiments conducted by Battelle, Pacific Northwest Division to evaluate the use of ion exchange with SuperLig® 655 (SL-655) for sulfate ion removal from the waste currently stored in tank 241-AN-107 (referred to hereafter as AN-107). SL-655 is a developmental material prepared by IBC Advanced Technologies Inc. The resin incorporates a divalent cation (proprietary) as an exchange site for sulfate ion as it replaces two nitrate ions in the loading stage. The theoretical capacity of SL-655 is 0.8 meq sulfate per gram of dry resin. The test matrix used was an archived sample of actual AN-107 tank waste pretreated to remove Sr, Cs, and transuranic elements (TRU). The AN-107 waste is categorized as LAW Envelope C waste. Batch contacts and small-scale column testing were conducted in order to evaluate K_ds, column loading and elution characteristics, and physical properties of the eluates and effluents as delineated in the BNFL test specification.[1] This report summarizes the results of these studies.

[1] Test Specification for Evaluating Sulfate Separation from LAW Solutions, Rev. 0, September 13, 1999, TSP-W375-99-00012. Rev. 0.

2.0 TEST CONDITIONS AND EXPERIMENTAL PROCEDURES

2.1 Preparation of the AN-107 Test Sample

Testing was conducted with a sample of 241-AN-107 tank waste archived after previous examination (Hendrickson et. al., 1997). This sample was originally obtained as a grab sample in January of 1997. It was diluted with 0.53 M NaOH to approximately 5 M Na and 0.24 M OH^-. Cesium was removed using a crystalline silico-titanate ion exchanger. Following Cs removal, the free hydroxide measured 0.126 M. The cesium-decontaminated samples were transferred to the Radiochemical Processing Laboratory (RPL) and were archived in the Shielded Analytical Laboratory (SAL). A 1-L aliquot of this archived material was processed for Sr/TRU removal using 1 M $Sr(NO_3)_2$ and 1 M $NaMnO_4$ solutions (Hallen et al., 2000). After decontamination, the archive AN-107 could be contact-handled in a fume hood. The archive AN-107 feed composition is given in Table 2.1. The density of the AN-107 feed solution was determined to be 1.22 g/mL.

2.2 SuperLig® 655

Battelle received SuperLig® 655 (IBC Advanced Technologies, Inc. American Fork, Utah) material from batch 990805DHC-8-030 suspended in 0.25 M $NaNO_3$. An F-factor (the fraction of the resin's dry weight to the as-received material) of 0.454 was experimentally determined using duplicate aliquots of ca. 2.8-g resin that were weighed, dried at 95°C, and re-weighed.

2.3 Batch Contacts

The batch contact study was conducted according to the Test Plan BNFL-29953-056.[2] As-received SL- 655 was taken and the particles drained to a damp dryness. Nominally 0.33 g samples of the damp SL-655 (possessing an estimated ca. 0.12 meq. sulfate capacity per test sample) were weighed into tarred glass vials. To these resin aliquots, 5-mL aliquots (containing an estimated ca. 0.2 meq. sulfate) of archived AN-107 solution were added and the slurries were agitated for 4, 8, 16, 24, 72, and 96 hours. An archive AN-107 blank (no added resin) was similarly prepared and the test samples agitated for 96 hours. The temperature was not controlled but the temperatures at the beginning (nominally 22.7°C) and the end (22.7°C to 28.7°C) of the agitation process were recorded. A summary of resin masses, AN-107 masses, contact times, and temperatures is provided in Table 2.2. After agitation was complete, the samples were filtered through 0.2-0.8-µm syringe filters and the filtrate submitted for sulfate analysis by ion chromatography.

2.4 Small-scale Ion Exchange Column Testing

The ion exchange study was conducted according to the Test Plan BNFL-29953-054.[3] A slurry of the SL-655 resin in aqueous 0.25 M $NaNO_3$ was loaded into a column providing a resin bed 2.5-cm diameter by 5.75 cm high (L/D =2.3 with an approximate volume of 29 mL). A schematic of the ion exchange system is provided in Figure 2.1. The processing steps, solution matrices, volumes, and flow rates are summarized in Table 2.3. All processing was conducted at room temperature, 23.8°C.

[2] BNFL-29953-056, Rev. 0, Batch Contact Test Instructions for SL-655 and Archive AN-107 Sample, 9/16/99.
[3] BNFL-29953-054, Rev. 0, Test Instructions for Sulfate Removal Column Testing Using Envelope C (AN-107 Archive) Solution, 9/10/99.

Table 2.1. 241-AN-107 Feed Composition

Analyte	Concentration µg/mL	MRQ µg/mL	Analyte	Concentration µCi/mL	MRQ µCi/mL
Cr	13	15	Cs-137	2.67E-02	9.00E+00
TOC	14,000	1500	Tc-99	5.61E-02	1.50E-03
TIC	6800	1500	Additional opportunistic analytes		
Cl	930	3000	Sr-90	6.87E-01	NMRQ
F(1)	3600	1500	Am-241	4.52E-03	NMRQ
PO_4	1400	2500	Total alpha	5.13E-03	NMRQ
SO_4	3920(2)	2300	Co-60	5.72E-02	NMRQ
Additional opportunistic analytes			Sb-125	5.62E-04	NMRQ
Al	133	NMRQ	SnSb-126	2.87E-04	NMRQ
B	21	NMRQ	Cs-134	<2E-4	NMRQ
Ba	<1	NMRQ	Eu-154	1.10E-02	NMRQ
Ca	162	NMRQ	Eu-155	7.95E-03	NMRQ
Cd	26	NMRQ			
Co	[2.1]	NMRQ			
Fe	3	NMRQ			
K	723	NMRQ			
La	<0.5	NMRQ			
Mg	<1	NMRQ			
Mn	[2.1]	NMRQ			
Mo	15	NMRQ			
Na	114,200	NMRQ			
Nd	[1.3]	NMRQ			
Ni	222	NMRQ			
P	203	NMRQ			
Rh	[5]	NMRQ			
Ru	[15]	NMRQ			
Si	101	NMRQ			
Sn	<15	NMRQ			
Sr	91	NMRQ			
Ti	<0.3	NMRQ			
U	[41]	NMRQ			
W	[73]	NMRQ			
Y	<0.5	NMRQ			
Zn	6	NMRQ			
Zr	[1.2]	NMRQ			
NO_2	28,000	NMRQ			
NO_3	111,000	NMRQ			

Uncertainties typically do not exceed +/-15% (2-σ).

Note: bracketed values indicate uncertainties greater than +/-15% (2-σ).

1) Fluoride concentration is suspect because of retention time shift and peak shape anomaly
2) Based on an average of 5 determinations.

Table 2.2. SuperLig® 655 Batch Contact with 241-AN-107

Vial ID	SuperLig® 655 mass (g)	AN-107 mass (g)	Contact time (h)	Start temp. (°C)	End temp. (°C)
AAN-C	0.0	6.1655	96	NR	28.0
AAN55-04	0.3305	6.1933	4	22.7	23.9
AAN55-08	0.3300	6.2000	8	22.7	28.7
AAN55-16	0.3299	6.1733	16	NR	22.7
AAN55-24	0.3299	6.1725	24	NR	28.7
AAN55-72	0.3305	6.2380	72	24.0	28.0
AAN55	0.3304	6.1883	96	NR	28.0
AAN55-D	0.3287	6.1962	96	NR	28.0

NR: not recorded

Table 2.3. Summary of Ion Exchange Column Processing Steps

Process Step	Solution	Total Volume, Column Volumes (mL)		Flow Rate in Column Volumes per Hour (mL/hr)	
		target	actual	target	actual
Column Preparation	0.1M NaOH + 0.25M $NaNO_3$	3 (90)	3 (90)	3 (90)	3.1 (90)
Loading	AN-107 Feed	10 (300)	11.2 (327)	3 (90)	3.0 (88)
Feed Displacement	0.1M NaOH + 0.25M $NaNO_3$	2 (60)	2.0 (59.5)	3 (90)	3.0 (88)
Column Washing	0.25M $NaNO_3$	2 (60)	2.1 (60)	3 (90)	3.1 (89)
Elution	0.5M HNO_3	10 (300)	10.7 (310)	1 (30)	1.1 (31)
Elution Rinse	0.25M $NaNO_3$	2 (60)	2.1 (62)	1 (30)	1.1 (31)
Regeneration	0.1M NaOH + 0.25M $NaNO_3$	2 (60)	2.1 (60)	3 (90)	3 (90)

NOTE: The column volume is based on a resin bed volume of 29.1 mL with a height 5.75 cm.

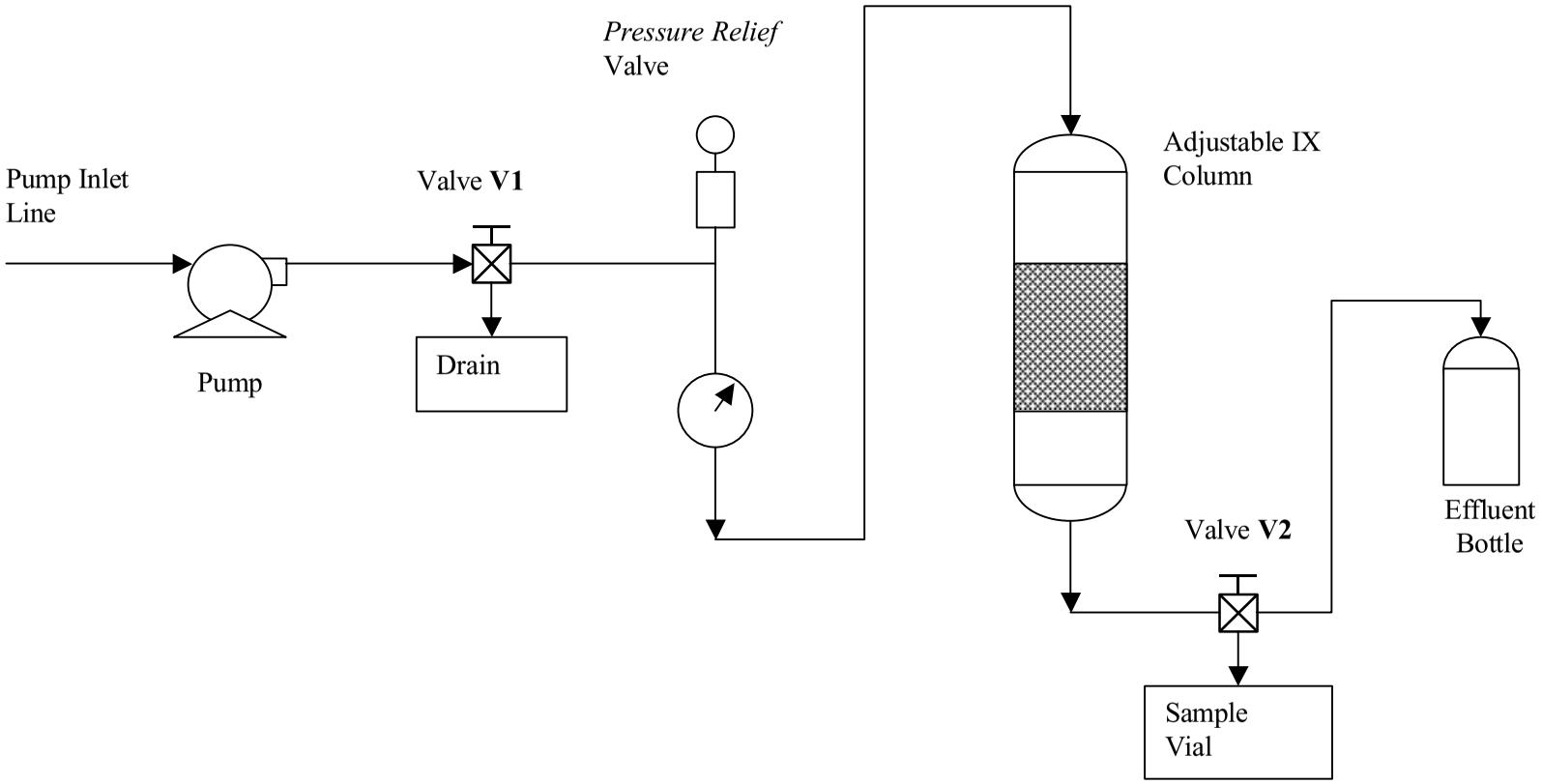


Figure 2.1. Schematic of the Sulfate Ion Exchange Column Setup

The column was initially conditioned with 90-mL (3 column volumes) of a solution consisting of 0.1 M in NaOH and 0.25 M in $NaNO_3$. Next, the resin was loaded with 327-mL of AN-107. Throughout the sample loading stage, 15-mL increments (0.5 column volumes) of the column effluent were collected. Some leakage around the top and bottom o-rings on the column was observed. The leakage at the bottom was collected while the leakage at the top was collected above the adjustable plunger. After sample loading was complete, the sample bed height measured 5.5-cm. The 15-mL column effluent samples were sub-sampled for sulfate analysis. The remaining fractions were combined and sub-sampled for GEA, ^{90}Sr, ^{99}Tc, TOC, TIC, IC and Cr. The TOC, TIC, ^{99}Tc, and several ^{90}Sr analyses were later cancelled per request by BNFL.

Following completion of the resin loading with AN-107, a column feed displacement solution 0.1 M in NaOH and 0.25 M in $NaNO_3$ was passed through the SL-655 resin bed and the effluent collected in two 15-mL fractions followed by two 30-mL fractions. Next, a column wash using 0.25 M $NaNO_3$ was collected in two 30-mL fractions. The first 15-mL feed displacement fraction was analyzed only by IC. The remaining feed displacement and column wash fractions were analyzed by GEA, IC and ICP.

To remove any remaining sulfate on the column the SL-655 resin next was eluted with ten, ≈30-mL volumes of 0.5 M HNO_3. Each of these volumes was collected separately. During this initial elution process, the column plugged and went completely dry. The column plugging was attributed to the leakage of feed during the loading step around the adjustable plunger at the top. This feed appeared to drain back into the column where it was neutralized by the acid, resulting in precipitation of some of the dissolved metals which plugged the column. The plug was removed and the air bubbles in the resin bed were removed with a plastic Pasteur pipette causing some resin bed mixing. This incident most affected the first two elution fractions. Some leakage at the bottom of the column also occurred during this elution step and was repaired. Sub-samples of each elution fraction were analyzed by GEA, IC, and ICP. The remaining elution sample fractions, excluding the first and second, were combined and submitted for ICP, GEA, IC, and ^{90}Sr analyses. The TIC, TOC, total alpha, and ^{99}Tc analyses were cancelled per request by BNFL.

The column was finally rinsed with two, ≈30-mL volumes 0.25 M $NaNO_3$, with the eluate being collected in two 30-mL increments. The two samples were analyzed by GEA, IC and ICP. Finally, the column was regenerated with two 30-mL volumes of a solution 0.1 M in NaOH and 0.25 M in $NaNO_3$. This effluent was collected in a single container.

3.0 RESULTS AND DISCUSSION

3.1 Batch Contact K_d Study

Within experimental error, the sulfate concentration did not change from the initial sulfate concentration of 3920 µg/mL, even after a 96-hour contact time with SL-655 resin. For all contacts the average final sulfate concentration was 3900 ± 47 µg/mL (1 standard deviation). The initial and final aqueous sulfate concentrations are essentially equal, and thus the measured sulfate K_ds become effectively 0, as described by the following formula:

$$K_d = \frac{(C_o - C)}{C} \times \frac{V}{m_r}, \qquad (3.1)$$

where C_o = initial aqueous sulfate concentration, C = final aqueous sulfate concentration, V = solution volume, and m_r = dry resin mass.

From the uncertainty associated with the IC measurements, a maximum possible K_d can be estimated. The uncertainty associated with the IC analysis is claimed to be 15% or less. Assuming, then, that the final aqueous sulfate concentration is 15% less than the initial aqueous concentration and that this difference is bound up in the resin (which would correspond to only 25% of the resin's capacity), the application of equation 3.1 would lead to a maximum possible K_d of only about 6.

3.2 Ion Exchange Column Test

The sulfate concentration during the AN-107 loading phase was monitored for each half-column volume (15-mL). The sulfate loading/breakthrough curve, as determined by the concentration/initial concentration ratio (C/C_o) as a function of column volume, is given in Figure 3.1. The sulfate concentration changes only in the initial loading stages through the first 2 column volumes. This decrease in sulfate concentration is probably attributable to simple mixing of the sulfate containing AN-107 feed with the non sulfate containing column conditioning solution of 0.1 M NaOH/0.25 M $NaNO_3$. After the initial loading stages, the sulfate concentration remained consistent with the feed sulfate concentration (within the error of the analytical method), indicating that no exchange onto the column resin was taking place.

The subsequent feed displacement samples manifested reduced sulfate concentrations consistent with sample dilution in the effluent. The first column volume of 0.5 M HNO_3 elution resulted in a spike in sulfate concentration. This could indicate the stripping of a small amount of sulfate exchanged onto the resin during the loading phase. The amount of stripped sulfate is small, coming to only about 0.1 meq. of sulfate ion, which is only the amount of sulfate contained in about 2.6 mL or 0.089 column volumes of the AN-107 feed solution with a sulfate concentration of 3900 µg/mL. The sulfate concentration changes during the various parts of this column test are illustrated in Figure 3.2, which shows the sulfate concentration found in solution relative to the feed sulfate concentration.

Other measured analytes displayed similar behavior relative to loading and recovery. Analyte concentrations (C) and C/C_o (where C_o is the AN-107 feed concentration) are shown in Table 3.1. The column effluent concentrations are virtually identical to the AN-107 feed concentrations (Table 3.1) and the C/C_o values are close to 1.0. The ICP analysis shows that a significant amount of Ba was eluted with the 0.5 M HNO_3, leveling off at a nominal concentration of ca. 200 µg/g-eluate. Because Ba was not significantly present in the AN-107 load solution, the most likely source of the Ba must

be from the SL-655. During the load stage, Ba appears to have remained with the ion exchange resin. However, even during the feed displacement and column wash stages, detectable quantities of barium were observed in the eluate. Iron and manganese also eluted from the column in higher concentrations than the load solution. No evidence for any significant retention of the radionuclides ^{137}Cs, ^{90}Sr and ^{241}Am was observed. Not all column volume fractions are represented in the table, however analytical details of these samples can be found in Appendix B.

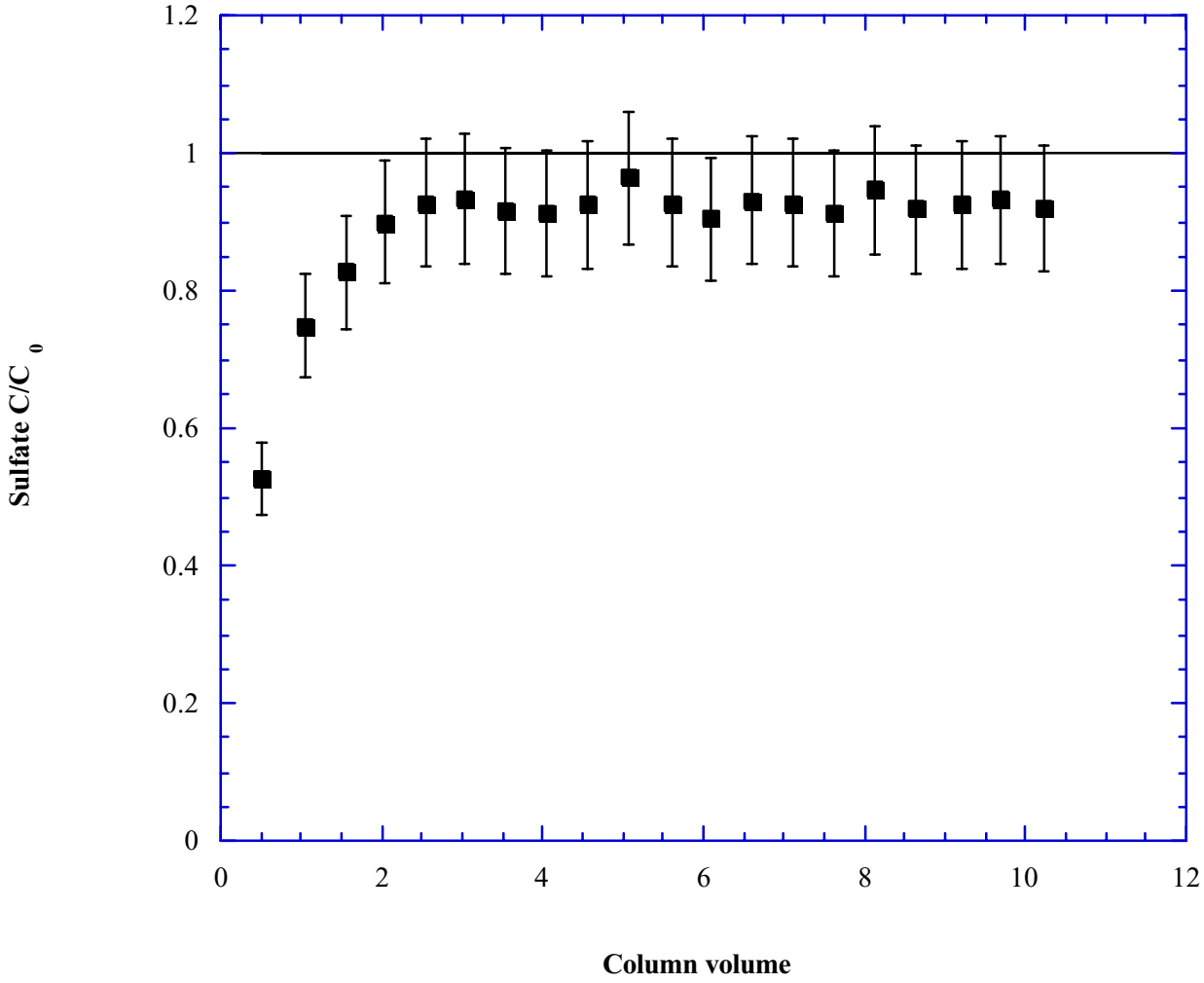
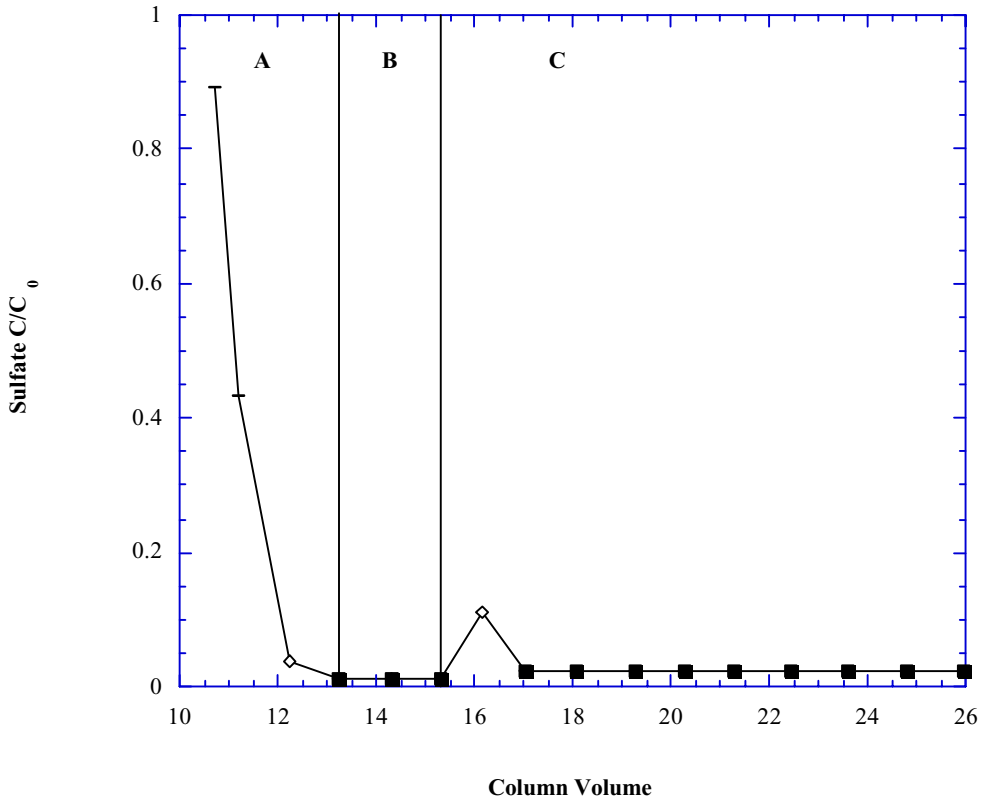


Figure 3.1. Sulfate Concentration Changes During Loading of Superlig® 655 Resin with an Archived Waste Sample from Tank AN-107



Eluent A represents Feed Displacement solution 0.25 $NaNO_3$-0.25M NaOH.
Eluent B represents Column Wash solution 0.25M $NaNO_3$.
Eluent C represents Elution solution 0.5M HNO_3.
The first 10 column volumes consisted of the AN-107 loading.

Figure 3.2. Sulfate Concentration Profiles in Eluates Relative to Feed Sulfate Concentration

Table 3.1. Cation, Anion, and Radioisotope Measurements for SL-655 Ion Exchange Column Wash and Elution Cycles

				Feed Displacement 0.25M $NaNO_3$-0.25M NaOH						Wash 0.25M $NaNO_3$				Sulfate Elution 0.5M HNO_3								
	AN-107 Feed	SO_4 effluent composite		*Column Volume* 11.2		12.25		13.25		*Column Volume* 14.3		15.3		*Column Volume* 16.2		17.1		18.1		19.1		MRQ
Analyte	µg/mL	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL
Al	133	125	0.94	69.0	0.52	5.8	0.04	2.95	0.02	1.8	0.01	<0.12		83.7	0.63	32.8	0.25	29.2	0.22	[8.6]	0.06	75
Ba	<1	[1.2]		129.0		26		9.76		8.37		8.89		478		1280		523		459		78
Ca	162	152	0.94	81.9	0.50	6.9	0.04	[1.4]	0.01	[1.1]	0.01	[1.9]	0.01	110	0.68	144	0.89	85	0.52	98	0.61	150
Cd	26	25	0.93	12.9	0.49	0.90	0.03	[0.05]	0.002	<0.03		<0.03		14.2	0.54	5.07	0.19	[3.2]	0.12	<0.4		7.5
Co	[2.1]	[2.]	0.99	<1.3		<0.1		<0.1		<0.1		<0.1		<2		<2		<2		<2		30
Cr	13	12	0.92	6.3	0.48	0.38	0.03	<0.04		<0.04		<0.04		7.21	0.55	[3.3]	0.25	[2.1]	0.16	<0.5		15
Cu	18	15	0.85	10.5	0.58	1.3	0.07	[0.08]	0.004	[0.22]	0.01	0.58	0.03	9.76	0.54	7.34	0.41	[2.1]	0.12	<0.6		17
Fe	3	[2.4]	0.77	1.1	0.34	0.11	0.04	<0.05		<0.05		<0.05		[2.2]	0.70	8.77	2.80	8.62	2.75	[2.0]	0.64	150
K	723	606	0.84	[473]	0.65	162	0.22	152	0.21	107	0.15	78.3	0.11	[77]	0.11	[260]	0.36	[190]	0.26	[150]	0.21	75
La	<0.5	<1		<1.3		<0.1		<0.1		<0.1		<0.1		<2		<2		<2		<2		35
Mn	[2.1]	<1		<1.3		<0.1		<0.1		<0.1		[0.53]	0.26	[3.5]	1.69	54.4	26	41.1	20	[4.1]	2	150
Na	114200	95600	0.84	56652.5	0.50	10485	0.09	7790	0.07	6200	0.05	5220	0.05	77900	0.68	32600	0.29	14600	0.13	2310	0.02	75
Ni	222	211	0.95	117.2	0.53	8.8	0.04	[0.59]	0.003	[0.26]	0.00	[0.2]	0.00	124	0.56	42.2	0.19	27.4	0.12	[2.3]	0.01	30
Pb	77	64	0.83	39.6	0.52	2.7	0.04	<0.2		<0.2		<0.2		38.3	0.50	[7.0]	0.09	<3		<3		300
Si	101	131	1.30	301.0	2.98	81.7	0.81	102	1.01	64.1	0.63	15.6	0.15	563	5.57	[62]	0.61	[96]	0.95	[80]	0.79	170
Sn	<15	<30		<38		<3		<3		<3		<3		<37		<37		<37		<37		1500
Ti	<0.3	<0.5		<0.62		<0.05		<0.05		<0.05		<0.05		<0.62		<0.62		<0.62		<0.62		17
U	[41]	[47]	1.13	<51		<4		<4		<4		<4		<50		<50		<50		<50		600
Zn	6	[5.8]	1.00	[5.2]	0.89	0.38	0.07	1.5	0.26	[0.12]	0.02	[0.37]	0.06	[3.5]	0.61	[2.7]	0.47	[1.9]	0.33	16.8	2.91	16.5
TOC	14000																					1500
TIC	6800																					1500
Cl	930	940	1.01	560	0.60	<50		<25		<25		<25		90		<50		<50		<50		3000
F[1]	3600	3400	0.94	1900	0.53	140	0.04	<25		<25		<25		400		120		100		<50		15000
PO_4	1400	1300	0.93	<200		<100		<50		<50		<50		<100		<100		<100		<100		2500
SO_4	3920[2]	3500	0.89	1700	0.43	150	0.04	<50		<50		<50		430		<100		<100		<100		200
	µCi/mL	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL	C/Co	µCi/mL
^{137}Cs	2.67E-2	2.39E-2	0.90	1.64E-2	0.61	5.93E-3	0.22	4.71E-3	0.18	3.42E-3	0.13	2.30E-3	0.09	1.38E-3	0.05	1.67E-3	0.06	1.30E-3	0.05	1.04E-3	0.04	9.00E+0
^{241}Am	4.52E-3	4.45E-3	0.98	1.87E-3	0.41	1.28E-4	0.03	<3E-5		<2E-5		1.10E-5	0.002	2.27E-4	0.05	1.48E-3	0.33	4.05E-4	0.09	6.20E-5	0.01	7.75E-4
^{90}Sr	6.87E-1	6.02E-1	0.88	3.49E-1	0.51	4.85E-2	0.07	7.38E-3	0.01	5.60E-3	0.01	6.51E-3	0.01	5.04E-2	0.07	3.92E-1	0.57	1.29E-1	0.19	3.33E-2	0.05	3.00E-1
Additional opportunistic analytes																						
	µg/mL	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL	C/Co	µg/mL
B	21	25	1.19			12		13.6	0.64	9.37	0.44	4.84	0.23									NMRQ
Mg	<1																					NMRQ
Mo	15	14	0.93			0.42																NMRQ
Nd	[1.3]																					NMRQ
P	203	177	0.87	158	0.78	40		5.03	0.02	[1.1]	0.01	[0.9]	0.00	99.6	0.49	109	0.54	36.9	0.18	[4.6]	0.02	NMRQ
Rh	[5]																					NMRQ
Ru	[15]																					NMRQ
Sr	91	60	0.65			5.8		1.05	0.01	0.726	0.01	0.844	0.01	35.4	0.39	280	3.07	93.6	1.03	24.4	0.27	NMRQ
W	[73]	[71]	0.97																			NMRQ
Y	<0.5																					NMRQ
Zr	[1.2]	[1.1]	0.91																			NMRQ
NO_2	28000	27000	0.96	16000	0.57	1200		<50						2700	0.10							NMRQ
NO_3	111000					23000																3000

Uncertainties typically do not exceed +/-15% (2-σ).

Note: bracketed values indicate uncertainties greater than +/-15% (2-σ).

1) Fluoride results are suspect because of retention time peak shift and peak shape.

2) Based on an average of 5 determinations.

4.0 CONCLUSIONS

The SL-655 resin was ineffective in removing sulfate from an archived sample of waste from tank AN-107. Both batch contact and small column tests indicate that insignificant quantities of sulfate from 241-AN-107 are bound by the SL-655. Taking into account the precision of the sulfate analyses, the sulfate batch K_d values for of SL-655 are determined to be between 0 and 6 and the column tests indicate that no more than 0.09 column volumes of sulfate of the 10 column volumes of AN-107 feed passed through the system were retained by the resin. In addition, the elution of significant quantities of barium from the SL-655 during the process is troubling. If the assumption is made that the presence of one equivalent of barium in the resin is required to bind one equivalent of sulfate, then the capacity equivalent of 0.25 grams of dry resin was lost in this one test cycle.

Finally, it should be noted that, if the performance of SL-655 is tied to the affinity of Ba for sulfate, the poor results shown in the batch and column tests are consistent with the results from concomitant studies examining selective precipitation of sulfate by barium from the same 241-AN-107 solution. In these precipitation tests, the presence of large amounts of carbonate in the AN-107 solution were shown to inhibit barium precipitation of sulfate, even when Ba:sulfate ratios were significantly greater than were employed in this study. However, a much more effective precipitation of sulfate from carbonate depleted AN-107 solutions was observed. Should there be any interest in trying to further apply SL-655 to sulfate removal of Hanford tank supernatants, an initial processing step to remove carbonate might enhance column performance.

5.0 REFERENCES

Hallen, R. T., K. P. Brooks, and L. Jagoda. 2000. *Demonstration of the Sr/TRU Removal Process with Archived AN-107 Waste*, BNFL-RPT-026, PNWD-3033, Battelle, Pacific Northwest Division, Richland, Washington.

Hendrickson, D.W. 1997. *Hanford Complexant Concentrate Cesium Removal Using Crystalline Silicotitanate*, SESC-EN-RPT-005, SGN Eurisys Services Corporation Richland, Washington.

APPENDIX A

Appendix A: Test Instructions

APPENDIX B

Appendix B: Analytical Data

APPENDIX C

Appendix C: Sample Spreadsheets

Table C.1. Batch Contacts: Archive AN-107 Contacted with Superlig®-655 (SL-655)

Performed 09/16/99 through 09/20/99, as per " Batch Contact Test Instructions
for SL-655 and Archive AN-107 Sample", BNFL-TI-29953-056
Distribution coefficient calculation: Kd = [(C_0-C1) / C1] x [V / (M x F)]

Archive AN-107 density = 1.22 g/mL
SL-655 F factor = 0.454

Sample ID	resin mass (g)	solution mass (g)	solution volume (mL)	sulfate conc., M	Kd (mL/g)
AAN-C	0	6.1655	5.054	3949.1	
AAN55-04	0.3305	6.1933	5.076	3886.7	0.543
AAN55-08	0.33	6.2	5.082	3953.7	-0.039
AAN55-16	0.3299	6.1733	5.060	3843.6	0.927
AAN55-24	0.3299	6.1725	5.059	3905	0.381
AAN55-72	0.3305	6.238	5.113	3979.1	-0.257
AAN-55	0.3304	6.1883	5.072	3891	0.505
AAN55D	0.3287	6.1962	5.079	3873.9	0.661

Table C.2. Column Flow Testing of SL-655 for Sulfate Removal from Archive AN-107

feed density =	1.22	g/mL								
Archive AN-107 sulfate concentrations										
AN-107 start	3632.7		Bed volume =	29.1	mL					
AN-107 start	4244.2									
AN-107 start	4024.4									
AAN-C-A	3949.1									
99-2663 SO4 IX Feed	3763.9									
Average	3922.9									
Standard Deviation	236.5									
							Sulfate			
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co			
	hr	g	mL	ug/mL	BV	BV	average			
99-2663 SO4 IX Feed							3922.9	feed		
99-2664 AN-SO-L1A	0.20	18.1994	14.9	2100	0.51	0.51	53.5%			
99-2665 AN-SO-L2A	0.21	19.3336	15.8	2900	0.54	1.06	73.9%			
99-2666 AN-SO-L3A	0.14	18.0048	14.8	3200	0.51	1.56	81.6%			
99-2667 AN-SO-L4A	0.17	17.287	14.2	3500	0.49	2.05	89.2%			
99-2668 AN-SO-L5A	0.17	17.5912	14.4	3600	0.49	2.54	91.8%			
99-2669 AN-SO-L6A	0.18	17.7314	14.5	3700	0.50	3.04	94.3%			
99-2670 AN-SO-L7A	0.15	17.9241	14.7	3600	0.50	3.55	91.8%			
99-2671 AN-SO-L8A	0.17	17.7684	14.6	3600	0.50	4.05	91.8%			
99-2672 AN-SO-L9A	0.17	18.0581	14.8	3600	0.51	4.55	91.8%			
99-2673 AN-SO-L10A	0.17	18.5403	15.2	3800	0.52	5.08	96.9%			
99-2674 AN-SO-L11A	0.17	18.5183	15.2	3600	0.52	5.60	91.8%			
99-2675 AN-SO-L12A	0.17	17.2941	14.2	3500	0.49	6.08	89.2%			
99-2676 AN-SO-L13A	0.17	18.1606	14.9	3700	0.51	6.59	94.3%			
99-2677 AN-SO-L14A	0.17	17.9832	14.7	3600	0.51	7.10	91.8%			
99-2678 AN-SO-L15A	0.17	18.1539	14.9	3600	0.51	7.61	91.8%			
99-2679 AN-SO-L16A	0.17	18.4088	15.1	3700	0.52	8.13	94.3%			
99-2680 AN-SO-L17A	0.17	18.3436	15.0	3600	0.52	8.65	91.8%			
99-2681 AN-SO-L18A	0.18	19.8415	16.3	3600	0.56	9.20	91.8%			
99-2682 AN-SO-L19A	0.16	17.3692	14.2	3700	0.49	9.69	94.3%			
99-2683 AN-SO-L20A	0.17	18.8238	15.4	3600	0.53	10.22	91.8%			
99-2684 AN-SO-L21A	0.17	18.032	14.8	3500	0.51	10.73	89.2%			
99-2685 AN-SO-L22A	0.17	17.063	14.0	1700	0.48	11.21	43.3%			
avaerage eff composite				3500			89.2%			
Total	3.72		326.6	ave flow rate =	3.01	BV/hr				

Bed height changed from 6 cm at start to 5.5 cm at end. Average bed volume used.

Performed 9/15/99 - 9/18/99 per "Test Instructions for Sulfate Removal Column Testing Using Envelope C (AN-107 Archive) Solution", BNFL-TI-29953-054, Rev 1.

Samples L1-L20 were composited. L21 and L22 were partially feed displacement solution.

Table C.2. Column Flow Testing of SL-655 for SO4 Removal from Archive AN-107 (cont')

0.25 M NaNO3/0.25 M NaOH Feed Displacement								
approximate density of 0.25 M NaNO3/0.25M NaOH =					1.024	g/mL	Sulfate	
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2687 AN-SO-FD1	0.33	31.0753	30.3	150	1.04	12.25	3.8%	
99-2688 AN-SO-FD2	0.34	29.8419	29.1	<50	1.00	13.25	<1.3%	
Total	0.7		59.5	ave flow rate =	3.02	BV/hr		
0.25 M NaNO3 wash								
approximate density of 0.25 M NaNO3 =			1.012	g/mL			Sulfate	
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2689 AN-SO-CW1 MS	0.34	31.0753	30.7	<50	1.05	14.30	<1.3%	
99-2690 AN-SO-CW2 MS	0.32	29.8419	29.5	<50	1.01	15.32	<1.3%	
Total	0.66		60.2	ave flow rate =	3.13	BV/hr		
Elution								
approximate density of 0.5 M nitric acid =			1.014	g/mL		Sulfate		
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2692 AN-SO-E1-A	1.00	25.1152	24.8	427	0.85	0.85	10.9%	
99-2693 AN-SO-E2-A	0.88	26.3731	26.0	<100	0.89	1.74	<2.5	
99-2694 AN-SO-E3-A	1.00	30.1769	29.8	<100	1.02	2.76	<2.5	
99-2695 AN-SO-E4-A	1.00	35.0744	34.6	<100	1.19	3.95	<2.5	
99-2696 AN-SO-E5-A	1.00	29.9812	29.6	<100	1.01	4.97	<2.5	
99-2697 AN-SO-E6-A	1.00	29.0696	28.7	<100	0.98	5.95	<2.5	
99-2698 AN-SO-E7-A	1.06	34.4395	34.0	<100	1.17	7.12	<2.5	
99-2699 AN-SO-E8-A	0.96	34.0742	33.6	<100	1.15	8.27	<2.5	
99-2700 AN-SO-E9-A	1.02	36.1764	35.7	<100	1.22	9.49	<2.5	
99-2701 AN-SO-E10-A	1.00	33.6707	33.2	<100	1.14	10.63	<2.5	
99-2702 SO4 Eff Composite A				<100			<2.5	
						ave flow rate =	1.07	BV/hr
Total	9.92	hr	309.8	mL				

Table C.2. Column Flow Testing of SL-655 for SO4 Removal from Archive AN-107 (cont')

Elution rinse							
approximate density of 0.25 M NaNO3		1.012	g/mL				
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co
	hr	g	mL	ug/mL	BV	BV	average
99-2703 AN-SO-ER1	1.01	31.1126	30.7	<100	1.06	1.06	<2.5
99-2704 AN-SO-ER2	0.99	31.4171	31.0	<100	1.07	2.12	<2.5
Total	2.00		61.8	ave flow rate =	1.06	BV/hr	
Regeneration			Note: BDL; below detection limit				
approximate density of 0.25 M NaNO3/0.25M NaOH =				1.024			
	mass	volume	Sulfate	Bed volumes	C/Co		
	g	mL	ug/mL	BV			
99-2705 AN-SO-REGN	61.4488	60.0	<100	2.06	<2.5		
		Note: BDL; below detection limit					

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APPENDIX A

Appendix A: Test Instructions

Batch Contact Test Instructions for SL-655 and Archive AN-107 Sample

BNFL Project Document Number: 29953-056, rev 0

Effective Date: September 16, 1998

Controlling Test Plan: 29953-03

Author Approval: D E Kurath 9/16/99
D.E. Kurath. Date

Technical Reviewer: David Z Blanchard 9/16/99
D.L. Blanchard Date

Objective: To perform the batch contact tests with SL-655 and archive AN-107 sample that has been treated for Sr/TRU removal.

CAUTION: FOLLOW THE INSTRUCTIONS LISTED BELOW.

Materials: Distilled de-ionized water
Waste samples (archive AN-107 filtrate composite)
Superlig 655 for SO_4 removal (abbrev. SL-655)

Equipment: Standard laboratory glassware and plasticware
Syringe filters, 0.2-0.8 um (10-12)
Thermometer to record temperature in lab 410 and 507
Splash shield for separation of equipment for radiological work in fume hoods
20 mL scintillation vials
Shaker table

Wastes: A sample of archive AN-107 (envelope C) will be used. The actual AN-107 will be tested at a later date.

Location: Solution and materials preparations will be done in labs in Pacific Northwest National Laboratory's Radiochemical Processing Laboratory (RPL). Actual waste sample preparations and batch contact tests will be performed in the lab 410 and 507 of the RPL.

Starting Date:

Balance No.:_____ Location: _____ Next Calibration Date:

Balance No.:_____ Location: _____ Next Calibration Date:

Balance No.:_____ Location: _____ Next Calibration Date:

Balance No.:_____ Location: _____ Next Calibration Date:

Balance No.:_____ Location: _____ Next Calibration Date:

Section A: Feed Characterization

A1.___ Locate the actual waste bottle in lab 410 fume hood. Record the actual labels and the appearance (color, cloudiness, precipitates, etc.).

Envelope C (AN-107) Label(s):

Appearance:

Dose rate if given:

A2.___ If data are available, determine the SO_4 concentration using the feed analytical characterization data collected for these samples. Record the data report used and show the calculations here.

Report(s) Used: _____

A3.___ If data are available, determine the CrO_4 concentration using the feed analytical characterization data collected for these samples. Record the data report used and show the calculations here.

Report(s) Used: _____

Sulfate Batch Contacts

The SL-655 material is untested. BNFL has directed PNNL to use a V/M of approximately 15 mL per gram, so 5mL/0.33 g will be used. The resin is stored in 1 M NaNO3 and is not to be dried or washed with water but will be weighed damp. Three unspiked samples will be run with one control.

Section B: Vial Preparation

Note: For each resin, the samples for the contacts should all be weighed in one session.

B1.___ Label, tare and weigh materials into 20 mL scintillation vials using Tables B.1. The SL-655 should be damp but not saturated with the solution it is currently stored in.

B2.___ Label, tare and weigh materials into 20 mL scintillation vials using the following table for the SO_4 batch contacts.

Table B.1: Material Contact Vial Weights - SO_4 Batch Contacts.

Vial ID	IX Material	Vial Mass (incl. Cap) (g)	Vial + cap + resin mass (g)	Resin Mass (g)	Required Resin Mass (g)	Date
AAN-C	None	16.8750			-	9/16/99
AAN55	SL-655	16.8697	17.2030	0.3304	0.330±0.002	9/16
AAN55-D	SL-655	16.8604	17.1891	0.3287	0.330±0.002	
AAN55-04	SL-655	17.0890	17.4195	0.3305	0.330±0.002	
AAN55-08	SL-655	16.8669	17.1969	0.3300	0.330±0.002	
AAN55-16	SL-655	16.9226	17.2525	0.3299	0.330±0.002	
AAN55-24	SL-655	16.8330	17.1629	0.3299	0.330±0.002	
AAN55-48	SL-655	17.0436	17.3747	0.3311	0.330±0.002	
AAN55-72	SL-655	16.9354	17.2659	0.3305	0.330±0.002	

F factors — ~~16.9726~~ one — wrem — dry @ 95°C — wet mass — dry mass

555F1	16.9726	19.8206	18.2656 g	2.8488	1.293
555F2	16.9891	19.8818	18.2933 g	2.8727	1.3042

f factor

555F1 ~~5FFF1~~ 0.454

555F2 0.454

Section C: Batch Contacts

The contacts will be performed in Room 507 using a shaker on the bench.

C1.___ Pipette 5 mL of the archive AN-107 sample into the vials as shown in Table C.1 below. Note that the sample will be pipeted into the vials at different times, and that this experiment requires 4 days to complete. Make sure to start this experiment on a Monday, or Thursday and make sure that you will be here to start and to collect samples at the appropriate times (see Table C.1), and that you have RCT coverage as appropriate.

C2.___ Check the masses of each capped vial with solution when the solution is added, and record in Table C.1 below. Also record the actual date and time for each.

C3.___ Place the capped vial in the shaker table on the bench top immediately. Follow all appropriate radiological procedures. Shaker set on low ~ 180 rpm

C4.___ Record the room temperature (rm 507) in Table C.1 each time you start a contact. Also transfer the figure for the mass of each vial from Table B.1, column "Vial Mass" to Table C.1, column "Mass," and determine the mass of solution added to the vial.

C5.___ Prepare for collection of the samples by assembling 9 syringe filter setups, and erecting a clear splash shield in the hood. Keep some extra syringe filter stuff handy in case you need more.

C6.___ Remove the vials from the shaker after the target contact time specified in Table C.1. Record the actual date and time that they were removed, and check the mass of each vial.

C7.___ Immediately separate the solution from the SL655 resin by filtering the solution through a clean syringe filter into the appropriate "-A" vial (i.e., AAN-C into AAN-C-A, etc.). Perform the filtration behind the splash shield, and cap vials immediately.

C8.___ Record the room temperature in Table C.1 each time you stop one of the contacts. Also transfer the figure for the mass of each vial from Table D.1, column "Vial Mass" to Table C.1, column "Mass," and determine the mass of solution in the vial.

C9.___ The filtered samples should be submitted for IC anion analysis on Monday, Sept 20. These samples will be batched with other samples requiring IC to determine sulfate.

Table C.1: Material Contact Weights and Start Times for SO_4 Kd versus Time Batch Contacts.

Vial ID	Target contact time, (hrs)	Target start time	Target stop time	Start Date, Time	End date, time	Vial +cap resin (g)	Vial + cap + resin + Sltn (g)	Sltn Net (g)	Temp at start (°C)	Temp at stop (°C)
AAN-C	none	9/16/99 11:00	9/20/99 11:00	9/16/99 11:50	9/20/99 11:54	16.8750	23.0405	6.1655		23.0
AAN55	96	9/16/99 11:00	9/20/99 11:00	9/16/99 11:50	9/20/99 11:51	17.2001	23.3884	6.1883		23.0
AAN55-D	96	9/16/99 11:00	9/20/99 11:00	9/16/99 11:50	9/20/99 11:57	17.1891	23.3853	6.1962		28.0
AAN55-04	4	9/17/99 8:30	9/17/99 12:30	9/17/99 07:40	9/17/99 11:40	17.4195	23.6128	6.1933	22.7	23.9
AAN55-08	8	9/17/99 7:45	9/17/99 15:45	9/17/99 07:35	9/17/99 15:35	17.1969	23.3969	6.2000	22.7	28.7
AAN55-16	16	9/16/99 16:00,	9/17/99 8:00	9/16/99 16:00	9/17/99 08:00	17.2525	23.4258	6.1733		22.7
AAN55-24	24	9/16/99 15:30	9/17/99 15:30	9/16/99 16:00	9/17/99 16:00	17.1629	23.3354	6.1725		28.7
~~AAN55-48~~ Delete	~~48~~	~~9/20/99 8:00~~	~~9/22/99 8:00~~			17.3747				
AAN55-72	72	9/17/99 11:00	9/20/99 11:00	9/17/99 11:30	9/20/99 11:45	17.2659	23.5039	6.2380	24.0	28.0

measured in triplicate.

Working Copy

TEST INSTRUCTIONS FOR SULFATE REMOVAL COLUMN TESTING USING ENVELOPE C (AN-107 ARCHIVE) SOLUTION

BNFL Project Document Number: 29953-054, Rev. 0
Effective Date: 09/10/1999
Controlling Procedure/Test Plan Number: 29953-003

Proj 29953
Task ~~2.12.3.1~~ 12
T 2.12.3.1

Author: DE Kurath for
JR Bontha

Technical reviewer: David L. Blanchard 9-14-99
~~DE Kurath~~
DL BLANCHARD

SYSTEM DESCRIPTION

The ion exchange column system is diagramed in Figure A.1. It consists of a small column containing SL-655 ion exchanger, a small metering pump and two valves. Valve 1 is placed at the outlet of the pump and may be used to eliminate air from the system or isolate the columns from the pump. Valve 2 is primarily used for obtaining samples or redirecting the effluent to the collecting vessel.

The term "sample position" will be used to denote directing flow into a sample bottle. Closed denotes no flow. Valve 1 and 2 are closed when the handles point away from the sample port or the flow line.

NOTE: Valve 1 should always be in the flow position throughout the entire experiment unless otherwise specified.

The process testing steps are:

Process Step	Solution	Total Volume, mL (Column Volumes)	Flow Rate in Column Volumes per Hour (mL/hr)	Est. Time in Hours
Column Preparation	0.1M NaOH + 0.25M $NaNO_3$	90 (3)	3 (90 mL/hr)	1 hr
Loading	AN-107 Feed	300 (10)	3 (90 mL/hr)	3.33 hrs
Feed Displacement	0.1M NaOH + 0.25M $NaNO_3$	60 (2)	3 (90 mL/hr)	0.67 hrs
Column Washing	0.25M $NaNO_3$	60 (2)	3 (90 mL/hr)	0.67 hr
Elution	0.5M HNO_3	300 (10)	1 (30 mL/hr)	10 hrs
Elution Rinse	0.25M $NaNO_3$	60 (2)	1 (30 mL/hr)	1 hr
Regeneration	0.1M NaOH + 0.25M $NaNO_3$	60 (2)	3 (90 mL/hr)	0.67 hrs

NOTE: The column volume is based on a volume of 30 mL with a height ~ 5.9-cm or 2.33-inches.

CAUTION: Water or chelating compounds (e.g., EDTA) that are not associated with metals should not contact with SuperLig® 655 because the divalent cation may be extracted and precipitate in the contacting solution.

CAUTION: Carefully monitor the pressure during all column operations to ensure the pressure remains below 40-45 psi. Normal operating pressure is less than 5 psig.

CAUTION: It is difficult to remove air from the system, especially from the ion exchange bed. Valve 2 should be closed prior to turning off the pump and opening or loosening of the plunger so that the system doesn't drain and draw air into the beds. It is possible to recover from air in the beds up to the point at which the experiment has been started. **Once the experiment is started, air in the resin beds can not be removed with out disrupting the resin bed, which will invalidate the experiment and about $200k worth of work.**

USEFUL INFORMATION

Density of feed = 1.2281 g/mL @24 °C(determined with a 25 mL volumetric flask)
Density of 0.1 M NaOH + 0.25M $NaNO_3$@ 25 °C= 1.008 g/mL.
Density of 0.25 M $NaNO_3$ @ 25 °C= 1.001 g/mL.
Density of 0.5 M nitric acid @ 25 °C = 1.014 g/mL

PRE TEST PREPARATIONS

1. Label and tare the 20 and 40-mL collection vials shown in Tables A.1 to A9.

2. Record the information about the balances to be used

(A) Balance	362-06-01 - 040	Location	lab 410	Calibration	8/17/99 due 8/2000
(B) Balance	_____	Location	_____	Calibration	_____
(C) Balance	_____	Location	_____	Calibration	_____

3. Obtain an initial tare weight of a ~~500~~ <250-mL Bottle and Cap labeled "AN-107 SO4 IX Effluent Composite": _____ g.

4. Obtain the initial tare weight of a ~~500~~ <250-mL Bottle and Cap labeled "AN-107 IX Acid Eluant Composite": _____ g.

NOTES:

Sample dose - 4 mr/hr at ~~2~~ 2"
1.5 mr/hr at hood face

COLUMN PREPARATION

NOTE: The Superlig 655 resin is provided in a solution containing 0.25M $NaNO_3$. Water or chelating compounds (e.g., EDTA) that are not associated with metals should not contact with SuperLig 655 because the divalent cation may be extracted and precipitate in the contacting solution.

NOTE: The pump calibration curve is shown in Figure A2.

5. Record the height of the bed: ~6 cm.

6. Record the Room Temperature: 24.8

7. Obtain the tare weight of the Waste Bottle: — g.

8. Obtain the tare weight of the Waste Bottle with the Cap: — g.

9. Insert the effluent line from the Column into the Waste Bottle.

10. Insert the pump inlet line into the 0.1M NaOH and 0.25M $NaNO_3$ solution.

11. Set the pump controller to ~~5~~ 26 .78 (90 mL/hr).

12. Turn Valve 2 to the flow position to direct flow to the waste bottle.

13. Immediately turn the Pump ON and pump for 1 hour.
    Start Time & Date: 5:30 / 9/15/99
    End Time & Date: 6:30 / 9/15/99

14. Close Valve 2 and immediately turn Pump OFF. * Valve closed, Turn to sample when loading starts.

15. Obtain mass of Waste Bottle + 0.1M NaOH and 0.25M $NaNO_3$: — g.

    Mass of 0.1M NaOH and 0.25M $NaNO_3$ pumped = — g.

16. Record the height of the bed: ~6 cm cm.

NOTES:

COLUMN LOADING

NOTE: Prior to the start of the loading cycle, ensure that the tare weight of the Vial's + Cap's listed in Table A1 and A2 is determined and recorded.

17. Using a pipette, transfer 20-mL sample of the starting feed material into the 20-mL vial labeled ~~AN-SO-L0~~ SO4-1X-FEED. Record the weight of the Vial + Cap + Sample in Table A1.
18. Record the following information:

Resin Bed Volume: (6 cm)

Resin Bed Color: Yellow brown

Room Temperature: 23.8°C

Other Observations: —

19. Insert the pump inlet line into the AN-107 Feed solution.
20. Ensure that Valve 2 is in the no flow position
21. Ensure the pump controller is set to ~~1.4~~ 78 (90 mL/hr) and initiate pumping.

Start Time & Date: 7:42 AM, 9-16-99

22. Immediately open Valve 2 to allow solution to flow into the waste bottle
23. After 40 minutes, close Valve 2 and turn the Pump OFF.
24. Place Vial AN-SO-L1 under Valve 2, turn the pump ON and immediately turn Valve 2 to the sampling position
25. Record time of sampling in Table A1
26. After approximately 15-mL of the feed (takes ~ 10 min) is collected in the sample vial, turn the Pump OFF, quickly replace the vial AN-SO-L(n) with AN-SO-L(n+1), and turn the Pump ON.
27. Record stop time of collecting sample AN-SO-L(n) and start time of collecting AN-SO-L(n+1) sample in Table A1.
28. Record the weight of the Vial + Cap + Sample in Table A1.
29. Repeat Steps 26 to 28 until 10 CV (or 300-mL) of feed has been pumped through the column.

NOTE: Samples should be taken in the order given in Table A1. In all, 20 samples of the ion exchange effluent will be collected.

30. After the loading is completed, close Valve 2 and turn the pump OFF.

31. Record the Time and Date the pump is stopped and loading is complete: ~ 11:30 AM, 9-16-99

32. Record the height of the bed: 5.5 cm.

NOTES:

8:00AM Some feed went to waste bottle.

8:15 AM MUST HAVE BEEN SOME CHANNELING, OR AT LEAST BOUNDARY BETWEEN RESIDUAL NaOH AND FEED WAS NOT DISTINCT, BECAUSE THE LAST DROPS OF SO-L1 WERE A DIFFERENT DENSITY THAN THE REST OF THE LIQUID IN THE VIAL - OBVIOUS AS THE DROP FELL INTO THE LIQ IN THE VIAL - COULD SEE IT MIXING IN W/ THE REST OF THE LIQUID. THIS IS NOT THE CASE W/ SO-L2. ~~ALL~~ DROPS LOOK TO BE ~~THE~~ ALL THE SAME DENSITY. WHEN SO-L1 WAS STARTED, THE EFFLUENT WAS BROWN, SO SOME FEED WAS GETTING THROUGH.

10:55 NOTICE THAT BED HAS SHRUNK TO ~~7.5cm~~ ~5.5cm

11:15:00AM PUMP OFF, ~~FEED~~ INLET TUBE SWITCH TO FEED DISPLACEMENT SOLUTION (0.1 M NaOH, 0.25 M $NaNO_3$)

11:16:20AM PUMP BACK ON, SAMPLE SO-L20 STARTED

11:45 LOOKED LIKE FEED DISPLACEMENT SOLUTION WAS MORE DENSE THAN FEED. DROPS SANK TO BOTTOM OF VIAL AS VIAL NEARED FULL.

COLUMN STARTED LEAKING AT ~9:30 A.M. LOST <1 mL, THEN PLACED VIAL "LEAK" IN PLACE TO CATCH.

FEED LEAKED AROUND BOTH TOP & BOTTOM O-RINGS OF COLUMN. ~5 THREADS ON TOP (LIQ LEVEL). BOTTOM VOL UNKNOWN.

Preparation of the Loading Samples for Analysis

NOTE: All loading samples will be sub-sampled for analysis and the remaining samples will be mixed to prepare a composite of the feed effluent.

NOTE: Analytical vials need to be loaded out of the fume hood and therefore, need to be uncontaminated.

NOTE: Analytical sample preparation can be done at the completion of the experiment.

33. Prepare the analytical samples as indicated in Table A2.

34. After the analytical sample preparation is complete, transfer the remaining solution from each vial into the ~~500~~ 4250 mL bottle labeled "AN-107 SO4 IX Effluent Composite" – Tare = 52.16899 g (w/cap)

35. Cap and determine the weight of the effluent bottle: ——— g

    Mass of the processed waste stream = ——— g

36. Gently shake the effluent bottle to mix the contents.

37. Determine the tare weight of a 20-mL sample vial labeled "SO4 Effluent Composite" and record in Table A2.

38. Carefully pipette 20-mL of the composite into the vial.

39. Determine the weight of the Vial + Cap + Composite Sample and enter in Table A2.

NOTES:

FEED DISPLACEMENT

NOTE: After the loading cycle is completed, the column must be washed to displace feed. This is accomplished by pumping 2 CV (60 mL) of 0.1 M NaOH + 0.25M $NaNO_3$ solution through the column at a flow rate of 3 CV/hr and collecting the effluent in 1 CV increments (i.e. two samples of approximately 30 mL each will be collected).

NOTE: Feed displacement with a 0.25M $NaNO_3$ solution containing 0.1M NaOH removes any aluminate that might precipitate on the column during the column washing or acid elution steps.

NOTE: The feed displacement samples will be directly loaded out of the fume hood for analysis and therefore, care must be taken to ensure that the samples are uncontaminated.

40. Using a clean pipette, collect 20-mL of the 0.1 M NaOH + 0.25M $NaNO_3$ in a tared vial labeled the same and record the weight in Table A9.

41. Place the pump inlet line into the 0.1M NaOH + 0.25M $NaNO_3$solution.

42. Adjust the pump controller to ~~3.9~~ 78 (90 mL/hr).

43. Record the tare weight of the Sample Vials + Caps in Table A3.

44. Record the Room Temperature: 23.4°C @ 11:49:52

45. Place the vial labeled "AN-SO-FD-1" under the Valve 2.

46. Start the pump and immediately turn Valve 2 to the sampling position.

47. Record the Start Time and Date of the feed displacement: 11:46:20 AM (1st Feed Disp. vial).

48. After one column volume (~ 30 mL) of sample is collected in the vial, turn the Pump OFF, quickly replace vial "AN-SO-FD-1" with "AN-SO-FD-2", and turn the Pump ON.

49. After one CV (~ 30 mL) of sample is collected in the vial, close Valve 2, and turn Pump OFF.

50. Record Finish Time and Date of feed displacement: 12:27:00

51. Record the height of the bed: 5.5 cm.

NOTES:

12:08:00 INLET TUBE SWITCHED TO FEED DISPLACEMENT SOLUTION

NOTE: SAMPLES COLLECTED IN TABLES A3, A4, A5, and A7 HAVE A VOLUME OF 30-mL. FOR THIS REASON THE SAMPLE VIALS FOR ALL OF THESE SAMPLES SHOULD HAVE A VOLUME OF 40-mL.

9-16-99

Table A3. Data Log Sheet for Feed Displacement Samples using 0.1M NaOH + 0.25M $NaNO_3$ Solution.

Sample ID	Target Volume mL	Mass of Vial + Cap (tare), g	Time Sampling Started	Time Sampling Ended	Mass of Sample + Vial + Cap, g
AN-SO-FD1	30	25.6542	11:46:30 AM	12:06:30	56.4568
AN-SO-FD2	30	25.5798	12:06:30 AM	12:27:00	55.7880

SWITCHED TO WASH SOLUTION

Table A4. Data Log Sheet for Column Rinse Samples using 0.25M $NaNO_3$ Solution.

Sample ID	Target Volume, ML	Mass of Vial + Cap (tare), g	Time Sampling Started	Time Sampling Ended	Mass of Sample + Vial + Cap, g
AN-SO-CW1	30	25.7375	12:28:20	12:48:40	56.8128
AN-SO-CW2	30	25.9240	12:49:40	1:09:00	55.7659

9-17-99

Table A.5. Data Log Sheet for Elution Samples.

Sample #	Target Volume ML	Mass of Vial + Cap, g	Time Started	Time Ended	Mass of Sample + Vial + Cap, g
AN-SO-E1	30	25.7358	4:55AM 9-17-99	5:55AM 9-17-99	50.8510
AN-SO-E2	30	25.9101	5:55AM	6:48AM	52.2832
AN-SO-E3	30	25.6276	8:32:00AM	9:32:00 AM	55.8045
AN-SO-E4	30	25.6779	9:32:00AM	10:32:00 AM	60.7523
* AN-SO-E5	30	25.8912	10:38:00 AM	11:38:20 AM	55.8724
AN-SO-E6	30	25.5841	11:38:20 AM	12:38:00 PM	54.6537
AN-SO-E7	30	25.8081	1:30 PM	2:33:30 PM	~~60.2476~~ 60.2476
AN-SO-E8	30	25.8829	2:33:30PM	3:31:00 PM	59.9571
AN-SO-E9	30	25.6793	3:31:00PM	4:32:00 PM	61.8557
AN-SO-E10	30	25.6507	4:34:00 PM	5:34:00 PM	59.3214

Pump off
Pump on
Pump spd ↑ to 30
↓ 29 → 30

SWITCH TO ELUTION RINSE ON #10.

LEAK2 16.7146
SO4 Elute loop ≈200mL 52.2322 251.3
16.8376 30.9460
Precip Vol.

* Flow pump off for 6 minutes to remove air from stop bed & ensure all resin is ~~at~~ in vial with nitric acid. Liquid level was very near top of bed anyway.

COLUMN WASHING

NOTE: After the feed is flushed through the column, the column is washed with 2CV of 0.25M $NaNO_3$ solution at a flow rate of 3CV/hr and collecting the effluent in 1 CV increments (i.e. two samples of approximately 30 mL each will be collected).

NOTE: Flushing with 0.25M $NaNO_3$ solution removes any hydroxide might have been left behind on the column during the feel displacement step.

NOTE: The column washing samples will be directly loaded out of the fume hood for analysis and therefore, care must be taken to ensure that the samples are uncontaminated.

52. Record the tare weight of the sample bottles in Table A4.

53. Record the Room Temperature: _____

54. Using a clean pipette, collect 20-mL of the 0.25M $NaNO_3$ solution in a tared vial labeled the same and record the weight in Table A9.

55. Place the pump inlet line in the 0.25M $NaNO_3$ bottle.

56. Place the sample vial AN-SO-CW1 under Valve 2 in a sample holder.

57. Ensure that the pump controller is set at ~~3.9~~ 7.8 (90 mL/hr).

58. Start the pump and immediately turn Valve 2 to the sampling position.

59. Record the Start Time and Date of the column ~~washing~~ rinse: _____.

60. After one column volume (~ 30 mL) of sample is collected in the vial, turn the Pump OFF, quickly replace vial "AN-SO-CW-1" with "AN-SO-CW-2", and turn the Pump ON.

61. After one CV (~ 30 mL) of sample is collected in the vial, close Valve 2 and turn Pump OFF.

62. Record Finish Time and Date of column washing: _____

63. Record the height of the bed: _____cm.

NOTES:

COLUMN ELUTION

NOTE: After the feed is flushed through the column and the column is washed, it will be eluted with 10CV of 0.5M HNO_3. The eluant flow rate is 1CV/hr and will be collected in increments of 1CV (i.e. a total of 10 samples will be collected).

64. Using a clean pipette, collect 20-mL of the 0.5M HNO_3 acid eluant into the vial labeled "0.5M HNO_3", and record weight in Table A9.

65. Record the Room Temperature: 22.0°C @ 4:51 AM, 9-17-99

66. Record the tare weight of the Vials + Caps in Table A5.

67. Place the vial number AN-SO-E1 under Valve 2 in a sample holder.

68. Set the pump controller at ~~1.7~~ 26 (1 CV/hr or 30 mL/hr).

69. Turn the Pump ON and turn Valve 2 to the sampling position.

70. Record the Time and Date the elution is initiated: 4:55 AM, 9-17-99.

71. Collect 1 CV (30 mL) in each of the vials listed in the Table A5. Each vial should take 1 hour to collect. Record the mass of each Vial + Cap after the sample is obtained.

NOTE: During switching of the sample collecting vials, turn the Pump OFF, replace the vial, and turn the Pump ON.

72. When the elution is complete, close Valve 2 and turn the Pump OFF.

73. Record the time and date the elution is finished: _____.

NOTES:

9-17-99

4:50 AM FEED THAT LEAKED AROUND TOP O-RING HAS RUN BACK DOWN ONTO BED. 1ST ELUTION SAMPLE MAY NOT BE VERY REPRESENTATIVE.

5:50 AM FLOW RATE TOO SLOW - TURN PUMP SPEED UP TO 30.

6:50 AM BED IS DRY! ELUANT FILLED UP ABOVE O-RING. MUST BE BAD CONNECTION IN ADJUSTABLE PLUNGER. PUMP OFF AT 6:48 AM. SAMPLE VIAL ONLY ½ FILLED. PROBLEM PROBABLY BEGAN W/ 1ST VIAL.

8:32 AM PUMP BACK ON. THIRD SAMPLE COLLECTION STARTED.

Sulfate Column Plugging Incident
9-17-99

All times are AM.

4:50 Start elution. Pump set at 26, liquid dripping from valve into sample vial (E1). Feed (brown liquid) clearly still above O-ring of top plunger.

5:50 Switch to second sample vial (E2). Flow rate appears too slow – sample not up to the 30 mL mark on vial.Still clearly feed above O-ring of top plunger. Increase pump speed to 30.

6:48 Pump turned off. Sample vial only half full. No drips into column. Bed completely dry. Top level of liquid above O-ring has now risen out of sight behind connectors at top of column. Also lighter color – guess that the feed initially present mixed with the eluant. Guess that the top plunger frit is plugged. Apparently liquid is leaking out above frit and O-ring.

Loosen top plunger O-ring so liquid will flow down around it onto top of bed, and remove plunger. Air bubbles in bed removed using long plastic Pastuer pipet. Bed somewhat mixed during this procedure. While fooling with top plunger, force liquid into vial E2. Cloudy. Some solids settle to bottom of vial. Almost a full BV in E2 now.

Frit and screen removed from top plunger and then flow tested – looks good. Remove liquid on top of column (very cloudy - placed in vial "Precip volume") and re-insert plunger. Air between top plunger plastic-to-metal connection and bottom end of top plunger (where frit was).

8:32 Pump restarted. New sample vial (E3). Pump speed still at 30. Liquid dripping into vial.

9:32 Full (30 mL) sample collected in E3. Switch to E4.

12:38 Pump off because of high leak rate at bottom of column. Fittings tightened. Restart at 12:46. Leak not fixed -

1:49 Pump back on. Tightened up fittings on bottom plunger, + added some teflon tape to fitting threads on rod. Continue collecting in E7

4:34 INLET TUBE SWITCHED TO ELUTION RINSE SOLUTION AT START OF #10 ELUTION SAMPLE COLLECTION.

5:00PM T = 25°C

9-18-99 12:01 PM NO PRESSURE BUILDUP OR GAS GENERATION APPARENT.

Preparation of the Elution Samples for Analysis

NOTE: Each Elution sample will be sub-sampled for analysis and the remaining samples will be mixed to prepare a composite of the eluant.

NOTE: Elution samples for analysis will be samples will be directly loaded out of the fume hood and therefore, care must be taken to ensure that the samples are uncontaminated.

74. Prepare the analytical samples as indicated in Table A6.

75. After the analytical sample preparation is complete, transfer the remaining solution from each vial into the 500 mL bottle labeled "AN-107 IX ACID Eluant Composite"

76. Cap and determine the weight of the eluant bottle: _____g

    Mass of the eluant = _____g

77. Gently shake the eluant bottle to mix the contents.

78. Determine the tare weight of a 20-mL sample vial labeled "SO4 Eluant Composite" and record in Table A6.

79. Carefully pipette 20-mL of the composite into the vial.

80. Determine the weight of the Vial + Cap + Composite Eluant Sample and enter in Table A6.

NOTES:

Only composite ~~#~~ 3 2 - 10. Not 1, 2.

ELUTION RINSE

NOTE: After the column is eluted, it is washed with 2CV of 0.25M $NaNO_3$ solution at a flow rate of 1CV/hr and collecting the effluent in 1 CV increments (i.e. two samples of approximately 30 mL each will be collected).

NOTE: The elution rinse samples will be directly loaded out of the fume hood for analysis and therefore, care must be taken to ensure that the samples are uncontaminated.

81. Place the pump inlet line in the 0.25M $NaNO_3$ solution.

82. Record the Room Temperature: 24.0 °C 9-18-99 Bed height ≈ 6 cm

83. Record the tare weight of the vials in Table A7.

84. Place the vial labeled AN-SO-ER1 in the sample collection position under Valve 2 in a sample holder.

85. Set the pump controller to 3.9 (1 CV/hr, 30 mL/hr).

86. Turn the Pump ON and immediately turn Valve 2 to the sampling position.

87. Record Start Time and Date of Eluant Rinse: 1:26 PM 9-18-99

88. Collect 1 CV volume (~30 mL or sampling for 1 hr) in vial AN-SO-ER1, then turn the pump OFF, replace the vial "AN-SO-ER-1" with vial "AN-SO-ER2", and then turn the Pump ON.

89. Collect 1 CV volume (~30 mL or sampling for 1 hr) in vial AN-SO-E1-R2.

90. Close Valve 2 and turn the Pump OFF.

91. Record End Time and Date of Eluant Rinse: _____

92. Record the height of the bed: _____ cm.

NOTES:

2:46:00 INLET TUBE SWITCHED TO REGN SOLTN (0.1M NaOH/0.25M $NaNO_3$)

COLUMN REGENERATION

NOTE: After the elution solution is displaced, the column is washed with 2CV of 0.1M NaOH + 0.25M $NaNO_3$ solution at a flow rate of 3CV/hr and collecting the effluent in a single bottle.

93. Place the pump inlet line in the ~~0.25M~~ 0.1M NaOH + 0.25M $NaNO_3$ solution bottle.

94. Record the Room Temperature: 24.8°C @ 3:30PM 9-18-99

95. Record the tare weight of a 100-mL sample bottle labeled "SO IX Column REGN": 24.5457.

96. Place the "SO IX Column REGN" bottle under Valve 2.

97. Set the pump controller to ~~7.4~~ 7.8 (90 mL/hr, 3 CV/hr).

98. Turn on the pump and turn Valve 2 to the sample position.

99. Record the Start Time and Date: 3:29:00PM 9-18-99

100. Run the system for 0.67 hours (40 min).

101. Turn the pump OFF and close Valve 2.

102. Record the Finish Time and Date: 4:09:00PM 9-18-99

103. Record the mass of the regeneration effluent bottle + contents: 85.9945.

104. Pipette 20-mL of the regeneration effluent solution into a tared vial labeled "AN-SO-REGN"

105. Determine and record weight of Vial + Cap + Sample in Table A8.

106. Record the height of the bed: ~6.0 cm.

NOTES:

POTENTIAL PROBLEMS AND SOLUTIONS

PRESSURE RISE: If the pressure as indicated on the pressure gauge starts to rise a block in the flow is indicated. Inspect the valves to ensure that they are not closed. If the valves are all open a plug in the system is indicated. Attempt to isolate the location of the plug. The most likely place is the screen in the bottom of the columns. Contact the task leader before taking action to unplug the system.

AIR ABOVE THE RESIN BED AND BELOW THE PLUNGER: Air above the resin bed and below the plunger may be removed as follows. Close the valve after the column. Loosen the plunger by turning the white disk at the top of the column assembly. If you look at the disk from below, it is loosened by turned in the clockwise direction. Pump some fluid into the system with the valve closed. The pressure may rise slightly. The plunger may have to be forced down to expel air. Do not disrupt the bed. It is best to wait until you see liquid above the bed before forcing the plunger down. Once air is expelled retighten plunger by turning disk in the same way.

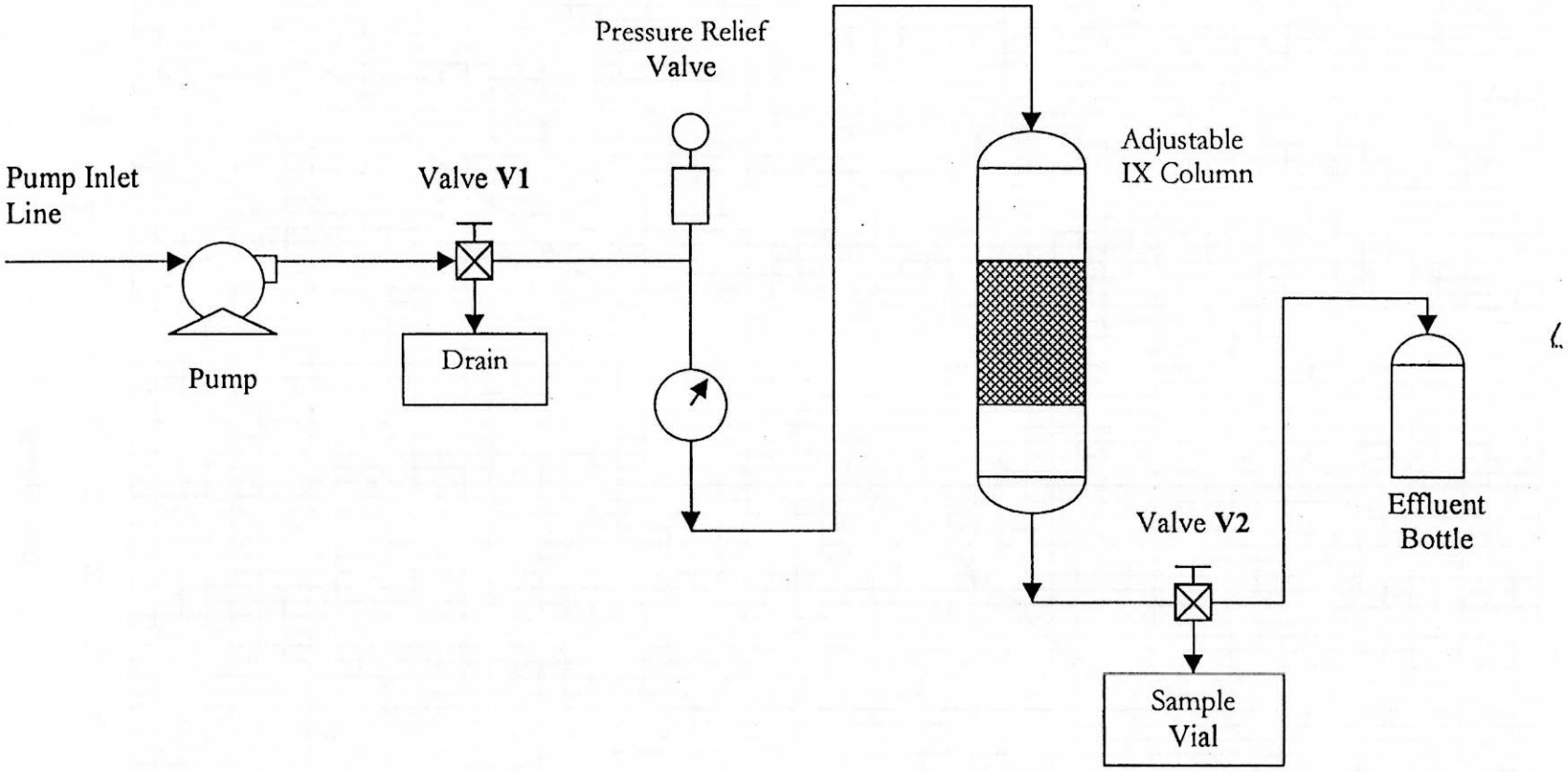


Figure A1. Schematic of the Sulfate Ion Exchange Column Setup

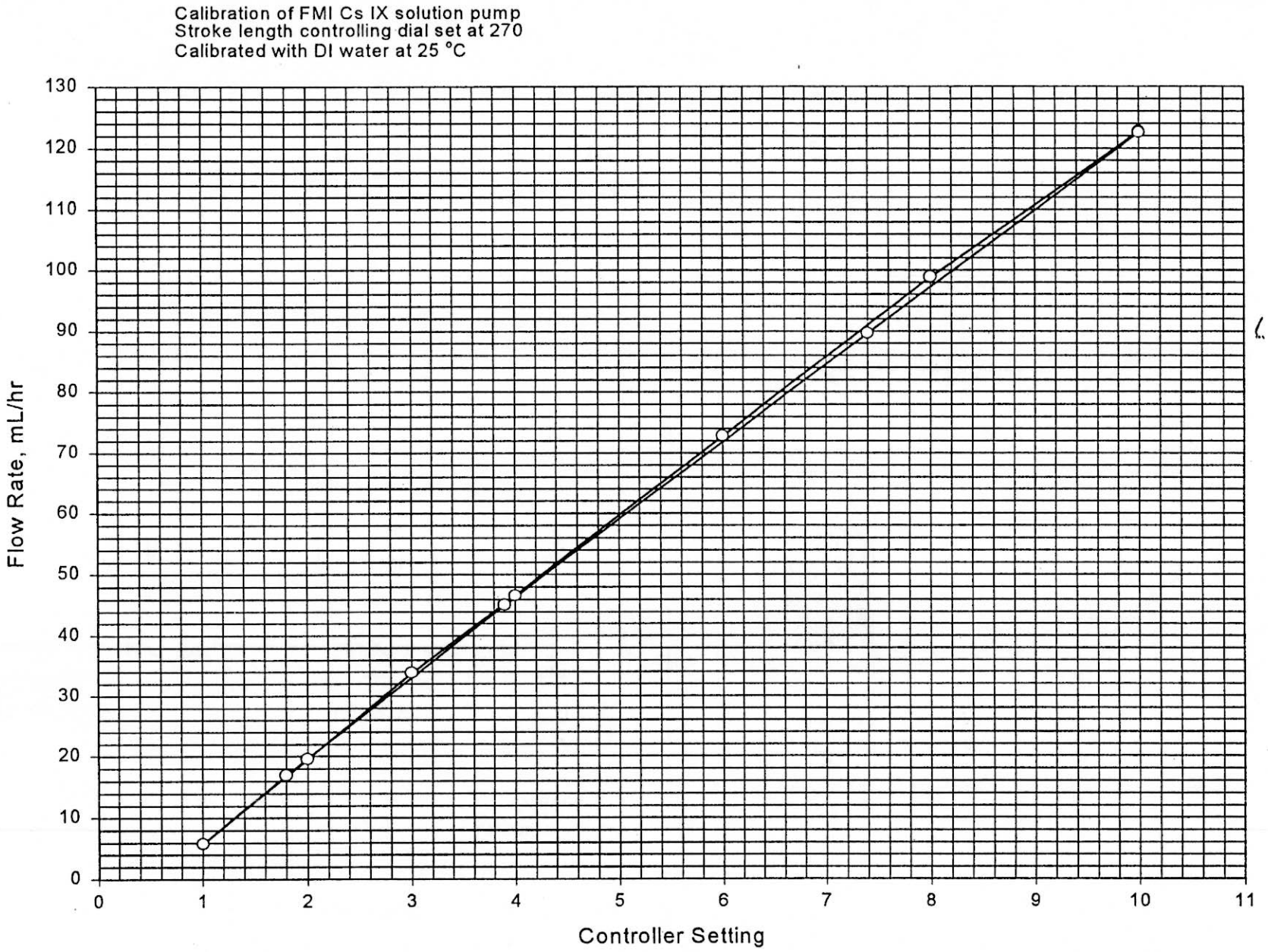


Figure A2. Pump Calibration

Table A-1. Data Log Sheet for Column Loading Samples.

9-16-99

Sample ID SO_4-1X-FEED	Time from Start of Feed (min)	Target Volume mL 20 ml	Mass Of Vial + Cap (Tare), g 25.8727	Time Sampling Started	Time Sampling Ended	Mass Of Sample + Vial + Cap g	Mass Of Sample g	Flow Rate and Notes
~~AN-SO-L0~~	0	~~7.5~~	~~16.9872~~	--	--			
AN-SO-L1	10	15	~~17.0389~~	8:02 AM	8:14:00 AM	34.9892		
AN-SO-L2	20	15	16.7456	8:14:00 AM	8:26:30 AM	36.0792		
AN-SO-L3	30	15	16.9862	8:26:30 AM	8:35:00 AM	34.9910		
AN-SO-L4	40	15	16.7869	8:35:00 AM	8:45:00 AM	34.0739		
AN-SO-L5	50	15	16.8703	8:45:00 AM	8:55:00 AM	34.4615		
AN-SO-L6	60	15	16.7958	8:55:00 AM	9:06:00 AM	34.5272		
AN-SO-L7	70	15	16.8358	9:06:00 AM	9:15:00 AM	34.7599		
AN-SO-L8	80	15	16.8767	9:15:00 AM	9:25:00 AM	34.6451		
AN-SO-L9	90	15	16.7759	9:25:00 AM	9:35:00 AM	34.8340		
* AN-SO-L10	100	15	16.9592	9:35:00 AM	9:45:00 AM	35.~~4323~~ 4995 *		
AN-SO-L11	110	15	16.9140	9:45:00 AM	9:55:00 AM	35.4323		
AN-SO-L12	120	15	16.7678	9:55:00 AM	10:05:00 AM	34.0619		← DI & CLOSE
AN-SO-L13	130	15	16.7870	10:05:00 AM	10:15:00 AM	34.9476	~	
AN-SO-L14	140	15	16.8745	10:15:00 AM	10:25:00 AM	34.8577		
AN-SO-L15	150	15	16.8289	10:25:00 AM	10:35:00 AM	34.9828		
AN-SO-L16	160	15	16.7849	10:35:00 AM	10:45:00 AM	35.1937		
AN-SO-L17	170	15	16.7771	10:45:00 AM	10:55:00 AM	35.1207		
AN-SO-L18	180	15	16.8925	10:55:00 AM	11:05:30 AM	36.7340		
AN-SO-L19	190	15	16.7714	11:05:30 AM	11:15:00 AM	34.1406		
AN-SO-L20	200	15	16.8464	11:16:20 AM	11:26:30 AM	35.6702		SWITCH TO FEED DIS

16.7898 –

SWITCH TO FEED DISP.

16.9159 — —

LEAK

AN-SO-L21 16.9351 11:26:30AM 11:36:30AM 34.9671 ——→ START TO SEE DENSITY CHANGE

AN-SO-L22 16.8803 11:36:30AM 11:46:30AM 33.9433

AN-SO-L23 16.7814 — —

* AN-SO-L10 Tot mass = 35.4995 g

Table A-2. Column Loading Samples Diluted for Analysis

9-16-99

Sample ID	Target Transfer Volume mL	Mass of Vial + Cap g	Mass of Vial + Cap + Sample g
AN-SO-L1A	7.5	16.7403	25.2687
AN-SO-L2A	7.5	16.8947	25.7839
AN-SO-L3A	7.5	16.9463	25.9554
AN-SO-L4A	7.5	16.9458	26.0312
AN-SO-L5A	7.5	16.9226	26.0398
AN-SO-L6A	7.5	16.8520	25.9579
AN-SO-L7A	7.5	16.7669	25.8826
AN-SO-L8A	7.5	16.8616	25.9789
AN-SO-L9A	7.5	16.7651	25.8837
AN-SO-L10A	7.5	16.9020	26.0385
AN-SO-L11A	7.5	16.7728	25.9100
AN-SO-L12A	7.5	16.7354	25.8601
AN-SO-L13A	7.5	16.9198	26.0400
AN-SO-L14A	7.5	17.0130	26.1421
AN-SO-L15A	7.5	16.9947	26.1255
AN-SO-L16A	7.5	16.8594	25.9904
AN-SO-L17A	7.5	16.7141	25.8720
AN-SO-L18A	7.5	16.8359	25.9769
AN-SO-L19A	7.5	16.8624	25.9954
AN-SO-L20A	7.5	16.8194	25.9584
SO4-Effulent-Composite	20.0	25.8407	61.9312
AN-SO-L21A	7.5	~~16.935~~ 16.8362	25.9795
AN-SO-L22A	7.5	16.8829	25.2733

SO_4 Effluent bottle composite

Pu.

Leah

195,061 0570

22.1358

9-17-99

Table A.6. Data Log Sheet for Preparation of Elution Samples for Analysis.

Sample ID	Target Transfer Volume, mL	Mass of Vial + Cap g	Mass of vial + Cap + Sample, g
AN-SO-E1A	7.5	16.9139	24.6403
AN-SO-E2A	7.5	16.8484	24.4408
AN-SO-E3A	7.5	16.9556	24.5815
AN-SO-E4A	7.5	16.8356	24.4342
AN-SO-E5A	7.5	16.9857	24.6388
AN-SO-E6A	7.5	16.9295	24.5131
AN-SO-E7A	7.5	16.7974	~~23.4701~~ 24.4152
AN-SO-E8A	7.5	16.6233	24.2381
AN-SO-E9A	7.5	17.0238	24.6218
AN-SO-E10A	7.5	16.7933	24.3949
SO4-Eluant-Composite	20.0	25.8640	46.2034

9-18-99

Table A.7. Data Log Sheet for Elution Rinse Samples (0.25M $NaNO_3$)

Sample ID	Target Volume mL	Mass of Vial + Cap (Tare), g	Time Started	Time Ended	Mass of Vial + Cap + Sample, g
AN-SO-ER1	30	25.6043	1:26:00 PM	2:26:30 PM	56.7169
AN-SO-ER2	30	25.5047	2:26:30 PM	3:26:00 PM	56.9218

INLET TUBE SWITCHED TO 0.1M NaOH 0.25M $NaNO_3$ @ 2:46:00

Table A.8. Data Log Sheet for Column Regeneration Sample (0.1M NaOH + O.25M $NaNO_3$)

Sample ID	Target volume mL	Mass of vial + cap g	Mass of Sample + Vial + Cap, g
AN-SO-REGN	20.0	16.8240	32.0069
SO IX COL REGN (125 mL plas bottle)	60 mL	24.5457	85.9945

Table A.9. Chemical Supply Samples – Archive Vials

Sample ID	Target volume mL	Mass of vial + cap g	Mass of Sample + Vial + Cap, g
0.1 M NaOH + 0.25 M $NaNO_3$	20		
0.25 M $NaNO_3$	20		
0.5 M HNO_3	20		

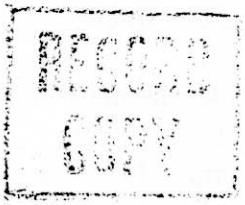
APPENDIX B

Appendix B: Analytical Data

Sulfate IC / Archive AN-107

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

29953
Task 12
T2.12.3.3



WO/Project: W48416 and W48418 / 29953
Client: D. Kurath

ACL Numbers: 99-02655 through 99-02708
ASR Number 5520

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Analyst: MJ Steele **Analysis Date:** September 21-23, 1999

M&TE: IC system (WD25214); Mettler AT400 Balance (360-06-01-031) See Chemical Measurement Center 98620 RIDS for IC File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: MJ Steele

Approval: Michael W Lumetta **Date** 10-25-99

Notes:

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

Final Results:

The liquid samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5520, with sample 99-2655 through 99-2684 for sulfate only. The samples were diluted at the IC workstation up to 2,000-fold to ensure that all anions were within the calibration range. The anion results are presented in the Table 1 and Table 2 below.

Q.C. Comments:

Besides the matrix spike QC, the following are results of analytical quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan. No duplicates were analyzed with these samples; however, similar samples from other samples submitted under the same ASR produced precision and accuracy results within acceptance criteria.

System Blank/Processing Blanks: Over 20 system blanks were process during the analysis of the samples. With the exception of only four nitrate values, no anions were detected above reportable concentrations in the system blanks. The four nitrate blanks measured about two times the instrument detection limit and were due primarily to the high nitrate samples analyzed.

Quality Control Calibration Verification Check Standards: Over 20 mid-range verification standards were analyzed throughout the analysis runs. Except for a few values, the reported results for all analytes of interest were recovered within the acceptance criteria of ±10% for the verification standard. For most of the values exceeding the acceptance criteria, no recoveries exceeded ±15% of the standard values; however, some nitrate values exceeded 125% recovery. These nitrate values were obtained for samples requiring only sulfate results, and are considered for information only.

Table 1: Samples for Sulfate Only

Lab ID	SAMPLE ID.	SO_4 ug/ml
99-2655	AAN-C-A	3,900
99-2656	AAN55-A	3,900
99-2657	AAN55-D-A	3,900
99-2658	AAN55-4A	3,900
99-2659	AAN55-08-A	4,000
99-2660	AAN55-16A	3,800
99-2661	AAN55-24-A	3,900
99-2662	AAN55-72-A	4,000
99-2663	SO4 IX Feed	3,800
99-2664	AN-SO-L1A	2,100
99-2665	AN-SO-L2A	2,900
99-2666	AN-SO-L3A	3,200
99-2667	AN-SO-L4A	3,500
99-2668	AN-SO-L5A	3,600
99-2669	AN-SO-L6A	3,700

Lab ID	SAMPLE ID.	SO_4 ug/ml
99-2670	AN-SO-L7A	3,600
99-2671	AN-SO-L8A	3,600
99-2672	AN-SO-L9A	3,600
99-2673	AN-SO-L10A	3,800
99-2674	AN-SO-L11A	3,600
99-2675	AN-SO-L12A	3,500
99-2676	AN-SO-L13A	3,700
99-2677	AN-SO-L14A	3,600
99-2678	AN-SO-L15A	3,600
99-2679	AN-SO-L16A	3,700
99-2680	AN-SO-L17A	3,600
99-2681	AN-SO-L18A	3,600
99-2682	AN-SO-L19A	3,700
99-2683	AN-SO-L20A	3,600
99-2684	AN-SO-L21A	3,500

Table 2: Samples for Fluoride, Chloride, Nitrite, Nitrate, Phosphate, and Sulfate

Lab ID	SAMPLE ID.	F ug/ml	Cl ug/ml	NO_2 ug/ml	NO_3 (2) ug/ml	PO_4 ug/ml	SO_4 ug/ml
99-2685	AN-SO-L22A	1,900 (1)	560	16,000	OvrRng	< 200	1,700
99-2686	SO4 Eff Composite	3,400 (1)	940	27,000	OvrRng	1,300	3,500
99-2687	AN-SO-FD1	140 (1)	< 50	1,200	23,000	< 100	150
99-2688	AN-SO-FD2	< 25	< 25	< 50	OvrRng	< 50	< 50
99-2689	AN-SO-CW1	< 25	< 25	< 50	OvrRng	< 50	< 50
	AN-SO-CW1 MS Recovery	108%	97%	117%	n/a	101%	105%
99-2690	AN-SO-CW2	< 25	< 25	< 50.0	OvrRng	< 50	< 50
99-2692	AN-SO-E1-A	400 (1)	90	2,700	OvrRng	< 100	430
99-2693	AN-SO-E2-A	120 (1)	< 50	< 100	OvrRng	< 100	< 100
99-2694	AN-SO-E3-A	100 (1)	< 50	< 100	OvrRng	< 100	< 100
99-2695	AN-SO-E4-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2696	AN-SO-E5-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2697	AN-SO-E6-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2698	AN-SO-E7-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2699	AN-SO-E8-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2700	AN-SO-E9-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2701	AN-SO-E10-A	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2702	SO4 Eff Composite A	< 50	< 50	< 100	32,000	< 100	< 100
99-2703	AN-SO-ER1	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2704	AN-SO-ER2	< 50	< 50	< 100	OvrRng	< 100	< 100
99-2705	AN-SO-REGN	< 50	< 50	< 100	16,000	< 100	< 100
99-2706	Leak	120 (1)	< 50	< 100	OvrRng	< 100	150
99-2707	Leak 2	1,600 (1)	490	13,000	OvrRng	< 200	1,600
99-2708	Precip Volume	1,200 (1)	320	8,800	OvrRng	< 200	1,200

(1) Quantified by IC system as fluoride; however, slight retention time peak shift and peak shape suggest significant organic anion interference. High probability that little or no fluoride is actually present in the samples.

(2) For samples not requiring nitrate analysis, samples were not diluted sufficiently to obtain a nitrate results; however, all nitrate values exceeded the calibration upper limit, indicated high nitrate concentrations.

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

Project: 29953
Client: G. Lumetta, and D. Kurath

ACL Number(s): 99-2650 through 99-2705

Client ID: "AAN107-Start" through "AN-SO-REGN"

ASR Number: 5520

Total Samples: 25

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

Analyst: D.R. Sanders

Analysis Date (Filename): 11-05-99 (A0551) & 11-24-99 (A0559)

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 Wagner 01-10-00
Reviewed by

M D Thomas 01-10-00
Concur

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

Twenty-five radioactive liquid or precipitate samples, **AN107-Start** through **AN-SO-REGN** (ACL# 99-2650 through 99-2705), were analyzed by ICPAES after preparation by the Sample Receiving and Processing Lab (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 1 to 12 gram sample aliquots (weighed) were processed and diluted to a final volume of 10ml or 20ml. Concentrations reported for liquids and precipitates have been corrected for process and final volumes. Both are reported in µg/g except the process blank, which is reported in µg/ml.

An equivalent blank estimate may be calculated by multiplying the concentration listed for the blank by the "multiplier" listed at the top of each sample data column divided by the anlytical dilution factor (ADF). The ADF value is the number following the "@" symbol associated with the ACL-sample number. If the "@" symbol is not present, the ADF value is equal to one (1). Volumes and weights have been recorded on bench sheets and included with this report.

Specific analytes of interest requested are listed in table 2-1. "Analytical Requirements for AN-107 Solution, Concentrate, Wash Solution, and Solids Separated from Archive AN-07 Sample" and Table 1-2 "Analytical Requirements for SO4 Column Eluate Solution" included with the ASR-5520. Analytes include: Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mn, Mo, Na, Ni, Pb, Si, Sn, Ti, U, and Zn.

Samples contained mostly high concentrations of sodium. Other analytes measured were generally much lower in concentration.

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 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) except silicon in sample **AN-SO-CW2** (ACL# 99-2690). The RPD for Silicon was about 39% which is above the tolerance limit of 20%. All other analyte concentrations above EQL in the sample were less than about 7% except boron which was about 90% (not an analyte of interest).

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 was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except as follows.

Potassium was slightly high (<14%) in two of seven QC_MCVA check standard measurements. Single element reagent standards of potassium at 100 ppm measured at the beginning and end of the run were well within the tolerance limit.

One of seven sodium measurements of QC_MCVA was somewhat high (+17%) and also one of two measurements in QC_SSTMCV (+13%). Sodium measurement of SRM-1643d, a NIST reference solution, was within tolerance limits at a concentration similar to that in QC_MCVA.

One of three tin measurements of QC_MCVB was low by about 14%. Measurement of a single element reagent standard of tin at 2 ppm was well within tolerance limit.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes of interest was within tolerance of ± 5% accuracy except sodium in one of two measurements (+7%).
A single element sodium standard of 1000 ppm measured at the beginning and at the very end of the run was within tolerance limits.

Process Blank:

All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration. Calcium, nickel and zinc were detected in the process blank but were below EQL.

Laboratory Control Standard (LCS):

None prepared.

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

Comments:

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

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

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

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

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

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

Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	1.0 / 99-2650-PB / Process Blank / 11/5/99 ug/mL	8.2 / 99-2650 @5 / AN107 START / 11/5/99 ug/g	36.4 / 99-2651 @10 / AN107-1 / 11/5/99 ug/g	37.8 / 99-2652 @10 / AN107-2 / 11/5/99 ug/g	2.0 / 99-2653 / AN107-PPT1 / 11/5/99 ug/g
0.025	Ag	–	–	–	–	–
0.060	Al	–	109	288	218	2.22
0.250	As	–	–	–	–	–
0.050	B	–	17.3	51.4	35.2	5.51
0.010	Ba	–	–	–	–	–
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	[0.27]	133	355	235	[4.3]
0.015	Cd	–	21.7	56.7	48.1	0.457
0.200	Ce	–	–	–	–	–
0.050	Co	–	[1.7]	[4.6]	[3.8]	–
0.020	Cr	–	10.8	29.0	24.2	[0.22]
0.025	Cu	–	14.7	37.3	30.0	[0.30]
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	–	2.57	[6.5]	[4.5]	–
2.000	K	–	593	1,560	1,350	[11]
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	[1.7]	[2.0]	–	–
0.050	Mo	–	12.4	33.3	27.6	[0.25]
0.150	Na	–	93,600	179,000	87,800	2,990
0.100	Nd	–	[1.1]	–	–	–
0.030	Ni	[0.044]	182	491	414	4.09
0.100	P	–	166	423	235	3.14
0.100	Pb	–	62.9	168	143	[1.4]
0.750	Pd	–	–	–	–	–
0.300	Rh	–	[3.9]	–	–	–
1.100	Ru	–	[12]	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	82.9	238	[120]	18.4
1.500	Sn	–	–	–	–	–
0.015	Sr	–	74.8	191	107	1.64
1.500	Te	–	–	–	–	–
1.000	Th	–	–	–	–	–
0.025	Ti	–	–	–	–	–
0.500	Tl	–	–	–	–	–
2.000	U	–	[34]	[86]	–	–
0.050	V	–	–	–	–	–
2.000	W	–	[60]	[160]	[140]	–
0.050	Y	–	–	–	–	–
0.050	Zn	[0.068]	4.73	[16]	[11]	[0.54]
0.050	Zr	–	[1.0]	[3.8]	[2.8]	–

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=		2.0	23.3	16.6	2.0	2.0
ALO#=		99-2654	99-2685 @5	99-2686 @10	99-2687	99-2688
Client ID=		AN107-PPT2	AN-20-L22A	SO4-Effluent-Comp.	AN-SO-FD1	AN-SO-FD2
et. Limit	Run Date=	11/5/99	11/5/99	11/5/99	11/5/99	11/5/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	2.68	64.2	104	5.73	2.95
0.250	As	--	--	--	--	--
0.050	B	5.09	49.2	20.8	11.8	13.6
0.010	Ba	[0.034]	120	[0.98]	25.1	9.76
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	6.07	76.2	126	6.81	[1.4]
0.015	Cd	0.598	12.0	20.5	0.882	[0.050]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	[1.7]	--	--
0.020	Cr	[0.30]	5.90	10.1	[0.37]	--
0.025	Cu	[0.35]	9.75	12.6	1.26	[0.080]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.057]	[1.0]	[2.0]	[0.11]	--
2.000	K	[15]	[440]	503	159	152
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	[0.34]	[6.7]	11.7	[0.41]	--
0.150	Na	3,580	52,700	79,300	10,300	7,790
0.100	Nd	--	--	--	--	--
	Ni	5.21	109	175	8.64	[0.59]
	P	5.47	147	147	39.2	5.03
0.100	Pb	[1.8]	36.8	52.7	2.64	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	18.7	280	109	80.3	102
1.500	Sn	--	--	--	--	--
0.015	Sr	3.45	45.8	49.5	5.70	1.05
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	[39]	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	[59]	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[0.24]	[4.3]	[4.8]	[0.37]	1.50
0.050	Zr	--	--	[0.92]	--	--

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=	2.0	2.0	2.0	2.0	2.0
	ALO#=	99-2689	99-2690	99-2690-DUP	99-2703	99-2704
	Client ID=	AN-SO-CW1	AN-SO-CW2	AN-SO-CW2	AN-SO-ER1	AN-SO-ER2
Det. Limit	Run Date=	11/5/99	11/5/99	11/5/99	11/5/99	11/5/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	1.80	--	--	2.09	[0.48]
0.250	As	--	--	--	--	--
0.050	B	9.37	4.84	1.80	6.57	6.30
0.010	Ba	8.37	8.89	8.90	152	196
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[1.1]	[1.9]	[1.4]	9.62	[3.6]
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	[0.041]	--
0.025	Cu	[0.22]	0.582	0.578	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	0.829	[0.46]
2.000	K	107	78.3	76.7	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	[0.43]	--
0.050	Mn	--	[0.53]	--	[0.11]	--
0.050	Mo	--	--	--	--	--
0.150	Na	6,200	5,220	5,500	4,070	5,340
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.26]	[0.20]	[0.20]	[0.26]	[0.23]
0.100	P	[1.1]	[0.90]	[0.93]	[0.38]	[0.27]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	64.1	15.6	10.3	[3.0]	[1.5]
1.500	Sn	--	--	--	--	--
0.015	Sr	0.726	0.844	0.840	[0.053]	--
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	[0.12]	[0.37]	[0.23]	[0.26]	[0.16]
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 PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	2.0 / 99-2705 / AN-SO-REGN / 11/5/99 / ug/g				
0.025	Ag	--	--	--	--	--
0.060	Al	[1.0]	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	7.17	--	--	--	--
0.010	Ba	217	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[1.8]	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	--	--
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	7,180	--	--	--	--
0.100	Nd	--	--	--	--	--
	Ni	[0.064]	--	--	--	--
	P	[0.20]	--	--	--	--
0.100	Pb	[1.1]	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	34.5	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[0.53]	--	--	--	--
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 PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	24.2	24.7	24.7	24.8	24.7
	ALO#=	99-2692 @5	99-2693 @5	99-2694 @5	99-2695 @5	99-2696 @5
	Client ID=	AN-SO-E1-A	AN-SO-E2-A	AN-SO-E3-A	AN-SO-E4-A	AN-SO-E5-A
Det. Limit	Run Date=	11/24/99	11/24/99	11/24/99	11/24/99	11/24/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	83.7	32.8	29.2	[8.6]	[7.7]
0.250	As	[11]	--	--	--	--
0.050	B	108	84.2	89.4	82.1	82.7
0.010	Ba	478	1,280	523	459	435
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	110	144	85.1	98.3	[51]
0.015	Cd	14.2	5.07	[3.2]	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	7.21	[3.3]	[2.1]	--	--
0.025	Cu	9.76	7.34	[2.1]	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[2.2]	8.77	8.62	[2.0]	[1.1]
2.000	K	[77]	[260]	[190]	[150]	[110]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[3.5]	54.4	41.1	[4.1]	--
0.050	Mo	[8.1]	[1.6]	--	--	--
0.150	Na	77,900	32,600	14,600	2,310	245
0.100	Nd	--	--	--	--	--
0.030	Ni	124	42.2	27.4	[2.3]	--
0.100	P	99.6	109	36.9	[4.6]	--
0.100	Pb	38.3	[7.0]	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	563	[62]	[96]	[80]	[66]
1.500	Sn	--	--	--	--	--
0.015	Sr	35.4	280	93.6	24.4	4.60
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	[3.5]	[2.7]	[1.9]	16.8	[4.7]
0.050	Zr	--	[1.7]	--	--	--

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

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

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

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

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	24.6 99-2697 @5 AN-SO-E6-A 11/24/99 ug/g	24.5 99-2698 @5 AN-SO-E7-A 11/24/99 ug/g	24.6 99-2699 @5 AN-SO-E8-A 11/24/99 ug/g	24.6 99-2700 @5 AN-SO-E9-A 11/24/99 ug/g	24.8 99-2701 @5 AN-SO-E10-A 11/24/99 ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	[7.4]	[14]	[8.7]	[8.7]	[8.5]
0.250	As	--	--	--	--	--
0.050	B	88.6	90.2	94.4	91.5	92.8
0.010	Ba	411	472	435	408	408
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	87.5	128	82.1	[59]	83.1
0.015	Cd	--	[0.87]	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	[0.53]	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.96]	[2.3]	[2.2]	[1.3]	[1.9]
2.000	K	[71]	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	215	2,960	272	305	861
[illegible]	Nd	--	--	--	--	--
	Ni	--	9.29	--	[0.95]	--
0.100	P	--	[6.3]	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[75]	[87]	[81]	[85]	[89]
1.500	Sn	--	--	--	--	--
0.015	Sr	[1.6]	[3.5]	[0.41]	--	--
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	[7.6]	[2.9]	[5.4]	[4.3]	16.8
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 PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	24.7					
	ALO#=	99-2702 @5					
	Client ID=	SO4-ELUANT-COMP-A					
et. Limit	Run Date=	11/24/99					
ug/mL)	(Analyte)	ug/g					
0.025	Ag	--	--	--	--	--	
0.060	Al	[7.1]	--	--	--	--	
0.250	As	--	--	--	--	--	
0.050	B	31.0	--	--	--	--	
0.010	Ba	464	--	--	--	--	
0.010	Be	--	--	--	--	--	
0.100	Bi	--	--	--	--	--	
0.250	Ca	[59]	--	--	--	--	
0.015	Cd	[0.54]	--	--	--	--	
0.200	Ce	--	--	--	--	--	
0.050	Co	--	--	--	--	--	
0.020	Cr	--	--	--	--	--	
0.025	Cu	--	--	--	--	--	
0.050	Dy	--	--	--	--	--	
0.100	Eu	--	--	--	--	--	
0.025	Fe	[2.3]	--	--	--	--	
2.000	K	[73]	--	--	--	--	
0.050	La	--	--	--	--	--	
0.030	Li	--	--	--	--	--	
0.100	Mg	--	--	--	--	--	
0.050	Mn	[5.7]	--	--	--	--	
0.050	Mo	--	--	--	--	--	
0.150	Na	2,540	--	--	--	--	
0.100	Nd	--	--	--	--	--	
0.030	Ni	[5.1]	--	--	--	--	
0.100	P	[6.5]	--	--	--	--	
0.100	Pb	--	--	--	--	--	
0.750	Pd	--	--	--	--	--	
0.300	Rh	--	--	--	--	--	
1.100	Ru	--	--	--	--	--	
0.500	Sb	--	--	--	--	--	
0.250	Se	--	--	--	--	--	
0.500	Si	[33]	--	--	--	--	
1.500	Sn	--	--	--	--	--	
0.015	Sr	15.3	--	--	--	--	
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	[3.5]	--	--	--	--	
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).

ACL number	client ID	description	vial	aliquot (ml)	sample + vial	sample wt. (g)	mL of each acid	final volume
99-2650	AN107 START	AN107 SUPERNATE -20ML	8.1198	10	20.2734	12.1536	1.1	20
99-2651	AN107-1	AN107 SUPERNATE - 10ML	8.1071	2	10.8547	2.7476	0.5	10
99-2652	AN107-2	AN107 SUPERNATE - 2ML	8.0633	2 / 1.5	10.7068	2.6435	0.6	10
99-2653	AN107-PPT1	Precipitate - redissolved - 20 ml	8.0800	10	18.0253	9.9453	1.1	20
99-2654	AN107 -PPT2	Precipitate - redissolved 0- 20 ml	8.1355	10	18.1172	9.9817	1.0	20
99-2685	AN-SO-L22A	AN-107 supernate	8.1188	2	10.2693	2.1505	0.5	10
99-2686	SO4-Effulent-	AN-107 supernate	8.0159	10	20.0696	12.0537	1.1	20
99-2687	AN-SO-FD1	0.1 M NaOH/0.25 M NaNO3	8.0734	10	18.2572	10.1838	1.0	20
99-2688	AN-SO-FD2	0.1 M NaOH/0.25 M NaNO3	8.1228	10	18.0604	9.9376	1.0	20
99-2689	AN-SO-CW1	Water/dilute caustic/NaNO3	8.0897	10	18.1402	10.0505	1.0	20
99-2690	AN-SO-CW2	Water/dilute caustic/NaNO3	8.0697	10	17.9753	9.9056	1.0	20
99-2690 DUP	AN-SO-CW2	Water/dilute caustic/NaNO3	8.3154	10	18.4109	10.0955	1.0	20
99-2703	AN-SO-ER1	0.25 M NaNO3	8.0862	10	17.9977	9.9115	1.0	20
99-2704	AN-SO-ER2	0.25 M NaNO3	8.0567	10	18.0884	10.0317	1.0	20
99-2705	AN-SO-REGN	0.1 M NaOH/0.25 M NaNO3	8.0815	10	18.1536	10.0721	1.0	20

A0551 11-05-99

Corrected

Battelle
Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	5520
CLIENT	Lumetta

Corrections

1. 99-2652 Acid Digest used TEA fraction
2ml – 0.5 ml used for Sr^{90} Dilution ∴
wt is correct but Vol = ≈ 1.5ml

2. ICP dilutions were the remainder of the Sr^{90} dilutions. The 1ml aliquots were not used for any determinations

analyst MJ Steele date 12/29/99

reviewer _____ date _____

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

99-2650
10/22/99

Client : Kurath/Lumetta

Cognizant Scientist: L R Greenwood Date : 10/22/99

Concur : T Trang-Le Date : 10/22/99

Procedure: PNL-ALO-420, 421

Measured Activities (µCi/ml) and 1-sigma error

ALO ID Client ID	Alpha Error %	Sr-90 Error %
99-2650 AN107 Start	5.12E-3 3%	6.87E-1 3%
99-2650 REP AN107 Start	5.27E-3 3%	7.46E-1 3%
RPD	3%	8%
99-2651 AN107-1	8.70E-3 3%	1.88E+0 4%
99-2652 AN107-2	1.66E-2 2%	2.16E+0 3%
99-2653 AN107-PPT1	1.11E-4 24%	1.19E-2 3%
99-2654 AN107-PPT2	1.81E-4 19%	2.68E-2 3%
99-2685 AN-SO-L22A		3.49E-1 3%
99-2686 SO4-Effulent Composite		6.02E-1 3%
99-2687 AN-SO-FD1		4.85E-2 3%
99-2688 AN-SO-FD2		7.38E-3 3%
99-2689 AN-SO-CW1		5.60E-3 3%
99-2690 AN-SO-CW2		6.51E-3 3%
99-2692 AN-SO-E1-A		5.04E-2 3%
99-2693 AN-SO-E2-A		3.92E-1 3%
99-2694 AN-SO-E3-A		1.29E-1 3%

Measured Activities (µCi/ml) and 1-sigma error

ALO ID Client ID	Alpha Error %	Sr-90 Error %
99-2695 AN-SO-E4-A		3.33E-2 3%
99-2695 Rep AN-SO-E4-A		3.37E-2 3%
RPD		1%
99-2696 AN-SO-E5-A		6.15E-3 3%
99-2697 AN-SO-E6-A		2.21E-3 3%
99-2698 AN-SO-E7-A		4.76E-3 3%
99-2699 AN-SO-E8-A		4.76E-4 5%
99-2700 AN-SO-E9-A		1.34E-4 14%
99-2701 AN-SO-E10-A		7.72E-5 23%
99-2702 SO4-Eluant-Composite A		2.19E-2 3%
99-2703 AN-SO-ER1		1.14E-4 16%
99-2704 AN-SO-ER2		7.76E-5 23%
99-2705 AN-SO-REGN		<4.E-5
Spike	100%	106% 101%
Matrix Spike*	43%	89% 111%
Blank	<3.E-5	<5.E-6 <8.E-6

*Note: The total alpha matrix spike recovery was low due to high mass absorption, indicating a potential low bial for the total alpha results.

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

99-2650
10/12/1999

29953
Task 12
T2.12.3.3

Client : Kurath/Lumetta

Cognizant Scientist: D.K. Kurath

Concur : T Trang-le

Date : 10/12/99

Date : 10/12/99

Procedure: PNL-ALO-450

Measured Activities (μCi/ml) and 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-2650 AN107 Start	5.72E-2 1%	5.62E-4 19%	2.87E-4 7%	<2.E-4	2.67E-2 2%	1.10E-2 2%	7.95E-3 3%	4.52E-3 5%
99-2651 AN107-1	1.63E-1 1%	9.54E-4 30%	6.56E-4 9%	<4.E-4	7.74E-2 2%	2.42E-2 2%	1.75E-2 3%	1.11E-2 6%
99-2652 AN107-2	3.07E-1 1%	1.32E-3 27%	1.51E-3 6%	<6.E-4	1.49E-1 2%	3.62E-2 2%	2.66E-2 3%	1.92E-2 5%
99-2653 AN107-PPT1	1.02E-3 2%	<3.E-5	<9.E-6	<2.E-5	4.56E-4 3%	2.70E-4 3%	2.10E-4 7%	9.71E-5 22%
99-2654 AN107-PPT2	1.31E-3 2%	<2.E-5	7.32E-6 17%	<8.E-6	6.07E-4 2%	2.83E-4 2%	2.06E-4 5%	1.18E-4 15%
99-2685 AN-SO-L22A	2.90E-2 2%	<3.E-4	1.35E-4 16%	9.69E-5 49%	1.64E-2 2%	4.50E-3 3%	3.19E-3 5%	1.87E-3 15%
99-2686 SO4-Effulent Composite	5.53E-2 1%	3.65E-4 22%	2.41E-4 7%	<2.E-4	2.39E-2 2%	1.06E-2 2%	7.58E-3 3%	4.45E-3 5%
99-2687 AN-SO-FD1	2.64E-3 2%	<5.E-5	<2.E-5	<2.E-5	5.93E-3 2%	2.61E-4 4%	2.03E-4 8%	1.28E-4 22%
99-2688 AN-SO-FD2	2.24E-4 2%	<3.E-5	<9.E-6	<4.E-6	4.71E-3 2%	1.07E-5 18%	<2.E-5	<3.E-5
99-2689 AN-SO-CW1	1.04E-4 2%	<3.E-5	<8.E-6	<3.E-6	3.42E-3 2%	8.50E-6 21%	<2.E-5	<2.E-5
99-2690 AN-SO-CW2	6.73E-5 2%	<2.E-5	<3.E-6	<2.E-6	2.30E-3 2%	1.38E-5 11%	<1.E-5	1.10E-5 42%
99-2692 AN-SO-E1-A	6.48E-3 2%	3.82E-5 36%	2.49E-5 12%	<2.E-5	1.38E-3 2%	5.91E-4 3%	4.14E-4 5%	2.27E-4 16%
99-2693 AN-SO-E2-A	2.02E-3 2%	<5.E-5	<2.E-5	<2.E-5	1.67E-3 2%	4.29E-3 1%	3.09E-3 3%	1.48E-3 6%
99-2694 AN-SO-E3-A	1.36E-3 2%	<3.E-5	8.81E-6 27%	<2.E-5	1.30E-3 2%	1.08E-3 2%	8.06E-4 3%	4.05E-4 7%
99-2695 AN-SO-E4-A	3.25E-4 2%	<3.E-5	<6.E-6	<7.E-6	1.04E-3 2%	9.65E-5 5%	6.81E-5 13%	6.20E-5 26%
99-2696 AN-SO-E5-A	9.28E-5 3%	<2.E-5	<7.E-6	<6.E-6	7.94E-4 2%	1.51E-5 17%	1.21E-5 33%	1.68E-5 35%
99-2697 AN-SO-E6-A	5.21E-5 5%	<2.E-5	<5.E-6	<6.E-6	6.04E-4 2%	1.28E-5 27%	<2.E-5	2.06E-5 36%

Measured Activities (μCi/ml) and 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-2698 AN-SO-E7-A	3.75E-4 2%	<2.E-5	<7.E-6	<7.E-6	5.44E-4 2%	8.18E-5 5%	5.11E-5 10%	4.03E-5 19%
99-2699 AN-SO-E8-A	4.66E-5 7%	<2.E-5	<7.E-6	<7.E-6	2.87E-4 3%	<2.E-5	<2.E-5	<2.E-5
99-2700 AN-SO-E9-A	2.40E-5 8%	<2.E-5	<5.E-6	<5.E-6	1.66E-4 3%	<2.E-5	<2.E-5	<2.E-5
99-2701 AN-SO-E10-A	1.98E-5 14%	<3.E-5	<7.E-6	<7.E-6	9.56E-5 6%	<2.E-5	<2.E-5	<2.E-5
99-2702 SO4-Eluant-Composite A	2.75E-4 2%	<8.E-6	<4.E-6	<3.E-6	5.82E-4 2%	1.55E-4 2%	1.16E-4 4%	5.41E-5 9%
99-2703 AN-SO-ER1	3.21E-5 4%	<6.E-6	<3.E-6	<3.E-6	8.09E-5 4%	6.73E-6 17%	<7.E-6	<9.E-6
99-2704 AN-SO-ER2	1.19E-5 5%	<4.E-6	<2.E-6	<2.E-6	5.71E-5 3%	<3.E-6	<4.E-6	<5.E-6
99-2705 AN-SO-REGN	2.70E-5 3%	<3.E-6	<1E-6	<2.E-6	2.53E-5 4%	<3.E-6	<3.E-6	<3.E-6

APPENDIX C

Appendix C: Sample Spreadsheets

Table C.1. Batch Contacts: Archive AN-107 Contacted with Superlig®-655 (SL-655)

Performed 09/16/99 through 09/20/99, as per " Batch Contact Test Instructions for SL-655 and Archive AN-107 Sample", BNFL-TI-29953-056
Distribution coefficient calculation: $Kd = [(C_0-C1) / C1] \times [V / (M \times F)]$

Archive AN-107 density = 1.22 g/mL
SL-655 F factor = 0.454

Sample ID	resin mass (g)	solution mass (g)	solution volume (mL)	sulfate conc., M	Kd (mL/g)
AAN-C	0	6.1655	5.054	3949.1	
AAN55-04	0.3305	6.1933	5.076	3886.7	0.543
AAN55-08	0.33	6.2	5.082	3953.7	-0.039
AAN55-16	0.3299	6.1733	5.060	3843.6	0.927
AAN55-24	0.3299	6.1725	5.059	3905	0.381
AAN55-72	0.3305	6.238	5.113	3979.1	-0.257
AAN-55	0.3304	6.1883	5.072	3891	0.505
AAN55D	0.3287	6.1962	5.079	3873.9	0.661

Table C.2. Column Flow Testing of SL-655 for Sulfate Removal from Archive AN-107

feed density =	1.22	g/mL									
Archive AN-107 sulfate concentrations											
AN-107 start	3632.7		Bed volume =	29.1	mL	Bed height changed from 6 cm at start to 5.5 cm at end. Average bed volume used.					
AN-107 start	4244.2										
AN-107 start	4024.4										
AAN-C-A	3949.1										
99-2663 SO4 IX Feed	3763.9										
Average	3922.9										
Standard Deviation	236.5										

Performed 9/15/99 - 9/18/99 per "Test Instructions for Sulfate Removal Column Testing Using Envelope C (AN-107 Archive) Solution", BNFL-TI-29953-054, Rev 1.

	time	mass	volume	Sulfate	Bed volumes	cumulative	Sulfate C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2663 SO4 IX Feed							3922.9	feed
99-2664 AN-SO-L1A	0.20	18.1994	14.9	2100	0.51	0.51	53.5%	
99-2665 AN-SO-L2A	0.21	19.3336	15.8	2900	0.54	1.06	73.9%	
99-2666 AN-SO-L3A	0.14	18.0048	14.8	3200	0.51	1.56	81.6%	
99-2667 AN-SO-L4A	0.17	17.287	14.2	3500	0.49	2.05	89.2%	
99-2668 AN-SO-L5A	0.17	17.5912	14.4	3600	0.49	2.54	91.8%	
99-2669 AN-SO-L6A	0.18	17.7314	14.5	3700	0.50	3.04	94.3%	
99-2670 AN-SO-L7A	0.15	17.9241	14.7	3600	0.50	3.55	91.8%	
99-2671 AN-SO-L8A	0.17	17.7684	14.6	3600	0.50	4.05	91.8%	
99-2672 AN-SO-L9A	0.17	18.0581	14.8	3600	0.51	4.55	91.8%	
99-2673 AN-SO-L10A	0.17	18.5403	15.2	3800	0.52	5.08	96.9%	
99-2674 AN-SO-L11A	0.17	18.5183	15.2	3600	0.52	5.60	91.8%	
99-2675 AN-SO-L12A	0.17	17.2941	14.2	3500	0.49	6.08	89.2%	
99-2676 AN-SO-L13A	0.17	18.1606	14.9	3700	0.51	6.59	94.3%	
99-2677 AN-SO-L14A	0.17	17.9832	14.7	3600	0.51	7.10	91.8%	
99-2678 AN-SO-L15A	0.17	18.1539	14.9	3600	0.51	7.61	91.8%	
99-2679 AN-SO-L16A	0.17	18.4088	15.1	3700	0.52	8.13	94.3%	
99-2680 AN-SO-L17A	0.17	18.3436	15.0	3600	0.52	8.65	91.8%	
99-2681 AN-SO-L18A	0.18	19.8415	16.3	3600	0.56	9.20	91.8%	
99-2682 AN-SO-L19A	0.16	17.3692	14.2	3700	0.49	9.69	94.3%	
99-2683 AN-SO-L20A	0.17	18.8238	15.4	3600	0.53	10.22	91.8%	
99-2684 AN-SO-L21A	0.17	18.032	14.8	3500	0.51	10.73	89.2%	
99-2685 AN-SO-L22A	0.17	17.063	14.0	1700	0.48	11.21	43.3%	
avaerage eff composite				3500			89.2%	
Total	3.72		326.6	ave flow rate =	3.01	BV/hr		

Samples L1-L20 were composited. L21 and L22 were partially feed displacement solution.

Table C.2. Column Flow Testing of SL-655 for SO4 Removal from Archive AN-107 (cont')

0.25 M NaNO3/0.25 M NaOH Feed Displacement								
approximate density of 0.25 M NaNO3/0.25M NaOH =					1.024	g/mL	Sulfate	
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2687 AN-SO-FD1	0.33	31.0753	30.3	150	1.04	12.25	3.8%	
99-2688 AN-SO-FD2	0.34	29.8419	29.1	<50	1.00	13.25	<1.3%	
Total	0.7		59.5	ave flow rate =	3.02	BV/hr		
0.25 M NaNO3 wash								
approximate density of 0.25 M NaNO3 =			1.012	g/mL			Sulfate	
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2689 AN-SO-CW1 MS	0.34	31.0753	30.7	<50	1.05	14.30	<1.3%	
99-2690 AN-SO-CW2 MS	0.32	29.8419	29.5	<50	1.01	15.32	<1.3%	
Total	0.66		60.2	ave flow rate =	3.13	BV/hr		
Elution								
approximate density of 0.5 M nitric acid =			1.014	g/mL		Sulfate		
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co	
	hr	g	mL	ug/mL	BV	BV	average	
99-2692 AN-SO-E1-A	1.00	25.1152	24.8	427	0.85	0.85	10.9%	
99-2693 AN-SO-E2-A	0.88	26.3731	26.0	<100	0.89	1.74	<2.5	
99-2694 AN-SO-E3-A	1.00	30.1769	29.8	<100	1.02	2.76	<2.5	
99-2695 AN-SO-E4-A	1.00	35.0744	34.6	<100	1.19	3.95	<2.5	
99-2696 AN-SO-E5-A	1.00	29.9812	29.6	<100	1.01	4.97	<2.5	
99-2697 AN-SO-E6-A	1.00	29.0696	28.7	<100	0.98	5.95	<2.5	
99-2698 AN-SO-E7-A	1.06	34.4395	34.0	<100	1.17	7.12	<2.5	
99-2699 AN-SO-E8-A	0.96	34.0742	33.6	<100	1.15	8.27	<2.5	
99-2700 AN-SO-E9-A	1.02	36.1764	35.7	<100	1.22	9.49	<2.5	
99-2701 AN-SO-E10-A	1.00	33.6707	33.2	<100	1.14	10.63	<2.5	
99-2702 SO4 Eff Composite A				<100			<2.5	
						ave flow rate =	1.07	BV/hr
Total	9.92	hr	309.8	mL				

Table C.2. Column Flow Testing of SL-655 for SO4 Removal from Archive AN-107 (cont')

Elution rinse							
approximate density of 0.25 M NaNO		1.012	g/mL				
	time	mass	volume	Sulfate	Bed volumes	cumulative	C/Co
	hr	g	mL	ug/mL	BV	BV	average
99-2703 AN-SO-ER1	1.01	31.1126	30.7	<100	1.06	1.06	<2.5
99-2704 AN-SO-ER2	0.99	31.4171	31.0	<100	1.07	2.12	<2.5
Total	2.00		61.8	ave flow rate =	1.06	BV/hr	
Regeneration			Note: BDL; below detection limit				
approximate density of 0.25 M NaNO3/0.25M NaOH =				1.024			
	mass	volume	Sulfate	Bed volumes	C/Co		
	g	mL	ug/mL	BV			
99-2705 AN-SO-REGN	61.4488	60.0	<100	2.06	<2.5		
		Note: BDL; below detection limit					

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