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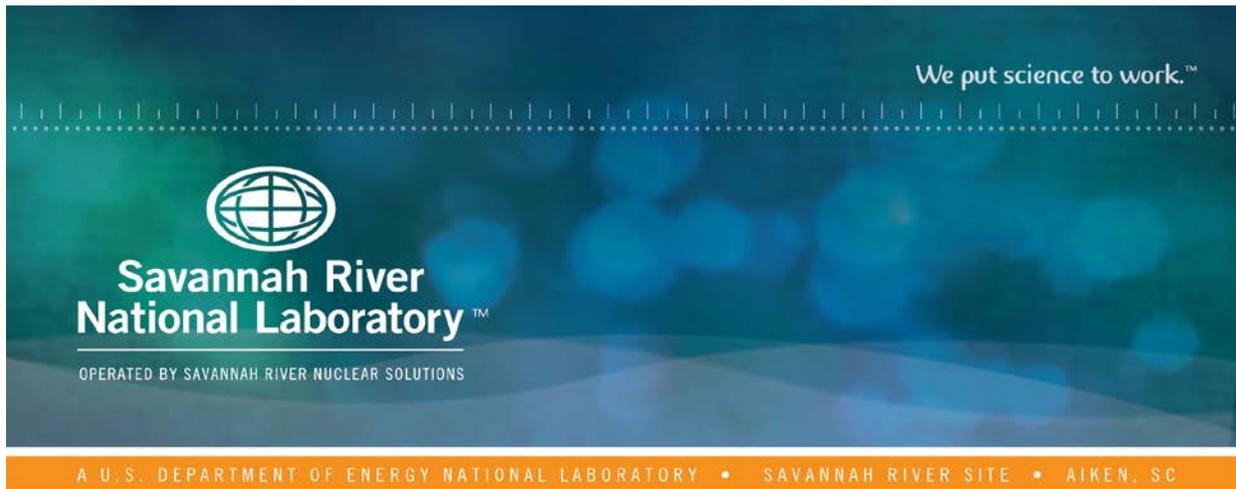
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# Solvent Hold Tank Sample Results for MCU-15-914-915-916: December 2015 Monthly Sample

F. F. Fondeur

D. H. Jones

March 2016

SRNL-STI-2016-00125, Revision 0



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# Solvent Hold Tank Sample Results for MCU-15-914- 915-916: December 2015 Monthly Sample

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March 2016

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## EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) received one set of Solvent Hold Tank (SHT) samples (MCU-15-914-915-916), pulled on 12/22/2015 for analysis. The samples were combined and analyzed for composition. Analysis of the composite sample MCU-15-914-915-916 indicated the TiDG, Isopar<sup>TM</sup>L, and MaxCalix are at nominal levels. The modifier concentration is 3% below its nominal concentration. This analysis confirms the addition of TiDG, MaxCalix, and modifier to the solvent in November 2015. Based on the current monthly sample, the levels of TiDG, Isopar<sup>TM</sup>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, the Fourier transform infra-red spectroscopy (FTIR) method detected trace levels (a few ppm) of amides (more indicative of bacteria than a possible degradation product of TiDG). In addition, up to  $18 \pm 4$  micrograms of mercury per gram of solvent (or 14.8  $\mu\text{g/mL}$ ) was detected in this sample.

The current gamma concentration level (8.48E4 dpm/mL) confirmed that the gamma concentration has returned to the previous level where the process operated normally 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( <i>tert</i> -octylbenzo-crown-6)
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- <i>alt</i> -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	<i>N,N',N''</i> -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.<sup>1</sup> On December 22, 2015, Operations personnel pulled and delivered three samples from the SHT (MCU-15-914, MCU-15-915, and MCU-15-916) 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 3 solvent components at the same time to generate a solution of the appropriate composition that approximates the blend of cocktail<sup>2</sup> 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 November 28, 2015, a trim addition was made to MCU that was 10.02 E3 grams of modifier and 765 grams of TiDG in 18.6 gallons of Isopar<sup>TM</sup>L.

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

<b>Event</b>	<b>Date</b>
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 <sup>TM</sup> L added to MCU	March 6, 2015
9 gallons of Isopar <sup>TM</sup> 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 <sup>TM</sup> 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
October solvent trim added to MCU	October 28, 2015
SHT sample MCU-15-815-816-817-818-819-820	November 29, 2015
SHT sample MCU-914-915-916	December 22, 2015
November solvent trim added to MCU	November 28, 2015

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-15-914, MCU-15-915, and MCU-15-916 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.

**Table 2-2 Nominal concentrations of the relevant components in NGS Blend<sup>2</sup>**

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

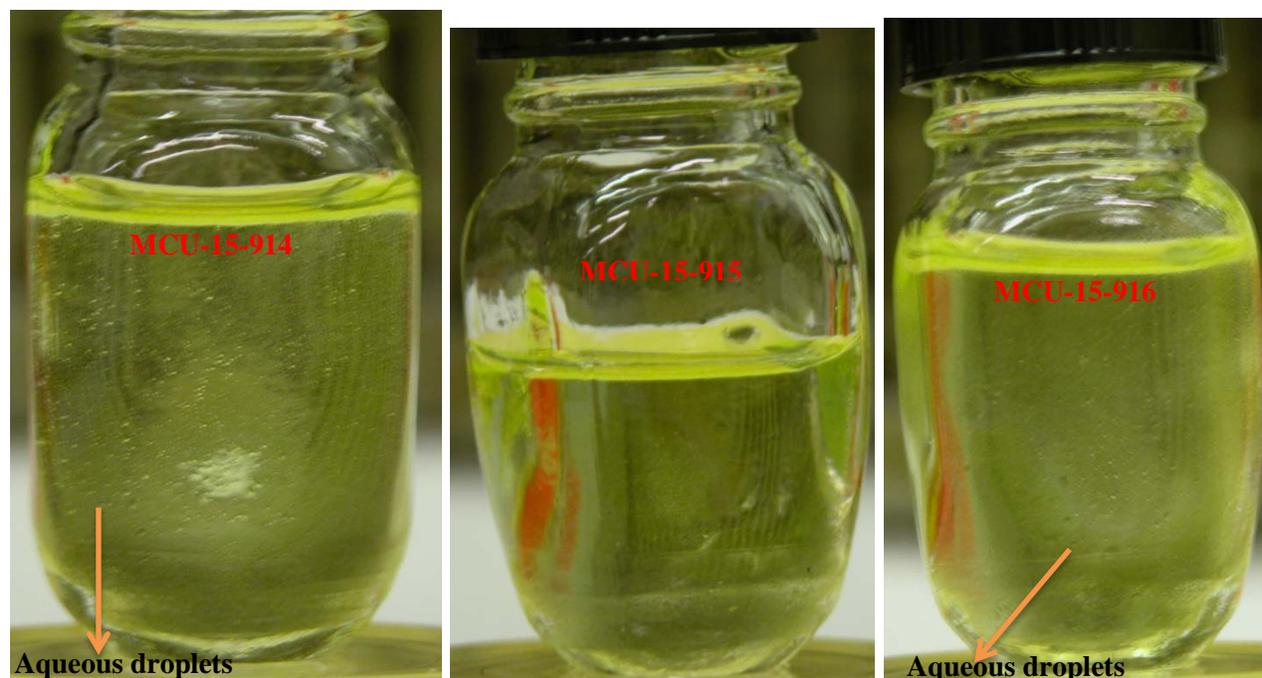
Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

## 3.0 Results and Discussion

Each sample (MCU-15-914, MCU-15-915, and MCU-15-916) was visually examined. Most were found to contain a single phase liquid with no apparent solids contamination or cloudiness. Minute aqueous droplets were found on the walls of the p-nut vials from sample MCU-15-914 and MCU-15-916. These were not considered to compromise the quality of the solvent since these droplets were immiscible in the solvent and remained on the walls after the emptying the p-nuts vial. 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-15-914-915-916). Table 3-1 contains the results for the MCU-15-914-915-916 composite sample.

### *Isopar<sup>TM</sup> L and Modifier Levels*

A density measurement of the sample gave a result of 0.8283 g/mL (0.12% RSD) (or 0.8249 g/mL at 25 °C when corrected for temperature using the CSSX temperature correction formula) for MCU-15-914-915-916 at 21 °C. The calculated density (0.8249 g/mL) for MCU-15-914-915-916 is less than 1% below the calculated density for the standard sample (0.835 g/mL at 25 °C for the scratch blend made in the laboratory)<sup>i</sup>. Using the density as a starting point, we know that the concentration level of the Isopar<sup>TM</sup>L component in the sample should be slightly above its nominal value (within analytical uncertainties) and the modifier concentration should be slightly below its nominal value.



**Figure 1. Typical appearance of the three MCU-15-914, MCU-15-915, and MCU-15-916**

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 (3% 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.

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. 2 for recent modifier concentrations from HPLC measurements). The modifier level appears to trend up and down possibly reflecting randomness in the process of mixing, sampling and analyzing it. 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. They added up to  $0.829 \pm 0.019$  g/mL. Their sum is consistent with the measured and temperature corrected (to 25 °C) value of 0.825 g/mL, and also with the measured and corrected to 25 °C mass concentration (density) of the standard (0.835 g/mL).

**Table 3-1 Sample Results for MCU-15-914-915-916**

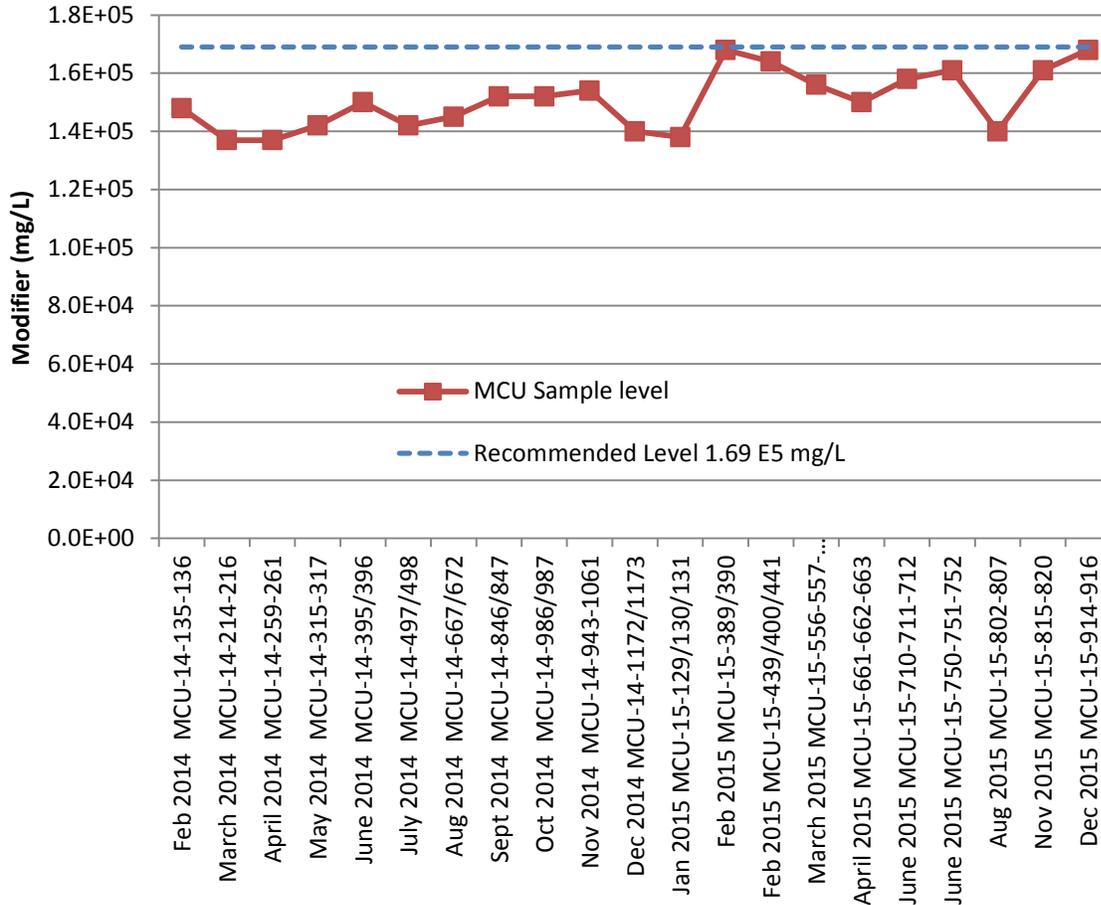
Analysis	Method	LIMS #	Result (mg/L) <sup>#</sup>	Nominal* Result (mg/L)	% of (Result ÷ Nominal Result)
Isopar <sup>®</sup> L	FT-HNMR	NA	6.23E+05	6.23E+05	100
Isopar <sup>®</sup> L	FTIR	NA	6.23E+05		100
Isopar <sup>®</sup> L	Density*	NA	6.31E+05		101
Average <sup>s</sup>	All	NA	6.31E+05		101
Modifier	HPLC	300320446	1.68E+05	1.69E+05	99
Modifier	FT-HNMR	NA	1.56E+05		92
Modifier	FTIR	NA	1.61E+05		96
Modifier	Density*	NA	1.47E+05		87
Average <sup>s</sup>	All	NA	1.50E+05		88
TiDG <sup>‡</sup>	Titration	NA	1.15E+03	1.44E+03	80
Average <sup>s</sup>	All	NA	1.15E+03	1.44E+03	80
trioctylamine	Titration	NA	3.05E+02	5.30E+02	58
Average <sup>s</sup>	All	NA	3.05E+02	5.30E+02	58
MaxCalix	FT-HNMR	NA	4.37E+04	4.44E+04	98
MaxCalix	HPLC	300320446	4.50E+04		101
Average <sup>s</sup>	All	NA	4.45E+04		100
BOBCalixC6	HPLC	300320446	2.80E+03	4.03E+03	69
Average <sup>s</sup>	All	NA	2.80E+03	4.03E+03	69
Density (g/mL)	Direct Measurement	NA	0.8249	0.835	99

<sup>#</sup> 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, 14% for Isopar<sup>™</sup> L, and 20% for TiDG. N/A = Not Applicable. Density estimations assume the combined weight percent of TiDG, MaxCalix, BOBCalixC6, and TOA to be approximately 6%.

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

$$s = \frac{\sum_i \left( \frac{x_i}{\delta_i} \right)^2}{\sum_i \left( \frac{1}{\delta_i^2} \right)}; \quad x_i \text{ stands for the concentration obtained at a given method and } \delta_i \text{ is the corresponding uncertainty.}$$

<sup>‡</sup> No TiDG value was estimated by FTHNMR due to an aged (questionable) standard.

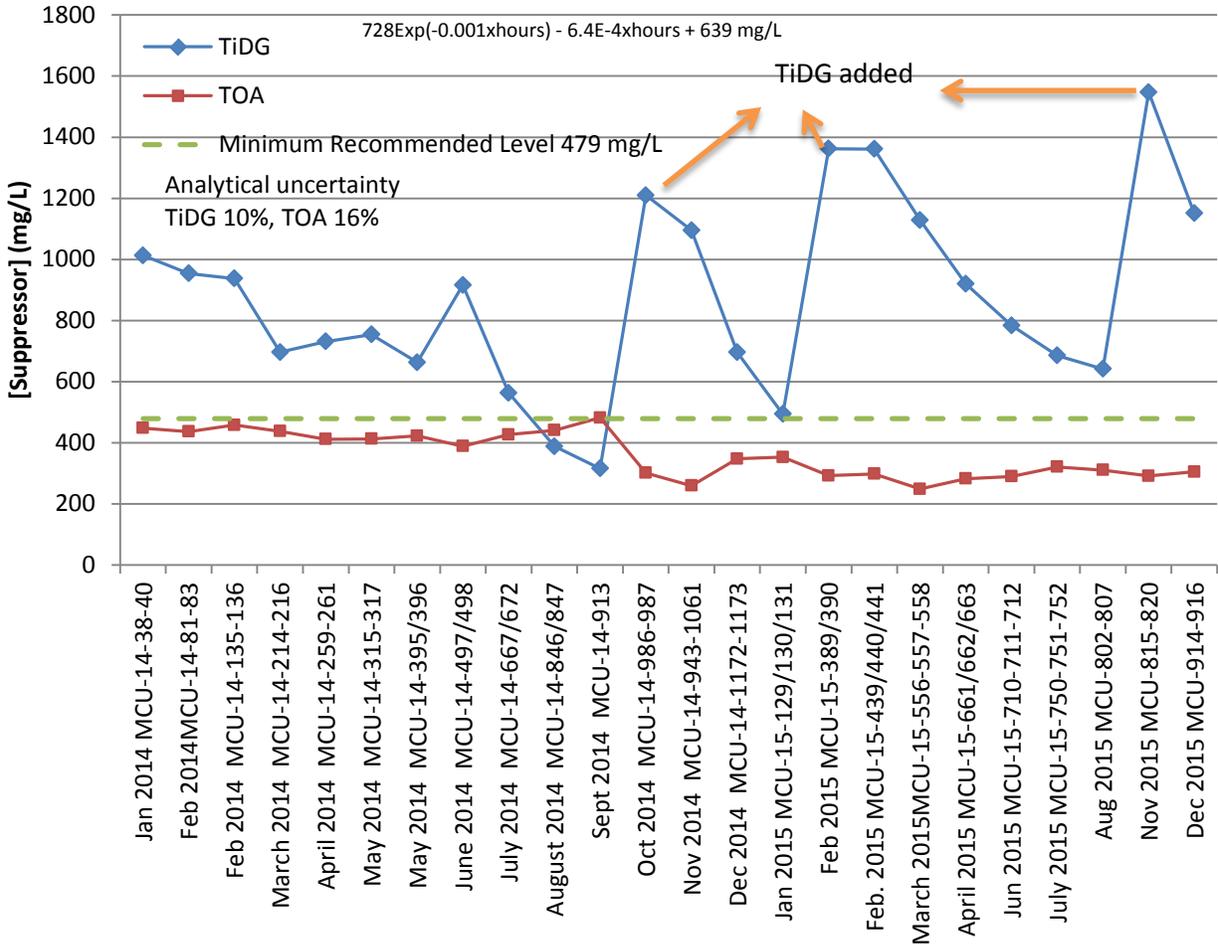


**Figure 2 Modifier level in the solvent as measured by HPLC (one sigma is 10%).**

*Suppressors Levels*

The average TiDG concentration level ( $1.15 \pm 0.1 \text{ E3 mg/L}$ ) is at 80 % of its nominal value of 1440 mg/L confirming the rapid depletion of the trim addition to the solvent done in December 2015 (a noticeable spike in the TiDG concentration level was observed in Fig. 3). The suppressor concentration is above the minimum recommended operating level (479 mg/L) and thus, the solvent did not require a TiDG addition at the time sample MCU-15-914-915-916 was collected. Inferring from past TiDG concentration level trends (see Fig. 3) 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 end of March or early April 2016. This drop is due to the combined effect of auto-decomposition and phase transfer to the aqueous phases (salt solution and boric acid). The TOA concentration appears to have increased slightly  $305 \pm 49 \text{ mg/L}$  (in the previous sample the TOA level was at 292 mg/L). The difference between this and last month measurement is within analytical error. Looking at the slope of the TOA concentration data, it appears to trend upwards with time after the trim addition (TiDG). The TOA's concentration level trend appears to anti-correlate with the TiDG's concentration level trend (or the disappearance of Isopar™ L due to evaporation). This anti-correlation is believed to be due to the combined effects of titration of TiDG's decomposition products, dilution effects from the trimming, and to the evaporation of Isopar™ L. Since MCU no longer adds TOA, a drop in TOA concentration is expected with time. The drop in the TOA concentration level, right after the trim addition, is possibly due to the dilution effect of the trim (see Fig. 3). Based on previous historical trends

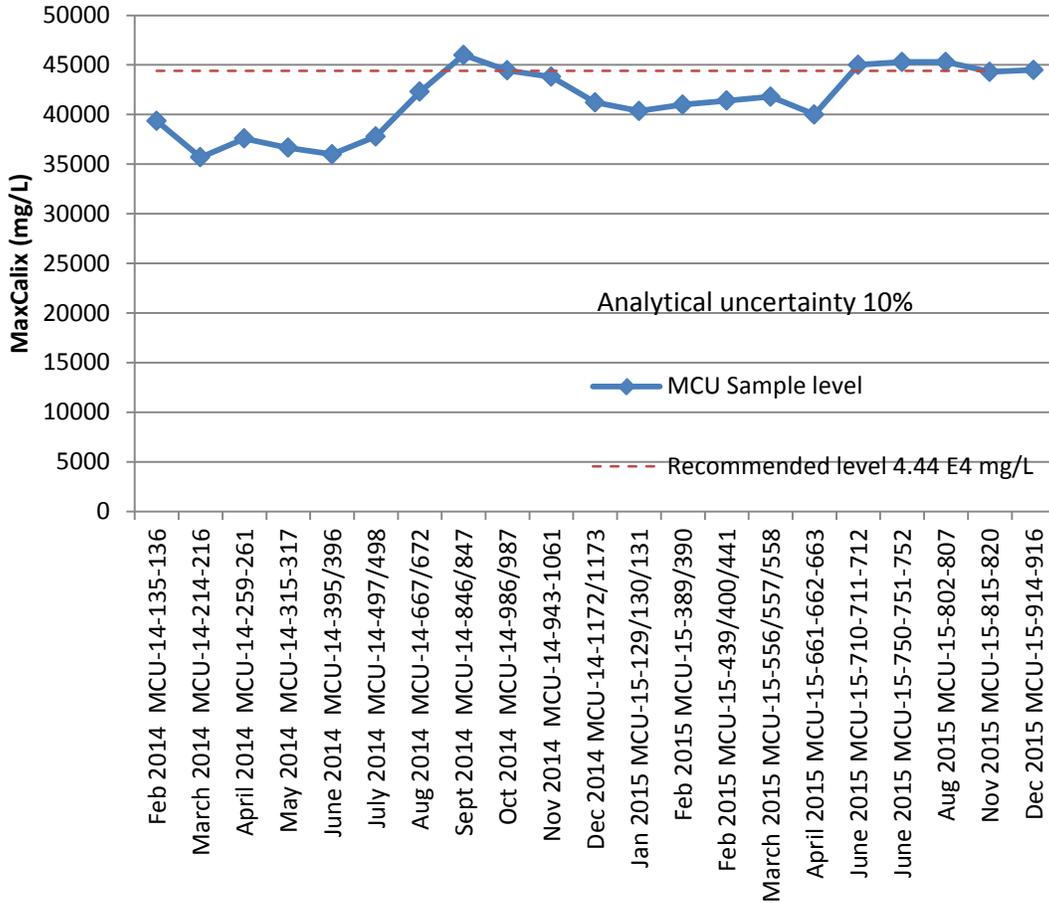
the TOA concentration is expected to increase. The increase is perhaps due to TiDG degradation into primary amines, which have previously been identified as degradation products of the suppressor when heated.<sup>3</sup> The primary amine degradation products would likely have a similar pKa to the TOA (tertiary amine) making the equivalent points coincide.<sup>4</sup>



**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 MaxCalix level is 4.45E4 mg/L ( $\pm 13\%$ ) and that is nearly the same as the nominal concentration and it is within the 95% confidence level of the analytical measurement as shown in Figure 4. The residual concentration of BOBCalixC6 level is currently at 69% of the level measured when the NGS was implemented in late FY13.



**Figure 4. 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 measurement of MCU-15-914-915-916 is 8.48E4 dpm/mL ( $\pm 5\%$ ). This level of activity indicates a return to normal levels previously seen in early 2015 (see Fig. 5). It confirms an end to the upward trend in the data that was first detected in the June 2015. In the recent past, an apparent correlation was observed between poor extraction and/or stripping performance of the solvent and high gamma activity levels in the solvent. The current gamma level in the solvent indicates the process has return to previous normal levels.

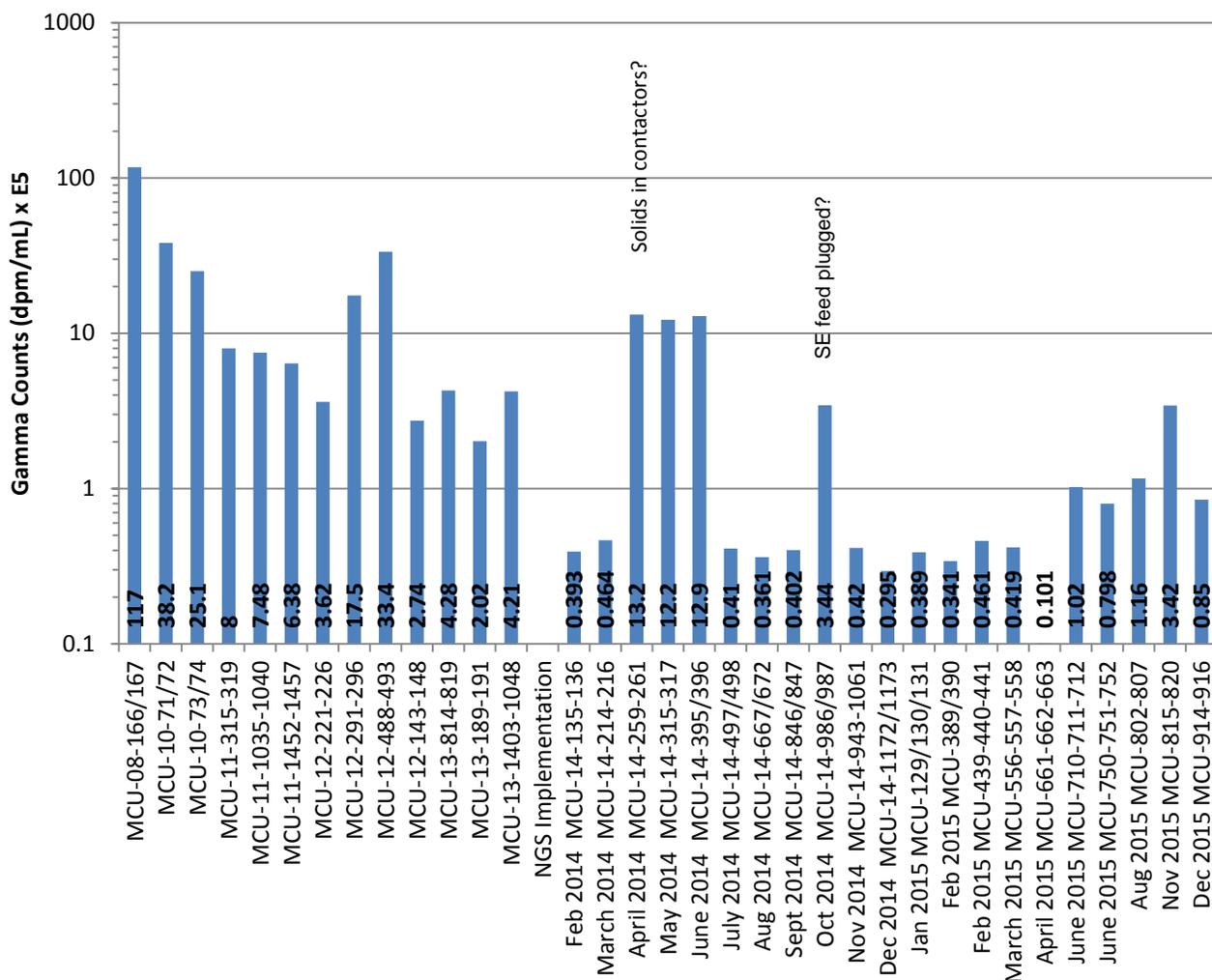


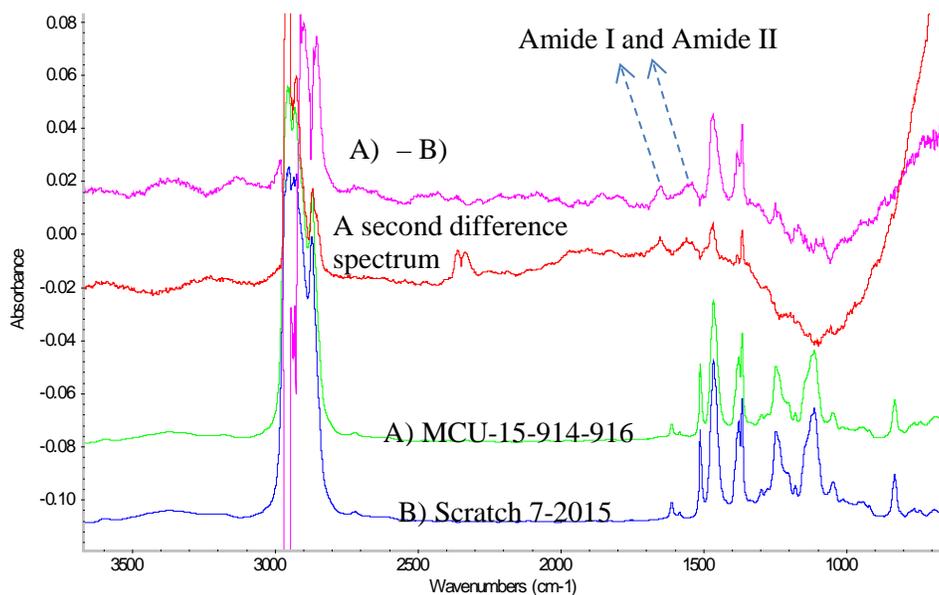
Figure 5. The gamma count of selected SHT samples. One standard deviation is 5%.

### Impurities

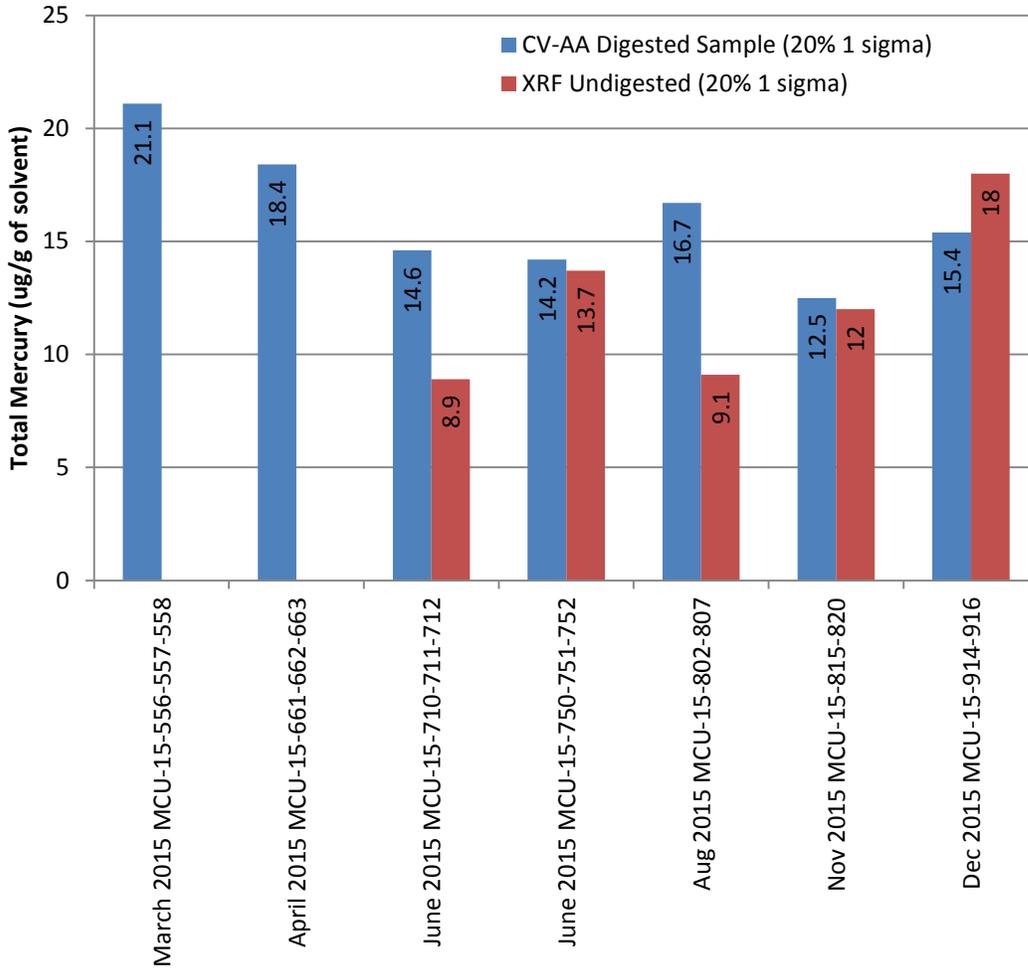
No impurities were seen at the 1000 ppm level or higher as indicated by the SVOA method ( $\pm 20\%$  uncertainty). No impurities were observed in the HNMR spectrum. However, evidence of amides (RCO-NHR) in this sample after subtracting the scratch solvent spectrum (see Fig. 6). The spectral at 1650 and 1550  $\text{cm}^{-1}$  can also be assigned to a protein source like bacteria. However, the 3380  $\text{cm}^{-1}$  band should be the strongest band for bacteria. Another amide source may be a degradation byproduct from TiDG. The frequent addition of TiDG (see Fig. 3) is possibly raising the TiDG's decomposition products in the solvent. At this point, the observed amide could be bacteria.

A few mL of the blended solvent was digested and analyzed for mercury (by the cold vapor method and the XRF method). The CV-AA method failed to detect any mercury in this sample (and further inquiry is being pursued to validate this finding). However, the XRF method detected a total mercury level in the solvent of  $18 \mu\text{g}/\text{g}_{\text{solvent}}$  (20% st. dev.) or  $14.8 \mu\text{g}/\text{mL}_{\text{solvent}}$ . If the XRF value is true, it is significantly higher than the solubility of metallic Hg in dodecane ( $\sim 3 \text{ ppm}$ )<sup>5</sup> 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.<sup>6</sup> For 200 gallons of solvent (757.1 L) and assuming a density of 0.825 g/mL, the solvent could contain a total of  $11 \pm 2$  g of mercury. A comparison of this measurement with previous month data is shown in Fig. 7. Based on the Mercury XRF data, there appears to be a correlation between the level of TiDG and the level of mercury in the solvent but further data is needed. If the correlation is true, it may be due to the complexing ability of TiDG and its decomposition products for heavy metals.



**Figure 6. Possible amide presence in the solvent**



**Figure 7. Total mercury in recent SHT samples. One standard deviation is 20%. CVAA = Cold Vapor Atomic Absorption Spectrometry. XRF =X-ray Fluorescence.**

*Recommendation*

The current analysis indicates the solvent has a slightly lower modifier and TiDG level, but nominal levels of Isopar™L and MaxCalix relative to the standard. 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 to continue the periodic surveillance of and trimming additions to the solvent.

#### 4.0 Conclusions

SRNL received one set of SHT samples (MCU-15-914-915-916), pulled on 12/22/2015 for analysis. The samples were combined and analyzed for composition. Analysis of the composite sample MCU-15-914-915-916 indicated the TiDG, Isopar<sup>TM</sup>L, and MaxCalix are at nominal levels. The modifier concentration is 3% below its nominal concentration. This analysis confirms the addition of TiDG, MaxCalix, and modifier to the solvent on November 28, 2015. Based on the current monthly sample, the levels of TiDG, Isopar<sup>TM</sup>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 SVOA. No impurities were observed in the HNMR. However, the FTIR method detected trace levels (a few ppm) of amides (more indicative of bacteria than a possible degradation product of TiDG). In addition, up to  $18 \pm 4$  micrograms of mercury per gram of solvent (or  $14.8 \mu\text{g/mL}$ ) was detected in this sample.

The current gamma concentration level ( $8.48\text{E}4$  dpm/mL) confirmed the process may have return to previously normal levels.

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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