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Solvent Hold Tank Sample Results for MCU-16-701-702-703: May 2016 Monthly Sample and MCU-16-710-711-712: May 2016 Superwashed Sample

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D. H. Jones
August 2016
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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) received one set of Solvent Hold Tank (SHT) samples (MCU-16-701, MCU-16-702 and MCU-16-703), pulled on 05/23/2016 and another set of SHT samples (MCU-16-710, MCU-16-711, and MCU-16-712) were pulled on 05/28/2016 after the solvent was superwashed with 300 mM sodium hydroxide for analysis. Samples MCU-16-701, MCU-16-702, and MCU-16-703 were combined into one sample (MCU-16-701-702-703) and samples MCU-16-710, MCU-16-711, and MCU-16-712 were combined into one sample (MCU-16-710-711-712). Of the two composite samples MCU-16-710-711-712 represents the current chemical state of the solvent at MCU. All analytical conclusions are based on the chemical analysis of MCU-16-710-711-712. There were no chemical differences between MCU-16-701-702-703 and superwashed MCU-16-710-711-712. Analysis of the composited sample MCU-16-710-712-713 indicated the Isopar™L concentration is above its nominal level (102%). The modifier (CS-7SB) is 16% below its nominal concentration while the TiDG and MaxCalix concentrations are at and above their nominal concentrations respectively. The TiDG level has begun to decrease and it is 7% below its nominal level as of May 28, 2016. Based on this current analysis, 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 the solvent samples by the SVOA. No impurities were observed in the HNMR. One small particle of sec-butyl phenol was observed floating in MCU-16-712 possibly dislodged from the solvent by the superwashing process (an indication this process is working). Also, up to 21.1 ± 4.2 ug/$g_{solvent}$ (or 17 ug/$mL_{solvent}$) of mercury was detected in this sample (as determined by the CV-AA method). XRF analysis of undigested MCU-10-710-711-712 sample detected 16.6 ± 3.3 ug/$g_{solvent}$ of mercury. The higher mercury concentration in the solvent (as determined in the last four monthly samples) is possibly due to the higher mercury concentration in Salt Batch 8 (Tank 49H).

The current gamma level (1.26E5 dpm/mL (±5%)) confirmed that the gamma concentration is consistent with previous level where the process operated normally and as expected.

The similar component concentrations between the two composite samples (MCU-16-701-702-703 and MCU-16-710-711-12) shows that the superwashing step (cleaning with 300 mM sodium hydroxide solution) did not affect the main components of the MCU solvent.

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

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

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
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
Sigma	One standard deviation (sample deviation)
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 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 May 23, 2016, Operations personnel pulled and delivered three samples from the SHT (MCU-16-701, MCU-16-702, and MCU-16-703) for analysis. Then MCU personnel proceeded to wash (super wash) the solvent with a 300 mM sodium hydroxide solution. After washing the solvent, another three samples were pulled and delivered from the SHT (MCU-16-710, MCU-16-711, and MCU-16-712) to determine the effect of washing on solvent composition. These samples are also 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 (September 2015) 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 April 29, 2016, a trim addition was made to MCU that included 9.9 lbs of MaxCalix, 7.9 lbs of modifier and 2.0 lbs of TiDG in 143.4 lbs of Isopar™L.

Table 2-1 Log of recent trims to the MCU solvent and sample pull-out dates

Event	Date
February solvent trim added to MCU	February 22, 2015
SHT sample MCU-15-389-390	February 25, 2015
SHT sample MCU-15-439-440-441	February 28, 2015
10 gallons of Isopar™L added to MCU	March 6, 2015
9 gallons of Isopar™L added to MCU	March 13, 2015
SHT sample MCU-15-556-557-558	March 16, 2015
SHT sample MCU-15-661-662-663	April 2, 2015
10 gallons of Isopar™L added to MCU	May 6, 2015
SHT sample MCU-15-710-711-712	June 15, 2015
SHT sample MCU-15-750-751-752	June 22, 2015
SHT sample MCU-15-802-803-804-805-806-807	August 31, 2015
November solvent trim added to MCU	November 28, 2015
SHT sample MCU-15-815-816-817-818-819-820	November 29, 2015
14 gallons of Isopar™L added to MCU	December 21, 2015
SHT sample MCU-15-914-915-916	December 22, 2015
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

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-16-701, MCU-16-702, and MCU-16-703 were composited to make one sample (MCU-16-701-702-703) before use. Similarly, MCU-16-710, MCU-16-711, and MCU-16-712 were composited to make sample MCU-16-710-711-712. 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.

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

Component	mg/L	Molar
MaxCalix	~ 44,400	~ 0.0465
BOBCalixC6*	< 4,030	< 0.0035
TOA*	< 530	< 0.0015
Modifier	~ 169,000	~ 0.50
TiDG	~1440	~ 0.003
Isopar™L	~ 623,000	~ 74 wt%

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

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

Visual Inspection of the P-nut Vials

Each sample (MCU-16-701, MCU-16-702, MCU-16-703, MCU-16-710, MCU-16-711, and MCU-16-712) was visually examined. All samples were visibly translucent and clear. A 0.5 mm x 0.5 mm particle was observed in MCU-16-712 and it appears to be sec-butyl phenol with some nitrile rubber (see Fig. 2). All samples had a pH value of 5.5. No unusual reactions, solids, foaming, or immiscible layers were observed after combining the respective samples into their composites (MCU-16-701-702-703 and MCU-16-710-711-712). Table 3-1 contains the results for the MCU-16-701-702-703 composite sample and Table 3-2 contains the results for the MCU-16-710-711-712 composite sample.

Isopar™ L and Modifier Levels

A density measurement of the sample gave a result of 0.8292 g/mL (0.07% RSD) (or 0.8232 g/mL at 25 °C when corrected for temperature using the Caustic-Side Solvent Extraction (CSSX) temperature correction formula)[3] for MCU-16-701-702-703 at 18 °C (see Table 3-1). Similarly, the measured density for MCU-16-710-711-712 was 0.8275 g/mL (0.06% RSD) (or 0.8228 g/mL at 25°C) at 19.5 °C (see Table 3-2). The calculated density (0.823 g/mL) for MCU-16-701-702-703 and for MCU-16-710-711-712 (0.823 g/mL) were about 1.5% below the calculated density for the standard sample (0.835 g/mL at 25 °C for the scratch blend made in the laboratory).[2] Using the density as a starting point, we know that the concentration level of the Isopar™L component in the sample should be slightly above its nominal value (within analytical uncertainties) and the modifier concentration should be below its nominal value.

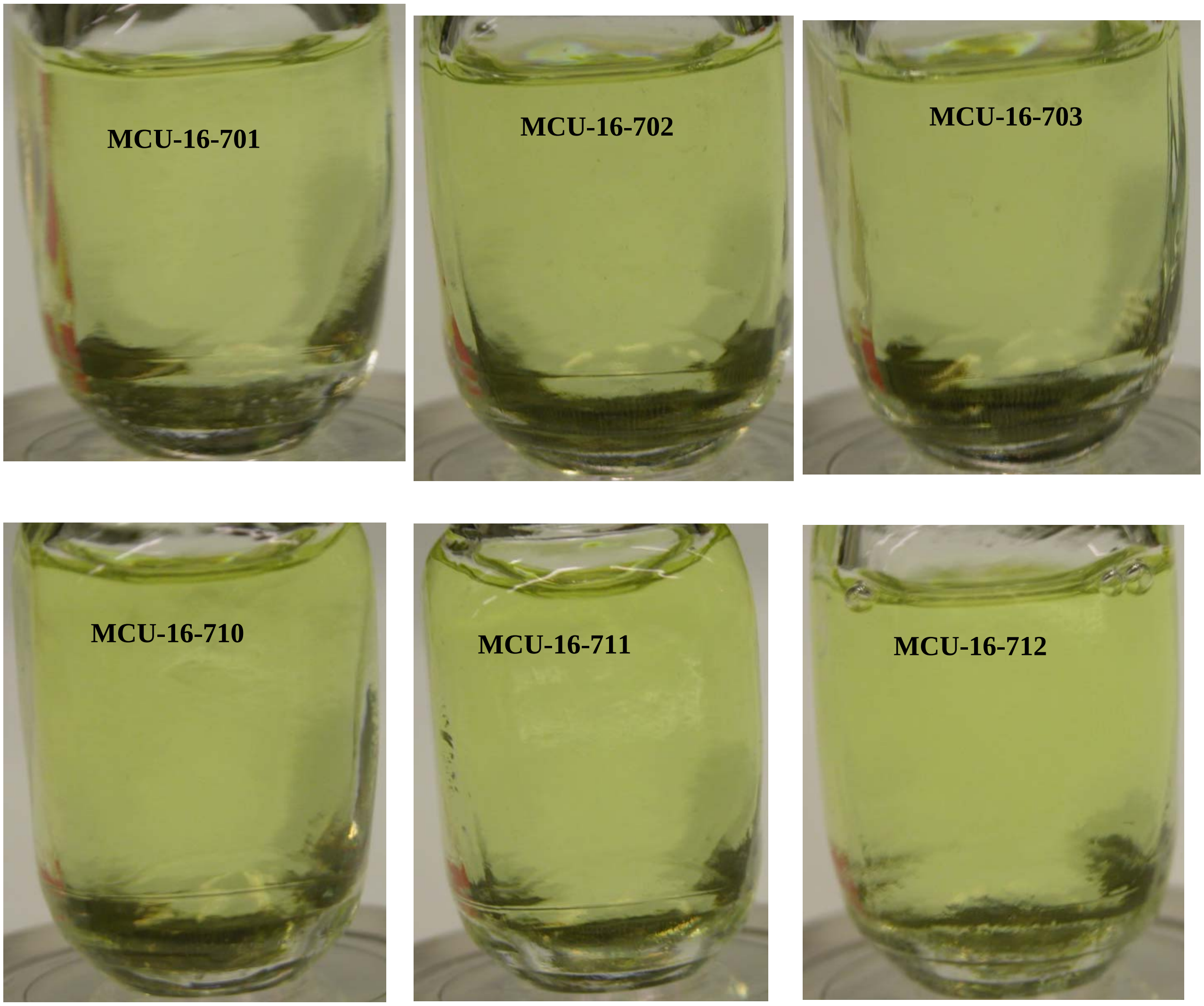

Figure 1. Typical appearance of the six MCU-16-701, MCU-16-702, and MCU-16-703, MCU-16-710, MCU-16-711, and MCU-16-712

An examination of Table 3-1 shows that the Isopar™ L concentration in MCU-16-701-702-703 is above its nominal value (~ 2%) in both samples (MCU-16-701-702-703 and MCU-16-710-711-712) while the modifier concentration was correspondingly 15% and 16% lower than its nominal value. Of all the methods listed, density has the lowest uncertainty. Thus, the final reported values are closer to the density measurement. The data confirms (based on the Isopar™ data) the trim addition to MCU on April 29, 2016. Every component, except for the modifier possibly due to its high viscosity, is at or above their nominal values.

All measurements indicate the Isopar™ L level is slightly above its nominal value while the modifier concentration level is below its nominal value (see Fig. 3 for recent modifier concentrations from HPLC measurements). Looking at Fig.3, the modifier level precipitously dropped from the March sample possibly due to a combined effect from the trim addition done in April 2016 (dilution) and analytical uncertainties. The relatively lower modifier concentration explains why the measured density is slightly below the standard sample density. 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 and Table 3-2.

They added up to 0.829 ± 0.019 g/mL (MCU-16-701-702-703) and 0.828 ± 0.019 g/mL (MCU-16-710-711-712) respectively. Their sum is consistent with the measured and temperature corrected (to 25 ºC) value of 0.823 g/mL for MCU-16-701-702-703 and 0.823 g/mL for MCU-16-710-711-712. Also the sum of the analytical results from Table 3-1 and 3-2 are consistent with the measured and corrected to 25 °C mass concentration (density) of the standard (0.835 g/mL). With a lower modifier concentration, the solvent chemical properties are closer to that of Isopar™L; thus, expect normal emulsification/de-emulsification as well as rheology and phase carry-over at the centrifuge contactors (as seen in the past two years). The current modifier concentration is well above the minimum modifier concentration below which the extractant concentration may drop due to solubility limits. However, sufficient modifier was added to the solvent in April 2016 for the levels to reach 169,000 mg/L and it appears that mixing conditions rather than a removal mechanism is limiting the equilibrium concentration of the modifier in the solvent. Future samples may show the added modifier.

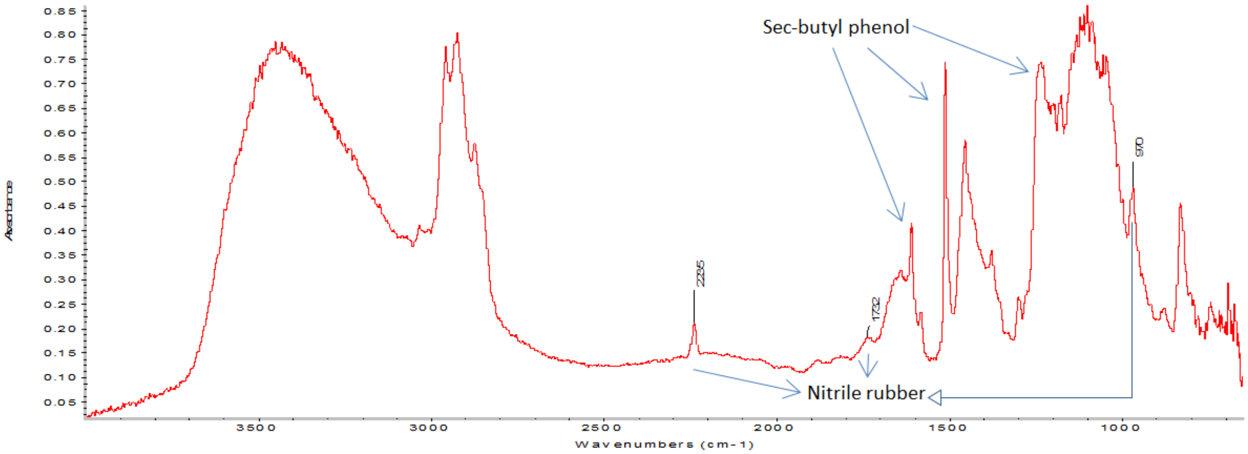

Figure 2. FTIR spectrum of a solid particle (0.5 mm x 1 mm) found in MCU-16-712.

Table 3-1. Sample Results for MCU-16-701-702-703

Analysis	Method	LW-AD-Proj-160531-1	Result (mg/L)[#]	Nominal[*] Result (mg/L)	% of (Result ÷ Nominal Result)
Isopar® L	FT-HNMR	NA	6.30E+05	6.23E+05	101
Isopar® L	FT-IR	NA	6.34E+05		102
Isopar® L	Density	NA	6.36E+05		102
Average[$]	All	NA	6.35E+05	6.23E+05	102
Modifier	HPLC	#1503	1.40E+05	1.69E+05	83
Modifier	FT-HNMR	NA	1.38E+05		82
Modifier	FT-IR	NA	1.51E+05		89
Modifier	Density	NA	1.44E+05		85
Average[$]	All	NA	1.43E+05	1.69E+05	85
TiDG[♠]	Titration	NA	1.35E+03	1.44E+03	94
Average[$]	All	NA	1.35E+03	1.44E+03	94
trioctylamine	Titration	NA	2.00E+02	5.30E+02	38
Average[$]	All	NA	2.00E+02	5.30E+02	38
MaxCalix	HPLC	#1503	4.60E+04	4.44E+04	104
MaxCalix	FT-HNMR	NA	4.73E+04		106
Average[$]	All	NA	4.64E+04	4.44E+04	105
BOBCalixC6	HPLC	#1503	2.52E+03	4.03E+03	63
Average[$]	All	NA	2.52E+03	4.03E+03	63
Density (g/mL)	Direct Measurement	NA	0.8232	0.835	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 uncertainties are 10% for the modifier and 13% for MaxCalix, and 14% for Isopar™ L. FTIR analytical uncertainties are 15% for Isopar® L and 10% for Modifier. 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.

[$] $x = \frac{\Sigma_1^i \left(x_i / \delta_i^2 \right)}{\Sigma_1^i \left(1 / \delta_i^2 \right)}$; 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.

Table 3-2. Sample results for MCU-16-710-711-712

Analysis	Method	LW-AD-Proj-160606-3	Result (mg/L)[#]	Nominal[*] Result (mg/L)	% of (Result ÷ Nominal Result)
Isopar® L	FT-HNMR	NA	6.41E+05	6.23E+05	103
Isopar® L	FT-IR	NA	6.34E+05		102
Isopar® L	Density	NA	6.35E+05		102
Average[$]	All	NA	6.35E+05	6.23E+05	102
Modifier	HPLC	#1699	1.39E+05	1.69E+05	82
Modifier	FT-HNMR	NA	1.40E+05		83
Modifier	FT-IR	NA	1.51E+05		89
Modifier	Density	NA	1.42E+05		84
Average[$]	All	NA	1.43E+05	1.69E+05	84
TiDG[♠]	Titration	NA	1.34E+03	1.44E+03	93
Average[$]	All	NA	1.34E+03	1.44E+03	93
trioctylamine	Titration	NA	2.10E+02	5.30E+02	40
Average[$]	All	NA	2.10E+02	5.30E+02	40
MaxCalix	HPLC	#1699	4.61E+04	4.44E+04	104
MaxCalix	FT-HNMR	NA	4.60E+04		104
Average[$]	All	NA	4.61E+04	4.44E+04	104
BOBCalixC6	HPLC	#1699	2.56E+03	4.03E+03	64
Average[$]	All	NA	2.56E+03	4.03E+03	64
Density (g/mL)	Direct Measurement	NA	0.8228	0.835	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 uncertainties are 10% for the modifier and 13% for MaxCalix, and 14% for Isopar™ L. FTIR analytical uncertainties are 15% for Isopar® L and 10% for Modifier. 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.

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[$] $x = \frac{\Sigma_1^i \left({x_i}/{\delta_i^2} \right)}{\Sigma_1^i \left({1}/{\delta_i^2} \right)}$; 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.

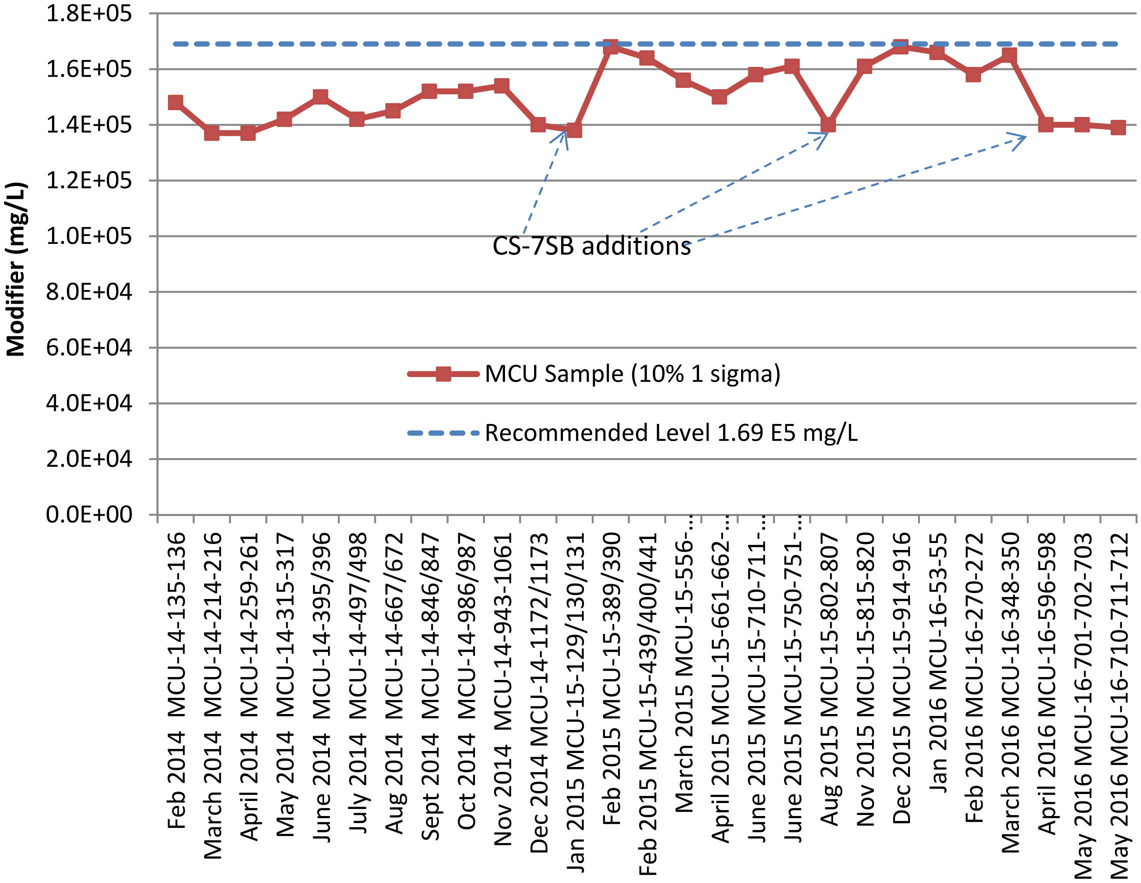

Figure 3. Modifier level in the solvent as measured by HPLC (one sigma is 10%).

Suppressors Levels

The average TiDG concentration level in MCU-16-701-702-703 is 1.35 ± 0.13 E3 mg/L and 1.34 ± 0.13 E3 mg/L in MCU-16-710-711-712. The TiDG level is at 94% and 93% respectively of its nominal value of 1440 mg/L in the two May samples. These levels confirmed the trim addition to the solvent done in April 2016 (the noticeable spike in the TiDG concentration level in the April sample in Fig. 4). The suppressor concentration is above the minimum recommended operating level (479 mg/L) and thus, the solvent does not require a TiDG addition at the time sample MCU-16-710, MCU-16-711 and MCU-16-713 were collected.

Inferring from past TiDG concentration level trends (see Fig. 4) and in the absence of new additions or new removal mechanisms (and assuming continuous steady operation), the TiDG concentration is expected to drop and reach the minimum recommended level sometime in late August 2016.

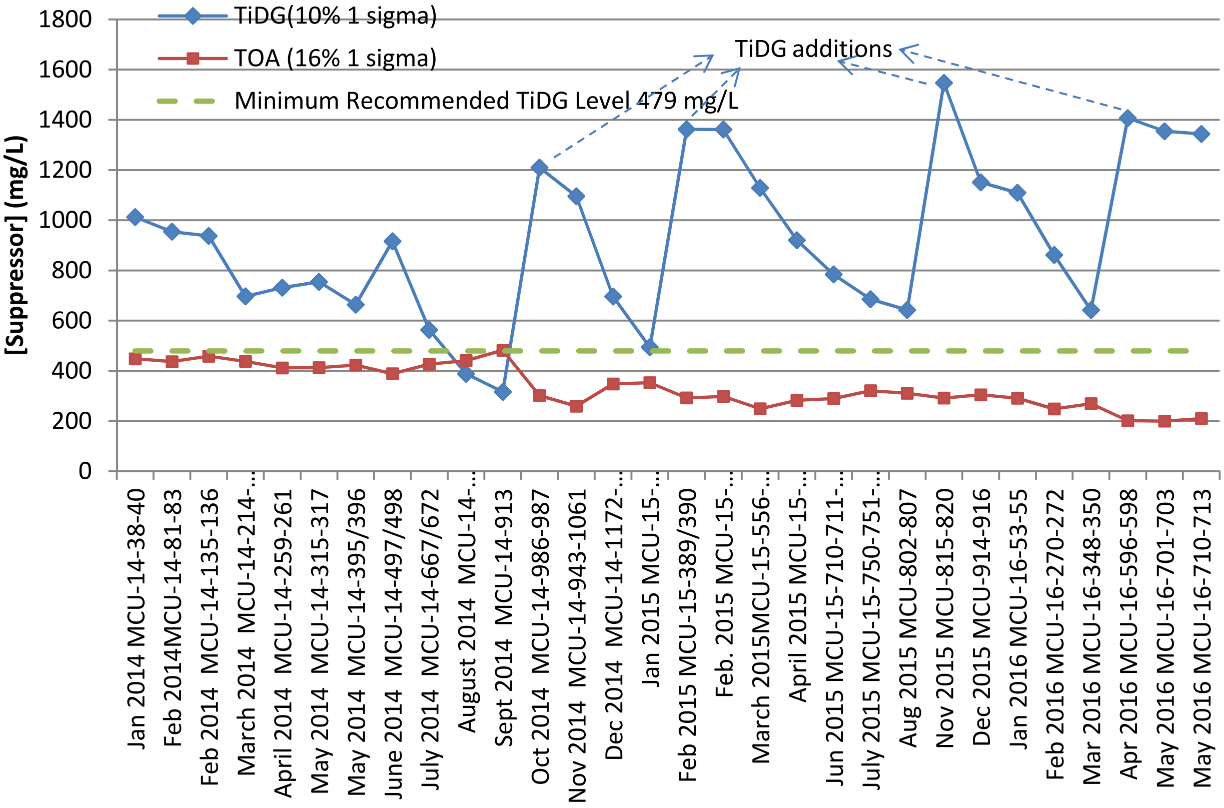

Figure 4. Suppressor concentration as measured by titration in SHT samples since NGS implementation. The minimum recommended level is 479 mg/L for TiDG.

The TOA concentration appears to have leveled. The TOA concentrations in MCU-16-701-702-703 and MCU-16-710-711-712 are 200 ± 32 mg/L and 210 ± 34 mg/L respectively in the previous sample the TOA level was at 269 mg/L). The differences between the current measurements and last month measurement are within the analytical error. Since MCU no longer adds TOA, a drop in TOA concentration is expected with time as observed in Fig. 4. However, the rate of TOA concentration decrease appears slower than expected perhaps due to TiDG degradation into primary amines, which have previously been identified as degradation products of the suppressor when heated (3 ºC, 25 ºC and 36 ºC).[4] The primary amine degradation products would likely have a similar pKa to the TOA (tertiary amine) making the equivalent points coincide.[5]

Extractant Levels

The average calculated MaxCalix level was 4.64E4 mg/L (±10%) and 4.61E4 mg/L (±10%) for the MCU-16-701-702-703 and the MCU-16-710-711-712 samples respectively. The MaxCalix concentration is slightly above its nominal value. The sudden drop in the MaxCalix concentration in the month of February 2016 is probably due to analytical variance (see Figure 5). However, the current MaxCalix concentration level is consistent with its historical trend (Fig. 5). The residual concentration of BOBCalixC6 levels in samples MCU-16-701-702-703 and MCU-16-710-711-712 were at 64% and 63% respectively of the level measured when the NGS was implemented in late FY13 (the concentration variability is due to analytical fluctuations). Since no BOBCalixC6 is added to the SHT, it cannot be explained at this time the constancy of the BOBCalixC6 concentration in the solvent.

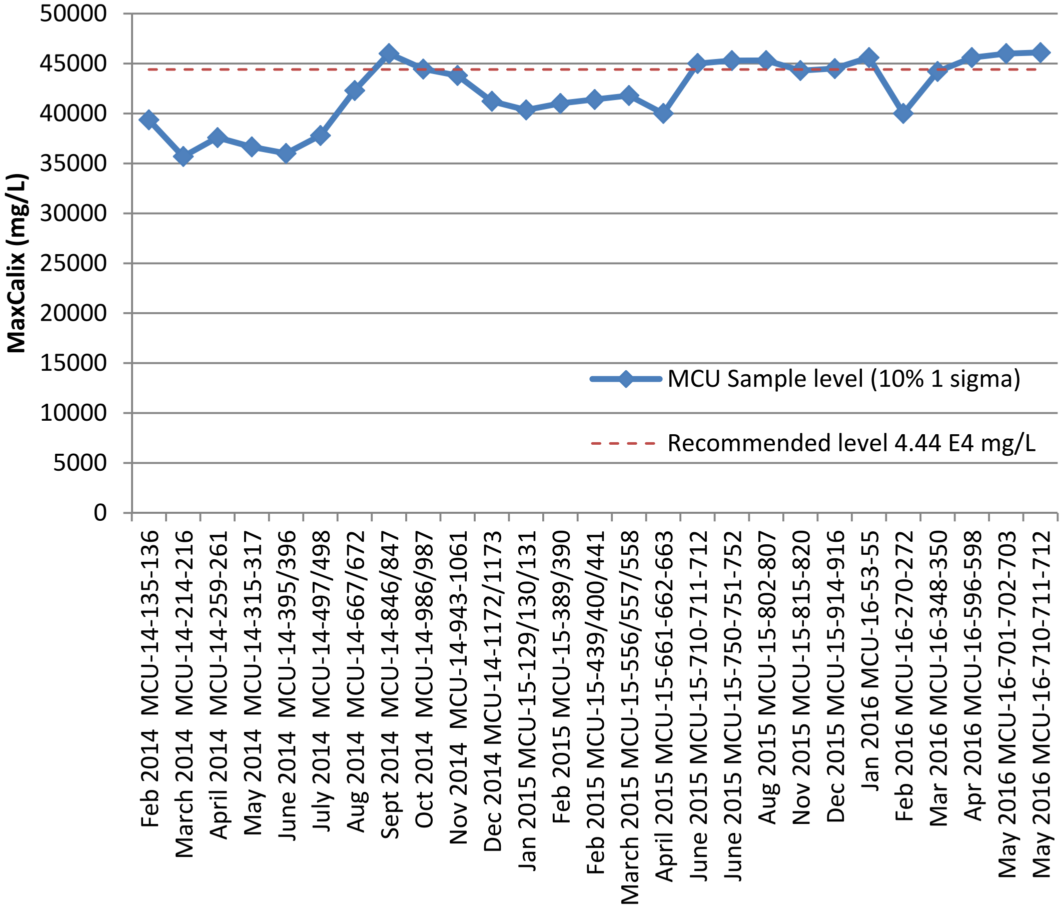

Figure 5. MaxCalix concentration as measured by HPLC and FT-HNMR of recent samples since NGS implementation (44,400 mg/L is the nominal concentration).

Gamma Level

The gamma measurements of MCU-16-701-702-702 and MCU-16-710-711-712 were 1.45E5 dpm/mL (±5%) and 1.26 E5 dpm/mL (±5%) respectively. These levels of activity are consistent with the previous gamma levels when the process was operating normally in late 2015. It confirms the steady state trend level observed since June 2015 (see Fig. 6).

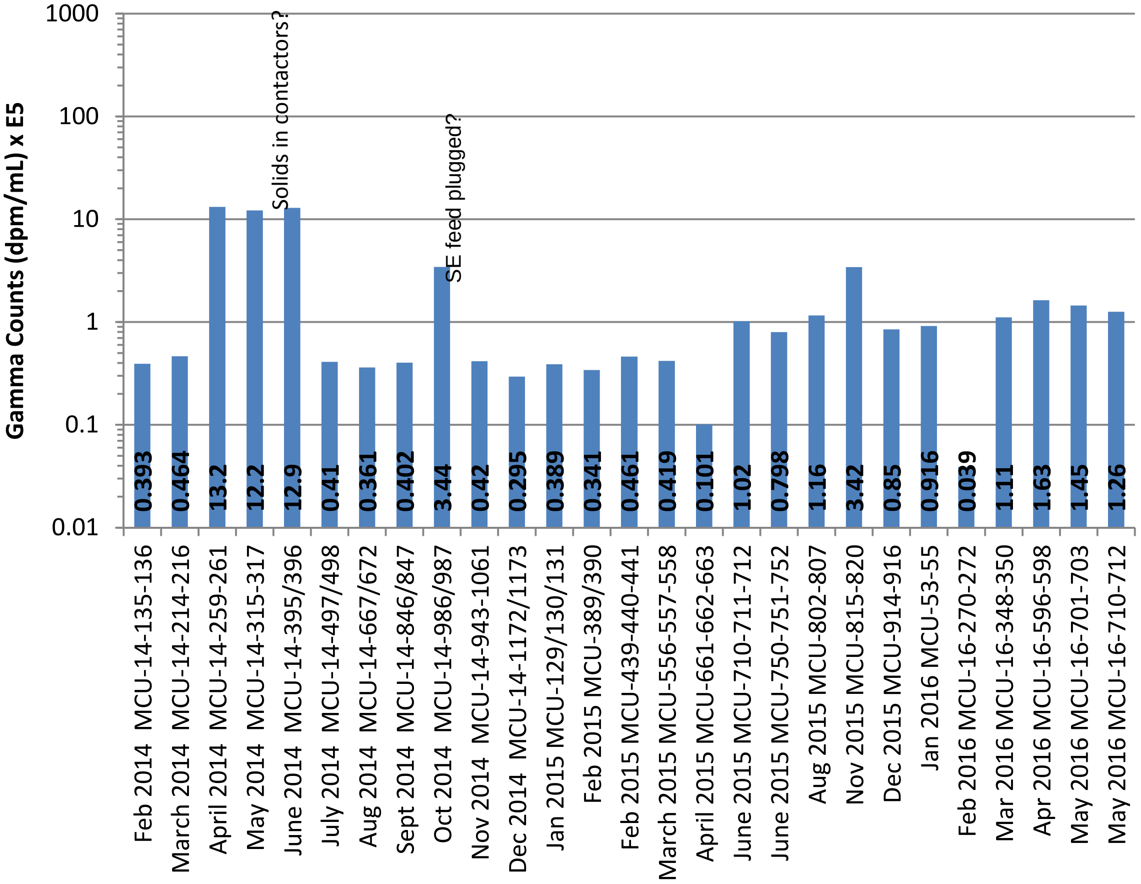

Figure 6. The gamma count of selected SHT samples. One sigma is 5%.

Impurities

No impurities were seen at the 1000 ppm level or higher as indicated by the SVOA method (± 20% uncertainty at 1 sigma). No impurities were observed in the HNMR spectrum.
A few mL of samples MCU-16-701-702-703 and MCU-16-710-711-712 were digested and analyzed for total mercury by the CV-AA method. The concentration of measured mercury by the CV-AA method was 21.6 ± 4.3 ug/$g_{solvent}$ and 21.1 ± 4.2 ug/$g_{solvent}$ (or 17 ug/$mL_{solvent}$) respectively. The mercury concentration of the undigested MCU-16-701-702-703 and MCU-16-710-711-712 samples by the XRF method were 15.1± 3.0 ug/$g_{solvent}$ and 16.6 ± 3.3 ug/$g_{solvent}$ respectively. Both results are similar when considering the error in the measurements

This level of mercury is significantly higher than the solubility of metallic Hg in dodecane (~3 ppm)[6] 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.[7] For 200 gallons of solvent (757.1 L) and assuming a density of 0.8241 g/mL, the solvent could contain a total of 9 ± 2 g of mercury. A comparison of this measurement with previous month confirms a positive trend in the mercury concentration in the solvent (data is shown in Fig. 7). The positive trend in Fig. 7 might be due to a higher mercury concentration in Salt Batch 8 (Tank 49H).

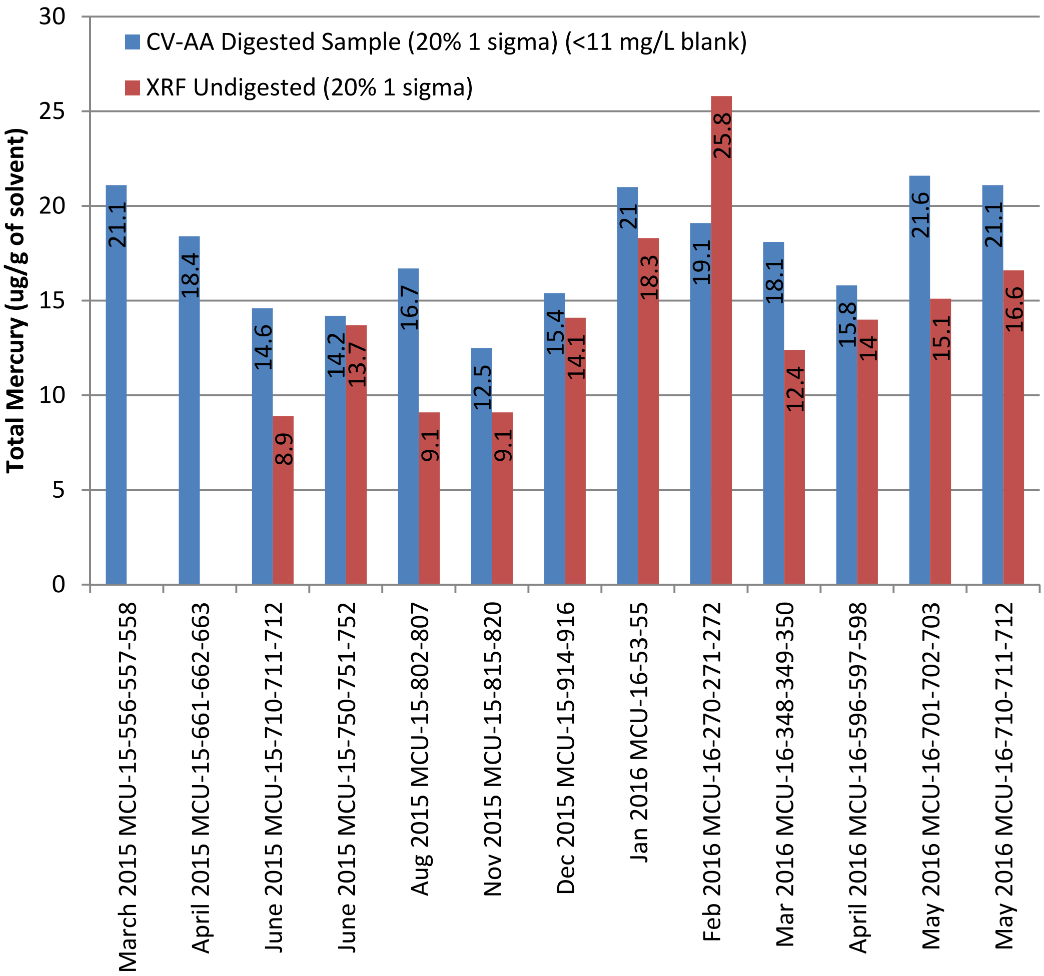

Figure 7. Total mercury in recent SHT samples. One sigma is 20%. CVAA = Cold Vapor Atomic Absorption Spectrometry. XRF =X-ray Fluorescence (20% one sigma).

Recommendations

The current analysis indicates the solvent has a lower modifier (84% of its nominal concentration) relative to the standard. Given that sufficient modifier was added to the solvent in April 2016, that future samples may show a rise in the modifier level. If the modifier level does not increase, it may indicate insufficient mixing or a new removal mechanism or an unrepresentative sample (absence of any simultaneous analytical biases). Consideration should be given to studies that examine the modifier dispersion into a spent and radioactive solvent following a trim addition. Given that the Isopar™L and MaxCalix levels are at or above their recommended nominal levels due to the April trim, it is believed that the modifier from the trim may be selectively dissolving (or sorbing and desorbing from surfaces) at a different rate than from the other trim's components. The lower MaxCalix concentration observed in the February 2016 sample was due to analytical measurement fluctuations. The TiDG, MaxCalix and Isopar™L levels are expected to trend downward with time. In order to remain two-sigma above the minimum recommended level, it is recommended the addition of modifier in the next solvent trim be made assuming that complete mixing of the existing modifier inventory in the solvent has occurred. In the case of a gross concentration of bacteria in the stripping solution, most likely to form when the strip solution is idled, filtration is recommended. Continuance of the periodic surveillance of the solvent to verify concentration and cleanliness is also recommended.

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 components concentration in the solvent.

4.0 Conclusions

SRNL received one set of SHT samples (MCU-16-701, MCU-16-702 and MCU-16-703), pulled on 05/23/2016 and another set of SHT samples (MCU-16-710, MCU-16-711, and MCU-16-712) pulled on 05/28/2016 after the solvent was superwashed with 300 mM sodium hydroxide for analysis. Samples MCU-16-701, MCU-16-702, and MCU-16-703 were combined into one sample (MCU-16-701-702-703) and samples MCU-16-710, MCU-16-711, and MCU-16-712 were combined into one sample (MCU-16-710-711-712). Of the two composite samples MCU-16-710-711-712 represents the current chemical state of the solvent at MCU. All analytical conclusions are based on the chemical analysis of MCU-16-710-711-712. There were no chemical differences between MCU-16-701-702-703 and superwashed MCU-16-710-711-712. Analysis of the composited sample MCU-16-710-712-713 indicated the Isopar™L concentration is above its nominal level (102%). The modifier (CS-7SB) is 16% below its nominal concentration while the TiDG and MaxCalix concentrations are at and above their nominal concentrations respectively. The TiDG level has begun to decrease and it is 7% below its nominal level as of May 28, 2016. Based on this current analysis, 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 the solvent samples by the SVOA. No impurities were observed in the HNMR. One small particle of sec-butyl phenol was observed floating in MCU-16-712 possibly dislodged from the solvent by the superwashing process (an indication this process is working). Also, up to 21.1 ± 4.2 ug/$g_{solvent}$ (or 17 ug/$mL_{solvent}$) of mercury was detected in this sample (as determined by the CV-AA method). XRF analysis of undigested MCU-16-710-711-712 sample detected 16.6 ± 3.3 ug/$g_{solvent}$ of mercury. The higher mercury concentration in the solvent (as determined in the last four monthly samples) is possibly due to the higher mercury concentration in Salt Batch 8 (Tank 49H).

The current gamma level (1.26E5 dpm/mL (±5%)) confirmed that the gamma concentration is consistent with the previous level where the process operated normally and as expected.

The similar component concentrations between the two composite samples (MCU-16-701-702-703 and MCU-16-710-711-12) shows that the superwashing step (cleaning with 300 mM sodium hydroxide solution)did not affect the main components of the MCU solvent.

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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