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Strontium-Transuranic Precipitation and Crossflow Filtration of 241-AN-102 Large C

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Table of Contents

Figures

Tables

1.0 SUMMARY

This work provides an important confirmation of the new strontium/permanganate precipitation process to achieve both acceptable filterability and decontamination for Envelope C (Tanks 241-AN-102 and 241-AN-107) wastes to be treated by the Hanford River Protection Project. As a bench-scale demonstration, a series of seven precipitation batches and crossflow filtration campaigns were performed to remove strontium-90 and transuranics from 16.5 liters of Tank 241-AN-102 “Large C” supernatant liquid containing entrained solids. In each batch, the Large C sample was caustic adjusted, strontium and permanganate precipitated, and crossflow filtered with entrained solids using a 2-foot long, 3/8” internal diameter, 0.1 micron pore size Mott crossflow filter tube. Test ranges for the transmembrane pressures and crossflow velocities were in the range of 30 to 70 psig (2.07 to 4.83 bar) and 9 to 15 ft/s (2.7 to 4.6 m/s), respectively.

The filtrate product was decontaminated for strontium by a factor exceeding 40 to produce a 1.0 μCi/ml product at 5.8 M sodium, far below the 8.4 μCi/ml maximum at 6.0 M Na design specification. This work also showed that strontium levels are reduced by simple isotopic dilution and strontium decontamination factors can be predicted *a priori*. Transuranics decontamination factors between 2.9 and 12.4 were achieved to produce filtrates below the 0.109 μCi/ml limit. This precipitation process is also metal selective where, for example, technetium-99 was not removed and was favorably non-selective towards aluminum. Washing of the high insoluble solids slurry with inhibited water showed simple dilution effects on most metals, although there was evidence that plutonium, calcium, strontium, and lead did leach somewhat. Lastly, no post-treatment precipitation in the product filtrate was observed after several days of storage in the SRTC Shielded Cells.

The slurry produced from the strontium/permanganate precipitation process was crossflow filterable producing fluxes similar to the design basis “optimum” of 3.8 m^3/m^2*day (0.065 gpm/ft^2) for a Tank AN-102 waste with 2% insoluble solids. At 1.5 wt% insoluble solids, increasing transmembrane pressure and increasing fluid axial velocity improved filter fluxes, with permeance maximized at lower transmembrane pressure and high axial velocity. Lower transmembrane pressure and higher axial velocities improved both filter fluxes and permeances at high insoluble solids levels (12-14 wt%) and when washing accumulated solids in the filter loop after Hanford material filtration. No significant differences in filter fluxes was found when the calcium nitrate-strontium nitrate-sodium permanganate data obtained in this study was compared with literature data for strontium nitrate-sodium permanganate strike. Lastly, a Cells Unit Filter operation shutdown and restart was found to improve filter flux like a backpulse up to approximately 7 wt% insoluble solids loadings.

Based on this work, further work is recommended to examine the possibility of meeting decontamination limits with reduced reagent levels. As decontamination far exceeded requirements, the total added oxide sent to the High Level Waste melter may be reduced.

Optimization of filtration rates by varying backpulse frequency was not part of the scope of this work and is also suggested.

2.0 INTRODUCTION

A scoping study by Herting[1] showed the efficacy of a strontium nitrate-ferric nitrate strike on the removal of strontium-90 and transuranics from caustic adjusted Hanford wastes. However, the ferric hydroxide precipitation resulting from this chemistry created filtration difficulties, leading to the discovery of a filterable calcium nitrate-strontium nitrate-sodium permanganate strike during an investigation of alternate chemistries with simulated waste at the Savannah River Technology Center (SRTC)[2]. An investigation at the Pacific Northwest National Laboratory (PNNL) also demonstrated sufficient removal efficiencies and filterability using a simpler strontium nitrate-sodium permanganate strike[3]. Based on this initial work, a small scale study with radioactive Hanford AN-102 (Envelope C) waste was conducted using lower strontium and permanganate quantities, and demonstrated acceptable filterability and Sr/TRU removal[4]. This new process reduces strontium-90 by isotopic dilution with inactive strontium nitrate addition through precipitation of strontium carbonate, with permanganate precipitating as manganese dioxide providing high lanthanide and transuranic decontamination. A detailed literature background supporting the new process is given in the second reference.

The purpose for this work was to demonstrate the efficacy of this new process on a bench-scale in semi-continuous operation, and to optimize filtration performance. This investigation, which represents the second radioactive crossflow filter testing of the strontium/permanganate process for Envelope C, was conducted according to a Technical Task Plan[5] written in response to the cited Task Specification[6]. This work also served as a confirmation of the filterability of the AN-102 supernatant liquid after precipitation without prefiltration to remove entrained solids. SRTC and PNNL have found that the entrained solids in Envelope C have been very difficult to filter and have activities sufficiently high for classification as High Level Waste.

3.0 EXPERIMENTAL AND RESULTS

3.1 Tank 241-AN-102 Sample History

The Hanford site contractor obtained approximately 14.25-liters of waste solution from tank 241-AN-102 during 1998. The waste samples were obtained by lowering sample bottles into the liquid phase of the waste, and are denoted as "grab samples" since the contents of Tank 241-AN-102 were not agitated prior to or during the sampling event. The Hanford site contractor then packaged these samples for shipment which the SRTC received in four separate deliveries from October through December, 1998. Summary information on the received samples is listed in Table 1.

Initially, each shipment of 8 samples were composited into 4 L polyethylene bottles. From the first shipment composite, approximately 1.5 L was withdrawn to create the

"Small C" sample. The remainder of "grab samples" was composited in a 25 L carboy, mixed well, and then characterized as the "Large C" sample as reported by Hay[7]. Of the composited sample, 10.1 L was then split into seven 4-liter polyethylene bottles and each bottle was diluted initially using inhibited water (0.01 M NaOH) to within 5% (see Table 12 of Hay's report) of the 6 M sodium target from above, and analyzed by ICP-ES. As solids dissolution may have caused an initial increase over the target, a second dilution was subsequently made to produce a ~16.4 L total final volume, and the results of this process are summarized in Table 2.

Table 1 Sample Identifications for Small C Composite

Jar Label	Number of Jars	Sample Date	Sample Type
2AN 98-43 to 2AN 98-48	6	07/21/98	Unaltered Grab Sample
2AN 98-49 to 2AN 98-54	6	07/22/98	Unaltered Grab Sample
2AN 98-58 to 2AN 98-62	5	08/10/98	Unaltered Grab Sample
2AN 98-63 to 2AN 98-67	5	08/11/98	Unaltered Grab Sample
2AN 98-68 to 2AN 98-72	5	08/12/98	Unaltered Grab Sample
2AN 98-LCOMP1,3,4,5,8	5	07/21/98	Liquid Core Composite

Table 2 Sodium Level Adjustment in Tank 241-AN-102 Samples

Bottle Name Suffix*	**Dil-1**	**Dil-2**	**Dil-3**	**Dil-4**	**Dil-5**	**Dil-6**	**Dil-7**
[Na+] after initial dilution	5.75	7.07	7.16	6.37	6.31	6.57	6.69
Volume, ml	2700	2100	2000	2400	1500	2200	2300
Total Na+, grams	357	341	329	352	218	332	354
DI water added, ml (second dilution)	0	350	375	0	0	200	250
New Vol., ml	2700	2450	2375	2400	1500	2400	2550
Final [Na+], M	5.75	6.06	6.03	6.37	6.31	6.02	6.04

*All names begin with "BNF-LC100-", e.g., BNF-LC100-Dil-1.
Total grams sodium = 2283 grams or 99.3 moles
Total final volume = 16.375 liters [average sodium molarity = 6.06]

3.2 Precipitation Recipes

Two precipitation recipes were studied in this work to further examine the two successful chemistries identified at PNNL and SRTC: 1) addition of non-radioactive $Sr(NO_3)_2$ and $NaMnO_4$ to take advantage of high filterability and reasonable Sr^{90} and TRU removal efficiencies[8,9], and 2) addition of $Ca(NO_3)_2$, non-radioactive $Sr(NO_3)_2$, and $NaMnO_4$ to balance higher removal efficiencies against lower filterabilities associated with Ca addition[10]. These were added after pH adjustment using concentrated NaOH.

The concentration of free OH- and each metal in the final mixture was adjusted upward by the quantities listed in Table 3 based on the most effective formulations known at the time (early November, 1999) at the SRTC[11]. The cation/anion additions were made using 17 M NaOH, 1 M $Ca(NO_3)_2$, 1 M $Sr(NO_3)_2$, and 1 M $NaMnO_4$ solutions, added to the feed solution in that order.

Table 3 Concentration Raise Targets for the Two Sr/TRU Precipitation Recipes

Cation/Anion \ Recipe	Concentration [M]	
	$Ca/Sr/MnO_4$	Sr/MnO_4
Free OH^-	0.875	0.875
Ca^{2+}	0.008	-------
Sr^{2+}	0.02	0.075
MnO_4^-	0.03	0.05

Table 4 Concentration of diluted feed and adjusted feed (assuming ideal mixing)

Cation/Anion \ Na conc.	Concentration [M]		
	6.4 M Na (diluted)	6.0 M Na (adjusted)	6.0 M Na [calc.] (PNNL, 1998[12,13])
Free OH^-	0.817*	0.763*	0.0972
Sr^{2+}	0	0	0
Ca^{2+}	0.008	0.0075	0.00782
* Erroneously high – see text			

Analysis of a 241-AN-102 sample after dilution to 6.4 M Na by Hay[14] showed the concentrations of the ions of interest in Table 4. Values relevant to the 6 M Na+ sample (assuming ideal mixing in the calculation) in the current work are shown as well. The last column of Table 4 presents the calculated diluted composition data from the 1998 PNNL analysis of the same grab samples. Micromolar concentrations of Sr-90 observed in both SRTC and PNNL analyses were neglected here. Hay believes the high SRTC free hydroxide result is due to a high bias caused by incomplete carbonate precipitation by the $SrCl_2$/titration method and/or from the presence of the organic complexing agents.

Based on the initial concentrations at 6.0 M Na in Table 4 and the concentration raise targets in Table 3 (target concentration = adjusted feed concentration + Table 3 quantity), quantities of each solution to add per liter of 6.0 M Na Large C Envelope adjusted feed was calculated by simultaneously solving a set of linear equations, assuming ideal mixing, for each recipe. The results of those calculations based on both SRTC and PNNL initial concentration results are shown in Table 5. Refer to Appendix B for the spreadsheet used for these calculations.

Table 5 Sr/TRU Precipitation Recipes – Calculated volumes of solution addition per liter 6.0 M Na Large C (AN-102) feed

Solution \ Recipe	Volume [ml]			
	Per SRTC feed composition		Per PNNL feed composition	
	$Ca/Sr/MnO_4$	Sr/MnO_4	$Ca/Sr/MnO_4$	Sr/MnO_4
17 M NaOH	64.1	73.3	58.6	63.8
1 M $Sr(NO_3)_2$	22.6	92.0	22.5	91.2
1 M $NaMnO_4$	33.9	61.3	33.8	60.8
1 M $Ca(NO_3)_2$	10.0	-----	10.0	-----

Mistakenly, the reagent addition volumes based on the SRTC analytical results were used instead of those based on PNNL results that were thought to be more accurate. Hence, an over-addition of the sodium hydroxide and precipitation reagent solutions resulted. Table 6 shows that the final expected concentrations of each species after addition likely did not change much beyond analytical uncertainty, compared to the target concentrations based on the PNNL feed composition data.

Table 6 Sr/TRU Precipitation Recipe Final Concentration of Target Species – Target vs. Expected for Each Precipitation Recipe

Solution \ Recipe	Molarity [M]			
	Target Concentration		Expected Concentration	
	$Ca/Sr/MnO_4$	Sr/MnO_4	$Ca/Sr/MnO_4$	Sr/MnO_4
Free OH-	0.972	0.972	1.05	1.10
Sr^{2+}	0.020	0.075	0.020	0.075
Mn^{x+}	0.030	0.050	0.030	0.050
Ca^{2+}	0.016	-----	0.016	-----
Na (resulting)	6.22	5.83	6.27	5.91

The deviation in the recipe calculation did not affect the resulting strontium and manganese concentrations, increases sodium concentration 0.05 – 0.08 M, and increases free hydroxide concentration by 0.07 – 0.12 M. While higher than the requested recipe, the resulting added free hydroxide is still very close to Rosencrance's recommended maximum addition of 1.0 M free hydroxide[15] found in his statistically-designed experiments with another sample of 241-AN-102.

3.3 Precipitation/Filtration Batch History

The seven partially-filled 4 liter carboys were used for seven precipitation batches, and close but not exactly on a one-to-one basis. As volumes in the carboys were approximate, a few hundred milliliters of feed left in the previous carboy was sometimes carried forward to the next batch. Batches 6 and 7 (Table 7) are the only batches with a significant mixing of different carboys. The best estimate for the actual quantities of each carboy fed into each of the seven precipitation batches is listed in Table 7.

The measured sodium levels at the bottom of Table 7 show that final dilution to the desired 6 M sodium level did very well. All bottles were within the +/- 10% analytical error for sodium measurement.

As solutions cannot easily be quantified and poured in the Shielded Cells where the CUF was operated, precipitation solutions were pre-measured and placed into individual bottles for one-time use. The pre-measured quantities used and listed in Appendix A were calculated based on the original experimental design volumes of 1.2 L and 2.4 L batch sizes rather than the actual volumes. As the second dilutions performed by Hay made the total amount of feed actually received larger than the (1.2 + 6*2.4 = 15.6 liter) plan (Figure 1), calculated final target ion concentrations (Table 7) through addition of the precipitating agents was slightly lower than planned. The process of adding these precipitation solutions raised the volume to be filtered from the approximately 16.5 L of 6.0 M Na Tank AN-102 supernatant liquid to nearly 19.8 L total precipitated slurry.

Table 7 Sodium Level Adjustment in Tank 241-AN-102 Samples

Precipitation batch #	**1**	**2**	**3**	**4**	**5**	**6**	**7**
~6.0 M Na AN-102 volume (L)	1.5	2.4	2.7	2.4	2.55	1.7 (+0.7 L CUF conc.)	3.1 (0.6 + 2.5)
AN-102 + precipitants volume (L)	1.66	2.94	3.24	2.94	3.09	2.2	3.64
Calculated Na	6.50	6.21	5.71	5.92	5.94	5.93	5.96
Calculated Free OH^-	0.88	1.10	1.00	1.10	1.05	1.32	0.90
Calculated Sr^{2+}	0.016	0.075	0.068	0.075	0.071	0.040	0.061
Calculated Mn^{x+}	0.025	0.050	0.045	0.050	0.048	0.041	0.040
Calculated Ca^{2+}	0.014	-----	-----	-----	-----	-----	-----
Bottle Used Suffix*	-DIL5	-DIL4	-DIL1	-DIL6	-DIL7	-DIL3	-DIL2
Bottle Na+ molarity	6.31	6.37	5.75	6.02	6.04	6.06	6.03

*All names begin with “BNF-LC100-“, e.g., BNF-LC100-Dil-1.

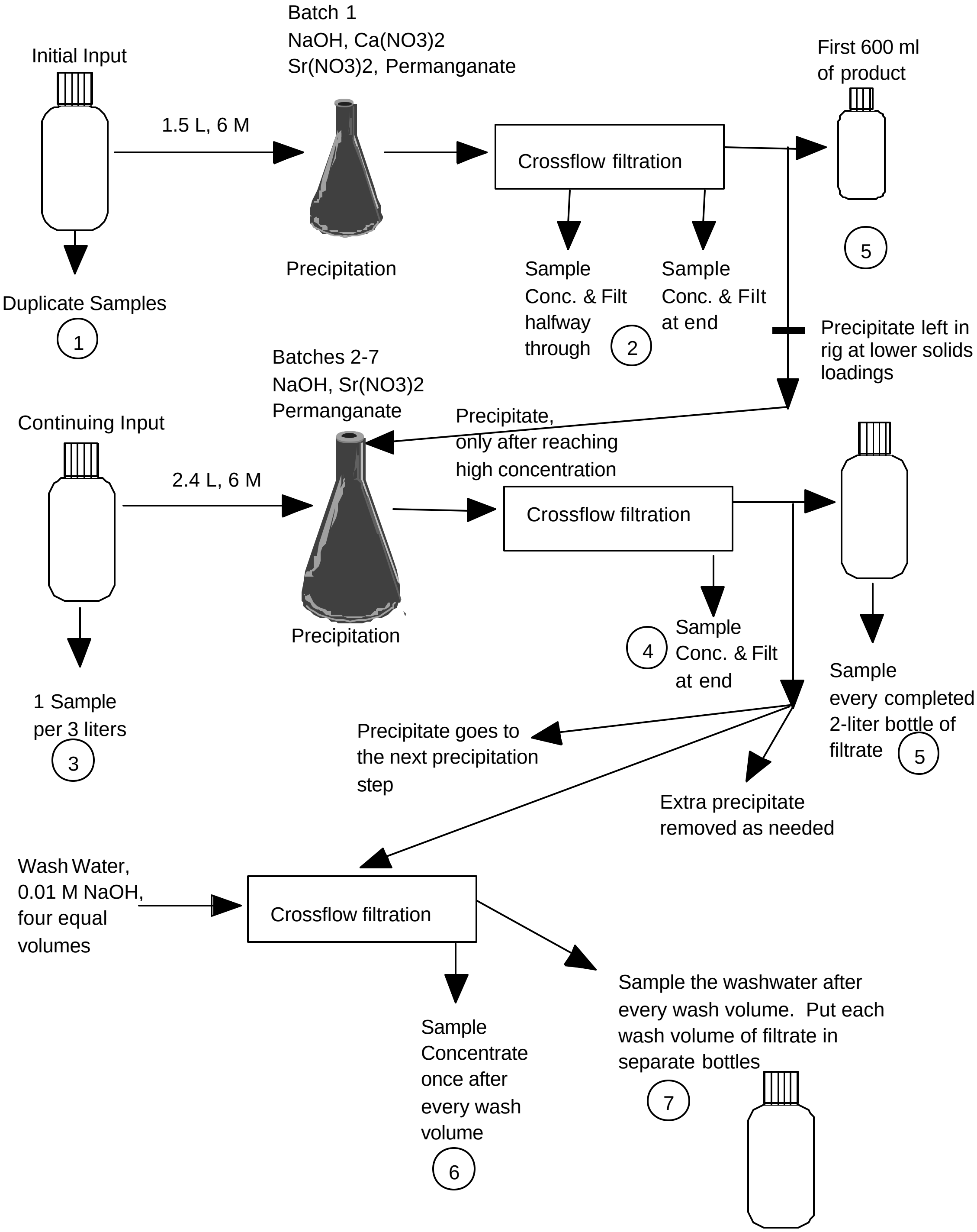


Figure 1 Sample Processing Flowsheet

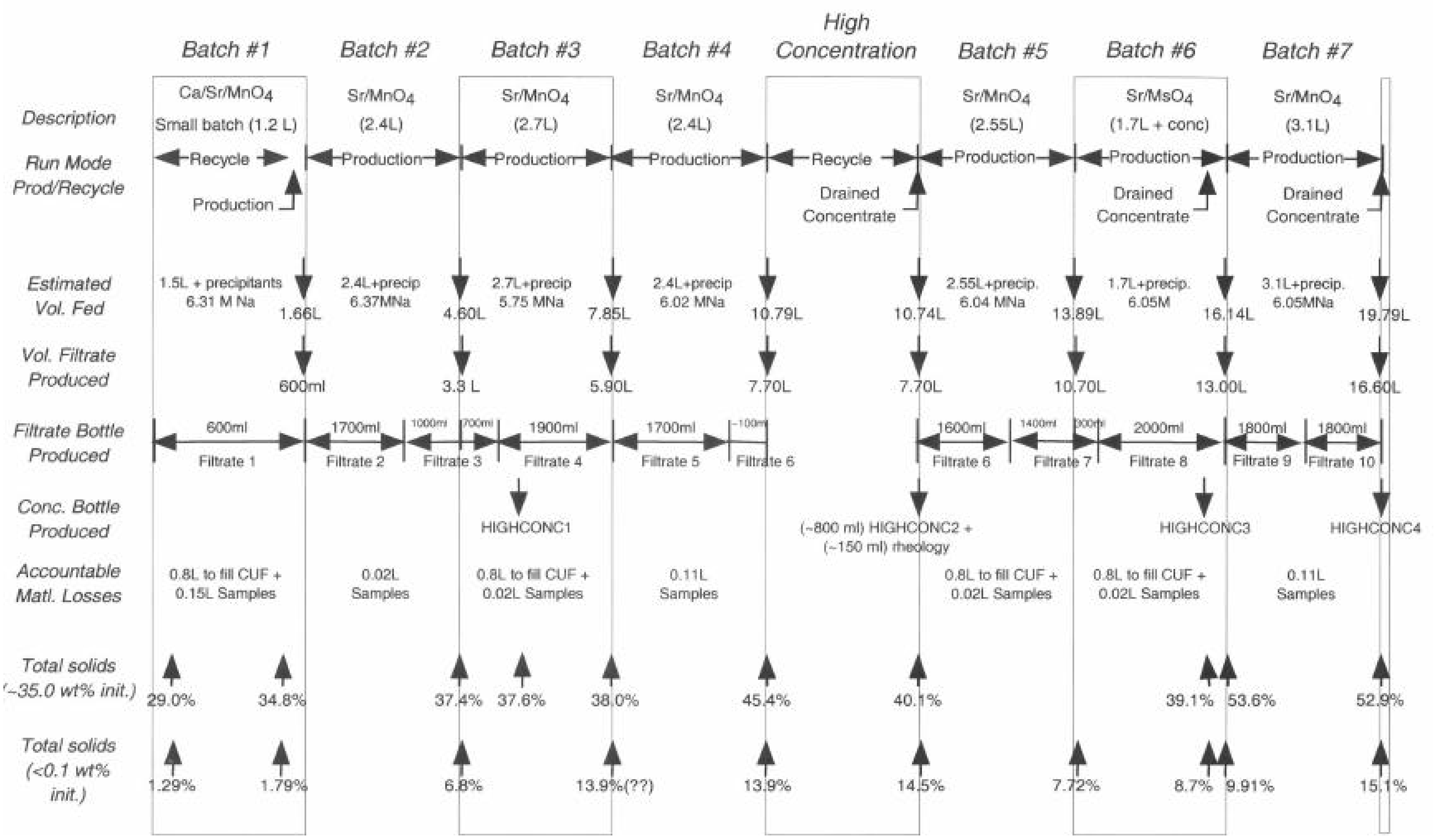


Figure 2. Run Data Summary

Figure 1 also shows the process flow and sampling. The Run Data summary (Figure 2) lists in graphical form the information in Table 7 and Figure 1, as well as the product filtrate bottles produced from each precipitation and filtration batch, when the crossflow filter unit was emptied of high insoluble solids content slurry due to low filtration fluxes, and other relevant operation and experiment material balance information.

Generally, the Cells Unit Filter (CUF) crossflow filter unit was operated by semi-continuously adding precipitated slurry as filtrate (or permeate) was produced continuously, allowing insoluble solids to accumulate in the filter loop. When pump and filter performance seriously deteriorated, the CUF was emptied (during Batch 3, before Batch 5, and during Batch 6) before precipitated feed material was added and filtration operations continued. At the end of Batch 7, the accumulated solids were washed in four batches by semi-continuously feeding 600 ml aliquots of inhibited water (0.01 M NaOH) as filtrate was produced continuously.

After the fourth wash campaign the washed solids/slurry was drained from the CUF rig as well as possible using the drain valves and briefly running the pump. The CUF was then flushed out using deionized (DI) water, then 1 M nitric acid, and DI water rinses. Then post-experiment clean water fluxes were lastly taken. Caustic cleaning solution was not used.

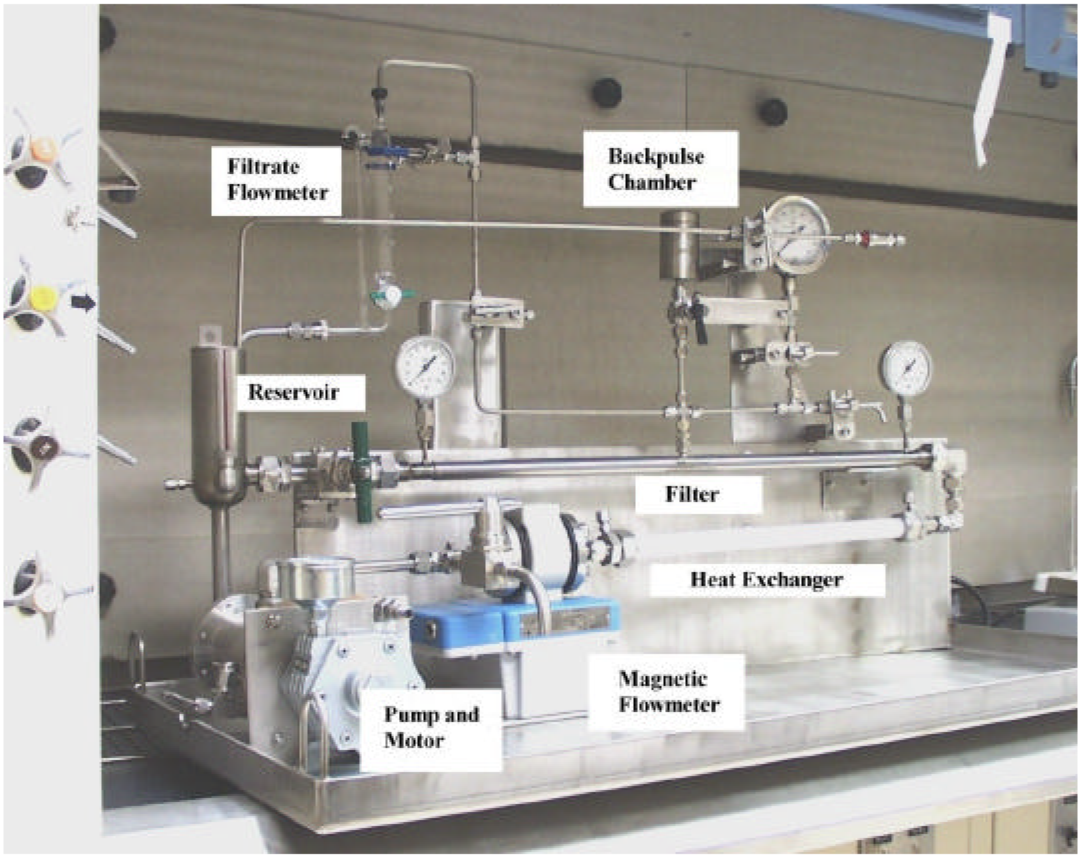
3.4 Apparatus

Crossflow filtration is a process where a slurry concentrate passes down a porous tube or channel under pressure, forcing permeate through the walls of the channel and at the same time sweeping concentrated solid cake off the filter medium. The process stands in contrast to dead-end filtration where the cake continues to build on the porous medium. Backpulsing of a crossflow filter (short reversal of the pressure across the medium) reverses the filtrate flow momentarily for extended cake removal so that filter flux is boosted.

Crossflow filtration was performed with a Cells Unit Filter (CUF) rig that was set up in Cell 16, B-Block, at SRTC. Figure 3 shows the unit without cooling tubes connected to the heat exchanger for clarity. Feed from the reservoir at the left goes to a progressive cavity pump. The pump is operated at variable speed by controlling air pressure to the air motor that drives it. Liquid is pumped through a magnetic flowmeter and heat exchanger that removes pump heat. It then passes down the center of a crossflow filter of 2-foot porous length. A throttle valve downstream drops fluid pressure back to atmospheric.

A 0.1 micron-pore Mott filter tube manufactured for "liquid" service was used in this work. The cold CUF work (ref. 1) found that Mott makes different sintered 0.1 micron stainless steel for "liquid" and "gas" service and that the "liquid" style is a requirement for our work. The filter has a two-foot active length, 3/8" ID bore, and 1/16" wall thickness. The filter elements planned for use in the RPP-WTP are also 0.1 micron-pore size Mott filter tubes. However, the RPP-WTP filter elements will be approximately 80 inches in length.

Figure 3. Cells Filter Unit in a Nonradioactive Laboratory



Filtrate production was measured with a graduated sightglass and stopwatch. A simple backpulse system can be charged with filtrate. Compressed air stored in the filtrate chamber forces reverse flow upon the filter medium. Standard Bourdon-type pressure gauges indicate pressure. A thermocouple mounted near the bottom of the reservoir measures slurry temperature directly. Details of the CUF are documented on six approved engineering drawings.[16]

The filter in this work was a 3/8-inch internal diameter, 2-foot long Mott Metallurgical sintered stainless steel filter. The nominal pore size was 0.1 micron. The single filter tube was mounted in a stainless steel housing of welded construction.

3.5 Filter Operation

General operation of the CUF follows an SRTC procedure.[17] Activities and data are recorded in a controlled laboratory notebook.[18] The rig is first cleaned by circulation of 1 M nitric acid, 1 M sodium hydroxide, or deionized water. These filter cleaning fluids were prefiltered with 0.22 micron nylon filters before use. 1 M sodium hydroxide was not used in the current work – only 1 M nitric acid with DI water flushes before and after were used for cleaning.

Clean water fluxes were taken after the rig was flushed with cleaning fluids. Transmembrane pressures were between 5 and 20 psid and fluxes were measured for periods of 20 minutes or more per the TTP, after initial backpulsing.

Operation of the CUF involved the following routine:

1. Filtrate was generated to fill the backpulse chamber. The chamber was air-pressurized to provide 45 psid overpressure. Filter concentrate pressure was reduced to a few psig for best backpulse effectiveness.
2. Two backpulses were performed before each set of conditions was run.
3. Conditions were set after the second backpulse while the filtrate valve was kept closed.
4. Each run started when the filtrate valve was opened slowly. Slowness was controlled by watching reduction of filtrate side pressure while the needle valve was opened.
5. Samples of concentrate and filtrate were taken at the beginning, middle, and end of the campaign.

RESULTS AND DISCUSSION

3.6 Precipitation Chemistry Results

3.6.1 Decontamination – Desired Targets for the Process

The purpose of the precipitation/filtration process is to decontaminate the waste entering the plant so the bulk of the mass, a liquid stream, will produce immobilized low-activity waste (ILAW) glass when vitrified. The transuranic limit here is 100 ηCi/gram of glass. For the pretreated AN-102 waste, the target waste sodium <u>oxide</u> loading in the ILAW glass was 17 wt%. This translates to a decontamination of 794 ηCi/gram of sodium or less, or 127 ηCi/ml of 7 molar sodium liquid.

The waste sodium oxide loading in the ILAW glass produced from the pretreated AN-102 sample was revised to 11.8wt% subsequent to conducting these tests. The waste sodium oxide loading was reduced to avoid the formation of a separate sulfur phase during the vitrification of the pretreated AN-102 waste. The maximum allowable Sr-90 and TRU concentrations in the pretreated AN-102 waste are 13.8ηCi/ml and 183.9ηCi/ml, respectively for 7M Na solution, based on the lower waste sodium oxide loading in the ILAW glass. For the 4.82M Na AN-102 sample, the maximum allowable Sr-90 and TRU concentrations in the pretreated AN-102 waste are 9.5ηCi/ml and 126.6ηCi/ml, respectively

The strontium-90 target is 20 Ci/cubic meter. This translates to an upper strontium level of 9.8 ηCi/ml for the same basis of 7 M sodium liquid.

The filtrate for most of the Large C campaign was nominally 6 M in sodium. The final composited product from the campaign was 4.82 M in sodium because filtrate from wash water was part of the composite. The limits for transuranics and strontium-90, and comparison with 241-AN-102 liquid are therefore:

Sodium concentration	**Total TRU (alpha)**	**Strontium-90**
6 M Sodium	109 ηCi/ml	8.4 μCi/ml
4.82 M Sodium	88 ηCi/ml	6.75 μCi/ml
6 M Na+ 241-AN-102	150 ηCi/ml *	53 μCi/ml
* Excludes Cm-244, which is at uncertain levels because of possible contamination in SRTC shielded cells.		

3.6.2 Decontamination – Strontium

Strontium-90 was tracked throughout the campaign and Figure 4 summarizes the results. The limit above is also shown on Figure 4. Instantaneous samples of filtrate were taken at the completion of each batch of precipitate. They are grab samples taken from the filtrate outlet. In contrast, “Bottle” samples are taken at the end of the filling of each filtrate collection bottle. They are thus more representative of a composite because the filtrate bottle has from 600 to 2000 ml of filtrate product when Bottle samples are taken.

It is seen that the process is robust in removing strontium-90 from the filtrate stream. In Batch #1, the target added concentration of Sr was only 0.02M. Calcium has previously been shown not to affect the removal of Sr-90 from Envelope C solutions. Even with this non-conservative level of strontium addition the process output was less than half of the tolerable limit.

A decontamination factor calculated from total strontium (not Sr-90) is shown on the y-axis in Figure 5. This is calculated by dividing 0.075 M by the measured total strontium measured in the filtrate. It would be comparable to the active decontamination factor in the case where isotopic mixing is good during the precipitation and mixing. The typical values of 40 to 50 during the campaign (average of 48) agree with the active data of Figure 4 because the feed Sr-90 activity was 50 μCi/ml. The typical activities of 1 μCi/ml shown in Figure 4 indicate a decontamination factor of approximately 50.

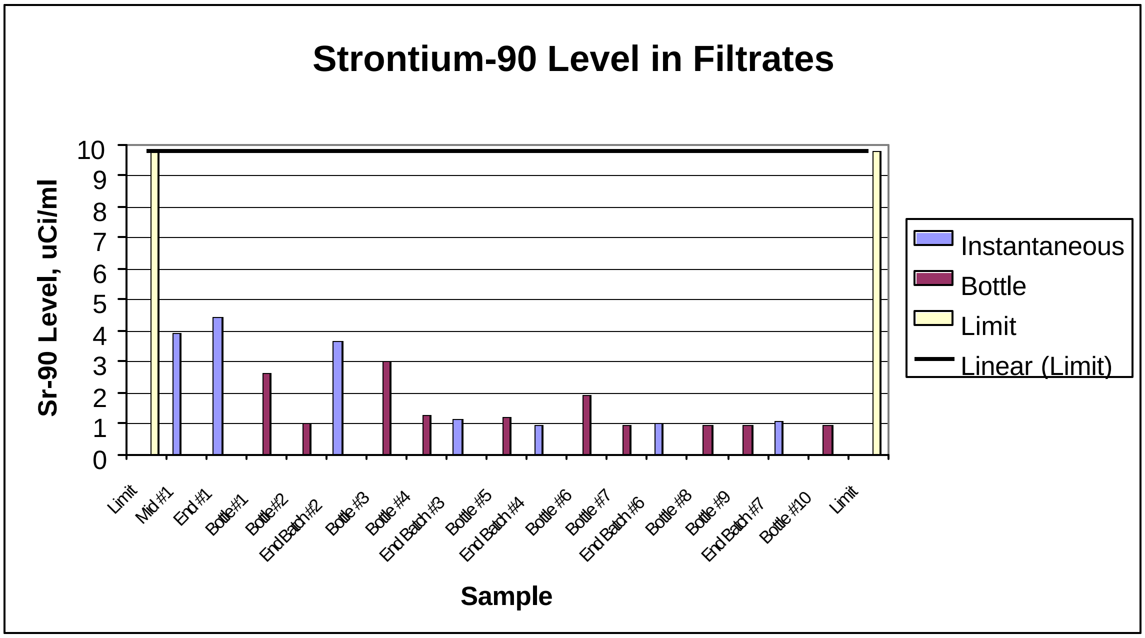


Figure 4 Strontium-90 Level in Filtrates

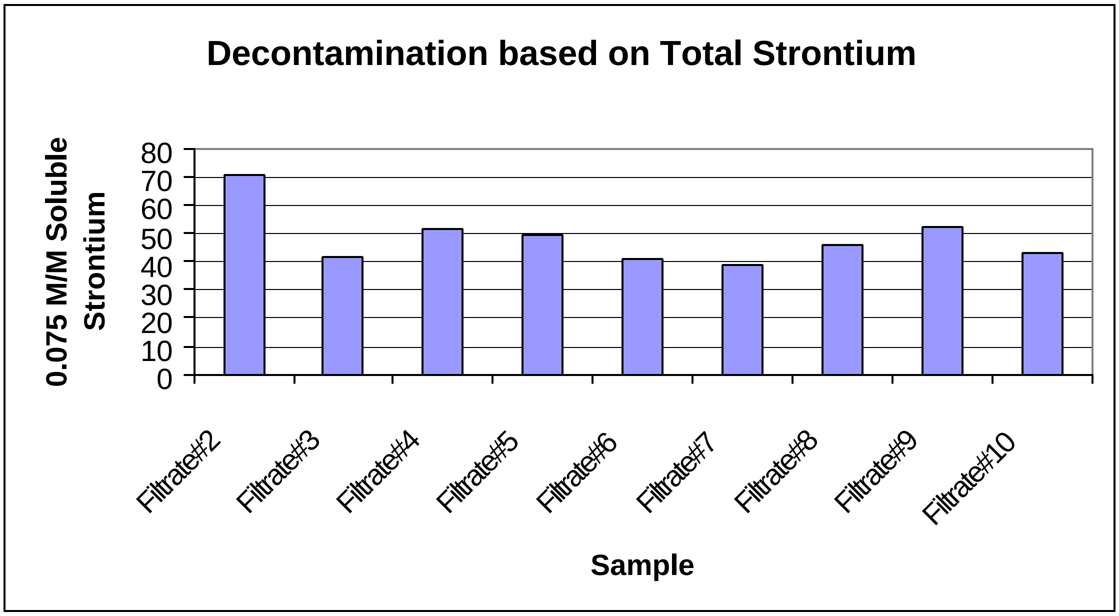


Figure 5 Decontamination based on Total Strontium

If incoming waste contains negligible total strontium on a molar basis and if the saturation level of soluble strontium in the liquid is known, then decontamination can be expressed by the equation below for perfect isotopic dilution.

DF based on total Strontium = (Ret + Sol) / (Sol * MDil)

where Sol = (sample volume + 1 M SrNO3 added volume) * (final total Sr concentration measured in filtrate), and

Ret = Sr retained in precipitate, which is ((1 Molar * 1 M SrNO3 added volume) – Sol), and MDil is the mass dilution ratio.

3.6.3 Decontamination – Transuranics

Figure 6 shows the activities of five transuranic species that were measured. It shows their relative contribution to the total as well. All filtrate product bottles were below the limit of 0.109 µCi/ml except for filtrate #8, which appears to have excess curium-244 contamination. The curium was an artifact of the work and is not present to significant levels in 241-AN-102 waste. Removal of the curium contamination from the total activity would show that the product was well below the limit in all cases. The Large C campaign thus demonstrated robust and ample removal of transuranics from the feed.

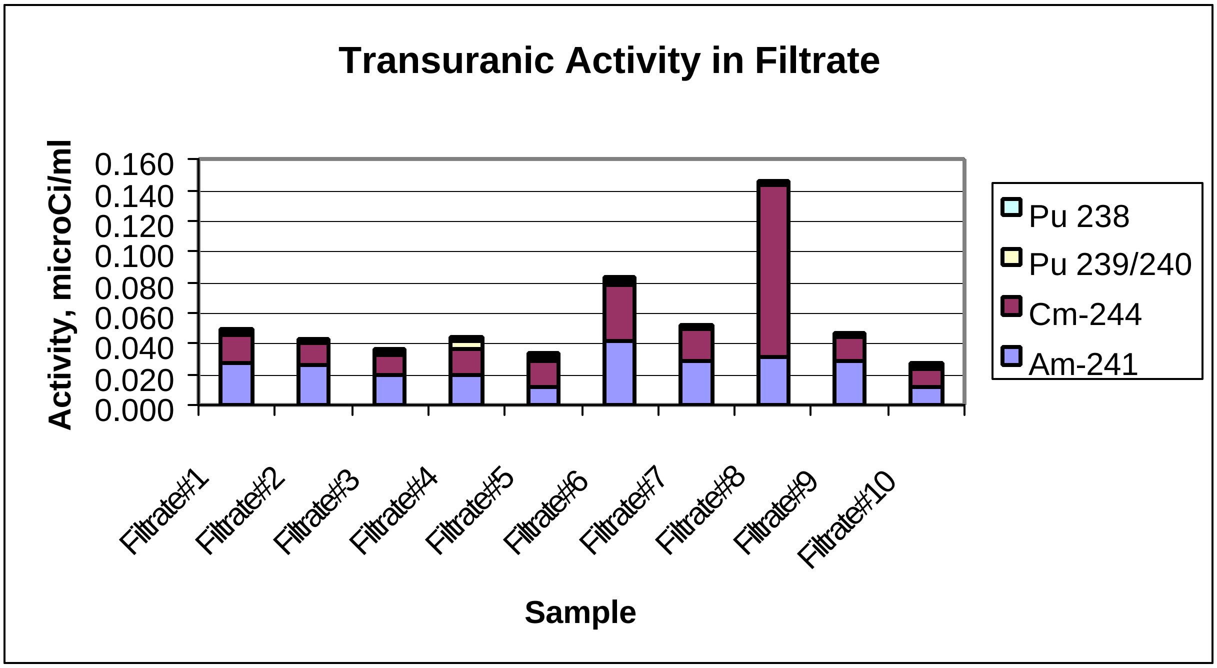


Figure 6 Transuranic Activity in Large C (AN102) Filtrates

Decontamination factor based on 6 M sodium feed is provided in Table 8. "Filtrate" samples are from the 1 or 2-liter composite collection bottles after each had been filled during the Large C filtration campaign. Feed activity levels used in the calculations were as found by Hay.[12] Removal of curium contamination was not calculated because of uncertainties in levels of that element in the input samples. The table shows a simple average and standard deviation for the ten decontaminations with equal weighting, then a volume-based weighted average is shown at the bottom. The volume weighted average was calculated by multiplying each filtrate bottle by its final volume, summing all ten, and dividing by a total product volume of 16.6 liters of filtrate product (wash solutions are excluded).

Table 8 Decontamination Factor for Transuranics in Large C Filtrate Samples

	Am-241	Pu 239/240
Filtrate#1	4.4	5.5
Filtrate#2	4.7	12.4
Filtrate#3	6.3	7.2
Filtrate#4	6.0	3.3
Filtrate#5	10.1	6.4
Filtrate#6	2.9	5.2
Filtrate#7	4.2	10.1
Filtrate#8	3.8	10.7
Filtrate#9	4.2	11.3
Filtrate#10	10.0	8.2
Average	5.7	8.0
Std. Dev.	2.5	3.0
Volume Weighted Average	5.7	8.2

3.6.4 Decontamination – Technetium-99

Technetium is clearly not removed from the waste by this precipitation process. This is a disappointment since technetium is a congener of manganese and might thus be expected to precipitate with it, forming a mixed oxide. Figure 7 shows technetium-99 from available batch (instantaneous) and filtrate (2-liter bottle composite) samples. It was measured by mass spectroscopy, which has been found to be more reliable than counting methods for this sample matrix. The technetium-99 level is consistent over these samples and its average value compares well with that of the feed. Hay reported 6.28 mg/liter +/- 1.4% at 6.42 M sodium. At 6 M sodium that would be 5.85 mg/liter which compares well with the average and standard deviation given on the figure.

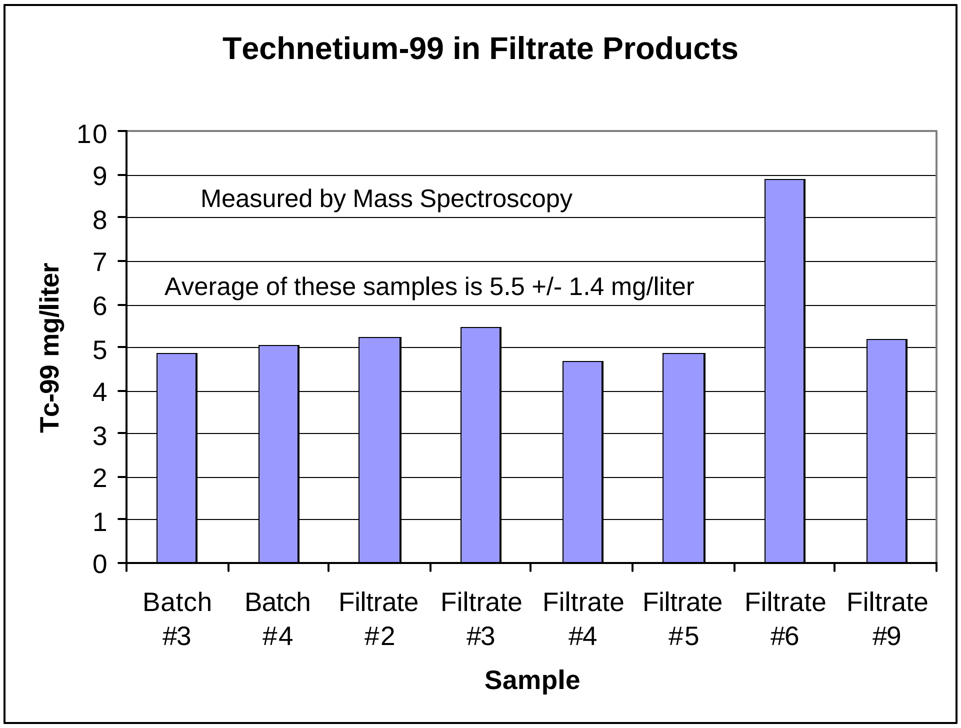


Figure 7 Technetium Levels in Product Filtrate Samples

This finding is confirmed by calculated quantities of the Tc-99 entering and leaving the precipitation process. Input into the experiment was 16.4 L of material at an average 6.06 M Na containing 5.93 mg/L Tc-99 for a total of 97.2 mg Tc-99. Leaving the experiment for ion exchange was nearly 18.9 L composited product (16.6 L filtrate + 2.3 L wash) containing 4.31 mg/L Tc-99 (King[19]) for a total of 81.5 mg Tc-99. An additional 14.4 mg Tc-99 can be accounted for through purged CUF concentrate and samples (approximately 3.2 L) containing an estimated 14 wt% solids and assuming a dilution factor of 1.13 from 5.93 mg/L due to addition of precipitating agents. The total Tc-99 effluent from the CUF was estimated to be 95.9 mg leading to a Tc-99 balance closed to within 1.5%, well within the 5% estimated analytical error. An alternate verification is an unchanged concentration ratio of K and Tc, two species not removed in the precipitation process, in the tank waste and the ion exchange feed material. Hay's feed characterization data[20] yields a K/Tc ratio of 555 (0.0352 M K/0.0000634 M Tc) and King's data gives a ratio of 574 (0.025 M K/0.0000435 M Tc). The 3.4% difference in the calculated ratio is again within the 5% estimated analytical error.

3.6.5 Components Showing Little Removal

The strontium-permanganate process leaves many components in the filtrate product with insignificant removal efficiency. Components like aluminum and sulfate are desired to stay in the filtrate because they are better handled in the Low Level Waste melter as opposed to the much smaller High Level Waste melter. It would be a benefit if other components like cesium and technetium were removed during precipitation, but they are handled by ion exchange columns just downstream of the precipitation process.

Table 9 shows splits of stable elements whose concentrations were measured in both the concentrate and filtrate during the campaign. Concentration ratios near unity show elements that are not separated very well. Strontium and manganese levels are artificially high because of the addition of these elements for precipitation.

It is interesting to note that barium, copper, iron, lead, tin, zirconium, and cadmium to some extent were separated more efficiently in the first batch than in the following six batches. This is despite the lower addition of strontium (0.02 M versus 0.075 M after mixing) and lower permanganate level (0.03 versus 0.05 M addition) for the first batch. Since this batch had added calcium these elemental levels provide evidence of the efficacy of calcium to increase removal of elements targeted by the process. It is fortunate that Batch 1 had two sets of samples so that this effect could be verified.

Table 9 Table of Slurry-Based Decontamination Factors for Metallic Elements

Element	BATCH 1-Mid	BATCH 1-End	BATCH 2	BATCH 3	BATCH 4	BATCH 5	BATCH 7
Al	1.0	1.1	0.3	0.7	1.2	0.6	0.1
B			1.2	0.9	1.1	1.1	1.1
Ba	21.6	25.6	7.6	0.3	5.5	4.8	9.6
Ca	2.4	2.5	2.6	1.5	1.9	1.6	1.8
Cd	1.4	1.6	1.0	0.9	0.8	0.8	0.8
Cr	2.0	2.2	1.8	1.3	1.1	1.5	1.9
Cu	2.6	3.0	0.7	0.4	0.5	0.5	0.5
Fe	48.0	80.1	3.7	2.5	10.2	7.6	5.1
Mg	5.0	5.0	25.4	2.6	49.8	2.5	1.4
Mn	3579.1	1841.5	2384.8	247.6	345.3	705.5	285.1
Mo	1.9	1.8	0.9	0.8	0.7	0.7	0.7
Na	1.0	1.1	1.0	1.0	0.9	0.9	0.7
Ni	1.2	1.4	1.3	0.9	0.9	1.0	0.9
P	1.2	1.2	1.1	0.9	0.8	0.9	0.8
Pb	4.1	5.7	1.9	0.7	1.1	1.1	1.2
Sn	4.6	4.2	0.2	0.4	0.6	0.4	0.2
Sr	35.0	34.8	108.8	8.0	38.9	36.2	49.5
Tc	bd	bd	0.5	0.5	bd	bd	bd
Zn	3.3	2.4	3.0	0.6	4.1	3.8	6.2
Zr	15.0	18.6	0.0	0.2	0.0	0.0	0.1

bd = below detection

Table 9 data from Batches 2 to 7 were averaged because they all used the same precipitation recipe. Results are tabulated in Table 10 for the leftmost data column under “This Work”. The results of other works are also shown for comparison where data are available. The other works referenced are as follows:

241-AN-102 Small C – Reference 4
Simulant 241-AN-107 Cold CUF – Reference 2
241-AN-107 PNNL – Reference 3

These works were compared partly to draw general conclusions on precipitation process performance across the variation represented by tank 241-AN-102 and tank 241-AN-107 materials. Elements not removed in either case are, for example, easily modeled in the process flowsheet without further effort.

Table 10 Elemental Split across the Filter - ICP-ES Data, Several Works

Sample:	AN-102 (This Work)			AN-102 Small C Ref. 4		Sim. AN-107 Cold CUF Ref. 2	AN-107 PNNL Ref. 3
	Average	St Dev					
Al	0.6	0.37		0.9		0.9	1.1
B	1.1	0.09		1.0			
Ba	5.5	3.11					
Ca	1.9	0.40		1.5		2.4	1.6
Cd	0.9	0.09		1.0			1.0
Cr	1.5	0.30		1.4			2.0
Cu	0.5	0.12		1.0		>10	
Fe	5.8	2.76		8.7		5.3	100
Mg	16.3	19.00					
Mo	0.8	0.09		0.9			
Na	0.9	0.10		1		1.0	1.0
Ni	1.0	0.16		1		1.0	1.0
P	0.9	0.11		0.9		1.1	1.0
Pb	1.2	0.41		1.4			2.5
Sn	0.4	0.16		0.4			
Sr	48.3	33.21		39.3		91	120
Tc	0.5						
Zn	3.5	1.81		2.4		1.3	
Zr	0.1	0.05		0.3			
Added Mn, M	0.05			0.044		0.04	0.05
Added Sr, M	0.075			0.066		0.075	0.075

Aluminum, boron, cadmium, molybdenum, sodium, nickel, and phosphorus are clearly not removed by this process and remain in the filtrate whether the feed is from tank 241-AN-102 or from tank 241-AN-107. Approximately half of calcium, chromium, lead, zinc are removed. The process always has a strong interaction with iron and removes at least 80 % of it.

Decontamination of strontium based on elemental totals compares well with decontaminations based on Sr-90 activity. The data from this set of samples thus supports the data shown in the Strontium Decontamination section which used total and Sr-90 data from other sample sets. This equality indicates that isotopic mixing in the process is complete. While this affirms the goal of activity reduction by isotopic dilution it also gives some credibility to the use of nonradioactive simulants to report a strontium decontamination where only data from total strontium measurements is available.

3.6.6 Analyses of Final Filtrate Products

All 6 M sodium and slurry wash filtrates were composited for the sake of delivery to the cesium ion exchange process. Table 11 shows how the composite greatly exceeds the decontamination for both strontium-90 and transuranics. An analysis of this grand composite is shown in Appendix C. The material composition is pretty much as expected, and decontamination numbers from this data set show the material met decontamination requirements.

Table 11 Radiochemical Data for Final Product Composite

ADS# 145153			
Radiochem	Dilution-Corrected		
	dpm/mL	µCi/ml	Limit for 4.8 M Na+
Sr-90	2.03E+06	0.9144	6.7 µCi/ml
	dpm/mL	ηCi /ml	
Pu-238	2.06E+03	0.9	
Pu-239-240	1.64E+03	0.7	
Am-241	2.31E+04	10.4	Transuranic Total must be
Cm-244	2.51E+04	11.3	Less than 87.6 ηCi /ml
Cs-137	3.34E+08	150.5 µCi/ml	
Cs-137 (µCi/ml)	150.52		

3.6.7 Chemical Analysis of Slurry Washing

The slurry concentrate in the CUF at the end of Batch #7 filtration was washed with inhibited water (0.01 M NaOH). Four 600-ml portions of washwater were used to generate four 600-ml samples of spent washwater. Slurry being washed was left in the rig between each 600-ml wash and was only removed at the end. Data from the four washwater analyses are presented below to show impacts of the washing sequence on chemistry and radioactive elements.

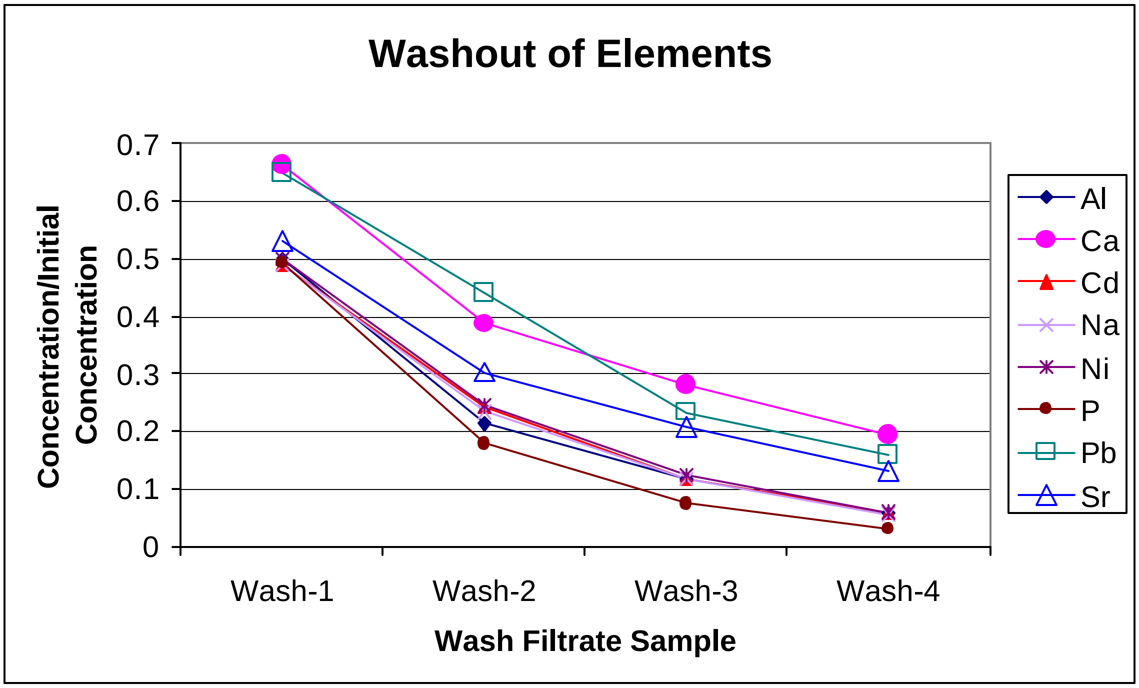


Figure 8 Washout of Elements

Elements that could be detected by ICP-ES in all four of the 600-ml “Wash-1” to “Wash-4” samples are shown in Figure 8. Two types of washout behaviors are clearly shown. These are separated in the figure by use of large versus smaller plot symbols.

Sodium, nickel, phosphorus, and cadmium appear to wash out by a dilution effect only, as if they have insignificant association with the solids in the slurry. These are shown with small plot symbols in Figure 8. The filtration rig inventory volume is estimated to be 800 ml, but the 600 ml of washwater passed through it for each “Wash-#” sample appears to reduce soluble concentration by half. This higher efficiency would be expected because the washwater was pumped in semi-continuously rather than all at once – the rig would not have been able to hold all 600 ml in a one-time addition. The permanganate process in all previous work did not remove these elements.

Calcium, strontium, and lead washed out with apparent bleed from the slurry solids. These are plotted with the larger symbols. The curves are higher than those of purely soluble elements, indicating that they are bleeding from the solids to some extent. Strontium is an important part of the slurry and would have this expected behavior. Calcium, as well, associates in some way with the manganese oxide-hydroxide solids in the slurry and thus washes out slowly. Lead has a removal factor of slightly above unity and is removed by the caustic wash solution as shown.

Manganese was below detection in the Wash-3 and -4 samples and was only 10 mg/l and 1.6 mg/l in the Wash-1 and Wash-2 samples, respectively. The form of manganese in the slurry was thus insoluble with respect to the washing operation. It did not bleed out significantly.

Figure 9 shows that sodium and Cs-137 wash out as totally-soluble components. These are plotted with heavy lines and both follow the same trend of halved concentration with each 600-ml wash. Americium-241, the most prevalent source of transuranic activity, is retained by the slurry solids and shows no significant bleed.

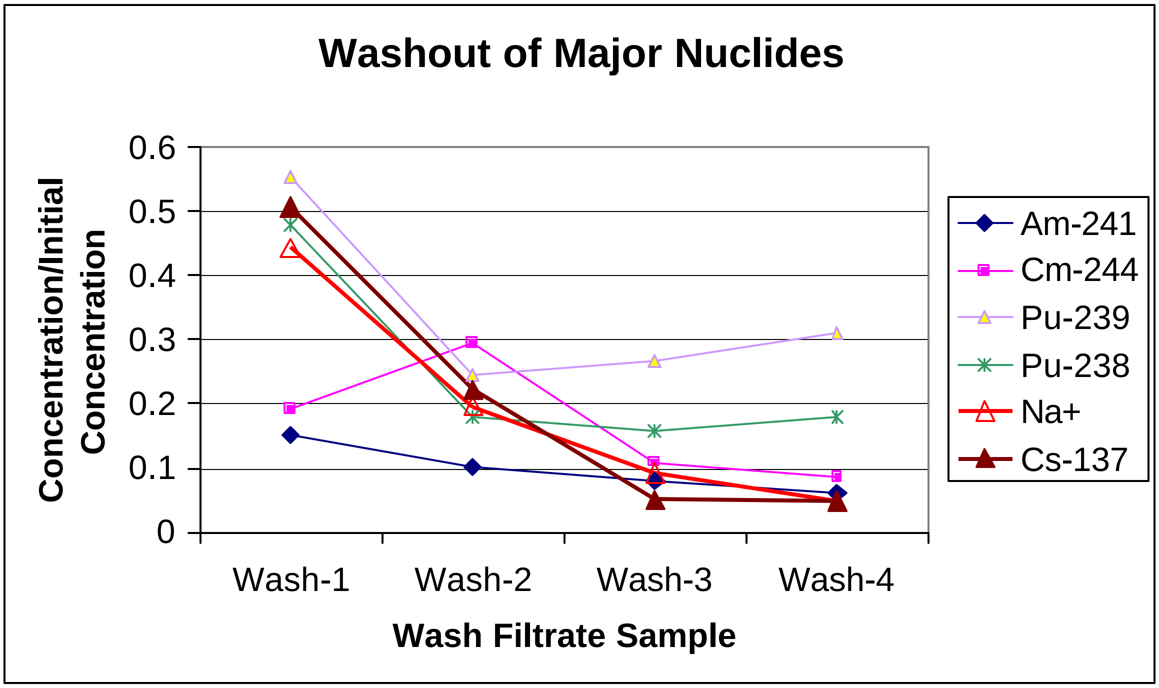


Figure 9 Washout of Major Nuclides

Plutonium behavior initially follows the trend of diluted soluble elements, but then seems to begin to wash out of the solids in the latter two washes. This is seen by its departure from the normal downward trend in Washes 3 and 4. Sodium and cesium follow a downward trend where concentrations are roughly halved with each new wash. The lower ionic strength of the slurry might have promoted this bleeding for plutonium. It is fortunate that plutonium contributes no more than about 10% of the total transuranic activity; the permanganate process thus retains the most important element (Am-241) during washing. Curium-244 behavior is uncertain at the first data point, but follows the soluble components beyond the first data point (average of two measurements). It shows little evidence of accelerated bleeding during slurry washing. This curium is not a normal part of complexant waste and contributes an insignificant amount of transuranic activity. Curium-244 has been found in higher levels alone in the sample used here due to inadvertent contamination of the 241-AN-102 sample within the SRTC hot cells.

Table 12 shows the absolute values of the product filtrate used to plot the two preceding dimensionless graphs. In all cases the measured values from filtrate sample bottles #8, #9, and #10 were averaged and the standard deviation of each initial value is presented as a percentage of the average value. The averaging was done so that an error could be calculated from experimental values. Bottles 8 to 10 were used because these were the last filtrate bottles, when the process using concentrated slurry would be running closest to steady state.

Table 12 Average Initial Concentration of Filtrate Product, Last Three Bottles

	Avg, mg/l	**St. Dev., %**			**Activity**	**Units**	**St. Dev., %**
Al	6898.0	5.6		Am-241	0.0105	µCi/ml	44.1
B	17.8	4.0		Cm-244	0.0048	µCi/ml	29.1
Ca	151.7	3.3		Pu-239	20.6	dpm/ml	18.1
Cd	31.6	2.3		Pu-238	9.7	dpm/ml	11.8
Co	3.1	29.4		Cs-137	10.6	µCi/ml	5.7
Cr	104.0	7.4					
Cu	12.8	3.3					
Fe	3.2	22.7					
La	3.8	23.6					
Mn	4.0	19.4					
Mo	30.8	0.6					
Na	141971.9	2.8					
Ni	207.6	2.3					
P	924.6	4.2					
Pb	82.7	5.6					
Si	12.3	63.1					
Sn	20.7	11.7					
Sr	141.0	9.9					

3.6.8 Filter Cleaning – Flush Solution Compositions

This section presents compositions of the flush solutions after they were used to clean the rig and crossflow filter. The cleaning protocol used water and 1 M nitric acid.

After the precipitate filtration/washing series the rig was drained of slurry. Several difficulties were encountered. The washed slurry product did not drain by gravity when the drain valve was opened. The valve was closed and it was then found that the slurry pump was hard to start. Pressure applied to the air motor was cycled up and down quickly and the pump was found to start when the air pressure was spiked to 110 psig, the maximum available pressure. 300 ml of slurry (less than half of the expected rig volume) was drained with the pump operating at slow speed. That volume was stored in its own sample bottle. This volume was replaced with 300 ml of inhibited water. It was circulated at 3.5 gpm (13.25 liters/min) for 10 minutes. This water drained slowly and appeared to contain a lot of slurry solids when it was drained. Only 300 ml drained again. This was replaced with 300 ml of inhibited water and the process repeated. This time 600 ml of inventory was drained and the rig was thus considered ready for a cleaning operation. Work up to this point was considered to be slurry removal.

The rig was charged with 700 ml of inhibited water. It was circulated for 30 minutes at 4 gpm (15 liters/min). There was no backpulsing. This liquid was drained and a sample named “Waterflush” was taken. The liquid was still black, but appeared to have water-like consistency.

The rig was then flushed with a full inventory of 1 M nitric acid for a total of one hour. The filter was backpulsed and fluxes were measured. This acid flush still contained visible black solids. The liquid was then drained and stored as Sample “1 M Nitric” flush. The rig was charged with an inventory of deionized water. This was left to sit overnight. This flush water wash was circulated for an hour the next day, and sampled. This was called the “Final Water” sample.

Figure 10 shows elemental compositions found after the named flush samples were acid digested and diluted to one thirtieth of their as-withdrawn concentration. The figure shows that the nitric acid flush had significant effect; acid concentrations of elements were not far below values found in the initial water flush and were significantly higher than those of the water that resided in the rig overnight.

Figure 11 shows that the nitric acid flush again dissolved many of the radioactive elements that were detected. Their level descends more quickly after the use of the acid. The only exception here was Tc-99, which seemed to bleed more uniformly into all three flush samples. The holdup mechanism for Tc-99 is unknown. Both sodium from the Figure 10 and Cs-137 here show a peak during the acid use. To get correct absolute values of activities from Figure 11 it is necessary to use the scale factors in the legend for two of the elements. The value of using them was the ability to note trends for all of the active elements on one figure.

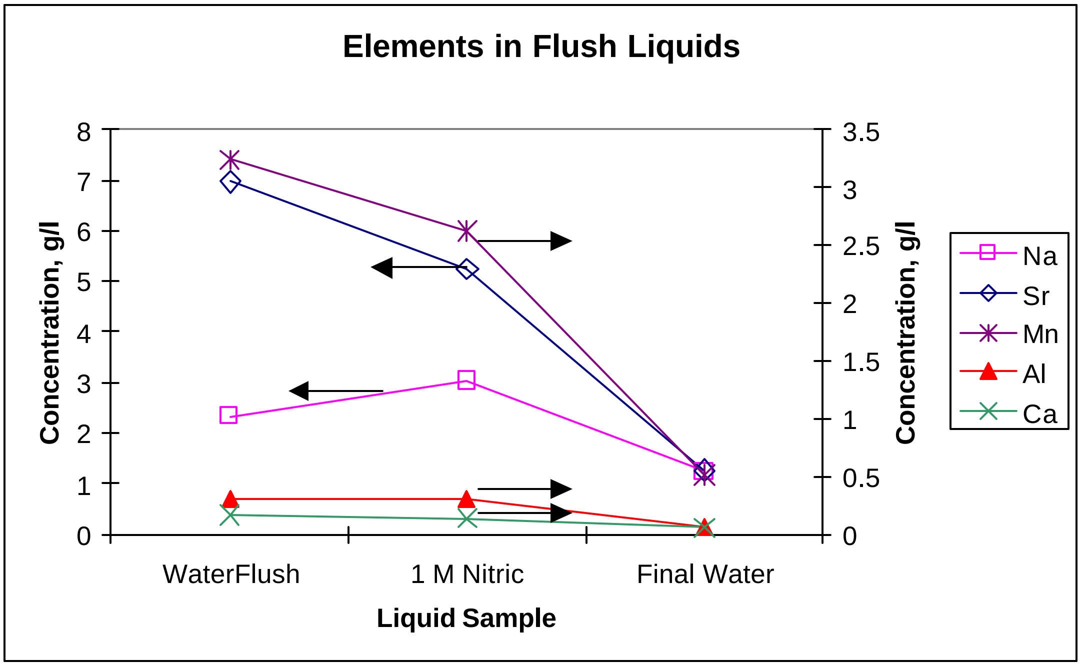


Figure 10 Elements Found in Flush Solutions

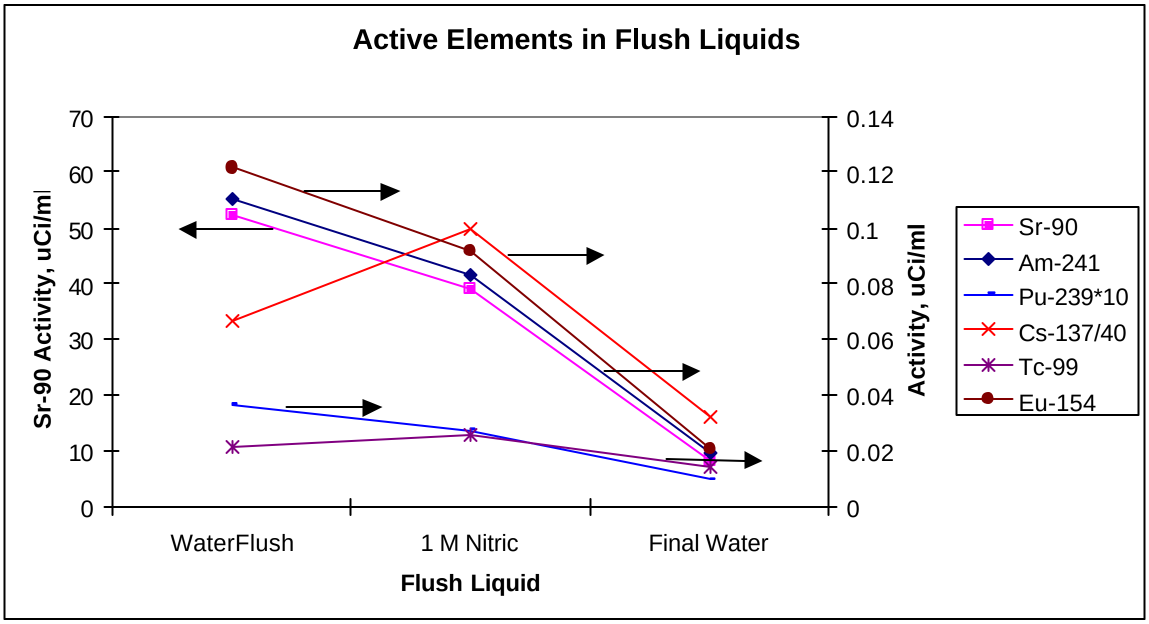


Figure 11 Active Elements in Flush Liquids

The trends noted above depend on the geometry of the filter loop, the number of deadlegs, tank heels, and internal surface per volume. The relative amount of internal surface is high for the small cells filter rig used here, and this would affect the amount of highly concentrated slurry stuck inside after the system is drained.

3.6.9 Final Compositions of Major Products of the Campaign

This campaign produced two major liquids of great significance to the RPP flowsheet – a composite of all filtrate plus washwater, and a washed strontium-manganese slurry. While many samples were taken and the data presented above, the analysis of the final products sent to other unit operations is included in this report for completeness.

The grand composite for the filtrate and spent wash is in Appendix C. The sodium level as measured was 4.82 M, addition of wash filtrate being the main reason for the dilution. The Sr-90 activity was over 7 times lower than the limit for this sodium level, and transuranic activity was 4 times below that limit. These great conservatisms for this large campaign show that the process is extremely robust and that good reductions in both strontium nitrate and sodium permanganate reagents can be tolerated for processing 241-AN-102 complexant waste. The inclusion of the washwater in the composite provided the benefit of lowering the sodium molarity for the sake of ion exchange – resin may float if the molarity is too high.

The washed slurry analysis in given in Appendix C. It is mainly strontium and manganese–bearing solids in a solution bearing residual sodium.

3.6.10 Observations on Post Precipitation

Various researchers have noted that the filtrate from permanganate precipitation appears to drop small amounts of black solid on standing. While no systematic study of this has been published, it was planned to watch the bottles of filtrate product from this campaign for such post-filtration precipitation solids.

In summary, some bottle discoloration, but no solids, was seen with the bottles of high sodium filtrate product. Free solid formation was seen with the lower sodium filtrate from washing. All Filtrate bottles #1 to #10 were checked after sitting in the shielded cells for two weeks after they were produced. No solids were seen in the bottles, but some discoloration of the polyethylene that was in contact with the solution in each bottle was observed. The filtrates were clear and straw-colored, while the discoloration was gray-black. Bottles containing filtrate from the first two of four washes formed visible solids within two days of standing. It was noted that the solutions in the bottles were light brown-yellow and clear. No post-filtration precipitation solids were captured or analyzed in this work.

3.7 FILTRATION RESULTS

3.7.1 Filtration Flux Data Summary

The table below summarizes the average filter fluxes obtained for each batch of precipitated Hanford AN-102 Large C waste, filter conditions and settings, and insoluble solids concentration average and range. The average flux and insoluble solids concentrations were calculated by integral average over the volume of filtrate produced.

Table 13. Summary of CUF Flux Data

Batch #	Precip. Chem	TMP	Axial Veloc.	Avg. Flux	Measurement Time (min)	Avg. IS (wt%)	Insol. Solids (IS) range (wt%)
1	Ca/Sr/Mn	**70 psig 4.8 bar**	**13.5 ft/s 4.1 m/s**	**0.074 gpm/ft^2 4.3 m^3/(m^{2}day)**	60	1.3	1.3
2	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.045 gpm/ft^2 2.7 m^3/(m^{2}day)	73	3.9	1.8 – 6.8
3	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.034 gpm/ft^2 2.0 m^3/(m^{2}day)	23	7.3	6.8 - 8.2
	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.030 gpm/ft^2 1.8 m^3/(m^{2}day)	59	8.2	7.1 – 9.4
4	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.021 gpm/ft^2 1.2 m^3/(m^{2}day)	110	11.0	9.4 – 13.0
	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.022 gpm/ft^2 1.3 m^3/(m^{2}day)	30	13.5	13.0 – 13.9
	Sr/Mn	**30 psig 2.1 bar**	**15.2 ft/s 4.6 m/s**	**0.016 gpm/ft^2 0.94 m^3/(m^2 day)**	30	14.5	14.5
5	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.023 gpm/ft^2 1.3 m^3/(m^{2}day)	48	4.3	3.6 – 5.0
	Sr/Mn	30 psig 2.1 bar	15.2 ft/s 4.6 m/s	0.025 gpm/ft^2 1.5 m^3/(m^{2}day)	30	5.5	5.0 – 6.0
	Sr/Mn	40 psig 2.8 bar	15.2 ft/s 4.6 m/s	0.027 gpm/ft^2 1.6 m^3/(m^{2}day)	43	6.8	6.0 – 7.7
6	Sr/Mn	30 psig 2.1 bar	9.1 ft/s 2.8 m/s	0.015 gpm/ft^2 0.9 m^3/(m^{2}day)	33	8.1	7.7 – 8.5
	Sr/Mn	30 psig 2.1 bar	15.2 ft/s 4.6 m/s	0.022 gpm/ft^2 1.3 m^3/(m^{2}day)	15	8.9	8.6 – 9.1
	Sr/Mn	50 psig 3.5 bar	12.2 ft/s 3.7 m/s	0.018 gpm/ft^2 1.1 m^3/(m^{2}day)	29	9.5	9.1 – 9.9
7	Sr/Mn	25 psig 1.7 bar	15.2 ft/s 4.6 m/s	0.021 gpm/ft^2 1.2 m^3/(m^{2}day)	30	10.3	9.9-10.6
	Sr/Mn	30 psig 2.1 bar	15.2 ft/s 4.6 m/s	0.023 gpm/ft^2 1.3 m^3/(m^{2}day)	30	11.0	10.6 – 11.4
	Sr/Mn	30 psig 2.1 bar	15.2 ft/s 4.6 m/s	0.019 gpm/ft^2 1.1 m^3/(m^{2}day)	156	13.1	11.4 - 14.7
	Sr/Mn	30 psig 2.1 bar	15.2 ft/s 4.6 m/s	0.015 gpm/ft^2 0.9 m^3/(m^{2}day)	34	14.7	14.5 – 15.0

Bold signifies maximum flux observed during test matrix

The remaining filtration discussion focuses on comparing specific conditions (e.g., different transmembrane pressures (TMP) and fluid axial velocity at constant insoluble solids contents, different insoluble solids contents for the same TMP and axial velocity, etc.) summarized in Table 13 above.

3.7.2 Pre- and Post-Run Clean Water Fluxes

Figures 12 and 13 show the permeate fluxes and permeances, respectively, of the Cells Unit Filter (CUF) before and after filtration of approximately 19.8 L of precipitated Large C (AN-102) feed material.

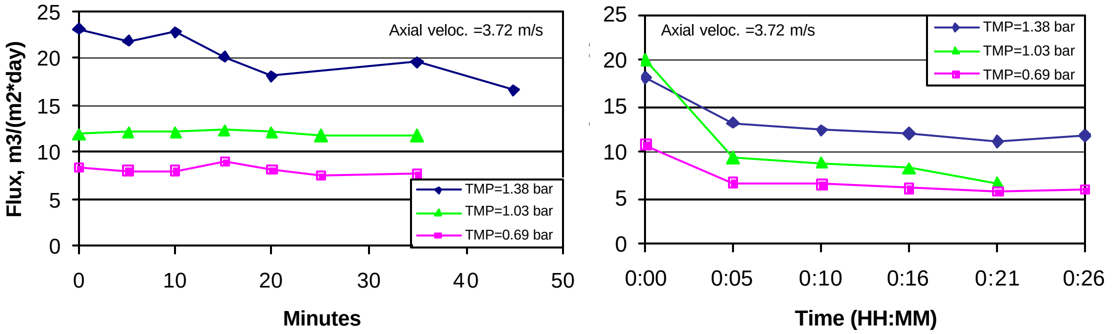


Figure 12 Clean water fluxes of the CUF from the filtration of deionized water before and after precipitated Large C filtration.

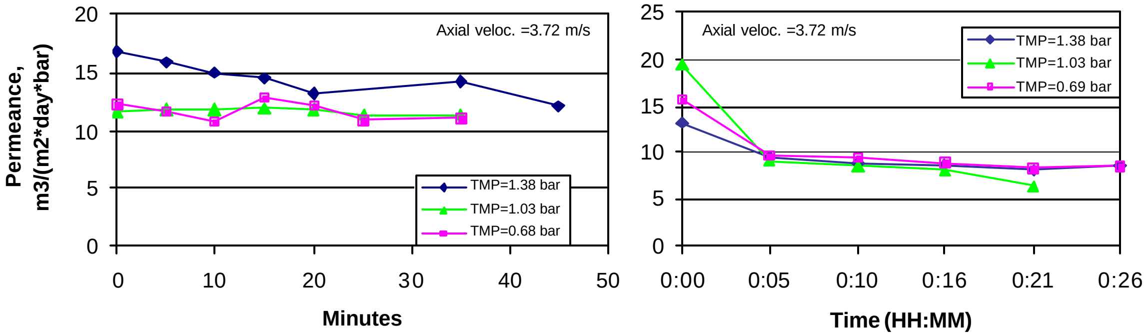


Figure 13 Clean water permeances of the CUF from the filtration of deionized water before and after precipitated Large C filtration.

Of the 19.8 L, approximately 16.4 L was an average 6.06 M Na Large C Envelope feed material. The axial fluid velocity for all clean water data was nominally 3.7 m/s (12 ft/sec), and transmembrane pressures (TMP) between 0.69 and 1.38 bar (10 and 20 psi) were tested. The pre-run clean water fluxes are of order half those seen for the small Envelope C precipitation and filtration study[21] performed with this equipment just prior, likely caused by some filter pore obstruction as a result of filtering entrained solids from an unprecipitated Small C AN-102 sample.

Figures 12 and 13 suggest an approximately 25-35% deterioration in the steady state filter performance as a result of the filtration of Hanford Large Envelope C waste. However, the comparable initial fluxes obtained in both cases after backpulsing the cross-flow filter indicates that the filtration of precipitated Large C did not permanently foul the 0.1 μm Mott filter through additional pore occlusion. As a result, no adjustment of the permeate flux and permeance data due to gradually deteriorating filter performance was deemed necessary. However, the rapid degradation of both permeate flux and permeance in the post-run data soon after backpulsing of the CUF does suggest that the post-run cleaning process used does leave some solids in the CUF, likely disturbed from accumulated material in "dead" spots expected in any experimental apparatus.

3.7.3 Ca Precipitation Data

To further assess the filterability of precipitate from the calcium-strontium-permanganate chemistry, a 1.2 L "small" batch of Large C (AN-102) was initially precipitated and filtered in the CUF immediately after emptying water from the initial clean water flux measurements. Total solids and insoluble solids contents for this precipitated solution were 35 wt.% and 1.29 wt.%, respectively. Without initially producing any filtrate, a test matrix[22] varying transmembrane pressure (2.06-4.83 bar, 30-70 psi) and axial velocity (2.77-5.03 m/s, 9-16 ft/s) was run, with the produced filtrate being recycled back to the CUF reservoir ("recycle" mode). Figure 14 shows all of the data in the order taken.

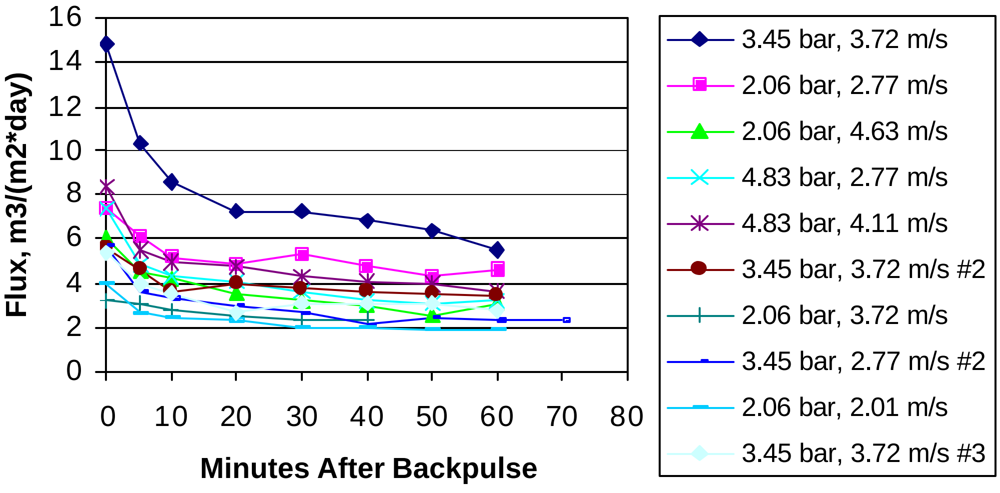


Figure 14 Permeate flux data from the Ca/Sr nitrate and permanganate precipitated solution filtration.

Comparison of fluxes from the first two conditions (3.45 bar and 3.72 m/s, 2.06 bar and 2.77 m/s) against identical or similar conditions subsequently run suggests that those fluxes are inordinately high due to the time necessary to build up a layer of solids onto the Mott filter. Excluding these two data sets from the analysis, the expected trends were obtained with fluxes increasing with both TMP (Figure 15) and axial velocity (Figure 16). Filter permeance is often used to measure power usage efficiency of the filtration process. Figure 17 shows that low TMP (2.06 bar, 30 psi) and high axial velocity (4.63 m/s, 15 ft/s) was the most favorable of the conditions studied.

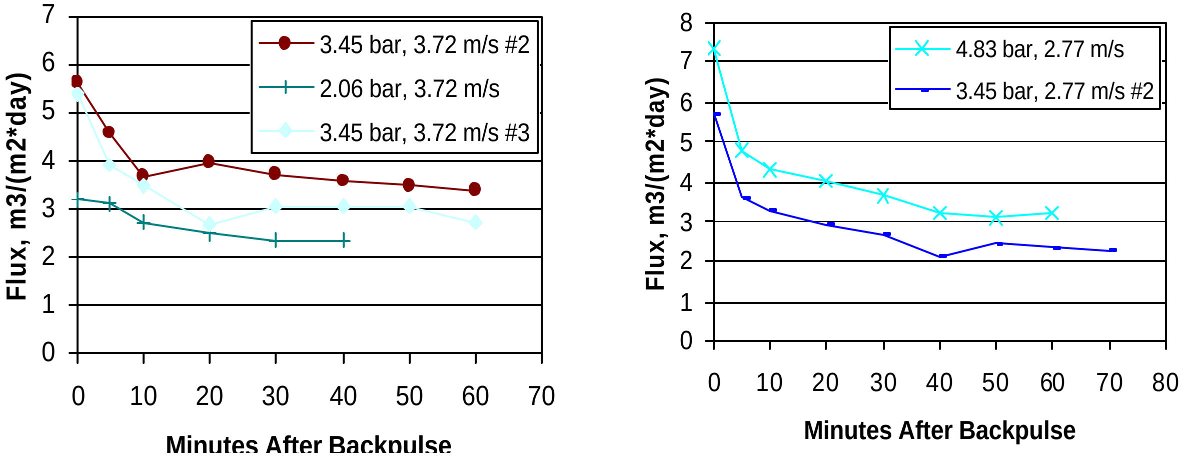


Figure 15 Permeate flux data from Ca/Sr nitrate and permanganate precipitate filtration as affected by filter transmembrane pressure.

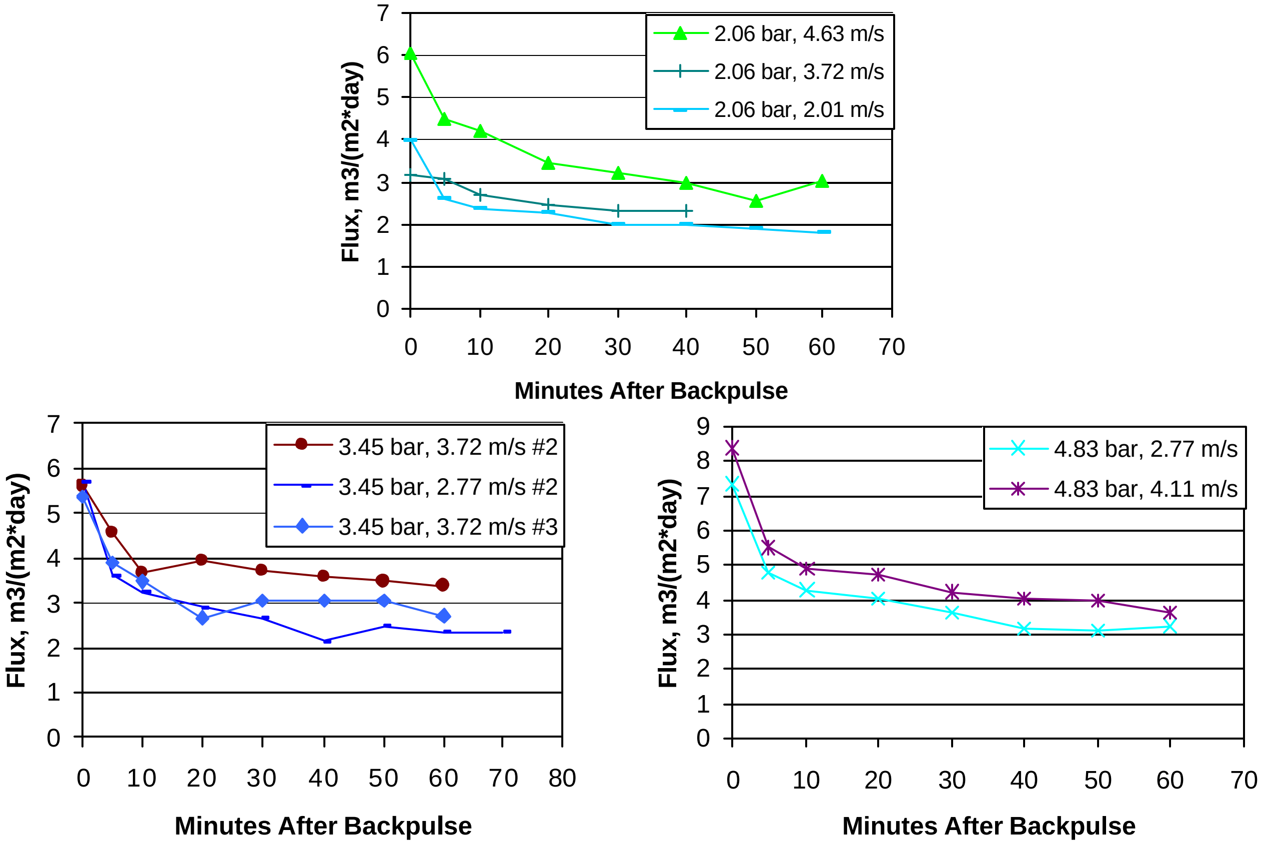


Figure 16 Permeate flux data from Ca/Sr nitrate and permanganate precipitate filtration as affected by filter axial velocity.

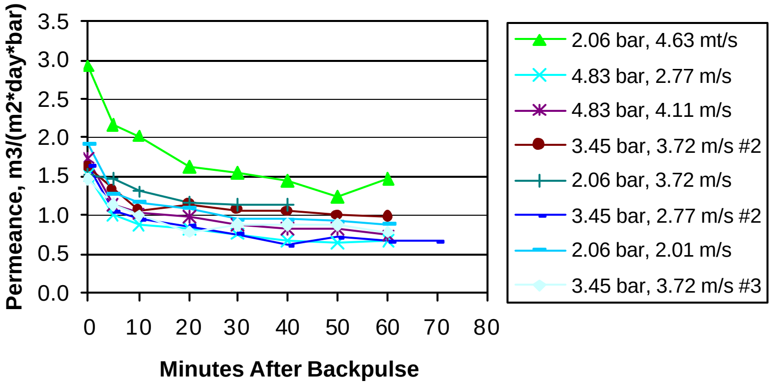


Figure 17 Permeance data from the Ca/Sr nitrate and permanganate precipitated solution filtration.

When compared to previous work at SRTC, the steady state fluxes obtained are comparable to the approximate 1.77 to 5.87 $m^3/(m^2*day)$ [0.03 to 0.1 gpm/ft^2] range obtained in the small C AN-102 study[23] where the strontium nitrate/sodium permanganate chemistry with no calcium was used in the exact same filtering equipment used in this work. Very similar steady state permeate flux (~2.05-4.40 $m^3/(m^2*day)$, ~0.035-0.075 gpm/ft^2) and permeance (~0.43-1.87 $m^3/(m^2*day*bar)$, ~0.0005 – 0.0022 $gpm/(ft^2*psi)$) ranges were obtained in the AN-107 simulant pilot-scale studies containing ~2 wt% insoluble solids performed at the Thermal Fluids Laboratory[24]. These steady state permeate fluxes are 3-4 times higher at the same operating conditions than those obtained immediately after backpulse when approximately 2.5 times more Ca and 30% more permanganate were added to a AN-107 simulant, and a 0.2 µm nominal pore diameter 1.27 cm (0.5 inch) ID Mott filter was used[25]. Investigators at PNNL cross-flow filtered an actual AN-107 sample precipitated at slightly higher pH (free OH^- raised by 1 M, rather than 0.875 M here) and demonstrated near steady-state permeate fluxes of order 4.7 to 5.9 $m^3/(m^2*day)$ [0.08 to 0.1 gpm/ft^2], although filter pore size of their filtration equipment was not reported[26].

3.7.4 High Insoluble Solids Concentration Flux Data

The only other portion of the filtration experiment where the produced filtrate was “recycled” back to the CUF reservoir was for the collection of permeate flux data under high insoluble solids loads. The purpose of these runs was to test the effect of changing conditions around the “standard” operating conditions of TMP = 3.45 bar (50 psi) and 3.72 m/s (12 ft/s) axial velocity. To produce a fluid with high insoluble solids content, freshly prepared feed material was semi-continuously pumped into the CUF reservoir to produce filtrate without concentrate being removed from the system, except for small samples. This process was carried out from mid-way through Batch #3 (about 60% feed remaining) to the end of Batch #4 to produce a fluid with a measured 45.4 wt.% total solids and 13.9 wt.% insoluble solids.

Upon comparison of the matrix operating conditions with the flux at "standard" conditions obtained just prior (Figure 18), raising the TMP from 3.45 to 4.83 bar (50 to 70 psi) has no effect on permeate flux and lowering the axial velocity only reduced the permeate production. Permeate flux and permeance increase when both the TMP is lowered to 2.06 bar (30 psi) and the axial velocity is increased from 3.72 to 4.63 m/s (12 to 15 ft/s).

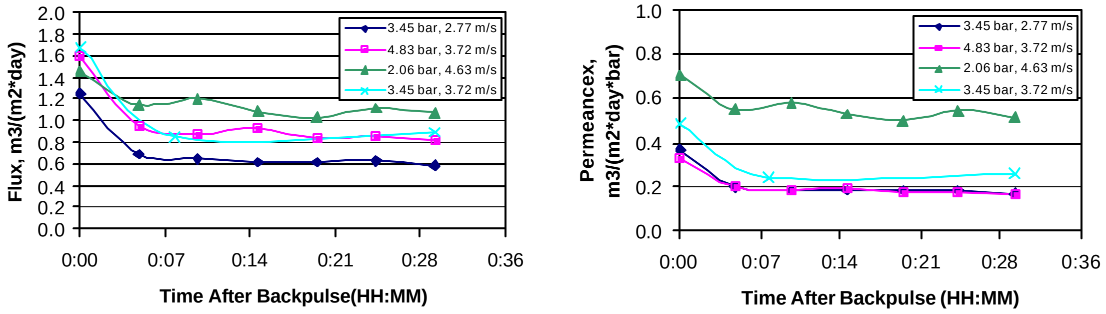


Figure 18 Permeate flux and permeance from filtration of 13.9 wt.% insoluble solids fluid after successive $Sr(NO_3)_2/NaMnO_4$ precipitations.

The permeate fluxes and permeances obtained experimentally are almost identical to those obtained for a 14 wt.% insoluble solids concentrate of precipitated AN-107 C-simulant filtered at TMP=2.06 bar (30 psi), 4.63 m/s (15 ft/sec) and TMP=3.45 bar (50 psi), 3.72 m/s (12 ft/sec) in the Thermal Fluids Laboratory[27].

3.7.5 Insoluble Solids Content and Effect of Backpulse

As expected (Figures 13-17), the filterability and the effect of the backpulse can be lessened as solids concentration rises. Some of these changes in filter performance and optimal filter operations observed were subtle. At low insoluble solids concentration, the backpulse can approximately double the initial permeate flux and permeance over the steady state values, independent of filter operating conditions. At high insoluble solids concentrations, the doubling effect is seen only for the lowest fluxes and permeances (Figure 18). At the "best" conditions (TMP=2.06 bar [30 psi] and 4.63 m/s [15 ft/s]), the backpulse effect is lessened to ~40-50% improvement, likely due to more rapid solids buildup on the filter at higher solids loading or thick higher consistency slurry being less easily removed from the filter surface.

As discussed earlier, "optimal" filtrate or permeate fluxes for low insoluble solids loadings were observed with high transmembrane pressure (TMP) and high axial velocity. However for high solids content, low TMP and high axial velocity showed the best permeate fluxes. In the case of filter permeance, low TMP and high axial velocity yielded the best values at both low and high solids concentration.

The results suggest two possible filtration strategies when concentrating insoluble solids: 1) initially begin with high TMP and high axial velocity, lowering TMP as solids concentration increases, or 2) filter continuously at low TMP and high axial velocity over the entire solids concentration range while accepting a sacrifice in permeate flux at lower solids contents.

3.7.6 Product Data Comparisons

Aside from the two periods discussed above where filtrate was recycled back to the CUF reservoir for low Ca/Sr/MnO_4 solids and Sr/MnO_4 high solids data, the CUF was almost always being run in “production” mode where permeate was being produced and moved to 2 L polyethylene filtrate bottles. There were long production periods where the same identical run conditions were used, allowing for an assessment of the change of filterability over a range of insoluble solids contents. Best examples are throughout the filtration of Batches 7 (~10-15 wt.% insoluble solids, 2.06 bar [30 psi], 4.63 m/s [15 ft/s]), 4 (~10-14 wt.% solids, 3.45 bar [50 psi], 3.72 m/s [12 ft/s]), and 2 (~2-7 wt.% insoluble solids, 3.45 bar [50 psi], 3.72 m/s [12 ft/s]).

Insoluble solids data was taken only for end-of-batch samples, and hence the insoluble solids content for much of the data was calculated. The insoluble solids were estimated by using the end-of-batch measurements and back-calculating by subtracting solids fed as precipitated AN-102 at the prior flux measurement. It was assumed that the volume of precipitated AN-102 fed equaled the volume of filtrate produced, and the volume of filtrate produced was obtained by trapezoid-rule integration between filtrate flux data points. Precipitated AN-102 feed solids content was estimated based on 2.4 L AN-102 material becoming 2.95 L of precipitated feed containing 1.5 wt.% solids, as was obtained in earlier studies. The quantity of solids formed was assumed constant in cases where the volume of AN-102 added to the precipitation recipe was larger than 2.4 L. Solids fed as precipitated AN-102 were estimated by multiplying volume of filtrate produced and the estimated precipitated feed insoluble solids content. An assumed CUF internal volume of 800 ml (estimated volume observed from repeated emptying and refilling of the CUF during the experiment), a precipitated AN-102 feed density of 1.26 g/ml (based on Na molarity and simulant data), and a 15-second period of filtrate production loss to fill empty CUF tubing during each flux measurement were also assumed in the calculation.

To illustrate the efficacy of this estimation method, the end of batch calculated insoluble solids contents were compared to analytically obtained results for Batches 1 and 6. Using end of batch measured insoluble solids concentrations of 6.8 and 15.1 wt%. for Batches 2 and 7, respectively, the calculated end of batch estimates of 1.1 and 9.5 wt%, respectively, compared favorably with the analytically obtained values of 1.8 and 9.9 wt%, respectively. The calculated estimates are thought to be low due to the expected filtrate volume over-estimation by trapezoid rule integration as well as the rough nature of the 1.5 wt% precipitated feed solids estimate, although there is also some uncertainty in the end and beginning of run insoluble solids analytical measurements.

Table 14. Measured vs. Calculated Insoluble Solids Content for CUF Concentrate (End of Precipitation Batch)

Batch #	End of Batch Insoluble Solids Content (wt%)	
	Measured	Calculated Backward
1	1.8	1.1
2	6.8	(HIGHCONC1*)
3	-----	9.4, 10.0
4	13.9, 14.5	(HIGHCONC2*)
5	7.7	(HIGHCONC3*)
6	9.9	9.5
7	15.1	-----
*Could not calculate due to uncertain quantities before draining CUF into bottle HIGHCONC*x*		

In the filtration of Batch 7, transmembrane pressure and axial velocity were nominally 2.06 bar (30 psi) and 4.63 m/s (15 ft/s), respectively. Figure 19 shows that increasing insoluble solids content reduces the permeate fluxes obtained when concentrating between calculated solids contents of 10.3 and 15.0 wt.%. Also a continuous decline in permeate flux as solids are concentrated was seen. The effect of CUF restarts without backpulsing was also examined (Figure 19) at high insoluble solids concentration, where restarts such as between the 12.1 and 12.2 wt.% solids data points do show some potential to restore higher permeate fluxes temporarily, but the improvements will likely be several times below that of a backpulse. Permeance data was found to mirror the permeate flux data. Concentrations ranged from 9.5 to 10.2 wt.% (calculated) using TMP=1.72 bar (25 psi) and axial velocity of 4.63 m/s (15 ft/s), not shown, was tested but yielded a lower steady state flux (~12.3 $m^3/(m^2*day)$ [~0.21 gpm/ft^2] compared to ~13.5 $m^3/(m^2*day)$ [~0.23 gpm/ft^2] at TMP= 2.06 bar [30 psi] immediately after) and caused large fluctuations (±10-15 psi) in P1 leading to test discontinuance.

Filtration of Batch 4, operated nominally at TMP=3.45 bar (50 psi) and 3.72 m/s (12 ft/s) axial velocity and concentrating the solution from a calculated 10.3 wt.% to an analytically measured 13.9 wt.%, confirms the observations at high insoluble solids concentration seen in Batch 7 filtration. Again, permeate flux decreased with increasing insoluble solids content, with a steady decline as insoluble solids levels increased, and CUF restarts without a backpulse only slightly increases permeate flux compared to a backpulse. Calculated permeances again mirrored the trends in the permeate flux. When Figures 19 and 20 are compared, very similar fluxes are obtained over the similar insoluble solids content ranges at both operating conditions.

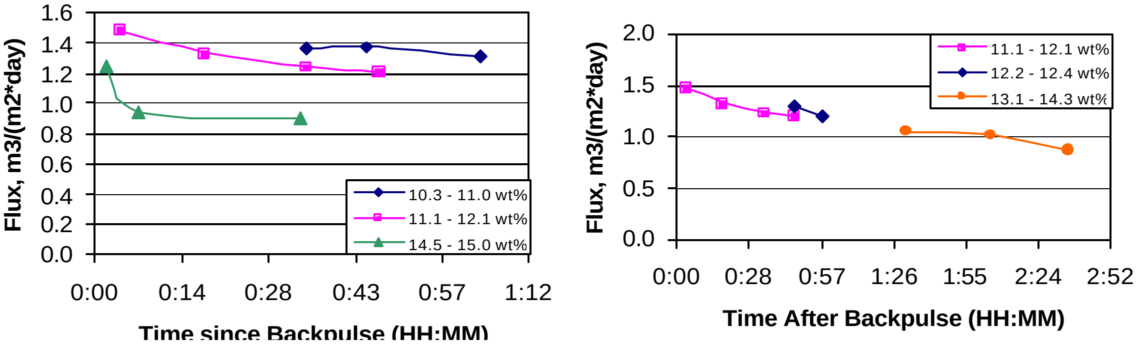


Figure 19 Permeate flux during filtration of Batch 7 as affected by high insoluble solids content, and as affected by restart without backpulse. TMP = 2.06 bar (30 psi), axial velocity = 4.63 m/s (15 ft/s), nominally.

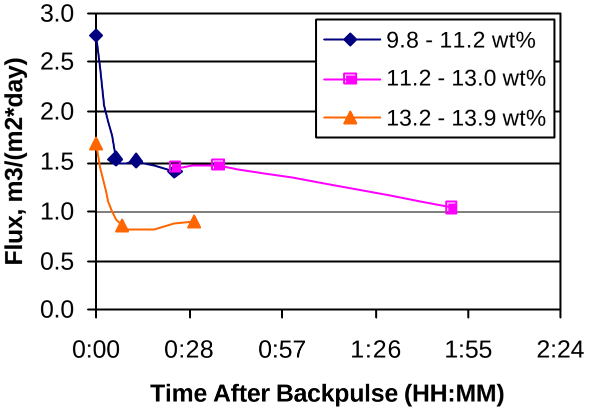


Figure 20 Permeate flux during filtration of Batch 4 as affected by high insoluble solids content, and as affected by restart without backpulse. TMP = 3.45 bar (50 psi), axial velocity = 3.72 m/s (12 ft/s), nominally.

A similar analysis was done for Batch #2 filtration data at lower insoluble solids loadings at TMP=3.45 bar (50 psi) and 3.72 m/s (12 ft/s) axial velocity. Figure 21 shows that filtration behavior is very different at low rather than high insoluble solids concentrations. The "spike" between the second (3.9 – 4.5 wt% insoluble solids, calculated) and third (4.5 – 6.3 wt% insoluble solids, calculated) curves illustrates that a CUF restart without a backpulse acts in the same way as a backpulse in restoring higher permeate fluxes. If the third curve is displaced left by 7 minutes, this and the first curve (1.1 – 3.8 wt% insoluble solids, calculated) overlap indicating that permeate fluxes are essentially independent of insoluble solids content in the ~1.5 – 6.5 wt% range.

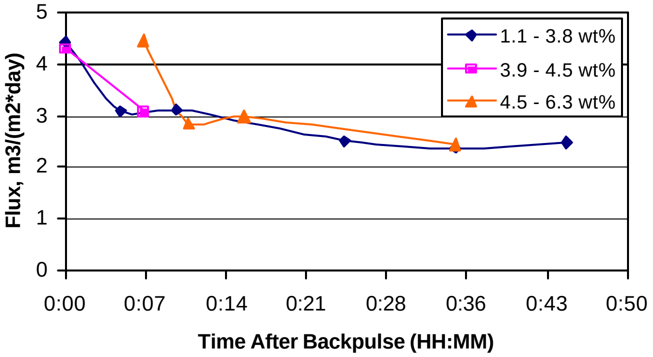


Figure 21 Permeate flux during filtration of Batch 2 as affected by low insoluble solids content, and as affected by restart without backpulse. TMP = 3.45 bar (50 psi), axial velocity = 3.72 m/s (12 ft/s), nominally.

The initial filtration of Batch 3 began without a backpulse and a simple restart of the CUF. Comparison of Figures 21 and 22 indicates that CUF restart begins to lose effectiveness as a backpulse substitute in restoring higher permeate fluxes at insoluble solids loadings above 6.8 wt%, and the steady state flux has also begun to drop. The calculated insoluble solids concentrations in Figures 21 and 22 are based on an analytically measured value of 6.8 wt% obtained for an end of Batch 2 filtration concentrate sample.

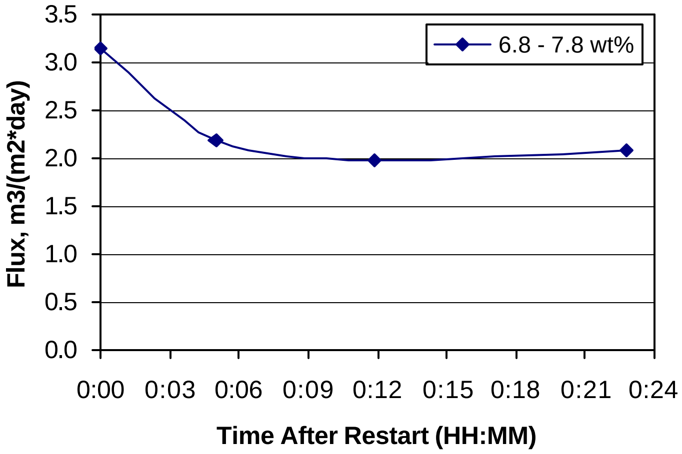


Figure 22 Permeate flux data at the beginning of Batch 3 filtration. TMP = 3.45 bar (50 psi), 3.75 m/s (12 ft/s) axial velocity, nominal.

Due to the difficulty in obtaining concentrate samples from the CUF for solids analysis, the opportunities to perform direct comparisons of the data without calculating insoluble solids content are very few. In the data collected, the most suitable comparison that can be made is of the high insoluble solids concentration flux data discussed previously (13.9 and 14.5 wt.% by independent analyses) and the flux data at the end of Batch 7 (finishing with 15.1 wt.%). Both were filtered at TMP = 2.06 bar (30 psi) and 4.63 m/s

(15 ft/s) axial velocity. The insoluble solids contents for both slurries were obtained analytically. Figure 23 compares filterability at the two conditions (permeance data mirrored the permeate flux data). The lower permeate flux at the end of Batch 7 is likely due to the slightly higher insoluble solids content.

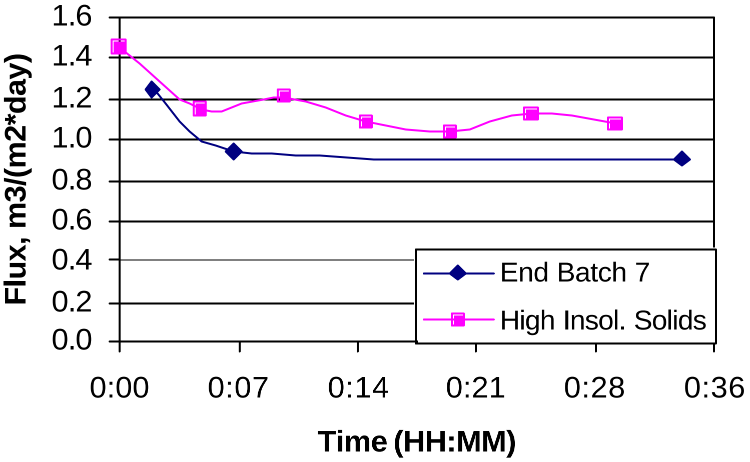


Figure 23 Permeate flux comparison between flux data taken at high insoluble solids concentration and at the end of Batch 7. TMP = 2.06 bar (30 psi), 4.63 (15 ft/s) nominal axial velocity.

3.7.7 Reduced Reagent Precipitation Batch

The optimization study by Rosencrance[28] recommended high hydroxide (1 M), strontium (0.075 M), and permanganate (0.05 M) addition to maximize Sr/TRU removal and filterability of treated AN-102. Currently, Sr-90 removal is thought to be removed by isotopic dilution and TRU removal by co-precipitation with manganese dioxide. To challenge these mechanisms and observe process sensitivity to precipitant addition levels, Batch #6 of the AN-102 Hanford waste was treated with a lower than recipe-specified precipitant quantities. Additionally as the full-scale process is expected to batch precipitate on top of recycled cross-flow filter concentrate until 20 wt% insoluble solids is attained in the precipitation tank, a test of the Sr/MnO_4 precipitation with pre-existing Sr/MnO_4 chemistry insoluble solids was also sought since this aspect has not been studied previously. Hence, a 700 mL portion of the concentrate produced in Batch #5 was included with 1.7 L fresh AN-102 feed material prior to addition of strontium nitrate and sodium permanganate in Batch #6.

In detail, at the end of Batch #5 with nearly 1 L feed remaining, the CUF was drained of concentrate and the CUF refilled with feed. The concentrate was then mixed with approximately 400 ml of remaining Batch #5 feed to yield approximately 1 L of slurry. Of this feed/concentrate mixture, approximately 300 ml was used to “top off” the CUF. To the remaining ~700 ml feed/concentrate, 1700 ml of untreated AN-102 material was added to the 4 L Erlenmeyer, followed by ~220 ml of ~0.3 M $Sr(NO_3)_2$ and ~147 ml ~0.4

M $NaMnO_4$. These reduced quantities of precipitating agents and feed translate to nominally ~42% $Sr(NO_3)_2$ and ~56% $NaMnO_4$ of the recipe cited in the Experimental section. The final total strontium and manganese concentrations (Table 7) of 0.040 and 0.041 M, respectively, represent a "middle-middle" addition level cited by Rosencrance above as acceptable for filterability.

Similar to the end of Batch #7 where permeate fluxes had slowed, filtration of Batch 6 was also interrupted and the CUF contents emptied due to slow permeate production rates. The steady state permeate flux in Batch #6 dropped to 0.56 $m^3/(m^2*day)$ [0.010 gpm/ft^2] by the time the calculated insoluble solids content in the CUF concentrate had risen to 10.1 wt.% (8.7 ± 0.4 wt.% by analytical measurement), compared to 0.9 $m^3/(m^2*day)$ [0.015 gpm/ft^2] when the concentrate insoluble solids content rose to 14.6 – 15.1 wt.% for Batch #7. Hence, the effect of precipitating with lower precipitating agent quantities appeared to significantly lower the filterability of the resulting precipitated slurry despite the contribution of filterable Batch #5 insoluble solids.

To help elucidate the quantity of insoluble solids formed as a function of precipitating reagent input, a comparison of measured and calculated insoluble solids concentration was performed for sample HIGHCONC3 as was done in the previous section for end-of-batch insoluble solids levels. HIGHCONC3 was a thick concentrate produced and drained from the CUF during Batch #6. In this case, the end of Batch #5 insoluble solids content of 7.7 wt% was used as a starting point, and the insoluble solids content was calculated forward based on filtrate production. A linear scaling assumption for precipitate generation was used, where an estimated 35% of the typical 1.5 wt% insoluble solids per batch was generated since 30% and 40% of the typical quantities of $Sr(NO_3)_2$ and $NaMnO_4$, respectively, were added. Although there is some uncertainty in the measurements (data ranged from 8.4 – 9.0 wt.%), the 10.1 wt% calculated insoluble solids content suggests that the linear scaling assumption likely overestimates insoluble solids production, and that a certain minimum precipitant addition threshold must be attained before significant precipitated solids are generated.

3.7.8 Insoluble Solids Wash Runs

At the end of the production of filtered precipitated feed, a high insoluble-solids content (15.1 wt%), highly-viscous concentrate was then washed with four aliquots of 0.01 M NaOH (inhibited water) to remove interstitial Hanford liquids. In each wash step, 600 ml 0.01 M NaOH was added to the CUF reservoir to produce ~600 ml of filtrate at TMP = 30 psi and 15 ft/s axial velocity, the optimal filtration conditions identified for high-insoluble solids content fluids. The only exception was Wash Run #4, where the "standard" conditions of 50 psig and 12 ft/s were used. Figure 24 shows that the low TMP and high axial velocity continued to be favorable compared to the "standard."

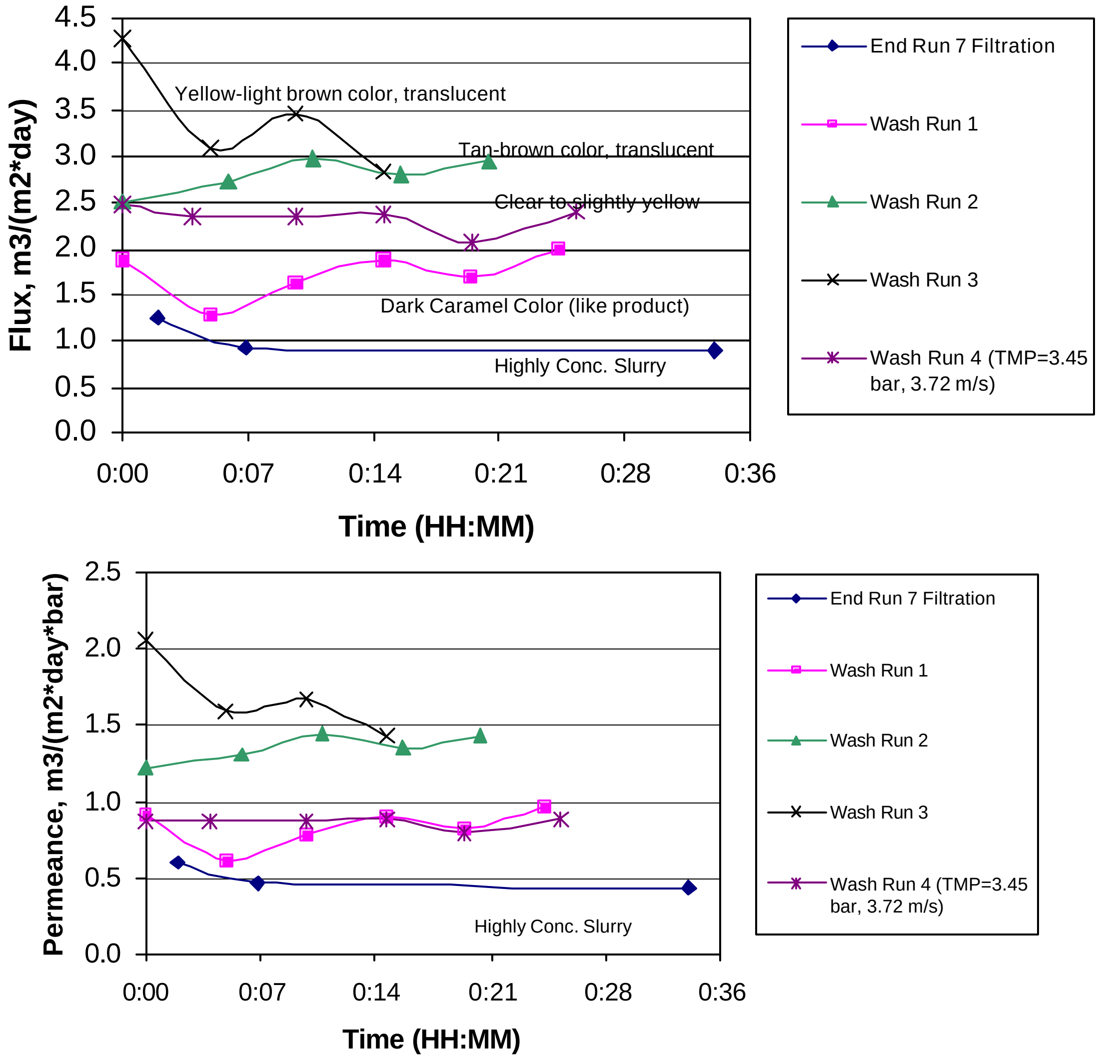


Figure 24 Permeate flux and permeance of each 0.01 M NaOH solution wash vs. the end of filtration results.

With each successive wash, permeate flux and permeance increased with the third wash yielding up to 3-4 times the flux obtained just at the end of precipitate filtration. Each wash also yielded successively lighter filtrates starting from a dark caramel to a yellowish-light brown liquid (uncorrected for color skew from Shielded Cell window and halogen lighting). Each filtrate produced was a translucent liquid, except for the second wash which showed only a slight trace of a “fluffy” dark solids at the bottom of the sample bottle. The CUF concentrate insoluble solids content drop from 15.1 wt% at the end of filtration to the 12.8 wt% (1 wt% std. dev.) at the end of the four washes, along with the successive loss of filtrate color, does suggest that some solids or salts are being removed during each wash step.

The total solids concentration of the CUF concentrate after washes was 16.6 wt% (0.16 wt% std. dev.), indicating that deactivated water (~0.04 wt% calculated total solids) has replaced of order 90-95% of the salt-laden supernatant liquid/filtrate. The expected replacement percentage was expected to be 95%, based on washing with 3 CUF internal volumes and assuming the CUF is well-stirred. Another interesting observation was that the CUF recirculation pump could not be easily restarted after completing the last wash indicating poor flow characteristics of the wash slurry.

3.7.9 Task Plan Deviations

As cited in Section 3.2 “Precipitation Recipes”, the added free hydroxide was 0.95 and 1.0 M for the $Ca/Sr/MnO_4$ and Sr/MnO_4 precipitation chemistry recipes, respectively, rather than the 0875 M additional free hydroxide target.

The cross-flow filtration experiments was performed per the specifications and Table 2 in the Technical Task Plan cited in the Introduction with a few deviations in the prescribed tests noted in Table 15 (next page). The transmembrane pressures in Tests 1-3 and 25 were reduced to obtain fluxes more similar to actual operating conditions. In Test 10, the transmembrane pressure was reduced from 40 to 30 psi (2.76 to 2.06 bar) as the system was found to be less sensitive to TMP than expected.

As TMP=50 psi (3.45 bar) and 12.2 ft/s (3.72 m/s) was determined to be the “optimum” conditions at that time (for Test 13), Tests 10-12 were modified to examine the effect of reduced axial velocity and/or transmembrane pressure. Test 14 was omitted as increased TMP was not found earlier to increase permeate flux, and Tests 15-16 were not performed as these duplicated earlier conditions.

As dewatering in Test 17 was performed at TMP=50 psi (3.45 bar) and 12.2 ft/s (3.72 m/s), Tests 18-20 became a study to quantify the effects of increased TMP (Test 20 unchanged), reduced axial velocity (Test 19), and a combination of reduced TMP and higher axial velocity (Test 18) at higher insoluble solids loading.

Tests 21-22 were deemed unnecessary, since TMP=30 psi (2.06 bar) and 15.2 ft/s (4.63 m/s) was observed to yield the highest permeate flux and the CUF pump was beginning to labor.

Rheology measurements for samples of insoluble solids concentrates remaining in the CUF after filtration of Batches 1, 4 and 7 were performed, and the results are cited in a separate report by Rosencrance[29].

Table 15 Technical Task Plan (Table 2) with deviations noted in parentheses

Test no.	Sample	Transmembrane pressure (psi)	Crossflow Velocity (ft/s)
1	Demin water	30 **(10)**	12.2
2	Demin water	40 **(15)**	12.2
3	Demin water	55 **(20)**	12.2
4	Feed	50	12.2
5	Feed	30	9.1
6	Feed	30	15.2
7	Feed	70	9.1
8	Feed	70	12.2 – 15.2
9	Feed	50	12.2
10	Feed	40 **(30)**	12.2
11	Feed	55 **(50)**	12.2 **(9.1)**
12	Feed	70 **(30)**	12.2 **(6.6)**
13	Feed	optimum from 4-12 **(50)**	9.1 **(12.2)**
14	Feed	optimum from 4-12 **(omitted)**	15.2 **(omitted)**
15	Feed	55 **(omitted)**	12.2 **(omitted)**
16	Feed	30 **(omitted-see 12)**	6.6 **(omitted-see 12)**
17	De-watering	optimum from 4-12	Optimum from 4-16
18	Solids Concentration	40 **(30)**	12.2 **(15.2)**
19	Solids Concentration	55 **(50)**	12.2 **(9.1)**
20	Solids Concentration	70	12.2
21	Solids Concentration	optimum from 18-20 **(omitted)**	9.1 **(omitted)**
22	Solids Concentration	optimum from 18-20 **(omitted)**	15.2 **(omitted)**
23	De-watering	optimum from 18-22 **(30)**	Optimum from 18-22 **(15.2)**
24	Acid Clean	N/A	N/A
25	Demin water (post acid clean)	40 **(10, 15, 20)**	12.2

4.0 CONCLUSIONS

Precipitation Chemistry Study

- The most significant observation on decontamination was that the level of reagent additions were found to be conservatively large. The low level composite filtrate had about one seventh the Sr-90 activity and one quarter of the TRU activity permitted for low level glass loading. This shows that reduced reagent addition will be permissible for the flowsheet.

- Removal or passage of elements match past observations. Al, B, Cd, Mo, Na, Ni, S, and P were not removed. Very small amounts of Ca, Cr, Pb, and Zn were removed. About 80% of the Fe is removed.

- This process does not remove technetium-99 from the solution.

- Am-241 does not wash out of the slurry during washing; plutonium shows some washout behavior.

- Nitric acid showed some effectiveness over water alone in dissolving residual matter in the filter rig.

Filterability Study

- At low insoluble solids levels, high transmembrane pressure and high axial velocity favors filterability, although the effect of transmembrane pressure is smaller. Permeance data suggests highest energy usage efficiency at low transmembrane pressure and high axial velocity.

- At high insoluble solids levels (~14 wt%), low transmembrane pressure (2.06 bar, 30 psi) and high axial velocity (4.63 m/s, 15 ft/s) favors filterability.

- Cells Unit Filter (CUF) restart is equivalent to a backpulse up to ~7 wt.% insoluble solids, in providing higher initial fluxes.

- Washing of solids filterability is best at low transmembrane pressure and high axial velocity, with flux increasing with increased rinsing with inhibited water.

- No significant difference in filterability was observed when the calcium nitrate-strontium nitrate-sodium permanganate strike data obtained was compared with previous literature for the strontium nitrate-sodium permanganate chemistry.

- Although no data was obtained to directly address the design basis filter flux "optimum" of 3.8 m^3/m^2*day (0.065 gpm/ft^2) for a Tank AN-102 waste with 2% insoluble solids (P. Townson, BNFL Memorandum CCN #011449, March 17, 2000), similar filter fluxes were experimentally obtained. Extensive data at 1.3 wt% insoluble solids presented in this study suggests the optimum is possible, but production data at 1.1 – 3.8 wt% insoluble solids was not encouraging. However, this experimental data was taken with largely unprecipitated solids which are expected to be less filterable than those from the Sr/MnO_4 chemistry.

5.0 RECOMMENDATIONS

- Future testing of the strontium and permanganate process should focus on reduction of added strontium and permanganate while still producing low level filtrate. Reduced levels of sodium hydroxide in the initial caustic adjustment would also provide a benefit of adding less sodium to the low level glass. It is possible that the precipitation reagents do not consume free hydroxide, though some is needed to assure that aluminum does not precipitate.
- Darkening of filtrate product bottles indicated some post-precipitation mechanism. This mechanism needs further study.
- Precipitation at lower temperatures need investigation for practicality. While not a focus of the current study, some room temperature precipitation has been tested in past "beaker" studies and would simplify the process.
- Rheology of the (sheared) precipitate at 15-20 wt% insoluble solids needs study. The 15 wt% material caused pumping difficulties in the narrow tubing of the cells filter unit.
- Backpulse frequency needs to be investigated to improve overall average filter fluxes.

6.0 REFERENCES

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APPENDIX A

SAMPLE AND ANALYSIS PLAN AND

BATCH VOLUME ESTIMATES

Basic information on the sample bottles, estimated sample and batch volume, and analysis tables is listed in this appendix. Volumes were estimated beforehand and do not necessarily match the campaign exactly. The following analyses were used in the program:

- ICP-ES: Inductively-Coupled Plasma Emission Spectroscopy provides over a dozen elements, mostly metals.
- AA(element): Atomic Absorption measures (element) concentration.
- Gamma: Gamma counting measures Cs-137, Eu-154, Eu-155, Co-60.
- Sr-90 was measured by a counting method.
- Am-241, plutonium, and curium were measured by a TTA counting technique. A related series of procedures for the same sample provides these.
- ISE: Ion selective electrode for Cl, F
- IC Anions: Ion Chromatography for common anions
- Tc-99 was measured by Inductively-Coupled Plasma Mass Spectroscopy.
- Uranium was measured by a wet chemical method commonly called “Chemcheck”.
- Weight percents were measured with standard filtration, drying, and weighing methods.

A “lollipop” is a shielded sample bottle. The shielding is of at least half an inch plastic that is effective in reducing alpha-beta dosage to extremities.

Large C Sr/TRU Precipitation and Filtration

Bottle and Sample List (Product and Samples

Sample Bottle Description	(BNF-LC080-) Feed Bottle Label	Bottle Content (ml)	Bottle Size (ml)	Special Notes	Desired Sample Analyses Acid digest	ICP-ES	AA (Na)	gamma (5000 sec,w/ Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	total alpha (TK-50 rad screen)	suspended solids wt.%	total solids	chemchek	rheology	density	total dissolved solids	TIC/TOC
Run 1 Filtrate	FILTRATE1	1218.74	1000	Excess liquid stuck in CUF																		
Run 1 Filtrate Digested	FILTR1DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 1 Digested Filtrate SampleA	FILTR1ADSA	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X															
Run 1 Digested Filtrate SampleB	FILTR1ADSB	10	15	10 ml DIGEST in 15 ml "Lollipop"				X2	X	X	X	X	X2		X2							
Run 1 Mid-run Concentrate Digested	MIDRUNCONC1DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 1 Mid-run Digested Conc. SampleA	MIDRUNCONC1ADSA	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X											X				
Run 1 Mid-run Digested Conc. SampleB	MIDRUNCONC1ADSB	10	15	10 ml DIGEST in 15 ml "Lollipop"				X	X	X	X	X	X									
Run 1 Mid-run Concentrate Sample	MIDRUNCONC1	25	60	To Charles Coleman, Cell 8												X2	X			X		
Run 1 Mid-run Filtrate Digested	MIDRUNFILT1DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 1 Mid-run Digested Filtrate SampleA	MIDRUNFILT1ADSA	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X											X				
Run 1 Mid-run Digested Filtrate SampleB	MIDRUNFILT1ADSB	10	15	10 ml DIGEST in 15 ml "Lollipop"				X	X	X	X	X	X									
Run 1 End of run Concentrate Digested	ENDRUNCONC1DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 1 End of run Digested Conc. SampleA	ENDRUNCONC1ADSA	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X											X				
Run 1 End of run Digested Conc. SampleB	ENDRUNCONC1ADSB	10	15	10 ml DIGEST in 15 ml "Lollipop"				X	X	X	X	X	X									
Run 1 End of run Concentrate Sample	ENDRUNCONC1	100	125	To Charles Coleman, Cell 8												X2	X		X	X		
Run 1 End of run Filtrate Digested	ENDRUNFILT1DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 1 End of run Digested Filtrate Samp.A	ENDRUNFILT1ADSA	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X											X				X
Run 1 End of run Digested Filtrate Samp.B	ENDRUNFILT1ADSB	10	15	10 ml DIGEST in 15 ml "Lollipop"				X	X	X	X	X	X	X								
	Total filtrate in bottle(ml)=	1218.74	(assumes no losses to equipment)																			
	Total samples volume (ml)	138																				

Sample Bottle Description	(BNF-LC080-) Feed Bottle Label	Bottle Content (ml)	Bottle Size (ml)	Special Note	Desired Sample Analyses Acid digest	ICP-ES	AA (Na)	gamma (5000 sec,w/ Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	total alpha (TK-50 rad screen)	suspended solids wt.%	total solids	chemchek	rheology	density(HLC)	total dissolved solids	TIC/TOC
Run 2 Filtrate		2927.96		(see below for container)																		
Run 2 End of run Concentrate Digested	ENDRUNCONC2DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 2 End of run Digested Conc. Sample	ENDRUNCONC2ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 2 End of run Concentrate Sample	ENDRUNCONC2	10	30	To Charles Coleman, Cell 8												X	X			X		
Run 2 End of run Filtrate Digested	ENDRUNFILT2DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 2 End of run Digested Filtr. Sample	ENDRUNFILT2ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
	Total filtrate produced(ml)=	2927.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	16																				

Sample Bottle Description	(BNF-LC080-) Feed Bottle Label	Bottle Content (ml)	Bottle Size (ml)	Special Note	Desired Sample Analyses Acid digest	ICP-ES	AA (Na)	gamma (5000 sec,w/ Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	total alpha (TK-50 rad screen)	suspended solids wt.%	total solids	chemchek	rheology	density	total dissolved solids	TIC/TOC
Run 3 Filtrate		2927.96		(see below for container)																		
Run 3 End of run Concentrate Digested	ENDRUNCONC3DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 3 End of run Digested Conc. Sample	ENDRUNCONC3ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 3 End of run Concentrate Sample	ENDRUNCONC3	10	30	To Charles Coleman, Cell 8												X	X			X		
Run 3 End of run Filtrate Digested	ENDRUNFILT3DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 3 End of run Digested Filtr. Sample	ENDRUNFILT3ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
	Total filtrate produced(ml)=	2927.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	16																				

Sample Bottle Description	(BNF-LC080-) Feed Bottle Label	Bottle Content (ml)	Bottle Size (ml)	Special Note	Desired Sample Analyses Acid digest	ICP-ES	AA (Na)	gamma (5000 sec,w/ Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	total alpha (TK-50 rad screen)	suspended solids wt.%	total solids	chemchek	rheology	density	total dissolved solids	TIC/TOC
Run 4 Filtrate		2837.96		(see below for container)																		
Run 4 End of run Concentrate Digested	ENDRUNCONC4DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 4 End of run Digested Conc. Sample	ENDRUNCONC4ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 4 End of run Concentrate Sample	ENDRUNCONC4	100	125	To Charles Coleman, Cell 8												X	X		X	X		
Run 4 End of run Filtrate Digested	ENDRUNFILT4DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 4 End of run Digested Filtr. Sample	ENDRUNFILT4ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X	X				X				X
	Total filtrate produced(ml)=	2837.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	106																				

	(BNF-LC080-)	Bottle	Bottle		Desired Sample Analyses			(5000 sec,w/							(TK-50 rad	suspended	total				dissolved	
Sample Bottle Description	Feed Bottle Label	Content (ml)	Size (ml)	Special Note	Acid digest	ICP-ES	AA (Na)	Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	screen)	solids wt.%	solids	chemchek	rheology	density	solids	TIC/TOC
Run 5 Filtrate		2927.96		(see below for container)																		
Run 5 End of run Concentrate Digested	ENDRUNCONC5DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 5 End of run Digested Conc. Sample	ENDRUNCONC5ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 5 End of run Concentrate Sample	ENDRUNCONC5	10	30	To Charles Coleman, Cell 8												X	X			X		
Run 5 End of run Filtrate Digested	ENDRUNFILT5DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 5 End of run Digested Filtr. Sample	ENDRUNFILT5ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
	Total filtrate produced(ml)=	2927.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	16																				

															total							
								gamma							alpha						total	
	(BNF-LC080-)	Bottle	Bottle		Desired Sample Analyses			(5000 sec,w/							(TK-50 rad	suspended	total				dissolved	
Sample Bottle Description	Feed Bottle Label	Content (ml)	Size (ml)	Special Note	Acid digest	ICP-ES	AA (Na)	Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	screen)	solids wt.%	solids	chemchek	rheology	density	solids	TIC/TOC
Run 6 Filtrate		2927.96		(see below for container)																		
Run 6 End of run Concentrate Digested	ENDRUNCONC6DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 6 End of run Digested Conc. Sample	ENDRUNCONC6ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 6 End of run Concentrate Sample	ENDRUNCONC6	10	30	To Charles Coleman, Cell 8												X	X			X		
Run 6 End of run Filtrate Digested	ENDRUNFILT6DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 6 End of run Digested Filtr. Sample	ENDRUNFILT6ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
	Total filtrate produced(ml)=	2927.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	16																				

															total							
								gamma							alpha						total	
	(BNF-LC080-)	Bottle	Bottle		Desired Sample Analyses			(5000 sec,w/							(TK-50 rad	suspended	total				dissolved	
Sample Bottle Description	Feed Bottle Label	Content (ml)	Size (ml)	Special Note	Acid digest	ICP-ES	AA (Na)	Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	screen)	solids wt.%	solids	chemchek	rheology	density	solids	TIC/TOC
Run 7 Filtrate		2837.96		(see below for container)																		
Run 7 End of run Concentrate Digested	ENDRUNCONC7DIGEST	30	60	Pipette 5 ml, acid digestion by ADS	X																	
Run 7 End of run Digested Conc. Sample	ENDRUNCONC7ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X					X				
Run 7 End of run Concentrate Sample	ENDRUNCONC7	100	125	To Charles Coleman, Cell 8												X	X		X	X		
Run 7 End of run Filtrate Digested	ENDRUNFILT7DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																		
Run 7 End of run Digested Filtr. Sample	ENDRUNFILT7ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X	X	X				X				X
	Total filtrate produced(ml)=	2837.96	(assumes no losses to equipment)																			
	Total samples volume (ml)=	106																				

	(BNF-LC080-)	Bottle	Bottle		Desired Sample Analyses			(5000 sec,w/							(TK-50 rad	suspended	total				dissolved
Sample Bottle Description	Feed Bottle Label	Content (ml)	Size (ml)	Special Note	Acid digest	ICP-ES	AA (Na)	Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99	Cm	screen)	solids wt.%	solids	chemchek	rheology	density	solids
Used NaOH, Wash Volume 1	WASH1	600	1000																		
Used NaOH, Wash Vol. 1 Digested #1	WASH1DIGEST1	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 1 Samp#1	WASH1ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 1 Digested #2	WASH1DIGEST2	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 1 Samp#2	WASH1ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 1 Sample	WASH1SAMP	25	30	Sample at end of run																	X
Used NaOH, Wash Volume 2	WASH2	600	1000																		
Used NaOH, Wash Vol. 2 Digested #1	WASH2DIGEST1	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 2 Samp#1	WASH2ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 2 Digested #2	WASH2DIGEST2	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 2 Samp#2	WASH2ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 2 Sample	WASH2SAMP	25	30	Sample at end of run																	X
Used NaOH, Wash Volume 3	WASH3	600	1000																		
Used NaOH, Wash Vol. 3 Digested #1	WASH3DIGEST1	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 3 Samp#1	WASH3ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 3 Digested #2	WASH3DIGEST2	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 3 Samp#2	WASH3ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 3 Sample	WASH3SAMP	25	30	Sample at end of run																	X
Used NaOH, Wash Volume 4	WASH4	600	1000																		
Used NaOH, Wash Vol. 4 Digested #1	WASH4DIGEST1	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 4 Samp#1	WASH4ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 4 Digested #2	WASH4DIGEST2	30	60	Add 1 ml sample to 29 ml 0.01 M KOH																	
Used NaOH Digested, Wash Vol. 4 Samp#2	WASH4ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X									
Used NaOH, Wash Vol. 4 Sample	WASH4SAMP	25	30	Sample at end of run																	X
Wash Concentrate, Wash Vol. 1 Digested#1	WASHCONC1DIGEST1	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 1 Sample #1	WASHCONC1ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate, Wash Vol. 1 Digested#2	WASHCONC1DIGEST2	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 1 Sample #2	WASHCONC1ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate Sample, Wash Vol. 1	WASHCONC1SAMP	10	30	To Charles Coleman, Cell 8												X2	X2			X2	
Wash Concentrate, Wash Vol. 2 Digested#1	WASHCONC2DIGEST1	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 2 Sample #1	WASHCONC2ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate, Wash Vol. 2 Digested#2	WASHCONC2DIGEST2	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 2 Sample #2	WASHCONC2ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate Sample, Wash Vol. 2	WASHCONC2SAMP	10	30	To Charles Coleman, Cell 8												X2	X2			X2	
Wash Concentrate, Wash Vol. 3 Digested#1	WASHCONC3DIGEST1	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 3 Sample #1	WASHCONC3ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate, Wash Vol. 3 Digested#2	WASHCONC3DIGEST2	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 3 Sample #2	WASHCONC3ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate Sample, Wash Vol. 3	WASHCONC3SAMP	10	30	To Charles Coleman, Cell 8												X2	X2			X2	
Wash Concentrate, Wash Vol. 4 Digested#1	WASHCONC4DIGEST1	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 4 Sample #1	WASHCONC4ADS1	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate, Wash Vol. 4 Digested#2	WASHCONC4DIGEST2	30	60	Pipette 5 ml, acid digestion by ADS	X																
Digested Wash Conc., Vol. 4 Sample #2	WASHCONC4ADS2	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X	X	X	X	X						X			
Wash Concentrate Sample, Wash Vol. 4	WASHCONC4SAMP	10	30	To Charles Coleman, Cell 8												X2	X2			X2	
End of Project Concentrate, Bottle 1	HIGHCONC1	???	1000																		
End of Project Concentrate, Bottle 2	HIGHCONC2	???	1000																		

	(BNF-LC080-)	Bottle	Bottle		Desired Sample Analyses			(5000 sec,w/					
Sample Bottle Description	Feed Bottle Label	Content (ml)	Size (ml)	Special Note	Acid digest	ICP-ES	AA (Na)	Co-60 scan)	Sr-90	Am-241	Pu-239/240	Pu-238	Tc-99
Filtrate, Bottle 2	FILTRATE2	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 2 (Digested)	FILTRATE2DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 2	FILTRATE2ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 3	FILTRATE3	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 3 (Digested)	FILTRATE3DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 3	FILTRATE3ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 4	FILTRATE4	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 4 (Digested)	FILTRATE4DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 4	FILTRATE4ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 5	FILTRATE5	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 5 (Digested)	FILTRATE5DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 5	FILTRATE5ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 6	FILTRATE6	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 6 (Digested)	FILTRATE6DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 6	FILTRATE6ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 7	FILTRATE7	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 7 (Digested)	FILTRATE7DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 7	FILTRATE7ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 8	FILTRATE8	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 8 (Digested)	FILTRATE8DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 8	FILTRATE8ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 9	FILTRATE9	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 9 (Digested)	FILTRATE9DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 9	FILTRATE9ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 10	FILTRATE10	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 10 (Digested)	FILTRATE10DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 10	FILTRATE10ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 11	FILTRATE11	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 11 (Digested)	FILTRATE11DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 11	FILTRATE11ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 12	FILTRATE12	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 12 (Digested)	FILTRATE12DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 12	FILTRATE12ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2
Filtrate, Bottle 13	FILTRATE13	1700	2000	Graduated, baled top and bottom									
Filtrate, Bottle 13 (Digested)	FILTRATE13DIGEST	30	60	Add 1 ml sample to 29 ml 0.01 M KOH									
Digested Filtrate Sample, Bottle 13	FILTRATE13ADS	10	15	10 ml DIGEST in 15 ml "Lollipop"	X	X	X	X2	X	X	X	X	X2

Large C Sr/TRU Precipitation and Filtration					
Bottle and Sample List (Feed Materials)					
Run 1		Run volume (liter)	1.2		
		(BNF-LC080-)	Bottle	Bottle	
	Feed Bottles Description	Feed Bottle Label	Content (ml)	size (ml)	Special Notes
	1.2 l Large C Sample (6 M Na)	FEED1	1200	2000	baled top&bottom
	17 M NaOH	NAOH1	76.872	100	
	1 M Ca(NO3)2	CANIT1	12.012	30	
	1 M Sr(NO3)2	SRNIT1	27.12	60	
	1 M NaMnO4	PERMANG1	40.74	60	
		Total volume (ml) =	1356.74		
Run 2		Run volume (liter)	2.4		
			Bottle	Bottle	
	Feed Bottles Description	Feed Bottle Label	Content (ml)	size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED2	2400	4000	
	17 M NaOH	NAOH2	175.94	250	
	1 M Sr(NO3)2	SRNIT2	220.85	250	
	1 M NaMnO4	PERMANG2	147.17	250	
		Total volume (ml) =	2943.96		
Run 3		Run volume (liter)	2.4		
			Bottle	Bottle	
	Feed Bottles Description	Feed Bottle Label	Content (ml)	size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED3	2400	4000	
	17 M NaOH	NAOH3	175.94	250	
	1 M Sr(NO3)2	SRNIT3	220.85	250	
	1 M NaMnO4	PERMANG3	147.17	250	
		Total volume (ml) =	2943.96		
Run 4		Run volume (liter)	2.4		
			Bottle	Bottle	
	Feed Bottles Description	Feed Bottle Label	Content (ml)	size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED4	2400	4000	
	17 M NaOH	NAOH4	175.94	250	
	1 M Sr(NO3)2	SRNIT4	220.85	250	
	1 M NaMnO4	PERMANG4	147.17	250	
		Total volume (ml) =	2943.96		
Run 5		Run volume (liter)	2.4		
			Bottle	Bottle	
	Feed Bottles Description	Feed Bottle Label	Content (ml)	size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED5	2400	4000	
	17 M NaOH	NAOH5	175.94	250	
	1 M Sr(NO3)2	SRNIT5	220.85	250	
	1 M NaMnO4	PERMANG5	147.17	250	
		Total volume (ml) =	2943.96		

Large C Precipitation and Filtration Reagent Bottle List and Volumes

Run 6		Run volume (liter)	2.4		
	Feed Bottles Description	Feed Bottle Label	Bottle Content (ml)	Bottle size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED6	2400	4000	
	17 M NaOH	NAOH6	175.94	250	
	1 M Sr(NO3)2	SRNIT6	220.85	250	
	1 M NaMnO4	PERMANG6	147.17	250	
		Total volume (ml) =	2943.96		
Run 7		Run volume (liter)	2.4		
	Feed Bottles Description	Feed Bottle Label	Bottle Content (ml)	Bottle size (ml)	Special Notes
	Large C Sample (6 M Na)	FEED7	2400	4000	
	17 M NaOH	NAOH7	175.94	250	
	1 M Sr(NO3)2	SRNIT7	220.85	250	
	1 M NaMnO4	PERMANG7	147.17	250	
		Total volume (ml) =	2943.96		

APPENDIX B

CALCULATIONS

Calculations below were made for reagent additions

Envelope Large C Dilution Calculation Check						
	AN-102					
Precipitation with Ca(NO3)2						
	solution	volume to add (liters)	solution conc.			
	VNaOH	0.064058214	17 M NaOH		Assume Initial solution Env. C (AN-102) solution containing ~10 M Na diluted with DI H2O	
	VCaNO3	0.010014656	1 M Ca(NO3)2		to make 6 M Na, 0.7633 M free OH-, 0.0075 M Ca2+, and 0 M Sr2+ soln.	
	VSrNO3	0.022601459	1 M Sr(NO3)2			
	VMnO4	0.033948615	1 M NaMnO4			
		(per liter 6.0 M Na Large C)				
		Equ 1	9.92707E-06		(0.7633+0.875M)*(1l+VNaOH+VCaNO3+VSrNO3+VMnO4) = 0.7633M(1l)+17M*VNaOH	
		Equ 2	9.99986E-06		(0.0075+0.0080))M(1l+VNaOH+VCaNO3+VSrNO3+VMnO4)=0.0075M(1l) + 1M(VCaNO3)	
		Equ 3	1.09998E-05		0.02M(1l+VNaOH+VCaNO3+VSrNO3+VMnO4)=0M(1l)+1M(VSrNO3)	
		Equ 4	-2.99267E-05		0.03M(1l+VNaOH+VCaNO3+VSrNO3+VMnO4)=0M(1l)+1M(VMnO4)	
	Target	Sum Eq. 1-4	1E-06			
		Just for information, the new Na concentration is:			6.269986	M
						Na Conc. = (6.0M(1l)+17M*VNaOH)/(1l+VNaOH+VCaNO3+VSrNO3+VMnO4)
		Initial volume of 6 M Large C:		1	liter	
		Final volume after additions:		1.130623	liter	
Precipitation with no Ca(NO3)2						
	solution	volume to add (liters)	solution conc.			
	VNaOH	0.073312902	17 M NaOH		Assume Initial solution Env. C (AN-102) solution containing ~10 M Na diluted with DI H2O	
	VSrNO3	0.092021192	1 M Sr(NO3)2		to make 6 M Na, 0.7633 M free OH-, 0.0075 M Ca2+, and 0 M Sr2+ soln.	
	VMnO4	0.061321794	1 M NaMnO4			
		(per liter 6.0 M Na Large C)				
		Equ 1	1.1E-05		(0.7633+0.875M)*(1l+VNaOH+VSrNO3+VMnO4) = 0.7633M(1l)+17M*VNaOH	
		Equ 2	-2.19999E-05		0.075M(1l+VNaOH+VSrNO3+VMnO4)=0M(1l)+1M(VSrNO3)	
		Equ 3	1.1E-05		0.05M(1l+VNaOH+VSrNO3+VMnO4)=0M(1l)+1M(VMnO4)	
	Target	Sum Eq. 1-4	1.24146E-10			
		Just for information, the new Na concentration is:			5.907377	M
						Na Conc. = (6.0M(1l)+17M*VNaOH)/(1l+VNaOH+VCaNO3+VSrNO3+VMnO4)
		Initial volume of 6 M Large C:		1	liter	
		Final volume after additions:		1.226656	liter	

APPENDIX C

FINAL PRODUCT COMPOSITE ANALYSES

Main products from the Large C precipitation and filtration campaign are provided on the pages C-2 and C-3, following. The filtrate product was sampled after all 10 filtrate bottles plus four bottles of 600-ml of wash solution were composited. This product was then forwarded to cesium ion exchange.

The washed slurry product was acid-digested with a final dilution factor of 30 to create the samples for analysis. Acid dissolution changes some analytes like anions so final analysis is not as extensive as that for filtrate. However, the metals and radioactive elemental analysis is valuable data for evaluating the process. It was planned that this product would be mixed with an Envelope D high level sludge for a vitrification test.

Table C-1. Measured Composition of the Grand Filtrate Product Composite

300145154

ChemChek	mg/L	Dilution-Corrected mg/L
U	0.126	12.6

300145155

ICP-ES Analyte		mg/L		Dilution-Corrected mg/L	
Ag	<	0.006	<	0.6	
Al		57.75		5775	
B		0.156		15.6	
Ba	<	0.002	<	0.2	
Ca		0.979		97.9	
Cd		0.241		24.1	
Co		0.016		1.6	
Cr		0.93		93	
Cu		0.097		9.7	
Fe		0.025		2.5	
La	<	0.011	<	1.1	
Li	<	0.002	<	0.2	
Mg		0.003		0.3	
Mn		0.012		1.2	
Mo		0.229		22.9	
Na		1107.809		110780.9	4.82 M Na
Ni		1.624		162.4	
P		8.282		828.2	
Pb		0.581		58.1	
Si		0.084		8.4	
Sn		0.175		17.5	
Sr		1.471		147.1	
Ti	<	0.002	<	0.2	
V	<	0.003	<	0.3	
Zn		0.027		2.7	
Zr		0.01		1	

300145153

Radiochem	dpm/mL	Dilution-Corrected dpm/mL	uCi/ml
Sr-90	2.03E+04	2.03E+06	0.9144
Pu-238	2.06E+01	2.06E+03	0.0009
Pu-239-240	1.64E+01	1.64E+03	0.0007
Am-241	2.31E+02	2.31E+04	0.0104
Cm-244	2.51E+02	2.51E+04	0.0113
Cs-137	3.34E+06	3.34E+08	150.5225
Cs-137 (uCi/mL)	1.5052	150.52	

300145156

ISE		mg/L		Dilution-Corrected mg/L
F	<	10	<	1000
Cl		18.1		1810

300145156

OH	Molarity	Dilution-Corrected Molarity	
Free OH	0.0125	1.25	
Total OH	0.0117	1.17	**Total less than free

300145157

IC Anion		mg/L		Dilution-Corrected mg/L			
F		6		600	0.032	M	
Formate		42		4200	0.093	M	
Chloride		25		2500	0.071	M	
Nitrite		303		30300	0.659	M	
Nitrate		824		82400	1.329	M	
Phosphate	<	100	<	10000			
Sulfate		50		5000	0.052	M	
Oxalate	<	100	<	10000			
					3.54	M	Total Anion
					1.28	M Cation Excess (ICP-ES Na, Fre	

300145158

AA	mg/L	Dilution-Corrected mg/L		
Na	1119.12	111912	4.87	M Na
K	9.939	993.9	0.03	M K
		Total	4.89	M Cation
			1.36	M Cation Excess

Table C-2. Measured Composition of the Final Washed Precipitate Slurry

Element	g/liter
Al	1.037
B	0.006
Ba	0.026
Ca	0.717
Cd	0.003
Co	0.002
Cr	0.303
Cu	0.004
Fe	0.121
La	0.048
Li	0.001
Mg	0.001
Mn	13.728
Mo	0.002
Na	10.461
Ni	0.073
P	0.259
Pb	0.140
Si	3.681
Sn	0.005
Sr	30.306
Tc	0.003
Ti	0.001
V	0.001
Zn	0.019
Zr	0.023

Cs-137	2.79E+07	dpm/ml
Am-241	1.16E+06	dpm/ml
Cm-244	4.94E+05	dpm/ml
Pu-239	3.51E+04	dpm/ml
Pu-238	3.87E+04	dpm/ml
Sr-90	2.36E+08	dpm/ml
Total U*	6.9345	mg/liter

APPENDIX D

RAW ANALYTICAL DATA

Analytical data for the campaign is archived in this appendix. Note that all samples were diluted 30 times; concentrations and activities in this section need to be multiplied by 30 to obtain sample values.

Table D-1. Compositions for Digested Slurries (Concentrates) and Batch (Instantaneous) Filtrate Samples
Units: mg/liter

	Conc-1x	Conc-1xx	Conc-1b	Filt-1b	Conc-1c	Filt-1c	Conc-2	Filt-2	Conc-3	Filt-3
Al	13.932	13.083	222.912	212.919	209.328	192.448	68.277	218.455	149.747	214.049
B	873.614	921.330	13977.82	0.587	14741.28	0.511	0.744	0.613	0.551	0.591
Ba	0.027	0.032	0.432	0.020	0.512	0.02	0.621	0.082	0.020	0.058
Ca	2.265	2.102	36.24	15.044	33.632	13.29	14.529	5.610	5.056	3.479
Cd	0.084	0.087	1.344	0.973	1.392	0.864	0.959	0.938	0.775	0.905
Co	0.050	0.089	0.8	0.060	1.424	0.06	0.050	0.240	0.050	0.186
Cr	0.415	0.422	6.64	3.390	6.752	3.058	6.436	3.527	4.692	3.491
Cu	0.068	0.067	1.088	0.411	1.072	0.357	0.340	0.468	0.145	0.413
Fe	0.234	0.546	3.744	0.078	8.736	0.109	1.355	0.363	0.443	0.179
La	0.110	0.110	1.76	0.080	1.76	0.08	0.364	0.611	0.110	0.511
Li	0.023	0.020	0.368	0.030	0.32	0.03	0.020	0.107	0.020	0.077
Mg	0.010	0.010	0.16	0.032	0.16	0.032	0.254	0.010	0.026	0.01
Mn	5.145	4.834	82.32	0.023	77.344	0.042	271.866	0.114	18.571	0.075
Mo	0.109	0.098	1.744	0.936	1.568	0.876	0.862	0.951	0.707	0.868
Na	288.018	279.515	4608.288	4552.684	4472.24	4048.125	4759.038	4749.198	3874.785	3976.731
Ni	0.497	0.510	7.952	6.558	8.16	5.94	7.922	6.225	5.264	6.054
P	2.273	1.965	36.368	29.568	31.44	26.727	35.118	32.010	26.918	30.944
Pb	0.601	0.706	9.616	2.330	11.296	1.998	4.947	2.570	1.754	2.577
Si	9.135	7.911	146.16	0.361	126.576	0.29	152.036	1.046	89.192	0.803
Sn	0.191	0.156	3.056	0.668	2.496	0.598	0.171	0.767	0.247	0.684
Sr	4.955	4.857	79.28	2.267	77.712	2.234	477.871	4.394	40.845	5.104
Tc			0	0.070	0	0.07	0.125	0.276	0.094	0.188
Ti	0.047	0.044	0.752	0.020	0.704	0.02	0.020	0.135	0.020	0.091
V	0.068	0.063	1.088	0.040	1.008	0.04	0.030	0.223	0.030	0.162
Zn	0.030	0.030	0.48	0.147	0.48	0.203	0.315	0.104	0.055	0.09
Zr	0.872	0.547	13.952	0.928	8.752	0.47	0.056	1.223	0.082	0.483

Table D-2. Compositions for Digested Slurries (Concentrates) and Batch (Instantaneous) Filtrate Samples
Units: mg/liter

	Conc-5	Filt-5	Conc-6	Filt-6	Conc-7	Filt-7
Al	33.698		124.32	223.046	21.244	247.486
B	0.698		0.641	0.57	0.628	0.587
Ba	0.193		0.095	0.02	0.192	0.020
Ca	9.399		7.741	4.964	9.235	5.057
Cd	0.853		0.819	0.969	0.813	1.083
Co	0.050		0.05	0.085	0.050	0.100
Cr	5.738		4.929	3.389	6.249	3.257
Cu	0.209		0.201	0.384	0.201	0.446
Fe	0.637		0.685	0.09	0.575	0.113
La	0.155		0.11	0.11	0.110	0.110
Li	0.020		0.02	0.02	0.020	0.020
Mg	0.077		0.025	0.01	0.014	0.010
Mn	163.461		95.951	0.136	158.806	0.557
Mo	0.706		0.722	0.978	0.689	1.050
Na	4192.144		4246.906	4700.967	3715.728	5234.326
Ni	6.606		6.156	6.374	6.191	7.213
P	29.428		26.688	29.217	27.762	34.310
Pb	3.273		2.539	2.378	3.235	2.668
Si	144.580		93.134	0.314	126.611	0.287
Sn	0.150		0.247	0.611	0.151	0.818
Sr	311.051		169.736	4.684	263.598	5.324
Tc	0.101		0.07		0.081	
Ti	0.020		0.02	0.02	0.020	0.020
V	0.030		0.03	0.035	0.030	0.030
Zn	0.181		0.113	0.03	0.186	0.030
Zr	0.040		0.04	1.144	0.040	0.497

Note: Data for the Filt-5 sample (instantaneous grab sample of Batch #5 filtrate) are not available because of a dilution error for the ICP-ES method.

Table D-3. Filtrate Product Bottle Compositions for the Ten Bottles

ALL ENTRIES ARE mg/liter

Below Det.:	Element	Bottle1	Bottle 6	Bottle 7	Bottle 9	Bottle 3	Bottle 2	Bottle 4	Bottle 5	Bottle 8	Bottle 10
	Al	7318.26	6720.945	7069.56	6549.09	6577.41	5865.12	5867.55	6000.06	6826.62	7318.26
	B	18.39	16.935	17.13	17.01	18.93	17.49	15.93	16.59	17.97	18.39
x	Ba	0.6	0.3	0.6	0.6	2.55	2.37	2.4	2.28	0.87	0.6
	Ca	151.71	112.185	121.44	156.72	140.58	232.98	96.84	100.05	146.67	151.71
	Cd	32.34	28.425	30.06	30.9	28.71	26.19	24.09	25.17	31.59	32.34
	Co	2.7	2.46	2.61	2.52	7.56	7.35	6.51	6.3	4.2	2.7
	Cr	95.22	104.43	108.21	109.77	111.24	97.2	96	99.84	106.89	95.22
	Cu	12.99	11.49	12.12	12.3	14.31	13.53	12.15	12.81	13.05	12.99
	Fe	2.76	3.03	2.67	2.82	7.17	6.54	6.39	6.45	4.05	2.76
x	La	3.3	1.68	3.3	3.3	19.62	17.88	18.33	17.46	4.86	3.3
x	Li	0.6	0.3	0.6	0.6	3.33	3.03	3.15	3.03	0.99	0.6
x	Mg	0.3	0.15	0.3	0.3	0.3	0.42	0.33	0.3	0.3	0.3
	Mn	3.84	3.615	2.31	3.33	7.68	2.34	2.52	5.37	4.86	3.84
	Mo	30.6	27.75	29.52	30.93	28.32	25.86	24.54	25.29	30.84	30.6
	Na	141383.04	137299.59	138780.69	146209.08	143753.22	119324.67	135310.65	136062.48	138323.7	141383.04
	Ni	212.88	190.53	200.88	206.43	193.83	175.38	162.06	171.18	203.34	212.88
	P	946.71	926.25	940.65	879.99	966.54	842.43	840.15	867.93	947.16	946.71
	Pb	80.04	67.59	73.47	80.04	85.92	84.6	74.94	83.94	88.05	80.04
	Si	9.09	9.36	8.28	6.63	33.12	30.57	27.96	27.63	21.09	9.09
	Sn	20.82	19.185	22.08	18.15	19.5	18.69	18.39	17.25	22.98	20.82
	Sr	153.48	160.2	170.25	125.97	159.57	93.03	127.59	133.23	143.4	153.48
x	Ti	0.6	0.3	0.6	0.6	4.08	3.75	3.84	3.72	1.38	0.6
x	V	0.9	0.555	0.99	0.9	6.84	6.54	6.27	6.21	3.03	0.9
x	Zn	0.9	0.45	0.9	0.9	2.91	3.54	3.36	2.79	0.9	0.9
	Zr	7.83	12.75	7.5	4.98	14.55	16.32	21.54	22.32	28.08	7.83
	Filt Bottle #	1	6	7	9	3	2	4	5	8	10

Table D-4. Filtrate Activities for Ten Bottles

Large	ADS LIMS #	Sr-90	Sample µCi/ml
C	Filtrate#1	1.96E+05	2.65
Product	Filtrate#2	7.68E+04	1.04
	Filtrate#3	2.24E+05	3.03
	Filtrate#4	9.45E+04	1.28
	Filtrate#5	8.69E+04	1.17
	Filtrate#6	1.43E+05	1.93
	Filtrate#7	6.83E+04	0.92
	Filtrate#8	7.08E+04	0.96
	Filtrate#9	7.04E+04	0.95
	Filtrate#10	6.86E+04	0.93

	Tc-99,	mg/liter
Batch #3	4.86	
Batch #4	5.04	
Filtrate #2	5.22	
Filtrate #3	5.46	
Filtrate #4	4.68	
Filtrate #5	4.86	
Filtrate #6	8.88	
Filtrate #9	5.19	
Avg, St. dev.	5.52375	1.378363

Table D-5. Filtrate and Wash Solution Activities
Units for “Sample” (dilution corrected) are microcuries/ml

ADS LIMS #	Am-241	Sample	Cm-244	Sample	ADS #	Pu239/240	Sample	Pu238	Sample
Filtrate#1	2010	0.02716	1360	0.01838	135150	206	0.00278	138	0.00186
Filtrate#2	1890	0.02554	1130	0.01527	135202	91	0.00123	91	0.00123
Filtrate#3	1.39E+03	0.01878	1.04E+03	0.01405	135203	1.56E+02	0.00211	1.02E+02	0.00138
Filtrate#4	1.46E+03	0.01973	1.25E+03	0.01689	135204	3.40E+02	0.00459	2.29E+02	0.00309
Filtrate#5	8.74E+02	0.01181	1.29E+03	0.01743	135205	1.75E+02	0.00236	1.24E+02	0.00168
Filtrate#6	3.07E+03	0.04149	2.71E+03	0.03662	135206	2.18E+02	0.00295	1.57E+02	0.00212
Filtrate#7	2.10E+03	0.02838	1.59E+03	0.02149	135207	1.12E+02	0.00151	1.05E+02	0.00142
Filtrate#8	2.33E+03	0.03149	8.26E+03	0.11162	135208	1.05E+02	0.00142	7.12E+01	0.00096
Filtrate#9	2.08E+03	0.02811	1.18E+03	0.01595	135209	1.00E+02	0.00135	8.33E+01	0.00113
Filtrate#10	8.77E+02	0.01185	8.85E+02	0.01196	135210	1.38E+02	0.00186	9.03E+01	0.00122
Wash #1	3.10E+02	0.00419	2.68E+02	0.00362	135214	4.88E+01	0.00066	3.49E+01	0.00047
Wash #2	2.17E+02	0.00293	4.07E+02	0.00550	135216	2.88E+01	0.00039	1.55E+01	0.00021
Wash #3	1.52E+02	0.00205	2.23E+02	0.00301	135218	9.08E+00	0.00012	5.97E+00	0.00008
Wash #4	1.05E+02	0.00142	8.92E+01	0.00121	135220	2.63E+01	0.00036	1.47E+01	0.00020

Table D-6. Digested Slurry Compositions for the Four Washes – Duplicate Measurements
Units: mg/liter

ADS#	WashConc-1 3-135222	WashConc-1 3-135223	WashConc-2 3-135224	WashConc-2 3-135225	WashConc-3 3-135226	WashConc-3 3-135227	WashConc-4 3-135228	WashConc-4 3-135229
Al	43.745	43.82	16.164	20.806	29.335	36.54	39.796	29.358
B	0.295	0.338	0.132	0.239	0.192	0.257	0.191	0.206
Ba	0.334	0.313	0.24	0.732	0.503	0.828	0.968	0.787
Ca	10.619	10.546	6.851	8.697	14.492	22.251	25.61	22.187
Cd	0.291	0.23	0.036	0.043	0.05	0.101	0.093	0.077
Co	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Cr	6.443	5.82	2.888	3.596	6.204	9.573	10.93	9.302
Cu	0.085	0.1	0.03	0.03	0.051	0.111	0.126	0.116
Fe	1.909	2.454	1.363	1.451	3.129	3.847	4.661	3.436
La	0.355	0.483	0.328	0.437	1.001	1.192	2.175	1.038
Li	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Mg	0.021	0.02	0.01	0.02	0.06	0.096	0.047	0.044
Mn	244.247	303.447	152.388	190.727	326.716	442.463	502.911	412.278
Mo	0.215	0.178	0.06	0.06	0.06	0.06	0.06	0.06
Na	1529.064	1197.5	242.86	326.915	268.59	491.735	359.683	337.737
Ni	3.072	2.895	0.926	1.181	1.699	2.481	2.661	2.237
P	10.595	9.31	2.772	3.531	4.734	7.902	9.017	8.268
Pb	2.356	2.6	1.123	1.532	2.723	4.477	5.021	4.281
Si	109.469	94.879	150.702	100.128	152.007	134.974	152.329	93.097
Sn	0.168	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Sr	434.051	474.349	321.065	397.209	684.145	975.395	1131.351	889.058
Tc	0.085	0.085	0.07	0.07	0.07	0.07	0.07	0.107
Ti	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
V	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Zn	0.231	0.273	0.174	0.189	0.339	0.567	0.672	0.598
Zr	1.808	0.658	0.299	0.45	0.505	0.753	0.953	0.603

Table D-7. Digested Slurry Activities for the Four Washes – Duplicate Measurements
Units: dpm/ml, though Uranium is mg/liter

	WashConc-1	WashConc-1	WashConc-2	WashConc-2	WashConc-3	WashConc-3	WashConc-4	WashConc-4
ADS#	3-135222	3-135223	3-135224	3-135225	3-135226	3-135227	3-135228	3-135229
Cs-137	4.20E+06	3.50E+06	6.51E+05	9.46E+05	7.68E+04	1.23E+06	9.95E+05	8.62E+05
Eu-154						4.31E+04		
Eu-155						2.10E+04		
Co-60								
Am-241	1.55E+04	1.50E+04	1.20E+04	1.45E+04	2.56E+04	3.57E+04	4.24E+04	3.46E+04
Cm-244	5.65E+03	6.14E+03	6.28E+03	5.66E+03	1.04E+04	1.37E+04	1.71E+04	1.58E+04
Pu-239	4.99E+02	5.34E+02	5.14E+02	4.96E+02	7.07E+02	1.17E+03	1.23E+03	1.11E+03
Pu-238	4.64E+02	5.48E+02	1.59E+03	4.41E+02	1.14E+03	9.85E+02	1.08E+03	1.50E+03
Sr-90	6.02E+06	7.70E+06	4.96E+06	6.02E+06	1.14E+07	1.64E+07	1.57E+07	
Total U	0.162	0.2108	0.1204	0.1361	0.1562	0.251	0.288	0.1743

Table D-8. Wash Filtrate ICP-ES Data – Duplicate Measurements
Units: mg/liter

	Wash-1	Wash-1	Wash-2	Wash-2	Wash-3	Wash-3	Wash-4	Wash-4
ADS #>	3-135214	3-135215	3-135216	3-135217	3-135218	3-135219	3-135220	3-135221
Al	122.457	107.351	46.765	50.932	27.741	26.262	13.287	13.792
B	0.265	0.237	0.078	0.109	0.05	0.05	0.05	0.05
Ba	0.020	0.02	0.02	0.02	0.022	0.02	0.025	0.02
Ca	3.530	3.163	1.956	1.952	1.475	1.365	1.002	0.966
Cd	0.547	0.487	0.243	0.268	0.123	0.121	0.068	0.054
Co	0.067	0.05	0.061	0.059	0.071	0.053	0.068	0.05
Cr	0.739	0.703	0.351	0.496	0.604	0.585	0.282	0.271
Cu	0.248	0.236	0.13	0.136	0.087	0.094	0.062	0.048
Fe	0.063	0.064	0.042	0.049	0.04	0.03	0.041	0.03
La	0.110	0.11	0.11	0.11	0.11	0.11	0.11	0.11
Li	0.020	0.02	0.02	0.02	0.024	0.02	0.025	0.02
Mg	0.010	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Mn	0.262	0.416	0.01	0.097	0.015	0.01	0.01	0.01
Mo	0.549	0.632	0.205	0.331	0.127	0.103	0.065	0.062
Na	2471.210	2151.436	1037.82	1173.648	549.071	555.784	260.836	269.969
Ni	3.634	3.292	1.654	1.727	0.834	0.898	0.383	0.441
P	15.876	14.407	5.154	5.827	2.199	2.364	0.739	1.062
Pb	1.760	1.816	1.14	1.293	0.691	0.596	0.477	0.398
Si	0.190	0.205	0.161	0.188	0.236	0.172	0.219	0.16
Sn	0.341	0.398	0.231	0.152	0.15	0.15	0.15	0.15
Sr	2.666	2.328	1.736	1.108	1.019	0.926	0.576	0.653
Ti	0.020	0.02	0.02	0.02	0.035	0.028	0.04	0.022
V	0.030	0.039	0.052	0.056	0.076	0.06	0.083	0.057
Zn	0.030	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Zr	0.115	0.26	0.04	0.049	0.064	0.054	0.076	0.046

Table D-9. Wash Filtrate Activities – Duplicate Measurements
Units: dpm/ml

	Wash-1	Wash-1	Wash-2	Wash-2	Wash-3	Wash-3	Wash-4	Wash-4
ADS #>	3-135214	3-135215	3-135216	3-135217	3-135218	3-135219	3-135220	3-135221
Cs-137	6.95E+06	6.85E+06	2.75E+06	3.30E+06	1.38E+06	1.40E+06	6.13E+05	7.21E+05
Eu-154								
Eu-155								
Co-60	1.76E+03	1.63E+03	5.29E+02	8.45E+02			1.57E+02	1.99E+02
Am-241	3.10E+02	2.22E+02	217	142	152	130	105	113
Cm-244	2.68E+02	2.00E+02	407	312	223	44.4	89.2	120
Pu-239	48.8	77.9	28.8	27.5	9.08	51.7	26.3	44.9
Pu-238	34.9	43.4	15.5	13.7	5.97	19.7	14.7	14.7
Tc-99	2.57E+04	1.47E+04		1.77E+04				
Sr-90	5.35E+04	4.52E+04	2.98E+04	2.97E+04	1.75E+04	1.71E+04	9.83E+03	1.00E+04

Table D-10. Flush Liquid Activities
Units: dpm/ml

	WaterFlush	WaterFlush	1 M Nitric	1 M Nitric	Final Water	Final Water
ADS #>	136882	136884	136885	136886	136887	136888
Sr-90	3.92E+06	3.84E+06	2.40E+06	3.39E+06	5.22E+05	7.13E+05
Am-241	8330	8050	6240	6030	1470	1380
Cm-244	3070	3300	2450	2450	584	581
Pu-239	290	251	232	172	80	67
Pu-238	323	163	192	159	76	156
Cs-137	2.00E+05	1.94E+05	2.92E+05	2.98E+05	9.74E+04	9.16E+04
Tc-99	1440	1760	1840	2020	1620	475
Eu-154	9.01E+03	8.97E+03	6.79E+03	6.78E+03	1.75E+03	1.31E+03

Table D-11. Flush Liquid Compositions, mg/liter

	WaterFlush	WaterFlush	1 M Nitric	1 M Nitric	Final Water	Final Water
ADS #>	882	884	885	886	887	888
Al	9.98	9.787	10.17	10.517	2.048	1.981
B	0.05	0.05	0.05	0.050	0.05	0.05
Ba	0.138	0.133	0.096	0.073	0.02	0.02
Ca	5.59	5.567	4.499	4.603	1.908	1.87
Cd	0.03	0.03	0.03	0.030	0.03	0.03
Ce	3.841	3.573	3.15	3.150	3.15	3.15
Co	0.05	0.05	0.05	0.050	0.05	0.05
Cr	1.972	1.907	1.479	1.494	0.308	0.272
Cu	0.03	0.03	0.03	0.030	0.228	0.223
Fe	1.057	1.042	1.018	1.038	0.304	0.276
La	0.2	0.165	0.11	0.110	0.11	0.11
Li	0.02	0.02	0.02	0.020	0.02	0.02
Mg	0.038	0.034	0.034	0.034	0.068	0.067
Mn	108.695	107.173	85.714	88.975	17.084	17.042
Mo	0.06	0.06	0.06	0.060	0.06	0.06
Na	78.572	77.186	98.868	103.003	41.249	41.138
Ni	0.546	0.479	0.501	0.424	0.07	0.07
P	1.716	1.629	1.407	1.407	0.263	0.451
Pb	0.54	0.492	0.373	0.480	0.28	0.28
Si	0.16	0.16	0.16	0.160	0.16	0.16
Sn	0.15	0.15	0.15	0.150	0.15	0.15
Sr	233.232	231.35	171.216	178.044	41.95	41.952
Ti	0.02	0.02	0.02	0.020	0.02	0.02
V	0.03	0.03	0.03	0.030	0.03	0.03
Zn	0.118	0.111	0.099	0.096	0.059	0.052
Zr	0.125	0.095	0.086	0.075	0.04	0.04

APPENDIX E

RAW FILTRATION DATA

The raw data from the filtration work is archived in this section.

Batch #1 Data

Ca Precipitation/Filtration Flux Data											
Large C precipitation/filtration Run #1											
11/10/99 - 11/12/99											
TMP=50 psig, 12.2 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	87	50	3.80	30	9.61	3.12	187.30	0.252	14.782	0.0050	4.288
5	85	50	3.76	20	9.18	2.18	130.72	0.176	10.316	0.0035	2.993
10	84	50	3.93	20	11.04	1.81	108.70	0.146	8.578	0.0029	2.488
20	85	50	3.98	20	13.17	1.52	91.12	0.123	7.191	0.0025	2.086
30	86	50	4.01	20	13.15	1.52	91.25	0.123	7.202	0.0025	2.089
40	86	50	3.86	20	13.95	1.43	86.02	0.116	6.789	0.0023	1.969
50	86	50	4.00	20	15.00	1.33	80.00	0.108	6.314	0.0022	1.831
60	84	50	4.07	20	17.42	1.15	68.89	0.093	5.437	0.0019	1.577
TMP=30 psig, 9.1 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	85	30	3.07	20	12.90	1.55	93.02	0.125	7.341	0.00417	3.549
5	85	30	3.00	20	15.33	1.30	78.28	0.105	6.178	0.00351	2.987
10	83	30	3.11	20	18.27	1.09	65.68	0.088	5.184	0.00294	2.506
20	83	30	3.03	20	19.62	1.02	61.16	0.082	4.827	0.00274	2.334
30	85	30	3.05	20	18.10	1.10	66.30	0.089	5.232	0.00297	2.530
40	86	30	3.00	20	20.12	0.99	59.64	0.080	4.707	0.00267	2.276
50	84	30	3.05	20	21.88	0.91	54.84	0.074	4.328	0.00246	2.093
60	84	30	3.06	20	20.82	0.96	57.64	0.078	4.549	0.00258	2.199
TMP=30 psig, 15.2 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	87	30	5.11	20	15.65	1.28	76.68	0.103	6.051	0.00344	2.926
5	87	30	5.06	20	21.11	0.95	56.85	0.076	4.486	0.00255	2.169
10	87	30	4.98	20	22.53	0.89	53.26	0.072	4.203	0.00239	2.032
20	83	31	5.04	20	27.39	0.73	43.81	0.059	3.458	0.00190	1.618
30	85	30	5.13	20	29.41	0.68	40.80	0.055	3.220	0.00183	1.557
40	83	30	5.14	20	31.50	0.63	38.10	0.051	3.006	0.00171	1.454
50	81	30	5.06	20	37.16	0.54	32.29	0.043	2.549	0.00145	1.232
60	84	30	5.13	20	30.88	0.65	38.86	0.052	3.067	0.00174	1.483
TMP=70 psig, 9.1 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	85	70	2.98	20	12.88	1.55	93.17	0.125	7.353	0.00179	1.523
5	81	70	2.98	20	19.83	1.01	60.51	0.081	4.776	0.00116	0.990
10	83	70	2.97	20	22.16	0.90	54.15	0.073	4.274	0.00104	0.885
20	83	70	2.88	20	23.51	0.85	51.04	0.069	4.028	0.00098	0.835
30	83	70	2.86	20	26.00	0.77	46.15	0.062	3.642	0.00089	0.755
40	82	70	2.92	20	29.52	0.68	40.65	0.055	3.208	0.00078	0.665
50	83	70	2.90	20	30.36	0.66	39.53	0.053	3.119	0.00076	0.646
60	85	70	2.93	20	29.41	0.68	40.80	0.055	3.220	0.00078	0.667
TMP=70 psig, 13.5 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	86	70	4.35	20	11.31	1.77	106.10	0.143	8.374	0.0020	1.735
5	87	70	4.30	20	17.21	1.16	69.73	0.094	5.503	0.0013	1.140
10	81	70	4.29	20	19.22	1.04	62.43	0.084	4.927	0.0012	1.021
20	87	70	4.51	20	19.96	1.00	60.12	0.081	4.745	0.0012	0.983
30	87	70	4.36	20	22.42	0.89	53.52	0.072	4.224	0.0010	0.875
40	86	70	4.34	20	23.49	0.85	51.09	0.069	4.032	0.0010	0.835
50	88	70	4.37	20	23.70	0.84	50.63	0.068	3.996	0.0010	0.828
60	88	70	4.41	20	26.40	0.76	45.45	0.061	3.587	0.0009	0.743

TMP=50 psig, 12.2 ft/s fluid velocity (Run #2)								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	86	50	3.83	20	16.92	1.18	70.92	0.095	5.597	0.0019	1.624
5	84	50	3.89	20	20.67	0.97	58.06	0.078	4.582	0.0016	1.329
10	86	50	3.94	20	25.88	0.77	46.37	0.062	3.659	0.0012	1.061
20	86	50	3.95	20	23.92	0.84	50.17	0.067	3.959	0.0013	1.148
30	86	50	3.91	20	25.58	0.78	46.91	0.063	3.702	0.0013	1.074
40	83	50	3.90	20	26.47	0.76	45.33	0.061	3.578	0.0012	1.038
50	84	50	3.85	20	27.38	0.73	43.83	0.059	3.459	0.0012	1.003
60	87	50	4.05	20	27.84	0.72	43.10	0.058	3.402	0.0012	0.987
TMP=30 psig, 12.2 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	80	30	4.04	20	29.86	0.67	40.19	0.054	3.172	0.0018	1.533
5	86	30	3.98	20	30.83	0.65	38.92	0.052	3.072	0.0017	1.485
10	86	30	4.13	20	35.18	0.57	34.11	0.046	2.692	0.0015	1.301
20	86	31	4.08	20	38.34	0.52	31.30	0.042	2.470	0.0014	1.156
30	86	30	4.19	20	40.44	0.49	29.67	0.040	2.342	0.0013	1.132
40	88	30	4.18	20	40.43	0.49	29.68	0.040	2.342	0.0013	1.132
50											
60											
TMP=50 psig, 9.1 ft/s fluid velocity (Run #2)								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	83	50	3.04	20	16.62	1.20	72.20	0.097	5.698	0.0019	1.653
5	84	50	3.12	20	26.27	0.76	45.68	0.061	3.605	0.0012	1.046
10	84	50	3.16	20	29.07	0.69	41.28	0.056	3.258	0.0011	0.945
20	84	50	3.15	20	32.36	0.62	37.08	0.050	2.927	0.0010	0.849
30	85	51	2.97	20	35.51	0.56	33.79	0.045	2.667	0.0009	0.758
40	79	50	3.16	20	44.48	0.45	26.98	0.036	2.129	0.0007	0.618
50	84	50	3.12	20	38.56	0.52	31.12	0.042	2.456	0.0008	0.712
60	84	50	3.04	20	40.73	0.49	29.46	0.040	2.325	0.0008	0.674
70	85	50	3.02	20	40.95	0.49	29.30	0.039	2.313	0.0008	0.671
TMP=30 psig, 6.6 ft/s fluid velocity								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	84	30	2.17	20	23.80	0.84	50.42	0.068	3.979	0.0023	1.924
5	82	30	2.23	20	35.84	0.56	33.48	0.045	2.642	0.0015	1.278
10	82	30	2.15	20	39.53	0.51	30.36	0.041	2.396	0.0014	1.158
20	84	30	2.29	20	41.53	0.48	28.89	0.039	2.280	0.0013	1.102
30	80	30	2.18	20	47.76	0.42	25.13	0.034	1.983	0.0011	0.959
40	84	30	2.20	20	47.78	0.42	25.12	0.034	1.982	0.0011	0.958
50	84	30	2.25	20	49.81	0.40	24.09	0.032	1.901	0.0011	0.919
60	84	30	2.19	20	52.16	0.38	23.01	0.031	1.816	0.0010	0.878
TMP=50 psig, 12.2 ft/s fluid velocity (Run #3)								Permeate	Permeate		
		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
0	85	53	3.75	20	17.60	1.14	68.18	0.092	5.381	0.0017	1.473
5	84	50	4.00	20	24.12	0.83	49.75	0.067	3.926	0.0013	1.139
10	82	50	4.15	20	27.30	0.73	43.96	0.059	3.469	0.0012	1.006
20	84	50	3.93	20	35.28	0.57	34.01	0.046	2.684	0.0009	0.779
30	84	50	4.01	20	30.92	0.65	38.81	0.052	3.063	0.0010	0.888
40	85	50	4.01	20	31.18	0.64	38.49	0.052	3.037	0.0010	0.881
50	84	50	4.00	20	31.16	0.64	38.51	0.052	3.039	0.0010	0.882
60	87	50	4.11	20	34.88	0.57	34.40	0.046	2.715	0.0009	0.788
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2			
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372					
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321					
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s										
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s										

Batch #2 Data

Large C Precipitation/Filtration Flux Data																
Large C precipitation/filtration Run #2, Large Batch #1																
11/13/99																
															Calculated	
TMP=50 psig, 12.2 ft/s fluid velocity									Permeate	Permeate				Adjusted	Wt.%	
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance	Filtrate	Filtrate	Insoluble	
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)	Produced (ml)	Produced (ml)	Solids	
															1.07	
8:05 AM	0	77	50	3.80	20	21.32	0.94	56.29	0.076	4.442	0.00151	1.289	56.3	22.2	1.11	
8:10	0:05	80	50	3.75	20	30.66	0.65	39.14	0.053	3.089	0.00105	0.896	238.6	208.8	1.50	
8:15	0:10	86	50	3.70	20	30.50	0.66	39.34	0.053	3.105	0.00106	0.901	196.2	166.4	1.81	
8:30	0:25	83	50	3.79	15	28.28	0.53	31.82	0.043	2.512	0.00086	0.729	533.8	510.8	2.77	
8:40	0:35	87	50	3.98	15	30.00	0.50	30.00	0.040	2.368	0.00081	0.687	309.1	286.6	3.31	
8:50	0:45	87	50	3.99	15	28.48	0.53	31.60	0.043	2.494	0.00085	0.723	308.0	285.1	3.84	
	BACKPULSE															
8:57	0:00	87	50	3.71	15	16.58	0.90	54.28	0.073	4.284	0.00146	1.243	54.3	25.7	3.89	
9:04	0:07	85	50	3.94	20	30.55	0.65	39.28	0.053	3.100	0.00106	0.899	327.5	297.6	4.45	
	Shutdown for product bottle change, NO BACKPULSE															
9:33	0:07	86	50	3.62	20	21.27	0.94	56.42	0.076	4.452	0.00152	1.292	56.4	22.3	4.49	
9:37	0:11	86	50	3.87	15	24.86	0.60	36.20	0.049	2.857	0.00097	0.829	185.2	161.2	4.79	
9:42	0:16	87	50	3.98	15	23.74	0.63	37.91	0.051	2.992	0.00102	0.868	185.3	160.8	5.09	
10:01	0:35	88	48	4.23	15	29.03	0.52	31.00	0.042	2.447	0.00087	0.739	654.7	631.9	6.28	
10:10	Stopped												279.0	279.0	6.8	(measured)
																Calculation assumes 1.26 g/ml slurry density and
												TOTAL =	3105.3	3058.5		800 ml CUF internal volume
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2						adjusted for		
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372								recycle during		
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321								readings and time		
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s													to fill tubing		
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s													to flowmeter		
													Estimated wt.% solids in feed		1.50	Assumed 2.4 L produces 1.5 wt.% solids in usual recipe
																Assumed same amt. solids produced with 2.4 L feed

Batch #3 Data

Large C Precipitation/Filtration Flux Data

Large C precipitation/filtration Run #3, Large Batch #2

11/15/99

TMP=50 psig, 12.2 ft/s fluid velocity

Time	Running Time (min)	Temp (F)	(P1-P2) TMP (psi)	F1 (gpm) Filter Flow	F2 vol. (ml) Filtrate Vol.	F2 time Time (sec)	F2 mL/sec	F2 ml/min	Permeate Flux GPM/ft*ft	Permeate Flux m3/(m2*day)	Permeance (gpm/ft2 psi)	Permeance m3/(m2*day*bar)	Filtrate Produced (ml)	Adjusted Filtrate Produced (ml)	Calculated Wt.% Insoluble Solids	
															6.80	(measured)
12:13 PM	0	85	53	3.77	15	22.60	0.66	39.82	0.054	3.143	0.00101	0.860	39.8	14.9	6.83	
12:18	0:05	83	50	3.83	15	32.47	0.46	27.72	0.037	2.188	0.00075	0.635	168.9	146.9	7.05	
12:25	0:12	86	48	3.98	15	35.81	0.42	25.13	0.034	1.983	0.00070	0.599	185.0	163.7	7.33	
12:36	0:23	86	50	3.92	15	33.90	0.44	26.55	0.036	2.095	0.00071	0.608	284.2	262.6	7.78	
12:40	Notice variation in pump sound. Slurry may be too thick.												265.5	265.5	8.23	
13:27	Restart to make product. Had to turn up pump a lot to restore flow and pressure															
13:30	Flow dropped to 3.4 gpm (air press = 90 psig, normally 60)															
13:41	0:14	88	51	4.25	15	49.12	0.31	18.32	0.025	1.446	0.00048	0.411	256.5	236.9	8.63	
Dropped flow to assess compressibility																
13:47	0:20	86	30	4.06	10	60.82	0.16	9.87	0.013	0.779	0.00044	0.376	93.0	80.6	8.77	(submitted sample, no data obtained)
Shut down.												TOTAL	1292.9			vs. ~1200 ml observed during changeover
HIGHCONC1 drawn from spigot. Filtrate #4 had 0.5 liter in bottle at this time.																
14:17	Restart after reloading fresh material and backpulsing once.														7.10	(unknown)
14:30	0:13	84	52	3.73	15	21.62	0.69	41.63	0.056	3.285	0.00108	0.916	541.2	515.8	7.97	
14:35	0:18	87	50	3.80	15	29.24	0.51	30.78	0.041	2.429	0.00083	0.705	181.0	158.3	8.24	
14:55	0:38	87	50	3.71	15	34.21	0.44	26.31	0.035	2.076	0.00071	0.602	263.1	241.5	8.66	
15:01	0:44	87	48	3.97	15	37.27	0.40	24.15	0.032	1.906	0.00068	0.576	151.4	130.3	8.88	
15:06	0:49	85	49	3.91	15	38.75	0.39	23.23	0.031	1.833	0.00064	0.543	118.4	97.6	9.04	
15:11	0:54	85	51	3.87	15	38.33	0.39	23.48	0.032	1.853	0.00062	0.527	116.8	95.9	9.21	
15:16	0:59	87	52	4.05	15	38.55	0.39	23.35	0.031	1.842	0.00060	0.514	117.1	96.2	9.37	(from Run 4 calculation)
																Calculation assumes 1.26 g/ml slurry density and
												TOTAL =	2781.8	2506.7		800 ml CUF internal volume
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2						adjusted for		
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372								recycle during		
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321								readings and time		
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s													to fill tubing		
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s													to flowmeter		
													Estimated wt.% solids in feed		1.36	Assumed 2.4 L produces 1.5 wt.% solids in usual recipe
																Assumed same amt. solids produced with 2.7 L feed
														(~800 ml of		(i.e., assume simple dilution effect)
														feed lost to		
														refill CUF)		

Batch #4 Data

Large C Precipitation/Filtration Flux Data

Large C precipitation/filtration Run #4, Large Batch #3

11/15/1999 - 11/16/1999

TMP=50 psig, 12.2 ft/s fluid velocity

Time	Running Time (min)	Temp (F)	(P1-P2) TMP (psi)	F1 (gpm) Filter Flow	F2 vol. (ml Filtrate Vol	F2 time Time (sec)	F2 mL/sec	F2 ml/min	Permeate Flux GPM/ft*ft	Permeate Flux m3/(m2*day)	Permeance (gpm/ft2 psi)	Permeance m3/(m2*day*bar)	Filtrate Produced (ml)	Adjusted Filtrate Produced (ml)	Calculated Wt% Insoluble Solids	
															9.37	
	Worked down old material, and finished adding Batch 4 material												233.5	233.5	9.81	
11:33 PM	0	85	50	3.93	15	25.61	0.59	35.14	0.047	2.773	0.00095	0.805	35.1	11.4	9.83	
11:39 PM	0:06	87	50	4.02	15	46.20	0.32	19.48	0.026	1.537	0.00052	0.446	327.7	307.9	10.41	
11:45 PM	0:12	87	47	3.98	15	47.24	0.32	19.05	0.026	1.504	0.00055	0.464	231.2	211.4	10.80	
11:57 PM	0:24	85	49	4.04	10	33.65	0.30	17.83	0.024	1.407	0.00049	0.417	221.3	206.8	11.19	
	Shutdown for evening															
	Restarted after loading ice															
10:35 AM	0:24	85	48	3.89	15	49.19	0.30	18.30	0.025	1.444	0.00051	0.436	18.3	0	11.19	
10:49 AM	0:38	85	51	4.02	10	32.41	0.31	18.51	0.025	1.461	0.00049	0.416	404.9	390.3	11.92	
12:01 PM	1:50	85	48	4.17	10	45.73	0.22	13.12	0.018	1.035	0.00037	0.313	348.0	334.7	12.55	
	Ran for production - Started filling Filtrate6 bottle (3:42 to 4:00)												236.2	236.2	12.99	
	Stopped for ice and restarted at 5:58 PM															
	Backpulsed 1X															
6:05 PM	0:00	84	50	3.51	15	42.34	0.35	21.26	0.029	1.678	0.00057	0.487	148.8	128.5	13.23	
6:13 PM	0:08	83	50	3.73	5	27.95	0.18	10.73	0.014	0.847	0.00029	0.246	128.0	120.3	13.46	
6:35 PM	0:30	86	50	3.87	5	26.54	0.19	11.30	0.015	0.892	0.00030	0.259	242.4	234.6	13.9	(measured)
	Shutdown and took some samples (filtrate?)															Calculation assumes 1.26 g/ml slurry density and
	Ran next for high solids concentration flux points (recycle mode presumably)															800 ml CUF internal volume
												TOTAL =	2575.3	2415.4		
														adjusted for		
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2						recycle during		
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372								readings and time		
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321								to fill tubing		
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s													to flowmeter		
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s															
													Estimated wt.% solids in feed		1.50	Assumed 2.4 L produces 1.5 wt.% solids in usual recipe
																Assumed same amt. solids produced with 2.4 L feed

High Insoluble Solids Concentration Run Data

Large C Precipitation/Filtration Flux Data												
Large C precipitation/filtration Run #4, Large Batch #3												
11/16/99												
After filtering 2 of 3 liters of solution, filter fluxes dropped very low (concentrated solids about 8-10X). Did test matrix like in TTP.												
TMP=70 psig, 12.2 ft/s fluid velocity									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (HH:MM)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
8:35 PM	0	86	70	3.90	10	29.51	0.34	20.33	0.027	1.605	0.00039	0.332
20:40	0:05	88	70	3.88	10	49.29	0.20	12.17	0.016	0.961	0.00023	0.199
20:45	0:10	85	70	3.72	10	53.38	0.19	11.24	0.015	0.887	0.00022	0.184
20:50	0:15	88	70	3.75	10	51.28	0.20	11.70	0.016	0.923	0.00022	0.191
20:55	0:20	86	70	3.88	10	56.41	0.18	10.64	0.014	0.839	0.00020	0.174
21:00	0:25	86	70	3.92	10	55.75	0.18	10.76	0.014	0.849	0.00021	0.176
21:05	0:30	86	70	3.93	10	57.17	0.17	10.50	0.014	0.828	0.00020	0.172
TMP=30 psig, 15.1 ft/s fluid velocity									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (HH:MM)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
9:17 PM	0	83	30	5.10	10	32.50	0.31	18.46	0.025	1.457	0.00083	0.704
21:22	0:05	88	30	5.20	10	41.43	0.24	14.48	0.019	1.143	0.00065	0.553
21:27	0:10	88	30	5.11	10	39.23	0.25	15.29	0.021	1.207	0.00069	0.584
21:32	0:15	88	30	5.09	10	43.40	0.23	13.82	0.019	1.091	0.00062	0.527
21:37	0:20	83	30	5.09	10	45.75	0.22	13.11	0.018	1.035	0.00059	0.500
21:42	0:25	86	30	5.13	10	42.17	0.24	14.23	0.019	1.123	0.00064	0.543
21:47	0:30	86	30	5.11	10	44.30	0.23	13.54	0.018	1.069	0.00061	0.517
TMP=50 psig, 9.1 ft/s fluid velocity									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (HH:MM)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
10:42 PM	0:00	85	50	3.25	10	37.67	0.27	15.93	0.021	1.257	0.00043	0.365
22:47	0:05	80	50	3.01	10	68.70	0.15	8.73	0.012	0.689	0.00023	0.200
22:52	0:10	86	50	3.15	10	73.50	0.14	8.16	0.011	0.644	0.00022	0.187
22:57	0:15	87	50	3.19	10	76.20	0.13	7.87	0.011	0.621	0.00021	0.180
23:02	0:20	86	50	3.06	10	76.11	0.13	7.88	0.011	0.622	0.00021	0.180
23:07	0:25	87	50	3.15	10	75.41	0.13	7.96	0.011	0.628	0.00021	0.182
23:12	0:30	86	50	3.16	10	80.12	0.12	7.49	0.010	0.591	0.00020	0.171
	Took rheology sample and HighConc2 sample.					(15.7, 13.3 = 14.5 wt.% avg. insoluble solids, measured)						
	Drained rig to best possible.											
	Refilled CUF with remainder of Batch 4											
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2				
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372						
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321						
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s											
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s											

Batch #5 Data

Large C Precipitation/Filtration Flux Data

Large C precipitation/filtration Run #5, Large Batch #4

11/17/99

TMP=50 psig, 12.2 ft/s fluid velocity									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
6:15 PM	0	87	50	3.70	10	17.33	0.58	34.62	0.047	2.732	0.00093	0.793
6:20 PM	0:05	83	50	3.80	5	17.26	0.29	17.38	0.023	1.372	0.00047	0.398
6:25 PM	0:10	87	50	3.88	5	17.55	0.28	17.09	0.023	1.349	0.00046	0.391
6:35 PM	0:20	85	50	3.87	5	17.41	0.29	17.23	0.023	1.360	0.00046	0.394
6:45 PM	0:30	87	50	3.85	5	18.47	0.27	16.24	0.022	1.282	0.00044	0.372
7:03 PM	0:48	86	50	3.89	5	19.37	0.26	15.49	0.021	1.222	0.00042	0.355
	Shutdown at 7:15, switched to Filtrate7 bottle											
7:33 PM	0:48	84	30	5.20	5	12.38	0.40	24.23	0.033	1.912	0.00109	0.925
7:38 PM	0:53	83	30	5.16	5	14.14	0.35	21.22	0.029	1.674	0.00095	0.810
7:43 PM	0:58	85	30	5.17	5	14.64	0.34	20.49	0.028	1.617	0.00092	0.782
7:53 PM	1:08	85	30	5.17	5	16.70	0.30	17.96	0.024	1.418	0.00081	0.685
8:03 PM	1:18	83	30	5.20	5	17.82	0.28	16.84	0.023	1.329	0.00075	0.642
	At 8:08, backpulsed 2X - filling for 2nd backpulse easier as indicated by filtrate											
	pressure better matching concentrate pressure better											
8:17 PM	1:18	86	40	5.15	5	10.18	0.49	29.47	0.040	2.326	0.00099	0.843
8:23 PM	1:24	86	40	5.15	5	13.71	0.36	21.88	0.029	1.727	0.00074	0.626
8:29 PM	1:30	86	40	5.17	5	13.48	0.37	22.26	0.030	1.756	0.00075	0.637
8:39 PM	1:40	86	40	5.07	5	15.23	0.33	19.70	0.026	1.555	0.00066	0.564
9:00 PM	2:01	86	40	5.16	5	16.46	0.30	18.23	0.025	1.438	0.00061	0.522
	Shutdown at 9:05 due to lack of ice											
	Discharged all concentrate to beakers for EndConc5 and Digest samples											
	Filled CUF with material from flask to leave 400 ml precipitated material											
	Made Batch 6 with 400 ml Batch 5 material + remaining concentrate + remaining filtrate to make 1 L											
	When turned on CUF, level dropped to transferred over ~300 ml more to CUF											
	Took remaining 700 ml for Batch 6											
						bars						
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2				
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				40 psig =	2.757898						
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				50 psig =	3.447372						
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s											
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s											

Batch #6 Data

Large C Precipitation/Filtration Flux Data

Large C precipitation/filtration Run #6, Large Batch #5

11/19/99

Starting with new batch + ~700 ml of mixed concentrate from Run #5

TMP=50 psig, 12.2 ft/s fluid velocity

Time	Running Time (min)	Temp (F)	(P1-P2) TMP (psi)	F1 (gpm) Filter Flow	F2 vol. (ml) Filtrate Vol.	F2 time Time (sec)	F2 mL/sec	F2 ml/min	Permeate Flux GPM/ft*ft	Permeate Flux m3/(m2*day)	Permeance (gpm/ft2 psi)	Permeance m3/(m2*day*bar)	Filtrate Produced (ml)	Adjusted Filtrate Produced (ml)	Calculated Wt.% Insoluble Solids	
Two backpulses before start															7.72	(measured)
8:38 AM	0	85	30	3.00	15	47.48	0.32	18.96	0.025	1.496	0.00085	0.723	19.0	0.0	7.72	
8:52	0:14	83	32	2.95	15	68.49	0.22	13.14	0.018	1.037	0.00055	0.470	96.3	78.0	7.88	
8:58	0:20	85	30	3.01	15	78.11	0.19	11.52	0.015	0.909	0.00052	0.440	172.6	154.8	8.20	
Shutdown to switch filtrate bottles (started Filtrate8)																
9:14	Restarted (no backpulse)															
9:19	0:25	84	32	3.02	5	28.52	0.18	10.52	0.014	0.830	0.00044	0.376	52.6	45.0	8.29	
9:27	0:33	85	30	2.90	5	31.73	0.16	9.45	0.013	0.746	0.00042	0.361	59.9	52.6	8.40	
9:32 Backpulse 2X					Noticed frequent adjustments to V1 (every few minutes at 9:19)								47.3	47.3	8.49	
9:39	Restarted															
9:41	0:02	85	30	5.06	10	29.58	0.34	20.28	0.027	1.601	0.00091	0.774	40.6	25.5	8.55	
9:49	0:10	86	30	4.95	10	31.86	0.31	18.83	0.025	1.486	0.00084	0.719	156.5	141.8	8.84	
9:54	0:15	83	30	5.12	10	33.28	0.30	18.03	0.024	1.423	0.00081	0.688	92.2	77.6	8.99	
10:00 Shutdown to change conditions																
10:04	Restart															
10:21	0:32	83	50	4.00	10	41.29	0.24	14.53	0.020	1.147	0.00039	0.333	247.0	233.4	9.47	
10:33	0:44	85	50	3.97	10	41.39	0.24	14.50	0.019	1.144	0.00039	0.332	174.2	160.5	9.80	
10:42 Shutdown to change conditions, optimize production																
10:51 Restart																
10:56	Reset to 20 psi, 5.5 gpm, no BP done															
11:00	0:48	83	20	5.42	5	68.86	0.07	4.36	0.006	0.344	0.00029	0.249	39.2	33.1	9.87	
This is too slow. Change back to 30 psi, ~5 gpm																
This done with filtrate valves closed.																
After 30 psid, 5 gpm set, we opened V4 and took flow point.																
11:05	0:53		30	5.02	5	42.43	0.12	7.07	0.010	0.558	0.00032	0.270	28.6	21.8	9.91	
We judged this too slow.													70.7	70.7	10.06	
11:15 We drained CUF to HIGHCONC3, put in more batch material into reservoir, put more ice in cooler.																
11:55 Backpulsed 1X																
11:59	Restart														8.11	(unknown)
12:01	0:02	82	30	5.09	10	30.22	0.33	19.85	0.027	1.567	0.00089	0.758	39.7	24.7	8.16	
12:15	0:16	86	30	5.09	10	30.36	0.33	19.76	0.027	1.560	0.00089	0.754	277.3	262.4	8.70	
12:45 Shutdown													592.9	592.9	9.91	(measured)
																Calculation assumes 1.26 g/ml slurry density and
												TOTAL =	2206.5	2022.0		800 ml CUF internal volume
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				20 psig =	1.378949		GPM = gallons/ft2						adjusted for		
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				30 psig =	2.068423								recycle during		
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				50 psig =	3.447372								readings and time		
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s													to fill tubing		
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s													to flowmeter		
														Estimated wt.% solids in feed	1.64	(700 ml feed/conc + 2.242 L from feed&precip agents)
														Batch #5 feed/conc solids (wt%)	5.20	(400 ml Batch #5 feed, 600 ml conc., both 1.26 g/ml)
														Batch #5 feed solids (wt%)	1.43	2.55 L diluted assuming same solids as 2.4 L
														Analytically measured Insol. Solids for concentrate (wt.%)	7.72	(from above)
														Estimated solids contribution by precipitation (wt%)	0.53	Assumed 2.4 L produces 1.5 wt.% solids in usual recipe
																Used 0.35 factor since 30% usual Sr and 40% usual MnO4 used
														(~800 ml of		
														feed lost to		
														refill CUF)		

Batch #7 Data

Large C Precipitation/Filtration Flux Data

Large C precipitation/filtration Run #7, Large Batch #6

11/19/1999 - 11/22/99

Time	Running Time (min)	Temp (F)	(P1-P2) TMP (psi)	F1 (gpm) Filter Flow	F2 vol. (ml Filtrate Vol.	F2 time Time (sec)	F2 mL/sec	F2 ml/min	Permeate Flux GPM/ft*ft	Permeate Flux m3/(m2*day)	Permeance (gpm/ft2 psi)	Permeance m3/(m2*day*bar)	Filtrate Produced (ml)	Adjusted Filtrate Produced (ml)	Calculated Wt.% Insoluble Solids	
6:00 PM	0	88	25	5.40	5	13.61	0.37	22.04	0.030	1.740	0.00119	1.009		-10.5	9.49	(9.91 wt.% measured)
18:05	0:05	84	25	5.10	5	18.03	0.28	16.64	0.022	1.313	0.00090	0.762	96.7	87.5	9.62	
18:10	0:10	85	26	5.31	5	17.03	0.29	17.62	0.024	1.390	0.00091	0.776	85.6	76.2	9.74	
18:15	0:15	85	25	5.40	5	19.33	0.26	15.52	0.021	1.225	0.00083	0.711	82.8	74.0	9.85	
18:30	0:30	86	24	5.28	5	19.51	0.26	15.38	0.021	1.214	0.00086	0.733	231.7	222.9	10.18	
6:34 PM Without stopping, changed conditions due to P1 gauge vibration																
18:35	0:35	84	29	5.03	5	17.41	0.29	17.23	0.023	1.360	0.00080	0.680	81.5	72.2	10.29	
18:45	0:45	84	30	5.14	5	17.23	0.29	17.41	0.023	1.374	0.00078	0.664	329.1	319.8	10.78	
19:04	1:04	84	30	5.10	5	18.14	0.28	16.54	0.022	1.305	0.00074	0.631	169.7	160.6	11.02	
Stopped to due lack of ice in cooler to cool pump.																
11:00 AM		Restarted after backpulsing 2X														
11:04	0:04	83	30	5.00	10	31.97	0.31	18.77	0.025	1.481	0.00084	0.716	75.1	60.4	11.11	
11:18	0:18	85	30	5.00	5	17.75	0.28	16.90	0.023	1.334	0.00076	0.645	249.7	240.5	11.48	
11:35	0:35	83	30	4.97	5	18.96	0.26	15.82	0.021	1.249	0.00071	0.604	278.2	269.2	11.88	
11:47	0:47	86	30	4.92	10	39.12	0.26	15.34	0.021	1.210	0.00069	0.585	187.0	173.1	12.14	
					~1600 ml left in Erlenmeyer flask											
12:52 Restarted, no backpulse																
12:53	0:47	84	29	5.06	5	18.21	0.27	16.47	0.022	1.300	0.00076	0.650	16.5	7.4	12.16	
13:04	0:58	84	30	5.01	5	19.57	0.26	15.33	0.021	1.210	0.00069	0.585	174.9	166.1	12.41	
13:15	Shutdown for window maintenance												168.6	168.6	12.66	
13:33	Restart with no backpulse				~1100 ml left in Erlenmeyer											
13:55	1:31	85	30	4.95	5	22.28	0.22	13.46	0.018	1.063	0.00060	0.514	296.2	287.9	13.10	
14:29	2:05	85	30	4.92	5	23.28	0.21	12.89	0.017	1.017	0.00058	0.492	448.0	439.8	13.76	
15:00	2:36	81	30	4.92	5	27.48	0.18	10.92	0.015	0.862	0.00049	0.417	369.0	361.2	14.31	
3:07 PM Slowed for backpulse				~700 ml left in Erlenmeyer at 3:00 PM									76.4	76.4	14.42	
		Looks like filter is clogged (slurry very thick), as sight glass level did not appear to drop														
3:15 PM	completed 2 backpulses. Stopped for ice.															
3:30 PM	Restarted				~500 ml left in Erlenmeyer flask											
15:32	0:02	84	30	5.09	5	18.97	0.26	15.81	0.021	1.248	0.00071	0.603	31.6	22.7	14.46	
15:37	0:07	82	29	5.06	5	25.28	0.20	11.87	0.016	0.937	0.00055	0.468	69.2	61.2	14.55	
16:04	0:34	87	30	4.91	5	26.21	0.19	11.45	0.015	0.903	0.00051	0.437	314.7	306.9	15.01	
16:09	Shutdown				Erlenmeyer emptied at 4:01 PM								57.2	57.2	15.1	(measured)
																Calculation assumes 1.26 g/ml slurry density and
												TOTAL =	3889.5	3701.2		800 ml CUF internal volume
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2						adjusted for		
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				50 psig =	3.447372								recycle during		
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				70 psig =	4.826321								readings and time		
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s													to fill tubing		
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s													to flowmeter		
													Estimated wt.% solids in feed		1.21	Assumed 2.4 L produces 1.5 wt.% solids in usual recipe
																Assumed same amt. solids produced with 3.1 L feed
																(i.e., assume simple dilution effect)

Wash Run Data

Wash Run Filter Flux Data												
Wash Runs 1-4												
11/29/1999 - 11/30/99												
Run 1									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
Pumped in ~200 ml of Wash solution, backpulsed 1X at 3:43 PM.												
3:46 PM	0	83	30	5	5	12.51	0.40	23.98	0.032	1.893	0.00108	0.915
15:51	0:05	86	30	4.95	5	18.53	0.27	16.19	0.022	1.278	0.00073	0.618
15:56	0:10	85	30	4.98	5	14.51	0.34	20.68	0.028	1.632	0.00093	0.789
16:01	0:15	87	30	5.04	5	12.59	0.40	23.83	0.032	1.881	0.00107	0.909
16:06	0:20	84	30	5.02	5	13.92	0.36	21.55	0.029	1.701	0.00097	0.822
16:11	0:25	84	30	5.16	5	11.86	0.42	25.30	0.034	1.996	0.00113	0.965
4:12 PM Stopped run as produced 600 ml in WASH1 bottle, took samples.												
Run 2									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
6:23 PM Backpulsed 1X												
6:26 PM	0	86	30	4.93	5	9.39	0.53	31.95	0.043	2.521	0.00143	1.219
18:32	0:06	86	30	5.06	5	8.71	0.57	34.44	0.046	2.718	0.00154	1.314
18:37	0:11	83	30	5.00	5	7.95	0.63	37.74	0.051	2.978	0.00169	1.440
18:42	0:16	84	30	5.01	5	8.42	0.59	35.63	0.048	2.812	0.00160	1.359
18:47	0:21	84	30	4.96	5	8.01	0.62	37.45	0.050	2.956	0.00168	1.429
6:48 PM Shutdown. Made 600 ml filtrate. Sampled system next morning.												
Run 3									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
12:54 PM Backpulsed 1X												
12:57 PM Established flow, but had to shutdown as peristaltic was not working. Tried to clean sight glass of reservoir to no avail.												
PUMP VERY HARD TO RESTART												
1:31 PM Reestablished flow conditions												
1:33 PM	0	88	30	4.95	5	5.53	0.90	54.25	0.073	4.281	0.00243	2.070
13:38	0:05	85	28	5.15	5	7.71	0.65	38.91	0.052	3.071	0.00187	1.591
13:43	0:10	83	30	5.01	5	6.85	0.73	43.80	0.059	3.456	0.00196	1.671
13:48	0:15	86	29	4.72	5	8.31	0.60	36.10	0.049	2.849	0.00167	1.425
1:52 PM Shutdown. Finished making 600 ml filtrate												
Run 4									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
4:17 PM Backpulsed 2X (pump stopped moving during first backpulse)												
4:21 PM Established flow conditions. Tried TMP=50 PSI, 12.2 ft/s but pump labored hard. Set as below.												
4:24 PM	0	81	41	3.83	5	9.54	0.52	31.45	0.042	2.482	0.00103	0.878
16:28	0:04	84	39	4.16	5	10.05	0.50	29.85	0.040	2.356	0.00103	0.876
16:34	0:10	86	39	4.19	5	10.12	0.49	29.64	0.040	2.340	0.00102	0.870
16:39	0:15	86	39	4.10	5	9.94	0.50	30.18	0.041	2.382	0.00104	0.886
16:44	0:20	85	38	4.00	5	11.35	0.44	26.43	0.036	2.086	0.00094	0.796
16:50	0:26	82	39	4.09	5	9.85	0.51	30.46	0.041	2.404	0.00105	0.894
4:52 PM Made 600 ml wash filtrate and stopped run.												
CUF pump did not start when tried to empty concentrate from CUF (tried up to ~85-90 psi air pressure).												
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				30 psig =	2.068423		GPM = gallons/ft2				
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				40 psig =	2.757898						
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				50 psig =	3.447372						
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s											
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s											

Post Filtration Clean Water Flux Data

Post-Run Clean Water Flux Data												
Post-run Clean Water Runs 1-4												
12/13/1999 - 12/16/1999												
Run 1									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
	Backpulsed 1X before run (12/15)											
10:07 AM	0:00	77	15	4.26	20	4.71	4.25	254.78	0.343	20.107	0.02285	19.442
10:12 AM	0:05	81	15	4.10	20	10.00	2.00	120.00	0.161	9.470	0.01076	9.157
10:17 AM	0:10	85	15	4.12	20	10.75	1.86	111.63	0.150	8.810	0.01001	8.518
10:22 AM	0:15	85	15	4.15	20	11.34	1.76	105.82	0.142	8.351	0.00949	8.075
10:27 AM	0:20	88	15	4.14	20	14.28	1.40	84.03	0.113	6.632	0.00754	6.413
Run 2									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
	Backpulsed 2X before run (12/16)											
2:40 PM	0:00	78	10	4.10	20	8.71	2.30	137.77	0.185	10.873	0.01853	15.770
2:45 PM	0:05	82	10	4.11	20	14.18	1.41	84.63	0.114	6.679	0.01138	9.687
2:50 PM	0:10	86	10	4.13	20	14.53	1.38	82.59	0.111	6.518	0.01111	9.453
2:56 PM	0:16	84	10	4.13	20	15.53	1.29	77.27	0.104	6.098	0.01039	8.845
3:01 PM	0:21	83	10	4.11	20	16.40	1.22	73.17	0.098	5.775	0.00984	8.375
3:06 PM	0:26	84	10	4.12	20	15.96	1.25	75.19	0.101	5.934	0.01011	8.606
Run 3									Permeate	Permeate		
	Running		(P1-P2)	F1 (gpm)	F2 vol. (ml)	F2 time	F2	F2	Flux	Flux	Permeance	Permeance
Time	Time (min)	Temp (F)	TMP (psi)	Filter Flow	Filtrate Vol.	Time (sec)	mL/sec	ml/min	GPM/ft*ft	m3/(m2*day)	(gpm/ft2 psi)	m3/(m2*day*bar)
3:17 PM	0:00	84	20	4.11	20	5.21	3.84	230.33	0.310	18.177	0.01549	13.182
3:22 PM	0:05	82	20	4.04	20	7.21	2.77	166.44	0.224	13.135	0.01119	9.525
3:28 PM	0:11	84	20	4.06	20	7.68	2.60	156.25	0.210	12.331	0.01051	8.943
3:32 PM	0:15	84	20	4.01	20	7.93	2.52	151.32	0.204	11.943	0.01018	8.661
3:38 PM	0:21	79	20	3.99	20	8.40	2.38	142.86	0.192	11.274	0.00961	8.176
3:42 PM	0:25	83	20	4.01	20	8.03	2.49	149.44	0.201	11.794	0.01005	8.553
	F1 = 2.2 gpm = 6.6 ft/s = 2.01 m/s				20 psig =	1.378949		GPM = gallons/ft2				
	F1 = 3 gpm = 9.1 ft/s = 2.77 m/s				15 psig =	1.034212						
	F1 = 4 gpm = 12.2 ft/s = 3.72 m/s				10 psig =	0.689474						
	F1 = 4.5 gpm = 13.5 ft/s = 4.11 m/s											
	F1 = 5 gpm = 15.2 ft/s = 4.63 m/s											