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FILTRATION OF A HANFORD AN-104 SAMPLE (U)

AUGUST 7, 2003

Savannah River Technology Center

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500

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FILTRATION OF A HANFORD AN-104 SAMPLE (U)

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LIST OF ACRONYMS

AA	Atomic Absorption
CUF	Cells Unit Filter
CVAA	Cold Vapor Atomic Absorption
HLW	High Level Waste
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectroscopy
ICPMS	Inductively Coupled Plasma Mass Spectroscopy
LAW	Low Activity Waste
LIMS	Laboratory Information Management System
N/A	Not Applicable
PUTTA	Plutonium Triphenyl Trifluoro Acetone
RPP	River Protection Project
SEM	Scanning Electron Microscope
SRTC	Savannah River Technology Center
TDS	Total Dissolved Solids
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TC	Total Carbon
TIS	Total Insoluble Solids
TMP	Transmembrane Pressure
WTP	Waste Treatment Plant

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ABSTRACT

The Savannah River Technology Center (SRTC) conducted ultrafiltration tests with samples from the Hanford Site's AN-104 tank. The test objectives were to measure filter flux during dewatering and the removal of soluble species during washing.

The filtration tests were conducted with the Cells Unit Filter (CUF) currently installed in Cell 16 of the SRTC High Activity Caves. Following filtration, personnel performed inhibited water washing to remove soluble species. Because of the limited volume of concentrated slurry, the washing was performed with a volumetric flask rather than a crossflow filter. Following the washing, personnel chemically cleaned the filter with 1 M nitric acid and periodically measured the clean water flux.

The results of the testing follow.

- The average measured flux of 0.085 gpm/ft^2 during dewatering exceeded the target of 0.03 gpm/ft^2. Note that a low insoluble solids content of only 0.9 wt% contributed to the high average flux.
- A statistically significant correlation was observed between filter flux and transmembrane pressure.
- The measured mean particle size was 0.8 – 1.7 micron.
- The filtrate viscosity measured 3.5 cp, and the slurry viscosity measured 3.9 cp at 0.9 wt % insoluble solids. The washed filtrate viscosity measured 1.3 cp. The washed slurry was concentrated to 2.2 wt% insoluble solids. Its viscosity measured 1.9 cp with a yield stress of 1.47 Pa.

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1.0 SUMMARY OF TESTING

1.1 OBJECTIVES

The SRTC conducted ultrafiltration tests with a sample from the AN-104 tank. The test objectives were to measure filter flux during dewatering and the removal of soluble species during washing. Single tube crossflow filtration tests produced indicative data on equipment performance (permeate flux).

1.2 CONDUCT OF TESTING

Filtration tests were conducted with the CUF currently installed in Cell 16 of SRTC High Activity Caves. The unit has a 2 ft long stainless steel Mott crossflow filter of 3/8" ID and 0.1 micron nominal pore size. The system can provide up to 16.5 ft/s crossflow velocity, along with up to 80 psi transmembrane pressure (TMP). Feed from the reservoir passes through a progressive cavity pump. The pump is operated at variable speed by controlling the air pressure supplied to the pump motor. The slurry is pumped through a magnetic flow meter and heat exchanger to remove heat. Ice water, contained in a 3-gallon Igloo cooler, removes heat from the system. The slurry then passes through the crossflow filter. A throttle valve downstream of the filter is used to adjust the filter TMP. The filtrate flow rate is measured with a calibrated sight glass and stopwatch. The system is equipped with a manual backpulse system. The feed, concentrate, and filtrate pressures are measured with standard Bourdon-type pressure gauges. A thermocouple mounted near the bottom of the feed reservoir measures the slurry temperature.

The feed sample for the tests was an AN-104 actual waste sample previously adjusted to 5 M sodium.[3]

During the testing, personnel controlled the feed slurry temperature to 25°C. The axial velocity and transmembrane pressure were controlled to Hanford Waste Treatment Plant (WTP) personnel-specified values.[1] Filtrate flux data was collected periodically.

Following filtration, personnel performed inhibited water washing to remove soluble species. Because of the limited volume of concentrated slurry, the washing was performed with a 100 mL volumetric flask rather than a crossflow filter.

Following the washing, personnel chemically cleaned the filter with 1 M nitric acid and periodically measured the clean water flux.

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

The objectives and success criteria for this task follow.[1]

- The product stream before and after washing contains 20 wt. % insoluble solids with compatible (will it pump or not) slurry rheology. The feed sample contained insufficient volume to reach 20 wt. % solids in the filtration test equipment. Therefore, the researchers cannot determine if 20 – 25 wt % insoluble solids slurries have compatible slurry rheology.
- No solids pass into the ultrafiltration permeate. No solids were observed in filtrate samples.
- Average flux is >0.03 gpm/ft^2 during dewatering. The average filter flux during dewatering was 0.085 gpm/ft^2, which is greater than the minimum flux.
- The wash factors for five AN-104 anions were determined and are shown in Table 2.8.
- Researchers could not return the filtration equipment to pre-test operating levels following cleaning with inhibited water and 1 M nitric acid.

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO MOSRLE60. SRTC has provided matrices to WTP demonstrating compliance of the SRTC QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRTC QA program with RW-0333P, Revision 10, NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices.

The specific quality requirements for this task are described in the Task Technical and Quality Assurance Plan.[2]

The measuring and test equipment used in the testing is in compliance with the SRS QA Program.

The methods used for performing the work and the data obtained from the work are reported in Laboratory Notebook WSRC-NB-2003-00031, “AN-104 CUF Filtration”.

The data collected and reported was verified by independent checking (Procedure E7, 2.31).

1.5 ISSUES

- Nitric acid cleaning. Personnel could not effectively clean the filter with 1 M nitric acid. Following cleaning with four batches of inhibited water, five batches of 1 M nitric acid, and two batches of 0.01M nitric acid, the filter flux remained significantly below the pre-run fluxes.
- A stable foam was formed in the CUF that overflowed the slurry tank and slurry tank sight glass during dewatering.

2.0 DISCUSSION

2.1 INTRODUCTION

The WTP Research &Technology Plan identified a sample from AN-104 tank as one of the waste solutions to be used to perform the filtration and sludge washing tests using the bench scale crossflow ultrafiltration unit (the CUF).[1] Washing tests were performed to assess the reduction in quantity of High Level Waste (HLW) by removing soluble components.

SRTC personnel characterized the waste sample. That work is described in the AN-104 Characterization Task Plan.[2] Following characterization, SRTC personnel processed that waste sample through the CUF to provide filter flux data, and washed the concentrated solids to provide design verification data to WTP. After the filtration testing, they chemically cleaned the filter with 1 M nitric acid.

2.2 EXPERIMENTS

2.2.1 Test Equipment

Filtration tests were conducted with the CUF currently installed in Cell 16 of SRTC High Activity Caves (see Figure 2-1). The unit has a 2 ft long stainless steel Mott crossflow filter of 3/8" ID and 0.1 micron nominal pore size. The system can provide up to 16.5 ft/s crossflow velocity, along with up to 80 psi TMP. Feed from the reservoir passes through a progressive cavity pump. The pump is operated at variable speed by controlling the air pressure supplied to the pump motor. The slurry is pumped through a magnetic flow meter and heat exchanger that removes heat. Ice water, contained in a 3-gallon Igloo cooler, removes heat from the system. The slurry then passes through the crossflow filter. A throttle valve downstream of the filter is used to adjust the filter feed pressure. The filtrate flow rate is measured with a sight glass and calibrated stopwatch. The system is equipped with a manual backpulse system. The feed, concentrate, and filtrate pressures are measured with standard Bourdon-type pressure gauges. A thermocouple mounted near the bottom of the feed reservoir measures the slurry temperature.

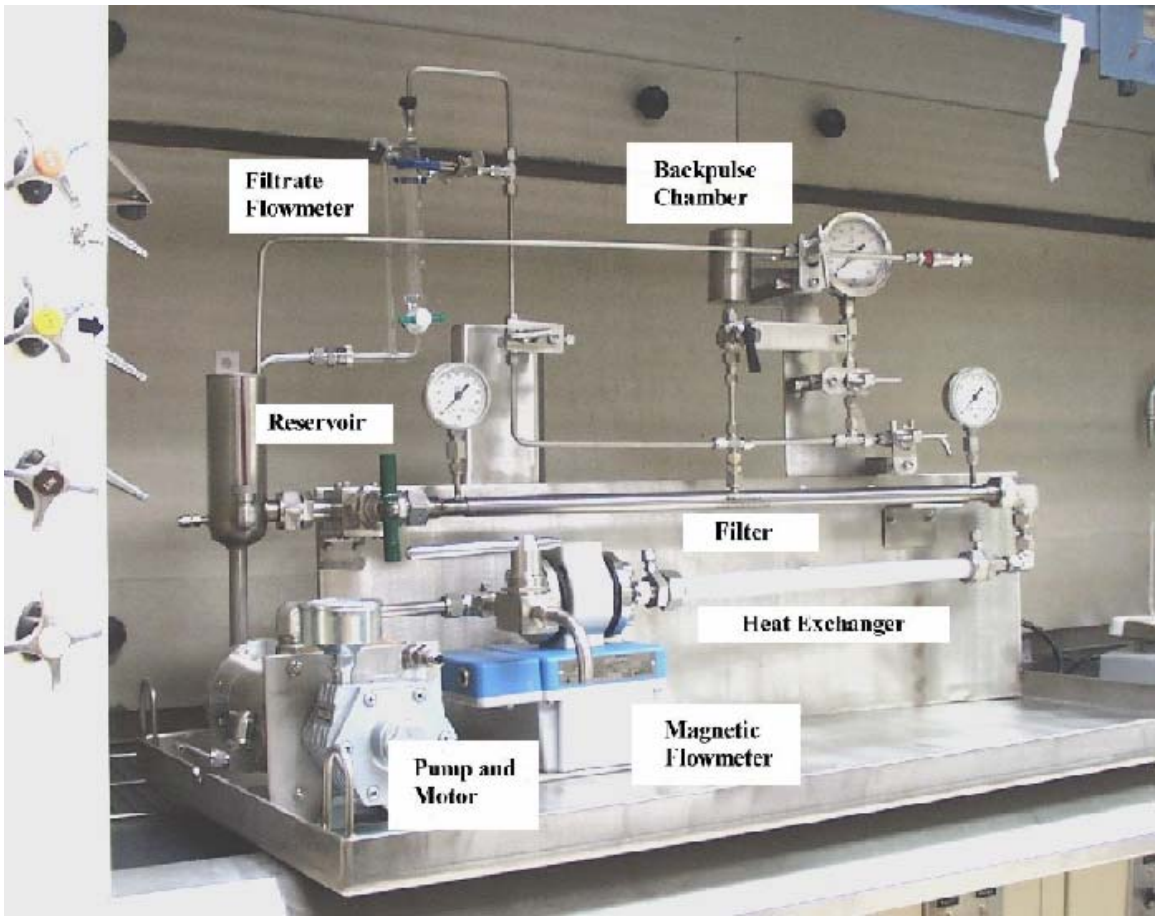

Figure 2-1 Cells Unit Filter (CUF)

2.2.2 Test Preparation

Researchers received a 6200 mL sample of Hanford AN-104 waste from the Characterization Team.[3] The sample contained 5 M sodium prior to the start of the filtration tests.

2.2.3 Filtration Tests

Personnel performed clean water flux tests with 0.01 M NaOH solution that was filtered through a 0.1 micron filter. The tests were performed at 11 ft/s axial velocity and 10 and 20 psi TMP. Following the clean water flux tests, personnel performed tests with 5 wt. % strontium carbonate at 11 ft/s axial velocity and 10, 20, and 30 psi TMP. Following the strontium carbonate test, they performed an additional clean water flux test at 11 ft/s axial velocity and 20 psi TMP.

Personnel added 4.7 liters of AN-104 slurry to the filter feed tank. They concentrated the feed slurry from ~ 0.07 wt. % to ~ 0.9 wt. % by reducing its volume. The dewatering step lasted 12 hours. During the dewatering process, the axial velocity was 11 ft/s, and the transmembrane pressure was 40 psi. Following the dewatering process, personnel conducted filtration matrix tests with the conditions shown in Table 2-1. Due to equipment limitations, three of the test conditions could not be met. Since the TMP has a greater effect on filter flux, the TMP was met at the expense of somewhat lower axial velocities for those three test conditions (See Appendix 2)

Table 2-1 Filtration Test Matrix Conditions

TMP (psi)	Axial Velocity (ft/s)
40	11
40	11
40	11
30	9
30	13
50	13*
50	9
40	11
40	7
40	15*
20	11
60	11*
40	11

* Could not achieve target velocity

About midway through the dewatering step, a stable foam was formed in the CUF and overflowed both the slurry tank and the slurry tank level sight glass. Foaming continued until the end of the dewatering step. No addition foaming occurred during the matrix flow tests. Figure 2-2 is a photograph of the foam at the end of the dewatering test. The equipment configuration with the small slurry tank may contribute to the occurrence of foam. This foam was still present five days later when it was rinsed off the unit with water. Further study would be required to determine the effect of foam on filter flux.

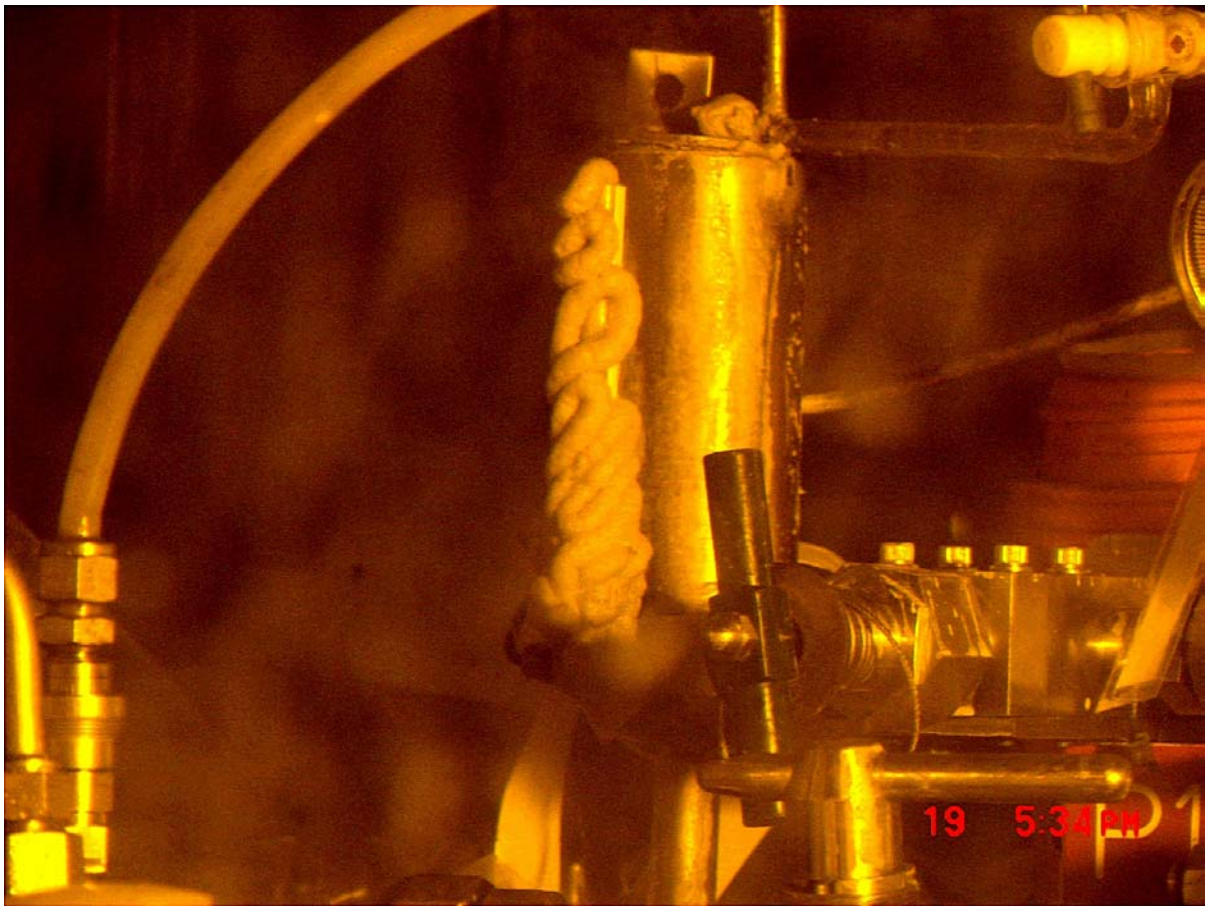

Figure 2-2 Foam from CUF

2.2.4 Washing Test

Because of the low volume of solids in the feed slurry, 94 mL of the 500 mL of feed slurry were decanted into a 100 mL volumetric flask. The washing process was performed in the volumetric flask rather than with the CUF.

To perform the wash, personnel added 9.5 mL of inhibited water to the slurry in the volumetric flask. They capped the flask and inverted and shook it to contact the slurry and wash water. The flask was then set in a pan to allow the solids to separate from the liquid by settling. Following the settling, 9.5 mL liquid was removed with a disposable pipette that had a "stop" attached to it that would only allow it to go so far down into the volumetric flask.

Personnel repeated this process for a total of twelve washes.

2.2.5 Sample Preparation and Analysis

During the testing, SRTC collected the following samples and performed the specified analyses.

- Unwashed Slurry following dewatering
  - TIC/TOC, IC(anions), rheology, insoluble solids, particle size
- Filtrate at the start of dewatering
  - Radionuclides and chemical constituents in Table 2-2, soluble solids, rheology, density
- Filtrate from wash 1, wash 4, and wash 8
  - Sodium, Cs-137
- Filtrate from washes 2,3,5,6,7,9-11
  - Sodium
- Filtrate from wash 12 (final wash)
  - Radionuclides and chemical constituents in Table 2-2, soluble solids
- Final solids slurry
  - Radionuclides and chemical constituents in Table 2-2, insoluble solids, rheology

Table 2-2 shows the analyses performed on slurry, filtrate, and wash solutions, along with the method used to perform the analysis.

Table 2-2 Radionuclides and Chemical Constituents

Analyte	Solids	Filtrate, Wash Solutions	Method
Cesium-137	X	X	Gamma counting
Strontium-90	X	X	Sr separation by Eichrom resin
Technetium-99	X	X	Eichrom disk separation, gamma counting, liquid scintillation counting
Americium-241	X	X	Cs removal, gamma count
Europium-154	X	X	Cs removal, gamma count
Europium-155	X	X	Cs removal, gamma count
Pu-239/240	X	X	PUTTA
Total Alpha	X	X	Cs removal, proportional counting
Total Beta	X	X	Rad screen
Ag	X	N/A	ICPES
Al	X	X	ICPES
Ba	X	X	ICPES
Ca	X	X	ICPES
Cd	X	X	ICPES
Co	X	X	ICPMS
Cr	X	X	ICPES
Cu	X	X	ICPES
Fe	X	X	ICPES
Hg	**	N/A	CVAA
K	X	X	AA
La	X	X	ICPES
Mg	X	X	ICPES
Mn	X	X	ICPES
Mo	X	X	ICPES, ICPMS
Na	X	X	ICPES, AA
Ni	X	X	ICPES
Pb	X	X	ICPES
Si	X	X	ICPES
Sr	X	X	ICPES
Ti	X	X	ICPES
U	X	X	ICPES, ICPMS
Zn	X	X	ICPES
TOC	**	X	TIC/TOC
TIC	**	X	TIC/TOC
Cl	**	X	IC
F	**	X	IC
NO3	**	X	IC
SO4	**	X	IC
PO4	**	X	IC
As	*	N/A	AA
Se	*	N/A	AA

* Only requested for final solids product.

** Performed on unwashed slurry only. These species expected to be removed during washing.

Personnel performed rheological measurements with a RV30/m5 rotoviscometer. They performed the measurements at 25 ºC with an NV double concentric rotor and cylinder. The rotor ramped from 0 – 2700 sec^{-1} shear rate in five minutes, held the 2700 sec^{-1} shear rate for one minute, and ramped from 2700 – 0 sec^{-1} in five minutes. Each sample was measured at least two times.

The filtrate and unwashed slurry data was fit with the following rheological model

$$\tau = \mu \gamma/1000$$

where τ is the shear stress (in Pascals), μ is the Newtonian viscosity (in centipoises), and γ is the shear rate (in sec^{-1}). The slope of shear stress versus shear rate yields the viscosity (in Pascal second). The factor of 1000 converts Pascal seconds to centipoise.

The data from the concentrated and washed slurry was fit with a Bingham plastic model described by the following equation

$$\tau = \tau_y + \eta \gamma/1000$$

where τ_y is the yield stress (in Pascal) and η is the consistency or infinite viscosity (in centipoises).

The total solids (both insoluble solids and soluble salts) were measured by heating at 115 +/- 5 ^oC until a constant dry weight was achieved. The samples were dried at 115 ^oC rather than 105 ^oC listed in the Test Specification because we have observed that this temperature is better for achieving a stable dry weight for samples with high dissolved salt content.

The weight % insoluble solids and weight % soluble solids were calculated after measuring the weight % total solids in the slurry and the weight % soluble solids in a filtered portion of the supernatant. This technique is used for determining the weight % insoluble solids rather than collecting and measuring the insoluble solids directly for two reasons: (1) it is less prone to experimental errors; and (2) it includes the water-soluble salts that would be dissolved during the water rinse of the solids to remove interstitial supernatant. The expression used for calculating the insoluble solids is:

$$IS = TS - (100-TS) \times \frac{(FS/100)}{(1-FS/100)}$$

where

IS = weight % insoluble solids in the slurry
TS = weight % total solids in the slurry
FS = weight % soluble solids in the filtered supernatant

The weight % soluble solids in the as-received slurry (SS) was then calculated from the difference in measured total weight % solids in the slurry (TS) and the calculated weight % insoluble solids (IS):

SS= TS-IS

Density measurements were performed by weighing a known volume of sample.

Slurry samples were collected, dried and then submitted for particle size analysis by scanning electron microscope (SEM). The Analytical Development Section performed the SEM analysis and provided SEM pictures to the authors which are shown in Appendix D. The analysis was performed at 41X, 333X, 2300X, and 4600X. Personnel measured the size of particles on each of the pictures.

Filtrate samples were dissolved prior to analysis to ensure that all components were soluble. The dissolution was performed by mixing 5 mL of sample, 5 mL of nitric acid, and 2 mL of hydrogen peroxide. The samples were capped, mixed, and heated to 115 ºC for two hours. After heating, the samples were cooled and diluted to 100 mL with deionized water. This procedure can be found in laboratory notebook WSRC-NB-2003-00031.

Acid digestion of the final solids slurry was performed as follows. Personnel mixed between 0.5 and 1 grams of the slurry, 3 mL of nitric acid, 9 mL of HCl, and 5 mL of HF in a Teflon pressure vessel. The vessel was capped, mixed, and placed into a 115 ºC oven for three hours. After three hours, the vessel was removed from the oven, allowed to cool, and the contents diluted to 1000 mL with 0.6 M boric acid. This procedure can be found in laboratory notebook WSRC-NB-2003-00031.

Water leaching of the solids was performed so TIC/TOC and anion analyses could be performed. About 115 mL of slurry from the CUF was split up among 3 centrifuge tubes and centrifuged at high speed for over an hour. All liquid was poured off; then the wet solids were weighed. Twenty five mL of deionized water was added to the solids in each centrifuge tube. After vigorously shaking the tubes, they were placed in an oven at 115 ^{o}C for three hours. After cooling, the water/solids solutions were diluted to about 40 mL including rinses of the centrifuge tubes. This procedure can be found in laboratory notebook WSRC-NB-2003-00031.

2.3 RESULTS

The test specification[1] calls for target minimum reportable quantities (MRQs) for analytes as listed in Appendix C. The MRQ is defined in this test specification as the reporting value that is at least 3 times above the Minimum Detection Limit (Activity). SRTC did not meet the MRQs of eleven analytes from the final washed slurry sample and two analytes, Sr and Am-241, from the filtrate analyses, so SRTC personnel contacted the BNI R&T representative to discuss the impact. Since there was such a low solids content in the slurry, per BNI R&T, SRTC did not repeat these analyses.

2.3.1 Filtration Test Results

Table 2-3 shows the clean water flux and strontium carbonate flux data. In previous tests with the radioactive CUF and a 0.1 micron filter, the clean water flux at 20 psi measured 0.5 – 1.0 gpm/ft2.[4,5,6] Since one measured clean water flux at 20 psi was 0.56 gpm/ft2, the filter was considered clean and the testing begun.

Table 2-3 Clean Water and Strontium Carbonate Flux

Feed	Temperature (°C)	Axial velocity (ft/s)	TMP (psi)	Flux (gpm/ft2)@25°C
Water	20.7	11.0	10	0.34
Water	20.7	11.0	20	0.63
SrCO3	21.0	11.1	10	0.25
SrCO3	22.4	11.0	20	0.44
SrCO3	23.0	11.2	30	0.62
Water	25.0	11.1	20	0.31

Figure 2-3 and Table A- 2 show the filter flux during dewatering. The average measured flux of 0.085 gpm/ft^2 exceeds the target of 0.03 gpm/ft^2.

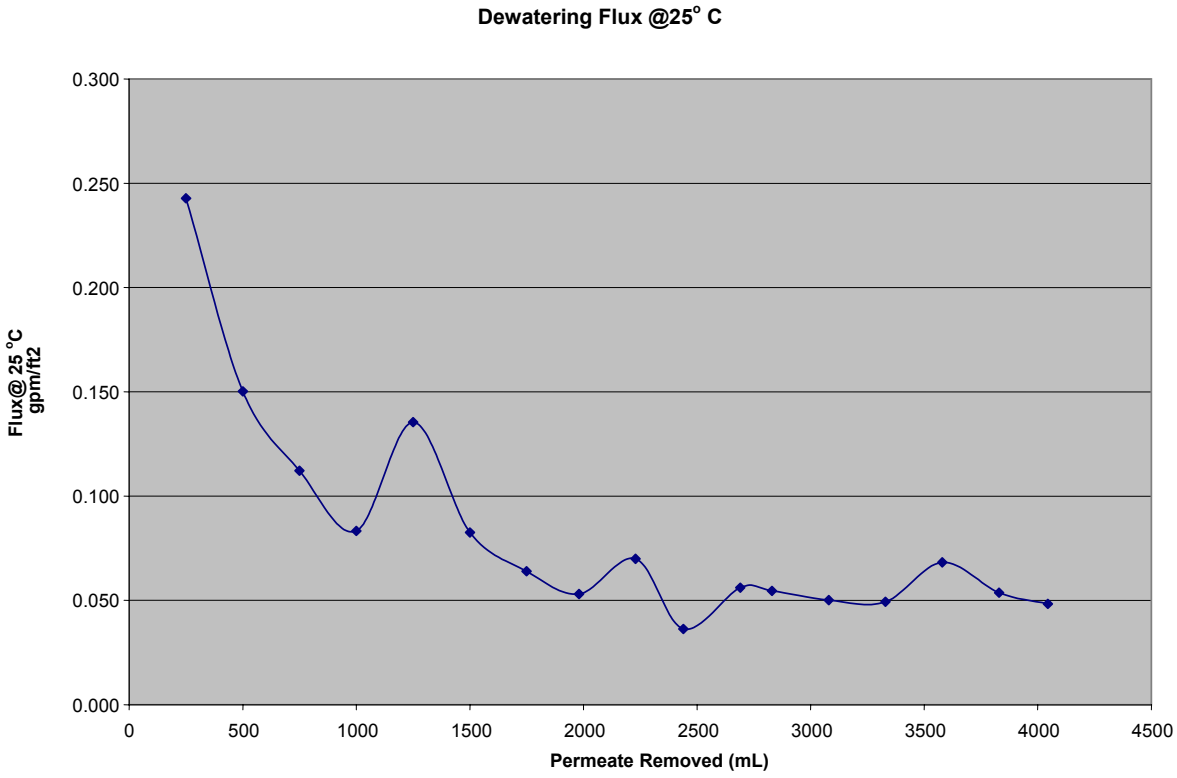

Figure 2-3 Filter Flux During Dewatering

Figure 2-4, Figure 2-5, and Table A- 1 show the filter flux during the matrix tests performed at 0.9 wt. % insoluble solids. In all cases, the filter flux is less than 0.03 gpm/ft^2. There is a correlation between transmembrane pressure and filter flux per the statistical analysis shown in Appendix B. There is no observed correlation between axial velocity and filter flux.

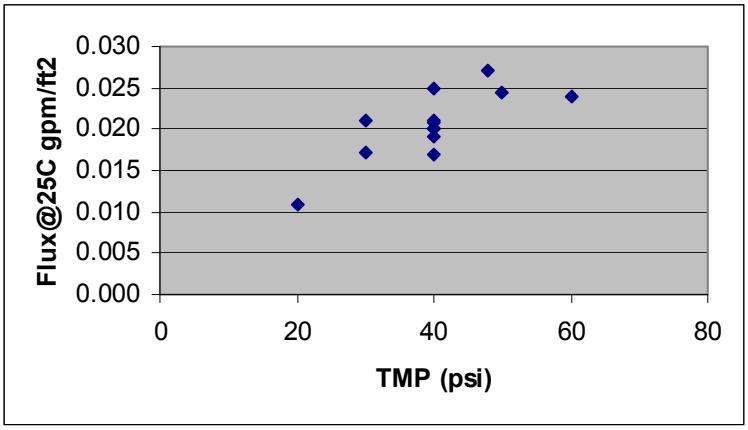

Figure 2-4 Filter Flux of 0.9 wt.% AN-104 Slurry

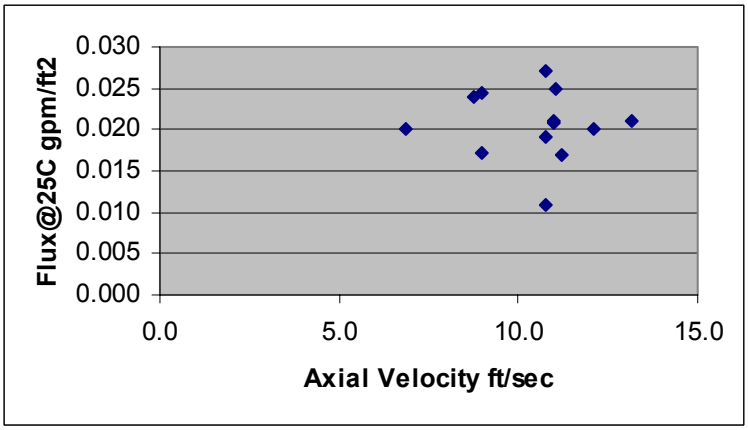

Figure 2-5 Filter Flux of 0.9 wt.% AN 104 Slurry

2.3.2 Washing Results

Table 2-4 shows the sodium and Cs-137 concentration in supernate samples collected during the washing. The sodium concentration in the supernate decreases fairly linearly throughout the washes as expected. The Na results vary widely in Table 2-5 between the AA and the ICPES methods for determining it. The Na value of 12,300,000 µg/mL as determined by AA is incorrect. The Cs-137 decreases steadily as expected.

Table 2-4 Washing Sample Results

Sample	Na (µg/mL)	Cs-137 µCi/g
Wash 1	106,000.	2.85E+02
Wash 2	100,000.	
Wash 3	90,100.	
Wash 4	82,100.	1.78E+02
Wash 5	75,900.	
Wash 6	68,200.	
Wash 7	60,000.	
Wash 8	55,300.	1.13E+02
Wash 9	50,000.	
Wash 10	47,000.	
Wash 11		
Wash 12	41200.	9.32E+01

Table 2-5 shows the composition of the wash solution 12. Wash solution 12 is the only wash that had the full suite of analyses performed on it. The units are per mL of wash solution.

Table 2-5 Washing Sample Results of Wash 12

K	**µg/mL**	875	**% Uncertainty**
Na-AA	**µg/mL**	12,300,000	
Na-ICPES	**conc, µg/mL**	41200	
Cs-137	**µCi/mL**	1.17E+02	2.4
Co-60	**µCi/mL**	1.20E-04	2.49
Eu-154	**µCi/mL**	6.30E-05	6.77
Eu-155	**µCi/mL**	3.45E-05	mda
Am-241	**µCi/mL**	1.62E-04	5.92
Gross Alpha	**µCi/mL**	1.10E-03	15
Hg	**µg/mL**	<0.110	
BROMIDE	**µg/mL**	<100	
CHLORIDE	**µg/mL**	1060	
FLUORIDE	**µg/mL**	<20	
FORMATE	**µg/mL**	183	
NITRATE	**µg/mL**	23200	
NITRITE	**µg/mL**	14300	
OXALATE	**µg/mL**	1040	
PHOSPHATE	**µg/mL**	<100	
SULFATE	**µg/mL**	960	
PU-238	**µCi/mL**	2.25E-04	38.9
PU-239/240	**µCi/mL**	7.34E-05	35.38
Beta	**µCi/mL**	1.39E+02	20
SR-90	**µCi/mL**	2.13E-01	12.4
TC-99	**µCi/mL**	3.17E-02	5.5
TIC	**µg/mL**	1740	
TOC	**µg/mL**	800	
TC	**µg/mL**	2540	

2.3.3 Analytical Results

2.3.3.1 Particle Size Data

Table 2-6 shows the results and Appendix D shows the actual pictures. The results shown are from the same sample, using different magnification and examining different fractions of the sample. The mean particle size varies widely, depending on the magnification of the SEM. In reviewing the pictures, the authors observed that at low magnification, the particles were extremely small. They had difficulty determining the location of the edges of the particles and therefore, determining their size. At magnification 2300X and larger, the mean particle size is approximately the same. The authors recommend using a mean particle size of 0.8 – 1.7 micron for the AN-104 sample.

Table 2-6 Particle Size of AN-104 Solids

Sample ID	# of particles	Avg. D, µ	Std Dev, µ	Max. D, µ	Min. D, µ
3-191232-10X-SE	61	4.8	1.5	9.4	2.4
3-193822-41X-SE	23	38	11	60	20
3-193282-41X-BS	26	39	11	67	19
3-193822-333X-BS	52	13	6	31	4
3-193822-2300X-SE	37	1.5	0.4	2.4	1.0
3-193822-2300X-BS	60	1.7	0.7	5.7	0.7
3-193822-4600X-SE	44	0.77	0.21	1.5	0.36
3-193822-4600-BS	59	1.29	0.30	2.18	0.73

2.3.3.2 Filtrate Data

The filtrate was visually inspected for solids and none were observed. Table 2-7 shows the composition of filtrate samples collected at the start of the dewatering process. The units are per mL of filtrate. Table 2-8 shows the anion data for both the filtrate after concentration and the filtrate at the end of washing.

Table 2-7 Chemical Composition of Filtrate Samples

LIMS		300194279	300194280	300194281
Sample ID		Filtrate 1	Filtrate 2	Filtrate 3
ICPES				
Ag	µg/mL	<1.95	<1.94	<1.65
Al	µg/mL	16128	13734	13734
B	µg/mL	35.2	20.7	24.1
Ba	µg/mL	<4.63	<4.61	<3.91
Ca	µg/mL	<5.60	<5.58	<4.75
Cd	µg/mL	<1.02	<1.01	<0.866
Ce	µg/mL	13.5	<6.5	<5.56
Cr	µg/mL	180	150	150
Cu	µg/mL	2.22	1.63	1.15
Fe	µg/mL	<0.488	<0.486	0.818
Gd	µg/mL	<5.71	<5.68	<4.83
La	µg/mL	2.12	<1.75	1.68
Li	µg/mL	<10.5	<10.4	<8.88
Mg	µg/mL	<1.30	<1.29	<1.09
Mn	µg/mL	<0.195	<0.194	<0.165
Mo	µg/mL	61.7	38.2	40.4
Na	µg/mL	139000	117000	116000
Ni	µg/mL	<3.29	<3.28	<2.78
P	µg/mL	562	498	462
Pb	µg/mL	14.2	13.2	11.0
S	µg/mL	1575	1298	1323
Sb	µg/mL	<92.1	<91.7	<78.0
Si	µg/mL	105	92.5	90.6
Sn	µg/mL	62.2	35.0	39.9
Sr	µg/mL	<1.85	<1.84	<1.57
Ti	µg/mL	<1.88	<1.86	<1.59
U	µg/mL	61.9	<55.0	<46.9
Zn	µg/mL	3.23	2.62	2.82
Zr	µg/mL	<5.56	<5.53	<4.71
AA				
K	µg/mL	2646	2054	2079
Na	µg/mL	197820	92988	101052
PUTTA				
Pu238	µCi/mL	3.60E-04	3.81E-04	5.57E-04
%Uncertainty		40.92	40.09	17.82
Pu239/240	µCi/mL	<1.02E-04	<8.63E-05	3.01E-04
		MDA	MDA	36.06

LIMS		300194279	300194280	300194281
Sample ID		Filtrate 1	Filtrate 2	Filtrate 3
RAD screen				
alpha count	µCi/mL	1.33E-01	1.30E-01	1.31E-01
% Uncertainty		7	7	7
beta count	µCi/mL	3.24E+02	2.72E+02	2.70E+02
% Uncertainty		15	15	15
Sr90 beta liq sint	µCi/mL	9.25E-02	1.06E-01	8.80E-02
% Uncertainty		9.9	9.6	9.7
Tc99 beta liq scnt	µCi/mL	1.02E-01	9.14E-02	8.23E-02
% Uncertainty		6.2	6.3	5.8
Cs-137	µCi/mL	2.67E+02	2.28E+02	2.24E+02
% Uncertainty		150.00%	1.6	1.5
Co	µg/mL	1.26E-01	1.26E-01	1.26E-01
Mo	µg/mL	3.40E+01	2.99E+01	3.02E+01
Am-241	µCi/mL	<1.80E-03	< 1.44E-03	<1.62E-03
Co-60	µCi/mL	3.02E-04	2.95E-04	2.82E-04
% Uncertainty		9	3.6	7.9
Eu-154	µCi/mL	5.11E-03	4.40E-03	4.43E-03
% Uncertainty		1.4	1.6	1.3
Eu-155	µCi/mL	4.81E-04	4.45E-04	3.60E-04
% Uncertainty		21.6	11.1	26.4
U-235	µg/mL	5.04E-02	5.04E-02	3.78E-02
U-238	µg/mL	8.19E+00	6.93E+00	7.06E+00

Table 2-8 Filtrate vs. Washed Filtrate Anion-Organic Data

Anion	Units	Unwashed Filtrate	Washed Filtrate	Wash Factors
F	µg/ml	66	46	0.70
Cl	µg/ml	3170	1100	0.35
NO2	µg/ml	43300	14000	0.32
NO3	µg/ml	75300	21800	0.29
PO4	µg/ml	1490	195	0.13
SO4	µg/ml	3810	1050	0.28
TC	µg/ml	6040	2760	0.46
TIC	µg/ml	3330	1620	0.49
TOC	µg/ml	2710	1140	0.42

2.3.3.3 Unwashed Solids Data

Table 2-9 shows the composition of final 0.9 wt.% insoluble solids slurry samples collected and prepared by water leaching. The units are per gram of wet solids after centrifugation and decantation.

Table 2-9 Chemical Composition of Unwashed Solids Sample by Water Leach

LIMS ID		300195377	300195378	300195379
User Sample ID		Leachate 1	Leachate 2	Leachate 3
ICA				
FLUORIDE	µg/g	6.68E+01	9.04E+01	6.24E+01
FORMATE	µg/g	4.34E+02	4.52E+02	4.68E+02
NITRITE	µg/g	2.48E+04	2.46E+04	2.80E+04
CHLORIDE	µg/g	2.07E+03	1.81E+03	2.03E+03
OXALATE	µg/g	2.91E+04	4.61E+04	2.79E+04
SULFATE	µg/g	1.90E+03	1.72E+03	1.87E+03
PHOSPHATE	µg/g	<334	<452	<311
NITRATE	µg/g	4.24E+04	4.57E+04	4.61E+04
TIC/TOC				
Total Inorganic Carbon	µg/g	2.64E+03	2.83E+03	2.38E+03
Total Organic Carbon	µg/g	1.03E+04	1.46E+04	1.00E+04
Total Carbon	µg/g	1.29E+04	1.74E+04	1.24E+04

2.3.3.4 Concentrated, Washed Slurry Data

Table 2-10 shows the composition of final solids slurry samples collected and prepared by acid digestion. The units are per gram of dried total solids. The high variability observed between samples is probably due to the large dilution of a small amount of solids.

Table 2-10 Chemical Composition of Final Solids Sample by Acid Digestion

LIMS #		300193853	300193854	300193855
Sample Name		WSLURY-1	WSLURY-2	WSLURY-3
ICPES				
Ag	µg/g	<465	<397	<360
Al	µg/g	24107	22441	49983
Ba	µg/g	<1105	<942	<853
Ca	µg/g	<1340	1234	4658
Cd	µg/g	<244	<208	<189
Ce	µg/g	<1571	<1340	<1214
Cr	µg/g	19177	15879	34682
Cu	µg/g	322.7	203.0	346.8
Fe	µg/g	1741	955	3104
La	µg/g	<418	<357	<324
Li	µg/g	<2503	<2132	<1935
Mg	µg/g	<309	<263	<238
Mn	µg/g	<46.6	<39.8	<36.0
Mo	µg/g	<3087	<2628	<2384
Na	µg/g	138728	127508	267936
Ni	µg/g	<785	<670	<609
P	µg/g	<4012	<3434	4318
Pb	µg/g	<1857	<1581	1731
Si	µg/g	16321	14111	22543
Sn	µg/g	82625	59164	57123
Sr	µg/g	1081	840	2265
Ti	µg/g	<449	<381	<347
U	µg/g	<13230	<11250	<10200
V	µg/g	751	513	401
Zn	µg/g	186.7	615.4	751.4
AA				
As	µg/g	<291	<248	<225
K	µg/g	3434	3434	5712
Na	µg/g	115607	109487	247535
Se	µg/g	<291	<248	<225
Hg	µg/g	<639	<544	<496
CS-137	µCi/g			
Am-241	µCi/g	5.33E-01	3.11E-01	5.67E-01
Uncertainty, %		7.75	18.68	6.38
PU-238	µCi/g	9.96E-02	5.58E-02	1.17E-01
Uncertainty, %		24.82	47.76	20
PU-239/240	µCi/g	7.89E-02	4.89E-02	9.39E-02
Uncertainty, %		18.1	43.93	17.81

LIMS #		300193853	300193854	300193855
Sample Name		WSLURY-1	WSLURY-2	WSLURY-3
ALPHA COUNT	µCi/g	5.74E+00	5.58E+00	7.83E+00
Uncertainty, %		20	20	20
BETA COUNT	µCi/g	1.00E+03	9.68E+02	1.84E+03
Uncertainty, %		10	10	10
SR90 BETA LIQ SCINT	µCi/g	2.40E+02	1.99E+02	3.78E+02
Uncertainty, %		10.1	10.8	10
TC99 BETA LIQ SCINT	µCi/g	2.05E-01	2.13E-01	3.05E-01
Uncertainty, %		15.1	12	8.9
Co	µg/l	3.85E+00	2.93E+00	5.75E+00
Co-60	µCi/g	9.02E-02	5.91E-02	1.23E-01
Uncertainty, %		24.6	24.3	9.4
Eu-154	µCi/g	<9.76E-02	<8.60E-02	4.73E-01
Eu-155	µCi/g	<1.33E-01	<1.12E-02	2.04E-01

2.3.4 Rheology

Table 2-11 shows the rheology data[8]. The samples were both unwashed and washed filtrate and slurry. The filtrate viscosity is 3.5 cp. The slurry yield stress measured 1.5 Pa, which is not very high and should not cause processing problems. However, because of the low volume of insoluble solids, personnel were unable to create a sample in sufficient quantity to even approach 15 wt% insoluble solids slurry. If the insoluble solids concentration had been higher, the yield stress would have been much higher.

Table 2-11 Rheology Data

Sample ID (AN-104)	Viscosity or Consistency (cP)	Yield Stress (Pa)
CUF Filtrate	3.5	0
CUF 0.4 wt% Slurry	3.85	0
CUF Washed Filtrate	1.3	0
CUF Washed 2 wt% Slurry	1.9	1.47

2.3.5 Density and Solids Concentration

Table 2-12 shows the density and solids data.

Table 2-12 Density and Solids Data

Sample	Density g/mL	Total Solids	Insoluble Solids	Soluble Solids
Filtrate	1.26	29.3 wt %	0	29.3 wt %
CUF Concentrated Slurry	N/A	30.0 wt %	0.9 wt %	29.1 wt %
1:5 Decanted, Washed CUF Slurry	N/A	11.6 wt %	2.2 wt %	11.62 wt %
Washed Slurry Supernate	N/A	13.6 wt %	0	13.6 wt %

2.3.6 Filter Cleaning

Table 2-13 shows the flux following filter cleaning. The axial velocity for all filter cleaning and flux testing is 11 feet per second. The filter was flushed four times with inhibited water initially. On the second, third, and fourth batch significant foaming was observed. The filter was then cleaned with five successive batches of 1M nitric acid, then two batches of 0.01M nitric acid. Values for inhibited after and strontium carbonate fluxes prior to the AN-104 run are placed in the PRE-Run Flux column for comparison with the Post AN-104 run fluxes after cleaning. The % change column shows the sign and the percentage change going from the PRE-run fluxes to the POST-run fluxes. In all cases, the POST-run fluxes were significantly less than the PRE-run fluxes. It will require further study for us to understand why the filter fluxes do not return to PRE-run conditions and what filter cleaning methods are required for this to be achieved.

Table 2-13 Filter Cleaning Data

Cleaning Agent	TMP (psi)	POST-Run Flux gpm/ft^2	PRE-Run Flux gpm/ft^2	% Change
Inhibited Water 1	40	0.132	N/A	N/A
Inhibited Water 2 **foamed**	40	0.079	N/A	N/A
Inhibited Water 3 **foamed**	40	0.042	N/A	N/A
Inhibited Water 4 **foamed**	40	0.026	N/A	N/A
1M Nitric Acid 1	40	0.282	N/A	N/A
1M Nitric Acid 2	40	0.163	N/A	N/A
1M Nitric Acid 3	40	0.45	N/A	N/A
1M Nitric Acid 4	40	0.439	N/A	N/A
1M Nitric Acid 5	40	1.19	N/A	N/A
0.01 M Nitric Acid 1	40	0.383	N/A	N/A
0.01 M Nitric Acid 2	40	0.445	N/A	N/A
Inhibited Water 5	40	0.531	N/A	N/A
Inhibited Water 6	10	0.108	.34	-67%
Inhibited Water 6	20	0.195	.63	-69%
Inhibited Water 6	30	0.34	omitted	N/A
SrCO3	10	0.112	.25	-55%
SrCO3	20	0.29	.44	-34%
SrCO3	30	0.15	.62	-76%
Inhibited Water 7	20	0.091	.31	-71%

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3.0 FUTURE WORK

Perform foaming study to determine the cause of foam and the impact of foam on filter flux.

It will require further study for us to understand why the filter fluxes do not return to PRE-run conditions and what filter cleaning methods would be required for this to be achieved.

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4.0 REFERENCES

1. P. S. Townson, "241-AN-104 LAW Entrained Solids Ultrafiltration Test Specification", 24590-PTF-TSP-RT-02-011, Rev. 0, October 17, 2002.
2. M. R. Poirier and P. R. Burket, "Task Technical and Quality Assurance Plan for AN-104 Active Waste Ultrafiltration", WSRC-TR-2002-00507, SRT-RPP-2002-00249, Rev. 0, November 19, 2002.
3. M.E. Stallings, C.J. Coleman, M.S. Hay, K.B. Martin, "Task Technical and Quality Assurance Plan for Compositing, Homogenizing, and Characterizing Samples from Hanford Tank 241-AN-104", WSRC-TR-2002-00386, SRT-RPP-2002-00199, rev. 0, September 9, 2002.
4. M. R. Poirier, P. R. Burket, and J. L. Siler, "Filtration of a Hanford AW-101 Waste Sample", WSRC-TR-2002-00530, Rev. 0 (SRT-RPP-2002-00263, Rev. 0), February 12, 2003.
5. J. R. Zamecnik, M. A. Baich, E. K. Hansen, and M. R. Poirier, "AN-102 Simulant Sr/TRU Precipitation and Ultrafiltration", WSRC-TR-2003-00056, Rev. 0 (SRT-RPP-2002-00231, Rev. 0), February 10, 2003.
6. M. R. Poirier, P. R. Burket, and J. L. Siler, "Filtration of a Hanford AY-102/C-106 Sample", WSRC-TR-2002-00530, Rev. 0 (SRT-RPP-2002-00240, Rev. A), June 6, 2003.
7. M.R. Poirier, WSRC-NB-2003-00031, "AN-104 CUF Filtration" Laboratory Notebook.
8. P.R. Burket, "RPP-WTP-AN-104 Filtration Rheology", SRTC-PDH-2003-00011, July 9, 2003.
9. "Procedure for the Operation of the Shielded Cells Unit Filter (U)", Manual number L12.1, procedure IWT-OP-088, February 9, 2001.

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APPENDIX A. FILTER FLUX DATA

Table A- 1 Filter Flux Data

Test 1.3										
Time (hr)	**Tubeside Exit Pressure (psi)**	**Filtrate Pressure (psi)**	**Slurry Flowrate (gpm)**	**Filtrate Flowrate (mL/min)**	**Tubeside Inlet Pressure (psi)**	**Slurry Temp (C)**	**Trans-membrane Pressure (psi)**	**Slurry Flow Vel (ft/s)**	**Flux@ Test Temp (gpm/ft^2)**	**Flux@ 25ºC (gpm/ft^2)**
0	40	0	3.74	N/A	40	25.2	40.5	10.9		
1	40	0	3.89	N/A	40	21.5	41	11.3	Flux	
2	40	0	3.78	N/A	40	23	41	11.0	Data	
3	40	0	3.65	N/A	40	24.3	40	10.6	Table A2	
4	40	0	3.84	N/A	40	23.6	40	11.2		Flux
5	40	0	3.87	N/A	40	26.6	40	11.2		Data
6	40	0	3.27	N/A	40	24.2	40	9.5		Table A2
7	40	0	3.19	N/A	40	25.7	40.5	9.3	Flux	
8	40	0	3.7	N/A	40	22.4	40	10.7	Data	
9	38	0	3.7	N/A	38	25.7	40	10.7	Table A2	
10	38	0	3.62	N/A	38	23.2	40	10.5		Flux
11	38	0	3.6	N/A	38	25.6	40	10.5		Data
12	38	0	3.64	N/A	38	26.9	40	10.6		Table A2
Test										
1.4	40	0	3.81	20.2	40	29.2	40	11.1	0.028	0.025
1.5	40	0	3.78	15	40	24.9	40	11	0.021	0.021
1.6	40	0	3.78	15	40	25.3	40	11	0.021	0.021
1.7	30	0	3.1	11.5	30	22.5	30	9	0.016	0.017
1.8	30	0	4.53	15.2	30	25.1	30	13.2	0.021	0.021
1.9	48	0	3.71	21.4	48	28.8	48	10.8	0.03	0.027
1.1	50	0	3.09	20.3	50	30	50	9	0.028	0.024
1.11	40	0	3.71	13.8	40	25	40	10.8	0.019	0.019
1.12	40	0	2.37	14.1	40	25	40	6.9	0.02	0.02
1.13	40	0	4.16	16.4	40	30.1	40	12.1	0.023	0.02
1.14	20	0	3.7	7.7	20	25	20	10.7	0.011	0.011
1.15	60	0	3.01	18.8	60	27.8	60	8.7	0.026	0.024
1.16	40	0	3.86	12.1	40	25.3	40	11.2	0.017	0.017

Table A- 2 Test 1.3 Dewatering Flux

AN-104 Average Dewatering Flux					
Volume of Permeate Removed (mL)	**Flux x 1000 gpm/ft^2**	**Volume x Flux**	**Temperature ^{o}C**	**Temperature Corrected @25^{o}C**	**Volume x Flux**
250	236	59000	24	243	60690
250	146	36500	24	150	37546
250	110	27500	24.3	112	28049
250	81	20250	24	83	20830
250	128	32000	23	136	33866
250	78	19500	23	83	20637
250	61	15250	23.3	64	16002
230	51	11730	23.6	53	12204
250	68	17000	24	70	17487
210	38	7980	26.6	36	7630
250	53	13250	23	56	14023
140	52	7280	23.3	55	7639
250	49	12250	24.2	50	12530
250	48	12000	24	49	12344
250	63	15750	22.2	68	17054
250	50	12500	22.5	54	13419
215	45	9675	22.5	48	10387
Average	**Average**	**Average**			
238	80	19377			20137
	Weighted Average of Flux			**Weighted Average of Flux**	
	@ Temp	0.081		@ 25 C	0.085

APPENDIX B. FILTER FLUX STATISTICAL DATA

JMP® Statistical Analysis of AN-104 Filtration Test Matrix Data

Response Flux@25
Whole Model

Summary of Fit

RSquare	0.647356
RSquare Adj	0.576827
Root Mean Square Error	0.00269
Mean of Response	0.020538
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.00013286	0.000066	9.1786
Error	10	0.00007237	0.000007	Prob > F
C. Total	12	0.00020523		0.0055

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	9	0.00007237	0.000008	.
Pure Error	1	0.00000000	0	Prob > F
Total Error	10	0.00007237		.
				Max RSq
				1.0000

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.000116	0.007148	-0.02	0.9874
TMP	0.0003581	0.000084	4.28	0.0016
Axial Vel	0.0006126	0.000504	1.21	0.2525

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
TMP	1	1	0.00013246	18.3025	0.0016
Axial Vel	1	1	0.00001067	1.4745	0.2525

Probability TMP < 0.05, TMP statistically significant
Probability Axial Velocity > 0.05, axial velocity not statistically significant.

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APPENDIX C. ANALYTICAL REQUIREMENTS

Analytical Requirements for Filtrate, Washed Solids, Wash Solutions

Analyte	Washed Solids Minimum Reportable Quantity (MRQ) µCi/gm	Filtrate, Wash Solutions Minimum Reportable Quantity (MRQ) µCi/mL
Cesium-137	6.0E-02	9.0E+00
Strontium-90	7.01E+01	1.5E-01
Technetium-99	**6E+00 µgm/gm**	1.5E-03
Americium-241	1.2E-03	7.2E-04
Europium-154	6.0E-02	2.0E-03
Europium-155	6.0E-02	9.0E-02
Pu-239/240	**6.0E+00 µCi/gm**	9.6E-03
Total Alpha/Beta	1.0E-03	2.3E-01
	µgm/gm	**µgm/mL**
Ag	2.0E+01	N/A
Al	3.3E+02	7.5E+01
As	6.0E+02	N/A
Ba	6.0E+02	7.8E+01
Be	1.5E+02	N/A
Ca	1.8E+02	1.5E+02
Cd	1.1E+01	7.5E+00
Co	3.0E+00	3.0E+01
Cr	1.2E+02	1.5E+01
Cu	1.8E+01	1.7E+01
Fe	1.4E+02	1.5E+02
Hg	3.0E+00	1.5E+00
K	1.5E+03	7.5E+01
La	6.0E+01	3.5E+01
Mg	5.4E+02	1.5E+02
Mn	3.0E+02	1.5E+02
Mo	6.0E+00	9.0E+01
Na	1.5E+02	7.5E+01
Ni	1.6E+02	3.0E+01
Pb	6.0E+02	3.0E+02
Sb	1.5E+02	N/A
Se	7.5E+02	N/A
Si	3.0E+03	1.7E+02
Sr	3.0E+02	6.5E-01
Ti	1.5E+02	1.7E+01
Tl	3.0E+01	N/A
U	6.0E+02	6.0E+02
Zn	6.0E+00	1.65E+01
TOC	6.0E+01	1.5E+03
TIC	3.0E+01	1.5E+02
Cl	2.3E+02	3.0E+00
F	7.5E+03	1.5E+02
NO3	*	3.0E+03
SO4	1.2E+03 (as S)	2.3E+03
PO4	6.0E+02 (as P)	2.5E+03

* Nitrate concentration in solids will not be measured.
N/A NOT APPLICABLE

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APPENDIX D. SCANNING ELECTRON MICROSCOPE PICTURES OF SOLIDS PARTICLES

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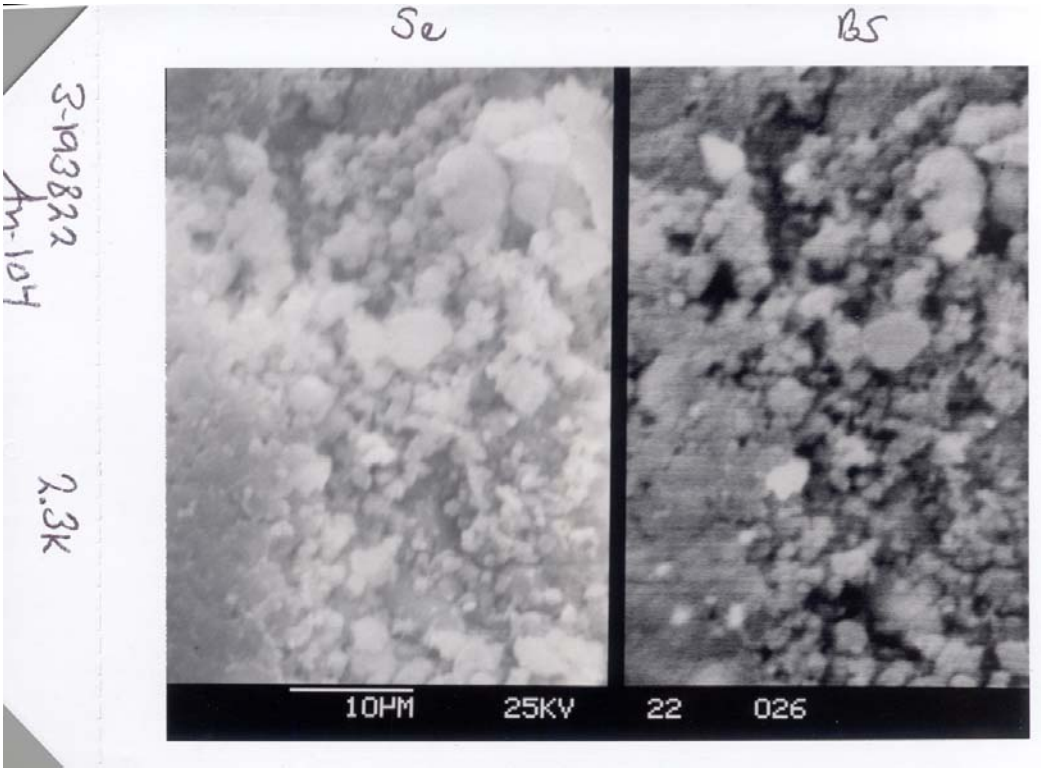

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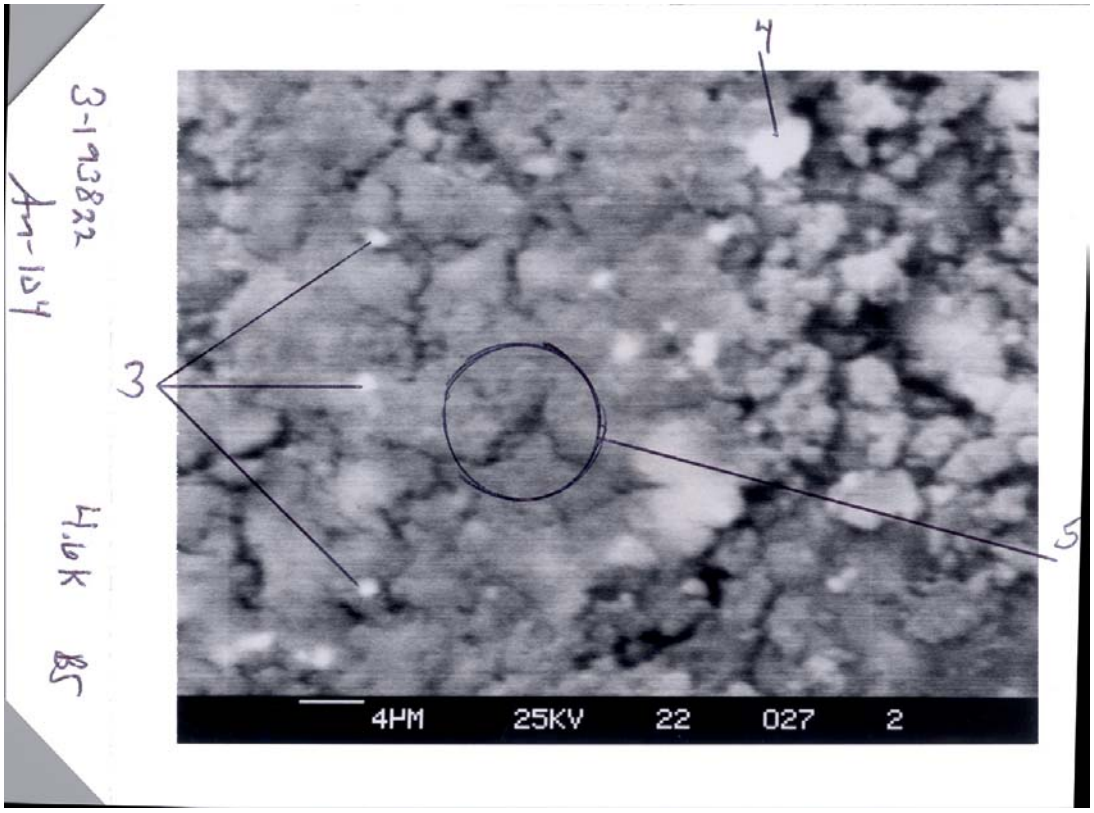

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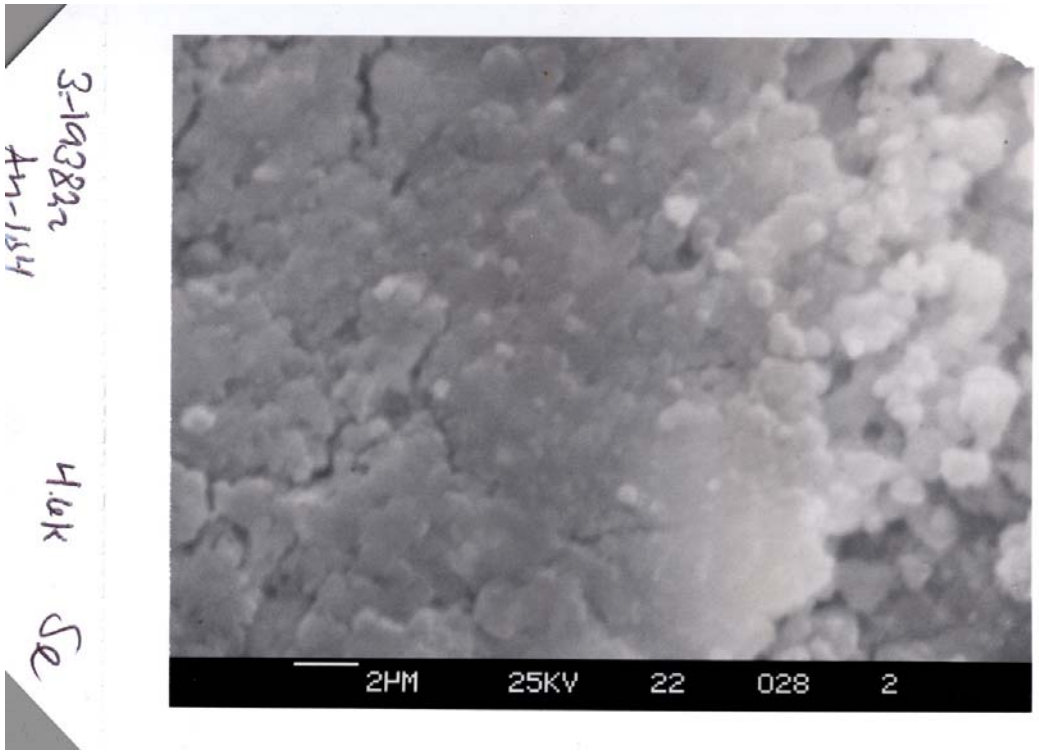

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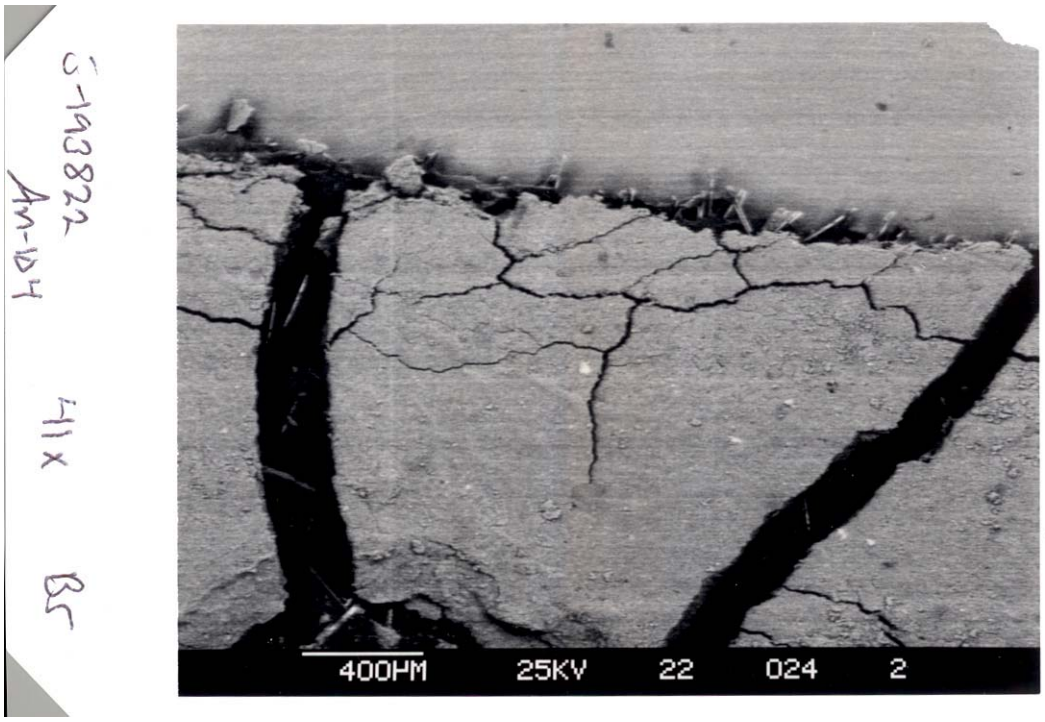
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Am-104
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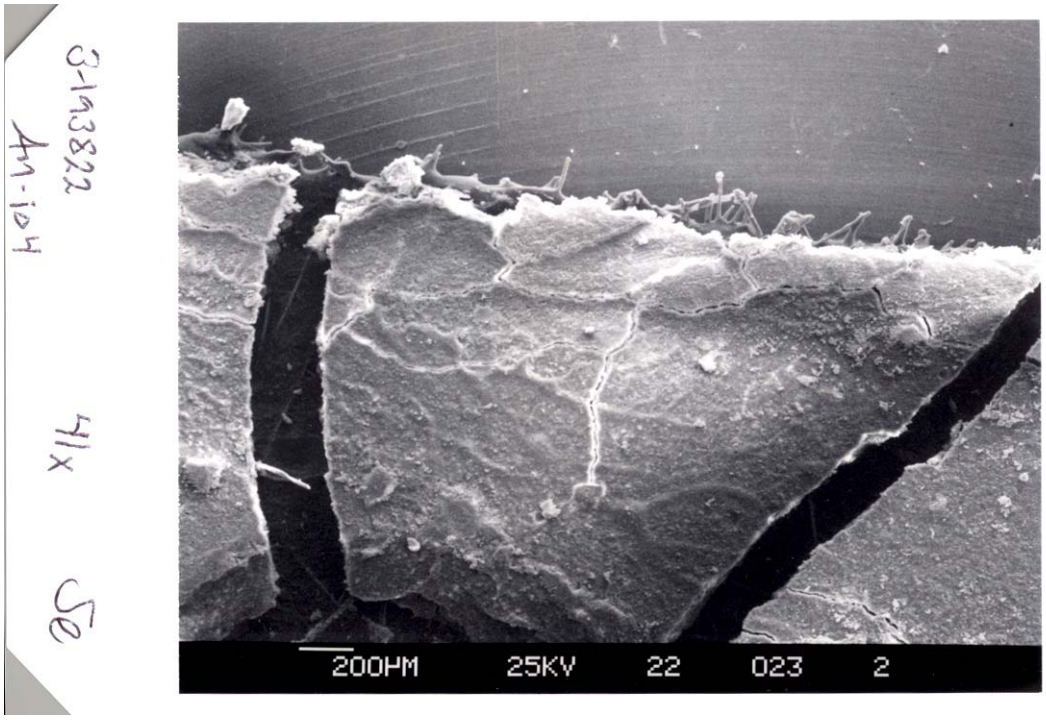
BS
400UM 25KV 22 024 2

41X SE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