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FILTRATION OF TANK 48H CONTENTS WITH A CELLS UNIT FILTER (U)

SUMMARY

This report documents the design, operation and results from tests using a small crossflow filter unit with Tank 48H material. A Cells Unit Filter (CUF), with a crossflow filter 1/3000 of the area of a filter used in the In-Tank Precipitation (ITP) process, was used to demonstrate filtration, decontamination, and washing of the material. The CUF was found to be useful as a predictor of plant scale-filter flux. Its versatility allowed the simulation of a slurry washing cycle to determine nuclide retentions. The unit ran well for the entire campaign and the following results were obtained:

* Filtrate was shown to meet Process Requirements for the analytes that were measured.

* Half-hour average filter fluxes for the unit using Tank 48H slurry ranged from 0.1 to 0.5 gpm/ft^2 at transmembrane pressures of from 5 to 25 psid. ITP produced comparable fluxes.

* Addition of 0.24 g/L of monosodium titanate caused filter fluxes to drop by 25%.

* Addition of a total of 0.48 g/L of monosodium titanate caused fluxes to drop by 25% from previous values and a total of 40% from the case where none was added.

* Cesium, initially at a level of 200,000 microCuries/L in the slurry, was reduced to between 1.5 and 14 microCuries/L in the filtrate.

* Plutonium loss to the filtrate was less than 1.9% of system inventory during a simulated slurry washing cycle.

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INTRODUCTION

The In-Tank Precipitation (ITP) Filtration building will be used to dewater and wash High Level Waste precipitate prepared in Tank 48H. The tank contained salt solution from Tank 38H along with material left from the 1983 In-Tank Salt Decontamination Demonstration.

High Level Waste Engineering (HLWE) requested Interim Waste Technology (IWT) to investigate filterability of High Level Waste precipitate in Technical Issue ITP-TI-271 as defined in Technical Task Request HLE-TTR-95049.[1] The Technical Task Plan outlines the strategy for this investigation.[2]

Samples from Tank 48H were taken to demonstrate the crossflow filtration of tetraphenylborate precipitate slurry made from these samples.[3] This demonstration involved measuring filter flux as a function of transmembrane pressure. This test is of interest because the feed will be actual ITP waste rather than a simulant. This work represents a best effort attempt to determine the difficulty of plant filter operation before the plant is started.

DESCRIPTION OF THE FILTER UNIT

A Cells Unit Filter (CUF) system was designed to perform remote filtration experiments with High Level Waste. A diagram of the unit is shown in Figure 1. The design drawings[4,5] incorporated the following constraints.

* Materials and instruments must be radiation-resistant. The use of Teflon and solid state electronics within the Shielded Cell was avoided. Materials included in the rig were stainless steel, EPDM rubber, ceramic, and glass.

* The sample volume must be as small as possible since acquisition of radioactive samples is difficult and expensive. The unit uses 400 mL of feed.

* The filter feed pump must deliver up to 30 psi of pressure at flows up to 5 gpm. It must be low shear to handle the precipitate slurry. A Moyno SP-23203 progressive cavity pump (stainless steel housing and EPDM stator) was selected.

* The pump driver had to provide sufficient power at adjustable rates. A Gast 1.75 hp vane air motor was selected.

* The rig had to be operable with Cells manipulators. Controls faced the observation window. Cells personnel provided recommendations for the design.

* Flow meters with wide operating ranges were required, and had to maintain calibration where liquid density might change. A magnetic flowmeter was used to measure slurry flow. A fill-and-drain graduated tube was used to measured filtrate flow.

EXPERIMENTAL

Filtration tests were performed with (1) feed slurry simulant to test the unit and (2) High Level Waste precipitate from Savannah River Tank 48H. Solids concentrations in the simulants were 1 and 8 wt %. Table 1 provides the recipe for the simulant. Table 2 provides the analysis and recipe for the Tank 48H composite sample. This recipe follows the calculation used for preparing ITP feed.[6]

The testing followed several segments, each of which is covered below.

Initial Testing

The CUF was operated with nonradioactive feed in a chemical hood to check operation and to obtain filter performance with a simulant. Simulants at 1 and 8 wt % were filtered. Half-hour data points were taken as follows.[7]

* Clean water fluxes were measured before slurry was added to the new unit.
* Slurry flow and pressure on the filter tubeside were set.
* The filter was backpulsed twice.
* Filtrate flow was established after the second backpulse. The half-hour timer was started and the first data point was recorded.
* Filtrate flow, slurry flow, tube and shellside pressures, and temperature were measured at five minute intervals for a half hour (total of 7 data points).
* The backpulse system was refilled with filtrate after the last data of the half-hour period was taken.
* New slurry flow and pressure conditions were set. After backpulsing twice with an initial pressure 30 psi above tubeside pressure, the next half-hour data set was obtained.
* The filter was laid up (i.e., slurry was left in the unit within a test phase).

Shielded Cells Operation - Filter Flux

The CUF was thoroughly flushed with water, 2 wt % oxalic acid and 2 wt % sodium hydroxide before it was inserted in Shielded Cell 10 of 773-A. Clean water flux was taken after the CUF was installed in the cell. The CUF was then used to obtain filtration data using 516 g (428 mL) of the precipitated Tank 48H sample. The slurry reservoir was judged to be about 2/3 full using a telescope and mirror.

Filtration data was taken under procedure IWT-OP-088.[8] The testing essentially followed the steps given in the Initial Testing section.

Shielded Cells Operation - Washing

The customer requested that an ITP washing cycle be simulated. The primary goal was to provide filtrate that could be analyzed for plutonium. Plutonium removal to meet Saltstone limits was to be verified. Table 3 gives the sequence of chemical additions. The cycle was patterned after the full scale mass balance (Case #4, Table IX of the reference, Corrosion Inhibitor Additions During Tank 48H Washing).[9]

Shielded Cells Operation - Monosodium Titanate Addition

Monosodium titanate (MST) was used in two test series. Goals of the work were to (1) determine the effect of MST on filtration fluxes, and (2) to measure any changes in plutonium concentration in the filtrate.

Each test series followed the same order: the filter was run to obtain two half-hour data points. The MST was then added and several more data points were taken at transmembrane pressures identical to those taken before the addition. The change in performance was thus expected to be a function of the addition rather then changes that might occur with filter layup.

PAST FILTRATION WORK WITH RADIOACTIVE MATERIALS

Filtration testing with radioactive materials for the In-Tank Precipitation process was first done by Lee and Kilpatrick.[10,11] An 18-inch long, 0.5 micron Mott filter tube of 0.375 inch inner diameter was used in a small system functionally similar to the CUF. A diaphragm pump was used was used to minimize shear. Use of a centrifugal pump had been found to cut fluxes from 0.3 to 0.1 gpm/ft^2. Feed was made from 10% Tank 37 and 90% Tank 38 supernate. Sodium titanate was added to a concentration of 0.5 g/L. Total slurry solids was about 1 wt %. Optimum flux at 5 to 8 minute backpulse duration was obtained at a slurry velocity of 6.5 ft/s. Flux was 0.33 gpm/ft^2 at a transmembrane pressure of 31 psid and a fluid velocity of 3.9 ft/s.

The Salt Decontamination Demonstration in Tank 48H included both nonradioactive startup and radioactive operation with Tank 24H material.[12,13] The 203 ft^2 filter bundle described previously was used to concentrate and wash the slurry. Fluxes of 0.12 to 0.15 gpm/ft^2 were obtained with the diaphragm pump and fluxes of 0.12 to 0.17 gpm/ft^2 were obtained with the centrifugal pump. Filtration of solids from 0.67 to 7.5 wt % was accomplished during the program.

ITP has operated with both nonradioactive and radioactive feed. ITP filters #1 and #3 were tested using nonradioactve feed in a temporary facility.[14] Feed from an 11,000 gal (working volume) tank could be recirculated to a plant filter using a low shear centrifugal pump. Filtrate could be recycled and was also used to fill the backpulse tank. Filter #3 was run in constant flux mode with slurry concentrations from 1.5 to 10 wt %. Data for filter #1 at clean water, 1.9 and 3.0 wt % slurry are available.[15] Filter #3 ran for over 30 hours without backpulsing using 1.5 wt % slurry and providing a flux of 0.27 gpm/ft^2.

RESULTS AND DISCUSSION

Filtration Tests

Data from the nonradioactive and radioactive filtration tests are shown in Figures 2 through 6. Before slurry testing, clean water fluxes were taken to indicate the cleanliness of the filter and flow system. Clean water flux was taken with the CUF using the new Mott filter, and again once the unit was placed in the Shielded Cell. Figure 2 shows that once the filter was used, cleaning restored the clean water flux data to about half the original slope. The Parallel Rheology Experimental Filter (PREF) data are shown for comparison. The slope of the used CUF filter water flux line is slightly greater than that of the used PREF filter.

Figure 3 shows CUF half-hour average flux data for all simulants. Time series data appear on Figures 4 and 5. The flux at a tubeside velocity of 4 ft/s looks slightly lower than that of the 3 ft/s data in Figure 3. These data are not offered as evidence for the historical finding that 3 ft/s is the optimum slurry velocity for ITP.[16,17] The 4 ft/s data were taken immediately after the 3 f/s data. The 4 f/s data may be lower because of a small amount of filter fouling over time or because of experimental uncertainties. The 8 wt % fluxes are one-third of the 1 wt % fluxes, the relative change being in agreement with past work.[18]

Figures 4 and 5 show that the flux decline over time within the half-hour test segments is similar for 1 wt % and 8 wt % slurry. The initial flux is typically twice the half-hour average flux. The decline over the first 5 minutes is substantial, and data appear to come to a steady state with much slower declines for the rest of the segment.

Figures 5 and 6, respectively, show a comparison of the time series and average flux behaviors of 1 wt % simulant with Tank 48H slurry. Figure 5 shows the CUF filter gave high fluxes on initial startup (round points) relative to simulant at 3 ft/s. Data are presented in chronological order; note the randomization of transmembrane pressure across the figure. The filter performance appears to decline over the first day of running Tank 48H material; note in both Figures 5 and 6 that the flux drops when transmembrane pressure (TMP) is increased from 20 to 24.5 psid. Figure 6 shows that the repeated TMP=15 psid point is much lower than the initial point. "Second day" data in Figure 5 show that the flux with Tank 48H material declines to values found for the simulant at 1 wt %. The overall observation is that the Tank 48H material gives high fluxes initially, taking hours of filter operation before the flux declines to values seen with simulant. If simulant had a time-dependent fouling effect on the filter, it was fast such that the level of filter fouling was constant for simulant runs.

Titanate Addition

Monosodium Titanate (MST) additions were tested to simulate the use of this material in Tank 48H.[19,20] The initial titanate addition was made before the washing cycle (see Table 3). Titanate, 0.24 g (dry basis)/L from Optima Drum #3, was added to the CUF inventory. Figure 7 and Table 4 show the effect on flux before and after the addition. Average flux decline caused by the addition was about 25%. This is not as dramatic as the 50% flux drop seen in work with simulant,[16] though the relative amount added here is smaller. Table 2 indicates that the composite sample had 0.361 g/L prior to the addition, that coming from the 1983 Salt Decontamination Demonstration.

A second titanate addition was made to study a higher level of MST. A 0.47 g/L total addition was targeted and 0.48 g/L was achieved. The level was achieved considering that 0.24 g/L had already been added. The new MST had been obtained from the 2 production lots to be used in Tank 48H, and it was mixed in proportion to the amounts to be added.

A relative flux decline was measured with this second addition. Unfortunately, this work was requested after the washing cycle had been completed - salt content was not as high as that of the Tank 48H contents. Figure 7 shows that the reduction of sodium by washing balances the effect of the first titanate addition. Reduction of the sodium level by washing reduced viscosity so that filter fluxes after the second addition were thought to be higher than they would have been at the initial salt concentration. Washing also removed some of the precipitated sodium tetraphenylborate solids in the slurry. Thus, only relative changes in filter flux are reported.

Washing Cycle

ITP requested a washing cycle be simulated to determine the effect of slurry washing on plutonium and cesium decontamination factor (DF). There was concern that plutonium might not be removed in the presence of tetraphenylborate alone, but that titanate was required. The 1982 Shielded Cells experiments and the 1983 Salt Decontamination Demonstration indicated that "alpha bleed-through" would not occur.[11,12] Nonetheless, the Cells Unit was used to confirm this.

The washing cycle shown in Table 3 is a volume-scaled process.[9] Figures 8, 9, and 10 show that the washing cycle did well in matching calculated values from the reference. Additions of inhibitor and wash water (matched by removal of filtrate) did well in keeping sodium, nitrate, and nitrite near anticipated values. Figure 9 uses one-third of the nitrate level given in the reference because nitrate in the composite sample was one third of that assumed in Walker's calculations. Sodium and nitrite in the composite sample matched Walker's assumptions.

Table 5 shows that plutonium was at detection limits in the filtrate. Results of this work are also presented elsewhere and show that less than 1.9% of the plutonium in Tank 48H would be expected to be lost during washing.[21]

Table 6 shows that cesium at all times remained well under the Saltstone acceptance limit of 100 μCi/L. Cesium level was expected to rise during washing because of a reduction in NaTPB that followed the sodium reduction. The level was estimated to average 36 μCi/L in Walker's calculation, but that calculation assumed higher solids and cesium loadings at the end of the concentration step.

Table 7 provides the inductively-coupled plasma mass spectroscopy (ICP-MS) results for the filtrates where available. These data are provided for future information, anticipating questions that may arise about the filtration behavior of various isotopes. Neptunium, mass 237, acts as a soluble species that washes out. Mass 238 appears to act in a similar fashion. Mass 239, which would include plutonium, is at the detection limit. Therefore, conclusions about the behavior of mass 239 isotopes cannot be drawn.

Comparison of CUF and ITP Filtration Performance

Figure 11 shows a comparison of CUF and ITP filter permeances where half-hour average data were available. The CUF and plant permeances using 1 wt % radioactive slurry were in good agreement under these conditions. CUF permeance with 1 wt % simulant having 0.21 g/L MST and 0.4 g/L sludge was about half of that from the ITP startup tests with 1.5 wt % simulant. A comparison of simulant data from the CUF at 8 wt % with ITP data at 9 wt % is favorable. The lower fluxes that the 1 wt % simulant provides relative to Tank 48H slurry at a range of transmembrane pressures is illustrated in Figure 6. It must be noted that some decline in filter performance was noted with time as seen in Figures 5 and 6. The data from Tank 48H material appear to trend downward to the values provided by the simulant at the end of two days of testing. However, Tank 48H slurry always demonstrated fluxes that were as good as or better than those of the simulant at similar wt %.

Comparisons of these and other small scale filter data obtained in support of ITP will be the subject of a future report.

Some experiments, especially those of the early 1980's, were not included in this comparison because they were run with 5-minute backpulsing frequencies. Half-hour average fluxes were not available. Comparisons with half-hour averages are considered more reliable because of the significant flux decline that filters exhibit in the first 5 minutes or so. The current work shows that with a permeance of 0.013 gpm/ft^2/psid at 1 wt %, the CUF data predicts that the ITP filter will meet its acceptance requirement flux of 0.25 gpm/ft^2 with a TMP of 19 psid.[14] At 8 wt %, the permeance of 0.004 gpm/ft^2/psid will allow the ITP filter to meet its acceptance requirement flux of 0.016 gpm/ft^2 at a TMP of 4 psid.

CONCLUSIONS

- Filtration with Tank 48H material provided fluxes higher than or as good as those of a 1 wt % simulant.

- While the CUF experiment was not expected to be an absolute indicator of filter flux, the CUF data clearly show that the ITP filter should meet its expected performance criteria with the batch 1 contents of Tank 48H.

- Addition of a total of 0.48 g/L of monosodium titanate caused fluxes to drop by 25% from previous values and a total of 40% from the case where none was added.

- The In-Tank Precipitation chemistry and filtration provided cesium removal to values under the Saltstone limit for both filtration and washing.

- Less than 1.9% of the plutonium inventory in the CUF slurry passed through the filter during washing.

- The 1/3000 scale CUF filter gave permeances that were reasonable when compared with ITP filter data.

QUALITY ASSURANCE

The experimental work is recorded in Notebook WSRC-NB-94-82. A technical task plan and QA plan were approved.[2,22]

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FIGURE 1. CELLS UNIT FILTER

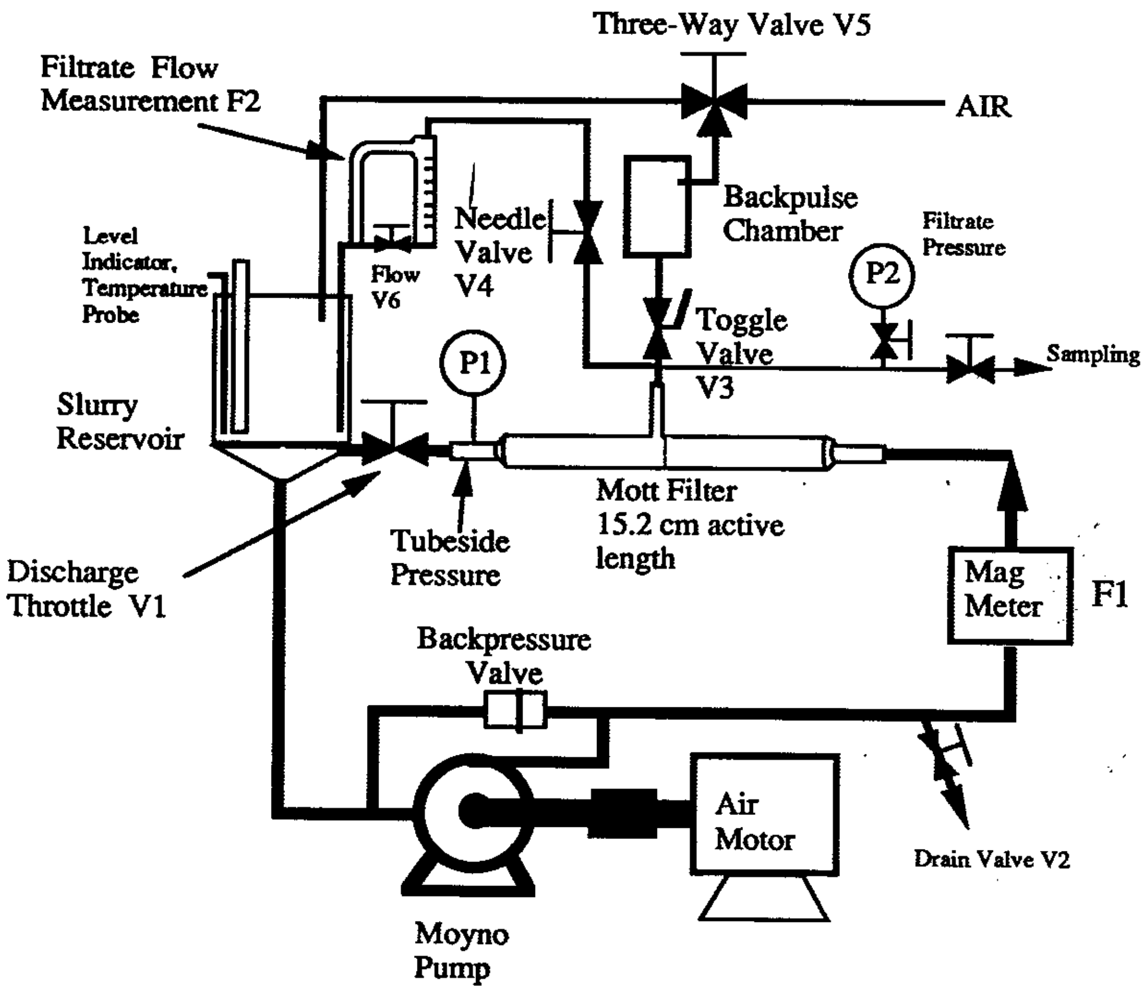


Figure 2. Clean Water Flux Data

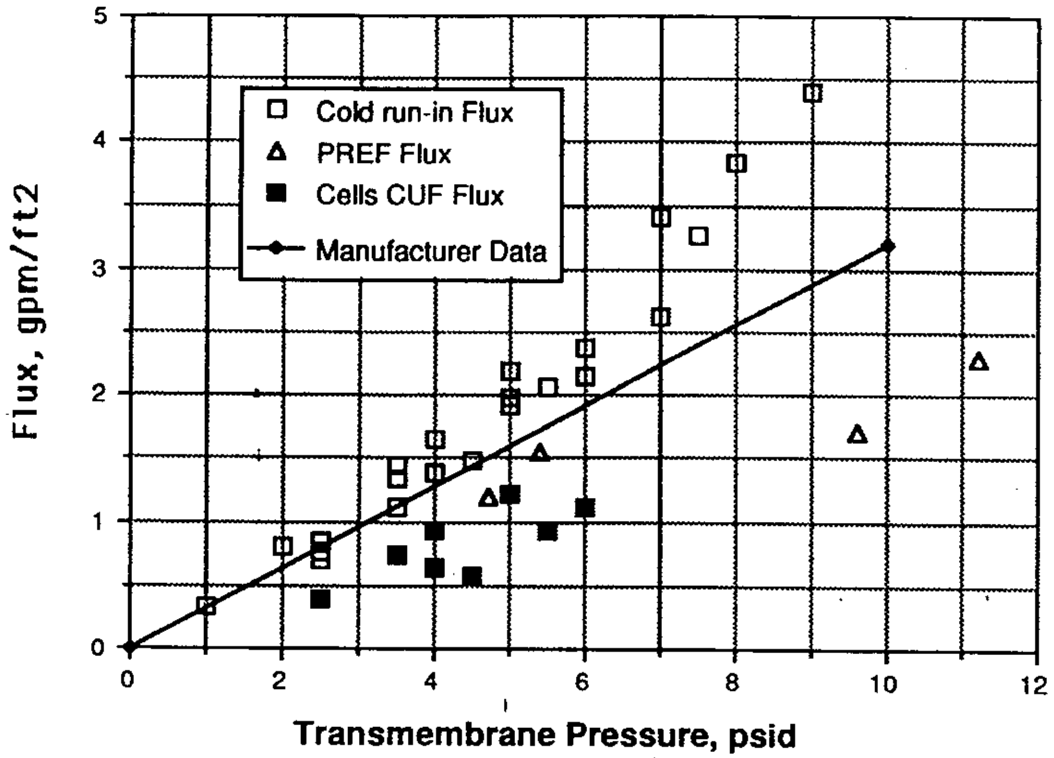
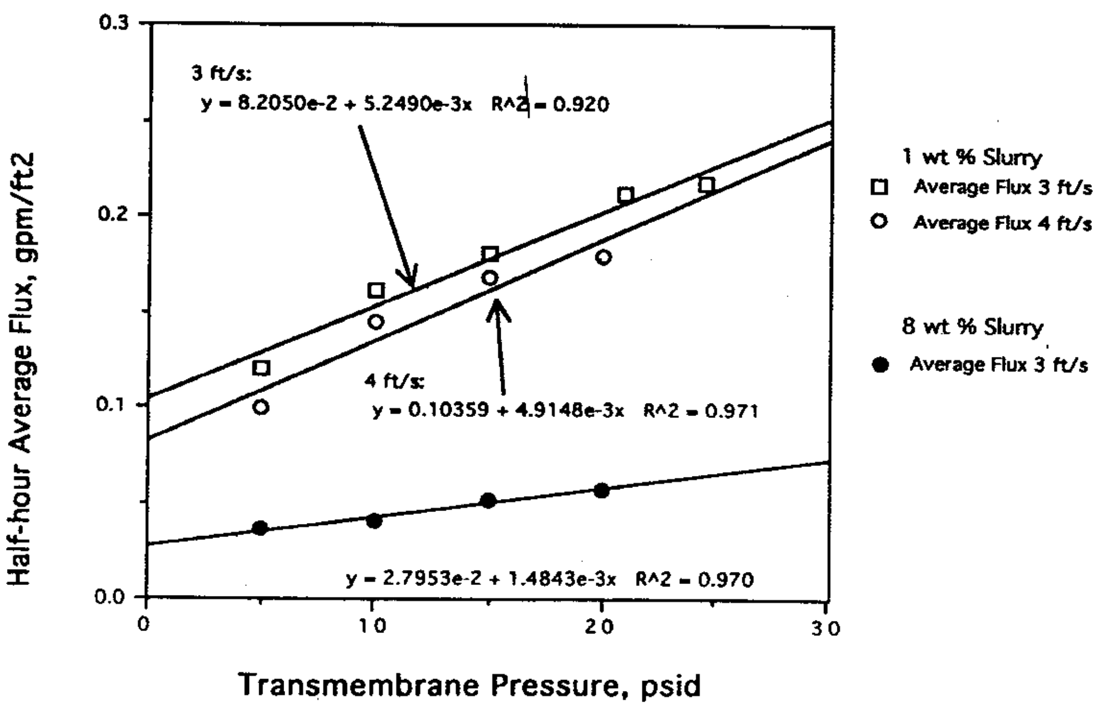


Figure 3. Flux Data with 1 and 8 wt % Simulant



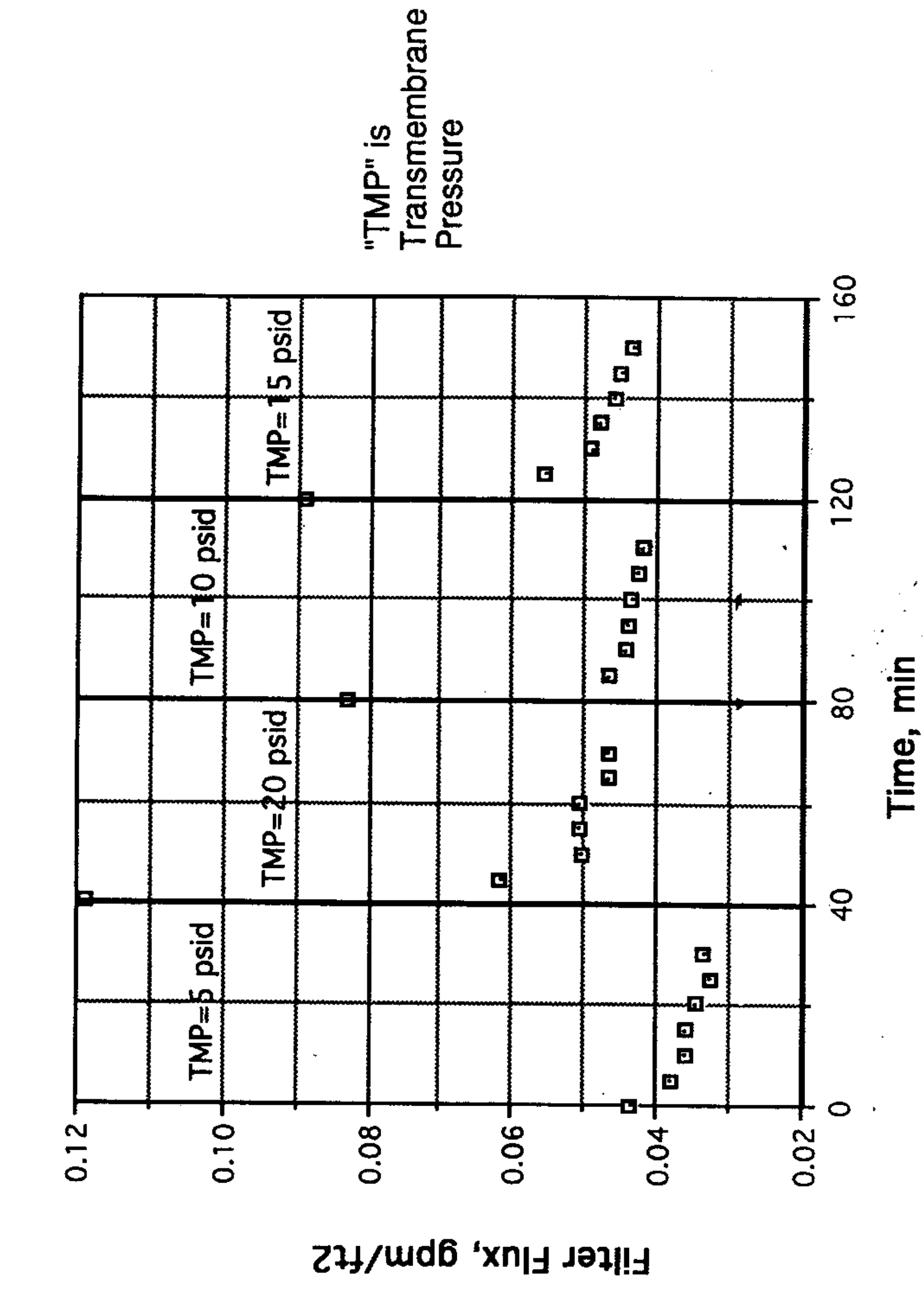


Figure 4. Flux vs Time for 8 wt % Simulant

Figure 5. Filter Fluxes vs Time for Tank 48H and Simulant Samples

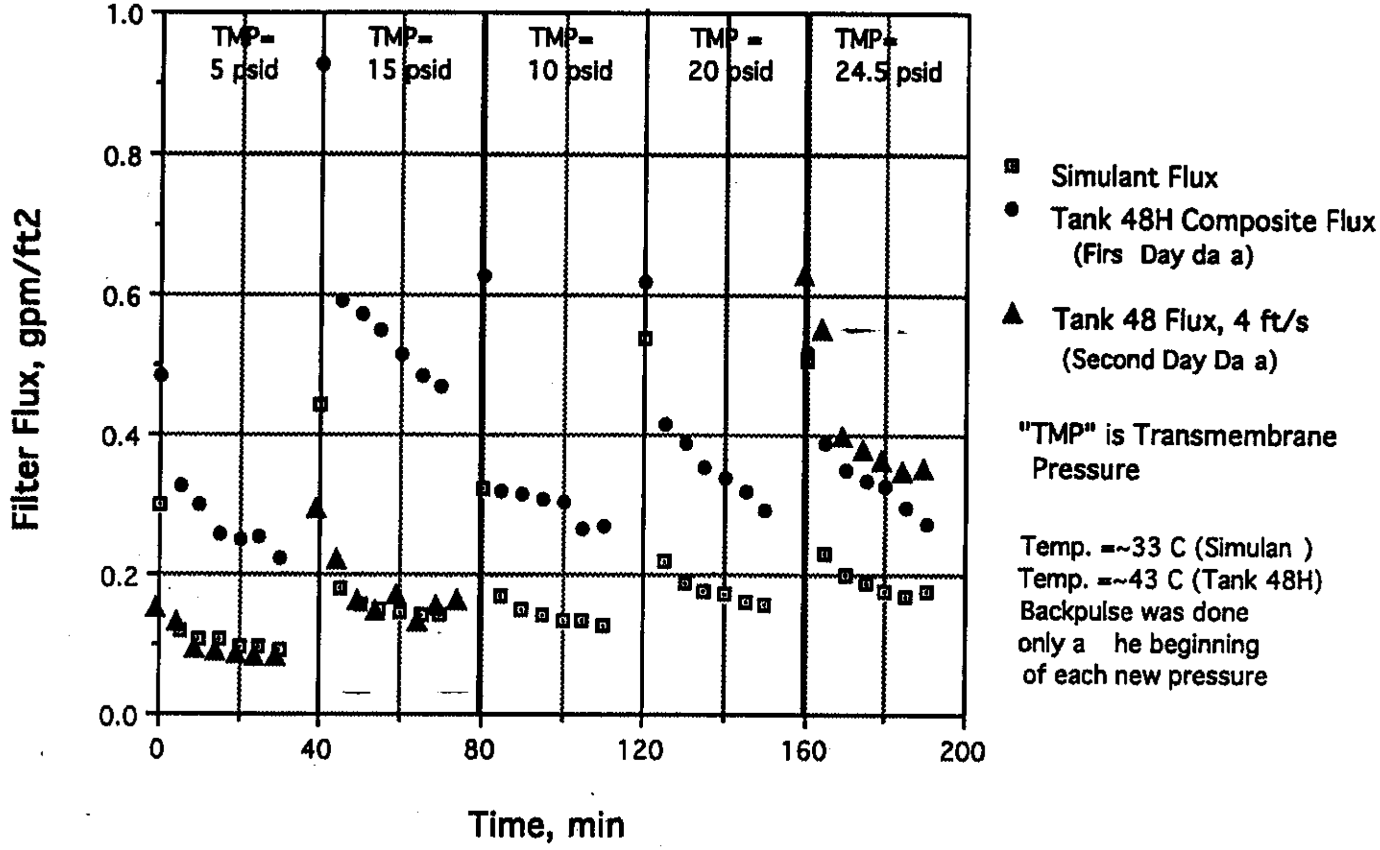


Figure 6. Comparison of Half-Hour Average Fluxes

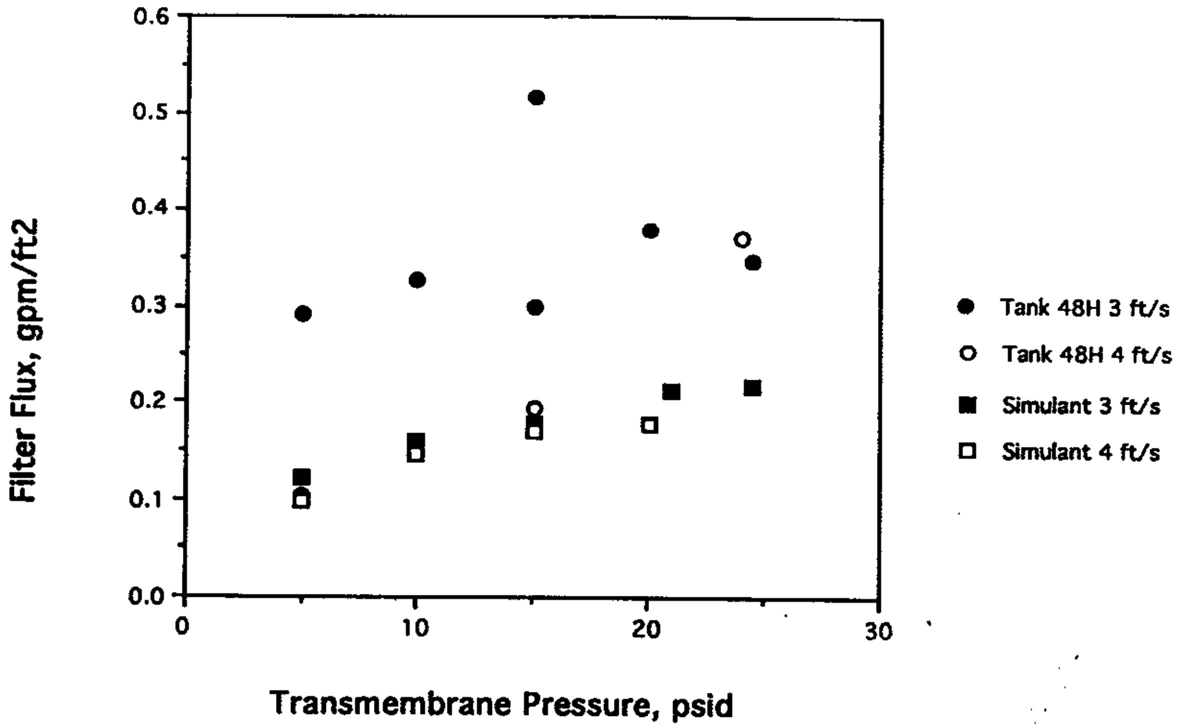


Figure 7. Cells Unit Filter, Effect of Added Monosodium Titanate

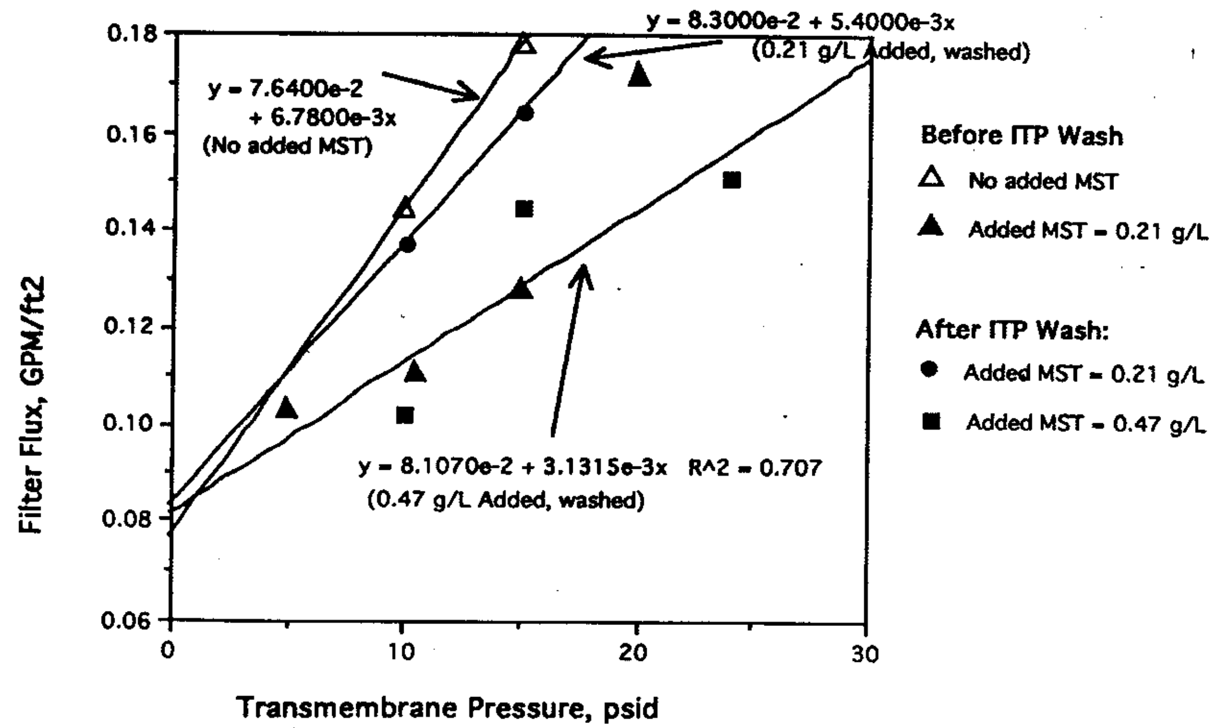


Figure 8. ITP Wash Cycle Sodium Concentration

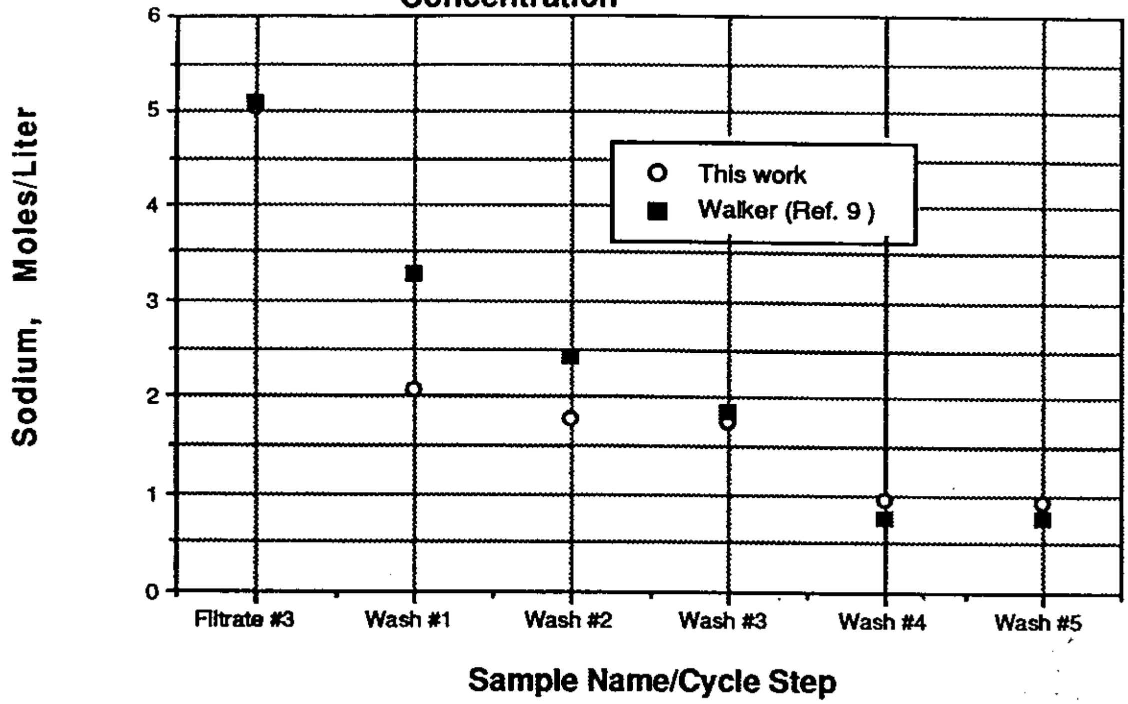


Figure 9. ITP Wash Cycle: Nitrate Concentration

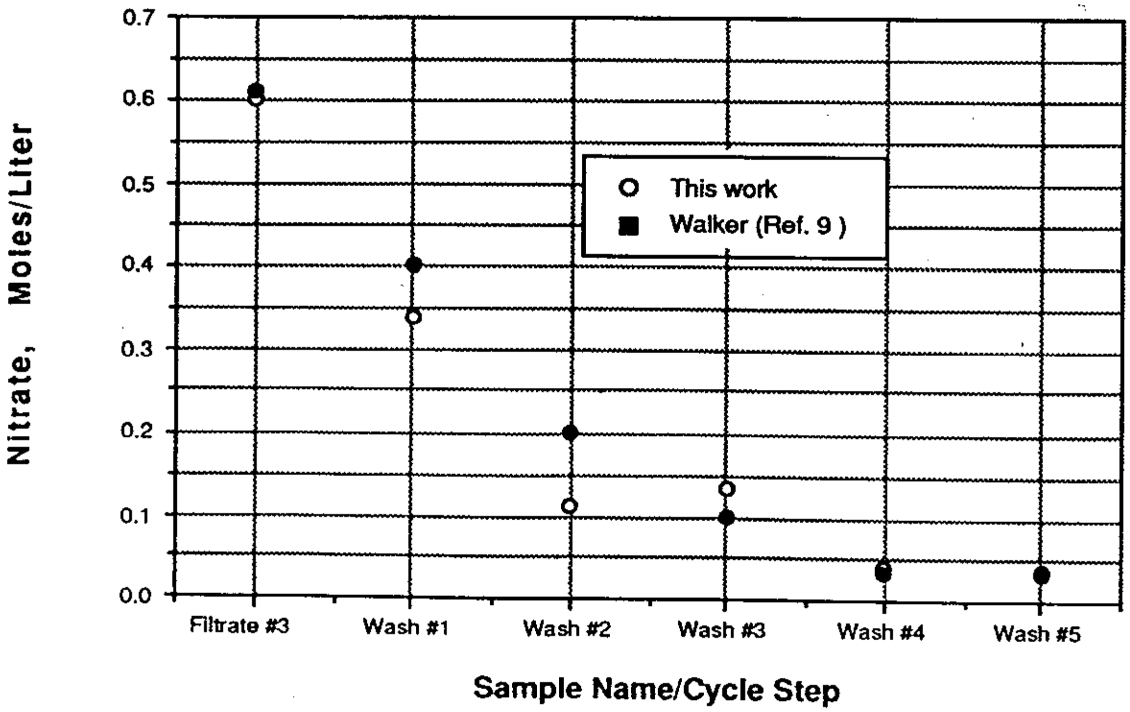


Figure 10. ITP Wash Cycle: Nitrite Concentration

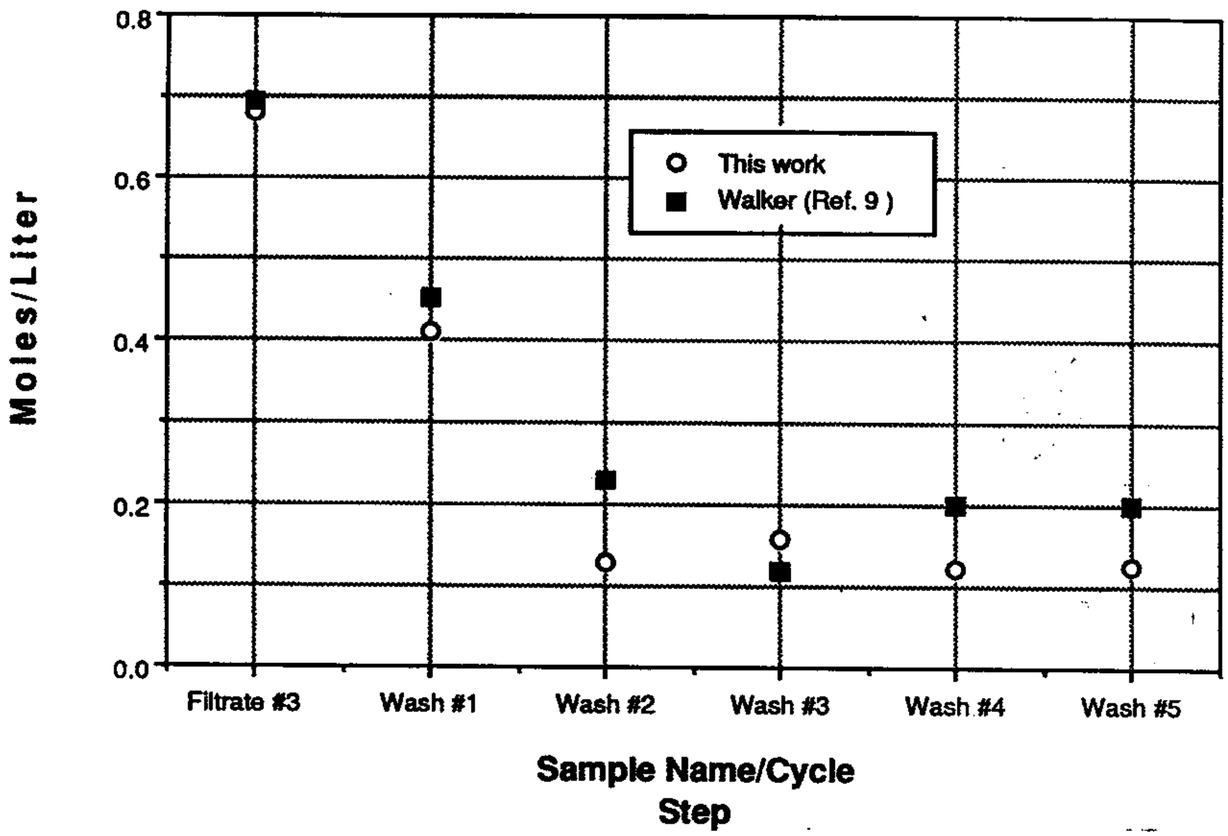


Figure 11. Filter Permeance Comparison

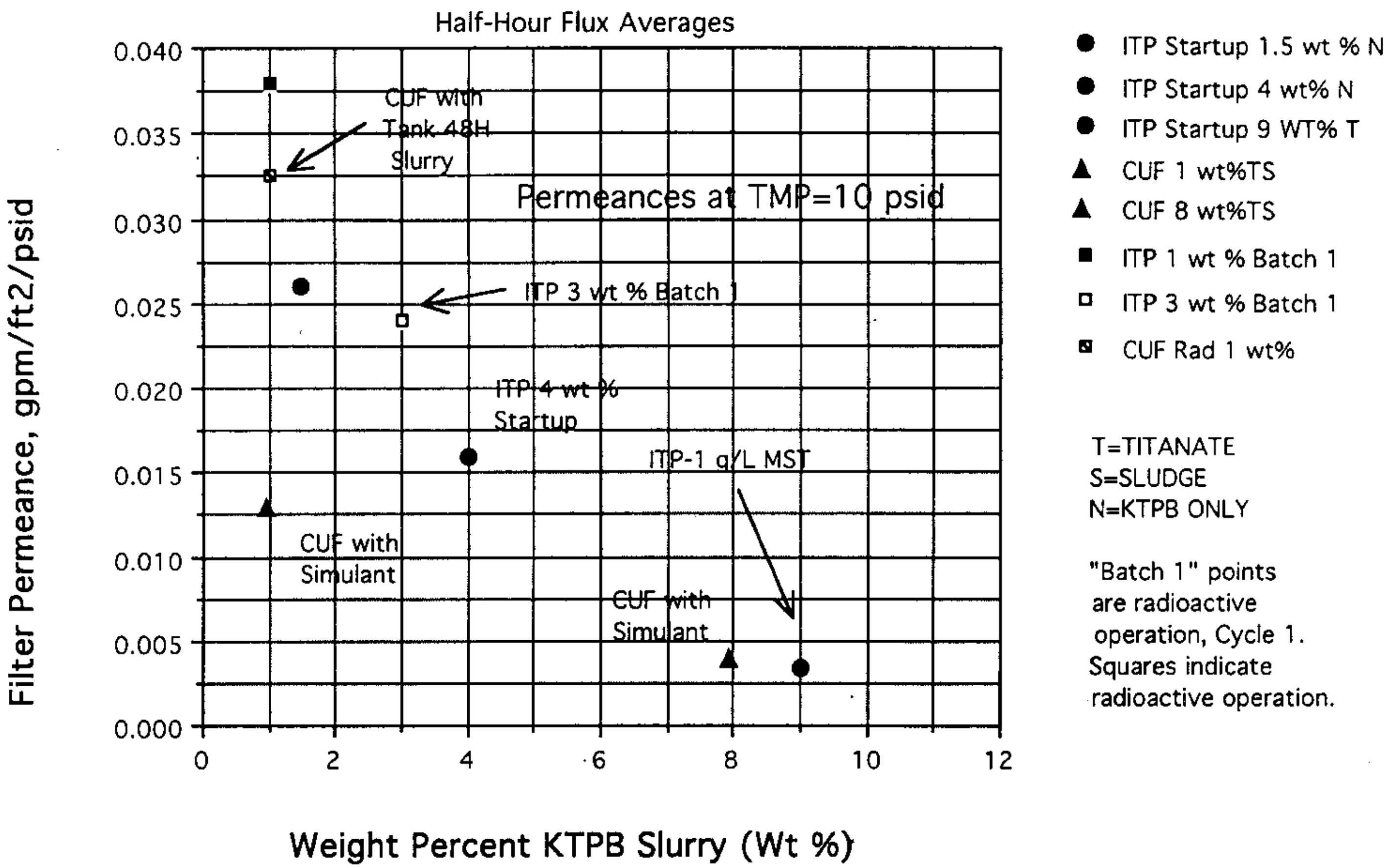


Table 1. Simulant Recipe

LIQUID PHASE

Component	Target Molarity
Na_2SO_4	0.13
$NaNO_2$	0.71
$NaNO_3$	1.11
NaOH	2.58
KNO_3	0.03
Na_2CO_3	0.17
$Al(NO_3)_3 * 9H_2O$	0.30

SOLIDS CONTENT

Component	Quantity
Potassium Tetraphenylborate	1 or 8 wt %
Monosodium Titanate	0.21 g/L per 1. wt %
Purex Sludge	400 mg/L per 1. wt %

Table 2. Tank 48H Sample Content

SUPERNATE (Composite Sample)

Component	Concentration
Na^+	5.04 M ± 0.6
NO_2^-	0.69 M ± 0.1
NO_3^-	0.6 M ± .09
K^+	0.03 M ± 0.001
OH^-	2.74 M
Cs-137	0.86 Ci/gal ± .02
Total wt % solids	0.3 ± 0.2 (3.62 g/L at sp. G = 1.207)
Titanium	173 mg/L ± 126
	(Translates to 0.36 g/L MST)
0.55 M NaTPB	(47.5+2.68) g added to 725.2 g supernate
MST additions	0.24 g/L, first addition
	0.24 g/L, second addition
	(Total 0.48 g/L added)

Table 3. Sequence of Runs and Samples

Date	Activity
4/19/95 - 5/23/95	Preliminary Checkouts
5/24/95 - 5/25/95	Water flux runs and 1 wt % slurry at 3 and 4 ft/s
5/30/95	Water flux runs and 8 wt % slurry at 3 ft/s
8/07/95 - 8/08/95	Water runs with the Cells-Installed Unit
8/16/95 - 8/17/95	Tank 48H slurry made and 517 g added to the CUF. 3 ft/s points taken. Sample filtrate #1 drawn.
8/18/95	4 ft/s points taken
8/23/95	2.73 g NaTPB solution added to the inventory. It had previously been precipitated to 0.03M K+. It should have been precipitated to 0.033 K+. Sample Filtrate #2 was taken during two points at 3 ft/s. 0.99 g Optima #3 MST added. Three points at 3 ft/s taken.
8/26/95	ITP Wash Cycle. Run order: *Take a data point and draw Sample Filtrate #3. *Wash with 157.5 mL water and draw sample Wash #1 *Add 25.2 mL of 19.2M NaOH *Wash with 220.5 mL water and draw sample Wash #2 *Add 18.9 mL of 19.2M NaOH *Wash with 204.7 mL water and draw sample Wash #3 *Add 25.2 mL of 7.5 M NaNO2 *Wash with 393.7 mL water and draw sample Wash #4 *Draw sample Wash #5
9/28/95	Draw sample Titanate Post-Wash #1. Add total of 0.1 g (dry basis) MST (Production runs 95QAB391 and 95QAB393). Draw sample Titanate Post-Wash #2 after taking 3 ft/s data.

Table 4: Effect of Titanate Addition on Filter Flux

Grams/Liter MST added		Flux, gpm/ft2, at		Percent of Previous Flux	
		10 psid	15 psid	10 psid	15 psid
0	(Before Wash cycle)	0.144	0.178	100	100
0.24	(Before Wash cycle)	0.110	0.127	76	71
0.24	(After Wash cycle)	0.137	0.164	100	100
0.48	(After Wash cycle)	0.102	0.144	75	88
Cumulative Reduction				57	62

Table 5. Plutonium-239 Measured in Filtrate Samples (ICP-MS)

SAMPLE	Pu-239, micrograms/liter
Initial filtrate before washing (Filtrate #3)	0.6
Filtrate after washing step 1 (Wash #1)	Not Detected
Filtrate after washing step 2 (Wash #2)	Not Detected
Filtrate after washing step 3 (Wash #3)	Not Detected
Filtrate after washing step 4 (Wash #4)	Not Detected
Replicate sample, filtrate after washing step 4 (Wash #5)	0.3

Table 6. Cesium-137 in CUF Filtrate

Cesium DF: Concentration in the feed was 213,000 microcuries/L

Filtrate #1:	6.1 uCi/L (precipitated to 0.03 M K+)
Filtrate #2:	1.5 uCi/L (precipitated to 0.033 M K+)
Filtrate #3:	3 uCi/L (Initial condition for ITP wash; expected flowsheet value)
Wash #1:	0.8 uCi/L
Wash #2:	1.5 uCi/L
Wash #3:	7.5 uCi/L
Wash #4:	14 uCi/L
Wash #5:	11 uCi/L (Second sample after #4)
Titanate Post-Wash #1:	3.3 uCi/L
Titanate Post-Wash #2:	1.9 uCi/L

Table 7. Isotopics of Filtrates

All values are in micrograms per liter.

Atomic Mass	Filtrate #1	Filtrate #2	Filtrate #3	Wash #1	Wash #2	Wash #3	Wash #4	Wash #5
230	0.4	0.2						0.3
231	0.4	0.4			0.3	0.5		
232	0.2	0.8	0.6	3.8	0.8	0.8		1.2
233	19.6	19.6	16.4	10.5	8.5	2.8	0.9	2.0
234	148.6	145.2	130.2	80.6	49.2	38.2	18.2	15.8
235	245.0	263.2	224.6	137.3	78.6	57.0	27.9	28.1
236	55.0	65.0	53.0	34.5	20.5	10.4	6.0	6.5
237	20.0	23.6	16.7	7.1	5.6	1.3	0.7	1.6
238	1455.4	1563.8	1270.6	804.1	500.4	307.3	166.0	157.8
239	0.4	0.6	0.6	0.3				
240	0.4	0.2	0.5	0.3	0.4	0.2		
241	0.4		0.4	0.2		0.6		
242	0.4	0.4		0.3	0.8	0.5		
243	0.2	0.4	0.2		0.2	0.3	0.2	
244	0.2		0.2		1.1			
245	0.2	0.4						
246		0.2	1.1		0.2	0.3	0.4	0.4
247				0.7	0.8		0.3	
248	0.4	0.6			0.2	0.3	0.4	0.2
249	0.4	0.4			0.6			