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Results of the SRNL Studies of the MCU Strip Effluent Coalescers

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October 2019
SRNL-STI-2019-00272, Revision 0

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EXECUTIVE SUMMARY

Three potential causes of the strip effluent (SE) coalescer (SEC) failure were investigated: inorganic fouling, organic fouling, and biological fouling. From the analysis of the SEC and leachate samples from the SEC, there is no evidence that the fouling is caused by inorganic precipitates, solids, or salts. The analysis of the SEC shows the presence of organic material such as sec-butylphenol and Modifier (1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-secbutylphenoxy)-2-propanol). The source of the sec-butylphenol would be from the decomposition of the Modifier in the solvent. The analysis of the SEC shows the presence of amide bonds that are likely from bacteria and other biological material. In addition, samples from the Strip Feed Tank (StFT) and the boric acid delivery tanker showed the presence of bacteria and other biological material.

The most likely cause of the SEC fouling is biofouling by bacteria and other organisms that form a biofilm on the SEC fibers. The biofilm would sorb organic material in the feed to the SEC, and not readily release this material. The bacteria would use this trapped organic material to grow the biofilm, which would decrease the porosity of the SEC. The biofilm would thereby impede SEC operation through biofouling.

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LIST OF ABBREVIATIONS

ACTL	Aiken County Technology Laboratory
AD	Analytical Development
ARP	Actinide Removal Process
BoBCalixC6	Calix[4]arene-bis(*tert*-octylbenzo-crown-6)
CDT	Contactor Drain Tank
dP	differential pressure
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
DWPF	Defense Waste Processing Facility
ETF	Effluent Treatment Facility
FTIR	Fourier Transform InfraRed
IC-A	ion chromatography – anions
ICPES	inductively-coupled plasma emission spectroscopy
ISDP	Interim Salt Disposition Project
MaxCalix	1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6
MCU	Modular Caustic-Side Solvent Extraction Unit
Modifier	1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-secbutylphenoxy)-2-propanol
MST	monosodium titanate
NGS	Next Generation Solvent
NMR	Nuclear Magnetic Resonance
PECMC	Portable Equipment Commodity Management Center
PPS	Poly Phenylene Sulfide
SB	Salt Batch
SE	Strip Effluent
SEC	Strip Effluent Coalescer
SEHT	Strip Effluent Hold Tank
SRNL	Savannah River National Laboratory
SSFT	Salt Solution Feed Tank
StFT	Strip Feed Tank
SVOA	Semi Volatile Organic Analysis
TiDG	Tris(isoDecyl)Guanidine, *N,N',N"*–tris(3,7-dimethyloctyl)guanidine
TTQAP	Task Technical and Quality Assurance Plan
TTR	Task Technical Request

1.0 Introduction

On May 22nd, 2018, the Modular Caustic-Side Solvent Extraction Unit (MCU) restarted operations using feed material from Salt Batch (SB) 10 following an approximately 16-month (January 2017 – May 2018) outage where the Defense Waste Processing Facility (DWPF) Melter was replaced and initial tie-ins for the Salt Waste Processing Facility (SWPF) were performed. During the startup, the differential pressure (dP) and the rate of dP increase across the SEC (SEC 1) were higher than anticipated. It should be noted that this SEC was the same element that had been installed before the outage. Operations were terminated after ~22 days of intermittent operation (~8 days of total operation) due to a loss of power from a lightning strike on the East Hill. Prior to the forced shut down, the pressure differential reached ~20 psid (at a salt feed flow rate of 6 gpm), with only ~4.2k gallons of SE material having been produced since resuming operations after the extended outage (and ~33.2k gallons of SE produced over the entire life of the coalescer).

The MCU performed four short "Processing Runs" between December 12-16th, 2017. Each of the four shifts performed one short Processing Run to demonstrate operator proficiency and plant operability. In total, ~3,500 gallons of salt feed (Salt Batch 9, SB9, material) were processed through MCU. This processing volume was limited both by the feed available prior to entering the extended outage in the system, and freeboard in each of the product hold tanks. The differential pressure rose ~3 psid over the course of these evolutions, a rate marginally higher than observed in the prior operations in January 2017. The SEC installed during these Processing Runs was initially installed in the plant in September 2016 and remained in place until replacement in June 2018. SB9 and SB10 material was processed through this same SEC media.

SEC 1 was replaced and a restart with a second coalescer element installed took place on July 2nd, 2018. Again, the rate of increase in dP across the SEC (SEC 2) was unusually high and operations were terminated (after ~19 days of intermittent operation, or ~13 days of flow) after the maximum observed dP reached ~24 psid (at a salt feed flow rate of 4 gpm), with 5.7k gallons of SE having been produced. SEC 2 was replaced and a restart with a third element installed took place on August 6th. SEC 1 had been flushed and discarded after being replaced, but SEC 2 was not flushed to retain as much material causing the pluggage issues as possible and was sent to Savannah River National Laboratory (SRNL) for analysis. While processing with the third SEC (SEC3), the dP rapidly increased at a higher rate than previously observed with either SEC 1 or SEC 2, and operations were terminated after just 15 hours, when the dP across the SEC reached ~25 psid (at a salt feed flow rate of 4 gpm) and only 369 gal of SE had been produced. Attempts to backflush the SEC by reversing flow path through the element did not improve (i.e., reduce) the rate of dP increase (nor the ultimate maximum dP reached).

SEC 3 was replaced, and a restart with a fourth element installed occurred on October 27th, 2018. SEC 3 was transported to SRNL for analysis. The initial dP across the SEC (SEC 4) increased to ~12 psi in a few hours, but then stabilized, only increasing to ~15 psi (at a salt feed flow rate of 4 gpm) after another ~22 hours (385 gallons of SE produced). After a temporary pause to sample and analyze the SE prior to transfer to DWPF, processing was continued starting on October 30th. The initial dP and rate of increase were roughly similar to the previous restart with a slow but

steady increase in the dP across SEC 4. Operations were terminated after ~18 hours and an additional 496 gallons of SE had been produced (~881 gallons over the life of SEC "4"), when the dP reached ~25 psid (at a salt feed flow rate of 4 gpm). SEC 4 was replaced and a restart with the fifth element installed occurred on November 28th. SEC 4 was transported to SRNL for analysis.

MCU shut down briefly on November 29th, 2018 due to a reduction in instrument air pressure (loss of process indications) but operations were resumed that same day. Operations were then paused to sample the SEHT (batch size of 528 gallons of SE), with a maximum dP during the run of ~3.05 psid. Due to a pump failure while attempting to transfer the SE batch, MCU entered an outage and, despite the improved performance between SEC 5 and the previous media, SEC 5 was pre-emptively replaced during the outage based on a Management Decision.

MCU resumed operations from the outage with SEC 6 installed on January 31st, 2019. SEC 6 was the first 40" element installed (all prior media were 20"). Operations were paused to sample the first three SEHT batches prior to transferring downstream. The increase in dP was slow during initial processing (salt feed flow was at 4 gpm) and reached ~13.2 psid on February 19th. The salt feed flow was increased to 6 gpm at this time and the dP increased ~15.9 psid in response. The dP continued to steadily increase until February 22nd when it reached ~25 psid while still processing salt at 6 gpm. The salt feed was then reduced to 4 gpm at this time and the dP dropped to ~18.9 psid in response. The SEC dP continued to increase though and reached ~20.4 psid that night before feed was swapped to DSS. An extended DSS recirculation was performed into February 23rd, and the SEC 6 dP increased to ~25.7 psid before flows were stopped. Approximately 7.25k gallons of SE were produced while processing with SEC 6.

SEC 6 was replaced and a restart with the seventh element installed occurred on March 9th, 2019. Operations were paused to sample the first two SEHT batches prior to transferring. MCU continued to operate continuously with minor interruptions in processing (e.g., Tank 50 level discrepancy, erratic DSSHA level indications, PVV pre-filter replacement). During operations the SEC 7 dP continued to increase at a slower rate than that observed for the previous media. Between the intermittent down times, salt feed was periodically adjusted between 4 gpm and 6 gpm, with no significant changes in dP (<2-3 psid change). MCU was forced down due to a scheduled outage at 512-S (upstream) on April 16th. The maximum dP across SEC 7 was ~18.8 psid, and approximately 14.2k gallons of SE were produced while it was installed. Due to the timing of the outage and maximum dP in relation to the operating limit, a Management Decision was made to pre-emptively replace SEC 7.

SEC 7 was replaced and a restart with the eighth element installed occurred on April 30th, 2019. Operations were paused to sample the first two SEHT batches prior to transferring. MCU continued to operate until May 11th when MCU was forced down due to excess rain water in-leakage into the process cell. The process sumps were then emptied until May 14th when processing resumed. MCU then continued to operate until May 22nd when lack of freeboard downstream at DWPF due to a pump issue drove MCU into an outage. Feed was swapped from salt to DSS on May 22nd and continued for a 24-hr recirculation period ending on May 23rd. During

operations, the SEC 8 dP continuously increased at a faster rate than observed with SEC 7. At the time of swapping to DSS the dP reached a max of ~15.8 psid. The dP dipped slightly once feed was swapped (< 2 psid decrease) but continued to increase during the extended DSS recirculation. The final dP across SEC 8 reached ~17.1 psid during the DSS recirculation. Approximately 7.6k gal of SE were produced with SEC 8 installed. Based on the rate of increase in SEC dP, the estimated duration to replace the pump and create sufficient freeboard, and the proximity in schedule to the final MCU shut down date for SWPF tie-in work to begin, a Management Decision was made to not resume MCU operations after the shut down on May 23rd. SEC 8 remained installed in the plant until the end of operations.

While the SECs were the prime targets of analyses, several other types of samples were sent to SRNL. These were feed samples or samples related to the feeds, salt solution, strip solution, flushes of the feed systems, or filtration materials of the same.

This work follows a customer Task Technical Request (TTR)[i] , for which a Task Technical and Quality Assurance Plan (TTQAP)[ii] was written.

2.0 Experimental Procedure

The SECs and salt solution samples were highly radioactive and were manipulated in the radiological cells. Samples of the strip and scrub solution feeds were non-rad and handled outside of the cells, as well as the filtration materials related to those samples. Dilution of liquid samples was minimized. Small solid samples of the SECs were removed for analysis that exhibited radioactively low enough to be handled in a radiological hood.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60 (Design Check). This is Production Support class work. The work, analyses, calculations, and technical review satisfy the customer defined QA requirements. For SRNL documents, the extent and type of review using the SRNL Technical Report Design Checklist is outlined in WSRC-IM-2002-00011, Rev. 2.[iii] Records for this work are contained in electronic notebook ELN-A4571-00084-36.

3.0 Results and Discussion

A variety of MCU troubleshooting samples were sent to SRNL for analyses. These are broken up into separate groups for discussion: scrub feed, salt solution feed, strip solution, flush samples, filter media, coalescer housing, coalescer inlet piping, and biological analyses.

3.1 Scrub Feed Sample

A single sample of the scrub feed solution (MCU-18-222) was sent on June 13th, 2018. The purpose of this sample was to verify chemical content and examine the scrub feed for evidence of contamination. The sample was measured for pH and density and sent to Analytical Development (AD) and analyzed for free hydroxide and cation content (Inductively Coupled Plasma Emission Spectroscopy - ICPES). The relevant results are listed in Table 1.

Scrub feed is nominally 0.03 M NaOH, which should give a pH of 12.5, a sodium concentration of 690 mg/L and a free hydroxide of 0.03 M. While the pH and sodium measurements are as expected, the free hydroxide is lower than expected (see Table 1). It is likely that the free hydroxide has reacted with atmospheric CO_2. Samples from the vendor should be screened to ensure the hydroxide specification is being met. Regardless, there is no indication of the scrub feed containing materials harmful to the coalescer operations.

Table 1. Relevant Results from Analysis of the Scrub Feed Sample

Analyte	Expected Results	Results
Density	~1 g/ml	0.9935 g/mL
pH	12.5	12
Free OH	0.03 M	0.00856 M
B	N/A	<0.0392 mg/L
Na	690 mg/l	659 mg/L
K	N/A	7.27 mg/L
Si	N/A	1.67 mg/L

The 1σ analytical uncertainty for each result is 10%.

3.2 Salt Solution Feed Samples

A single sample of the salt feed solution (MCU-18-237) from the Salt Solution Feed Tank (SSFT) was sent on June 29th, 2018. The purpose of this sample was to examine the salt feed for evidence of contamination and to compare to the previous SSFT sample. The sample was measured for density and sent to AD and analyzed for ^{137}Cs, cation content (ICPES) and anion content (Ion Chromatography). The relevant results are listed in Table 2. The comparable SB9 and SB10 qualification sample results (from the Tank 21H samples) are reported for comparison.

On August 29th, 2018, a second sample (MCU-18-367) from the SSFT was delivered to SRNL. This sample was analyzed in the same manner as the previous salt feed sample. The relevant results are listed in Table 3. The comparable SB9 and SB10 qualification sample results (from the Tank 21H samples) are reported for comparison. The results of this sample are comparable to the previous salt feed sample and give no indication of containing materials harmful to the coalescer operations.

The salt feed solution is comparable to the compositions of the SB9 and SB10 samples, which is expected given that the salt feed is from Tank 49H, which is a blend of SB9 and SB10. There is no indication of the salt feed containing materials harmful to the coalescer operations.

Table 2. Relevant Results from Analysis of the Salt Feed Sample MCU-18-237

Analyte	MCU-18-237	SB9 Feed [iv]	SB10 Feed [v]
Density	1.264 g/mL (23.9 °C)	1.250 g/mL (24.9 °C)	1.254 g/mL (24.9 °C)
Cs-137	4.85E+08 dpm/mL	5.43E+08 dpm/mL	2.75E+08 dpm/mL
Al	6739 mg/L	6010 mg/L	6770 mg/L
Cr	77.4 mg/L	67.2 mg/L	52.0 mg/L
K	577 mg/L	558 mg/L	425 mg/L
Na	156000 mg/L	144000 mg/L	140000 mg/L
Si	25.1 mg/L	12.9 mg/L	21.7 mg/L
fluoride	<624 mg/L	<100 mg/L	<100 mg/L
formate	<624 mg/L	127 mg/L	258 mg/L
chloride	705 mg/L	696 mg/L	722 mg/L
nitrite	37500 mg/L	31700 mg/L	34600 mg/L
nitrate	120000 mg/L	106000 mg/L	92200 mg/L
phosphate	<624 mg/L	455 mg/L	315 mg/L
sulfate	6300 mg/L	5660 mg/L	4350 mg/L
oxalate	<624 mg/L	453 mg/L	420 mg/L
bromide	<624 mg/L	not calculated	not calculated

The 1σ analytical uncertainty for each result is 10%, except the ^{137}Cs which is 5%, and 3% for density.

Table 3. Relevant Results from Analysis of the Salt Feed Sample MCU-18-367

Analyte	MCU-18-367	SB9 Feed [iv]	SB10 Feed [v]
Density	1.251 g/mL (23.9 °C)	1.250 g/mL (24.9 °C)	1.254 g/mL (24.9 °C)
Cs-137	5.21E+08 dpm/mL	5.43E+08 dpm/mL	2.75E+08 dpm/mL
Al	6540 mg/L	6010 mg/L	6770 mg/L
Cr	73.1 mg/L	67.2 mg/L	52.0 mg/L
K	569 mg/L	558 mg/L	425 mg/L
Na	152000 mg/L	144000 mg/L	140000 mg/L
Si	21.8 mg/L	12.9 mg/L	21.7 mg/L
fluoride	<133 mg/L	<100 mg/L	<100 mg/L
formate	<133 mg/L	127 mg/L	258 mg/L
chloride	759 mg/L	696 mg/L	722 mg/L
nitrite	39200 mg/L	31700 mg/L	34600 mg/L
nitrate	127000 mg/L	106000 mg/L	92200 mg/L
phosphate	493 mg/L	455 mg/L	315 mg/L
sulfate	6420 mg/L	5660 mg/L	4350 mg/L
oxalate	480 mg/L	453 mg/L	420 mg/L
bromide	<133 mg/L	not calculated	not calculated

The 1σ analytical uncertainty for each result is 10%, except the ^{137}Cs which is 5%, and 3% for density.

3.3 Strip Solution Feed Samples

Strip feed solution samples from the StFT were delivered to SRNL for analysis. The first sample, MCU-18-223, was delivered on June 13th, 2018, along with the scrub feed sample (Section 3.1). These samples were obtained by draining the feed line strainers downstream of each tank. The purpose of this sample was to examine the strip feed for evidence of contamination. The sample was measured for density pH, and Free Hydroxide, and sent to AD to be analyzed for cation content by ICPES. The relevant results are listed in Table 4.

Table 4. Relevant Results of Analyses of MCU-18-223.

Analyte	Strip Feed
Density	0.9965 g/mL (22.8 °C)
pH	7
Free OH	<0.005 M
B	94.3 mg/L
Na	2.45 mg/L
K	<0.587 mg/L
Si	3.4 mg/L

The 1σ analytical uncertainty for each result is 10%, except for pH, which is 1 unit and the density which is typically 3%.

The strip feed shows no signs of impurities and is about as expected for 10 mM boric acid.

A second strip feed sample (no sample ID given) was delivered to SRNL on September 4th, 2018. This sample contained the material purged from the strip feed line through the strainer immediately prior to obtaining MCU-18-223. This material was analyzed in addition to MCU-18-223 to determine any variance between the material that had been sitting in the strip feed line, and the fresh material collected in the sample. The sample was sent to AD and analyzed for cation content by ICPES and anion content by IC. The relevant results are listed in Table 5.

The strip feed shows no signs of inorganic impurities and is about as expected for 10 mM boric acid. For biological analyses for a strip feed sample, see section 3.9

Table 5. Relevant Results of Analyses of the September 2018 Strip Feed Strainer Purge Sample.

Analyte	Strip Feed (mg/L)
B	96.3
K	<5
Na	10.7
Si	2.06
Fluoride	<100
Formate	<100
chloride	<100
nitrite	<100
nitrate	<100
phosphate	<100
sulfate	<100
oxalate	<100
bromide	<100

The 1σ analytical uncertainty for each result is 10%.

3.4 Organic Extractions of SSFT Samples

Aside from the typical analyses of cation and anions in the SSFT samples, SRNL analyzed the organic content of these samples to look for any evidence of organic species that might explain the reason for the high pressure drops.

Two SSFT samples, MCU-18-237 and MCU-18-367 were each extracted with hexane, and the hexane extracts analyzed by Semi-Volatile Organic Analysis (SVOA).

In both cases, no organic species, down to a level of 1 mg/L, were detected.

3.5 Flush Samples

The MCU StFT was successfully cleaned during the coalescer replacement outage. Operations personnel pumped the StFT down to a minimal heel through a sock on the end of the transfer hose directly to a tanker trailer supplied by the Portable Equipment Commodity Management Center (PECMC). The contents of the tanker were then disposed of at the Effluent Treatment Facility (ETF). This sock sample was designated as Sample 1 (ACTL [Aiken County Technology Laboratory] Sample). Augusta Industrial and Maintenance personnel then proceeded with cleaning efforts, which consisted of pressure washing the inside of the StFT with water. Augusta Industrial then utilized a Pumper Truck to vacuum the heel from the StFT following the pressure washing. The material from the Augusta Industrial Pumper Truck was then transferred to the

PECMC trailer via a hose with a sock on the end to collect solids. This sample was designated as Sample 2A (ACTL Sample). Liquid held up in the sock after removal from the discharge of the hose contained StFT material and potentially foreign material from the Pumper Truck. This liquid/material was collected and designated as Sample 2B (ACTL Sample). Rags, towels and wipes were used to swipe solids from the bottom of the StFT and the rags, towels and wipes were sent to ACTL for analysis designated as Sample 3 (ACTL Sample). The flush material from the PECMC tanker was again dispositioned at ETF.

Figure 1 shows the flush sock from between the Augusta Industrial Pumper Truck and the PECMC trailer. The filter sock was lightly contaminated with what looked to be specks and clumps of dirt like material, brown in color.

Figure 1. The 7/30/2018 Flush Filter Sock (Sample 2A)

Some of the more available material was removed with a spatula (Figure 2).

Figure 2. Flush Solids (Sample 2A) from the 7/30/2018 Flush Filter Sock

While containing some of the filter sock fibers, most of the material looked and felt like mud. Several attempts were made to dissolve the solids in a variety of solutions. Deionized water, 3M nitric acid, 3 M, 6 M, and 12 M NaOH were all used to dissolve the solids. In each case, ~0.1 grams of solid were placed in a poly bottle with 30 mL of each solution. The suspensions were shaken by hand and allowed to sit. Over a period of two weeks, there was no evidence of significant dissolution in any of the solutions. A sample of this same material was then sent for aqua regia digestion, and then analyzed by ICPES (Table 6).

Table 6. Relevant Results of Analyses of Sample 2A

Analyte	Results (μg/g)
Al	1950
B	<500
Ca	4870
Cr	1400
Cu	5700
Fe	186000
Mg	1190
Mn	1090
Na	1400
Ni	1300
P	1600
Si	3110
Zn	1960

The 1σ analytical uncertainty for each result is 10%.

The largest result was iron and, with the corresponding nickel and chromium results, is most likely due to steel fines. The aluminum, calcium, sodium and silicon are likely from clays or dirt. The copper, manganese and zinc are possibly from metal piping and plumbing. The phosphorus could be from phosphate. The source of the magnesium is uncertain. The ICPES results seem to indicate external contamination (dirt, rust, etc.) from outside of MCU.

To provide cleaning of the tank from any residues, the StFT was pressure washed twice. After the washing, the tank was wiped down with several rags, towels and wipes that were then shipped to SRNL (Sample 3). A small sample of the dark colored solids isolated from the rags was sent to AD for digestion and ICPES analysis (Table 7).

Table 7. Relevant Results of Analyses of Sample 3

Analyte	Results (μg/g)
Al	3330
B	2560
Ba	98
Ca	9990
Cr	16600
Cu	662
Fe	105000
K	1250
Mg	3410
Mn	1690
Mo	382
Na	9100
Ni	10700
P	2700
Si	5050
Sr	443
Ti	929
Zn	1450
Zr	231

The 1σ analytical uncertainty for each result is 10%.

As with the previous sample of solids, the same general set of analytes is present, although the ratios are somewhat different. New to this sample is a non-trivial amount of boron (likely from the boric acid strip solution), potassium, strontium, and titanium. The potassium could be from tramp material, but the strontium and titanium are very odd. Neither of these is a corrosion product, nor should be present in the tank. Either these elements have been present as insoluble solids for some time (which would have been noted by their soluble presence is previous StFT or SE samples), or they are a relatively new introduction into the StFT via the strip feed delivery trucks or the pumper truck.

The MCU strip feed and effluent systems were flushed in a two-phase approach in September 2018. Phase 1 involved flushing the strip feed system, beginning at the StFT up to the strip contactor bank, and collecting the material in a tote staged outside the contactor room. Solids were observed at the bottom of the tote. Samples of both the liquid (MCU-18-440) and solids (MCU-18-441) in the tote were obtained and transported to SRNL. The tote material was then dispositioned at ETF. Phase 2 involved flushing the SE system, downstream of the strip contactor bank up through the outlet nozzle of CSSX-L-1248. This nozzle typically serves as the inlet to the SEC housing; however, for this flushing sequence the housing was removed, and a “dummy” Hanford connector

was installed on the end of the pipe to allow the flush material to drain into the MCU cell rather than be transferred downstream to DWPF. A sock was placed on the outlet of the dummy Hanford for the duration of the flush and solids were collected. The filter sock was retrieved and transported to SRNL.

Finally, on December 5th, 2018, a sample (MCU-18-440) of the flushed solution ("phase 1") from the strip feed system was delivered to SRNL. A sample of this material (which should strongly resemble strip feed) was sent directly for ICPES analysis (Table 8). As a comparison, the results from the last reported SEHT samples (MCU-18-361/362/363) are included.[vi]

Table 8. Relevant Results of Analyses of MCU-18-440 and MCU-18-361/362/363.

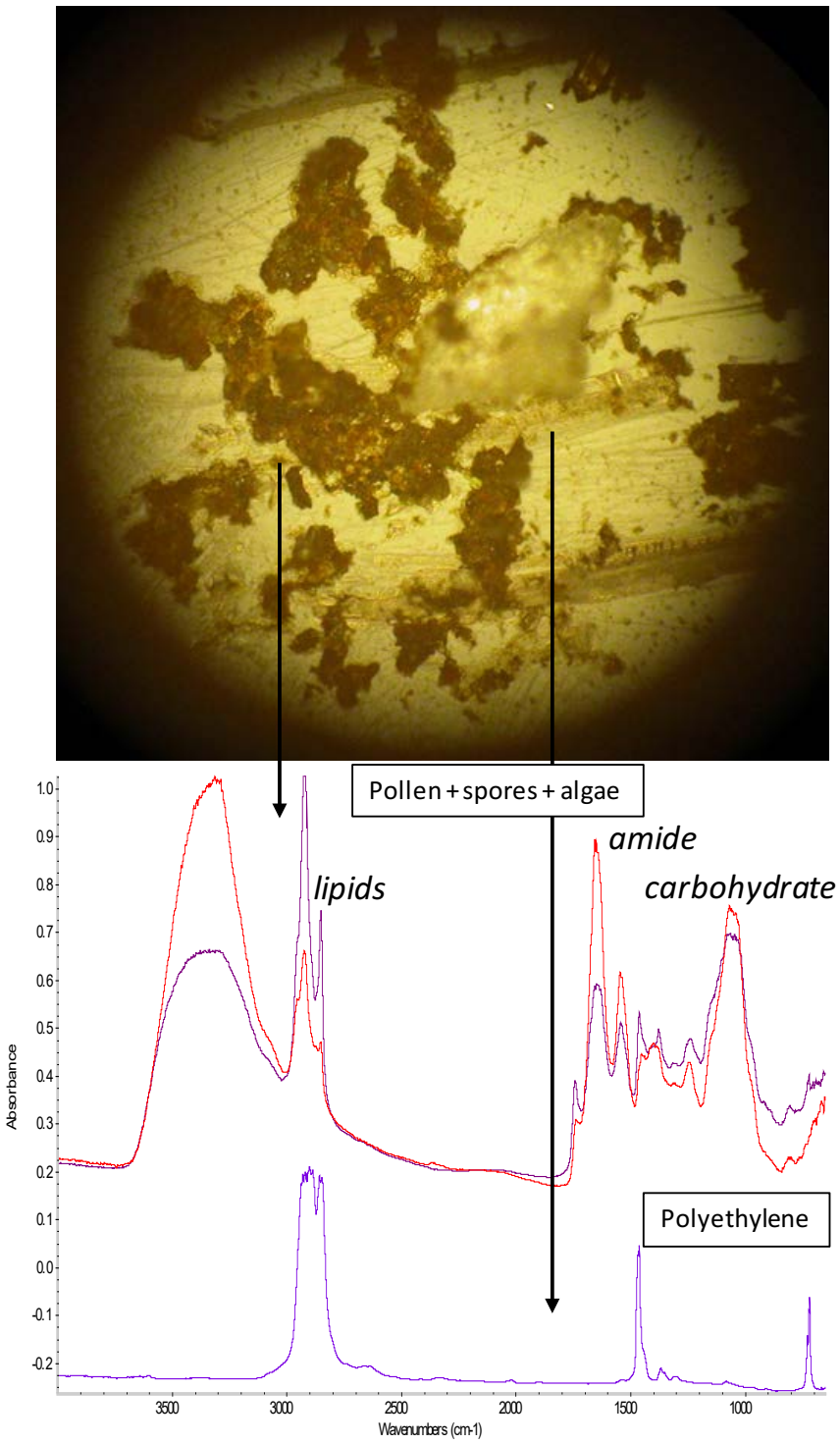
Analyte	Flush Results (mg/L)	SEHT Result (mg/L)
Al	1.68	<3.05
B	84.5	107
Ca	48.7	<0.683
K	<5.72	<21.7
Na	11.6	51.9
Si	1.97	<3.26
Sr	0.612	<0.08084

The 1σ analytical uncertainty for each result is 10%.

Since this material was fed directly from the StFT and did not go undergo any processing, this sample is more in-line with the boric acid feed specifications. While the boron value is lower than expected, this is not outside of historical precedent. The calcium and strontium in the flush sample are unexpected and likely from residual material from previous operations.

Solids from the phase 1 filtration were captured on a pig mat and were sent for Fourier-transform infrared (FTIR)/microscopy analysis (Figure 3). Other than the polyethylene filter sock material, there was abundant evidence of biological material, such as pollen, spores, and algae (as evidenced by the lipid, amide, and carbohydrate signatures).

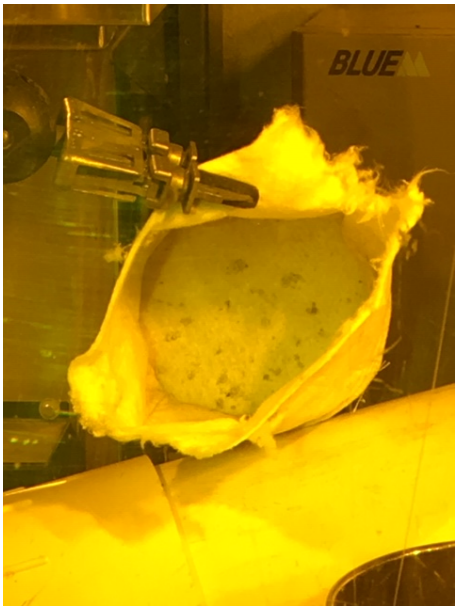
Figure 3. FTIR/Microscopy of Solids from Phase 1 Flushing (MCU-18-441)



A further flush ("phase 2") of the SE system through a filter sock was performed on September 25th, 2018. After the flushing, the filter sock and any solids on it was delivered to SRNL on

October 1st, 2018 and designated as MCU-18-418. The top half of the filter sock was roughly cut away, leaving the bottom dead-end half (Figure 4).

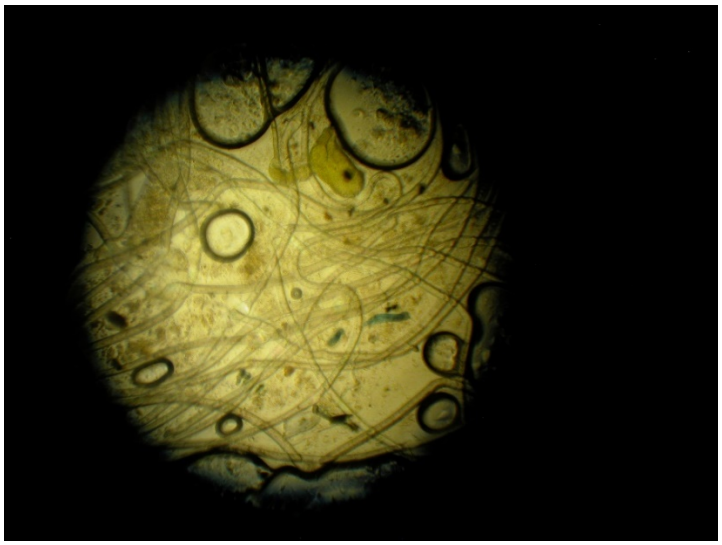
Figure 4. Bottom Half of the MCU-18-418 Filter Sock



Unexpectedly, there was little in the way of captured solids. Only small amounts of residual solids were scattered on the surface of the filter sock.

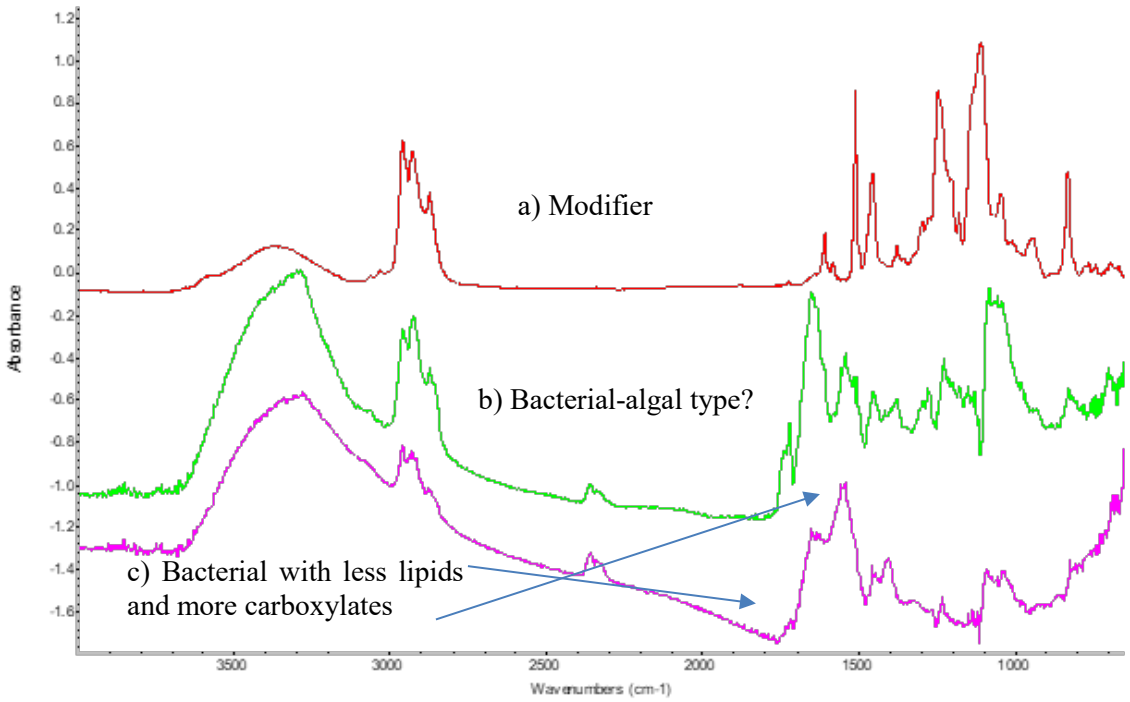
A section of the filter bottom was cut free and removed from the cells for several analyses. First, microscopy was used to study individual fibers (Figure 5).

Figure 5. Picture of the MCU-18-418 Filter Socks Fibers



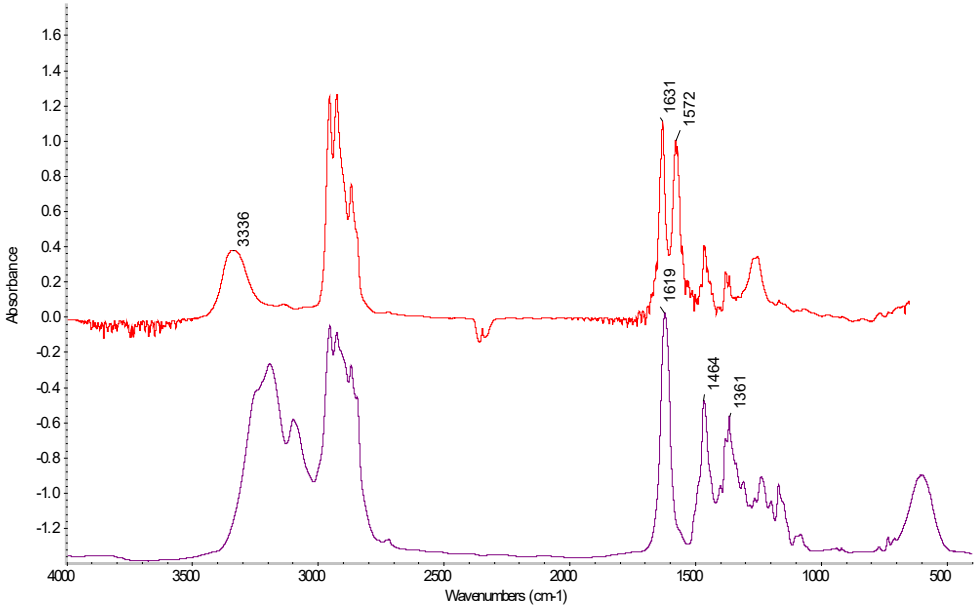
There was evidence of some material on parts of the fibers, and a FTIR of some of these fibers was performed (Figure 6).

Figure 6. FTIR of Several Spots on the MCU-18-418 Filter Sock



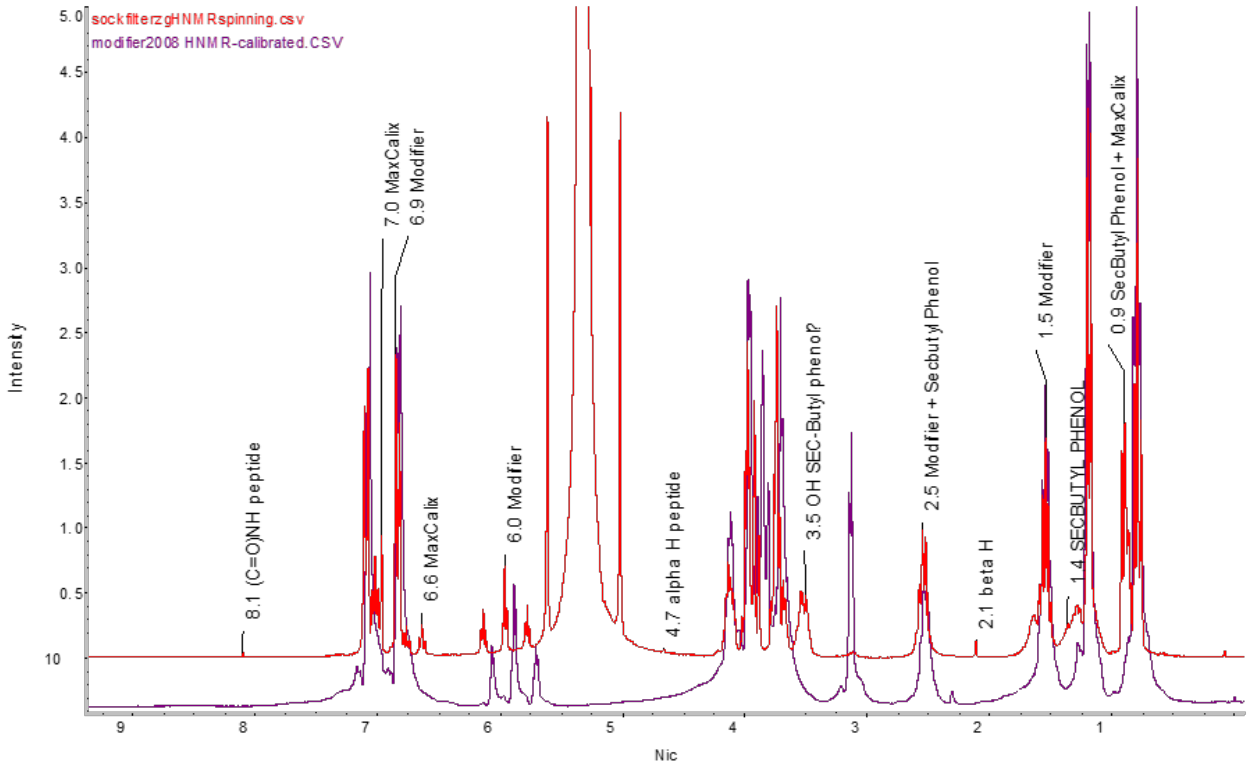
The largest component found in the filter was the Modifier (spectrum (a)). The second largest component found was characteristic of algal-like bacteria (with amide signals at 1550 and 1650 cm^{-1} and ester signals at 1740 cm^{-1} (spectrum (b)). Finally, minute amounts of a different bacteria, perhaps degraded bacteria with lesser lipid content but a relatively higher concentration of carboxylates was also detected (spectrum (c)). All the peaks found here are consistent with the spectrum of bacteria found in the literature.[vii] While it is conceivably possible that the amide signals are derived from suppressor, *N,N',N''*–tris(3,7-dimethyloctyl)guanidine (TiDG) degradation, peaks due to TiDG (purple spectrum in Figure 7) or its urea degradation product (red spectrum Figure 7) do not match the amide peaks observed in Figure 5. Rod-shaped bacteria were directly observed in MCU coalescer free liquid and MCU coalescer leach water associated with filamentous material in the samples.[xv]

Figure 7. TiDG and Its Degradation Product



A part of the removed filter sock was soaked with dichloromethane-D2 (CD_2Cl_2) to generate a sample suitable for Nuclear Magnetic Resonance (NMR) analysis. The NMR spectrum (in red) is shown in Figure 8, along with the spectrum of pure Modifier (in purple). The NMR spectrum identified most of the peaks to be related to solvent components (MaxCalix [1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6] or Modifier) or solvent component degradation products (*sec*-butyl phenol). The few remaining signals are interpreted to belong to biological entities such as peptides associated with microbial growth (at 8.1 and 4.7 wavenumbers).[viii]

Figure 8. ^{1}H NMR Analysis of the CD_2Cl_2 wash of the MCU-18-418 Filter Sock Solids



3.6 Filter Media Samples

The SEC is subject to fouling as the manner of construction allows it to act as a filter. When SEC2 arrived, it was agreed that SRNL should perform direct measurements on the fibers, in addition to leaching analytes from the fibers and analyzing the leaching solutions. In past efforts,[ix] SRNL leached slices of a coalescer in acids, water or caustic to liberate cations and anions as evidence of inorganic fouling. The current evolution followed the same protocols.

A ~1" wide ring of the coalescer material was cut from the perforated stainless-steel support tube. Once unrolled from the tube, each of the three coalescer rings was placed in a 1L poly bottle that contained one of three solutions: 3M nitric acid, deionized water, and 3M NaOH. The bottle was vigorously shaken for 1 minute and then allowed to sit undisturbed for up to ~1 week and supernate samples were periodically removed for cation and/or anion analysis. The relevant cation results (from ICPES) are reported in Table 9.

Table 9. ICPES Results for the SEC2 Leaching (mg/L)

Analyte	1 Day			7 Days		
	3M HNO3	DI Water	3M NaOH	3M HNO3	DI Water	3M NaOH
Al	24	<0.452	<9.04	24.9	<0.452	<9.04
B	4.78	3.2	<1.99	4.65	3.33	<1.99
Cr	1.47	<0.07	<1.39	1.57	<0.106	<2.13
Fe	14	<0.05	1.37	15.9	<0.05	1.78
Na	4.47	2.76	67400[1]	4.34	2.91	69900[2]
Ni	46.6	<0.254	<5.07	35.2	<0.495	<9.9
Si	10.1	<0.889	<17.8	11.1	0.653	<4.85
Zn	8.64	<0.05	2.25	8.38	<0.077	<1.55

The 1σ analytical uncertainty is 10% for all measurements in the above table.

In addition, the DI water leaching samples were analyzed via Ion Chromatography-Anions (IC-A). These results are reported in Table 10.

Table 10. IC-A Results for the SEC2 DI Water Leaching (mg/L)

Analyte	DI Water 1d	DI Water 7d
fluoride	<5	<5
formate	<5	<5
chloride	<5	<5
nitrite	<5	<5
nitrate	<5	<5
phosphate	<5	<5
sulfate	<5	<5
oxalate	<5	<5
bromide	<5	<5

The 1σ analytical uncertainty is 10% for all measurements in the above table.

Upon customer request a second set of leaching tests using 6.1 M NaOH, 9.6 M NaOH and 19 M NaOH were performed; using the same protocols as the previous leaching tests. The relevant cation results (from ICPES) are reported in Table 11.

From the leaching data, several conclusions can be drawn. First, the caustic leaching releases no or virtually no cations. Due to the high ionic strength of most of the caustic leaches, the only relevant cation seen is sodium, which is from the caustic. The water leaching also provided virtually no analytes, including anions, which precludes oxalate being present. Only the acid leaching provided a small amount of cations, which shows that only a trivial amount of inorganic

[1] The high Na values for the caustic leaching represent the added NaOH leaching solutions.

material is present, the most interesting of which is some evidence of stainless-steel fines (Cr, Fe, Ni).

Table 11. ICPES Results for the SEC2 Higher Caustic Leaching (mg/L)

Analyte	1 day			7 days		
	6.1M NaOH	9.6M NaOH	19M NaOH	6.1M NaOH	9.6M NaOH	19M NaOH
Al	<18.1	<45.2	<90.4	<18.1	<18.1	<18.1
B	<3.97	<9.94	<19.9	<3.97	<3.97	<3.97
Cr	<4.26	<10.6	<21.3	<4.26	<4.26	<4.26
Fe	<2.02	<5.04	<10.1	<11.8	<11.8	<11.8
Na [2]	148000	216000	426000	153000	242000	404000
Ni	<19.8	<49.5	<99	<19.8	<19.8	<19.8
Si	<9.7	<24.3	<48.5	<9.7	<9.7	<9.7
Zn	<3.09	<7.74	<15.5	<3.09	<3.09	<3.09

The 1σ analytical uncertainty is 10% for all measurements in the above table.

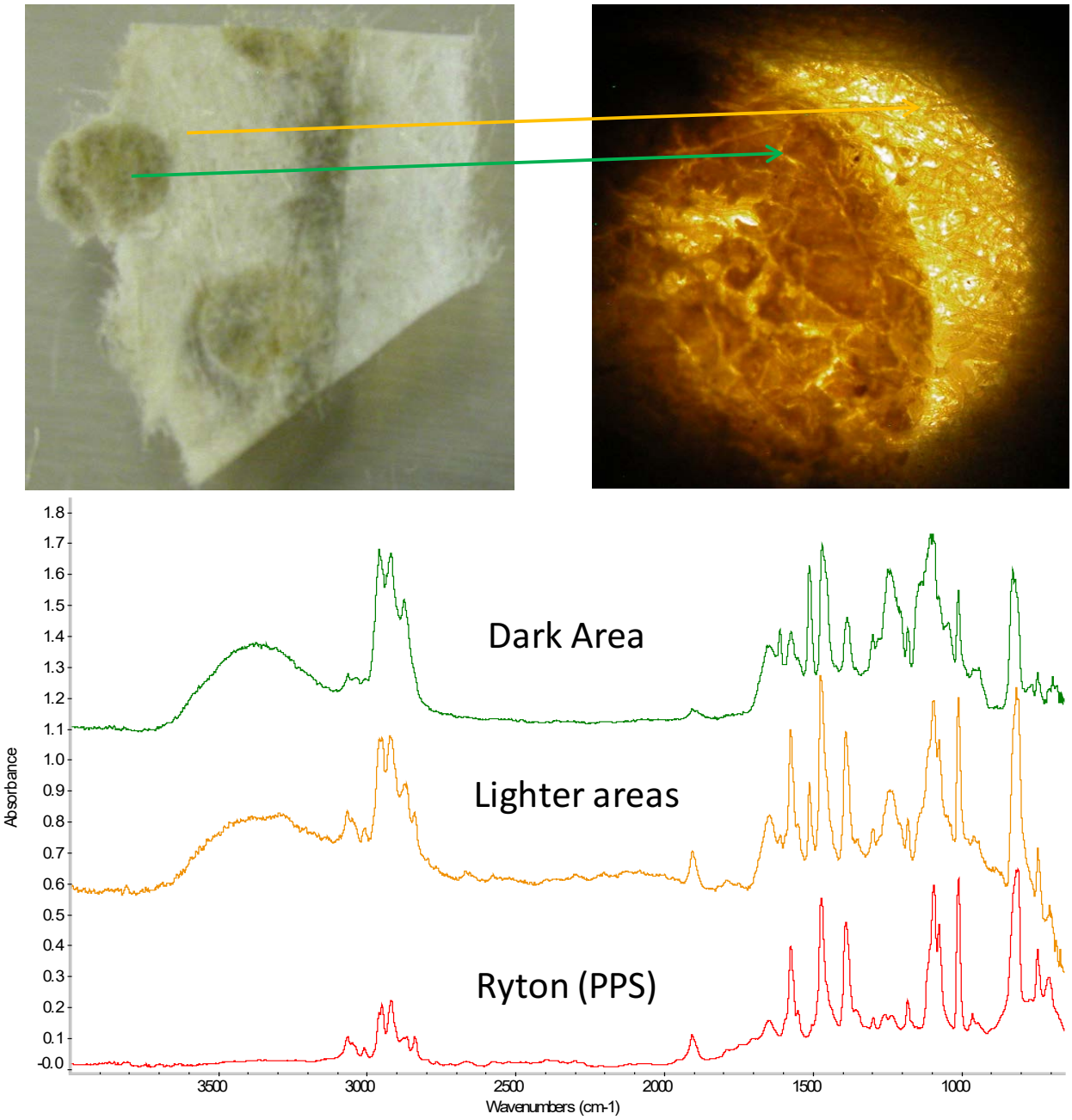
During operations, the coalescer is continually contacted with entrained organic solvent components and its degradation products. The hydrophobic coalescer material (polyphenylene sulfide (PPS)) preferentially contacts the solvent components over water. Previous analyses of coalescer slices have shown evidence of Modifier [3] on the coalescer fibers.[x] From SEC2, two attempts to analyze for organic materials have been made. First, a section of coalescer from the inner band (where any contaminants would be concentrated) was taken outside of the cells for microscopy and FTIR spectroscopy. An optical picture of this as-received piece and its corresponding infrared spectrum is shown in Figure 9. Figure 9 shows the transition region from the flow area disc to its neighboring area (see the two upper pictures).

The "dark" region which is the region directly over the holes in the perforated metal support structure is more opaque than its neighboring area. The "dark" region has a green tinge indicative of algal and/or cyanobacteria contamination (Figure 9). An infrared analysis of that area indicates the presence of amides (3300 and 1645 cm^{-1}) and sec-butyl phenol (1614, 1513, 1453, and the 1246 cm^{-1} peak which is more intense than the 1107 cm^{-1} peak in the Modifier). The amides (-C(O)-NH_2) could be from either suppressor (TiDG [4]) degradation or from bacteria.[xi] The sec-butyl phenol is from the degradation of the Modifier.[xii] The presence of Modifier is due to residual solvent which naturally adheres to the coalescer fibers. Much lower concentrations of these components were observed in the lighter colored areas neighboring the "dark" area.

For example, peak intensity of the amide (1645 cm^{-1}) and Modifier (1510 and 1240 cm^{-1}) is much lower relative to the peaks due to the PPS (1470 cm^{-1}) in the light area than in the plugged area.

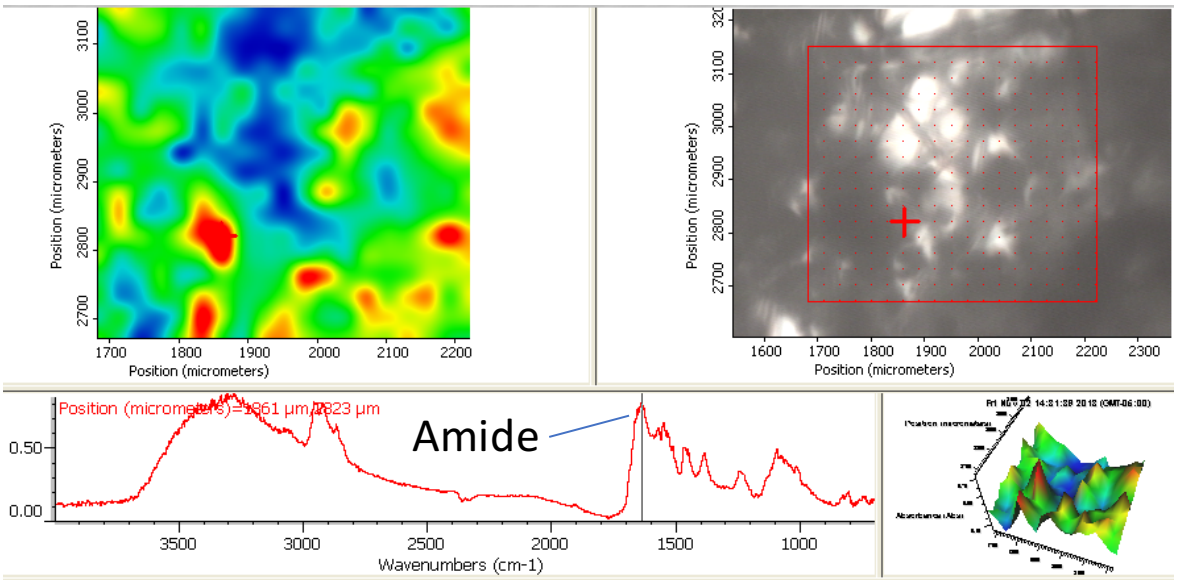
[2] The high Na values for the caustic leaching represent the added NaOH leaching solutions.
[3] Modifier is the common name for 1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-secbutylphenoxy)-2-propanol.
[4] TiDG is the common name for *N,N',N''*– tris(3,7-dimethyloctyl)guanidine.

Figure 9. Microscopy and FTIR of SEC2 Slice



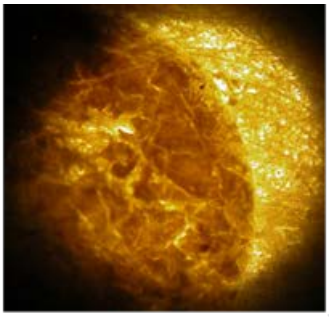
A closer examination of the amide signal was performed (Figure 10). The closeup FTIR seems to confirm that the amide is due to biological material and not a simple organic compound. The amide peak patterns observed in Figure 10 are typical of microbial colony metabolism.

Figure 10. Closeup Examination of the Amide Peak on SEC2



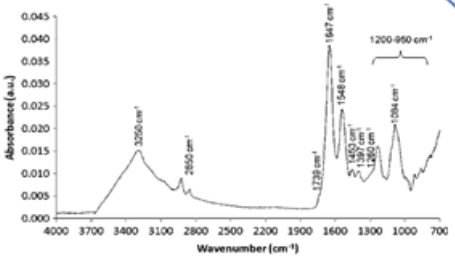
To further investigate the amide signal, two coalescer pieces were washed; one with dichloromethane, (CH_2Cl_2 – a polar solvent), the other with acetone (polar solvent). Both solvents were effective in removing the Modifier and sec-butyl phenol. The acetone wash was effective in removing the water. The coalescer pieces were much cleaner after both washes. An optical image and infrared analysis of the washed coalescer piece is shown in Figure 11. As can be seen in Figure 11, particulates remained in the "dark" and "light" areas of the coalescer. The chemical analyses indicate that the particulates are more closely related to microorganisms than to suppressor degradation products.

Figure 11. SEC2 Slice After Solvent Washing

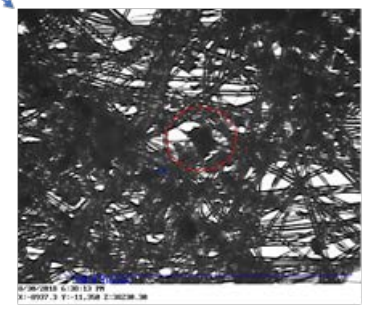


Picture of boundary between highly and lightly fouled sections. Even after solvent washing, fouling is evident. The dark (highly fouled) areas are the sections over the core perforations.

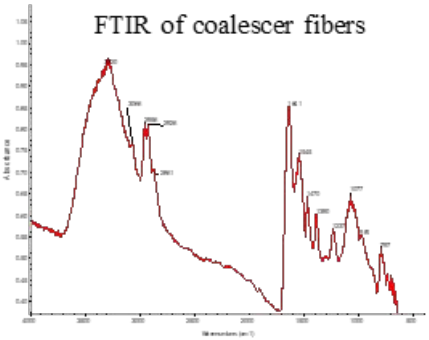
Literature FTIR of bacteria



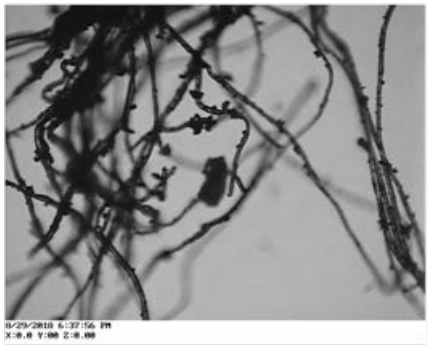
Under further magnification, particles can be noted – bacteria.



FTIR of coalescer fibers



Individual fibers are removed from the bulk and examined under magnification



Even after solvent washing, evidence of bacterial fouling remains.

Chemical analysis (FTIR) of the leachate only revealed Modifier, water, and sec-butyl phenol (and no TiDG degradation products or bacteria). Recall, that amines and urea are expected degradation products from TiDG degradation after contacting caustic aqueous solution. These products have different FTIR patterns (3337, 1614, 1580 cm^{-1}) compared to peptides (1645 and 1544 cm^{-1} or Amide I and II).

It was desired to corroborate the sec-butyl phenol signal. To do this, another leaching experiment was performed, except using CH_2Cl_2 as the solvent. After one day of leaching a sample was removed for High Performance Liquid Chromatography (HPLC) and SVOA analyses (Table 12). Comparing the individual component concentration ratios to the nominal solvent (right-most column), the Isopar-L™ is greatly depleted and the MaxCalix is relatively high compared to the Modifier.

Table 12. Results of the SEC2 CH_2Cl_2 Leaching (mg/L)

Analyte	SEC2 Leach Result	CSSX-NGS
MaxCalix [5]	9,130	47,800
Modifier	26,300	169,000
BOBCalixC6 [6]	<500	4,000
Sec-butyl phenol	180	0
Isopar-L™	862	610,000

The 1σ analytical uncertainty is 10% for all measurements in the above table, except for Isopar-L™ and sec-butyl phenol, which is 20%.

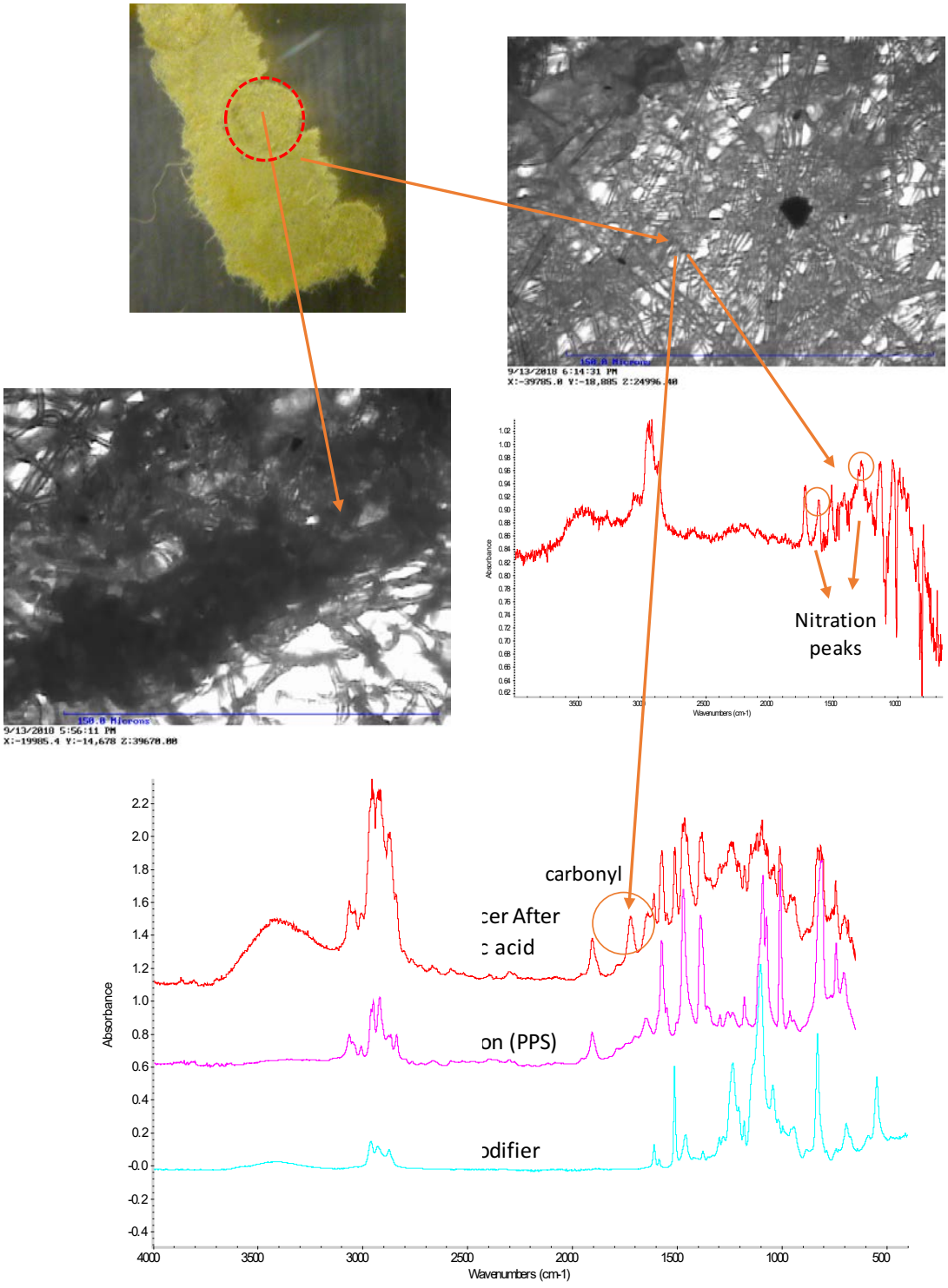
The sec-butyl phenol result of 180 mg/L would, if the sec-butyl phenol is evenly distributed across the coalescer, give a total of ~1.1 grams of sec-butyl phenol on the entire coalescer (180 mg/L per 1" slice × 0.3 L of leachate × 20 one-inch slices per coalescer).

To get a less obscured view of the as-received PPS fibers, two more sections from the coalescer were soaked in nitric acid – one in 3M nitric acid for 35 minutes (Figure 12), and one in 0.5M nitric acid for 24 hours (Figure 13).

Figure 12 shows that the nitric acid yellowed the PPS material, which by FTIR appears to correspond to some chemically altered Modifier (carbonyl peaks as evidence of oxidation). Otherwise, the bacterial/amide peaks as well as Modifier appear to be absent.

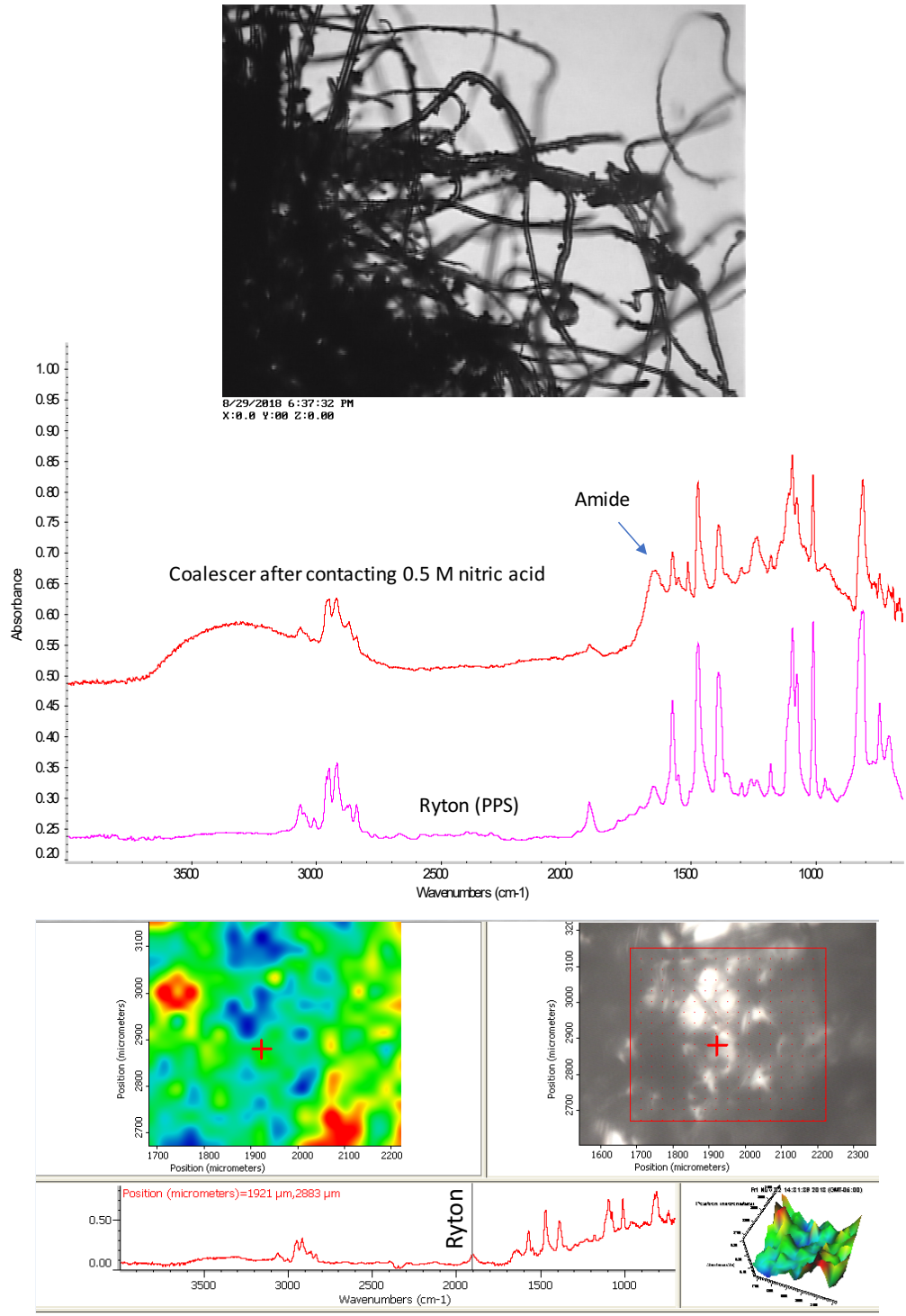
[5] MAXCalix is the common name for 1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arenebenzocrown-6
[6] BOBCalixC6 is the common name for Calix[4]arenebis(*tert*-octylbenzo-crown-6)

Figure 12. As-Received SEC2 After 35 Minutes Exposure to 3M Nitric Acid.



In comparison, the longer time exposure to 0.5M nitric acid did not appear to act on the PPS material, but did not completely strip away either the amide or Modifier traces (Figure 13).

Figure 13. As-Received SEC2 After 24 Hours Exposure to 0.5M Nitric Acid.

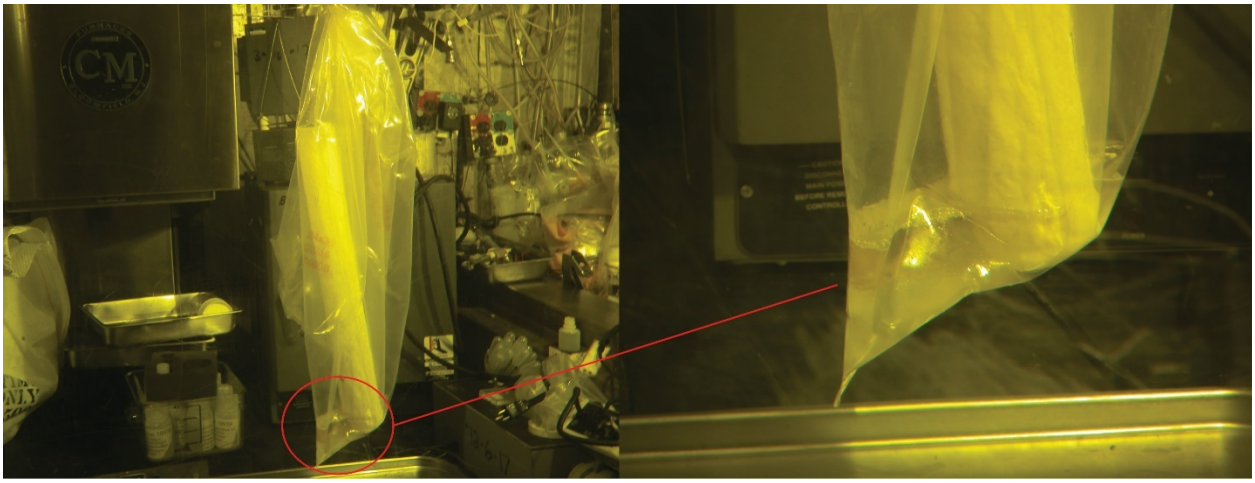


3.7 Coalescer Housing Liquid Samples

The SEC sits in a housing that can be isolated from the rest of the system. Liquid initially contained in the SEC media can drain out of it and still be retained in the housing when it is removed. Therefore, free liquids drained from the SEC or from the SEC housing are categorized together.

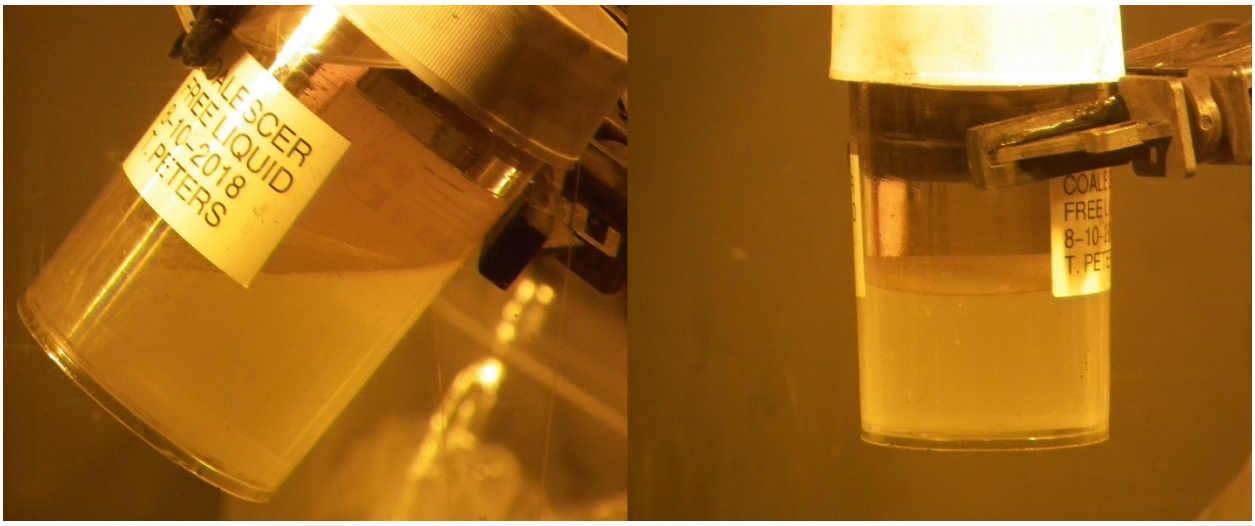
The first SEC to experience excessive pressure drops was not sent to SRNL for analysis since the media was back flushed and forward flushed which would have removed any material from the media. The second SEC was sent to SRNL and arrived on August 9th, 2018. The SEC arrived in a plastic bag (to prevent liquid from leaking into the larger shipping container) (Figure 14).

Figure 14. Free Liquids from the SEC2 Shipping Bag



There were two phases of liquid in the bag. The liquids were poured into a container for storage (Figure 15).

Figure 15. Two Phases of Liquids from the SEC2 Bag



The two distinct phases were more obvious in the container. The top organic phase was clear, while the lower aqueous phase was cloudy. A sample of the aqueous phase was analyzed via ICPES (Table 13).

Table 13. Selected ICPES Results for the SEC2 Aqueous Phase

Analyte	Result (mg/L)
Al	<9.99
B	79.3
Ca	<1.99
K	<59.7
Na	33.6
Si	<5.37

The 1σ analytical uncertainty for each result is 10%.

The aqueous phase is essentially diluted strip acid and is very similar to previous results (see Table 4).

On August 19th, 2018, the SEC housing containing SEC3 was opened at 299-H and the liquid in the SEC housing was sampled. This sample (MCU-18-356) was sent to SRNL for analysis and arrived on August 20th, 2018. The sample was just one phase; aqueous, and this material was sent for ICPES analysis (Table 14).

Table 14. Selected ICPES Results for MCU-18-356 (SEC3)

Analyte	Result (mg/L)
Al	<22
B	127
Ca	11.7
K	<107
Na	38.1
Si	<8.90

The 1σ analytical uncertainty for each result is 10%.

As with the SEC2 aqueous phase, this material closely resembles strip acid, although slightly high in the boron content.

From the SEC2 free liquids, a sample of the organic phase was analyzed via SVOA and HPLC (Table 15).

Table 15. Selected SVOA and HPLC Results for the SEC2 Organic Phase

Analyte	Result (mg/L)	Nominal Solvent Result (mg/L)
Modifier (HPLC)	227000	169000
MaxCalix (HPLC)	62500	46000
BoBCalixC6 (HPLC)	2040	NA
Impurities (SVOA)	<1	NA

The 1σ analytical uncertainty for the HPLC results are 10%, and 20% for SVOA.
"NA" indicates the analyte was not measured or has no nominal value.

The modifier and MaxCalix results are ~135% of nominal solvent concentration, indicating the solvent has suffered a corresponding loss of Isopar-L ™. The SVOA did not find any evidence of contaminants to a level of <1 mg/L.

SEC3 and SEC4 were shipped to SRNL and received on December 3rd, 2018. Both SECs and their shipping bags had two phases of free liquids in them, as with SEC2. It was noted that the PVC shipping tube had free liquid in the bottom of the tube, probably from a perforation in the plastic bags from SEC3. It was noted that the SEC3 bag contained very little free liquid and the SEC4 bag had some free liquid, but not as much as SEC2. The liquid was pumped from the PVC shipping tube into a polyethylene bottle and credited as SEC3 liquid since the SEC3 bag was free of liquid.[xiii,xiv]

It was noted that the organic phases were darker in color. The liquids (both phases) from each coalescer were transferred to a bottle for storage (Figure 16).

Figure 16. Free Liquids from SEC 3 and 4.

SEC3 had a greater amount of the organic phase and appeared to be slightly darker (likely from contact with the plastic shipping tube). Samples of each phase were removed for SVOA and HPLC analyses (Table 16).

Table 16. Selected SVOA and HPLC Results for the SEC3 and 4 Organic Phases

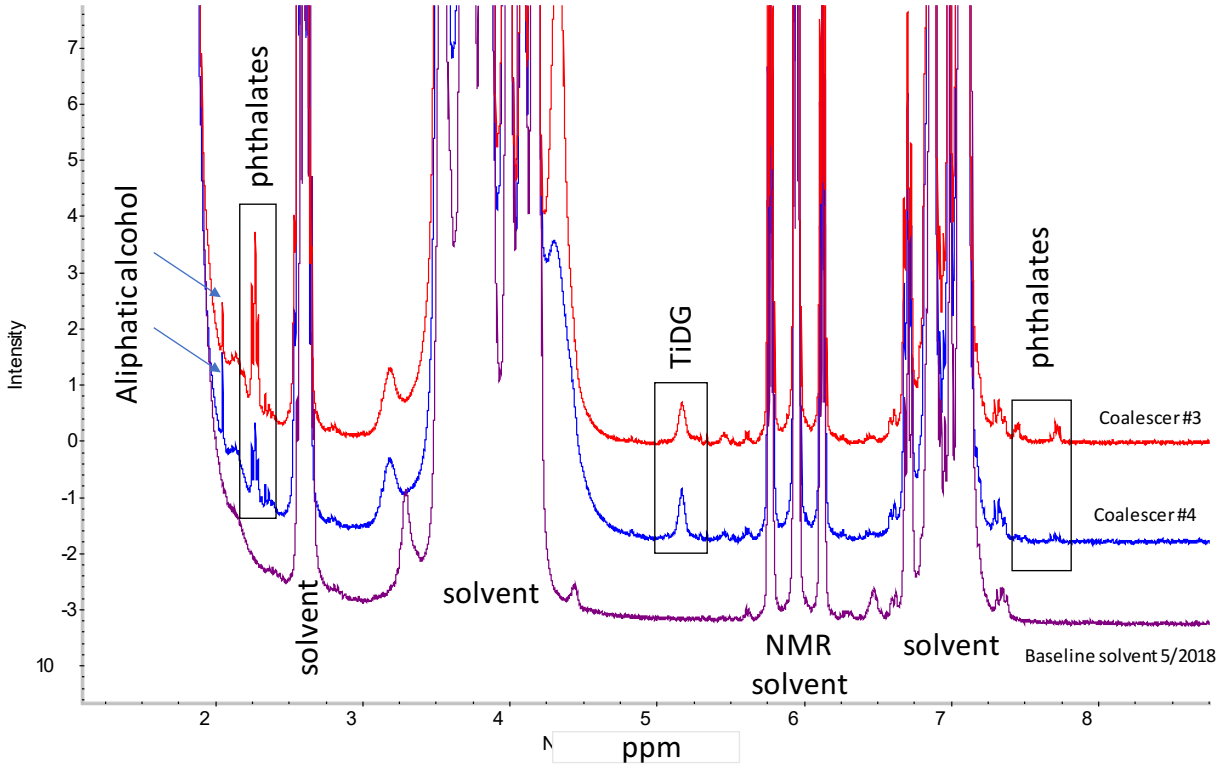
Analyte	SEC 3 Result (mg/L)	SEC 4 Result (mg/L)	Nominal Solvent Result (mg/L)
Modifier (HPLC)	176000	167000	169000
MaxCalix (HPLC)	52000	51200	46000
BoBCalixC6 (HPLC)	1490	1450	NA
Impurities (SVOA)	<1	<1	NA

The 1σ analytical uncertainty for the HPLC results are 10%, and 20% for SVOA.
"NA" indicates the analyte was not measured or has no nominal value.

As with the SEC2 organic phase, the analyses showed that the organic liquids from both SECs resembled solvent that had undergone some Isopar-L ™ losses. There was no indication of foreign organic species.

Further analyses of the two solutions was done using ^{1}H NMR (Figure 17) and FTIR (Figure 18).

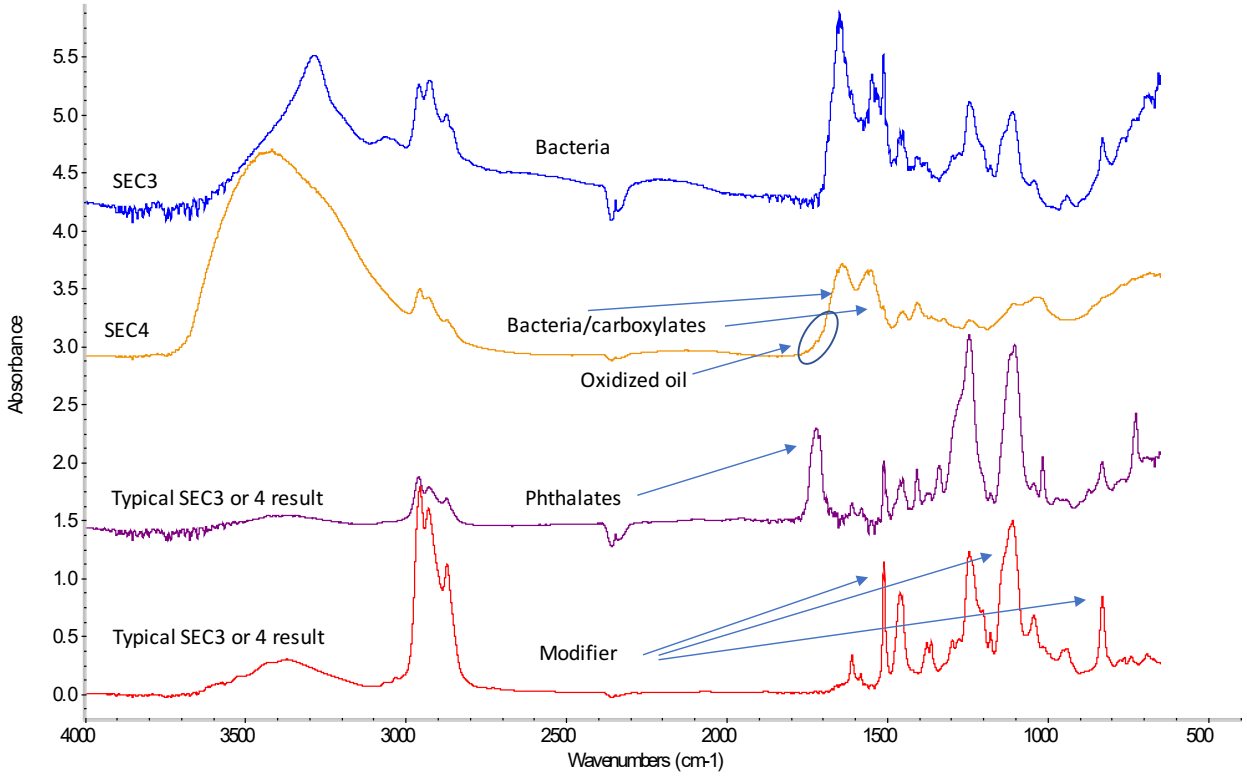
Figure 17. ^{1}H NMR of SEC3 and 4 Free Organic Liquids



The NMR of both solutions indicated that the sample matrix was the MCU solvent (broad regions at 2.75, 4, and 7 ppm), and the NMR lock solvent (6 ppm). Both SEC3 and SEC4 samples exhibited small peaks at ~7.75 and ~2.5 ppm attributed to phthalates which is attributed to contact with the plastic shipping bag (plasticizers).

The sample from SEC3 is roughly estimated to have a phthalate concentration of 480 ppm, while this cannot be estimated in the SEC4 sample due to the very weak signal. The aliphatic alcohol concentration is estimated to be 260 ppm in the SEC4 sample, and 670 ppm in the SEC3 sample. SRNL attributes both signals to the free liquids exposure to the shipping bag used in the transfer of each SEC. The color in each sample is likely due to some small impurity which is masked by the solvent peaks. These peaks and their concentrations are not found in the monthly SHT samples which are not exposed to the plastic shipping bag material.

Figure 18. FTIR of SEC3 and 4 Free Organic Liquids



The FTIR (Figure 18) corroborates the NMR assessment of the presence of Modifier and phthalates in each sample. Both SEC3 and 4 samples showed the presence of bacterial signals, although the SEC4 sample showed additional complexity possibly indicating a more diverse biological presence. In the SEC4 sample there was also some weak evidence of some sort of oxidized oil (shoulder on one of the carbonyl peaks at ~1600 cm^{-1}).

3.8 Coalescer Inlet Piping Sample L-1248

A liquid sample was taken from the piping between the SEC pumps and the SEC housing. This was taken after SEC3 was removed and before SEC4 was installed. This sample (MCU-18-417) was delivered to SRNL on September 25th, 2018.

The sample was initially analyzed by ICPES and IC-A (Table 17).

Table 17. Selected ICPES Results for MCU-18-417

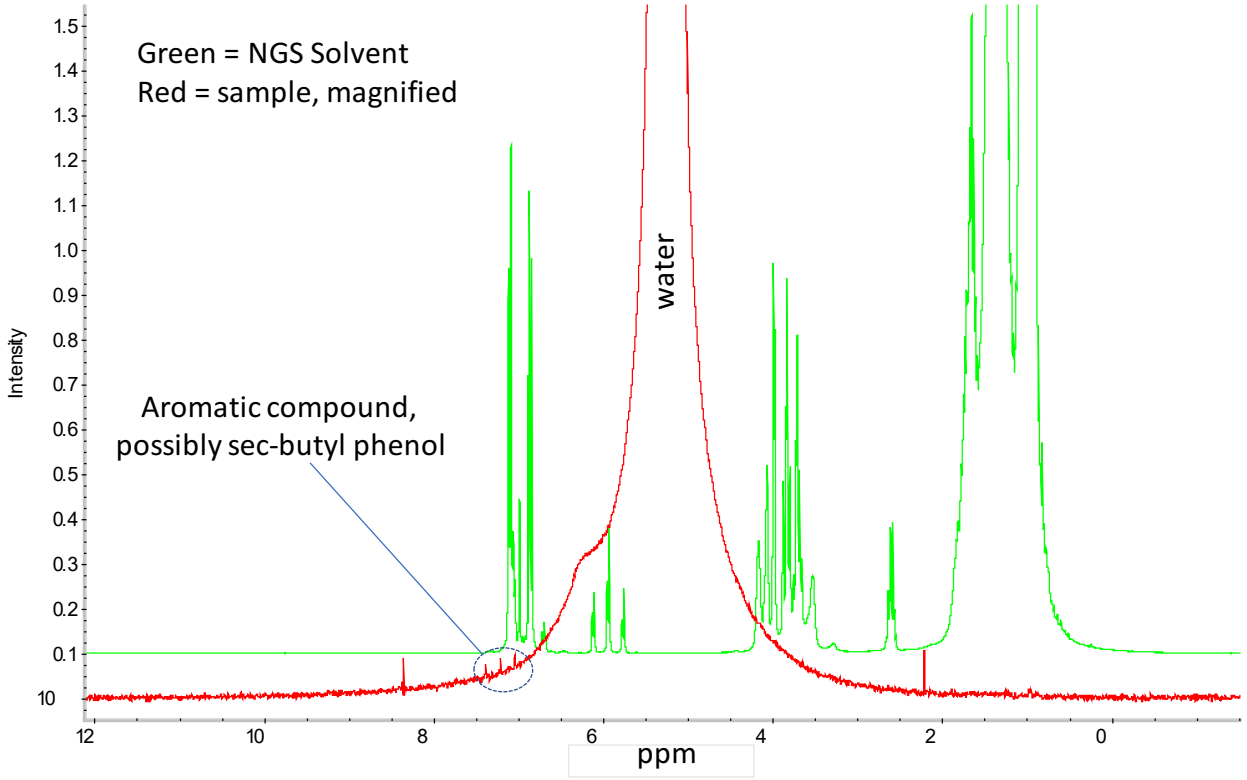
Analyte	Result (mg/L)
Nitrate	40800
Al	8.07
B	1.03
Ca	9.18
Fe	78.9
Cr	0.887
Na	3.53
Ni	1.80
Si	4.24

The 1σ analytical uncertainty for each result is 10%.

Given that nitric acid is no longer used with the current solvent formulation, the nitrate content (0.66 M) must be from the 3M nitric acid used in contactor cleaning which had been performed earlier in the month. The nitrate concentration is indicative that the nitric acid, as of the time of sampling, had not yet been adequately flushed and pumped out.

A further analysis of this sample via ^{1}H-NMR was performed (Figure 19). The NMR showed a very weak set of signals most likely corresponding to sec-butyl phenol, although slightly shifted due to the water solvation. An FTIR analysis was performed, but no organic signals could be clearly discerned.

Figure 19. ^{1}H-NMR of MCU-18-417



3.9 Biological Assays

SRNL received three samples from the MCU StFT on August 1st, 2018. Sample 1 was a plastic bag labeled "Strip Feed Solids MCU" and containing a filter bag and wipes. Sample 2 was a can labeled "Pumper Tank Contents" containing a filter bag and liquid on the bottom of the can. The filter bag material was labeled Sample 2A, and the liquid was labeled sample 2B. Sample 3 was a can labeled "Strip Feed Bottom Contents". Table 18 shows the results of the bacteria analysis. All the samples showed high microbial density.

Table 18. Microbial Density Results

Microbiological Density Results (cells per gram or mL)				
Bacteria Type	Sample 1 Strip Feed Solids	Sample 2A Pumper Tank Contents – Filter Bag	Sample 2B Pumper Tank Contents – Liquid	Sample 3 Strip Feed Bottom Contents
Total	>1,000,000	>1,000,000	>100,000	>1,000,000
Iron-related	>1,000,000	>1,000,000	>100,000	>1,000,000
Anaerobic	>1,000,000	>1,000,000	>100,000	>1,000,000
Acid Producing	>1,000,000	>1,000,000	>100,000	>1,000,000
Sulfate Reducing	10-100	1000-10,000	100-1000	10,000 -100,000

SRNL received another sample from MCU on August 10th, 2018. The sample was collected from the strip feed delivery tanker. Microbial analysis was performed on this sample and indicated a cell density of $10^6 – 10^8$ cells per Liter. The acid producing bacteria are a concern at these densities (>1,000,000 cells/mL) as they can cause pitting in system surfaces including stainless steel. The pitting in turn cause release of solids and provides a suitable environment for biofilm production.

A third set of samples were received on August 31st, 2018. These samples were from an SEC that was soaked in water to attempt to detach any microbes present. The cell densities for these samples were <1,000 bacteria/mL. While these bacterial densities are lower than the previous samples, the low bacterial density may be the result of the bacteria not readily detaching from the SEC when soaked in water. Based on these results, there is evidence of a microbial fouling problem in the MCU. More detailed discussion can be found in the report describing the analysis.[xv]

Additional samples of dilute boric acid from the MCU process and a filter containing dislodged material were collected and analyzed after a one-month incubation period. Table 19 shows the results.[xvi] The bacterial counts are high and indicate that biofouling could be occurring in the MCU.

Table 19. Bacterial Growth Studies After One Month Incubation at 20˚C

Microbial Test	Dilute boric acid (cells/ml)	Exudate on filter (cells/ml)
Low nutrient bacteria	$>10^5$	$>10^6$
Iron oxidizing bacteria	$<10^0$	10^3-10^4
Anaerobic bacteria	10^3-10^4	$>10^6$
Organic acid producing bacteria	10^4-10^5	$>10^6$
Sulfate reducing bacteria	$<10^0$	$<10^0$

Three MCU samples were sent from SRNL to Microbial Insights for molecular analysis. The objective of this analysis was to profile and identify the dominant members of the microbial community in these samples. These samples included an MCU Strip Feed Sample, an MCU Strip

Feed Sample grown in low nutrient conditions, and an MCU Strip Feed Sample grown in anaerobic conditions. The analysis looked at bacteria and fungi. The most dominant bacterial species were *Enterobacter, Comamonas, Erwinia,* and *Pseudomonas*. The most dominant fungi were *Cladosporium, Rhizophagus, Trametes, Malassezia,* and *Kodamaea*. These results indicate that the bacterial and fungi populations are versatile, robust, and are likely to survive under a variety of processing conditions within the MCU. More detailed analysis can be found in the technical report.[xvii]

4.0 Conclusions

Operation of the SEC causes solvent droplets to be captured by the coalescer fibers, grow (i.e., coalesce) by combining with other captured droplets, and detach from the fibers when the droplets achieve a larger size. These larger droplets rise more rapidly within the aqueous solution and separate in a downstream decanter. A pressure differential forces the SE through the coalescer media.

When compounds such as insoluble particles or organic droplets accumulate in the open void of the coalescer fibers, the dP increases. However, if the dP across a coalescer is too high, this can cause the coalescer to suffer physical failure (e.g., delamination of the fibers from the metal support structure) and lower efficiency for retaining / coalescing the solvent. Therefore, MCU maintains an upper limit to the dP across the coalescer, which is currently 28 psid. Coverage of the coalescer fibers with a biofilm as observed here would also limit efficiency.

There are four plausible sources of the increased pressure differential observed:

- Mechanical restrictions (blockages),
- Organic fouling (from organic solvent components or degradation products),
- Inorganic fouling (e.g., aluminosilicate or oxalate solids), or
- Biofouling (from bacteriological entities or biofilms).

SRNL is not chartered to test for mechanical issues. For the remaining three sources, all the data in this document were considered.

4.1 Organic Fouling

The MCU solvent is nominally composed of six components, five of which could have multiple degradation pathways – the sixth, Isopar-L ™ is an alkane and is unlikely to degrade to a noticeable degree. Organic degradation products that interact with the PPS fibers of a SEC could add a drag to the flow across the SEC and increase the pressure drop. Historically, the only clearly identified degradation product from the solvent has been low levels of sec-butyl phenol,[xii] which has not previously been identified as an issue with coalescer operations. Nevertheless, to search and identify any organic species that should not be present, multiple analyses at different points in the MCU system were examined. None of the samples identified an organic (not including biological)

compound present other than small amounts of sec-butyl phenol. In the absence of any indication of undesired organic compounds, SRNL feels that organic compounds are not the cause of the failures in the SEC.

4.2 Inorganic Fouling

Inorganic fouling has been the historical cause for difficulties in MCU operations.[ix, xviii] Aluminosilicate and oxalate salts have precipitated from solution and affected MCU operations in the past. However, SRNL has found no evidence of any atypical inorganic constituents in the feed solutions, the output solutions (DSSHT, SEHT), or from the fouled SEC2 itself currently. There is no evidence of any unusual constituents or concentrations of inorganic species from these samples. SRNL feels that inorganic fouling is not the reason for the SEC fouling.

4.3 Biofouling

The analysis of multiple samples from the MCU showed high concentrations of bacteria that are likely causing biofouling within the process, especially some of the strip feed samples. Samples of the water that a coalescer sample was soaked in showed much lower bacterial concentrations, indicating that the bacteria are not readily removed by a water soak. Molecular analysis of samples for bacteria and fungi showed diverse populations that would be versatile and robust. The biofouling could exist under a variety of MCU operating conditions and release inorganic (corroded materials) and organic (cell debris) solids that cause additional problems.

Leached samples of SEC2 suggest that nitric acid could be used to clear the biofouling from a coalescer. Relatively concentrated 3M nitric acid was shown to clear the biological amide signals from a sample of a coalescer when contacted for at least 30 minutes. However, this strength of acid also acted on the PPS fibers to some degree. More dilute 0.5M nitric acid did not clear the amide signals from a coalescer sample after 24 hours but did not act on the PPS material. Therefore, some range of acid between 0.5 and 3M at sufficient contact duration might be able to clear the amide signals, but without substantively degrading the PPS material.

Consideration should be given to locating a suitable biocide that is compatible with MCU operations. While tank cleaning has temporarily worked to reduce the biological loading, the effects do not last. Consideration should also be given to filtering the feed solution from the tanker trucks.

5.0 Recommendations

Given the issues with the SE coalescer, SRNL recommends that any SEC should be as long as possible to maximize the surface area.[xix]

To prevent the influx of organic materials which may promote biofouling (spores, pollen, insects, etc.), SRNL also recommends installing air filters and similar devices on air intakes to the various chemical feed tanks. Additionally, the strip feed should be filtered from the vendor trucks to eliminate possible sources of organic contamination.

6.0 References

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