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Solvent Hold Tank Sample Results for MCU-16-1363-1364-1365: November 2016 Monthly Sample

F. F. Fondeur

D. H. Jones

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REVIEWS AND APPROVALS

AUTHORS:

F. F. Fondeur, Advanced Characterization and Processing Date

D. H. Jones, Research Support Date

TECHNICAL REVIEW:

T. B. Peters, Advanced Characterization and Processing Date
Reviewed per Manual E7 Procedure 2.60

APPROVAL:

B. J. Wiedenman, Manager Date
Advanced Characterization and Processing

D. E. Dooley, Director Date
E&CPT Research Programs

E. A. Brass, Manager Date
MCU & Salt/Sludge Processing

EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) received one set of three Solvent Hold Tank (SHT) samples (MCU-16-1363-1364-1365), pulled on 11/15/2016 for analysis. The samples were combined and analyzed for composition. Analysis of the composite sample MCU-16-1363-1364-1365 indicated the Isopar™L concentration is at its nominal level (100%). The extractant (MaxCalix) and the modifier (CS-7SB) are 8% and 2 % below their nominal concentrations. The suppressor (TiDG) is 7% below its nominal concentration. A summary of the concentration of the relevant solvent components is shown below.

Sample	Sampling Date	Density at 25°C (g/mL)	Isopar™L (mg/L)	Modifier (mg/L)	MaxCalix (mg/L)	TiDG (mg/L)
MCU-16-1363-1365	11/15/2016	0.826	6.09 E5	1.66 E5	4.23 E4	1.34 E3
Scratch Blend at 25°C	Not Applicable	0.830	6.10 E5	1.69 E5	4.43 E4	1.54 E3

This analysis confirms the trim and Isopar™ additions to the solvent in November. 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. Periodic characterization and trimming additions to the solvent are recommended.

No impurities above the 1000 ppm level were found in this solvent by the Semi-Volatile Organic Analysis (SVOA). No impurities were observed in the Hydrogen Nuclear Magnetic Resonance (HNMR). However, there is evidence of possible chemical modification of the modifier with some missing a hydrogen atom at the tert-butyl group of the modifier. Another impurity observed in the samples was mercury. Up to 27 ± 5.4 micrograms of mercury per gram of solvent (or 22.3 µg/mL) was detected in this sample (the average of the CV-AA and XRF methods).

The current gamma level (1.76E5 dpm/mL) confirmed that the gamma concentration has returned to previous levels (as observed in the late 2015 samples) where the process operated normally and as expected.

The laboratory will continue to monitor the quality of the solvent in particular 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
FTIR	Fourier transform infra-red spectroscopy
HNMR	Hydrogen Nuclear Magnetic Resonance
HPLC	High Performance Liquid Chromatography
ISDP	Integrated Salt Disposition Project, NGS
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 Modular Caustic-Side Solvent Extraction Unit (MCU) switched to the Next Generation Solvent (NGS) flow sheet. Facility personnel implemented the switch by adding a non-radioactive, NGS "cocktail" containing the new extractant (MaxCalix) and a new suppressor (TiDG) to the SHT heel. The resulting "blend" solvent ("NGS Blend solvent") is essentially NGS with residual amounts of BOBCalixC6 and trioctylamine (TOA). SHT samples are sent to SRNL to examine solvent composition changes over time.[1] On November 15, 2016 Operations personnel pulled and delivered three samples from the SHT (MCU-16-1363, MCU-16-1364, and MCU-16-1365) for analysis. 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 at the same time to generate a solution of the appropriate composition that approximates the blend of cocktail[2] and heel solvent) was prepared in the lab (July 2016) and used for comparison and evaluation. 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. On November 8, 2016, a trim addition was made to MCU. [3]

Table 2-1 Log of recent trims to the MCU solvent and sample arrivals to SRNL

Event	Date
SHT sample MCU-16-53-54-55	January 25, 2016
SHT sample MCU-16-270-271-272	February 21, 2016
12 gallons of Isopar™L added to MCU	March 6, 2016
SHT sample MCU-16-348-349-350	March 30, 2016
10 gallons of Isopar™L added to MCU	March 31, 2016
April Solvent Trim added to MCU	April 29, 2016
SHT sample MCU-16-596-597-598	April 30, 2016
SHT sample MCU-16-701-702-703	May 23, 2016
SHT sample MCU-16-710-711-712 (washed with 300 mM caustic)	May 28, 2016
20 gallons solvent trim added to MCU	June 15, 2016
SHT sample MCU-16-1363-1364-1365	June 30, 2016
SHT sample MCU-16-991-992-993	July 23, 2016
SHT sample MCU-16-1033-1034-1035 (washed with 300 mM caustic)	July 28, 2016
20 gallons of Isopar™L added to MCU	August 21 , 2016
SHT sample MCU-16-1247-1248-1249	August 22, 2016
SHT sample MCU-16-1317-1318-1319	September 12, 2016
20 gallons solvent trim added to MCU	November 8, 2016
11 gallon Isopar™L added to MCU	November 13, 2016
SHT sample MCU-16-1363-1364-1365	November 15, 2016

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. MCU-16-1363, MCU-16-1364, and MCU-16-1365 were composited before use. Aliquots of the composited sample were removed to perform the following analysis: Density, SVOA, high performance liquid chromatography (HPLC), titration, gamma counting, CVAA, X-ray fluorescence (XRF), and Fourier-Transformed HNMR

(FT-HNMR). Results from analytical measurements were compared with the theoretical values shown in Table 2-2.

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

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	~1440	~ 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-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 (MCU-16-1363, MCU-16-1364, and MCU-16-1365) 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 (MCU-16-1363-1364-1365). Table 3-1 contains the results for the MCU-16-1363-1364-1365 composite sample.

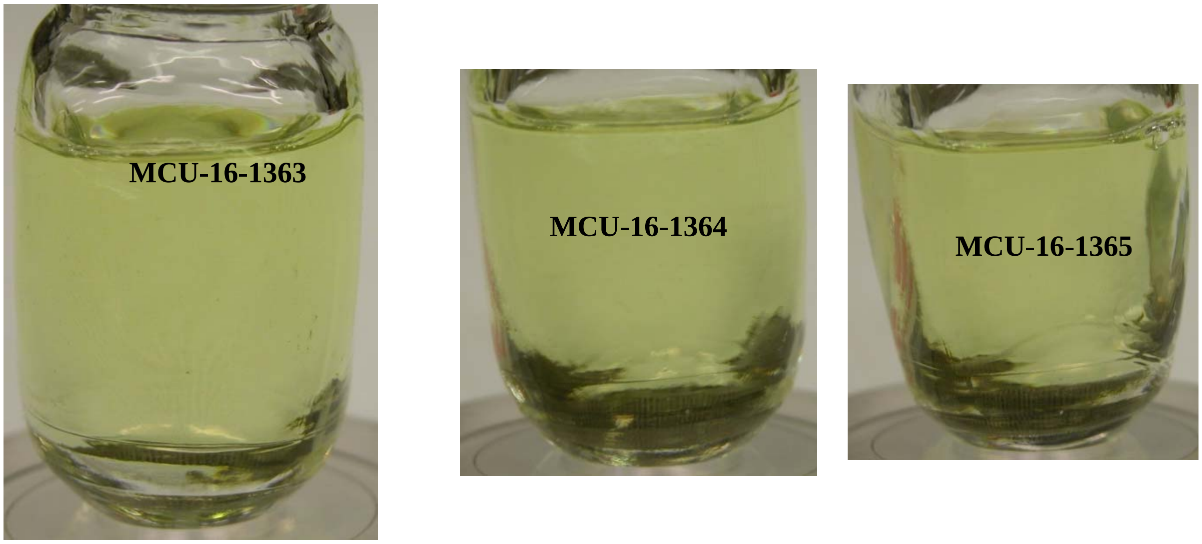

Figure 1. Typical appearance of the three MCU-16-1363, MCU-16-1364, and MCU-16-1365

Isopar™ L and Modifier Levels

Triplicate density measurements (by gravimetric and vibrating a filled tube method) of the sample gave an arithmetic average result of 0.831 g/mL (0.06% RSD) (or 0.826 g/mL at 25 °C when corrected for temperature using the CSSX temperature correction formula)[4] for MCU-16-1363-1364-1365 at 18.5°C. The calculated density (0.826 g/mL) for MCU-16-1363-1364-1365 is about 1% below the calculated density for the standard sample (0.830 g/mL at 25 °C for the scratch blend made in the laboratory).[2,4] Using the density as a starting point, we know that the concentration level of the Isopar™L component in the sample should be at (or slightly above) its nominal value (within analytical uncertainties) and the modifier concentration should be slightly below its nominal value.

An examination of Table 3-1 shows that the Isopar™L concentration is at its nominal value while the modifier concentration is correspondingly slightly lower (2% lower) than its nominal value. The Isopar™L concentration (at its nominal level) is consistent with the Isopar™L addition to the solvent on November 13. Of all the methods listed, density has the lowest uncertainty. Thus, the final reported values are closer to the density measurement. The last solvent trim addition to MCU was on November 8, 2016.

All measurements indicate the Isopar™L level is at its nominal value while the modifier concentration level is slightly below its nominal value (see Fig. 2 for recent modifier concentrations from HPLC measurements). Looking at Fig. 2, the modifier level appears to return to its nominal level observed right after the trim addition to the solvent on November 8, 2016. The trend in the modifier level correlates with the trend in the density measurements as expected (see Fig. 2).[4] The accuracies of the different measurements were within expectation as reflected in the total mass sum of the "average" results listed in Table 3-1. They added up to 0.821 ± 0.018 g/mL. Their sum is consistent with the measured and temperature corrected (to 25 ºC) value of 0.826 g/mL, and also with the measured and corrected to 25 °C mass concentration (density) of the standard (0.830 g/mL). The current modifier concentration is well above the minimum modifier concentration below which the extractant concentration may drop due to solubility limits. A HNMR of the modifier revealed a possible chemical modification of approximately 5% of the modifier. Specifically, there are 5% less hydrogen in the *sec*-butyl group (R_2CH-R) of the modifier than hydrogen in the terminal CF_2H. The area ratio from these two groups in the H-NMR spectrum measured 0.95 when it should be one (previous last two monthly samples had area ratio of 1.01 respectively).

Table 3-1 Sample Results for MCU-16-1363-1364-1365

Analysis	Method	LW-AD-Proj-160412-4 (Analytical Lab. Labeling)	Result (mg/L)[#]	Nominal[*] Result (mg/L)	% of (Result ÷ Nominal Result)
Isopar™L	FT-HNMR	NA	6.14E+05	6.10E+05	101
Isopar™L	FTIR	NA	6.22E+05		102
Isopar™L	Density	NA	6.08E+05		100
Average[$]	All	NA	6.09E+05	6.10E+05	100
Modifier	HPLC	LW4682	1.67E+05	1.69E+05	99
Modifier	FT-HNMR	NA	1.58E+05		94
Modifier	FTIR	NA	1.58E+05		94
Modifier	Density	NA	1.67E+05		99
Average[$]	All	NA	1.66E+05	1.69E+05	98
TiDG	Titration-SVOA[♣]	LW4681	1.20E+03	1.44E+03	83
TiDG[♠]	Titration	NA	1.38E+03	1.44E+03	96
Average[$]	All	NA	1.34E+03	1.44E+03	93
trioctylamine	SVOA	LW4681	3.10E+02	-	58[χ]
trioctylamine	Titration	NA	1.72E+02	-	32[χ]
Average[$]	All	NA	1.94E+02	-	37[χ]
MaxCalix	HPLC	LW4682	4.20E+04	4.60E+04[Δ]	91
MaxCalix	FT-HNMR	NA	4.29E+04		93
Average[$]	All	NA	4.23E+04	4.60E+04[Δ]	92
BOBCalixC6	HPLC	LW4682	1.79E+03	-	44[χ]
Average[$]	All	NA	1.79E+03	-	44[χ]
Density (g/mL)	Direct Measurement	NA	0.826	0.83	99

[#] Analytical uncertainty is 10% for HPLC. Titration method uncertainty is 10% for TiDG and 16% for TOA. Density results from the average of replicate volumetric trials typically have a percentage standard deviation of <3% between each value and the average. NMR analytical uncertainty is 10% for the modifier and 13% for MaxCalix, and 14% for Isopar™ L. N/A = Not Applicable. Density estimations assume the combined weight percent of TiDG, MaxCalix, BOBCalixC6, and TOA to be approximately 6%. All uncertainties are 1 sigma.

[*] Nominal value is the expected value for freshly prepared blended solvent with a target density of 0.8352 g/mL at 25 °C.

[$] Reported value for a MCU component is the weighted average of the values reported by the techniques that measured that component.

$$x = \frac{\sum_1^i \left(x_i / \delta_i^2 \right)}{\sum_1^i \left(1 / \delta_i^2 \right)};$$

x_i stands for the concentration obtained at a given method and δ_i is the corresponding uncertainty.

[♠] No TiDG value was estimated by FT-HNMR due to an aged (questionable) standard.

[♣] Trioctylamine obtained from the SVOA method was subtracted from the measured total base in the titration method to obtain a calculated TiDG concentration.

[χ] Percentages are relative to the initial TOA concentration of 530 mg/L and BOBCalixC6 concentration of 4030 mg/L in 2008.

[Δ] Value shown is the difference between 47,800 mg/L (50 mM MaxCalix) and the current BOBCalixC6 concentration (1,790 mg/L).

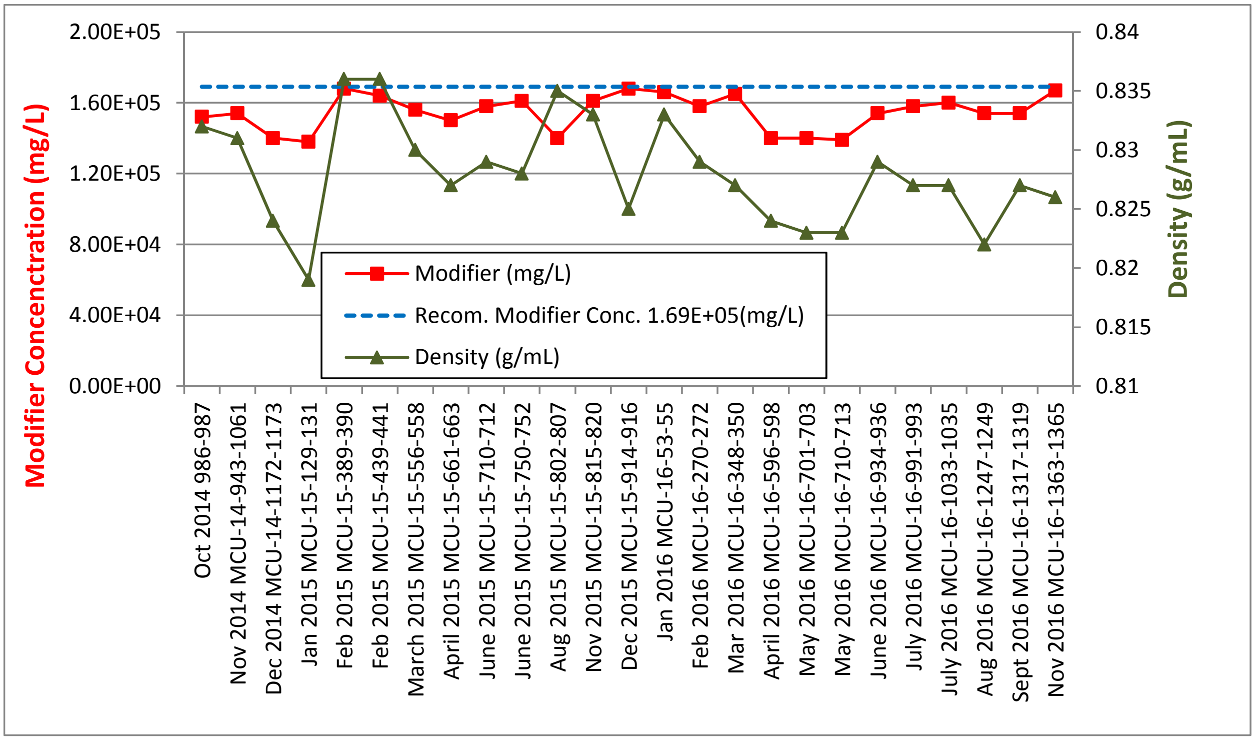

Figure 2. Modifier level in the solvent as measured by HPLC (one sigma is 10%).

Suppressors Levels

The average TiDG concentration level (1.34 ± 0.3 E3 mg/L) is at 93 % of its nominal value of 1440 mg/L confirming the solvent trim addition on November 8, 2016 (a noticeable spike in the TiDG concentration level was observed in Fig. 3 in the November 2016 SHT monthly sample). The suppressor concentration is above its minimum recommended operating level (479 mg/L). The TOA concentration appears to remain steady and it is currently at 194 ± 39 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 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).[5] The primary amine degradation products would likely have a similar pKa to the TOA (tertiary amine) making the equivalent points coincide.[6]

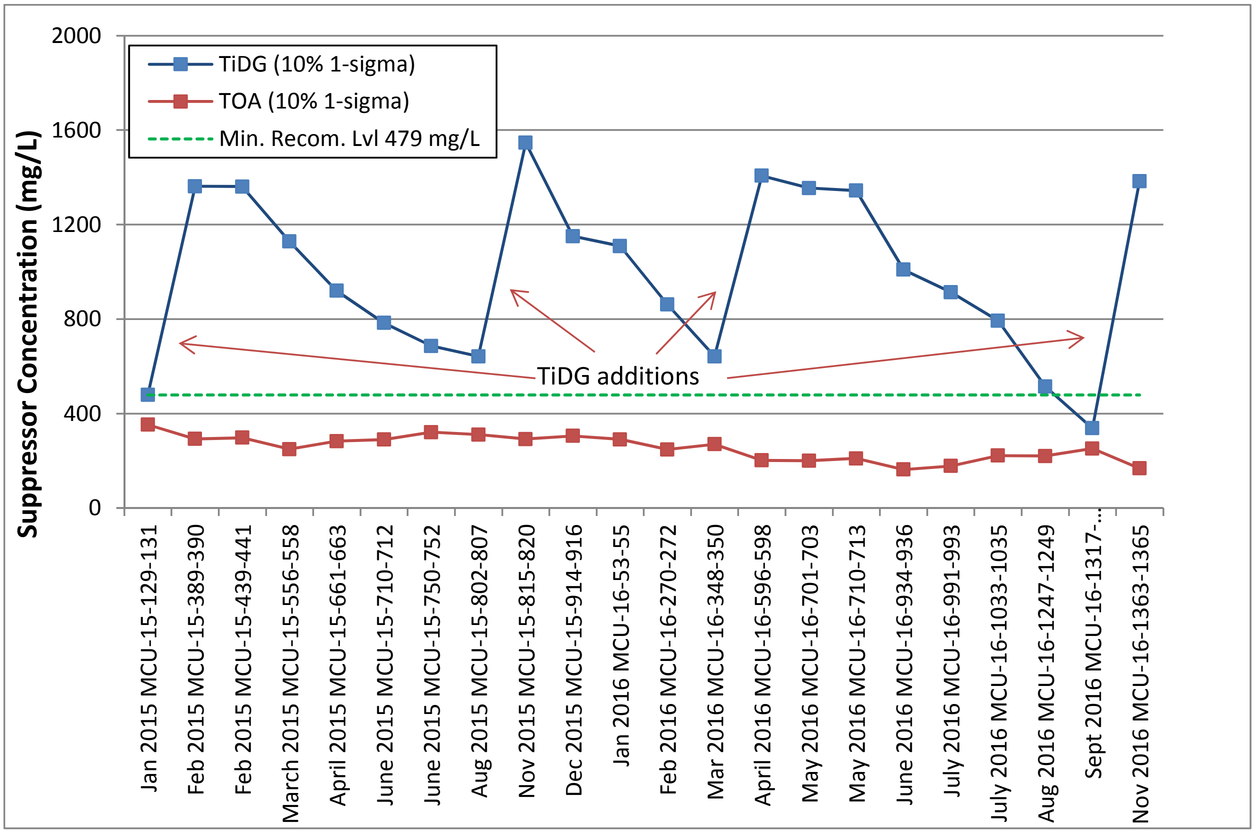

Figure 3. Suppressor concentration as measured by titration in SHT samples since NGS implementation. The minimum recommended level is 479 mg/L for TiDG.

Extractant Levels

The average calculated MaxCalix level is 4.23E4 mg/L (±10%) and it is at 8% below its nominal value. Note the current recommended value is the difference between 47,800 mg/L (50 mM MaxCalix) and the current BOBCalixC6 concentration in the SHT. The recent variation in the MaxCalix concentration seen in Fig. 4 is within the uncertainty range for this measurement. However, the current measurement appears to indicate a downward trend in the MaxCalix concentration possibly due to analytical, sampling or process variances since MaxCalix was added to MCU on November 8, 2016. The current "down and up" trend in the MaxCalix level has been previously observed after a solvent trim addition (see Fig. 4).

The residual concentration of BOBCalixC6 level is currently at 44% 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 the July 2016 sample (2.27E3 mg/L). 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.

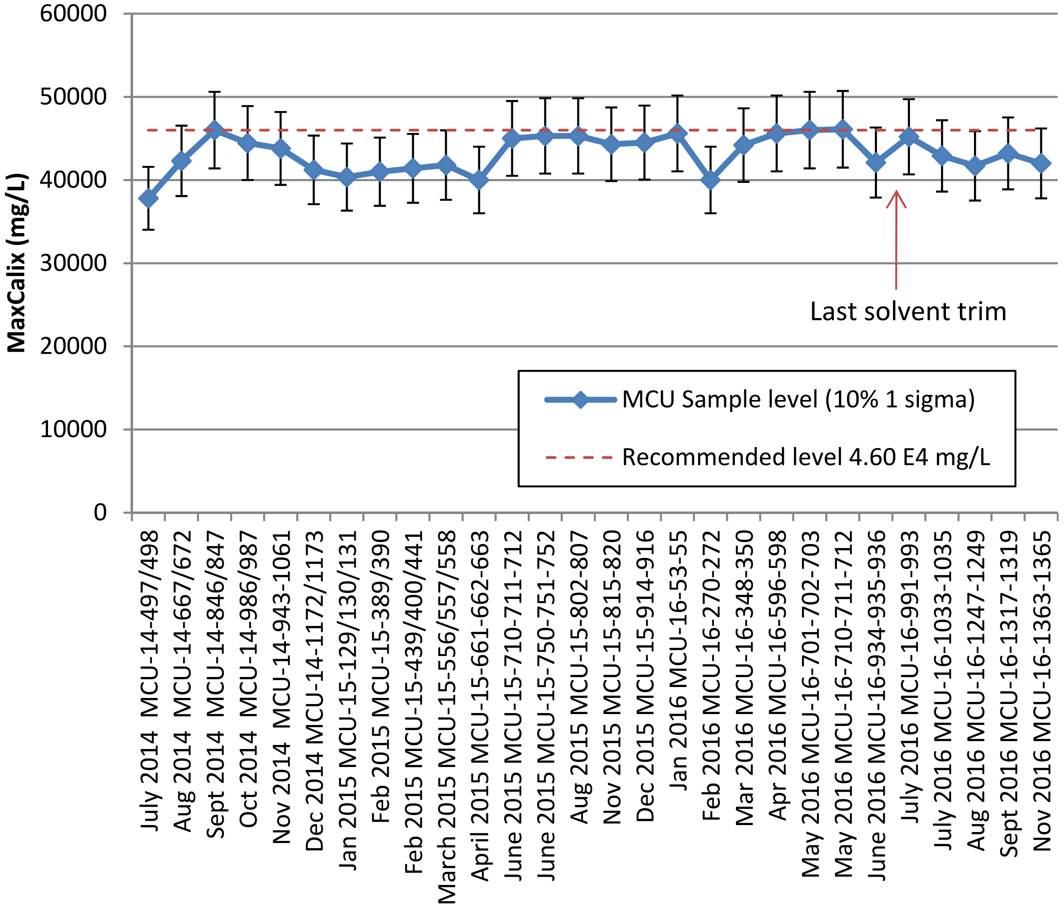

Figure 4. MaxCalix concentration as measured by HPLC and FT-HNMR of recent samples since NGS implementation (46,000 mg/L is the nominal concentration).

Gamma Level

The gamma measurement of MCU-16-1363-1364-1365 is 1.76E5 dpm/mL (±5%) or approximately 1E6 dpm/g $_{\text{modifier}}$ if the solvent is allowed to dry (for example, when the Isopar™L is allowed to evaporate as in the case of solvent misting). This level of activity is consistent with the previous gamma levels when the process was operating normally in late 2015 and in early 2016 (the low Gamma count from the February 2016 sample is unclear at this point). It confirms the steady state trend level observed since March 2016 (see Fig. 5).

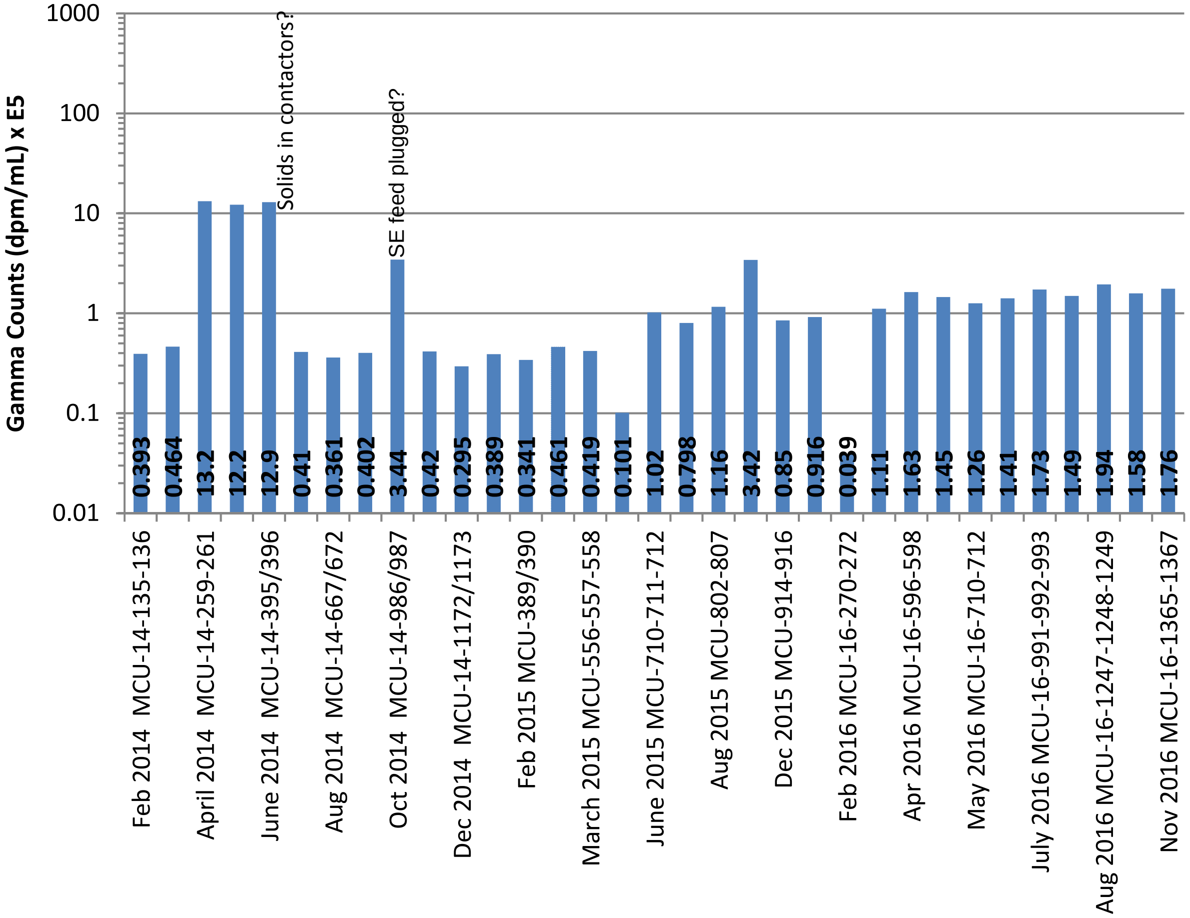

Figure 5. The gamma count of selected SHT samples. One sigma is 5%.

Impurities

No impurities were seen at the 1000 ppm level or higher as indicated by the SVOA method (± 20% uncertainty). No impurities were observed in the HNMR spectrum.

A few milliliters of MCU-16-1363-1364-1365 was digested and analyzed for total mercury by the CVAA method. The CV-AA method detected a total mercury concentration of 26.0 ug/$g_{solvent}$ of mercury. The XRF method also detected a concentration of 28.0 ug/$g_{solvent}$ of mercury of the undigested MCU-16-1363-1364-1365 sample. The average of both methods is 27 ± 5.4 ug/$g_{solvent}$ of mercury (or 22.3 ug/$mL_{solvent}$ at 25°C).

This level of mercury is significantly higher than the solubility of metallic Hg in dodecane (~3 ppm)[7], 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.[8] For 200 gallons of solvent (757.1 L) and assuming a density of 0.826 g/mL, the solvent could contain a total of 16.9 ± 3.4 g of mercury (based on the average of the two measurements). A comparison of this measurement with previous months confirms a positive trend in the mercury concentration in the solvent (data is shown in Fig. 6). Please note all the XRF data since November were renormalized and compensated for solvent density variation in this report. Thus, these values differ (slightly lower values) from previous reports. The positive trend in Fig. 6 might be due to a higher mercury concentration in Salt Batches 8 and 9 (Tank 49H). Also higher levels of total mercury (~109 ppm) were observed in Tank 50H third and fourth quarters surveillance samples.[10]

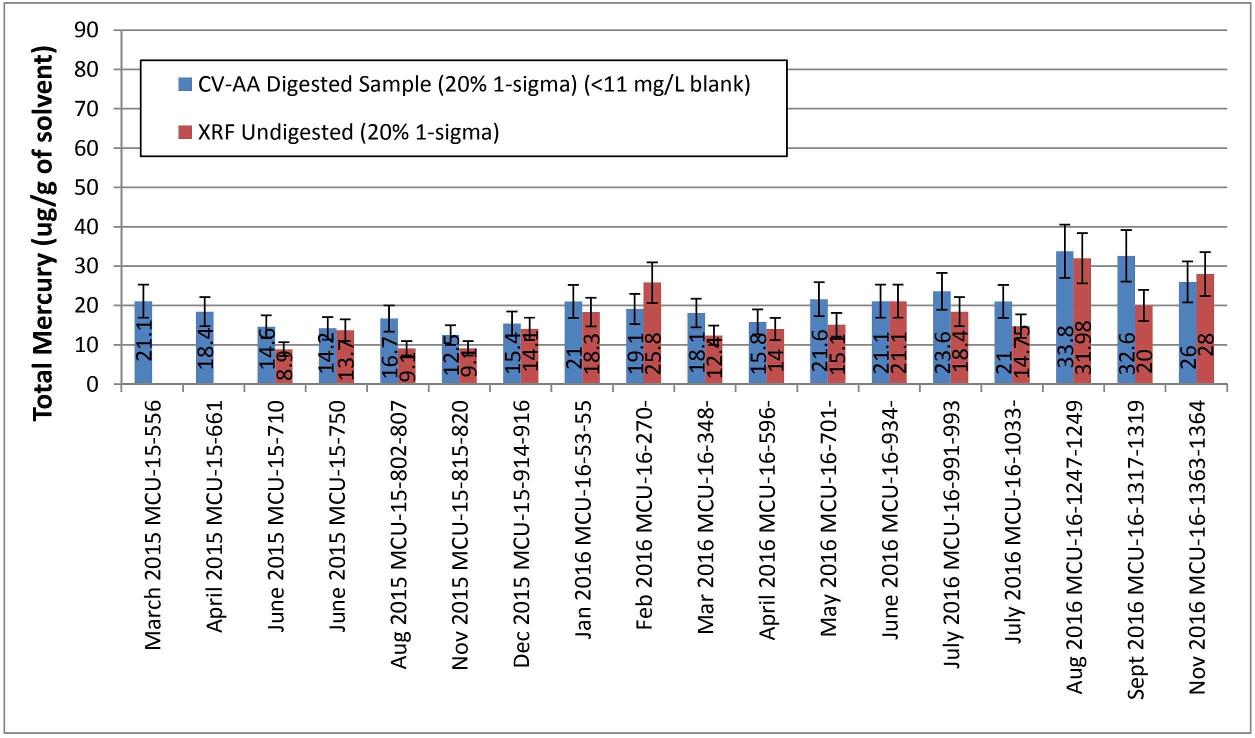

Figure 6. Total mercury in recent SHT samples. One sigma is 20%. CVAA = Cold Vapor Atomic Absorption Spectrometry. XRF =X-ray Fluorescence (20% one sigma).

Recommendations

The current analysis indicates the solvent has 92% of its nominal modifier and 93% of its suppressor (TiDG), but it has a nominal level of Isopar™L (100%) relative to the standard. The MaxCalix concentration is at 92% of 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 November sample, the solvent did not require any further 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

Savannah River National Laboratory (SRNL) received one set of three Solvent Hold Tank (SHT) samples (MCU-16-1363-1364-1365), pulled on 11/15/2016 for analysis. The samples were combined and analyzed for composition. Analysis of the composite sample MCU-16-1363-1364-1365 indicated the Isopar™L concentration is at its nominal level (100%). The extractant (MaxCalix) and the modifier (CS-7SB) are 8% and 2 % below their nominal concentrations. The suppressor (TiDG) is 7% below its nominal concentration.

This analysis confirms the Isopar™ addition to the solvent in November. This analysis also indicates the solvent did not required 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. Periodic characterization and trimming additions to the solvent are recommended. A solvent trim batch, containing TiDG, modifier and MaxCalix, was prepared by SRNL and was added to the SHT on December 14, 2016.

No impurities above the 1000 ppm level were found in this solvent by the SVOA. No impurities were observed in the HNMR. However, there is evidence of possible chemical modification of the modifier with some missing a hydrogen atom at the tert-butyl group of the modifier. Another impurity observed in the samples was mercury. Up to 27 ± 5.4 micrograms of mercury per gram of solvent (or 22.3 µg/mL) was detected in this sample (the average of the CV-AA and XRF methods).

The current gamma level (1.76E5 dpm/mL) confirmed that the gamma concentration has returned to previous levels (as observed in the late 2015 samples) where the process operated normally and as expected.

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

5.0 References

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