Solvent Hold Tank Sample Results for MCU-17-150-152 (July 2017) and MCU-17-153-155 (August 2017): Quarterly Report

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EXECUTIVE SUMMARY

A trend summary that includes the last two Solvent Hold Tank (SHT) monthly samples is shown; MCU-17-150-152 (July SHT) and MCU-17-153-155 (August SHT). Since the last SHT sample sent for analysis was the August sample the chemical state of the solvent is best approximated by the chemical analysis of the August SHT sample (MCU-17-153-155). This report mainly focused on the chemical analysis of the August SHT sample. The analysis data from the July SHT sample are presented in the "trend" plots of this report. Analysis of the August SHT sample (MCU-17-153-155) indicated that the modifier (CS-7SB) was 2% below but the extractant (MaxCalix) concentration was at its nominal recommended level (169,000 mg/L and 46,400 mg/L respectively). The suppressor (TiDG) level has decreased since the last measurement taken while the Modular Caustic-Side Solvent Extraction unit (MCU) was operating in January 2017, but has remained steady in the range of 666 (observed in April) to 715 mg/L (observed in the August 2017 sample) since February 2017, well above the minimum recommended level (479 mg/L), but below the nominal level. The "flat" trends observed in the TiDG, MaxCalix, modifier, and Gamma measurement are consistent with the solvent being idle since January 10, 2017. A strong correlation between density and modifier concentration in the solvent continues to be observed in the SHT samples.

This analysis confirms the Isopar™L addition to the solvent in January 2017. This analysis also indicates the solvent did not require further additions. Based on the current monthly sample, the levels of TiDG, Isopar™L, MaxCalix, and modifier are sufficient for continuing operation but are expected to decrease with time if the Modular Caustic-Side Solvent Extraction Unit (MCU) returns to processing radioactive liquid waste. Otherwise, the levels of these components will remain steady. A future Isopar™L trimming addition to the solvent is recommended when MCU resumes processing waste.

Two unknown impurities related to the modifier (but not sec-butyl phenol: a modifier degradation product observed before) at the 290 and 110 mg/L levels were observed in the August SHT sample by the Gas - Chromatography-Mass Spectrometry (GC-MS) method. They were observed in a second GC-MS re-run with a new column. Their identification can't be ascertained at this time. No impurities were observed in the Hydrogen Nuclear Magnetic Resonance (HNMR). Another impurity observed in the samples was mercury. Based on the August SHT sample, up to 23 ± 5 micrograms of mercury per mL of solvent was detected (the average of the Cold Vapor-Atomic Adsorption [CV-AA] 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 either a higher mercury concentration in Salt Batches 8 and 9 (Tank 49H) .

The gamma level (~ 2.0E4 dpm/mL) measured in the August SHT samples was one order of magnitude lower than the gamma levels observed in the December 2016 and January 2017 SHT samples. A similar level was observed in the July SHT sample (MCU-SHT-150-152). The gamma level has remained consistently steady since January 10, 2017 when MCU stopped processing radioactive liquid waste.

The laboratory will continue to monitor the quality of the solvent in particularly 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
GC-MS	Gas Chromatography-Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ISDP	Integrated Salt Disposition Project
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
RSD	Relative Standard Deviation or the absolute value of the Coefficient of Variation
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, the 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 BOBCalixC6 and trioctylamine (TOA). SHT samples are sent to the 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 two different SHT samples: MCU-17-150-151-152 and MCU-17-153-154-155. A summary report for each of the SHT samples was issued earlier.[2,3]

These samples are intended to verify that the solvent is within the specified composition range. A baseline "scratch" solvent was prepared in the lab in May 2017 and was used for comparison and evaluation. The scratch solvent is a preparation of all 6 solvent components to generate a solution of the appropriate composition that approximates the blend of cocktail[4] and heel solvent of the SHT back in 2013. The results from the analyses are presented in this document.

2.0 Experimental Procedure

2.1 Experimental Procedure

A summary of relevant and recent trims to the MCU solvent as well as the arrival date of the samples currently being studied are shown in Table 2-1. In January 2017, an Isopar™L addition was made to MCU and no further additions has been since.[5, 6]

Table 2-1 Log of recent trims to the 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 12, 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
23 gallons of Isopar™L added to MCU	January 18, 2017
SHT sample MCU-17-119-120-121	February 21, 2017
SHT sample MCU-17-122-123-124	March 18, 2017
SHT sample MCU-17-130-131-132	April 18, 2017
SHT sample MCU-17-133-134-135	May 2, 2017
SHT sample MCU-17-141-142-143-144-145-146-147-148-149	June 4, 2017
SHT sample MCU-17-150-151-152	July 10, 2017
SHT sample MCU-17-153-154-155	August 2, 2017

Samples shown in Table 2-1 were received in p-nut vials containing ~10 mL each (see Fig 1). Once taken into a radioactive hood, the samples were visually inspected and analyzed for pH. The p-nut vials for each monthly sample (SHT) were composited before use. Aliquots of the composited sample were removed to perform the following analysis: density, Semi-Volatile Organic Analysis (SVOA), high performance liquid chromatography (HPLC), titration, gamma counting, Cold Vapor Atomic Adsorption Spectrometry (CVAA), 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, CV-AA, 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 [6]

Component	mg/L	Molar
MaxCalix	~ 44,400$^{\spadesuit}$ to 47,800$^{\clubsuit}$	~ 0.0465 to 0.050
BOBCalixC6*	< 4,030	< 0.0035
TOA*	< 530	< 0.0015
Modifier	~ 169,000	~ 0.50
TiDG	~1440	~ 0.003
Isopar™L	~ 607,000$^{\clubsuit}$ to 613,000$^{\spadesuit}$	~ 73.05 to 73.69 wt%

*Values represent starting values when the NGS blend was implemented. These components are no longer added to or refurbished in MCU. $^{\clubsuit}$ Solvent composition is closer to a pure NGS formulation. $^{\spadesuit}$Solvent composition is closer to a 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.

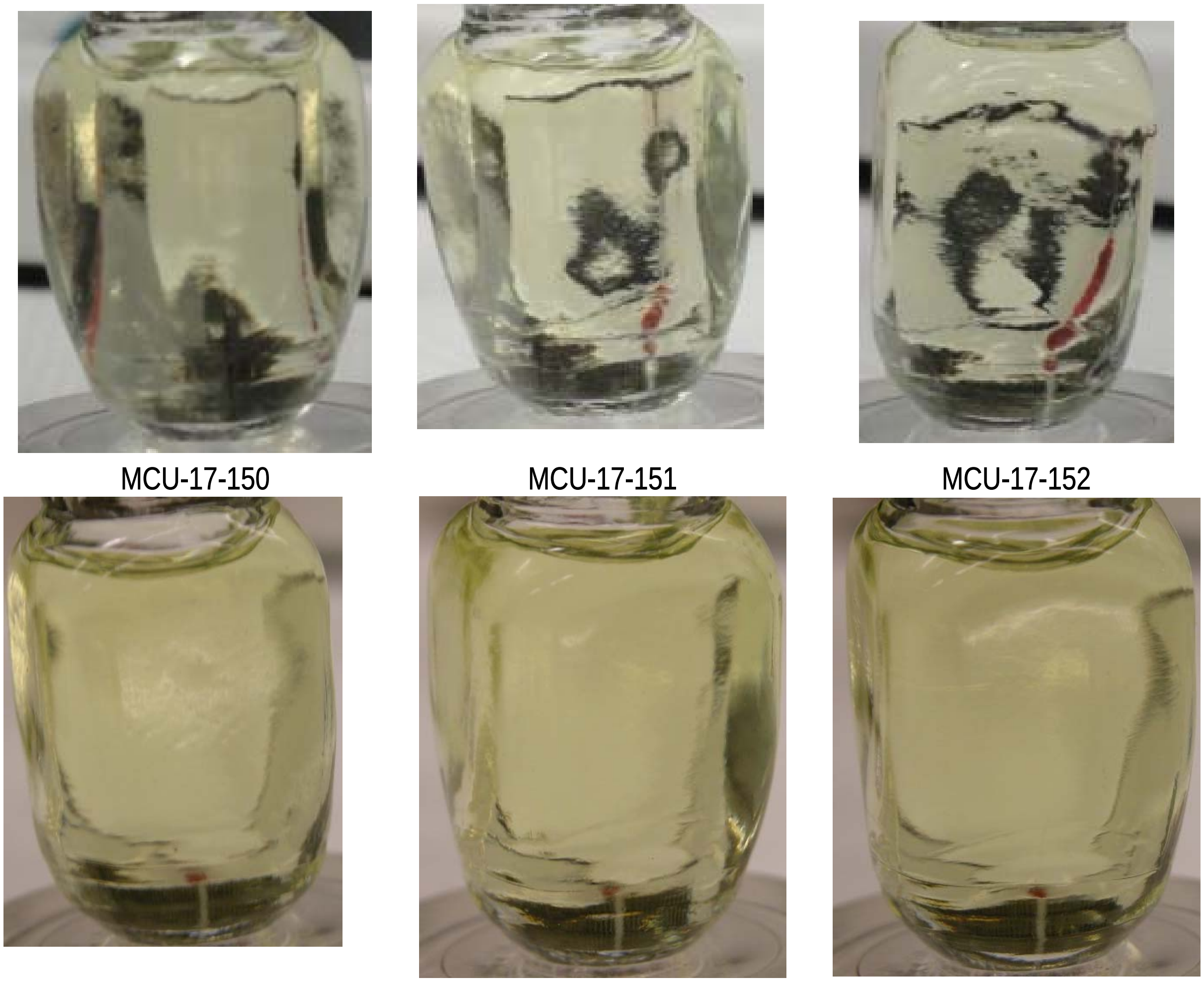
3.0 Results and Discussion

Each sample (and its corresponding p-nut vial) was visually examined. No immiscible phases or floating debris or foam or a film on the p-nut vial's walls were observed (see Fig. 1). Recall MCU suspended processing radioactive liquid waste January 10, 2017 and SHT samples collected since then should have no processing or contacted liquid waste history (for example minimal aqueous liquid carry over or minimal leached or evaporated components). 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 for each set of monthly SHT samples.

Modifier Levels and Density Measurements

Since MCU stopped processing radioactive liquid waste in January 2017, the chemical composition of the SHT is not expected to change with time (leaching, evaporation, chemical decomposition and/or reaction, and radiation damage rates are expected to be minimal). Based on the August sample, triplicate density measurements (by gravimetric and vibrating a filled tube methods) indicated the SHT sample density is the same as the baseline solvent at 25 °C, when corrected for temperature using the Caustic-Side Solvent Extraction (CSSX) temperature correction formula[7] (see Fig. 2). Furthermore, the measurement error intervals include the baseline solvent density value (the calculated density for the baseline solvent is 0.830 g/mL at 25 °C).[4]

The Isopar™L concentration (based on the August SHT sample analysis) is at its nominal level. Very slow evaporation is expected when the solvent is under stagnant conditions. The calculated modifier level is 2% below the recommended modifier level (1.69E05 mg/L) based on the August SHT sample analysis but the noise range in the data includes the recommended level (see Fig. 2). The recommended modifier level (1.69E05 mg/L) in Fig. 2 is well within the error intervals of the two MCU samples reported in this report. Both the density data and the modifier concentration correlate (excluding the March SHT sample result that appears to have an offset) with each other as expected; the solvent density is a concentration-weighed linear combination of the modifier and Isopar™L pure densities.[7] Other physical measurements of the August SHT samples such as viscosity (3.4 ± 0.1 cP) and surface tension (23 ± 3 dyn/cm) were similar to the baseline solvent.



MCU-17-150
MCU-17-151
MCU-17-152
MCU-17-153
MCU-17-154
MCU-17-155

Figure 1 A picture of sample MCU-17-150-151-152 (top row) and MCU-17-153-154-155 (bottom row).

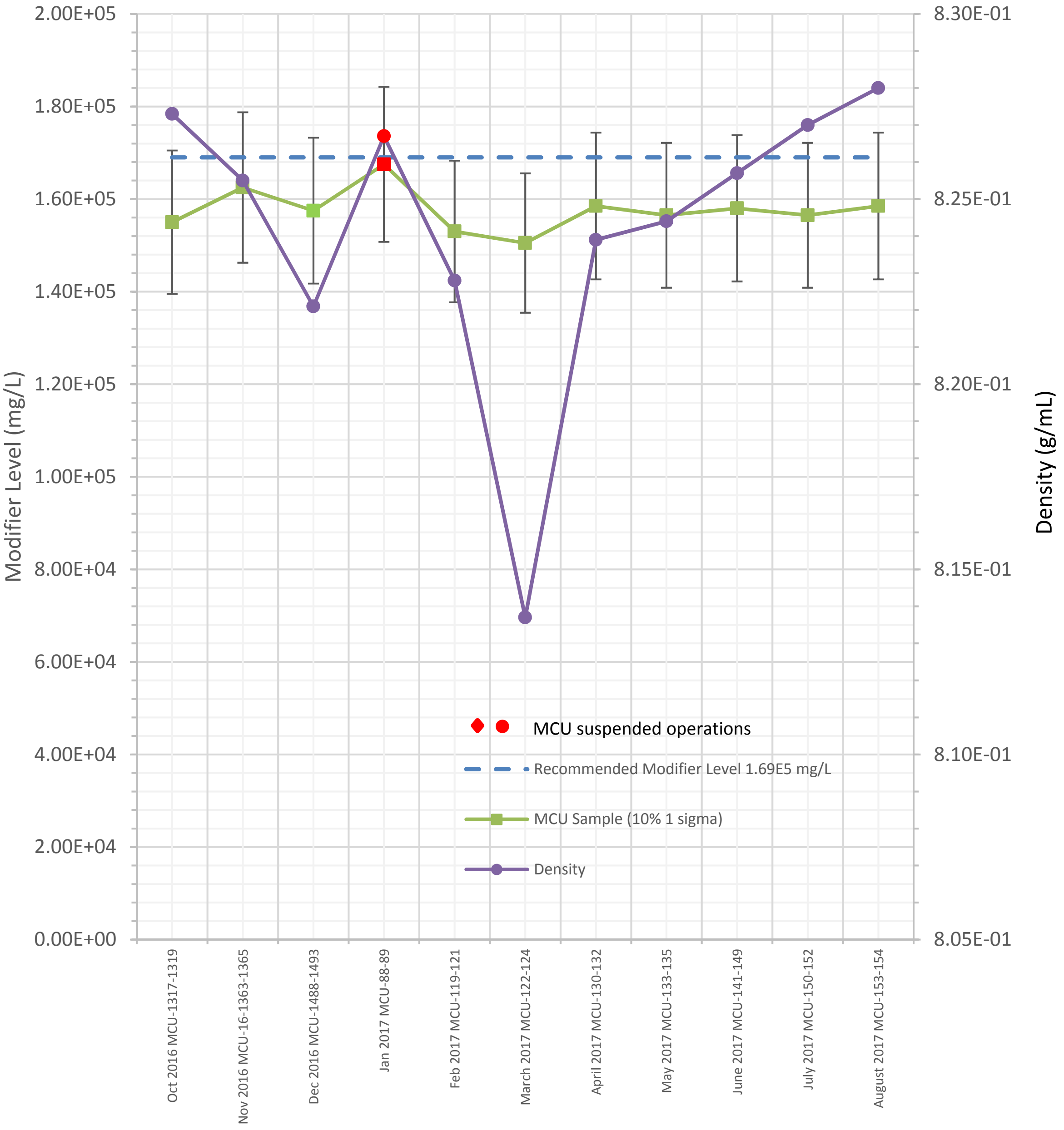


Figure 2. Modifier level in the solvent as measured by HPLC (one sigma is 10%).

Suppressors Levels

The average TiDG concentration levels for MCU-17-150-152 and MCU-17-153-155 are shown in Figure 3. As can be seen in Fig. 3, the TiDG level has remained steady between 666 and 715 mg/L since MCU suspended operations in January 10, 2017. Part of that trend is essentially "flat" after MCU stopped processing any aqueous solution. Thus, the "flat" trend reflects the TiDG degradation under low radiolysis and mild caustic conditions alone (without mass transfer losses to any aqueous solutions or thermal degradation). Since the solvent has been reading approximately 2E4 dpm/mL gamma since the February sample, it appears that there is insufficient total gamma energy (and/or dose rate) to cause any detectable systematic change in the TiDG concentration. Based on the August SHT sample, the suppressor concentration (715 ± 72mg/L) is above its minimum recommended operating level (479 mg/L), but below the nominal level. The TOA concentration appears to remain steady and it is currently (based on the August sample) at 179 ± 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 slower than expected degradation rate (or slower transfer rate to 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).[8] The primary amine degradation products would likely have a similar pKa to the TOA (or to the TiDG) making the equivalent points coincide[9], thereby indicating a higher concentration of TOA remaining in the solvent than is actually present.

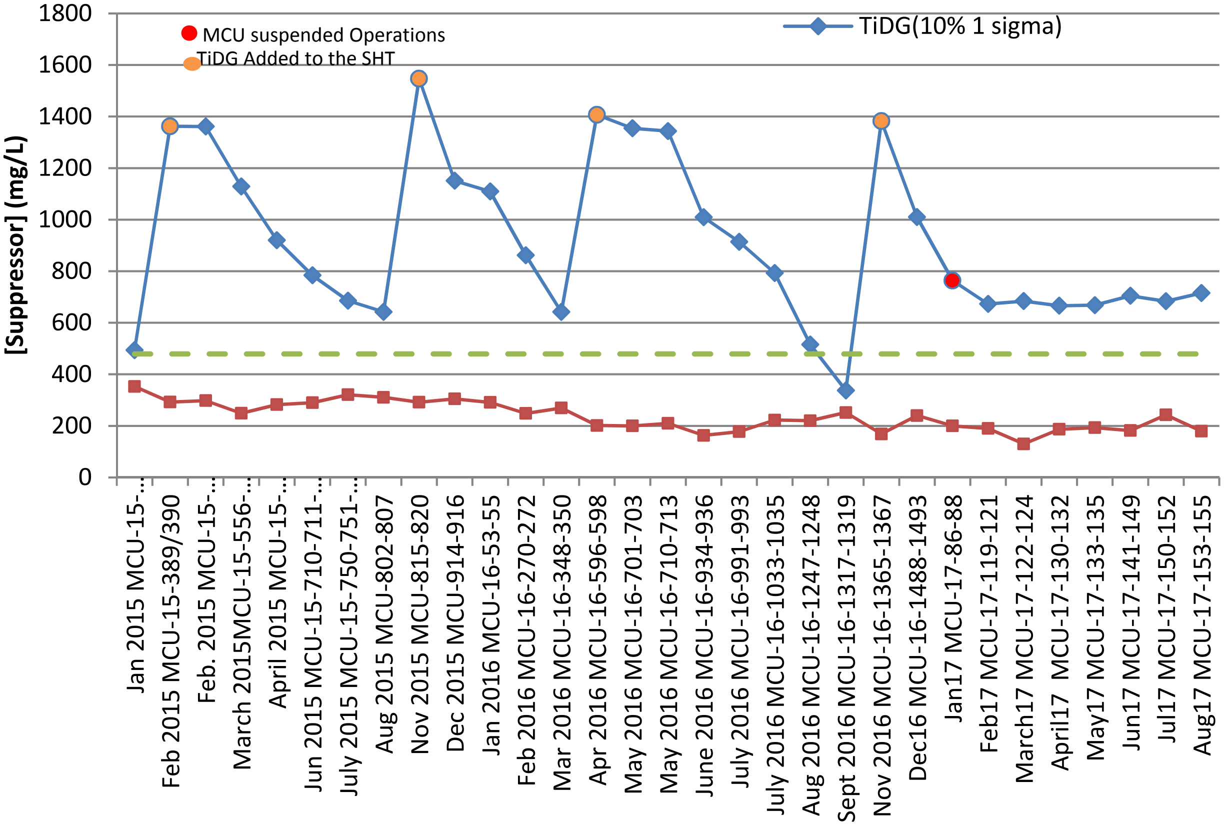


Figure 3. Suppressor concentration as measured by titration in the SHT samples since NGS implementation. The minimum recommended level is 479 mg/L for TiDG.

Extractant Levels

The calculated MaxCalix levels ranged from 4.57E4 to 4.65E4 mg/L in the last two SHT samples. That level of MaxCalix has been previously observed (see Fig. 4). Their error intervals included the recommended value (4.63E4 mg/L). Note the current recommended value is the difference between 47,800 mg/L (50 mM MaxCalix) and the current BOBCalixC6 concentration in the SHT (1.51E3 mg/L in the August sample). The "flat" trend in the MaxCalix level began after MCU stopped operations in January

2017. The recent variations in the MaxCalix concentration seen in Fig. 4 (a concentration spike in the January SHT sample) is within the uncertainty range for this measurement despite the addition of MaxCalix to the solvent on December 14, 2016. The uncertainty is possibly due to the aggregate of analytical, sampling, and process variances.

The residual concentration of BOBCalixC6 level is (based on the August sample) at 37% of the level measured when the NGS was implemented in late FY13 (the concentration variability is due to analytical fluctuations). This level is approximately the same level 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. Given that no BOBCalixC6 is added to the solvent, the level is expected to decrease with time.

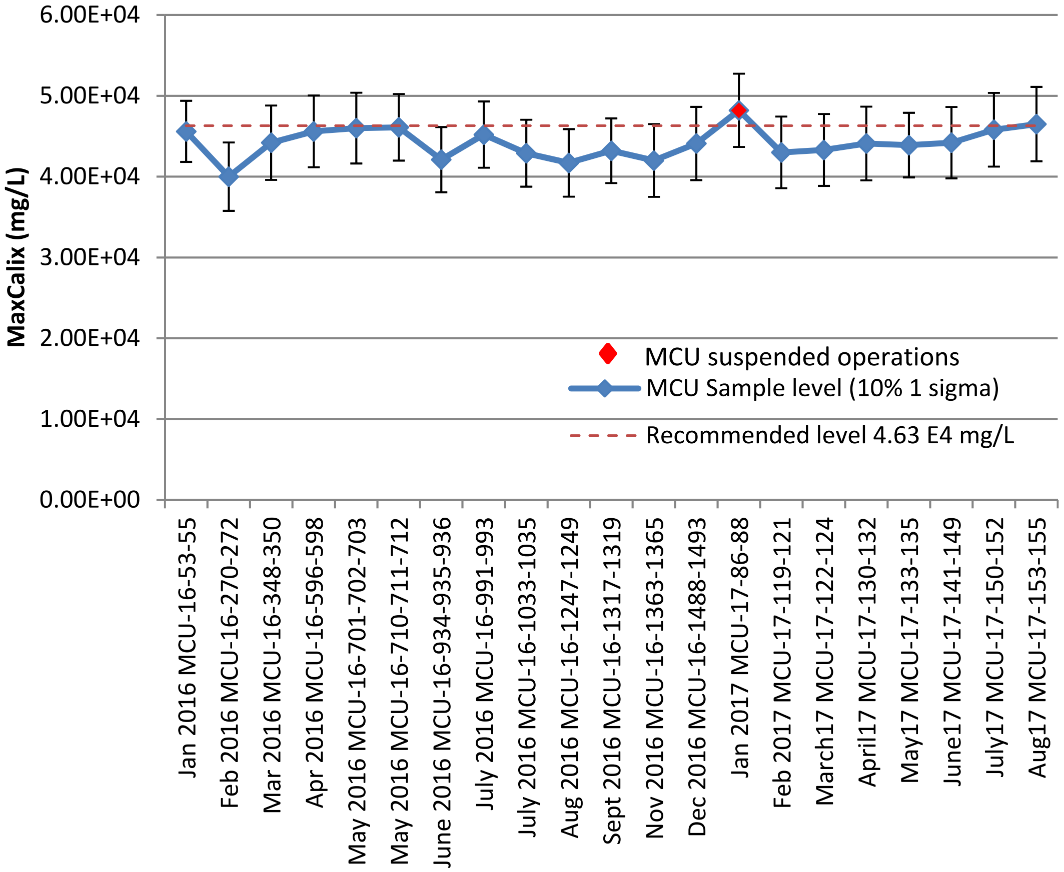


Figure 4. MaxCalix concentration as measured by HPLC and FT-HNMR of recent samples since NGS implementation (46,000 mg/L is the nominal concentration).

Gamma Level

The gamma measurements for the July and August samples are shown in Fig. 5 in relation to past gamma measurements. The gamma measurements for the SHT samples since February 2017 were one order of magnitude lower than the January measurement. This low gamma level has been observed previously but this time is probably due to MCU not processing salt solution since January 2017. It is possible that since the solvent has been sitting idle since January, that any radioactive aqueous solution carried-over has physically separated from the solvent. Thereby, reducing the total emitted energy level of the solvent.

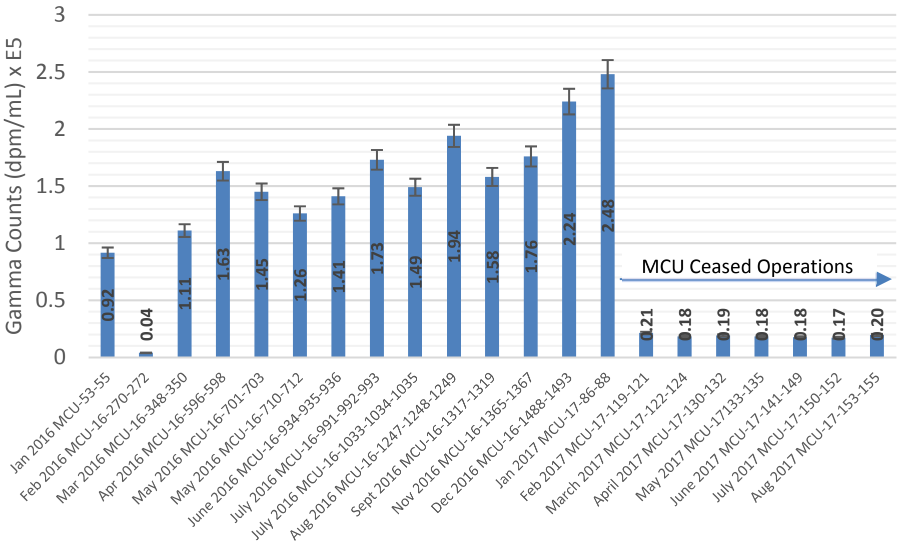


Figure 5. The gamma count of selected SHT samples. One sigma is 5%.

Impurities

A new column was installed in the GC-MS unit and samples from the Scratch May 2017, June, and August SHT samples were re-analyzed for impurities. No impurities were observed in the control sample (Scratch May 2017). However, two unknown impurities at the 290 and 110 mg/L level probably related to the modifier were observed in the June and August samples after a second re-run was conducted with a new column. No sec-butyl phenol was observed in the HPLC method. No impurities were observed in the HNMR spectrum and this indicates that further analysis is needed to confirm these impurities, especially if they are observed again in the next SHT sample.

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 July and August 2017 SHT samples were 20 ± 4 mg/L and 23 ± 5 mg/L respectively (based on the average of the CV-AA and XRF measurements). These levels are consistently lower than the levels observed on December 2016. The level of mercury observed in the July and August samples are significantly higher than the solubility of metallic Hg in dodecane (~3 ppm)[10], 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.[11] Based on the August SHT sample, for 200 gallons of solvent (757.1 L), the solvent could contain up to 17 ± 3 g of mercury (based on the August 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. 6). This may be consistent with the higher levels of total mercury (~109 ppm) observed in Tank 50H in the third and fourth quarters surveillance samples.[12] Please note all the CV-AA data since November 2015 were renormalized and compensated for solvent density variation in this report. Thus, these values differ (slightly lower values) from previous reports. The downward trend observed in Fig. 6 since the January 2017 SHT sample might be because the SHT solvent has not contacted any radioactive aqueous waste since January 10, 2017.

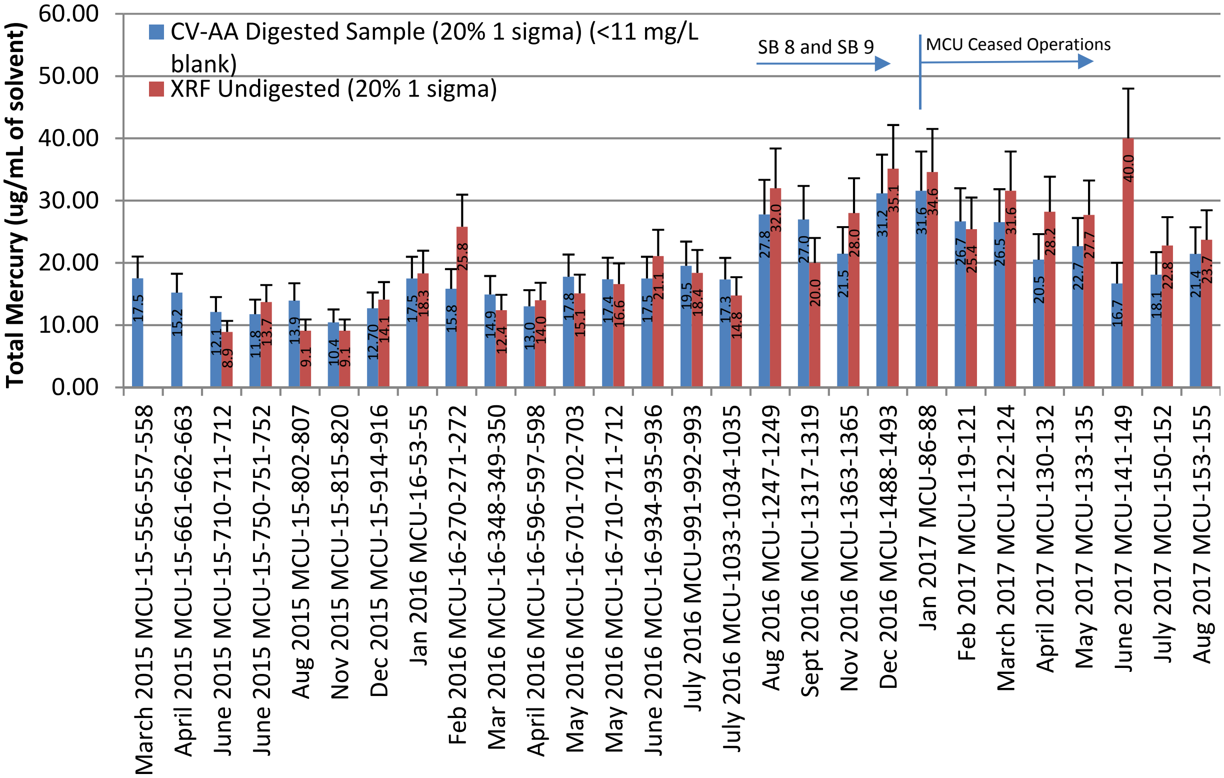


Figure 6. Total mercury in recent SHT samples. One sigma is 20%. CVAA = Cold Vapor Atomic Absorption Spectrometry. XRF =X-Ray Fluorescence (20% one sigma).

Recommendations

The August SHT sample analysis indicates the solvent was at 98% of its nominal modifier level, 50% of its suppressor (TiDG) nominal level, and at a nominal level of Isopar™L (100%) relative to the standard. The MaxCalix concentration was at its nominal level. The TiDG, MaxCalix, modifier, and Isopar™L amounts are expected to trend downward with time but at different rates. Based on the August sample, the solvent did not require any further trim addition. Two unknown impurities related to the modifier were observed in the August SHT sample that were not attributed to sec-butyl phenol in the GC-MS method. If the next SHT sample shows the type of impurity at the same level as in the August SHT sample. Further, analysis could be required to identify them.

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 that includes the last two SHT monthly samples is shown; MCU-17-150-152 (July SHT) and MCU-17-153-155 (August SHT). Since the last SHT sample sent for analysis was the August sample the chemical state of the solvent is best approximated by the chemical analysis of the August SHT sample (MCU-17-153-155). This report mainly focused on the chemical analysis of the August SHT sample. The analysis data from the July SHT sample are presented in the "trend" plots of this report. Analysis of the August SHT sample (MCU-17-153-155) indicated that the modifier (CS-7SB) was 2% below but the extractant (MaxCalix) concentration was at its nominal recommended level (169,000 mg/L and 46,400 mg/L respectively). The suppressor (TiDG) level has decreased since the last measurement taken while Modular Caustic-Side Solvent Extraction unit (MCU) was operating in January 2017, but has remained steady in the range of 666 (observed in April) to 715 mg/L (observed in the August 2017 sample) since February 2017, well above the minimum recommended level (479 mg/L), but below the nominal level. The "flat" trends observed in the TiDG, MaxCalix, modifier, and Gamma measurement are consistent with the solvent being idle since January 10, 2017. A strong correlation between density and modifier concentration in the solvent continues to be observed in the SHT samples.

This analysis confirms the Isopar™L addition to the solvent in January 2017. This analysis also indicates the solvent did not require further additions. Based on the current monthly sample, the levels of TiDG, Isopar™L, MaxCalix, and modifier are sufficient for continuing operation but are expected to decrease with time if MCU returns to processing radioactive liquid waste. Otherwise, the levels of these components will remain steady. A future Isopar™L trimming addition to the solvent is recommended when MCU resumes processing waste.

Two unknown impurities related to the modifier (but not sec-butyl phenol: a modifier degradation product observed before) at the 290 and 110 mg/L levels were observed in the August SHT sample by the GC-MS method. They were observed in a second GC-MS re-run with a new column. Their identification can't be ascertained at this time. No impurities were observed in the HNMR. Another impurity observed in the samples was mercury. Based on the August SHT sample, up to 23 ± 5 micrograms of mercury per mL of solvent was detected (the average of the CV-AA and XRF methods). The higher mercury concentration in the solvent (as determined in the last three-monthly samples) is possibly due to either a higher mercury concentration in Salt Batches 8 and 9 (Tank 49H).

The gamma level (~ 2.0E4 dpm/mL) measured in the August SHT samples was one order of magnitude lower than the gamma levels observed in the December 2016 and January 2017 SHT samples. A similar level was observed in the July SHT sample (MCU-SHT-150-152). The gamma level has remained consistently steady since January 10, 2017 when MCU stopped processing radioactive liquid waste.

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

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