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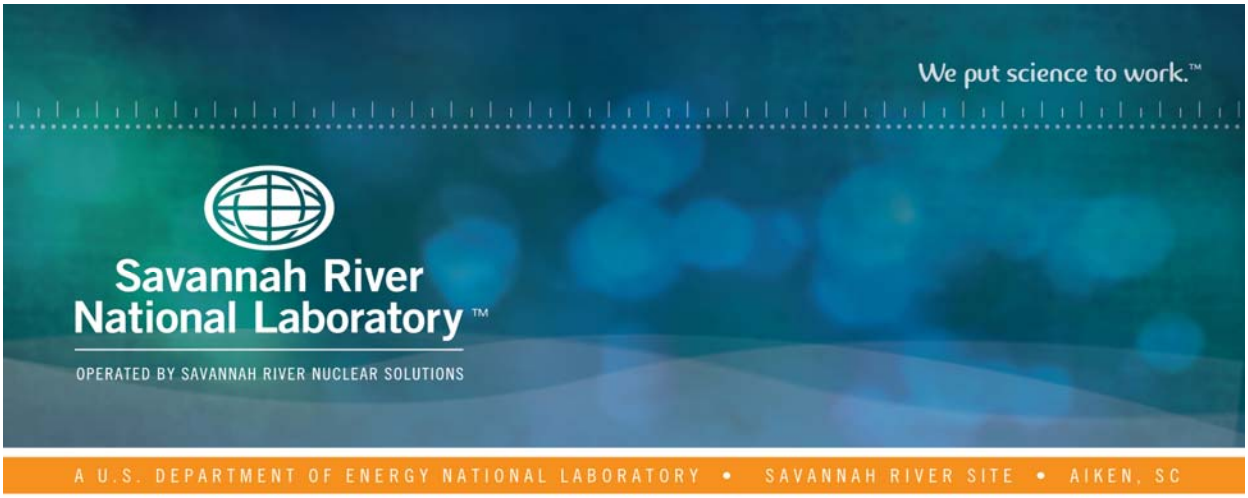

Solvent Hold Tank Sample Results for MCU-18-123-125 (April 2018), MCU-18-135-137 (May 2018), and MCU-18-192-197 (June 2018): Quarterly Report

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D. H. Jones

October 2018

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Savannah River National Laboratory™
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EXECUTIVE SUMMARY

A trend summary of three Solvent Hold Tank (SHT) monthly samples MCU-18-123-125, MCU-18-135-137, and MCU-18-192-197 are reported. Most of the conclusions are based on the June SHT sample (MCU-18-192-197). Analyses of the June SHT sample (MCU-18-192-197) indicated that the Modifier (CS-7SB) and the extractant (MaxCalix) concentrations were at the nominal recommended concentrations (169,000 mg/L and 46,400 mg/L respectively). The suppressor (*N,N',N"*–tris(3,7-dimethyloctyl)guanidine or TiDG) concentration has decreased since the April 2018 measurement (Modular Caustic-Side Solvent Extraction Unit or MCU resumed processing radioactive salt solution from May through August 2018) to 1,116 mg/L, well above the minimum recommended concentration (479 mg/L), but below the nominal concentration. The apparent "downwards" trends observed in the TiDG, MaxCalix, Modifier, and gamma measurement are consistent with the start up (and/or proficiency runs) of MCU on May 22, 2018 since these new concentrations are consistent with the concentrations observed when MCU was fully operational in 2016.

This analysis confirms the Isopar™ L addition to the solvent on April 28 and May 14, 2018. This analysis also indicates the solvent did not require further additions. Based on the current monthly sample, the concentrations of TiDG, Isopar™ L, MaxCalix, and Modifier are sufficient for continuing operation. Periodic characterization and trimming additions to the solvent are recommended.

The Semi-Volatile Organic Analysis (SVOA) did not detect any impurities. However, the Fourier-Transformed Hydrogen Nuclear Magnetic Resonance (FT-HNMR) analysis detected a high concentration of linear alkane phthalate (for example dibutyl phthalate), a typical plasticizer used in polyvinyl chloride polymers, in the solvent. The impurity concentration was highest in the April sample (~0.96 g/L) and below the detection limit in the June sample. The source of this impurity and how it ended up in the solvent is not known at this time. Another impurity observed in the samples was mercury. Based on the June SHT sample, up to 30 ± 6 micrograms of mercury per mL of solvent was detected (the average of the Cold Vapor Atomic Absorption [CVAA] and X-Ray Fluorescence [XRF] methods). The higher mercury concentration in the solvent (as determined in the last three monthly samples) is possibly due to the higher mercury concentration in Salt Batches 9 and 10 (SB 10 was recently added to Tank 49H in March 2017) or sampling of previously undisturbed areas of high mercury concentration in Tank 49H.

The gamma concentration (~2.42E5 dpm/mL) measured in the June SHT samples was consistent with previous values observed when MCU was fully operational (for example, between December 2016 and January 2017) and it was lower than the April SHT measurement. The "dip" in the gamma counts for the May 2018 SHT sample was due to Isopar™ L addition to MCU during April and May 2018.

The laboratory will continue to monitor the quality of the solvent for any new impurities or degradation of the solvent components.

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LIST OF ABBREVIATIONS

BOBCalixC6	Calix[4]arene-bis(*tert*-octylbenzo-crown-6)
CSSX	Caustic-Side Solvent Extraction
CVAA	Cold Vapor Atomic Absorption Spectrometry
FT-HNMR	Fourier Transform Hydrogen Nuclear Magnetic Resonance
HNMR	Hydrogen Nuclear Magnetic Resonance
HPLC	High Performance Liquid Chromatography
MCU	Modular Caustic-Side Solvent Extraction Unit
MaxCalix	1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6
NGS	Next Generation Solvent
PPS	Polyphenylene Sulfide
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
TiDG	*N,N',N"*–tris(3,7-dimethyloctyl)guanidine
TOA	Trioctylamine
XRF	X-Ray Fluorescence

1.0 Introduction

In late FY13, MCU implemented the Next Generation Solvent (NGS) flow sheet. Facility personnel added a non-radioactive, NGS "cocktail" containing the new extractant (MaxCalix) and a new suppressor (TiDG) to the SHT heel to implement the NGS flow sheet. The resulting "blend" solvent ("NGS blend solvent") is essentially NGS with residual amounts of calix[4]arene-bis(tert-octylbenzo-crown-6) (BOBCalixC6) and trioctylamine (TOA). For process monitoring, SHT samples are sent to Savannah River National Lab (SRNL) to examine solvent composition changes over time.[1] With the exception of Isopar™ L which is regularly added to the SHT due to its high vapor pressure, this report shows the cumulative chemical composition data, including impurities like mercury, of three SHT samples: MCU-18-123-125, MCU-18-135-137, and MCU-18-192-197 (6 P-nut vials). A summary report for each of the SHT samples was issued earlier.[2,3,4]

These samples are intended to verify that the solvent is within the specified composition range. A baseline "scratch" solvent – a scratch solvent is a preparation of all 6 solvent components of the composition that approximates the blend of cocktail[5] and heel solvent – was prepared in the lab (July 2017) and used for comparison and evaluation. The results from the analyses are presented in this document.

2.0 Experimental Procedure

2.1 Experimental Procedure

Table 2-1 lists a summary of relevant and recent trims to the MCU solvent as well as the arrival date of the samples currently being studied. On May 14, 2018, an Isopar™ L addition was made to MCU.[6]

Table 2-1 Log of recent trims to MCU solvent and SHT sampling dates

Event	Date
20 gallons solvent trim added to MCU	November 8, 2016
11 gallons Isopar™ L added to MCU	November 13, 2016
SHT sample MCU-16-1363-1364-1365	November 15, 2016
SHT sample MCU-16-122-127 (6 P-nut vials)	December 4, 2016
51 gallons solvent trim added to MCU	December 14, 2016
15 gallons Isopar™ L added to MCU	December 27, 2016
SHT sample MCU-17-86-87-88	January 9, 2017
18 gallons of solvent trim added to MCU	December 3, 2017
23 gallons of Isopar™ L added to MCU	January 18, 2017
SHT sample MCU-18-1-2-3	January 8, 2018
SHT sample MCU-18-18-19-20	February 22, 2018
SHT sample MCU-18-108-109-110	March 19, 2018
SHT sample MCU-18-123-124-125	April 24, 2018
15 gallons Isopar™ L added to MCU	April 28, 2018
10 gallons Isopar™ L added to MCU	May 14, 2018
SHT sample MCU-18-135-136-137	May 21, 2018
SHT sample MCU-18-192-193-194-195-196-197	June 18, 2018

Samples shown in Table 2-1 were received in P-nut vials containing ~10 mL each (see Figure 1). Once taken into a radioactive hood, the samples were visually inspected and analyzed for pH. Contents of the P-nut vials for each monthly sample (SHT) were composited before use. Aliquots of the composited sample were removed to perform the following analyses: Density, SVOA, High Performance Liquid Chromatography (HPLC), titration, gamma counting, CV-AA, XRF, and FT-HNMR. Results from analytical measurements were compared with the theoretical values shown in Table 2-2. Please note that the SVOA, HPLC, XRF, CVAA, Density, titration, and FT-HNMR results for each monthly SHT sample are shown in the monthly reports.

Table 2-2 Nominal concentrations of the relevant components in NGS Blend at 25 °C [5]

Component	mg/L	Molar
MaxCalix	~ 44,400♠ to 47,800♣	~ 0.0465 to 0.050
BOBCalixC6*	< 4,030	< 0.0035
TOA*	< 530	< 0.0015
Modifier	~ 169,000	~ 0.50
TiDG	~1,440	~ 0.003
Isopar™ L	~ 607,000♣ to 613,000♠	~ 73.05 to 73.69 wt %

*Values represent starting values when NGS blend was implemented. These components are no longer added to or refurbished in MCU.

♣ Solvent composition is closer to a pure NGS formulation.

♠ Solvent composition is closer to a Next Generation Solvent- Caustic Side Solvent Extraction (NGS-CSSX) blend formulation.

2.2 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.

3.0 Results and Discussion

Each sample (and its corresponding P-nut vial) was visually examined. No immiscible phases or floating debris or foam were observed (see Fig. 1). All samples had a pH value of 5.5. No unusual reactions, solids, foaming, or immiscible layers were observed after combining the samples into one vial for each set of monthly SHT samples.

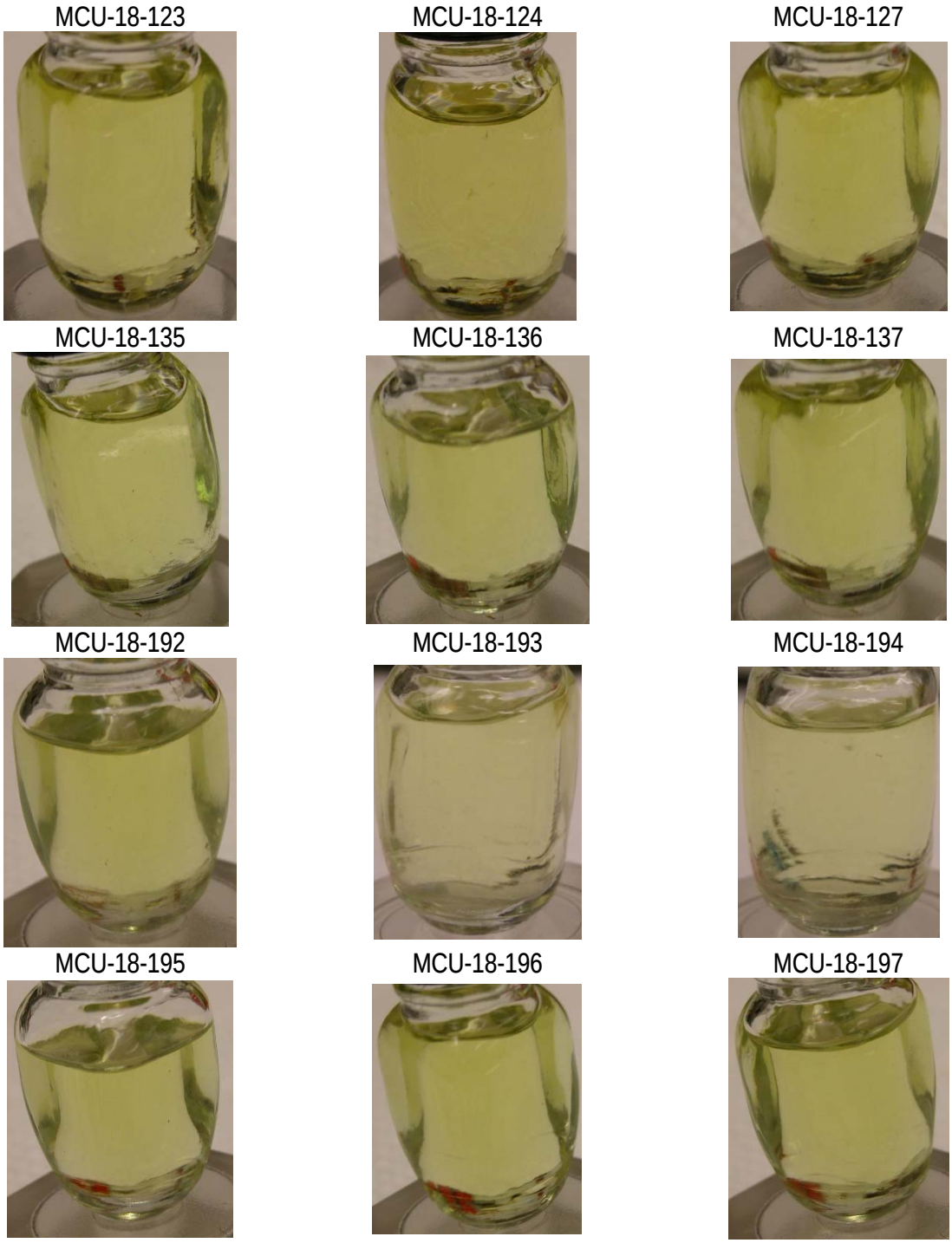

Figure 1 A picture of samples MCU-18-123-125 (top), MCU-18-135-137(middle) and MCU-18-192-197 (bottom).

Modifier Concentrations and Density Measurements

MCU suspended aqueous liquid waste processing in January 2017 and, except for processing ~3,780 gallons in December 2017, did not resume until May 2018, so the chemical composition of the SHT was not expected to change significantly during that time (since leaching, evaporation, chemical decomposition and/or reaction, and radiation damage rates are minimal). Based on the June sample, triplicate density measurements (by the vibrations of a filled tube method)[7] gave an arithmetic average similar to the baseline solvent at 25 °C when corrected for temperature using the CSSX temperature correction formula[8] (see Fig. 2), but the measurement error intervals include the calculated baseline solvent density value (0.830 g/mL at 25 °C); therefore, it cannot be concluded that the measurements are different from the baseline sample measurement.[2,4] The dip in the density (and also in the Modifier concentration) of the May sample is due to dilution (and insufficient mixing by the time the SHT was sampled in May) from the addition of Isopar™ L on April 28 and May 14. The density and Modifier values in the April sample were consistent with their previous concentrations when MCU was idled. Similarly, the Modifier concentration (1.71E5 mg/L) based on the June sample was similar to the recommended value (1.69E5 mg/L). The Isopar™ L concentration in the June sample was similar to the baseline solvent. Therefore, the calculated Isopar™ L concentration in the June sample is similar to the baseline solvent. Figure 2 shows the monthly Modifier concentration and density of the SHT samples. As expected, the curve pattern of the Modifier concentration mimic that of the density. The recommended Modifier concentration (1.69E05 mg/L) in Fig. 2 is well within the error intervals of the three MCU samples reported in this report. Both the density data and the Modifier concentration correlate with each other as expected; the solvent density is a concentration-weighed linear combination of the Modifier and Isopar™ L pure densities. Other physical measurements of the April, May, and June SHT samples such as viscosity and surface tension were also similar to the baseline solvent (see Fig. 3).

All measurements indicate (based on the June sample) the Isopar™ L concentration is at its nominal value while the Modifier concentration was 1% above its nominal value (see Fig. 2). Looking at Fig. 2, the Modifier concentration appears to be above its nominal concentration right after the trim addition to the solvent on December 3, 2017, but it dipped in the May 2018 sample due to Isopar™ L addition to the solvent. The Modifier concentration range then returned to its recommended concentration in the June 2018 sample after it contacted radioactive salt solution. The trend in the Modifier concentration correlates with the trend in the density measurements as expected (see Fig. 2).[8]

Other physical properties indicated the solvent composition is similar to that of the scratch solvent. Both the viscosity and surface tension of the April, May and June SHT solvent was similar to that of the scratch solvent (see Fig. 3).

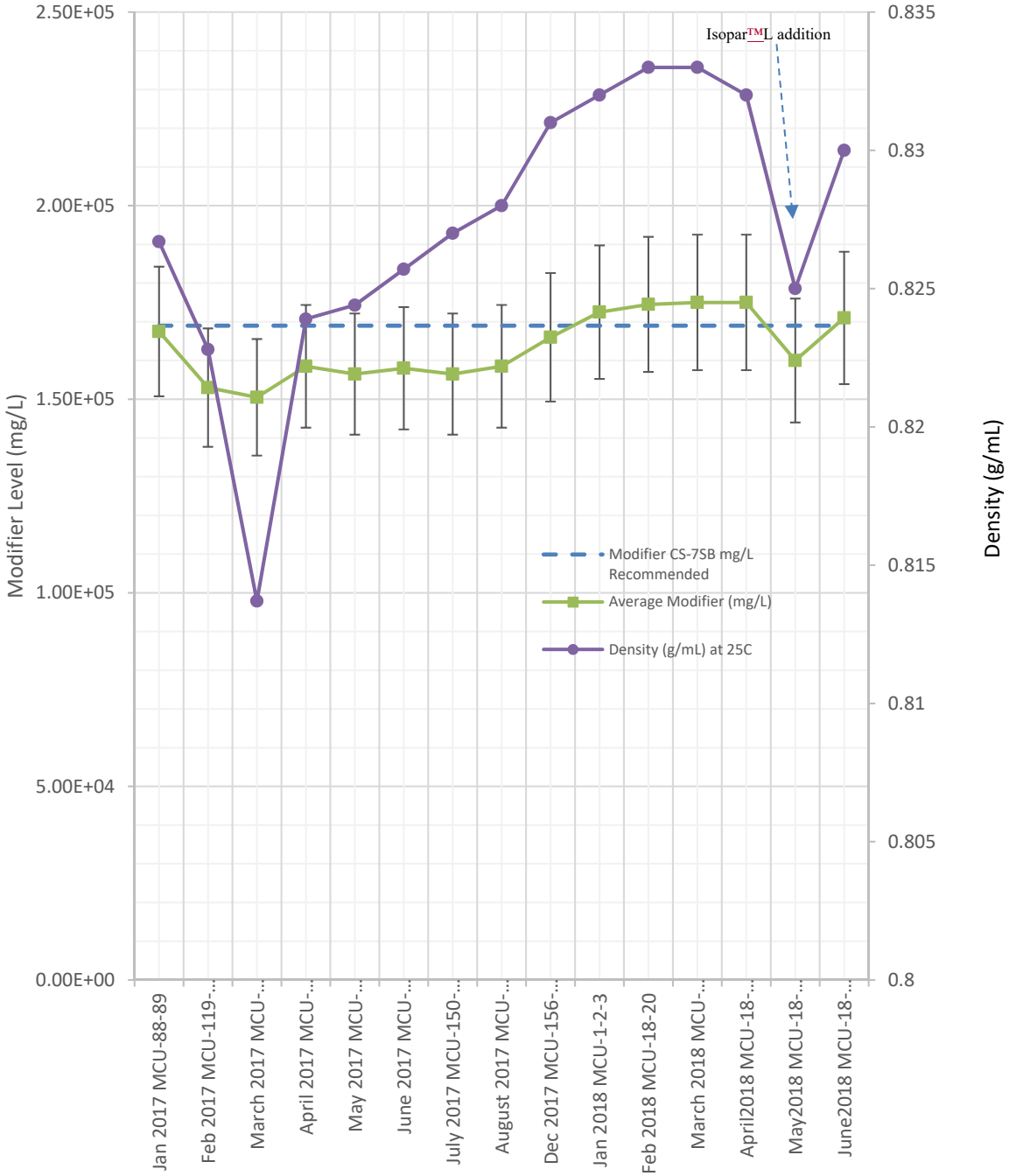

Figure 2. Modifier concentration in the solvent as measured by HPLC (one sigma is 10%).

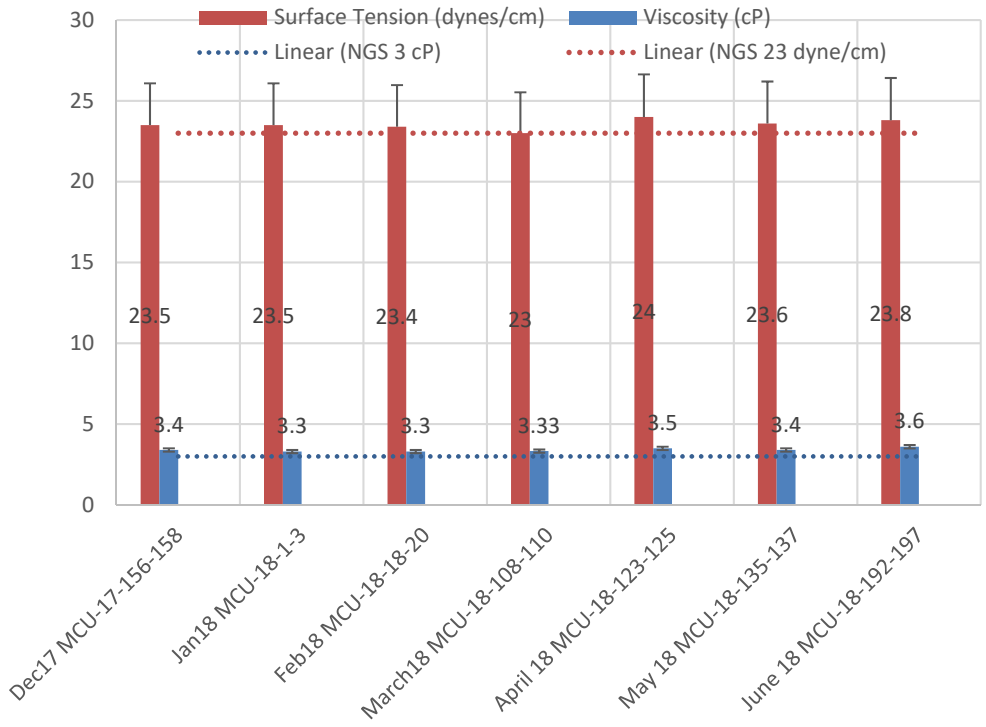

Figure 3. Viscosity and surface tension measurements of the last 7 SHT samples. The scratch blend measured a viscosity of 3±0.05 cP and a surface tension of 23±0.3 dynes/cm.

Suppressors Concentrations

The average TiDG concentrations for MCU-18-123-125, MCU-18-135-137, and MCU-18-192-197, are shown in Figure 4. As can be seen in Fig. 4, the TiDG concentration has steadily declined since MCU started operations in May 2018 (the April measurement was also lower probably due to Proficiency Runs at MCU in December 2017). This finding is consistent with the addition of the diluent and mass transfer when contacting aqueous solutions. It is also consistent with previous TiDG concentration drops in 2016. Based on the June SHT sample, the suppressor concentration (1,116 ± 86 mg/L) is above its minimum recommended operating concentration (479 mg/L), but below the nominal concentration (1,440 mg/L). The TOA concentration appears to remain steady and it is currently at 195 ± 28 mg/L. Since May 2016, the TOA level range can be estimated by 198 ± 29 mg/L. Since MCU no longer adds TOA, a drop in TOA concentration is expected with time. However, a detectable and steady TOA concentration persists with time, perhaps due to a slower than expected degradation rate, a slower transfer rate to the aqueous streams during operation, or the degradation of TiDG into primary amines, which have previously been identified as degradation products of the suppressor when heated (3 ºC, 25 ºC and 36 ºC).[9] The primary amine degradation products would likely have a similar pKa to the TOA (tertiary amine) making the equivalent points coincide, and therefore difficult to distinguish.[10]

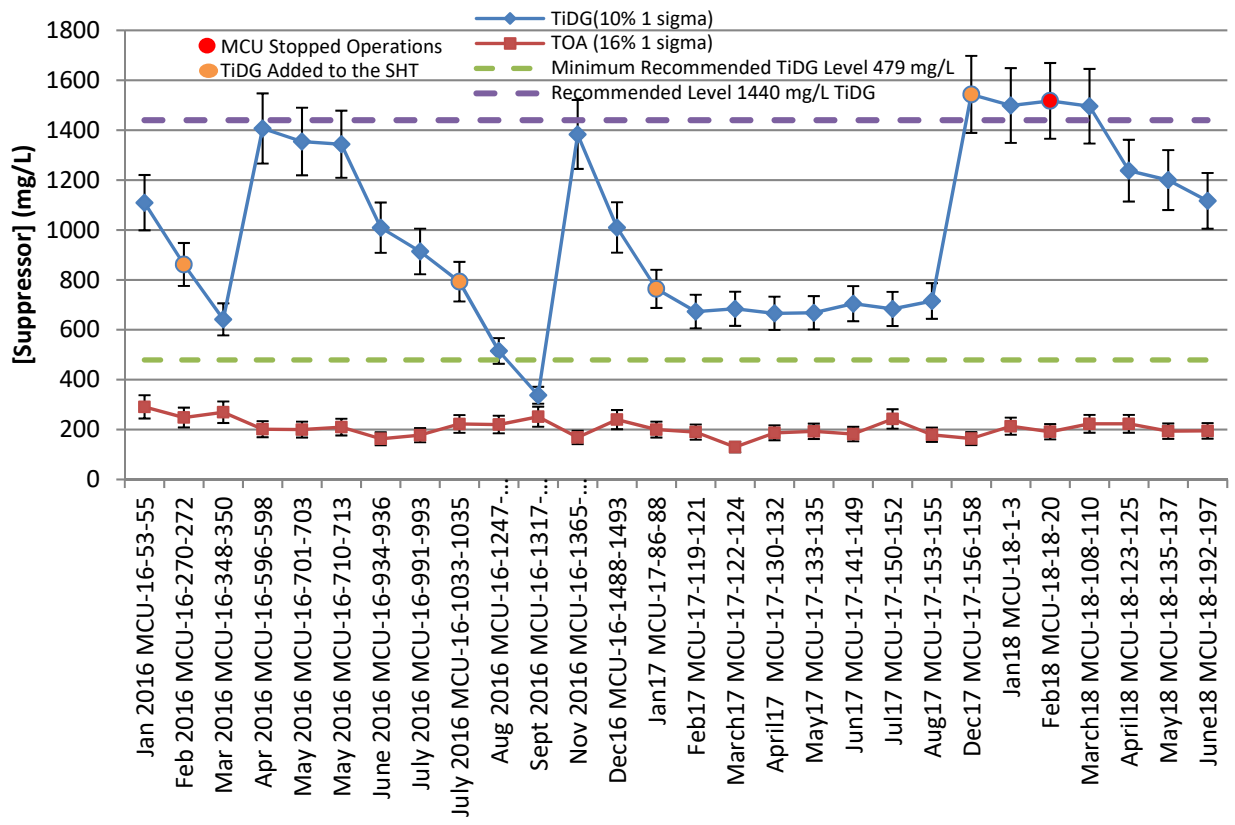

Figure 4. Suppressor concentration as measured by titration in the SHT samples since NGS implementation. The minimum recommended concentration is 479 mg/L for TiDG.

Extractant Concentrations

The calculated MaxCalix concentrations ranged from 4.83E4 to 4.36E4 mg/L in the last three SHT samples. That concentration of MaxCalix has been previously observed (see Fig. 5). Their error intervals include the recommended value (4.63E4 mg/L). However, a noted upward trend (from the trim addition in December 2017) above the recommended concentration was observed. Note the current recommended value is the difference between 47,800 mg/L (50 mM MaxCalix as referred to in Table 2.2) and the current BOBCalixC6 concentration in the SHT (1.41E3 mg/L in the June sample). The recent variations in the MaxCalix concentration seen in Fig. 5 (including a concentration increase since the January 2018 SHT sample) is within the uncertainty range for this measurement despite the addition of MaxCalix to the solvent on December 3, 2017. The uncertainty is possibly due to the aggregate of analytical, sampling, and process variances.

The residual concentration of BOBCalixC6 concentration is (based on the June sample) at 35% of the concentration measured when the NGS was implemented in late FY13 (the concentration variability is due to analytical fluctuations). This concentration is approximately the same concentration observed in previous samples. Since no BOBCalixC6 is added to the SHT, the variable trend in BOBCalixC6 concentration with time is more reflective of the analytical uncertainty (the standard deviation of the BOBCalixC6 concentration since January 2018 is 9.3% which is similar to the 10% method of uncertainty reported by HPLC). Since January 2018, the BOBCalixC6 concentration range can be estimated by 1.4 ± 1E3 mg/L. Given that no BOBCalixC6 is added to the solvent, the concentration is expected to decrease with time.

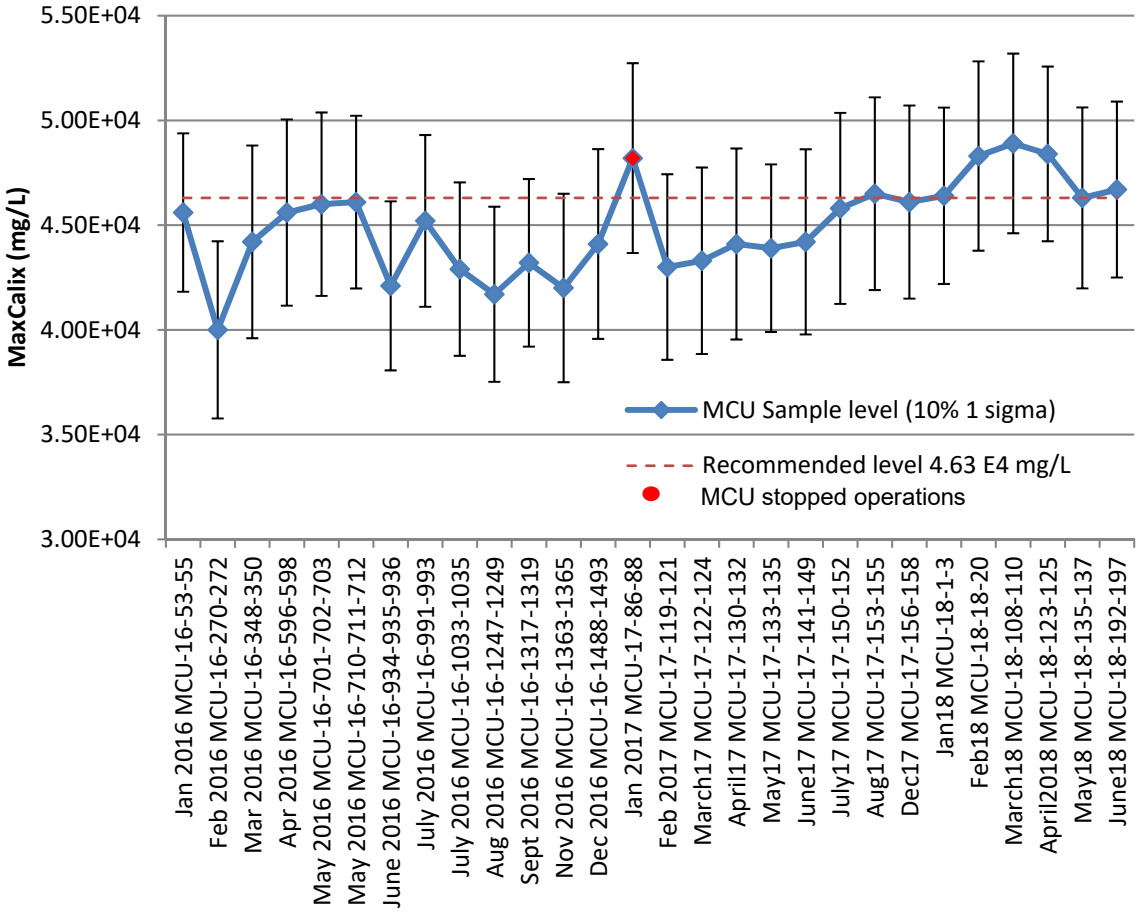

Figure 5. MaxCalix concentration as measured by HPLC and FT-HNMR of recent samples since NGS implementation (46,000 mg/L is the nominal concentration).

Gamma Measurements

The gamma measurements for the April, May, and June samples are shown in Fig. 6 in relation to past gamma measurements. The values in the May and June samples are consistent with previous levels observed during normal operation (for example in 2016). The gamma measurements for the May SHT sample were lower than the April and June samples, possibly due to several factors that include the Isopar™ L addition (12% dilution or 25 gallons of Isopar™ L to 200 gallons of solvent), processing start-up, and measurement imprecision. The gamma counts in the June 2018 SHT sample are consistent with routine MCU processing salt solutions before January 2017 and also indicate that additional stripping of the solvent by recirculating Decontaminated Salt Solution (DSS) did not reduce the gamma counts.

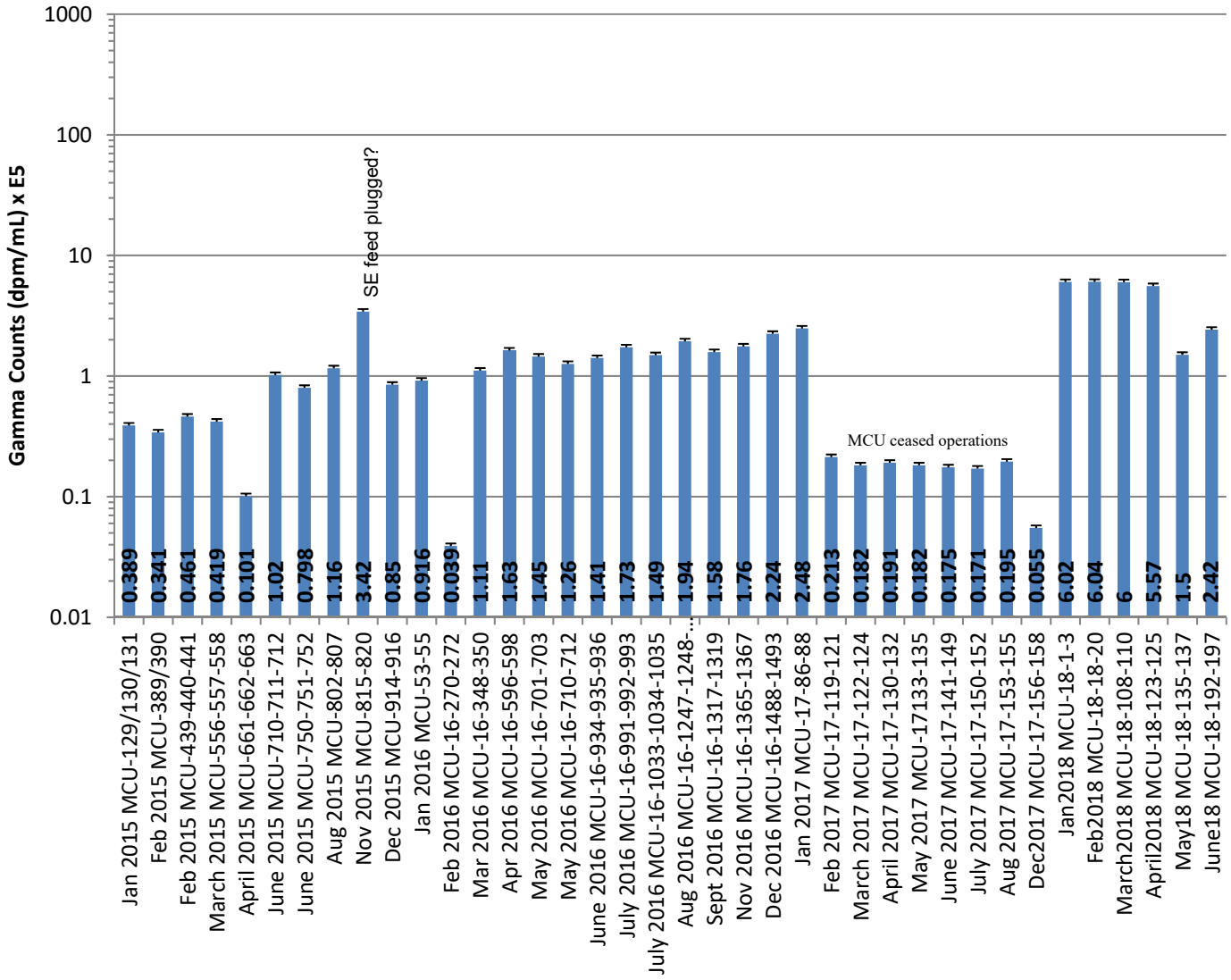

Figure 6. The gamma count of selected SHT samples. One sigma is 5%.

Impurities

No impurities were observed when performing the SVOA. However, the FT-HNMR analysis revealed the presence of three visible peaks (7.6, 7.4, and 4.3 ppm) not observed in the solvent spectrum (see Fig. 7). There is the potential that other new peaks may also be present, but if any are, they overlap with the solvent peaks and are indistinguishable. Assuming these peaks are associated with a new substance (and not a mixture), possible candidates with peaks in these locations include naphthalene, benzyl alcohol (from Modifier and/or MaxCalix), sec-butyl phenol, nitrosobenzene, phthalates, and phenyl sulfone (resin or degraded polyphenylene sulfide [PPS] from the coalescer). Based on peak characteristics, peak splitting, and existing chemical conditions in the Tank Farms and at MCU, the molecule that best fit those peaks is an aliphatic phthalate (see Appendix A). The peaks at 7.6 and 7.4 ppm are consistent with the CH in the aromatic ring of a phthalate. The peaks (triplet) at 4.52 ppm are consistent with an aliphatic CH_2 near an ester (aromatic). The concentration of this impurity is estimated to be 957 mg/L in the April SHT sample. This number was obtained by measuring the area under the peak at 7.4 and 7.7 ppm for the impurity (5,693 intensity*ppm) and that of the Modifier (254,405 intensity*ppm for the triplet between 6.2 and 5.7 ppm).

Then ratio the two areas and multiply by the Modifier concentration in the solvent which for the June SHT sample was 171,000 mg/L (accounting for the four hydrogens in the aromatic ring of the phthalate).

This impurity, if identified correctly, may have originated either from sample handling or the solvent contacted material containing the plasticizer like polyvinyl chloride.

SRNL ruled out that this impurity is from the degradation of the coalescer media (polyphenylene sulfide) used at MCU. A recent plugged Strip Effluent (SE) coalescer was sent to SRNL where it was determined that the coalescer media showed evidence of sulfoxide (-S=O) group formation (oxidation of the PPS) on the media fibers. It is possible that high beta-gamma radiation could promote further oxidation of the sulfide to S=O and then to $-SO_2-$ but that requires long exposure (several months) of the coalescer media to high radiation fields which is not tenable. MCU recently replaced the DSS coalescer media during the extended outage in August 2017; however, prior to installing the new media, the previous media had been in place since April 2015. It is possible that degraded PPS material may have contacted the solvent and that upon replacing that coalescer (no longer a source of it), the material was progressively removed with liquid waste processing. It is also possible that a compromised media, one that was not properly stored and exposed to UV radiation (solar), was put in service and it released oxidized product into the solvent during processing. The PPS could be damaged by exposure to air under high gamma radiation, but that rate of damage is much slower (unless, the gamma radiation hits metal and generates UV radiation). Therefore, it is unlikely that the impurity originated from degraded PPS.

SRNL also ruled out the impurity originating from the degradation of a cation resin given that spent resins (both anion and cation exchanged types) were historically deposited in the Tank Farm. An impurity from partially degraded cation-exchange resin could potentially interfere with the stripping of the cation cesium, if at elevated concentrations in the solvent, since it contains a sulfate group similar to sulfate soaps known to interfere with cesium stripping and an aromatic group that can increase this impurity solubility in the solvent. But we believe this mechanism is not possible since approximately 2/3 of the resin is digested before is added to the tanks. In addition, the upstream waste is first filtered through a 0.1 micron filter media that could remove it unless the resin has been digested at the molecular level. Sec-butyl phenol was not detected by this method due to interference in its spectrum with other components.

Thus, the only substance that explains the spectral features is a phthalate like molecule like dibutyl phthalate (a plasticizer used in polyvinyl chloride and other rigid polymers). We ruled out a laboratory source when the SHT samples are handled at the laboratory. We cannot explain at this time how the solvent received this impurity but based on the HNMR data, the chemical is below the HNMR detection level in the May and June samples indicating that the cleaning activities during that time were very efficient in removing it.

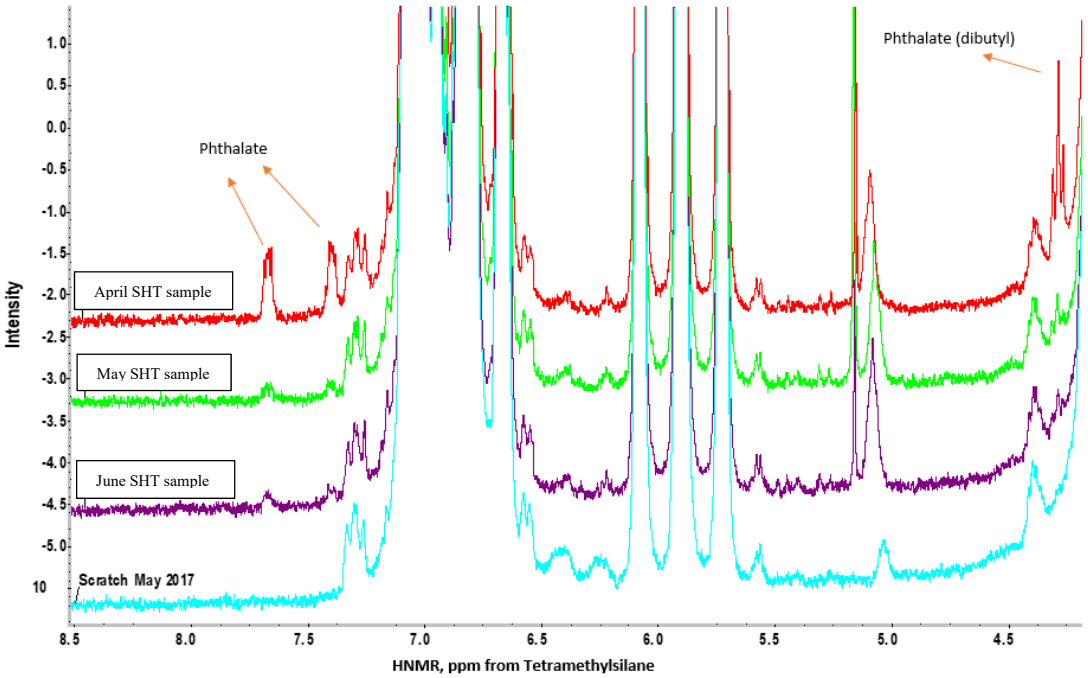

Figure 7. FT-HNMR spectra of the April, May, and June SHT samples. The FT-HNMR spectrum of the Scratch solvent is also shown (blue curve) for easier comparison.

Another impurity being tracked in the SHT solvent is the concentration of mercury. A few mL of each sample was analyzed by XRF and then digested and analyzed for total mercury by the CVAA method. The average mercury concentrations in the April, May, and June 2018 SHT samples were 24 ± 5 mg/L, 22 ± 4 mg/L, and 30 ± 6 mg/L, respectively. These concentrations are consistently lower than the concentrations observed in December 2016, when MCU last operated consistently. A second June SHT is being analyzed by XRF given the large result reported (40 mg/L).

The concentration of mercury observed in the April, May, and June samples is significantly higher than the solubility of metallic Hg in dodecane (~3 ppm),[11] implying that other solubility-enhancing mechanisms are at play (for example extraction by an extractant or sorption on trapped solids) or a more soluble form of mercury is present (organo-mercury like ethyl or dimethyl mercury). Organo-mercury compounds were recently detected in Tank 22H.[12] Based on the June SHT sample CVAA mercury measurements, for 200 gallons of solvent (757.1 L), the solvent could contain up to 24 ± 4 g of mercury (based on the June SHT sample measurement). A comparison of these measurements with previous months (especially 2016 samples) confirms a higher mercury concentration in the solvent (data is shown in Fig. 8). This may be consistent with the higher concentrations of total mercury (~109 ppm) observed in Tank 50H in the third and fourth quarters surveillance samples.[13] Please note all the XRF data since November 2017 were renormalized and compensated for solvent density variation in this report. Thus, these values differ (slightly lower values) from previous reports. The relatively lower mercury concentration observed in the May SHT sample might be due to the fact that Isopar™ L was added to the solvent (4/22 and 5/14) but the mercury concentration went up as observed in the June 2018 SHT sample consistent with the start-up of liquid waste processing at MCU.

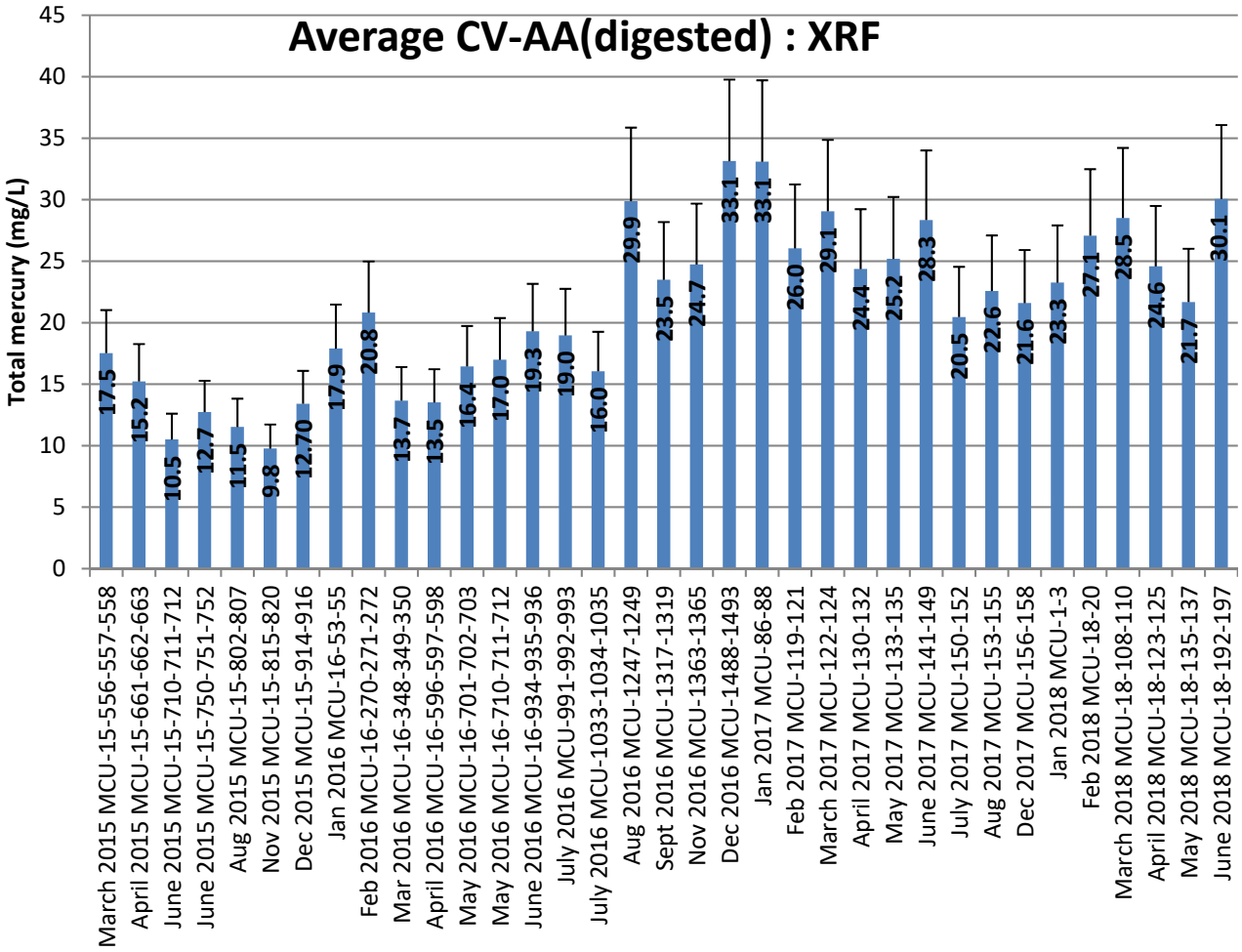

Figure 8. Total mercury in recent SHT samples. One sigma is 20%. CVAA = Cold Vapor Atomic Absorption Spectrometry. XRF =X-ray Fluorescence (20% one sigma).

Recommendations

The June SHT sample analysis indicated that the solvent's Modifier concentration was slightly above its recommended concentration (101%) and it contained 78% of the recommended concentration of the suppressor (TiDG). However, the solvent had an Isopar™ L concentration similar to the standard. The MaxCalix concentration was at its nominal concentration. The TiDG, MaxCalix, Modifier, and Isopar™ L concentrations are expected to trend downward with processing time but at different rates. Based on the June sample, the solvent did not require any further trim addition. We would investigate left over trimming solvent and p-nut vial caps for the presence of the phthalate plasticizer.

The temperature dependence of the current gravimetric density equation for solvent composition (originally obtained from CSSX solvent) needs reverification with the current NGS-CSSX solvent to improve the formula accuracy in extracting the component concentrations in the solvent.

4.0 Conclusions

A trend summary of three Solvent Hold Tank (SHT) monthly samples; MCU-18-123-125, MCU-18-135-137, and MCU-18-192-197 are reported. Most of the conclusions are based on the June SHT sample (MCU-18-192-197). Analyses of the June SHT sample (MCU-18-192-197) indicated that the Modifier (CS-7SB) and the extractant (MaxCalix) concentrations were at the nominal recommended concentrations (169,000 mg/L and 46,400 mg/L respectively). The suppressor (*N,N',N"*–tris(3,7-dimethyloctyl)guanidine or TiDG) concentration has decreased since the April 2018 measurement (Modular Caustic-Side Solvent Extraction Unit or MCU began processing radioactive salt solution from May through August 2018) to 1,116 mg/L, well above the minimum recommended concentration (479 mg/L), but below the nominal concentration. The apparent "downwards" trends observed in the TiDG, MaxCalix, Modifier, and Gamma measurement are consistent with the startup or Proficiency Runs) of MCU on May 22, 2018 since these new levels are consistent with the concentrations observed when MCU was fully operational in 2016.

This analysis confirms the Isopar™ L addition to the solvent on April 28 and May 14, 2018. This analysis also indicates the solvent did not require further additions. Based on the current monthly sample, the concentrations of TiDG, Isopar™ L, MaxCalix, and Modifier are sufficient for continuing operation but are expected to decrease with time. Periodic characterization and trimming additions to the solvent are recommended.

The SVOA did not detect any impurities. However, the FT-HNMR analysis detected a high concentration of linear alkane phthalate (for example dibutyl phthalate), a typical plasticizer used in polyvinyl chloride polymers, in the solvent. The impurity concentration was highest in the April sample (~0.96 g/L) and below the detection limit in the June sample. The source of this impurity and how it ended up in the solvent is not known at this time. Another impurity observed in the samples was mercury. Based on the June SHT sample, up to 30 ± 6 micrograms of mercury per mL of solvent was detected (the average of the CVAA and XRF methods). The higher mercury concentration in the solvent (as determined in the last three monthly samples) is possibly due to the higher mercury concentration in Salt Batches 9 and 10 (SB 10 was recently added to Tank 49H in March 2017) or sampling of previously undisturbed areas of high mercury concentration in Tank 49H.

The gamma concentration (~2.42E5 dpm/mL) measured in the June SHT samples was consistent with previous values observed when MCU was fully operational (for example, between December 2016 and January 2017) but it was lower than the April SHT measurement. The "dip" in the gamma measurement for the May 2018 SHT sample was due to Isopar™ L addition to MCU during April and May 2018.

The laboratory will continue to monitor the quality of the solvent for any new impurities or degradation of the solvent components.

5.0 References

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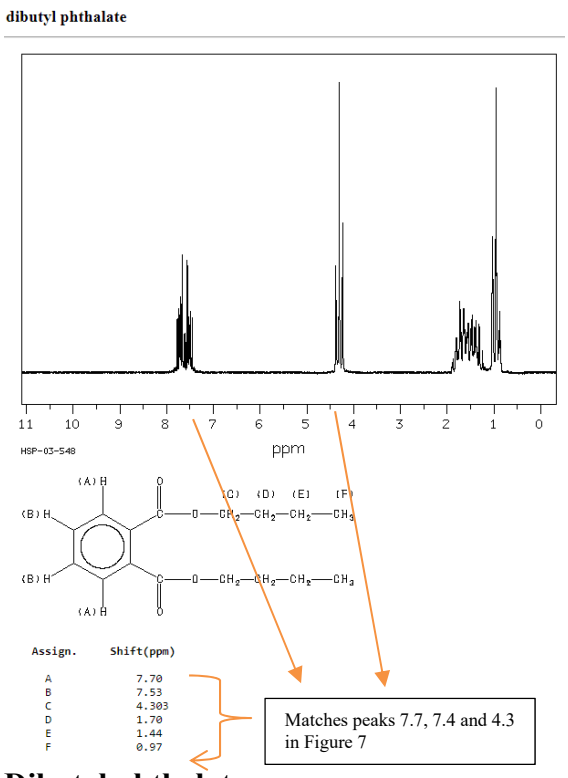
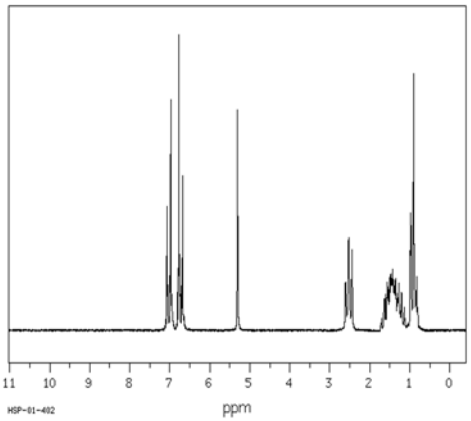
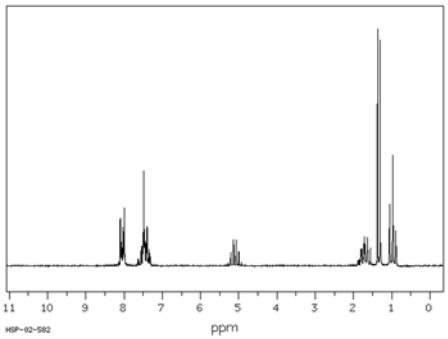
Appendix A. H-NMR spectra of different subtances that may explain the new peaks in Fig. 7.



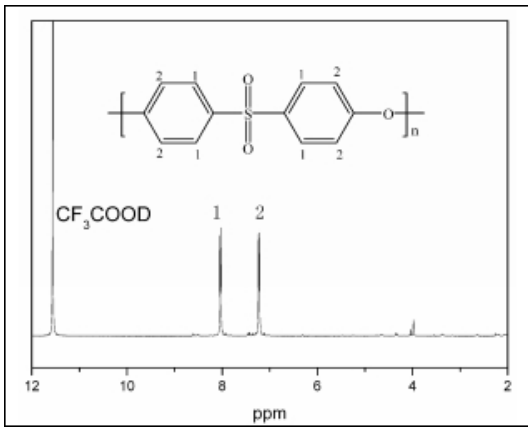
Dibutyl phthalate



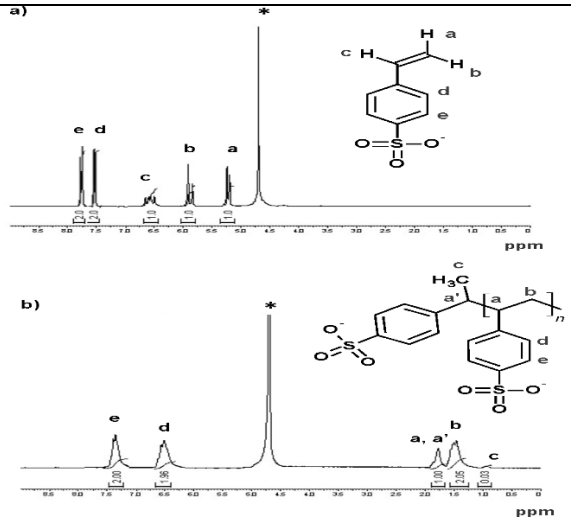
Sec-butyl phenol



Sec-butyl benzoate



Paraphenylene sulfone



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