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Solvent Hold Tank Sample Results for MCU-18-301-303 (July 2018), MCU-18-357-359-360 (August 2018), and MCU-18-402-410 (September 2018): Quarterly Report

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May 2019

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EXECUTIVE SUMMARY

A trend summary of three Solvent Hold Tank (SHT) monthly samples; MCU-18-301-302-303, MCU-18-357-359-360 and MCU-18-402-410 are reported. Most of the conclusions are based on the September SHT sample (MCU-18-402-410). Analyses of the September SHT sample (MCU-18-402-410) indicated that the Modifier (Cs-7SB) and the extractant (MaxCalix) concentrations were below their nominal recommended concentrations (169,000 mg/L and 46,400 mg/L respectively) by 5% and 8% 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 241 mg/L, well below the minimum recommended concentration (479 mg/L). The apparent “downwards” trends observed in the TiDG, MaxCalix, and Modifier measurements are consistent with the startup (or Proficiency Runs) of MCU on May 22, 2018; 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 July 2 and August 9, 2018. This analysis also indicates the solvent did require a trim addition. Based on the current monthly sample, the concentrations of Isopar™ L, MaxCalix, and Modifier are sufficient for continuing operation but are expected to decrease with time. The TiDG concentration was below its recommended level. Periodic characterization and trimming additions to the solvent are recommended.

The Semi-Volatile Organic Analysis (SVOA) did not detect any impurities. However, the Fourier Transform Hydrogen Nuclear Magnetic Resonance (FT-HNMR) analysis detected a low concentration of sec-butyl phenol (SBP) at levels of a few ppm. The impurity concentration was highest in the July sample and consistently detected in the August and September samples (but always a few ppm or less). The source of this impurity is due to the degradation of the Modifier. Another impurity observed in the samples was mercury. Based on the September SHT sample, up to 17 ± 3 micrograms of mercury per mL of solvent was detected (the average of the Direct Mercury Analysis (DMA) 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).

The gamma concentration ($\sim 2.3E5$ dpm/mL) measured in the September 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 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.

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LIST OF ABBREVIATIONS

BOBCalixC6	Calix[4]arene-bis(<i>tert</i> -octylbenzo-crown-6)
CV-AA	Cold Vapor-Atomic Absorption
CSSX	Caustic-Side Solvent Extraction
DMA	Direct Mercury Analysis
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- <i>alt</i> -25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6
NGS	Next Generation Solvent
PPS	Polyphenylene Sulfide
SBP	secbutylphenol
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
TiDG	<i>N,N',N''</i> -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-18-301-302-303, MCU-18-357-359-360, and MCU-18-402-410. 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 cocktail⁵ and heel solvent – was prepared in the lab (May 2018) 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 August 9, 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
18 gallons of solvent trim added to MCU	December 3, 2017
SHT sample MCU-18-1-2-3	January 8, 2018
SHT sample MCU-17-86-87-88	January 9, 2017
21 gallons of Isopar™ L added to MCU	January 18, 2017
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
14 gallons Isopar™ L added to MCU	July 2, 2018
SHT sample MCU-18-301-302-303	July 14, 2018
13 gallons Isopar™ L added to MCU	August 9, 2018
SHT sample MCU-18-357-359-360	August 20, 2018
SHT sample MCU-18-402-410	September 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 SHT sample were composited before use. The following physical and analytical measurements were performed on the samples: Density, SVOA, High Performance Liquid Chromatography (HPLC), titration, gamma counting, Direct Mercury Analysis (DMA), X-ray Fluorescence (XRF), and Fourier-Transformed Hydrogen Nuclear Magnetic Resonance (FT-HNMR). Results from analytical measurements were compared with the theoretical values shown in Table 2-2. Please note that

the SVOA, HPLC, XRF, DMA, 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 (Ref. 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 NGS - Caustic Side Solvent Extraction (NGS-CSSX) blend formulation.

♥ Assuming a molecular weight for caustic-washed TiDG of 479 g/mol (516 g/mol for TiDG*HCl).

2.2 Quality Assurance

The collected and analyzed data in this report meets the Quality Assurance classification level defined in SRNL-STI-2013-00536, Revision 2. The work presented in this report is exempted from RW-333P criteria. 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 floating debris or foam were observed (see Fig. 1). However, the bottom of the P-nut vials (MCU-18-301-302-303) showed a deposited layer that upon further investigation (by FTIR) was caustic water and Modifier with traces of sec-butylphenol (SBP). 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 Teflon container for each set of monthly SHT samples.

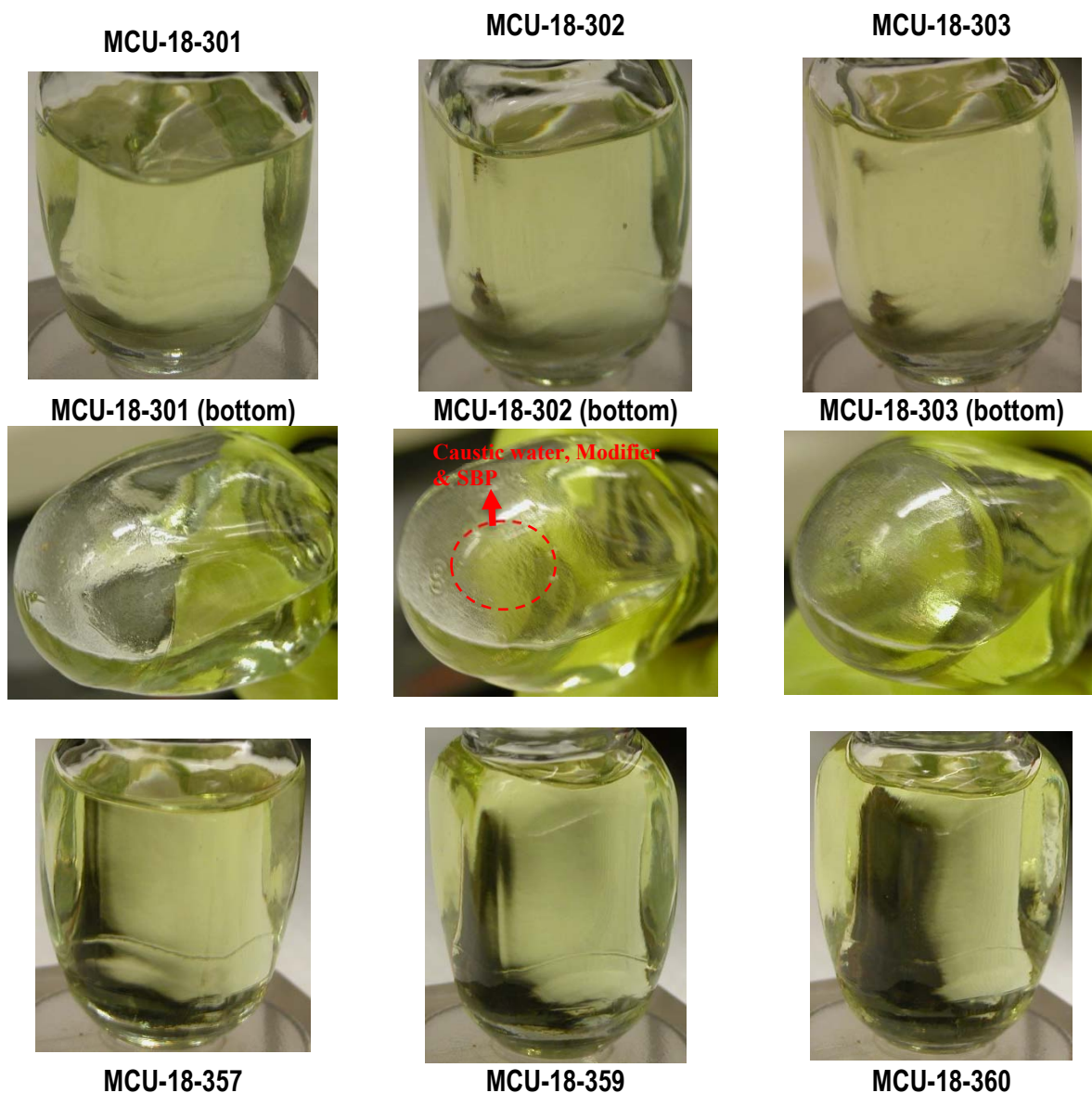


Figure 1 A picture of samples MCU-18-301-303 (top), the bottom of MCU-18-301, MCU-18-302 and MCU-18-303(middle), and MCU-18-357, MCU-18-359, and MCU-18-360 (bottom). No pictures of MCU-18-402-410 were taken due to a camera malfunction.

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 July-August-September results, there was a downward trend in the density (triplicate measurements corrected for temperature using the CSSX temperature correction formula) [see Fig. 2].^{7 8} 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 statistically different from the baseline sample measurement.^{2,4} The downward trend in the density (and also in the Modifier concentration) of the July-August-September samples is due to dilution (and possibly insufficient mixing by the time the SHT was sampled in August and September) from the addition of Isopar™ L on May 14. The density and Modifier values from the July-August-September samples were consistent with previous concentrations when MCU was idled. Similarly, the Modifier concentration level ($1.60 \pm 0.2E5$ mg/L) and its error interval (from the September sample) were below the recommended Modifier concentration level ($1.69E5$ mg/L) based on the September sample. The Isopar™ L concentration (not shown) in the September sample was similar to the baseline solvent. Both the density data and the Modifier concentration correlated with each other as expected (see Fig. 2).⁸ This is expected since the solvent density is a volume-weighted linear combination of the Modifier and Isopar™ L pure densities. Other physical measurements of the July, August, and September SHT samples such as viscosity and surface tension were also similar to the baseline solvent measurements (see Fig. 3) but a closer look at the viscosity and surface tension reveals a downward trend that is consistent with a lower Modifier concentration in the solvent from July to August 2018.

All measurements indicate (based on the July, August, and September samples) that the Isopar™ L concentration was at its nominal value. Isopar™ L is added to the solvent more frequently (compared to the Modifier) to compensate for its high evaporation rate.

Suppressors Concentrations

The average TiDG concentrations for MCU-18-301-302-303, MCU-18-357-358-359, and MCU-18-402 through 410, 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 TiDG mass transfer out of the solvent when contacting aqueous solutions. It is also consistent with previous TiDG concentration drops in 2016. Based on the September SHT sample, the suppressor concentration (241 ± 24 mg/L) is below its minimum recommended operating concentration (479 mg/L for caustic washed TiDG). The TOA concentration appears to have remained steady and it was at 223 ± 36 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 °C, 25 °C and 36 °C).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

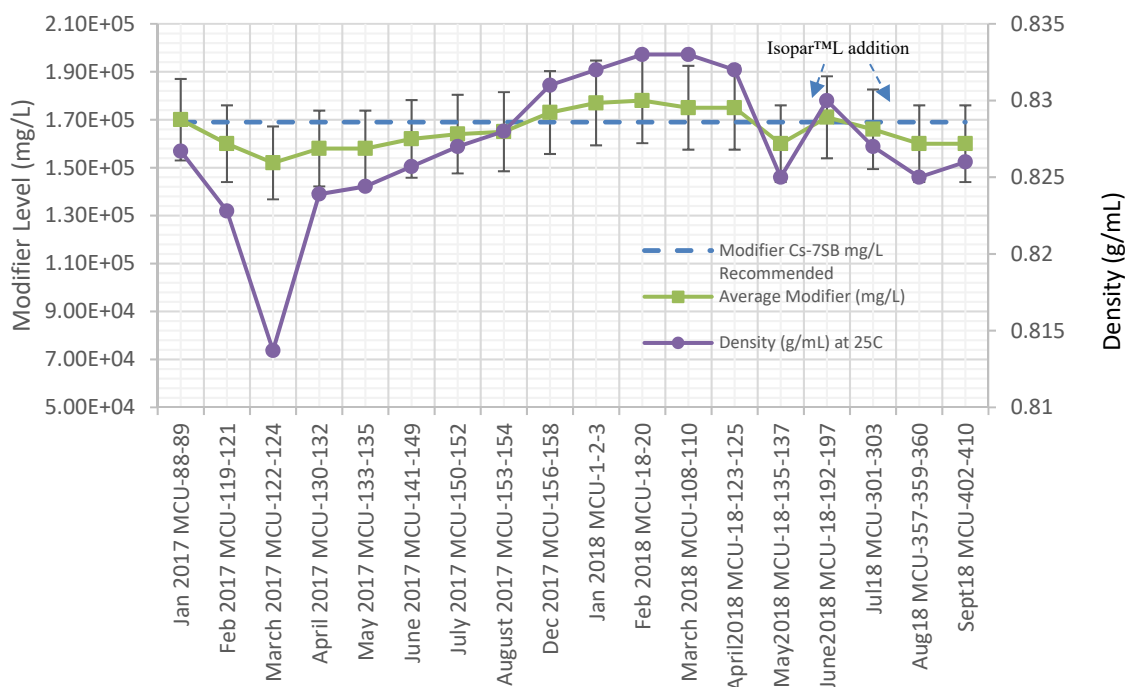


Figure 2. Modifier concentration in the solvent as measured by HPLC (one sigma is 10%).

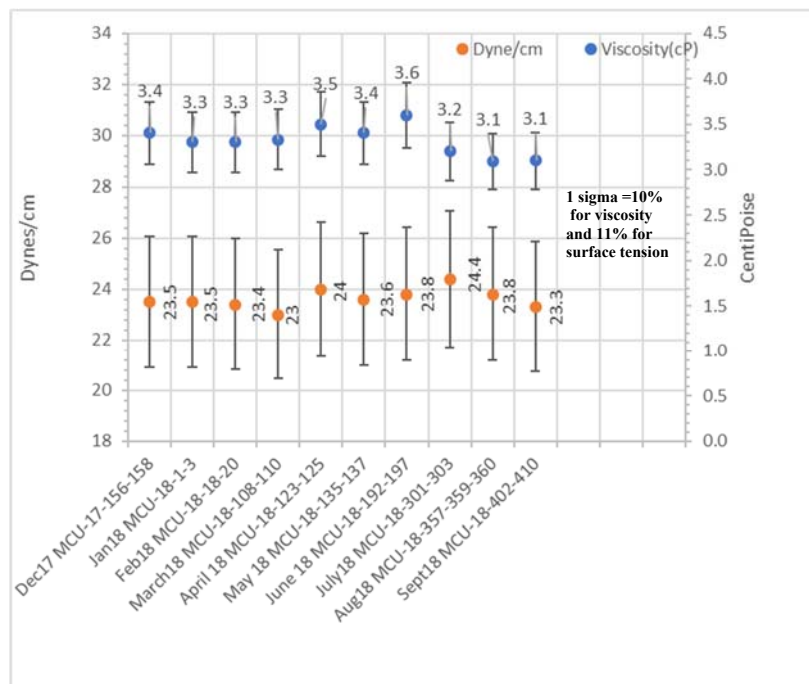


Figure 3. Viscosity and surface tension measurements of the last ten SHT samples. The scratch blend measured a viscosity of 3 ± 0.3 cP and a surface tension of 23 ± 0.3 dynes/cm.

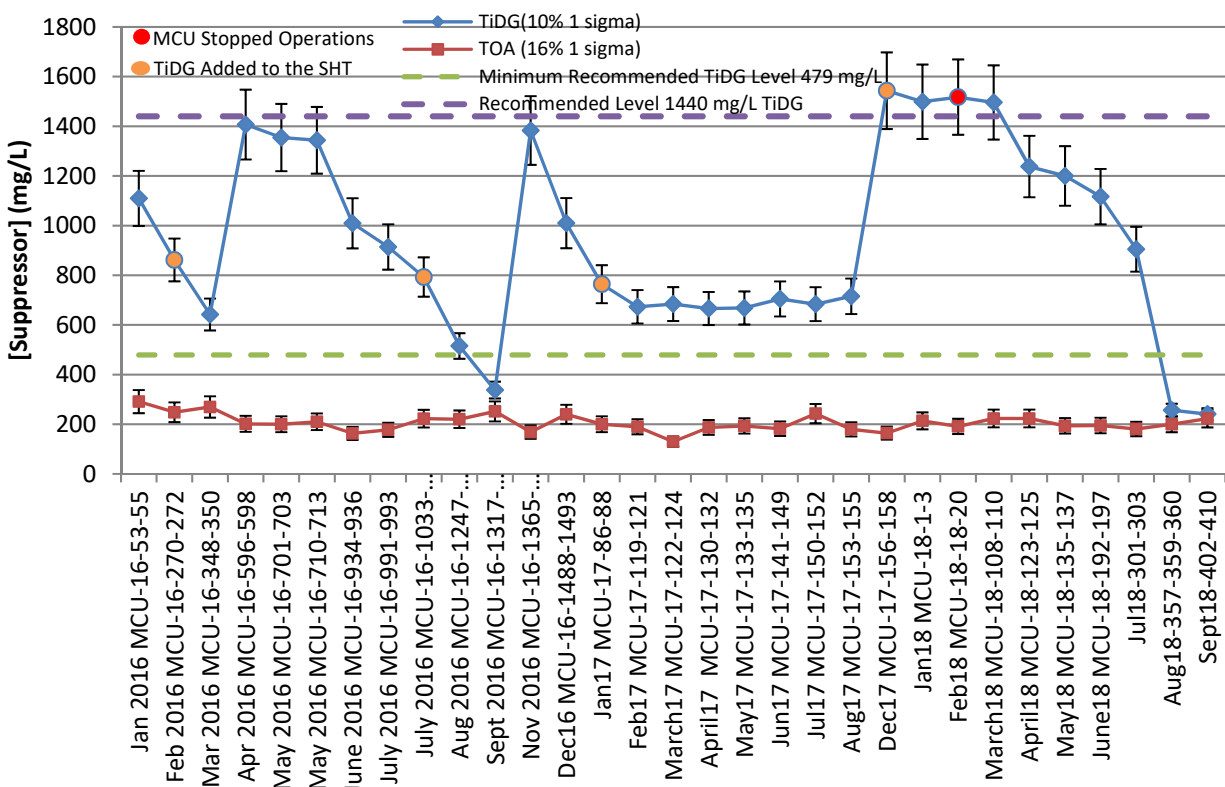


Figure 4. Suppressor concentration as measured by titration in the SHT samples since NGS implementation. The minimum recommended concentration is 479 mg/L for TiDG.

Extractant Concentrations

The calculated MaxCalix concentrations declined from 4.5E4 to 4.3E4 mg/L in the last three SHT samples. This decline is consistent with previous declines (April to September 2016 and November 2016 to January 2017) that occur after a trim addition to the solvent and subsequent contact with caustic solution. Please note the analytical error bars still overlap the recommended level of 4.65E4 mg/L. Those concentrations of MaxCalix have been previously observed (see Fig. 5) in September 2016. 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 BOBCalixC6 concentration in the SHT (1.31E3 mg/L in the September sample). The recent variations in the MaxCalix concentration seen in Fig. 5 are within the uncertainty range for this measurement despite the addition of MaxCalix to the solvent on December 3, 2017. The uncertainty is due to the aggregate of analytical, sampling, and process variances.

The residual concentration of BOBCalixC6 concentration is (based on the September sample) at 33% 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 $1.4 \pm 1\text{E}3$ mg/L. Given that no BOBCalixC6 is added to the solvent, the concentration is expected to decrease with time.

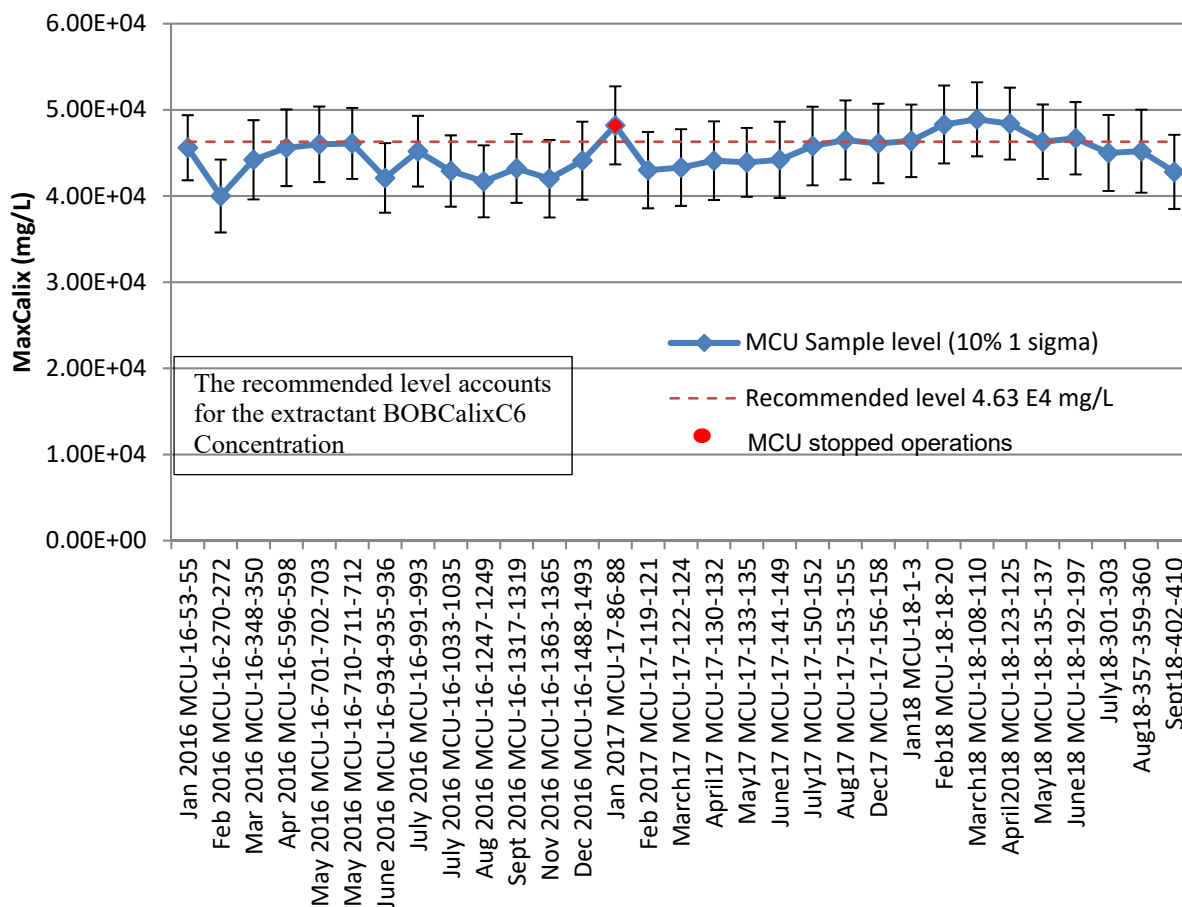


Figure 5. MaxCalix concentration as measured by HPLC and FT-HNMR of recent samples since NGS implementation (46,000 mg/L is the nominal concentration).

Gamma Measurements

The gamma measurements for the July, August, and September samples are shown in Fig. 6 in relation to past gamma measurements. The values in the August and September samples are consistent with previous levels observed during normal operation (for example in 2016). The variability in the gamma measurements is due to several factors that include the Isopar™ L addition (sometimes 12% dilution or 25 gallons of Isopar™ L to 200 gallons of solvent), processing start-up, and measurement imprecision. The gamma counts in the July, August, and September 2018 SHT samples are consistent with routine MCU processing salt solutions before January 2017. There is a positive correlation between the concentration of MaxCalix in the solvent and the gamma measurements as expected.

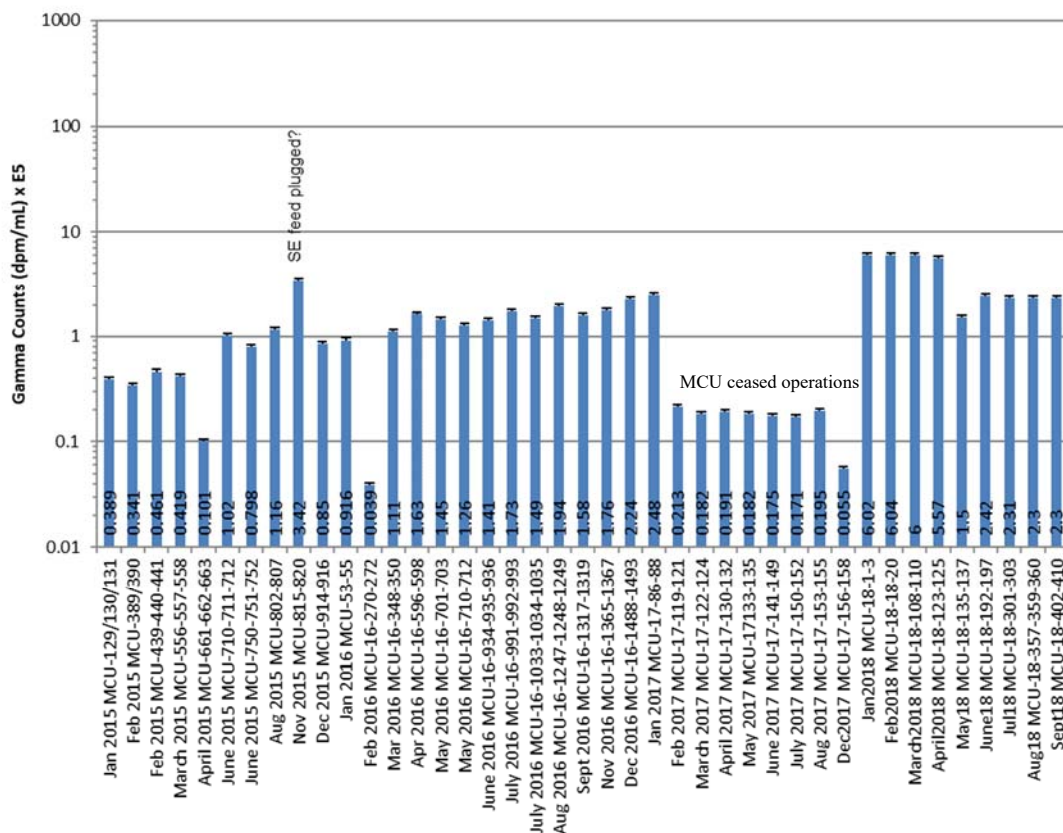


Figure 6. The gamma count of selected SHT samples. One sigma is 5%.

Impurities

No impurities were observed when performing the SVOA. However, the FT-HNMR analysis revealed the presence of three visible peaks (7.9, 7.6, and 2.5 ppm) not observed in the scratch solvent spectrum (see Fig. 7). These peaks are believed to be due to phthalates (possibly di-octylphthalate or ethylhexylphthalate a plasticizer commonly used with plastics) at the level of a few ppm. 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.

Another impurity tracked in the SHT solvent is the concentration of mercury. A few mL of each sample was analyzed by XRF and then another portion of the sample was diluted with Isopar™L for analysis by the total mercury by the DMA method. The average mercury concentrations in the July, August, and September 2018 SHT samples were 31 ± 6 ug/g, 32 ± 6 ug/g, and 28 ± 6 ug/g, respectively (see Fig. 8). Please note all mercury measurements prior to April 2018 were the average of the XRF and Cold-Vapor Atomic Absorption Spectrometry (CV-AA). After April 2018, Direct Mercury Analysis replaced the CV-AA mercury method. The July and August measurements are consistent with previous measurements. The September measurement is consistent with gamma measurements obtained since August 2016 (average mercury level of 29 ug/g if you average all measurements since August 2016) indicating a possible higher mercury concentration of the salt batches sent to ARP/MCU.

The concentration of mercury observed in the July, August and September samples is significantly higher than the solubility of metallic Hg in dodecane (~3 ppm),¹¹ implying that other solubility-enhancing

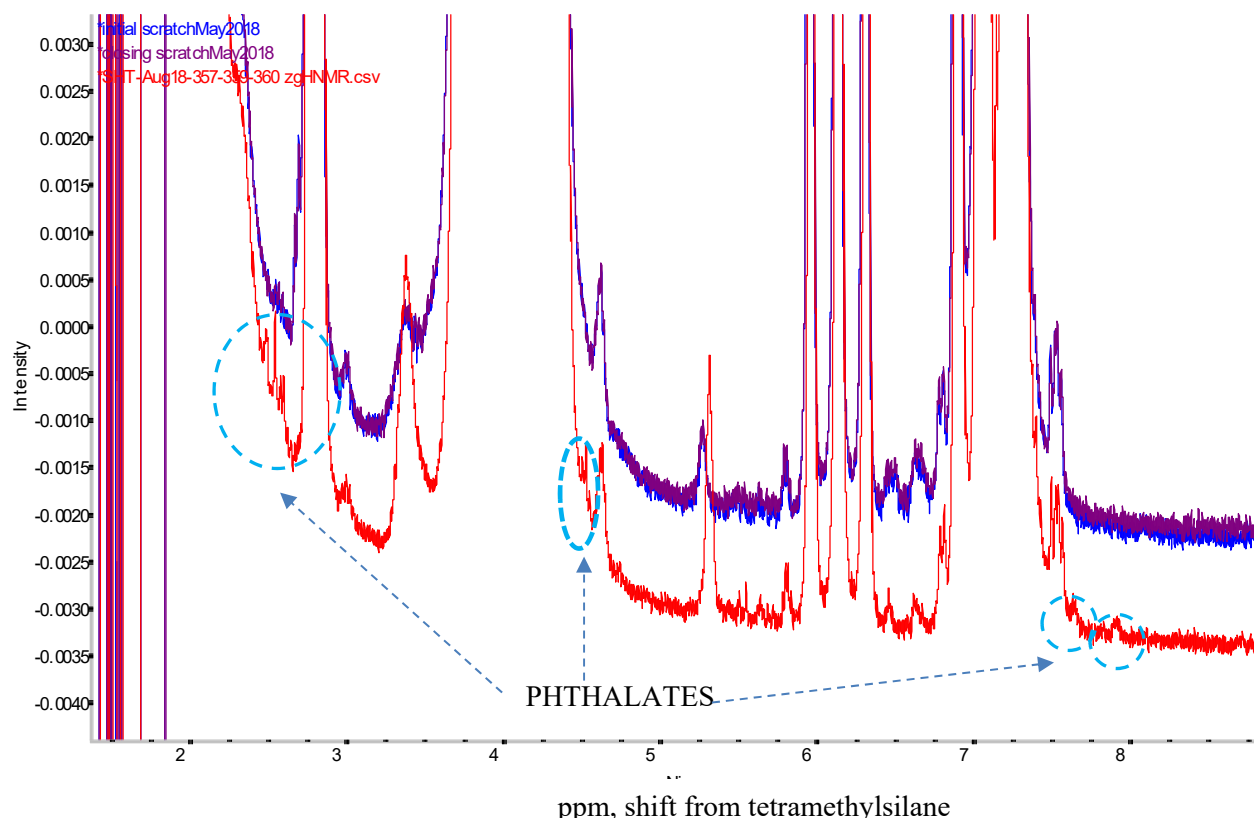


Figure 7 FT-HNMR of MCU-19-357-359-360 and of a scratch solvent made in May 2018

mechanisms are at play (for example extraction) 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 September SHT sample DMA mercury measurements, for 200 gallons of solvent (757.1 L), the solvent could contain up to 17 ± 3 g of mercury. 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 September SHT sample might be due to the fact that Isopar™ L was added to the solvent (4/22 and 5/14).

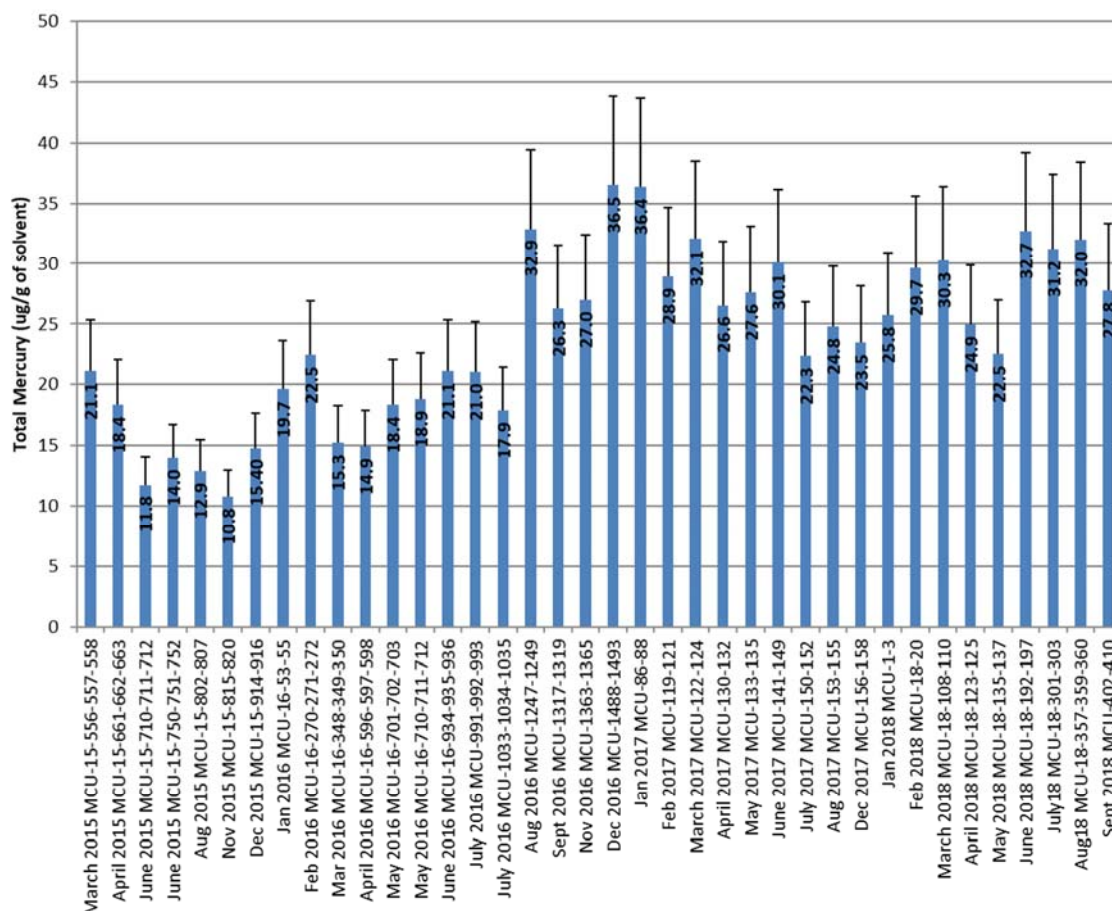


Figure 8. Total mercury in recent SHT samples. DMA = Direct Mercury Analysis (20% one sigma). XRF =X-Ray Fluorescence (20% one sigma).

Recommendations

The September SHT sample analysis indicated that the solvent's Modifier concentration was below its recommended concentration (95%) and it contained 17% of the recommended concentration of the suppressor (TiDG). However, the solvent had an Isopar™ L concentration slightly above (101%) the standard. The MaxCalix concentration was below its nominal concentration (92%). The TiDG, MaxCalix, Modifier, and Isopar™ L concentrations are expected to trend downward with processing time but at different rates. Based on the September sample, the solvent requires a trim addition.

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-18-301-302-303, MCU-18-357-359-360 and MCU-18-402-410 are reported. Most of the conclusions are based on the September SHT sample (MCU-18-402-410). Analyses of the September SHT sample (MCU-18-402-410) indicated that the Modifier (Cs-7SB) and the extractant (MaxCalix) concentrations were below their nominal recommended concentrations (169,000 mg/L and 46,400 mg/L respectively) by 5% and 8% 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 241 mg/L, well below the minimum recommended concentration (479 mg/L). The apparent “downwards” trends observed in the TiDG, MaxCalix, and Modifier measurements are consistent with the startup (or Proficiency Runs) of MCU on May 22, 2018; new levels are consistent with the concentrations observed when MCU was fully operational in 2016.

This analysis confirms the IsoparTM L addition to the solvent on July 2 and August 9, 2018. This analysis also indicates the solvent did require a trim addition. Based on the current monthly sample, the concentrations of IsoparTM L, MaxCalix, and Modifier are sufficient for continuing operation but are expected to decrease with time. The TiDG concentration was below its recommended level. Periodic characterization and trimming additions to the solvent are recommended.

The Semi-Volatile Organic Analysis (SVOA) did not detect any impurities. However, the Fourier Transform Hydrogen Nuclear Magnetic Resonance (FT-HNMR) analysis detected a low concentration of sec-butyl phenol (SBP) at levels of a few ppm. The impurity concentration was highest in the July sample and consistently detected in the August and September samples (but always a few ppm or less). The source of this impurity is due to the degradation of the Modifier. Another impurity observed in the samples was mercury. Based on the September SHT sample, up to 17 ± 3 micrograms of mercury per mL of solvent was detected (the average of the Direct Mercury Analysis (DMA) 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).

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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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