Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U. S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1) warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2) representation that such use or results of such use would not infringe privately owned rights; or
3) endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

MST Filterability Tests

M. R. Poirier
P. R. Burket
M. R. Duignan

March 2015

SRNL-STI-2015-00158, Revision 0

<div style="text-align:center">**DISCLAIMER**</div>

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

<div style="text-align:center">

Printed in the United States of America

Prepared for
U.S. Department of Energy

</div>

MST Filterability Tests

M. R. Poirier
P. R. Burket
M. R. Duignan

March 2015

Savannah River
National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

REVIEWS AND APPROVALS

AUTHORS:

_____
M. R. Poirier, Advanced Characterization and Process Technology   Date

_____
P. R. Burket, Engineering Process Development   Date

_____
M. R. Duignan, Engineering Process Development   Date

TECHNICAL REVIEW:

_____
C. J. Martino, Process Technology Programs, Reviewed per E7 2.60   Date

APPROVAL:

_____
F. M. Pennebaker, Manager   Date
Advanced Characterization and Process Technology

_____
S. L. Marra, Manager   Date
Environmental & Chemical Process Technology Research Programs

_____
E. J. Freed, Manager   Date
DWPF/Saltstone Facility Engineering

Summary

The Savannah River Site (SRS) is currently treating radioactive liquid waste with the Actinide Removal Process (ARP) and the Modular Caustic Side Solvent Extraction Unit (MCU). The low filter flux through the ARP has limited the rate at which radioactive liquid waste can be treated. Recent filter flux has averaged approximately 5 gallons per minute (gpm). Salt Batch 6 has had a lower processing rate and required frequent filter cleaning. Savannah River Remediation (SRR) has a desire to understand the causes of the low filter flux and to increase ARP/MCU throughput. In addition, at the time the testing started, SRR was assessing the impact of replacing the 0.1 micron filter with a 0.5 micron filter. This report describes testing of MST filterability to investigate the impact of filter pore size and MST particle size on filter flux and testing of filter enhancers to attempt to increase filter flux.

The authors constructed a laboratory-scale crossflow filter apparatus with two crossflow filters operating in parallel. One filter was a 0.1 micron Mott sintered SS filter and the other was a 0.5 micron Mott sintered SS filter. The authors also constructed a dead-end filtration apparatus to conduct screening tests with potential filter aids and body feeds, referred to as filter enhancers.

The original baseline for ARP was 5.6 M sodium salt solution with a free hydroxide concentration of approximately 1.7 M.[3] ARP has been operating with a sodium concentration of approximately 6.4 M and a free hydroxide concentration of approximately 2.5 M. SRNL conducted tests varying the concentration of sodium and free hydroxide to determine whether those changes had a significant effect on filter flux.

The feed slurries for the MST filterability tests were composed of simple salts (NaOH, $NaNO_2$, and $NaNO_3$) and MST (0.2 – 4.8 g/L). The feed slurry for the filter enhancer tests contained simulated salt batch 6 supernate, MST, and filter enhancers.

The conclusions from this work follow.

- The 0.5 micron filter produced the same or higher flux than the 0.1 micron filter. As the concentration of insoluble solids increased and the filter cake became more established, the difference became small. While this testing did not look at solids rejection by the filter, a 0.5 micron filter has larger pores and may allow more solid particles to pass through the filter.
- The "fewer fines MST" did not produce a higher flux than the "vendor MST", so efforts to remove the fine particles from the MST should not be pursued.
- The higher ionic strength, higher free hydroxide slurries produced higher flux than the lower ionic strength, lower free hydroxide slurries. Particle size data showed larger particles with the higher ionic strength, higher free hydroxide slurries. Additional laboratory-scale testing should be conducted to investigate this phenomenon.
- None of the filter aids or body feeds produced a significant increase in filter flux. One likely cause of this result is the solid particles being MST only. If testing identifies other solid particles contributing to the fouling at ARP, additional tests with filter aids or body feeds should be considered.
- Filtrate samples collected and examined visually showed that both pore size filters removed solid particles from the feed.
- Leaving the filter tubes filled between batches rather than draining the tubes produced an increase in the initial filter flux. SRR personnel should investigate implementing this practice at ARP.
- Periodically stopping the flow of slurry through a crossflow filter for even as little as 20 seconds appears to temporarily improve flux rate. Further testing should be performed to demonstrate the relationship between stopping time and flux improvement.
- The short duration on many of the tests conducted prevented the formation of a "good" filter cake. The test protocol was modified to allow more time for a "good" filter cake to develop. Future testing should use this protocol.

TABLE OF CONTENTS

Introduction

The Savannah River Site (SRS) is currently treating radioactive liquid waste with the Actinide Removal Process (ARP) and the Modular Caustic Side Solvent Extraction Unit (MCU). The low filter flux through the ARP has limited the rate at which radioactive liquid waste can be treated. Recent filter flux has averaged approximately 5 gallons per minute. Salt Batch 6 has had a lower processing rate and required frequent filter cleaning.[8] Savannah River Remediation (SRR) has a desire to understand the causes of the low filter flux and to increase ARP/MCU throughput. In addition, at the time the testing started, SRR was assessing the impacted of replacing the 0.1 micron filter with a 0.5 micron filter. This report describes testing of MST filterability to investigate the impact of filter pore size and MST particle size on filter flux and testing of filter enhancers to attempt to increase filter flux.

The original baseline for ARP was 5.6 M sodium salt solution with a free hydroxide concentration of approximately 1.7 M.[3] ARP has been operating with a sodium concentration of approximately 6.4 M and a free hydroxide concentration of approximately 2.5 M. SRNL conducted tests varying the concentration of sodium and free hydroxide to determine whether those changes had a significant effect on filter flux.

MST Filterability

SRR requested Savannah River National Laboratory (SRNL) personnel to conduct monosodium titanate (MST) filterability tests with a lab-scale crossflow filtration unit.[1,2] The purpose of the testing is to compare filter performance between existing "vendor supplied" MST and "reconditioned" MST[12], where "reconditioned" means this batch had "fewer fines"[a], and will be referred to as "fewer fines" MST. Another purpose of the testing is to compare the filterability of MST in a 0.1 μm Mott porous metal crossflow filter with the filterability of MST in a 0.5 μm Mott porous metal crossflow.

This document describes the data comparing filter flux of a 0.1 μm filter and a 0.5 μm filter using inhibited water (0.01 M NaOH), a salt solution containing 6.4 M sodium and 2.5 M free hydroxide, a salt solution containing 6.4 M sodium and 1.5 M free hydroxide, and a salt solution containing 5.6 M sodium and 1.7 M free hydroxide. The concentrations of solid particles ranged from 0.2 to 14.4 g/L to represent the initial and a mid-process values during filter operation.

Filter Enhancers

One potential method to increase filter flux is the introduction of filtration enhancers provided their addition does not have adverse consequences for the ARP/MCU or DWPF processes. Filtration enhancers have been studied in the past[4,5,6] and for some waste streams have shown potential to improve filter flux. Several filter aids and body feeds were evaluated in a dead-end filter with a simulant of Sludge Batch 6. Filtration enhancers are generally categorized as follows:

- Filter aids are substances that coat a filter to improve overall permeability.

- Body feeds are compounds made to react with a slurry to flocculate solids creating a more permeable cake.

A significant difference between previous filter enhancer tests[4] and the current one[7,13] is the waste stream to be filtered. The undissolved solids in the ARP feed to the crossflow filter are primarily MST solids at an initial concentration of 2.5 g/L (or approximately 0.2 wt%).[11] While there is some sludge in the feed stream it has been found[8] to be no more than 1/24th of the MST concentration.[11] The lower concentration and properties of solids may change the effect of filter enhancers on the solid particles.

---

[a] Because the attempts to "recondition" the MST to remove the fine particles were unsuccessful, tests were conducted with a batch of MST that had a lower fraction of fine particles.

For the current task[7] a search for filtration enhancers in the literature and industrial experiences was performed. The filter enhancer candidates were evaluated with dead-end filters to select the most promising enhancers for crossflow filter testing.

Experimental

Equipment

<u>Crossflow Filter</u>

SRNL personnel constructed a laboratory-scale filtration apparatus. Figure 1 shows the layout of the lab-scale filtration apparatus. The apparatus has an ~ 10 gallon feed tank with an impeller to mix the tank contents. The mixing system was not designed to be prototypic of the ARP; it was designed to suspend the MST particles in the feed slurry. A centrifugal pump[b] draws the slurry from the feed tank and pumps it into two parallel lines at ~ 3.0 gpm. Each line has a heat exchanger to control the temperature of the feed slurry to 25 ± 2 °C. The slurry flows past a tee where the two lines meet and the inlet pressure transducer is located. Beyond the tee there is one valve on each line which can be used to adjust the flow rate to each filter. Following each valve is a 0 – 5 gpm ± 0.1 gpm magnetic flowmeter which is used to measure the flow of slurry into each filter. The filters are located downstream of the flowmeters. After exiting the filters, the concentrated slurry streams are combined and returned to the feed tank. The concentrate line has a manual backpressure valve and an automated backpressure valve connected in parallel. The outlet from each of these valves returns the slurry to the bottom of the feed tank. All lines are ½" SS tubing except for the instrument lines to the pressure transducers which are ¼" SS tubing.

The filtrate leaves each filter through 3/8" and ¼" tubing. Pressure transducers measure the filtrate pressure immediately after each filter. A three way valve is positioned even with the top of a graduated tube for each filtrate line where the filtrate can be directed to the filtrate tank or to the 100 mL ±1 mL graduated tube which is used to manually measure the filtrate flowrate. For these tests, the filtrate flow was measured every 15 minutes. The filtrate flow could also be sent back to the feed tank by moving the tygon tubing from the filtrate tank to the feed tank.

One of the crossflow filters is a 0.1 µm pore size, 3/8 inch ID Mott® porous metal crossflow filter and the other is a 0.5 µm pore size, 3/8 inch ID Mott® porous metal crossflow filter. Both filters are 24 inches in length and constructed of sintered stainless steel. A computer was used to record the pressures, feed flow rates, and feed tank temperature as well as to control the automatic backpressure valve located after the filters. No secondary filter was installed in the filter system, because the purpose of the testing was to compare performance of the 0.5 µm crossflow filter with the 0.1 µm crossflow filter, and to evaluate the effect of using "reconditioned MST" on the crossflow filters.

<u>Dead End Filters</u>

Concurrent with the crossflow filter test a dead-end filter test was set up to screen potential filter aids and body feeds. Figure 2 shows the dead-end filter set up with multiple filters in parallel. The equipment included 5 filter stations with shut-off valves, a Banant Thermistor Data Logger (Model 600-1075) to measure temperature, a Pressure transducer (Paro Scientific DigiQuartz, Model 245A101), an Ashcroft pressure gauge (30 to 0 in. Hg), a McDaniels Controls pressure gauge, (0 to 60 psi and 30 to 0 in.Hg), a Welch vacuum pump (Thomas Industries, Model 1400 Duo Seal Vacuum Pump), a Toledo scale (0 to 1000 lb., Model 8142), and a Mettler Toledo balance (0.01 to 350 grams, Model AB304-S/FACT). To monitor and control the pressure, and to have five timers operating simultaneously a laptop computer was used. However, the actual data were manually taken from the computer after each test run.

---

[b] Initial testing utilized two progressive cavity pumps operating in parallel. When one of the pumps began leaking, the two progressive cavity pumps were replaced with a single centrifugal pump. The axial velocities and transmembrane pressures during the tests were not affected by the change in pumps.

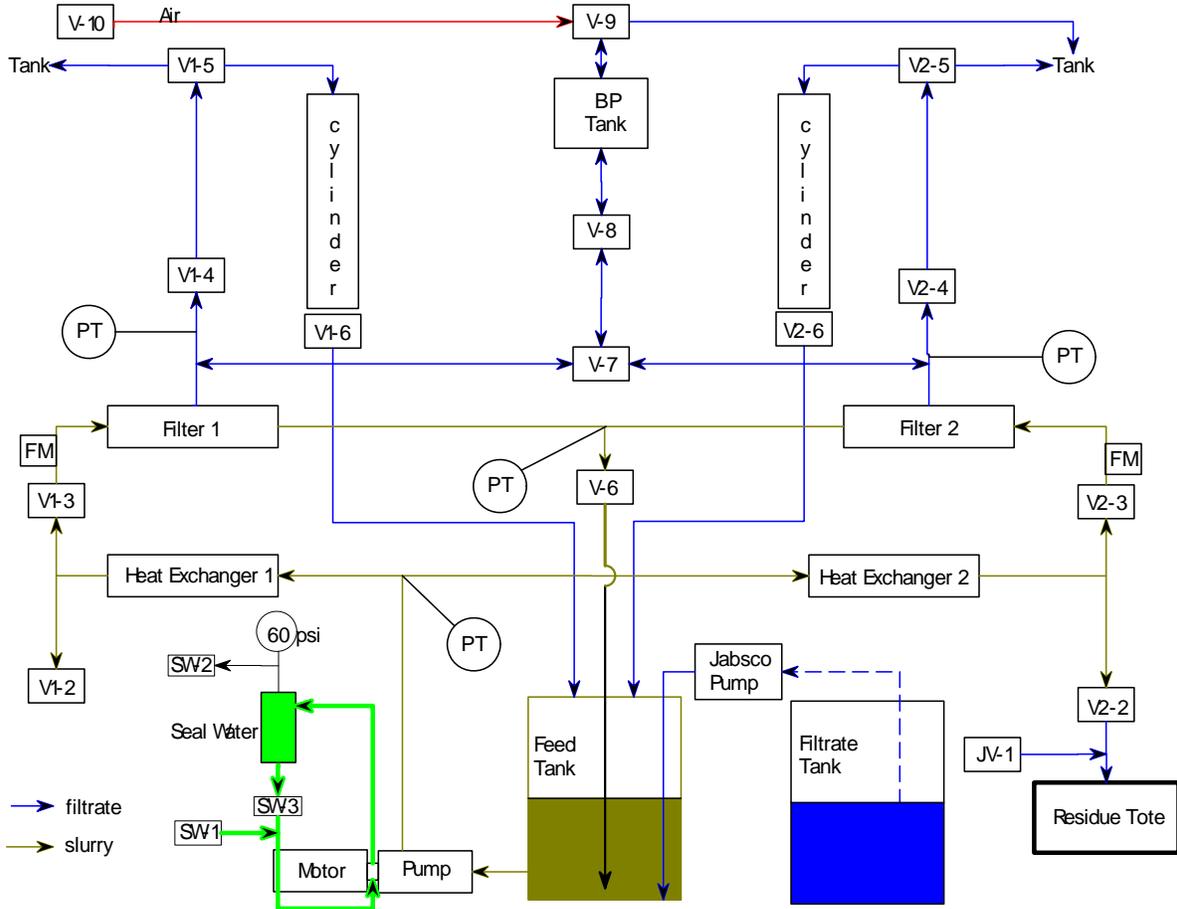**Figure 1. Schematic of Laboratory-Scale Crossflow Filter Unit**

Figure 2. Five 150 mL Nalgene Dead-End Filter with 0.45-micron Nylon Filters Connected to the same Vacuum Source and Vacuum Gauge

Feed Preparation

Table 1 shows four different supernate solutions for the MST filterability tests, and Table 2 shows the solids concentrations for the tests. The supernate is a simple salt solution composed of varying

concentrations of sodium hydroxide, sodium nitrite, and sodium nitrate. The salt solutions were prepared by dissolving the salts in deionized water. The simple salt solution was requested by SRR in the TTR to prevent salts such as sodium aluminate and sodium carbonate from confounding the effects of MST on the filter flux. The solid particles are monosodium titanate (MST). The MST concentrations were selected based on an MST strike concentration of 0.2 g/L. The 0.2 g/L initial concentration was selected, because the current ARP MST strikes are 0.2 g/L. The MST concentration was increased to mimic the concentration of MST in the ARP feed tank resulting from concentrating the insoluble solids. This approach neglects the heel in the ARP filter feed tank, but using a lower concentration of MST reduces the probability/amount of aggregation which would complicate identifying the impact of reducing the amount of fines in the MST on filter flux.

Table 1. Supernate Solutions for MST Filterability Tests

Feed	Sodium (M)	Hydroxide (M)	Nitrite (M)	Nitrate (M)
Inhibited water	0.01	0.01	0	0
High sodium, high hydroxide	6.4	2.5	0.5	3.4
Low sodium, Low hydroxide	5.6	1.7	0.5	3.4
High sodium, low hydroxide	6.4	1.5	0.5	4.4

Table 2. Initial Solids Concentration for MST Filterability Tests

MST Concentration (g/L)
0.2
0.4
0.6
0.8
1.8
2.8
3.8
4.8

Table 3 shows the composition of the salt solution used in the filter enhancer tests. The target sodium concentration for the supernate was 6.6 M, and the target free hydroxide concentration was 2.2 M. After the simulant was prepared, the density and viscosity were measured. The density was approximately 1.27 g/mL, and the viscosity was approximately 3.9 cP. Both values were below the targets of 1.3 g/mL and 4.7 cP, respectively. These physical properties of the liquid are important with respect to filtration, so more sodium hydroxide was added until the density was close to the target of 1.3 g/mL. When the density reached 1.31 g/mL, the viscosity slightly exceeded the target and ended up at 5.5 cP. At this point the supernatant was ready for the addition of undissolved solids.

Table 3. Concentration of Supernate Solution used for Filter Enhancer Tests

Species	Concentration (M)
Na[i]	7.5
K	0.0145
Cs	0.000135
OH	4.48
NO3	1.81
NO2	0.5
AlO2	0.25
CO3	0.155
SO4	0.072
F	0.031
Cl	0.022
PO4	0.009
SiO3	0.0033
C2O4	0.0039
MoO4	0.000193
[i] Sodium concentration was higher than 6.4 M because additional sodium hydroxide was added to increase density to 1.3 g/mL	
Free hydroxide concentration based on amount of sodium hydroxide added	

Due to the low fraction of sludge solids in the ARP feed and the fact that knowledge of those solids is very limited, they were not included for this phase of the test. The solid particles added to the supernatant were MST (i.e., no simulated sludge was added). The concentrations were 0.2 g/L which is the concentration used for each MST strike and a higher concentration to determine if there is effect due to a larger presence of solids. The higher concentration was 0.75 g/L.

Test Protocol

The MST Filterability crossflow filter tests were conducted as follows. Prior to testing, the apparatus was chemically cleaned with 0.5 M oxalic acid and 1 M nitric acid. Rather than using a prototypic ARP cleaning method, the filters were cleaned by draining the feed slurry from the filter system into the feed tank and removing the feed slurry from the feed tank. After the feed slurry was removed from the system, approximately 3 gallons of 0.5 M oxalic acid was added to the feed tank. The oxalic acid was recirculated through the filter system for at least 1 hour, which was drained into the feed tank, and removed. After the oxalic acid was removed, approximately 3 gallons of 1 M nitric acid was added to the feed tank. The nitric acid was recirculated through the filter system for at least 1 hour, drained into the feed tank, and removed. The filter system was flushed with deionized water until the pH was greater than 6. [All filter cleaning was conducted at ambient temperature] Following chemical cleaning, the filter system was run with deionized water to establish a clean water flux for each of the filters. The 0.1μm filter had a clean water flux of 0.23 – 0.36 gpm/ft^2, and the 0.5 μm filter had a clean water flux of 0.26 – 0.70 gpm/ft^2.

Following the clean water flux test, SRNL personnel prepared approximately 9 gallons of feed slurry containing supernate (see Table 1) plus 0.2 g/L of vendor MST. The slurry was run through the crossflow filters at an axial velocity of 8.7 ± 0.1 ft/s and a transmembrane pressure of 40 ± 2 psid. The axial velocities were kept the same by measuring the volumetric flow rate through each filter and adjusting a valve upstream of each filter to keep them equal. The transmembrane pressures were equal by design. The filter feed and concentrate lines of the two filters were connected to eliminate any differences in pressure between the two filters. The slurry was filtered until the feed reached the minimum level that could be pumped to the filters (~ 3 gallons) or the work shift ended before reaching that level. This process concentrated the slurry by a factor of 3 by dewatering the 9 gallons to 3 gallons. Prior to the next test, the filtrate was returned to the feed tank and additional MST was added to the feed tank to increase the MST concentration as described in Table 2. This process was repeated for a total of 4 batches. For each of the next four batches, MST was added to the feed tank to increase the MST concentration by 1.0 g/L slurry as described in Table 2. The filter unit was operated for a total of four batches at this MST addition rate, and an overall total of 8 batches.

This process was repeated for each of the supernate solutions shown in Table 1 with vendor MST and "fewer fines" MST.[c]

During the initial crossflow filter tests (inhibited water), the filter was drained overnight. Prior to the start of tests with 6.4 M sodium, 2.5 M free hydroxide, an automatic pressure control valve was installed on the system to aid in controlling the TMP. After operating two days successfully, the engineer noticed a sound upon shutdown since the discharge line from the automatic valve was not submerged in the feed tank liquid causing the simulant to drain from the filters. Based on this observation, the ends of the lines leaving from or returning to the feed tank were altered to ensure they remained submerged at all times. The shutdown procedure was altered to close the filtrate valve for each system prior to stopping the centrifugal pump. Thus the liquid was held in the filters while the system was in shutdown mode. This change also afforded a quicker startup since the filters no longer required a slow refill. It was believed that this mode of operation was more representative of longer term operation of the ARP filter.

After completing the filter enhancer evaluation with dead-end filters, one selected enhancer, hematite, was tested in the crossflow filters. Those filter enhancer crossflow filter tests were conducted as follows. Prior to testing, the apparatus was chemically cleaned with 0.5 M oxalic acid and 1 M nitric acid per the previously described protocol. Following chemical cleaning, the apparatus was run with deionized water to establish a clean water flux for each of the filters. The 0.1μm filter had a clean water flux of 0.33 gpm/ft^2, and the 0.5 μm filter had a clean water flux of 0.66 gpm/ft^2.

Following the clean water flux test, SRNL personnel prepared approximately 9 gallons of feed slurry containing supernate (see Table 3) plus 1.5 g/L of vendor MST. The slurry was run through the crossflow filters at an axial velocity of approximately 8.7 ft/s and a transmembrane pressure of approximately 40 psi. The axial velocity and transmembrane pressure were the same for both filters. The concentrated solids were recycled to the feed tank. For the first hour, the filtrate was recycled to the feed tank. After the first hour, the filtrate was collected in a separate container. If the volume of slurry in the feed tank decreased to 3 gallons, which increased the MST concentration by a factor of 3, the filtrate was recycled to the feed tank for the remainder of the shift. The next day, the filtrate was returned to the feed tank to dilute the slurry back to its starting concentration of MST, then additional MST was added to the feed tank to increase the MST concentration by 1.0 g/L slurry to 2.5 g/L. The filter was operated for another day. This process was run with 2.5 g/L slurry for a total run time of 5 days. For each of the next two days, MST was added to the feed tank to increase the MST concentration by 1.0 g/L slurry (i.e., 3.5 g/L and 4.5 g/L) for a total of eight (8) days of operation. This entire process was repeated so that one set of tests was conducted with only MST solids and another test was conducted with MST and hematite solids

---

[c] Because the attempts to "recondition" the MST to remove the fine particles were unsuccessful, tests were conducted with a batch of MST that has a lower fraction of fine particles.

(0.75 g/L hematite for 2.5 g/L MST) in order to have a good comparison. Additional hematite was added with each MST addition to keep the hematite:MST ratio constant.

The filter enhancer deadend filter test matrix was performed in three phases: baseline tests, tests with simulated salt solution and 0.2 g/L MST, and tests with simulated salt solution and 0.8 g/L MST. The feeds for the baseline tests were deionized water, inhibited water (0.01 M NaOH), simulated salt solution, simulated salt solution with 0.2 g/L MST, and simulated salt solution with 0.8 g/L MST.

Besides the different methods of applying the filter aids or body feeds, all of the deadend filter tests followed the same procedure. New, clean filter cups were connected to the apparatus. With all individual filter cup valves closed, the vacuum was applied to an initial vacuum in the system (target 75 Torr), as measured by the computer. The vacuum range was set from 70 to 80 Torr. The cups were filled with inhibited water and a light vacuum was applied to wet the filter media. Each filter cup was filtered until empty.

For the filter aids, which are applied directly to the filter surface, the filter aid was mixed with approximately 150 mL of inhibited water and gently poured into each filter cup. A mild vacuum was applied to each filter to draw the mixture through the filter and coat the filter surface with the filter aid. Following application of the filter aid, each filter cup is filled with 150 mL of slurry simulant.

The body feeds are mixed with the feed slurry before filtering. Five clean beakers were filled with 150 mL of slurry simulant. To each beaker the appropriate amount of body feed as listed in the test matrices shown in Appendix A was added and the contents stirred until well mixed. All body feeds were added directly to the slurry simulant as received, except the emulsion flocculants from Cytec Industries. Appendix B shows the vendor preparation necessary to properly use those body feeds. Each filter cup was gently filled with the contents of each beaker.

After the feeds slurries and filter system were prepared, the valve was opened to apply vacuum to the system. The volume of filtrate collected as a function of time was measured.

Results

MST Filterability

<u>0.01 M NaOH Salt Solution</u>
Figure 3 shows the filter flux of each filter as a function of elapsed run time in the tests with "vendor MST". The plot shows that initially, the 0.5 µm filter produced a larger flux than the 0.1 µm filter, but the difference is small. The likely reason for the higher initial flux measured with the 0.5 µm filter is that its larger pore size gives it less resistance to flow. Initially, no filter cake is present, so the lower filter resistance leads to higher filter flux. As a filter cake builds on each of the filters, the resistance of the filter cake becomes much larger than the resistance of the filter and controls filter flux. Once the filter cake controls filter flux, there is no significant difference in the flux between the two filters. For most of the run time, the difference was less than 10%. This result is consistent with the 2004 alternative filter media work, which showed less than a 5% difference in flux between a 0.1 µm Mott® porous metal filter and a 0.5 µm Mott® porous metal filter.[9] The results disagree with the 2003 testing in which the 0.1 µm Mott® porous metal filter produced a higher flux than the 0.5 µm Mott® porous metal filter.[10] While the results of this test contradict the results of the 2003 test, the feed for the 2003 test contained MST prepared to a different specification and also contained simulated sludge. The 2004 test was also performed with MST based on a different specification and a feed slurry containing simulated sludge, making direct comparison difficult.

Filtrate samples were collected and examined visually to look for solid particles. All samples, except for two, showed no visible solid particles. The two samples were from the 0.5 micron filter and occurred at

the same stage in the filtration process. Subsequent samples showed no visible solids. We are uncertain of the reason for the visible particles in the two samples.
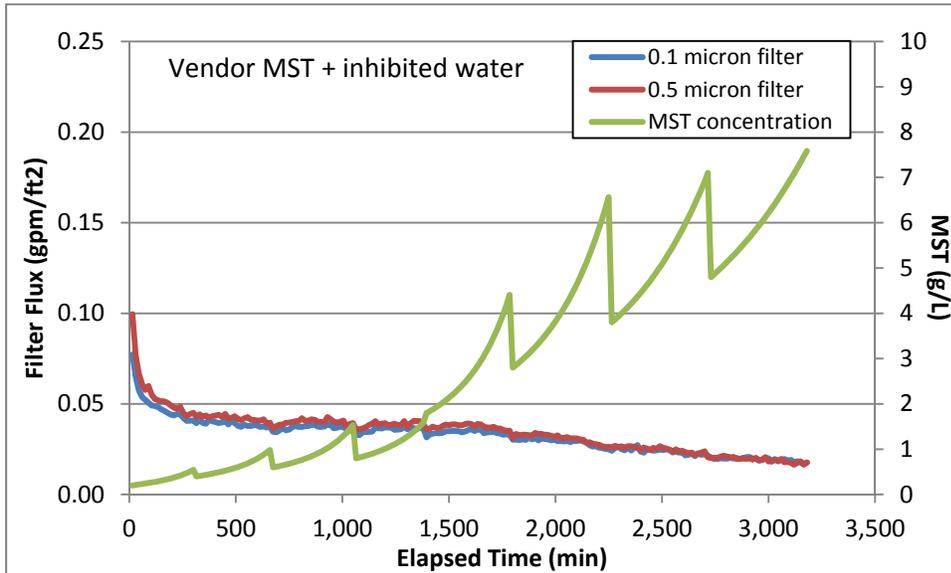

Figure 3. Filter Flux as a Function of Time with 0.01 M NaOH and "Vendor MST"

Figure 4 shows the filter flux of each filter as a function of elapsed run time in the tests with "fewer fines MST". The plot shows no significant difference in filter flux between the 0.1 and 0.5 micron filters and within measurement uncertainty, no different that the results for the "Vendor MST" test. Figure 3 and Figure 4 also show a cumulative concentration of MST in the slurry, assuming that it is a homogeneous slurry and disregarding any buildup of MST cake on the filters.
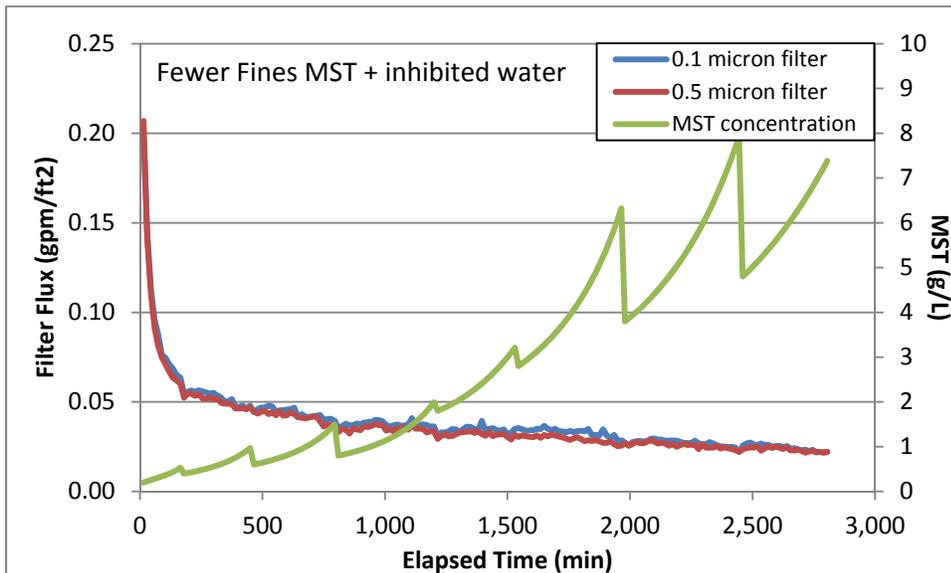

Figure 4. Filter Flux as a Function of Time with 0.01 M NaOH and "Fewer Fines MST"

Figure 5 compares the flux between "vendor MST" and "fewer fines MST". Initially, the "fewer fines" MST produces a larger flux, but after about 500 minutes, there is not a significant difference between the two. Based on an initial feed with no solids, an MST strike of 0.2 g/L, and a final MST feed

concentration of 4.8 g/L, this test (and the tests to be described later) represent the selected intervals of the first 24 batches of a cycle at ARP, but with inhibited water as the supernate.
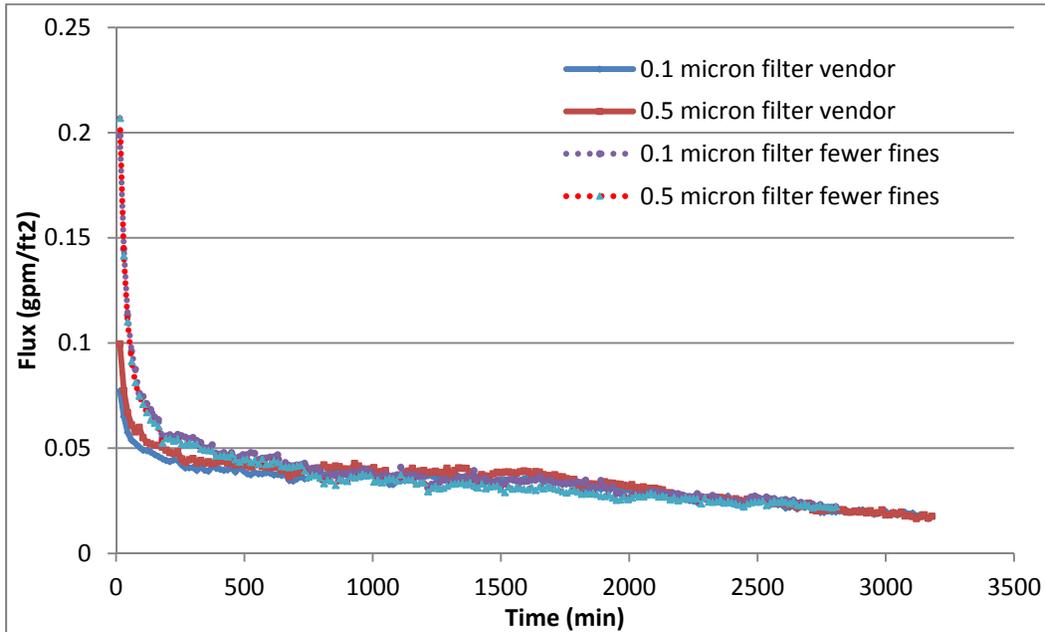

Figure 5. Filter Flux of "Vendor MST" with "Fewer Fines MST"

6.4 M Na, 2.5 M OH Salt Solution

Figure 6 shows the filter flux as a function of time for the feed slurry containing 6.4 M Na, 2.5 M OH Salt Solution and "Vendor MST". The plot shows higher flux with the 0.5 micron filter than the 0.1 micron filter. The difference is small near the end of the day for the three highest solids loadings. The reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

The initial flux at the start of each day is higher than the flux at the end of the previous day. This occurrence is likely due to the filter system not being drained of slurry after a batch and remaining filled overnight, resulting in the solid particles possibly diffusing away from the filter cake or the cake sloughing from portions of the filter tubes. However, for several subsequent batches, the initial flux increases from that of the prior batches. In addition, the average flux for a subsequent batches does not show a general decrease, as would be expected from increasing insoluble solids concentration. A decrease in the ending flux at the three highest solids loadings is observed. One plausible explanation for this result is that the filter cake did not have sufficient time to develop. Very large filter fluxes were measured in this test. Because of the large flux, the time to filter ~ 6 gallons was short. In addition, large differences are observed in the ending fluxes of the 0.1 and 0.5 μm filters. These differences could be an indication that a significant filter cake had not developed. As more MST was added and the filter ran longer (i.e., the last three batches), a significant filter cake developed, which controlled the filter flux for each filter. Based on these results, the operating protocol changed for the filter enhancer tests.

Comparing Figure 6 with Figures 3-5 shows that the filter flux is higher with 6.4 M sodium, 2.5 M free hydroxide than with inhibited water (0.01 M NaOH). This result is surprising given that the viscosity of the 6.4 M solution (~ 3 cp.) is significantly larger than the viscosity of inhibited water (~ 1 cp.). This result will be discussed later.
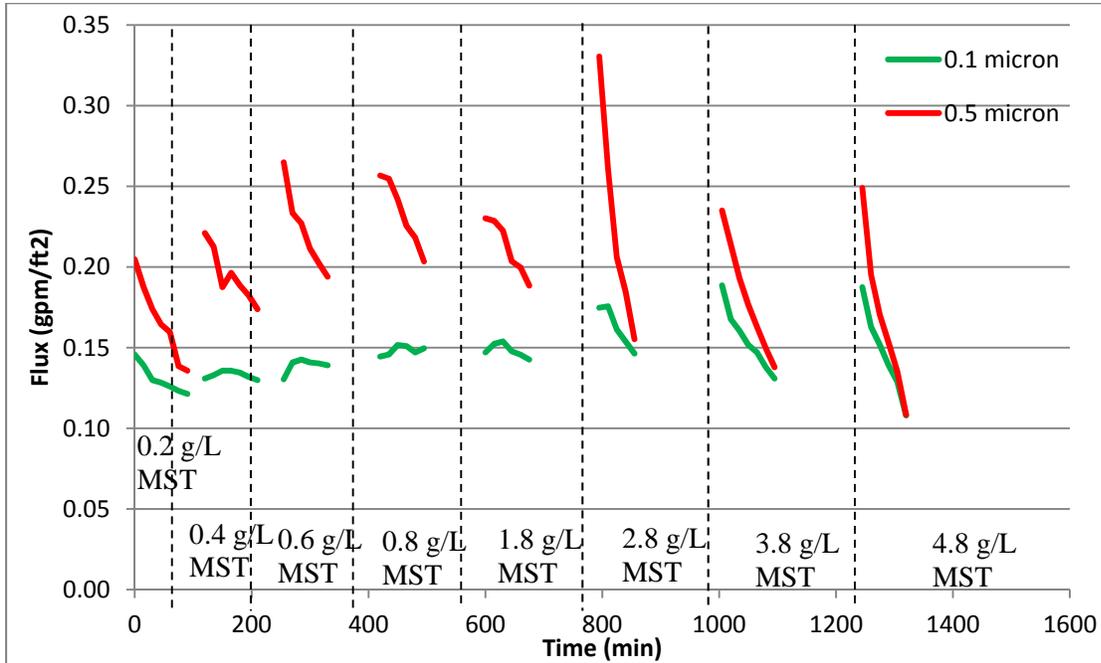
11

Figure 6. Filter Flux as a Function of Time with 6.4 M Na, 2.5 M OH Salt Solution and "Vendor MST"

Figure 7 shows the filter flux as a function of time for the feed slurry containing 6.4 M Na, 2.5 M OH Salt Solution and "Fewer Fines MST". The plot shows higher flux with the 0.1 micron filter than the 0.5 micron filter. The difference is small near the end of the day for the three highest solids loadings. The reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

In this test, the filter was drained overnight for the first three days, and then left filled with liquid overnight for the next five days. When the filter was drained overnight, the starting flux at the beginning of the next day was equal to the flux at the end of the previous day. When the filter system was left filled with liquid overnight, the starting flux at the beginning of the next day was higher than the flux at the end of the previous day. This observation of higher initial flux when the filters were left filled overnight is consistent with the "Vendor MST" results of Figure 6, where the filters were left filled each night. Subsequent tests used a protocol that included keeping the filters filled overnight.

The data in Figure 7 shows a decrease in flux with each batch, which is different from the results in Figure 6. A likely cause of this difference is that the filter system was not drained after the first three batches. Not draining the filter system allowed the cake that had formed to remain and develop. Because of the developed filter cake, the filter flux decreased with each batch.

Figure 7. Filter Flux as a Function of Time with 6.4 M Na, 2.5 M OH Salt Solution and "Fewer Fines MST"

Figure 8 compares the filter flux between the "vendor MST" and the "Fewer Fines MST". The plot shows higher flux with the "vendor MST" compared with the "Fewer Fines MST". Because of differences in operating protocol, comparing the vendor MST with the fewer fines MST is difficult.

Figure 8. Comparing Filter Flux of "Vendor MST" with "Fewer Fines MST"

5.6 M Na Salt Solution

Figure 9 shows the filter flux as a function of time for the feed slurry containing 5.6 M Na, 1.7 M OH Salt Solution and "Vendor MST". The plot shows higher flux with the 0.5 micron filter than the 0.1 micron

filter. The difference is small near the end of the day for the four highest solids loadings. The reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

The increase in flux observed at the end of the first four days is due to the feed flow being stopped, the filter system being allowed to sit with fluid for 5 – 10 minutes, and the filter being restarted. This stopping the flow may have allowed some of the filter cake to settle or diffuse away from the filter surface. Alternatively, this stopping and starting of the feed pump may have caused a shock to the filter system, which caused the filter cake to be dislodged.

Comparing Figure 9 with Figures 4 - 8 shows that the filter flux is higher with 5.6 M sodium than with inhibited water (0.01 M NaOH). This result will be discussed later.

Figure 9. Filter Flux as a Function of Time with 5.6 M Na Salt Solution and "Vendor MST"

Figure 10 shows the filter flux as a function of time for the feed slurry containing 5.6 M Na, 1.7 M OH Salt Solution and "Fewer Fines MST". The plot shows higher flux with the 0.5 micron filter than the 0.1 micron filter. The difference is small near the end of the day for the three highest solids loadings. The reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

The increase in flux observed during the middle and at the end on the last day is due to the feed flow being stopped, the filter system being allowed to sit with fluid for 5 – 10 minutes, and the filter being restarted.

Comparing Figure 10 with Figures 3-5 shows that the filter flux is higher with 5.6 M sodium than with inhibited water (0.01 M NaOH). This result will be discussed later.

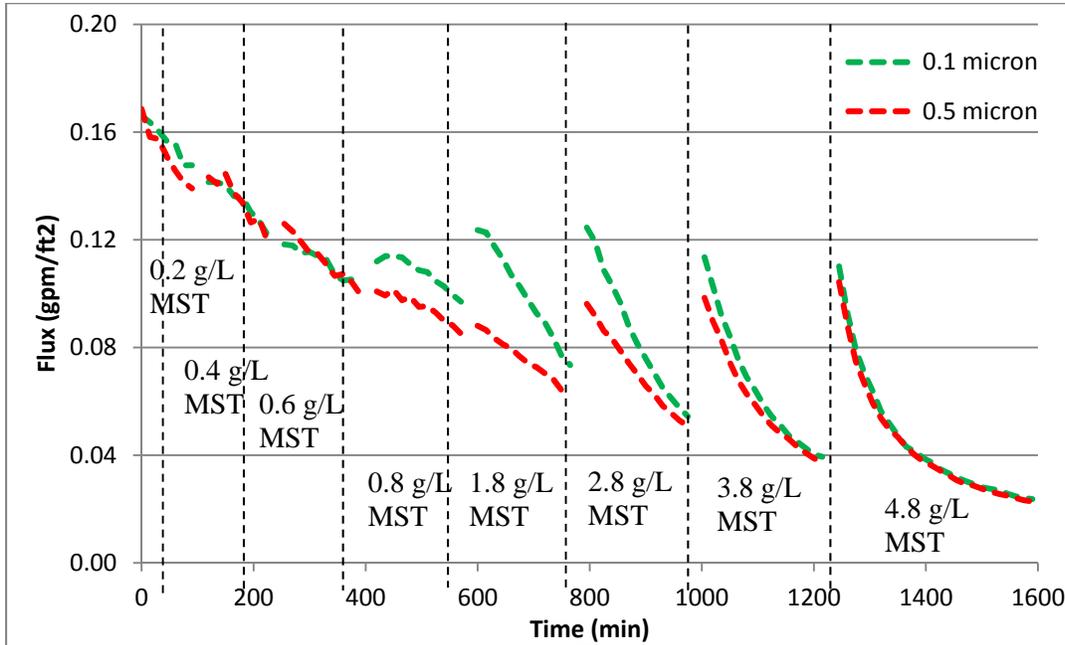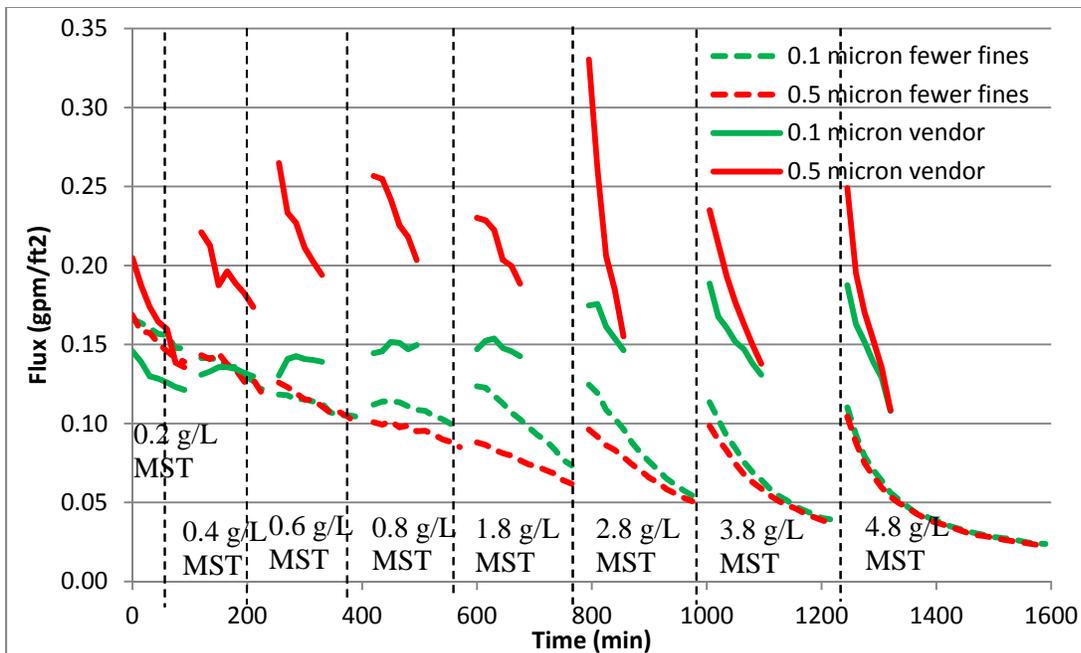**Figure 10. Filter Flux as a Function of Time with 5.6 M Na Salt Solution and "Fewer Fines MST"**

Figure 11 compares the filter flux between the "vendor MST" and the "Fewer Fines MST". The plot shows higher flux with the "vendor MST" at lower solids loading, but the results are inconclusive at the higher solids loadings.
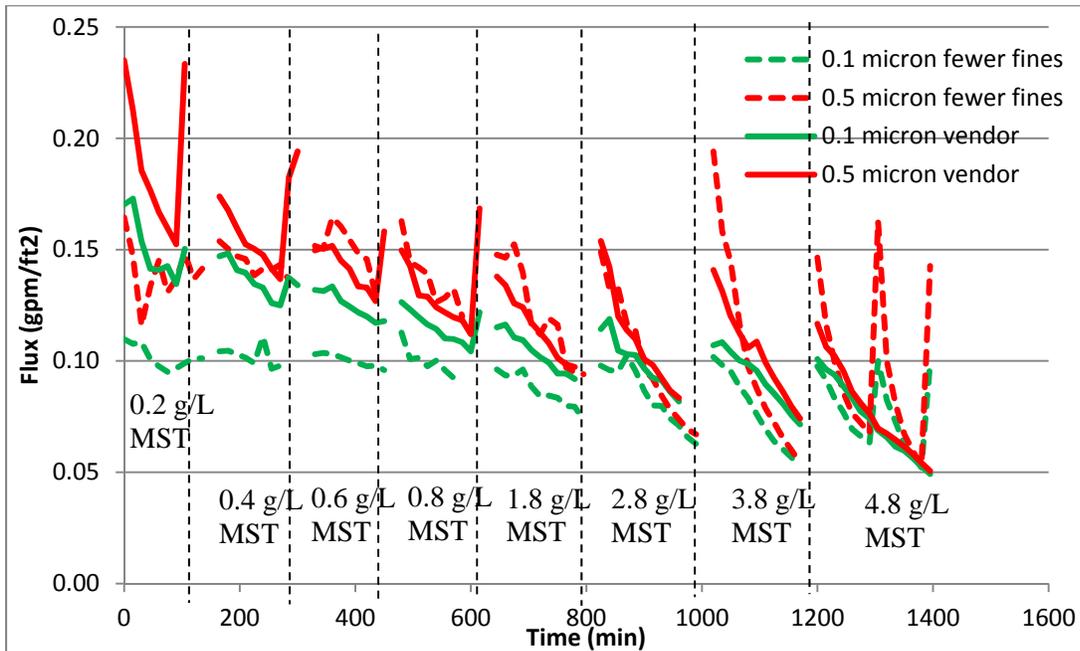
Figure 11. Comparing Filter Flux of "Vendor MST" with "Fewer Fines MST"

<u>6.4 M Na, 1.5 M OH Salt Solution</u>

Figure 12 shows the filter flux as a function of time for the feed slurry containing 6.4 M Na, 1.5 M OH Salt Solution and "Vendor MST". The plot shows higher flux with the 0.5 micron filter than the 0.1 micron filter. The difference is small near the end of the day for the four highest solids loadings. The

reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

Comparing Figure 12 with Figures 3-5 shows that the filter flux is higher with 6.4 M sodium than with inhibited water (0.01 M NaOH). This result will be discussed later.
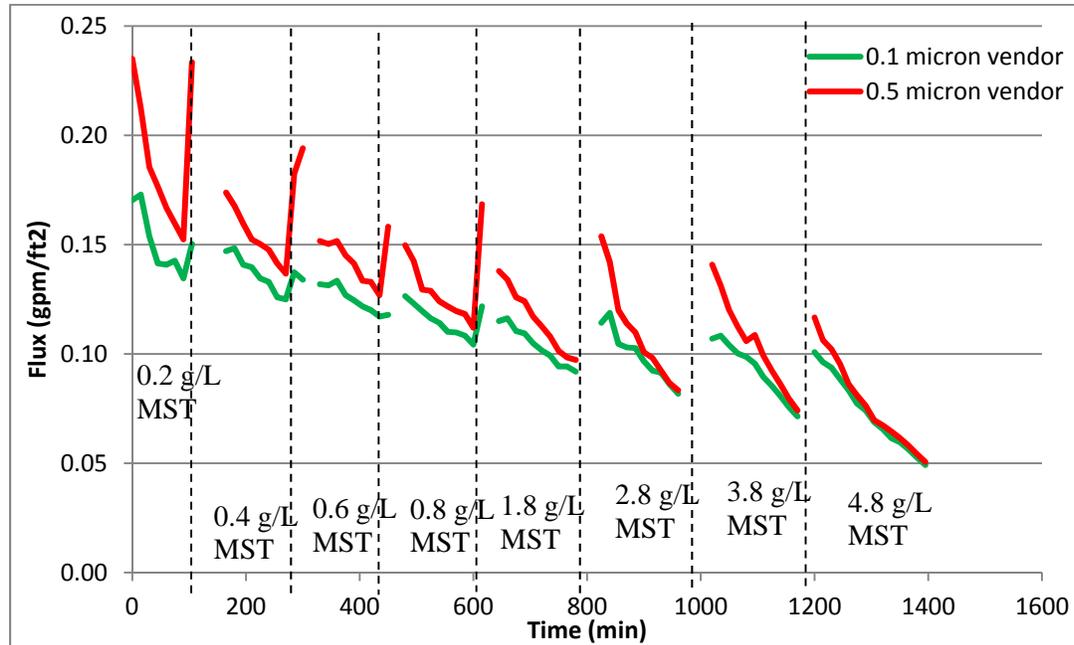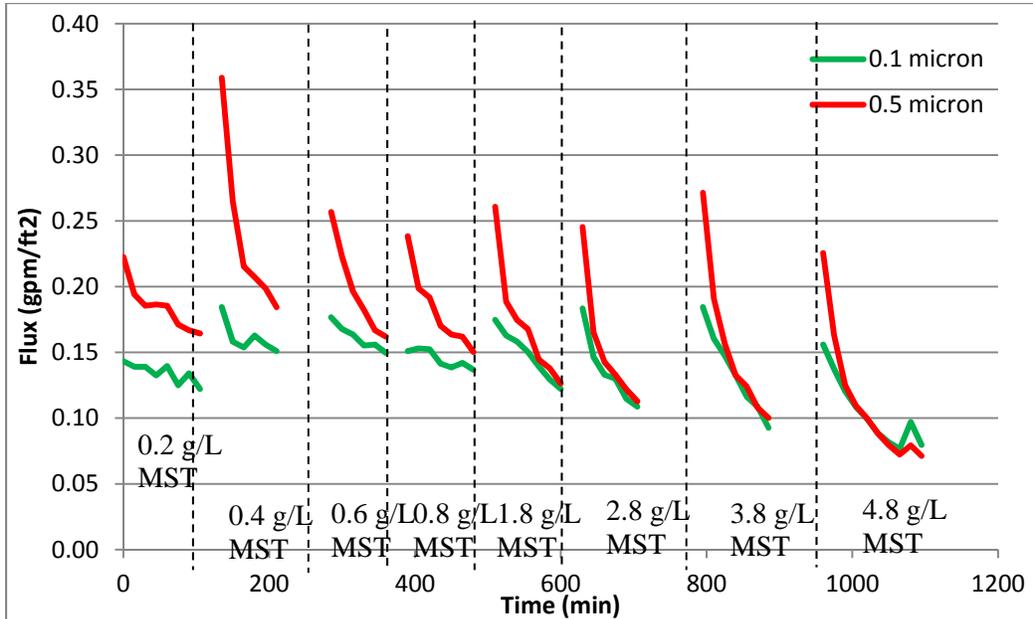

Figure 12. Filter Flux as a Function of Time with 6.4 M Na, 1.5 M OH Salt Solution and "Vendor MST"

Figure 13 shows the filter flux as a function of time for the feed slurry containing 6.4 M Na, 1.5 M OH Salt Solution and "Fewer Fines MST". The plot shows higher flux with the 0.5 micron filter than the 0.1 micron filter. The difference is small near the end of the day for the three highest solids loadings. The reason for this result is likely that a significant filter cake has formed and is the limiting resistance to filter flux.

Comparing Figure 13 with Figures 3-5 shows that the filter flux is higher with 6.4 M sodium than with inhibited water (0.01 M NaOH). This result will be discussed later.
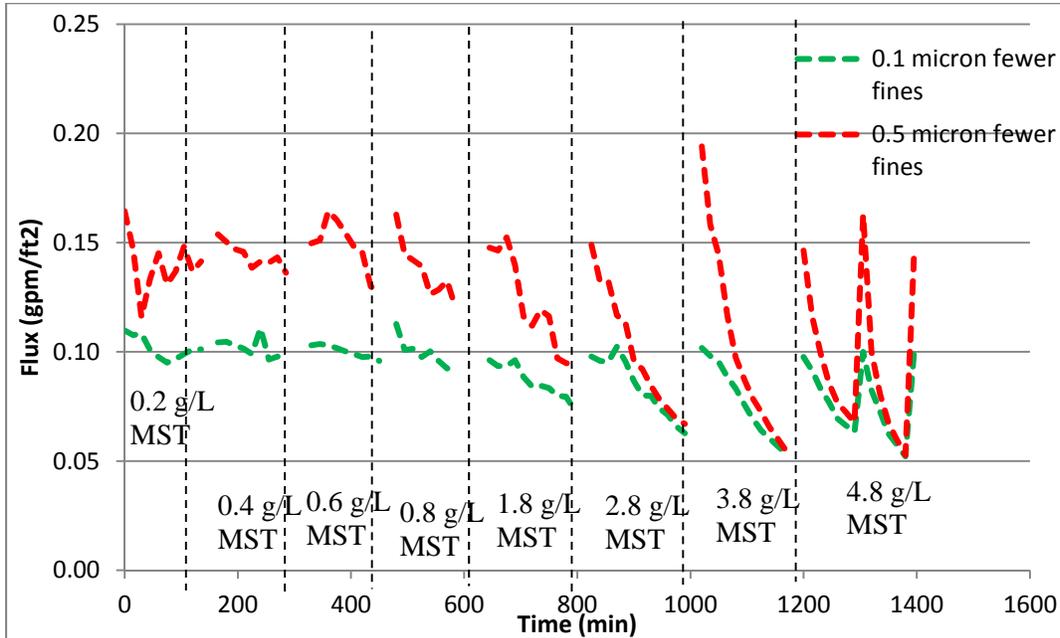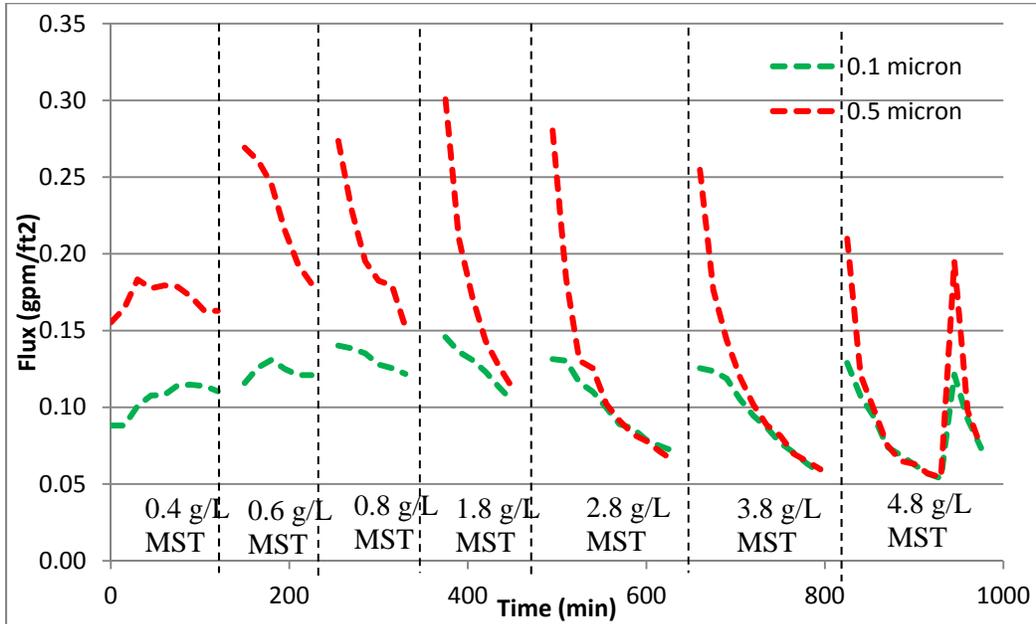
Figure 13. Filter Flux as a Function of Time with 6.4 M Na, 1.5 M OH Salt Solution and "Fewer Fines MST"

Figure 14 compares the filter flux between the "vendor MST" and the "Fewer Fines MST". The plot shows higher flux with the "vendor MST" compared with the "Fewer Fines MST".
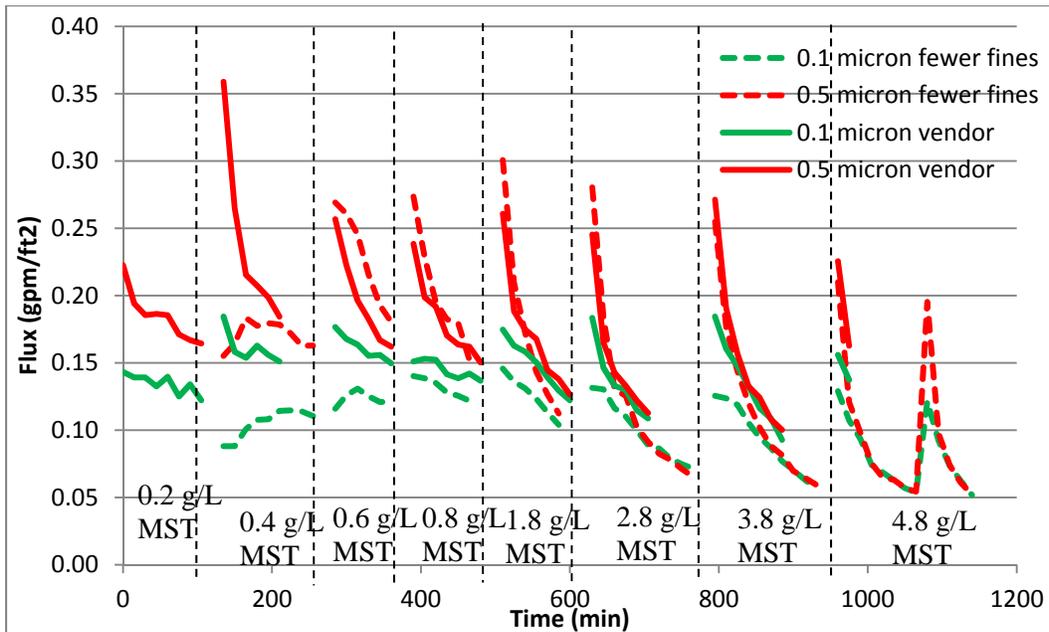
Figure 14. Filter Flux of "Vendor MST" with "Fewer Fines MST"

Particle Size Data

Following each test, feed samples were collected for particle size analysis. The particle size was measured by Microtrac with the carrier fluid being the corresponding salt solution. Table 4 shows the results. The table shows the volume average median particle size, the standard deviation, and the 10th percentile particle size. A larger volume average median particle size would produce a filter cake with less resistance to flow, and therefore, a larger filter flux. A larger standard deviation would allow the

particles to pack more tightly, producing a filter cake with higher resistance to flow, and therefore, a lower filter flux. A larger 10th percentile value would indicate fewer fine particles, which would lead to a higher filter flux.

The table shows significantly larger particle size with the 6.4 M and 5.6 M salt solutions compared with the inhibited water. The larger median particle size or the smaller fraction of fine particles could be the reason for the larger filter flux measured with the higher sodium salt solutions. With the 6.4 M sodium slurry, the data shows a larger particle size and fewer fines with the higher free hydroxide concentration.

The particle analysis was based on single samples from the slurries. Additional sample preparation and particle size analysis is needed to better understand the effect of ionic strength and free hydroxide concentration on MST particle size. However, the MST is typically qualified by measuring particle size in 0.01 M NaOH. A more applicable measurement may be to measure particle size in a solution with higher ionic strength and higher free hydroxide that is more representative of the actual feed processed.

Table 4. Particle Size of Feed Slurries

Feed	Volume Average (μm)	St Dev (μm)	10th percentile (μm)
0.01 M NaOH Fewer fines	4.6	1.8	2.5
6.4 M Na 2.5 M OH Fewer fines	12.1	5.5	4.9
6.4 M Na 2.5 M OH vendor	13.3	5.9	5.7
5.6 M Na vendor	8.3	3.7	3.6
5.6 M Na Fewer fines	6.2	2.8	2.8
6.4 M Na 1.5 M OH vendor	7.2	2.8	3.4
6.4 M Na 1.5 M OH Fewer fines	8.5	3.8	3.5

Effects of Liquid Filled vs Drained Filters During Shutdown

During the testing, researchers observed differences in filter performance if the filter system was left filled with fluid overnight as opposed to being drained overnight. The notable difference is in the startup filtrate flux. When the filters are drained, the next batch's filtrate flux will begin where the last batch's filtrate flux ended. When the filters are left filled with fluid, the next batch's filtrate flux starts out much higher than where the last batch's filtrate flux ended. This phenomenon was observed even after only 10 minutes of shutdown.

This observation was made with a single-tube laboratory-scale filter system. More work is needed to determine whether the ARP filters could be left filled with fluid between batches and whether comparable improvements would occur with a 144 tube filter unit. In addition, the ARP filters have a drain back to the LWPT when not recirculating fluid through the filter system.

Filter Enhancers

Filter Enhancer Tests with Dead-End Filters

A total of 81 dead-end filter tests were conducted to evaluate the filter enhancers obtained. The first set of tests was performed with five baseline solutions. The second set was performed with ten filter aids, which were conducted at two different concentrations of filter aid and MST. This resulted in 40 tests. Finally, the third set of tests was performed with nine different body feeds. Once again, they were tested at two different concentrations of body feed and MST concentration, which resulted in 36 tests. The results are discussed in the next three sections.

Baseline Solutions

Before testing the filter enhancers, five baseline solutions were filtered in the same type of dead-end filters to demonstrate the response of the filter cups. Inhibited water was included in the set because it is

commonly used to flush filters after acid cleaning in order to transition to alkaline waste streams and was used in this test campaign to both apply the filter aids to the filter media and wet the filter media before each test. To begin the tests, 150 mL of the five solutions was put into five side-by-side 0.45 micron media filter cups (see Figure 2). A vacuum of 75 ±5 Torr was applied to the common plenum onto which all five filter cups were attached. When the first filter cup was emptied the test was stopped, the time noted, and the volume of filtrate in each each cup was measured. Figure 15 shows the results, there is a clear distinction between the water and inhibited water solutions and the simulant solutions (with MST). The filter fluxes were approximately a factor of 6 greater than the salt-based solutions. The large sodium ion concentration and higher density and viscosity likely played a role in the lower flow rates for the salt simulant tests. Both of the slurries tested resulted in the same flux of 0.64 gpm/ft^2, but interestingly the supernatant of the salt solution, i.e., that contained no solids, had a filter flux slightly lower than the two slurries. Possibly the MST solids created a filter cake on the nylon filter medium to help improve the filter flux.
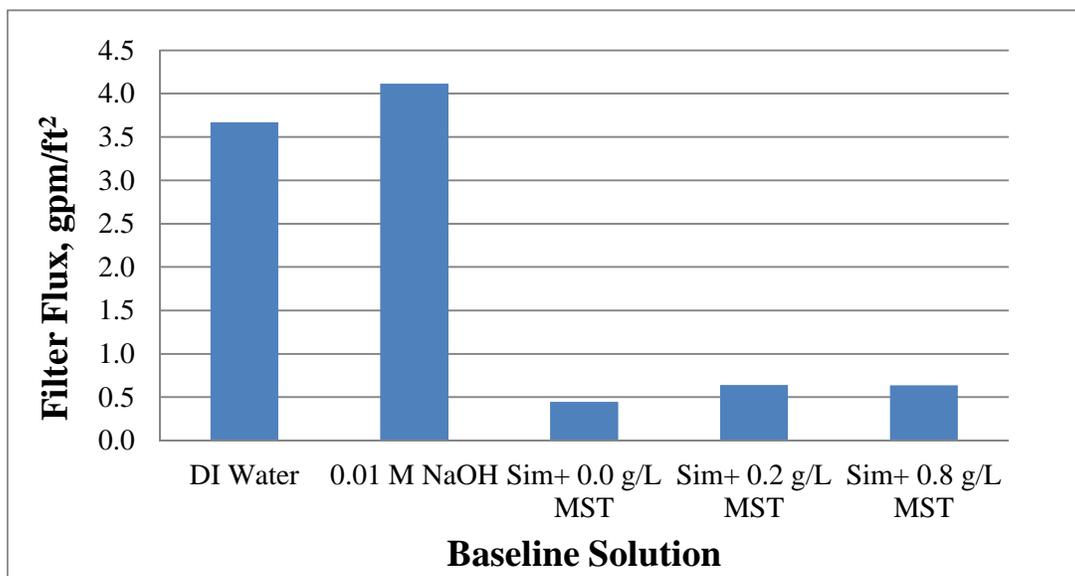
Figure 15. Dead End Test of Baseline Solutions: T ~ 21°C, Vacuum ~75 Torr

Filter Aids

The first set of filter enhancers to be tested was the filter aids. The results are shown in Figure 16 and are normalized by the filter fluxes for the appropriate slurry of salt simulant without a filter aid; see Figure 15, which coincidentally were the same, i.e., 0.64 gpm/ft^2. Unfortunately, the results did not show a clear improvement in filter flux. On one test of one filter aid there was an improvement, but the improvement was small, on the order of 5%, and likely within the uncertainty of the test. That was the DF-200, wood fibers, at its low concentration of 172 mg/150 mL of solution, see Test Matrix table in Appendix A for the MST concentration of 0.75 g/L. Therefore, for the 7.5 M Na simulated waste stream with up to 0.75 g/L of MST these filter aids do not improve the filter flux rate. No crossflow filter tests were performed with filter aids.
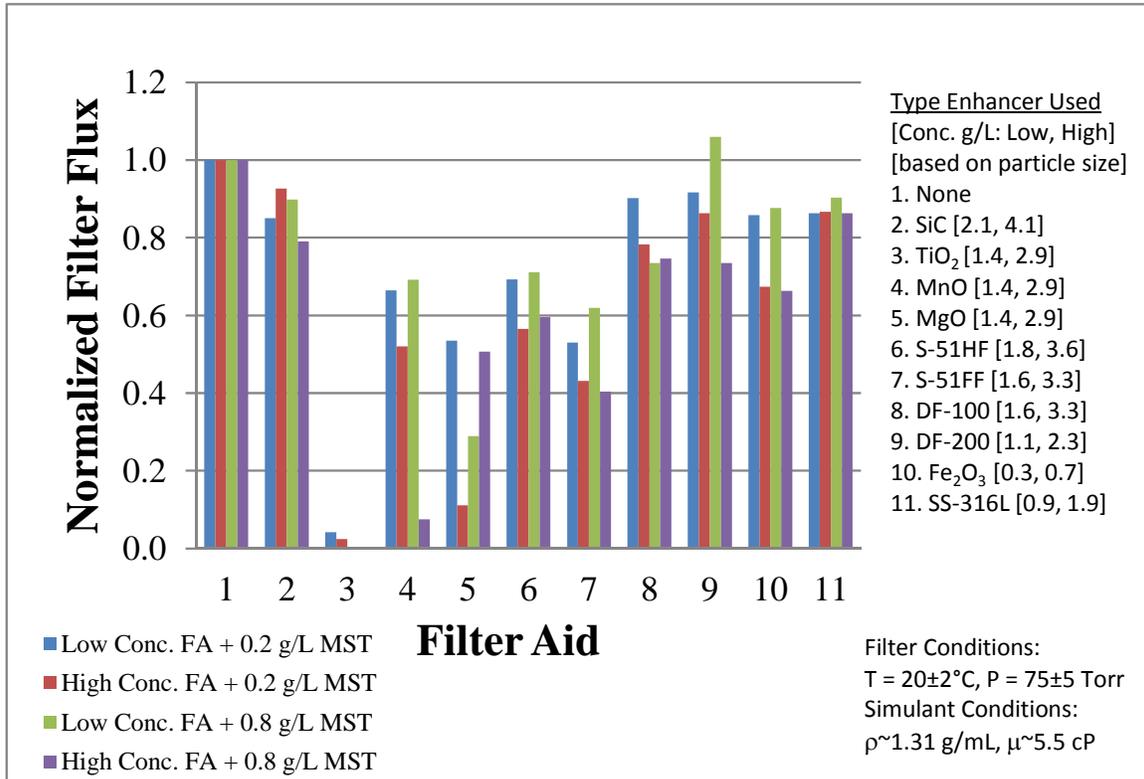
Figure 16. Normalized filter flux of filters coated with filter aids to filter flux without filter aids

Body Feeds

The second set of filter enhancers to be tested was the body feeds. The results are shown in Figure 17 and are normalized by the filter flux rate for the appropriate slurry of salt simulant without a body feed; see Figure 15, which coincidentally was the same, i.e., 0.64 gpm/ft². Once again, as for the filter aids, the results of the body feeds did not show a clear improvement in filter flux. There were two tests that showed a slight improvement, on the order of 10%, likely within the uncertainty of the test. That was the Iron Oxide (hematite) and one of the Cytec emulsion flocculants, i.e., HX-800. Those two body feed combinations had concentrations of 10 mg/150 mL of the 0.2 g/L MST solution and 75 mg/150 mL of the 0.8 g/L MST solution, respectively. See Test Matrix tables in Appendix A for the body feed and MST concentrations. Therefore, for the 7.5 M Na simulated waste stream with up to 0.8 g/L of MST these body feeds do not significantly improve the filter flux rate. The fact that no significant improvement was realized with these filter enhancers, as demonstrated in the past [4 - 7], may indicate that MST solids do not respond, e.g. flocculate, to the enhancers and only when a waste has significant sludge solids will the effect of such enhancement techniques be significant.
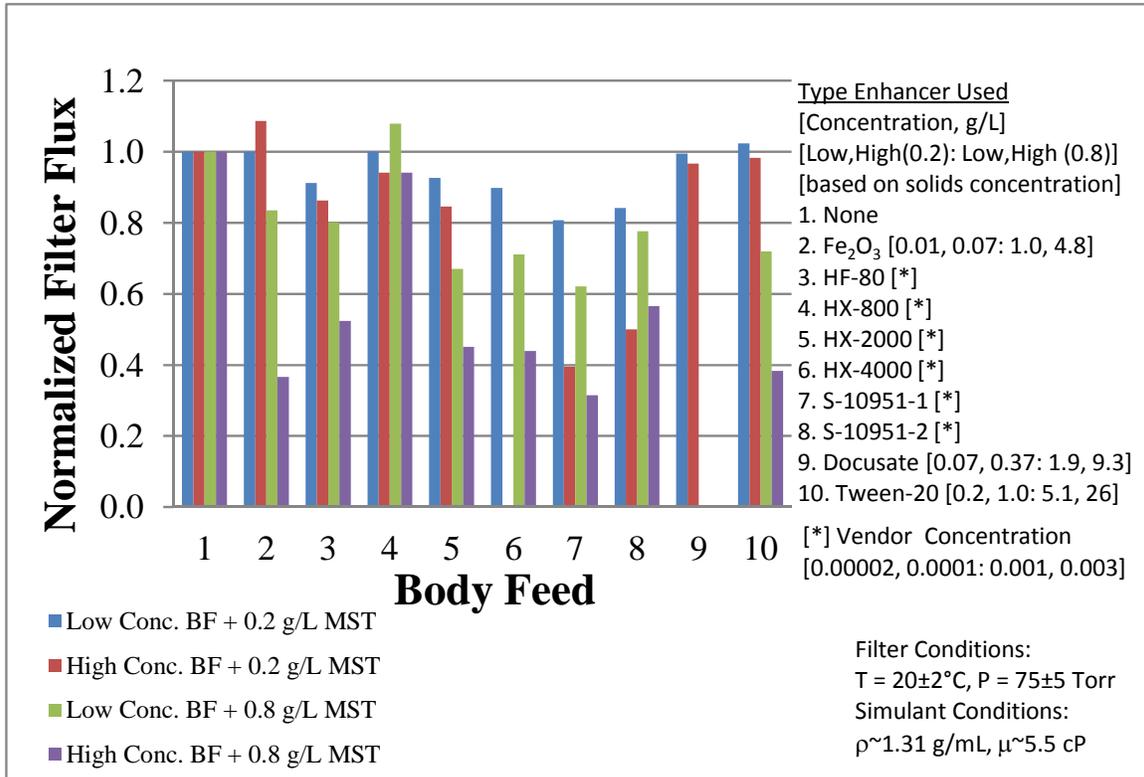
Figure 17. Normalized filter flux of simulant with body feeds to filter flux without body feeds

Crossflow Filter Tests

Because hematite produced a 10% improvement in deadend filter flux as a body feed, and is not anticipated to have an adverse effect on downstream processes, crossflow filter tests were performed with this filter enhancer as a body feed. Hematite was added directly to the 2.5 g/L MST simulant feed to attain a concentration of 0.75 g/L. The filtering procedure was similar to the previously described method, repeating the test with the same feed slurry on subsequent days for a total of four batches. Figure 18 shows the results.

With the 0.5 μm filter, the initial flux with hematite addition was lower than the flux without hematite addition. At the end of a batch, the difference was small. With the 0.1 μm filter, the initial flux with hematite addition was lower than the flux without hematite addition. At the end of a batch, the difference was small. The difference was smaller with the 0.1 μm filter than with the 0.5 μm filter.

The flux following hematite addition was lower than the flux with the slurry containing MST only. Thus, this testing did not show hematite to be an effective body feed.
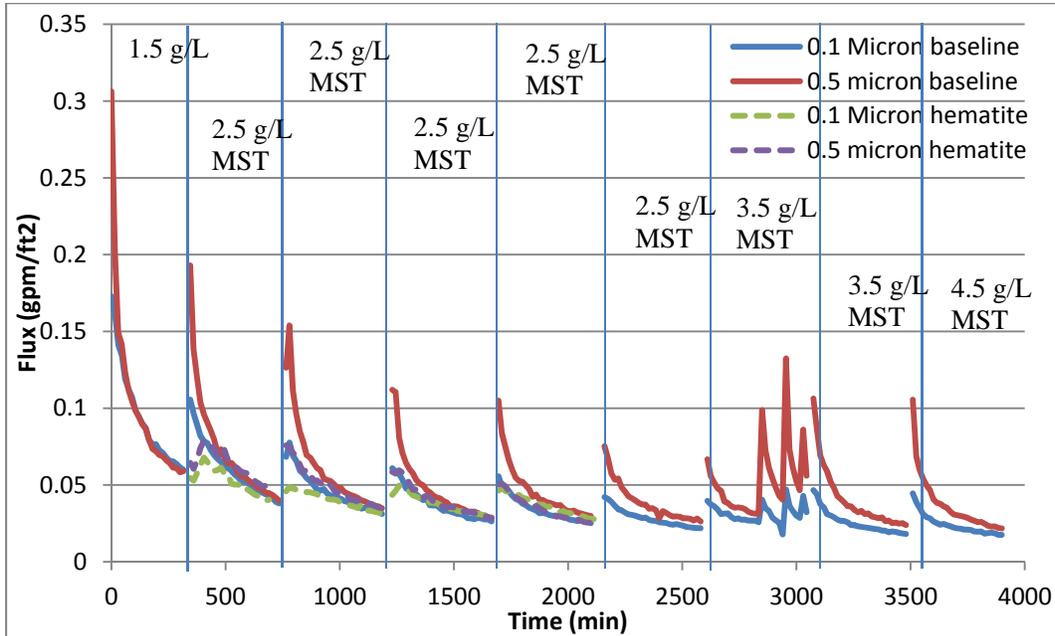
Figure 18. Effect of Hematite Addition as a Body Feed on Crossflow Filter Flux

Effects of Very Short Term Pump Shutdown (Passive Cleaning)

A test was conducted with the baseline slurry from the filter enhancer tests (see Figure 18) to investigate alternative methods to clean the crossflow filters. In this test, the filters were cleaned by stopping the filter feed pump for ~20 seconds (passive cleaning), backpulsing the filters, or stopping the filtrate flow and increasing the axial velocity (i.e., scouring).

Figure 19 shows the results. Both the stopping the pump for ~ 20 seconds and backpulsing provided a significant, but temporary, increase in filter flux. The scouring did not provide much increase in filter flux, but that result may be due to the size of the filter feed pump, which only increased the axial velocity from 8.7 ft/s to 10.2 ft/s.

Temporarily stopping and restarting the pump allowed some of the filtrate to drain back into the feed side of the filter, which could remove some of the filter cake. Alternatively, the "shock" of stopping and starting the filter feed pump may have disrupted and removed some of the filter cake.

While a significant, temporary increase in filter flux was observed following backpulsing, backpulsing at the ARP facility has not led to increases in filter throughput. One plausible reason is that the ARP facility is not conducive to backpulsing.

These observations were made with a single-tube laboratory-scale filter system. Additional work is needed to determine whether any of these techniques would increase filter flux in a 144-tube system. In addition, frequents stops and starts of the filter feed pump could reduce its operating life.
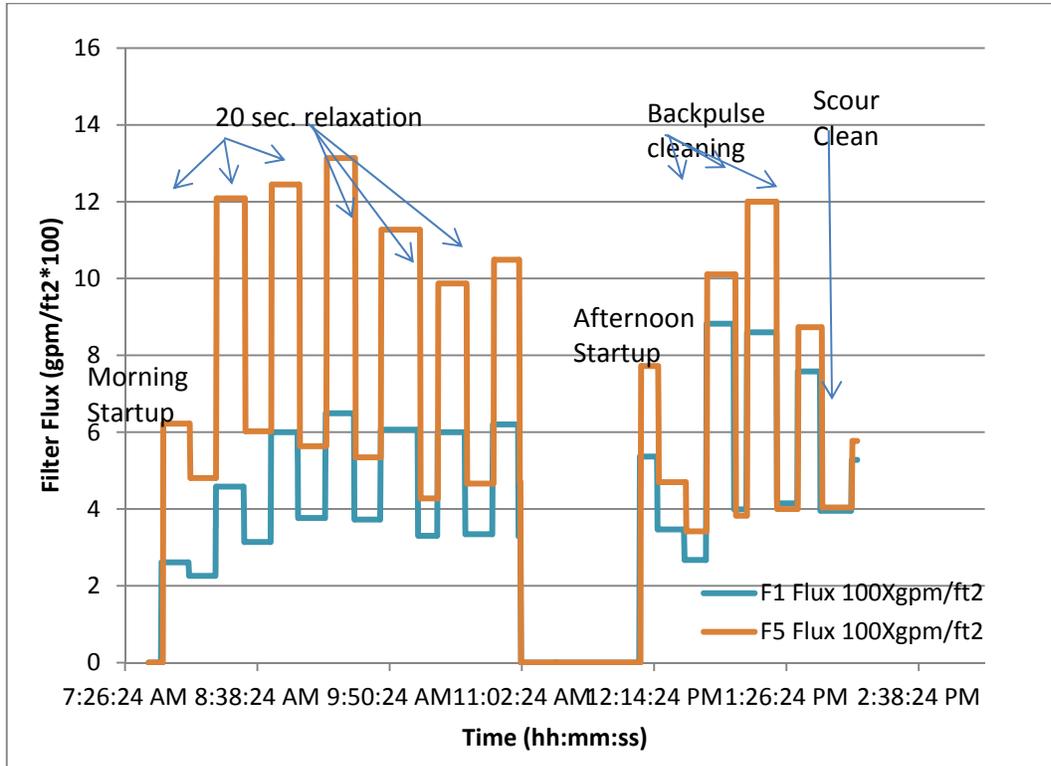
Figure 19 Cleaning Techniques with 13.5 g/l MST in ~7M Na

Discussion

Figure 20 shows the average flux for each of the eight combinations of feed slurries tested, i.e. the four supernatant chemistries (see Table 1) with both types of MST batches ("vender" and "fewer fines."). With the exception of the inhibited water data, the vendor MST produced higher flux than the "fewer fines" MST. With inhibited water, the differences are small. Based on these results, additional effort should not be spent trying to reduce the amount of fines in existing batches of MST.

The 0.5 micron filter produced approximately the same flux as the 0.1 micron filter, or a higher flux. At the end of the tests, the flux of the 0.5 micron filter was approximately equal to the flux of the 0.1 micron filter, because the filter cake is controlling the filter flux and the cake resistance is approximately the same for both filters.

There appears to be an effect of supernate composition on MST particle size measured. The increase in particle size with increased ionic strength could be the reason that the higher ionic strength (and higher viscosity) supernate solutions produced higher filter flux. There could also be an effect from the change in operating protocol during the tests.

Comparing the flux with 6.4 M sodium, 2.5 M free hydroxide, vendor MST with the flux for 6.4 M sodium, 1.5 M free hydroxide, vendor MST shows a higher flux with higher free hydroxide with both filters.[d] The particle size data (see Table 4) showed the 6.4 M sodium, 2.5 M free hydroxide solution to have a larger particle size than the 6.4 M sodium, 1.5 M free hydroxide solution, which could be the reason for the higher flux measured. Comparing the flux of 6.4 M sodium, 1.5 M free hydroxide with the flux for 5.6 M sodium, 1.7 M free hydroxide shows higher flux with the 6.4 M sodium, 1.5 M free

---

[d] Because of differences in operating protocol, a good comparison cannot be made with the "fewer fines" MST.

hydroxide solution for both filters and both sources of MST. Table 4 showed inconclusive results on relative particle size of the 6.4 M sodium, 1.5 M free hydroxide and 5.6 M sodium, 1.7 M free hydroxide solutions. The data in

Figure 20 suggests that increasing sodium concentration and increasing free hydroxide concentration could lead to higher filter flux with MST-containing slurries. However, this testing was performed with a simple salt solution, and the effect of higher ionic strength and free hydroxide concentration on salt solubility must be considered as well.
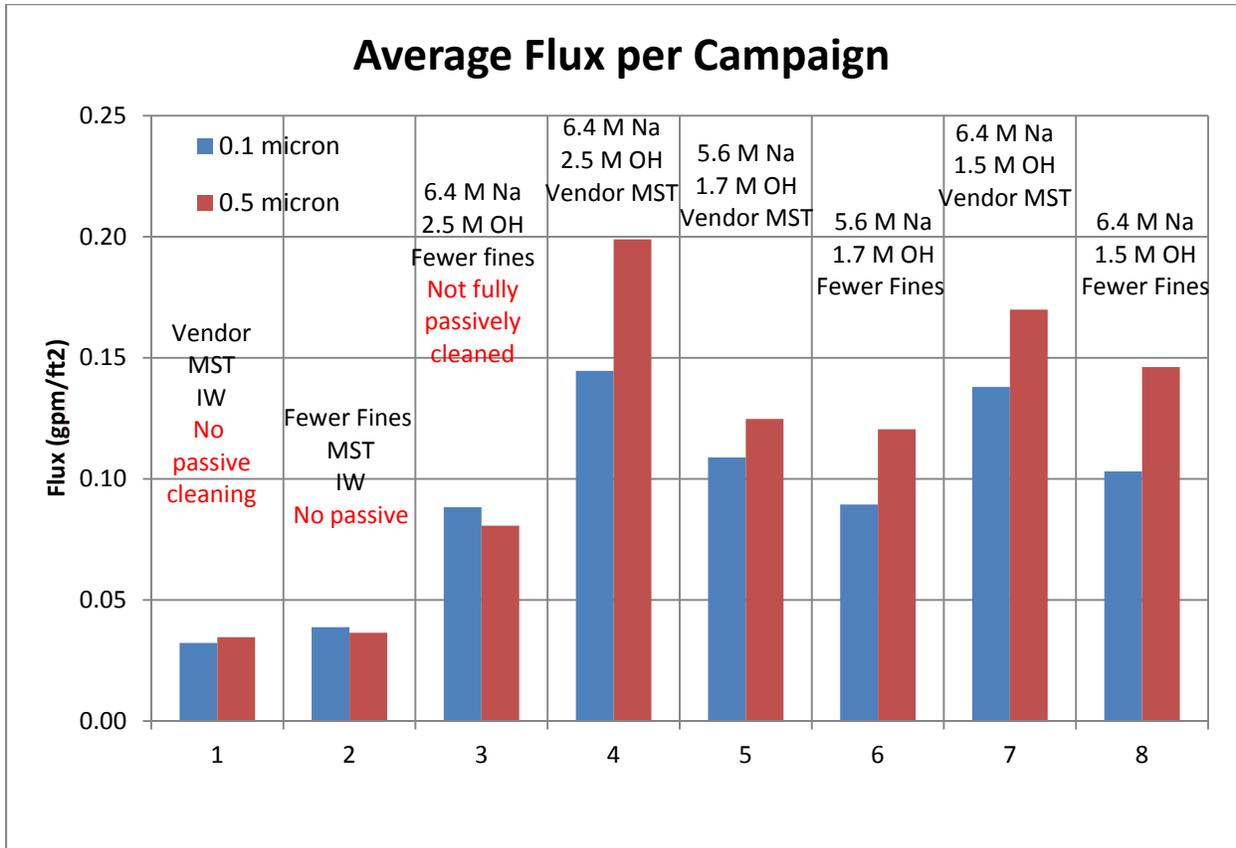
Figure 20. Average Filter Flux as a Function of Feed Slurry

Filtrate samples were collected and examined visually to look for solid particles. All samples, except for two, showed no visible solid particles. The two samples were from the 0.5 micron filter and occurred at the same stage in the filtration process with inhibited water. Subsequent samples showed no visible solids. We are uncertain of the reason for the visible particles in the two samples.

Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. The work is documented in a laboratory notebook. All M&TE was calibrated before the work started and will be calibrated after the work is complete.

Conclusions

The conclusions from this work follow.

- The 0.5 micron filter produced the same or higher flux than the 0.1 micron filter. As the concentration of insoluble solids increased and the filter cake became more established, the difference became small. While this testing did not look at solids rejection by the filter, a 0.5 micron filter has larger pores and may allow more solid particles to pass through the filter.
- The "fewer fines MST" did not produce a higher flux than the "vendor MST", so efforts to remove the fine particles from the MST should not be pursued.
- The higher ionic strength, higher free hydroxide slurries produced higher flux than the lower ionic strength, lower free hydroxide slurries. Particle size data showed larger particles with the higher ionic strength, higher free hydroxide slurries. Additional laboratory-scale testing should be conducted to investigate this phenomenon.
- None of the filter aids or body feeds produced a significant increase in filter flux. One likely cause of this is the solid particles being MST. If testing identifies other solid particles contributing to the fouling at ARP, additional tests with filter aids or body feeds should be considered.
- Filtrate samples collected and examined visually showed that both pore size filters removed solid particles from the feed.
- Leaving the filter tubes filled between batches rather than draining the tubes produced an increase in the initial filter flux. SRR personnel should investigate implementing this practice at ARP.
- Periodically stopping the flow of slurry through a crossflow filter for even as little as 20 seconds appears to temporarily improved flux rate. Further testing should be performed to demonstrate the relationship between stopping time and flux improvement.
- The short duration on many of the tests conducted prevented the formation of a "good" filter cake. The test protocol was modified to allow more time for a "good" filter cake to develop. Future testing should use this protocol.

References

1. I. Bodey, "Filterability Tests using SRR's reconditioned monosodium titanate and vendor supplied monosodium titanate", Z-TTR-S-00003, January 21, 2014.
2. M. R. Poirier and P. R. Burket, "Task Technical and Quality Assurance Plan for MST Filterability Tests", SRNL-RP-2014-00162, February 19, 2014.
3. E. Seufert and B. Voegtlen, "Two MST Strike Tanks at 241-96H Engineering Scope Document", CBU-SPT-2003-00216, December 11, 2003.
4. Duignan, M.R., Nash, C.A., and Poirier, M.R, "Crossflow Filtration: EM-31, WP-2.3.6," Doc. No. SRNL-STI-2011-00071, Rev. 0, February 2011.
5. Walker, W. and McCabe, D.J., "Oak Ridge National Laboratory Melton Valley Storage Tanks Waste Filtration Process Evaluation," Doc. No. WSRC-TR-97-00354, September 1997.
6. Poirier, M., "Improving the Filtration of Sludge/Monosodium Titanate Slurries by the addition of Flocculants," Doc. No. WSRC-TR-2001-00175, March 2001.
7. Duignan, M.R. and Poirier, M.R., "Task Technical and Quality Assurance Plan for Filter Enhancer Identification and Testing for the 512-S Crossflow Filter Facility," Doc. No. SRNL-RP-2014-00356, April 25 2014.
8. Martino, C.J., Herman, D.T., Pike, J.A., and Peters, T.B., "Actinide Removal Process Sample Analysis, Chemical Modeling, and Filtration Evaluation," Doc No. SRNL-STI-2013-00700, Rev 0, June 2014.
9. N. R. Mann, R. S. Herbst, T. G. Garn, M. R. Poirier, and S. D. Fink, "Alternative Ultrafiltration Membrane Testing for the SRS Baseline Process", INEEL/EXT-04-01933, June 2004.

10. M. R. Poirier, J. Siler, et.al, "Pilot-Scale Testing of a 0.1 Micron Filter with SRS Simulated High Level Waste", WSRC-TR-2003-00469, October 8, 2003.

11. C. J. Martino, D. T. Herman, J. A. Pike, and T. B. Peters, "Actinide Removal Process Sample Analysis, Chemical Modeling, and Filtration Evaluation", SRNL-STI-2014-00700,June 2014.

12. D. T. Herman, "Removal of Fines from MST", SRNL-L3100-2014-00149, July 11, 2014.

13. I. Bodey, "Filter Aid and Flocculant Identification and Testing Associated with the 512-S Facility", Z-TTR-S-00004, Rev. 0, March 13, 2014.

Appendix A. Test Matrices – Dead-End Filter Tests

The dead-end filter tests were performed in two basic groups: At the minimum expected MST loading of 0.2 g/L and an arbitrarily high MST loading of 0.75 g/L to see if the filter enhancer performed differently at a different concentration of solids.

Test Matrix for Dead-End Filter Test and Filter Enhancer List - As of 16 June 2014 - MST Concentration of 0.2 g/L

All dead-end filter will be Nalgene 150 mL Cup Filter with 0.45 micron nylon filter (Cat. No. 150-0045) with a membrane diameter of 50 mm (1,963.5 mm² or 0.021135 ft²)

Baseline Stream - Before enhancing filtration the filter flux with dead-end filtration will be evaluated

	Plan of Action:	To determine if filtrate flux is improved with a filter enhancer the filter flux through the filter is first needed without enhancement. A comparison will be made
		among deionized-filtered (0.2 micron) water, 0.01 NaOH, simulated waste with a solids loading of 0.2 wt% UDS, and simulated waste with a solids loading of
		5 wt% UDS. Furthermore, whichever of the two simulated waste streams filters slower will be used for testing.

Test No.	Base Stream	Product Name	Manufacturer/Supplier	Formula	CAS No.	Comment
BS-1	DI Water	Water		H2O	7732-18-5	Water
BS-2	0.01 M NaOH	Dilute Caustic	Multiple	NaOH	1310-73-2	Inhibited Water (IW)
BS-3	7.5 M Salt	Simulated Waste Stream	SRNL	Multiple	Multiple	ARP Feed at 0.2 wt% UDS
BS-4	7.5 M Salt	Simulated Waste Stream	SRNL	Multiple	Multiple	ARP Feed at 5 wt% UDS

Filter Aids - Applied directly to filter membranse surface before filtering waste stream (1)

mg/150 mL to use

	Plan of Action:	Each filter aid is be mixed in approximately 100 mL of solvent (or 0.01 NaOH or supernatant of slurry) with a mild vacuum to allow the mixture to drain and filter
		aid coat the filter. This process is stopped just before exposing the settled filter aid. Next all side by side filter cups are gently filled with 150 mL of the
		simulated waste stream and filtering is started at same time to determine filterabilty. This process is then repeated at the higher filter aid concentration.

Test No.	Filter Enhancer	Product Name	Manufacturer/Supplier	Fomula	CAS No.	SDS No.	Particle Size, d50, μm	Density at 20°C g/cc	Particle Size for addition cm	with membrane of diam(cm)= x 25 diam mg	5 x 50 diam mg	Comment
FA-1A	Silicon Carbide	Silicon Carbide	C.R. Laurence	SiC	409-21-2	34826-4	63 (220 grit)	3.20	0.0063	309		
FA-1B	Silicon Carbide	Silicon Carbide	C.R. Laurence	SiC	409-21-2	34826-4	63 (220 grit)	3.20	0.0063		619	
FA-2A	Titanium Oxide	Titanium Oxide	Alfa Aesar	TiO2 (anatase)	13463-67-7	11284-1	44 (-325 mesh)	3.90	0.0044	216		
FA-2B	Titanium Oxide	Titanium Oxide	Alfa Aesar	TiO2 (anatase)	13463-67-8	11284-1	44 (-325 mesh)	3.90	0.0044		432	
FA-3A	Manganese Oxide	Manganese Oxide	Johnson Matthey	Mn (IV) oxide	1313-13-9	31600-1	44 (-325 mesh)	5.03	0.0044	216		
FA-3B	Manganese Oxide	Manganese Oxide	Johnson Matthey	Mn (IV) oxide	1313-13-9	31600-1	44 (-325 mesh)	5.03	0.0044		432	
FA-4A	Magnesium Oxide	Magnesium Oxide	Sigma	MgO	1309-48-4	10718-1	44 (-325 mesh)	3.58	0.0044	216		
FA-4B	Magnesium Oxide	Magnesium Oxide	Sigma	MgO	1309-48-4	10718-1	44 (-325 mesh)	3.58	0.0044		432	
FA-5A	Activated Carbon	DARCO S-51HF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	40 to 55	0.50	0.0055	270		
FA-5B	Activated Carbon	DARCO S-51HF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	40 to 55	0.50	0.0055		540	
FA-6A	Activated Carbon	DARCO S-51FF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	30 to 50	0.50	0.0050	245		
FA-6B	Activated Carbon	DARCO S-51FF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	30 to 50	0.50	0.0050		491	
172	Cellulose Fiber	Dicaflock DF-100	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	50 (fiber length)	1.50	0.0050	245		
FA-7B	Cellulose Fiber	Dicaflock DF-100	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	50 (fiber length)	1.50	0.0050		491	
FA-8A	Cellulose Fiber	Dicaflock DF-200	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	35 (fiber length)	1.50	0.0035	172		
FA-8B	Cellulose Fiber	Dicaflock DF-200	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	35 (fiber length)	1.50	0.0035		344	
FA-9A	Iron Oxide	Iron Oxide	Brodmann	Fe2O3	1317-61-9	27660-1	9 to 10	5.10	0.0010	49		Could be body feed, too
FA-9B	Iron Oxide	Iron Oxide	Brodmann	Fe2O3	1317-61-9	27660-1	9 to 10	5.10	0.0010		98	Could be body feed, too
FA-10A	Stainless Steel Fiber	BEKINOX SF AISI 316L	Bekaert Fibre Technologies/National Filter Media	316L Stainless Steel	7440-02-0	47779-1-1	2 μm dia. X 3 mm long	8.00	0.0026	129		
FA-10B	Stainless Steel Fiber	BEKINOX SF AISI 316L	Bekaert Fibre Technologies/National Filter Media	316L Stainless Steel	7440-02-0	47779-1-1	2 μm dia. X 3 mm long	8.00	0.0026		257	

Body Feeds - Applied to waste steam before filtering to modify solids for a more permeable filter cake (2)

	Plan of Action:	Each body feed is be mixed in approximately 150 mL of the simulated waste steam and allowed to come to equilibrium for at least 30 minutes. Next all side
		by side filter cups are gently filled with the simulant and filtering is started at the same time to determine filterability. This process is repeated at the higher
		body feed concentration.

Test No.	Filter Enhancer	Product Name	Manufacturer/Supplier	Fomula	CAS No.	SDS No.	Particle Size, μm	Density at 20°C g/cc	Particle Size for addition cm	Body Feed mg/150 mL	Comment
BF-1A	Flocculent	Iron Oxide	Brodmann	Fe2O3	1317-61-9	27660-1	9 to 10	5.1	N/A	2 (3)	Could be a filter aid, too - MW=231.54
BF-1B	Flocculent	Iron Oxide	Brodmann	Fe2O3	1317-61-9	27660-1	9 to 10	5.1	N/A	10 (3)	Could be a filter aid, too - MW=231.55
BF-2A	Flocculent	HF-80	Cytec Ind. - Stamford Lab.	Cyfloc HF-80 Flocculent	Multiple	49564	Emulsion	N/A	N/A	0.003 (4)	Anionic Polyacrylamide
BF-2B	Flocculent	HF-80	Cytec Ind. - Stamford Lab.	Cyfloc HF-80 Flocculent	Multiple	49564	Emulsion	N/A	N/A	0.015 (4)	Anionic Polyacrylamide
BF-3A	Flocculent	HX-800	Cytec Ind. - Stamford Lab.	Cyfloc HF-800 Flocculent	Multiple	49528	Emulsion	N/A	N/A	0.003 (4)	Modified Polyacrylamide
BF-3B	Flocculent	HX-800	Cytec Ind. - Stamford Lab.	Cyfloc HF-800 Flocculent	Multiple	49528	Emulsion	N/A	N/A	0.015 (4)	Modified Polyacrylamide
BF-4A	Flocculent	HX-2000	Cytec Ind. - Stamford Lab.	Cyfloc HF-2000 Flocculent	Multiple	49536	Emulsion	N/A	N/A	0.003 (4)	Modified Polyacrylamide
BF-4B	Flocculent	HX-2000	Cytec Ind. - Stamford Lab.	Cyfloc HF-2000 Flocculent	Multiple	49536	Emulsion	N/A	N/A	0.015 (4)	Modified Polyacrylamide
BF-5A	Flocculent	HX-4000	Cytec Ind. - Stamford Lab.	Cyfloc HF-4000 Flocculent	Multiple	49633	Emulsion	N/A	N/A	0.003 (4)	Modified Polyacrylamide
BF-5B	Flocculent	HX-4000	Cytec Ind. - Stamford Lab.	Cyfloc HF-4000 Flocculent	Multiple	49633	Emulsion	N/A	N/A	0.015 (4)	Modified Polyacrylamide
BF-6A	Flocculent	Experimental S-10951-Lot#1	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.003 (4)	Modified Hydroxamated Polyacrylamide
BF-6B	Flocculent	Experimental S-10951-Lot#1	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.015 (4)	Modified Hydroxamated Polyacrylamide
BF-7A	Flocculent	Experimental S-10951-Lot#2	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.003 (4)	Modified Hydroxamated Polyacrylamide
BF-7B	Flocculent	Experimental S-10951-Lot#2	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.015 (4)	Modified Hydroxamated Polyacrylamide
BF-8A	Surfactant	Docusate Sodium Salt	Fluka	C20H37NaO7S	577-11-7	47761-1	Wax-like sheet	N/A	N/A	11 (5)	MST Flocculant - MW=444.56 g/mol
BF-8B	Surfactant	Docusate Sodium Salt	Fluka	C20H37NaO7S	577-11-7	47761-1	Wax-like sheet	N/A	N/A	56 (5)	MST Flocculant - MW=444.56 g/mol
BF-9A	Surfactant	Tween-20	Sigma	C58H114O26	9005-64-5	15411-1	Viscous Liquid	N/A	N/A	31 (5)	MST Flocculant - MW = 1226.71 g/mol
BF-9B	Surfactant	Tween-20	Sigma	C58H114O26	9005-64-5	15411-1	Viscous Liquid	N/A	N/A	153 (5)	MST Flocculant - MW = 1226.71 g/mol

Filter Aid and or Body Feed Tests at larger range of concentration or combination of filter aid + body feed

	Plan of Application:	This part of the test will be determined during the previous testing.
		If one or two filter aids or body feeds show promise they will be tested at two or three other concentrations.
		If both a filter aid and body feed show promise they may be tried together to determine is there is a additional benefit to use both at the same time.

Notes:	(1) Filter aids that show promise will be micrographed in a dry state and after the filter is coated.
	(2) Body feeds that show promise wil be micrographed after the filter is coated.
	(3) Arbitrary chosen initial body feed concentration of 10:1 mole ratio of Ti:Hematite based on surfactant experience (see Note 5). Hematite MW=156.69 g/mole
	For 150 mL filtered sample there will be 150 mL * 0.2 g/L MST = 0.03 grams of MST. The MW = 119.88 g/mole for MST (NaTiO3H) ; therefore, 0.03 g/119.88 g/mole =
	0.000250 mole Ti. Try a 20:1 ratio = 0.000250 mole Ti X (1/30) X 156.69 g/mole X 1000 ~ 2 mg for 150 mL and for a 4:1 ratio ~10 mg for 150mL.
	(4) Cytec recommends trying 100 g of flocculent and 500 g / ton of undissolved solids - A sample of 150 mL at 0.2 g/L MST has 0.03 g solids needing 0.003 mg and 0.015 mg
	of flocculent respectively (based on a ton = 1000 kg). However, Cytec requires a 0.1 wt% slurry of flocculents that is added to simulant therefore the amount of
	0.1 wt% flocculent slurry that contains 0.003 mg and 0.015 mg is 3 mg and 15 mg, respectively.
	(5) Experience in making larger MST is 10:1 Ti to Surfactant ratio. As shown in note 3 a 150 mL sample of simulant will have 0.000250 mole Ti. Try ratios 10:1 and 2:1 Ti:Surfactant.

	10:1 ratio	11	mg of BF-8A
	2:1 ratio	56	mg of BF-8B
	10:1 ratio	31	mg of BF-9A
	2:1 ratio	153	mg of BF-9B

Test Matrix for Dead-End Filter Test and Filter Enhancer List - As of 16 June 2014 - MST Concentration of 0.75 g/L

All dead-end filter will be Nalgene 150 mL Cup Filter with 0.45 micron nylon filter (Cat. No. 150-0045) with a membrane diameter of 50 mm (1,963.5 mm^2 or 0.021135 ft^2)

Baseline Stream - Before enhancing filtration the filter flux with dead-end filtration will be evaluated

	Plan of Action:	To determine if filtrate flux is improved with a filter enhancer the filter flux through the filter is first needed without enhancement. A comparison will be made among deionized-filtered (0.2 micron) water, 0.01 NaOH, simulated waste with a solids loading of 0.2 wt% UDS, and simulated waste with a solids loading of 5 wt% UDS. Furthermore, whichever of the two simulated waste streams filters slower will be used for testing.				
Test No.	Base Stream	Product Name	Manufacturer/Supplier	Formula	CAS No.	Comment
BS-1	DI Water	Water		H2O	7732-18-5	Water
BS-2	0.01 M NaOH	Dilute Caustic	Multiple	NaOH	1310-73-2	Inhibited Water (IW)
BS-3	7.5 M Salt	Simulated Waste Stream	SRNL	Multiple	Multiple	ARP Feed at 0.2 wt% UDS
BS-4	7.5 M Salt	Simulated Waste Stream	SRNL	Multiple	Multiple	ARP Feed at 5 wt% UDS

Filter Aids - Applied directly to filter membrane surface before filtering waste stream (1)

	Plan of Action:	Each filter aid is mixed in approximately 100 mL of solvent (or 0.01 NaOH or supernatant of slurry) with a mild vacuum to allow the mixture to drain and filter aid coat the filter. This process is stopped just before exposing the settled filter aid. Next all side filter cups are gently filled with 150 mL of the simulated waste stream and filtering is started at same time to determine filterabilty. This process is then repeated at the higher filter aid concentration.								mg/150 mL to use with membrane of		
								Density at 20°C	Particle Size for addition	diam(cm)= 5		
Test No.	Filter Enhancer	Product Name	Manufacturer/Supplier	Fomula	CAS No.	SDS No.	Particle Size, d$_{50}$, µm	g/cc	cm	x 25 diam mg	x 50 diam mg	Comment
FA-1A	Silicon Carbide	Silicon Carbide	C.R. Laurence	SiC	409-21-2	34826-4	63 (220 grit)	3.20	0.0063	309		
FA-1B	Silicon Carbide	Silicon Carbide	C.R. Laurence	SiC	409-21-2	34826-4	63 (220 grit)	3.20	0.0063		619	
FA-2A	Titanium Oxide	Titanium Oxide	Alfa Aesar	TiO2 (anatase)	13463-67-7	11284-1	44 (-325 mesh)	3.90	0.0044	216		
FA-2B	Titanium Oxide	Titanium Oxide	Alfa Aesar	TiO2 (anatase)	13463-67-8	11284-1	44 (-325 mesh)	3.90	0.0044		432	
FA-3A	Manganese Oxide	Manganese Oxide	Johnson Matthey	Mn (IV) oxide	1313-13-9	31600-1	44 (-325 mesh)	5.03	0.0044	216		
FA-3B	Manganese Oxide	Manganese Oxide	Johnson Matthey	Mn (IV) oxide	1313-13-9	31600-1	44 (-325 mesh)	5.03	0.0044		432	
FA-4A	Magnesium Oxide	Magnesium Oxide	Sigma	MgO	1309-48-4	10718-1	44 (-325 mesh)	3.58	0.0044	216		
FA-4B	Magnesium Oxide	Magnesium Oxide	Sigma	MgO	1309-48-4	10718-1	44 (-325 mesh)	3.58	0.0044		432	
FA-5A	Activated Carbon	DARCO S-51HF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	40 to 55	0.50	0.0055	270		
FA-5B	Activated Carbon	DARCO S-51HF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	40 to 55	0.50	0.0055		540	
FA-6A	Activated Carbon	DARCO S-51FF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	30 to 50	0.50	0.0050	245		
FA-6B	Activated Carbon	DARCO S-51FF	Norit Americas, Inc.	Powdered Activated Carbon	7440-44-0	31806-1	30 to 50	0.50	0.0050		491	
FA-7A	Cellulose Fiber	Dicaflock DF-100	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	50 (fiber length)	1.50	0.0050	245		
FA-7B	Cellulose Fiber	Dicaflock DF-100	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	50 (fiber length)	1.50	0.0050		491	
FA-8A	Cellulose Fiber	Dicaflock DF-200	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	35 (fiber length)	1.50	0.0035	172		
FA-8B	Cellulose Fiber	Dicaflock DF-200	Dicalite/Dicaperl Mineral Corp.	Cellulose	9004-34-6	47755-1	35 (fiber length)	1.50	0.0035		344	
FA-9A	Iron Oxide	Iron Oxide	Brodmann	Fe$_3$O$_4$ (+0.9% /Crystalline Silica)	1317-61-9	27660-1	9 to 10	5.10	0.0010	49		Could be body feed, too
FA-9B	Iron Oxide	Iron Oxide	Brodmann	Fe$_3$O$_4$ (+0.9% /Crystalline Silica)	1317-61-9	27660-1	9 to 10	5.10	0.0010		98	Could be body feed, too
FA-10A	Stainless Steel Fiber	BEKINOX SF AISI 316L	Bekaert Fibre Technologies/National Filter Media	316L Stainless Steel	7440-02-0	47779-1-1	2 µm dia. X 3 mm long	8.00	0.0026	129		
FA-10B	Stainless Steel Fiber	BEKINOX SF AISI 316L	Bekaert Fibre Technologies/National Filter Media	316L Stainless Steel	7440-02-0	47779-1-1	2 µm dia. X 3 mm long	8.00	0.0026		257	

Body Feeds - Applied to waste steam before filtering to modify solids for a more permeable filter cake (2)

	Plan of Action:	Each body feed is mixed in approximately 150 mL of the simulated waste steam and allowed to come to equilibrium for at least 30 minutes. Next all side by side filter cups are gently filled with the simulant and filtering is started at the same time to determine filterability. This process is repeated at the higher body feed concentration.									
								Density at 20°C	Particle Size for addition	
Test No.	Filter Enhancer	Product Name	Manufacturer/Supplier	Fomula	CAS No.	Particle Size, µm		g/cc	cm	Body Feed mg/150mL	Comment
BF-1A	Flocculent	Iron Oxide	Brodmann	Fe$_3$O$_4$ (+0.9% /Crystalline Silica)	1317-61-9	27660-1	9 to 10	5.1	N/A	145 (3)	Could be a filter aid, too - MW=231.54
BF-1B	Flocculent	Iron Oxide	Brodmann	Fe$_3$O$_4$ (+0.9% /Crystalline Silica)	1317-61-9	27660-1	9 to 10	5.1	N/A	724 (3)	Could be a filter aid, too - MW=231.54
BF-2A	Flocculent	HF-80	Cytec Ind. - Stamford Lab.	Cyfloc HF-80 Flocculent	Multiple	49564	Emulsion	N/A	N/A	0.075 (4)	Anionic Polyacrylamide
BF-2B	Flocculent	HF-80		Cyfloc HF-80 Flocculent	Multiple	49564	Emulsion	N/A	N/A	0.375 (4)	Anionic Polyacrylamide
BF-3A	Flocculent	HX-800	Cytec Ind. - Stamford Lab.	Cyfloc HF-800 Flocculent	Multiple	49528	Emulsion	N/A	N/A	0.075 (4)	Modified Polyacrylamide
BF-3B	Flocculent	HX-800	Cytec Ind. - Stamford Lab.	Cyfloc HF-800 Flocculent	Multiple	49528	Emulsion	N/A	N/A	0.375 (4)	Modified Polyacrylamide
BF-4A	Flocculent	HX-2000	Cytec Ind. - Stamford Lab.	Cyfloc HF-2000 Flocculent	Multiple	49536	Emulsion	N/A	N/A	0.075 (4)	Modified Polyacrylamide
BF-4B	Flocculent	HX-2000	Cytec Ind. - Stamford Lab.	Cyfloc HF-2000 Flocculent	Multiple	49536	Emulsion	N/A	N/A	0.375 (4)	Modified Polyacrylamide
BF-5A	Flocculent	HX-4000	Cytec Ind. - Stamford Lab.	Cyfloc HF-4000 Flocculent	Multiple	49633	Emulsion	N/A	N/A	0.075 (4)	Modified Polyacrylamide
BF-5B	Flocculent	HX-4000	Cytec Ind. - Stamford Lab.	Cyfloc HF-4000 Flocculent	Multiple	49633	Emulsion	N/A	N/A	0.375 (4)	Modified Polyacrylamide
BF-6A	Flocculent	Experimental S-10951-Lot#1	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.075 (4)	Modified Hydroxamated Polyacrylamide
BF-6B	Flocculent	Experimental S-10951-Lot#1	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.375 (4)	Modified Hydroxamated Polyacrylamide
BF-7A	Flocculent	Experimental S-10951-Lot#2	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.075 (4)	Modified Hydroxamated Polyacrylamide
BF-7B	Flocculent	Experimental S-10951-Lot#2	Cytec Ind. - Stamford Lab.	Superfloc Flocculent	Multiple	57493	Emulsion	N/A	N/A	0.375 (4)	Modified Hydroxamated Polyacrylamide
BF-8A	Surfactant	Docusate Sodium Salt	Fluka	C$_{20}$H$_{37}$NaO$_7$S	577-11-7	47761-1	Wax-like sheet	N/A	N/A	278 (5)	MST Flocculant - MW=444.56 g/mol
BF-8B	Surfactant	Docusate Sodium Salt	Fluka	C$_{20}$H$_{37}$NaO$_7$S	577-11-7	47761-1	Wax-like sheet	N/A	N/A	1391 (5)	MST Flocculant - MW=444.56 g/mol
BF-9A	Surfactant	Tween-20	Sigma	C$_{58}$H$_{114}$O$_{26}$	9005-64-5	15411-1	Viscous Liquid	N/A	N/A	767 (5)	MST Flocculant - MW = 1226.71 g/mol
BF-9B	Surfactant	Tween-20	Sigma	C$_{58}$H$_{114}$O$_{26}$	9005-64-5	15411-1	Viscous Liquid	N/A	N/A	3837 (5)	MST Flocculant - MW = 1226.71 g/mol

Filter Aid and or Body Feed Tests at larger range of concentration or combination of filter aid + body feed

Plan of Application:	This part of the test will be determined during the previous testing.
	If one or two filter aids or body feeds show promise they will be tested at two or three other concentrations.
	If both a filter aid and a body feed show promise they may be tried together to determine there is a additional benefit to use both at the same time.

Notes: (1) Filter aids that show promise will be micrographed in a dry state and after the filter is coated.

(2) Body feeds that show promise wil be micrographed after the filter is coated.

(3) Arbitrary chosen initial body feed concentration of 10:1 mole ratio of Ti:Hematite based on surfactant experience (see Note 5). Hematite MW=156.69 g/mole

For 150 mL filtered sample there will be 150 mL * 0.75 g/L MST = 0.1125 grams of MST. The MW = 119.88 g/mole for MST (NaTiO3H); therefore 0.1125 g/119.88 g/mole = 0.000938 mole Ti. Try a 1:1 ratio = 0.000938 mole Ti X (1/1.0) X 156.69 g/mole X 1000 ~145 mg for 150 mL and 0.2:1 ratio ~724 mg for 150mL.

(4) Tried a Cytec concentration of 667 g of flocculent and 3,333 g / ton of undissolved solids - A sample of 150 mL at 0.75 g/L MST has 0.1125 solids needing 0.075 mg and 0.375 mg of flocculent respectively (based on a ton = 1000 kg). However, Cytec requires a 0.1 wt% slurry of flocculents that is added to simulant therefore the amount of 0.1 wt% flocculant slurry that contains 0.075 mg and 0.375 mg is 75 mg and 375 mg, respectively.

(5) As shown in note 3 a 150 mL sample of simulant will have 0.000938 mole Ti. Try ratios 1.5:1 and 0.3:1 Ti:Surfactant.

Appendix B. Cytec Flocculent Preparation

The flocculating emulsions obtain from Cytec Industries need preparation before being mixed with the slurry to properly react with the solids. The procedure used to make up the mixture to add to the simulated waste stream is shown below:

###

Assumptions:

- 150 mL salt solution
- 0.2 g MST/L salt solution
- Flocculant dosage 500 g flocculant per 1000 kg of solids (i.e., MST)

Prepation of Concentrated Flocculant Solution:

- Add 1 gram of flocculant to 100 mL of make-up water. This produces a 1 wt% concentrated solution
- When needed, take a subsample of the concentrated solution and dilute 10X to produce a 0.1 wt% flocculant solution

Adding Flocculant to Salt Solution with 0.2 g MST/L:

- Prepare 150 mL salt solution containing 0.2 g MST/L salt solution
- (150 mL) (1:/1000 mL) (0.2 g/L) = 0.03 g MST
- (0.03 g MST) (500 g flocculant/1000 kg of MST) (1kg MST/1000 g MST) = 1.5 e-5 grams of flocculant
- 1.5 e-5 grams flocculant / (0.001 g flocculant/g solution) = 0.015 grams of solution to add to 150 mL of salt solution

Adding Flocculant to Salt Solution with 0.75 g MST/L:

- Prepare 150 mL salt solution containing 0.75 g MST/L salt solution
- (150 mL) (1:/1000 mL) (0.75 g/L) = 0.113 g MST
- (0.113 g MST) (500 g flocculant/1000 kg of MST) (1kg MST/1000 g MST) = 5.7 e-5 grams of flocculant
- 5.7 e-5 grams flocculant / (0.001 g flocculant/g solution) = 0.057 grams of solution to add to 150 mL of salt solution

###

These directions above were based on the procedure shown below obtain from vendor: Cytec Industries

PREPARATION OF FLOCCULANT SOLUTIONS FROM

WATER-IN-OIL EMULSIONS

{Provide by Cytec Industries on 6/10/2014}

Scope:

This procedure covers the preparation of a flocculant from the manufactured form (water-in-oil emulsion) to a useable aqueous solution.

Purpose:

Many flocculants used in the Bayer Process are manufactured in a form that does not allow them to be added to the process "as-is". Thus, they need to be prepared, generally converted to an aqueous form, before they can be tested. This procedure describes how to prepare an aqueous flocculant solution from an oil-in-water emulsion.

Highlights:

- Prepare at 0.5 to 1% "as is" concentration.
- Dilute ten times or as needed at time of testing. Prepare freshly diluted solutions each day.
- Concentrated solutions can be used up to 3 days provided there is no evidence of degradation.

Equipment needed:

- Beaker (250, 400, or 600 mL recommended)

- Magnetic stir plate, or approved blender/hand mixer

- Magnetic stir bar (1.5 inches)

- One hundred milliliter graduated cylinder

- Balance (accurate to 0.01g and 0.0001g)

<u>Procedure:</u>

1. Measure 100 ml make-up water (2% sodium hydroxide in deionized water) into a 250ml beaker (200 mL into a 600 mL beaker if larger volumes are needed).

2. Weigh desired amount of emulsion into a disposable syringe. This is the most accurate way (weight basis). A volumetric basis can also be used, measuring the volume of emulsion by the graduations on the syringe.

3. Stir water using a mechanical stirrer (overhead or magnetic) to create a vortex that extends to the bottom of the beaker

4. Rapidly add the emulsion to the shoulder of the vortex. Addition of emulsion should be at a controlled, uniform, rate.

5. Allow mixture to stir at high speed (750rpm with 1.5 – 2 inch (4-5cm) magnetic stir bar) for about 2 minutes then reduce speed to allow good movement of the solution surface (usually 200rpm will do) .

6. Continue to stir for a minimum of 1 hour (typically 2 hours for lab use) **before use ensuring no un-dissolved emulsion strings in solution otherwise remake solution.**

Distribution:

S. L. Marra, 773-A
T. B. Brown, 773-A
D. H. McGuire, 999-W
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)

E. A. Brass, 249-8H
C. K. Chiu, 704-27S
E. J. Freed, 704-S
A. G. Garrison, 248-9H
B. A. Gifford, 704-56H
K. L. Lang, 707-7E
D. J. Martin, 241-152H
A. R. Shafer, 704-27S

P. R. Jackson, DOE-SR, 703-46A
J. A. Crenshaw, 703-46A