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Solvent Hold Tank Sample Results for MCU-20-32-33-34 (September 2020): Quarterly Report

F. F. Fondeur

D. H. Jones

April 2021

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REVIEWS AND APPROVALS

AUTHORS:

_____
F. F. Fondeur, Separation Science & Engineering

_____
D. H. Jones, Research Support

TECHNICAL REVIEW:

_____
T. B. Peters, Separation Science & Engineering
Design Check Reviewed per Manual E7 Procedure 2.60

APPROVAL:

_____
B. J. Wiedenman, Manager, Separation Science & Engineering

_____
S. D. Fink, Director, Chemical Processing Sciences

_____
C. I. Aponte, Acting Manager, Tank Farm Facility Engineering

EXECUTIVE SUMMARY

The final characterization of Solvent Hold Tank (SHT) solvent sample from September 2020 (MCU-20-32-33-34) is reported and compared to past measurements. Analyses of the September 2020 SHT sample results in relation to the past measurements indicated that the Modifier (Cs-7SB), the Extractant (MaxCalix), the suppressor, and the gamma concentration were either steady or increasing due to Isopar™ L evaporation. The final relevant component concentration based on the September 2020 sample is shown below.

Component	**mg/L**
Isopar™L	603,900.0
Modifier(CS-7SB), 1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol	186,000.0
MaxCalix, 1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6	5,270.0
TiDG, *N,N',N''*–tris(3,7-dimethyloctyl)guanidine	836.0

The Semi-Volatile Organic Analysis (SVOA) and FT-HNMR did not detect any organic impurities. An impurity observed in the samples was mercury. Based on the September 2020 SHT sample, up to 22 ± 4 micrograms of mercury per gram of solvent (or 19 ± 4 mg/L) was detected.

The gamma concentration (~3.72 E4 dpm/mL) measured in the September 2020 SHT samples was consistent with previous values observed when MCU was idle (for example, between February 2017 and August 2017).

Modular Caustic-Side Solvent Extraction Unit (MCU) ceased operations in May 22, 2019 and entered a lay-up status. This report documents the last characterization analysis of the SHT solvent from MCU and is representing the analysis prior to approximately 128 gallons of SHT solvent being transferred to 30-gallon drums with 55-gallon drum overpacks for disposal.

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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
DMA	Direct Mercury Analysis
FT-HNMR	Fourier Transform Hydrogen Nuclear Magnetic Resonance
HNMR	Hydrogen Nuclear Magnetic Resonance
HPLC	High Performance Liquid Chromatography
MCU	Modular Caustic-Side Solvent Extraction Unit
MaxCalix	1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arene-benzocrown-6
NGS	Next Generation Solvent
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
SWPF	Salt Waste Process Facility
TiDG	*N,N',N''*–tris(3,7-dimethyloctyl)guanidine
TOA	trioctylamine
XRF	X-Ray Fluorescence

1.0 Introduction

In late FY13, MCU implemented the Next Generation Solvent (NGS) flow sheet. Facility personnel added a non-radioactive, NGS "cocktail" containing the new Extractant (MaxCalix) and a new Suppressor (TiDG) to the SHT heel to implement the NGS flow sheet. The resulting "blend" solvent ("NGS blend solvent") is essentially NGS with residual amounts of calix[4]arene-bis(tert-octylbenzo-crown-6) (BOBCalixC6) and trioctylamine (TOA). For process monitoring, SHT samples are sent to Savannah River National Laboratory (SRNL) to examine solvent composition changes over time.[1,2] 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 the September 2020 (MCU-20-32-33-34) solvent SHT sample. A summary report of the September SHT solvent sample was issued earlier.[3] This report examines the cumulative results from these and several past monthly reports.

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 (this includes the old extractant BoBCalix and the old suppressor TrioctylAmine that remained when solvent was converted to Next Generation Solvent [NGS]) of the composition that approximates the blend of cocktail[4] and heel solvent – was prepared in the lab (September 2018) and used for comparison and evaluation. The results from the analyses are presented in this document.

This report is the last characterization analysis of the SHT solvent. MCU entered a lay-up state and about 128 gallons of the SHT solvent were pumped into 55 gallons drums for future disposal.

2.0 Experimental Procedure

2.1 Experimental Procedure

Table 2-1 lists a summary of relevant and recent trims to the MCU solvent as well as the arrival date of the sample currently being studied. The MCU solvent has been placed in six 30-gallon drums for disposal in support of MCU transitioning into a lay-up state. No future monitoring samples are expected.

Table 2-1 Log of trims to MCU solvent for 2019 and SHT sampling dates

Event	Date
SHT special trim added	January 23, 2019
SHT sample MCU-19-2-3-4	January 26, 2019
9 gallons of Isopar™ L added to MCU	February 4, 2019
SHT sample MCU-19-83-84-85	February 17, 2019
11 gallons of Isopar™ L added to MCU	February 21, 2019
SHT sample MCU-19-138-139-140 special trim added	March 5, 2019
SHT sample MCU-19-208-209-210	March 19, 2019
9 gallons of Isopar™ L added to MCU	March 27, 2019
SHT sample MCU-19-366-367-368	April 13, 2019
SHT sample MCU-19-469-470-471	May 17, 2019
9 gallons Isopar™ L added to MCU	May 31, 2019
SHT sample MCU-19-524-525-526	June 23, 2019
SHT sample MCU-19-557-558-559	July 23, 2019
SHT sample MCU-19-560-561-562	August 13, 2019
SHT sample MCU-19-566-567-568	September 16, 2019
SHT sample MCU-19-569-570-571	December 16, 2019
SHT sample MCU-20-1-28	January 23, 2020
SHT sample MCU-20-29-30-31	June 8, 2020
SHT sample MCU-20-32-33-34	September 14, 2020
128 gallons of the SHT solvent placed in six 30-gallon drums with 55-gallon overpacks for future disposal	November 18, 2020

Samples shown in Table 2-1 were received in P-nut vials containing ~10 mL each (see Figure 1). Once taken into a radioactive hood, the samples were visually inspected and analyzed for pH. Contents of the P-nut vials for each monthly SHT sample were composited before use. Aliquots of the composited sample were removed to perform the following analyses: density, SVOA, HPLC, titration for TiDG, gamma counting, DMA, and FT-HNMR. Results from analytical measurements were compared with the theoretical values shown in Table 2-2. Please note that the HPLC, DMA, density, titration for TiDG, and FT-HNMR results for each SHT sample are shown in the respective monthly reports. All reported values were checked against the values obtained from a scratch solvent made in September 2018. All error bars represent one-sigma (one standard deviation). In the case of the physical measurements (density, surface tension, and viscosity measurements), the one-sigma was obtained from three replicates (observations). Suppressor concentration derived from titration was performed in duplicate. The one-sigma from the DMA measurement was obtained from duplicate observations (replicates). Therefore, the error bars shown in this report are the variations within replicates (or fidelity of the analytical measurements).

Table 2-2 Nominal concentrations of the relevant components in NGS Blend at 25 °C (Ref. 4)

Component	mg/L	Molar
MaxCalix	~ 44,400♠ to 47,800♣	~ 0.0465 to 0.050
BOBCalixC6*	< 4,030	< 0.0035
TOA*	< 530	< 0.0015
Modifier	~ 169,000	~ 0.50
TiDG	~1,440♥	~ 0.003
Isopar™ L	~ 607,000♣ to 613,000♠	~ 73.05 to 73.69 wt. %

*Values represent starting values when NGS blend was implemented. These components are no longer added to or refurbished in MCU.
♣Solvent composition is closer to a pure NGS formulation.
♠ Solvent composition is closer to an NGS-CSSX blend formulation.
♥Assuming a molecular weight for caustic-washed TiDG of 479 g/mol (516 g/mol for TiDG*HCl).

2.2 Quality Assurance

This work was performed under a stated Production Support request.[1] The recorded data, analysis, and conclusions satisfied previously published requirements.[2] Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 2.60 (design check requirements). 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 (see Figure 1). Floating debris was observed in MCU-20-32 and MCU-20-34. A Fourier-Transformed Infrared Resonance (FTIR) analysis revealed the debris to be of two types: one is an ester (possibly an acrylate material or a methoxy alkyd acid or a glycolate-like substance (like degraded ethylene glycol) but more characterization data is needed to make an identification) and the other material is a cellulose material containing an amine material (see Figure 2). The amine is probably degradation product from the suppressor. Based on the pliable consistency of the ester material it is possible that it might be a degradation product of degraded methacrylate with hydroxyl groups. This is a signature of a variety of common plastics. The solvent was slightly caustic (pH=8). SRNL believes the relatively high pH is evidence that the solvent contacted a caustic solution which is consistent with the caustic wash step of the solvent at MCU. No unusual reactions, solids, foaming, or immiscible layers were observed after combining the samples into one Teflon container for each set of monthly SHT samples.

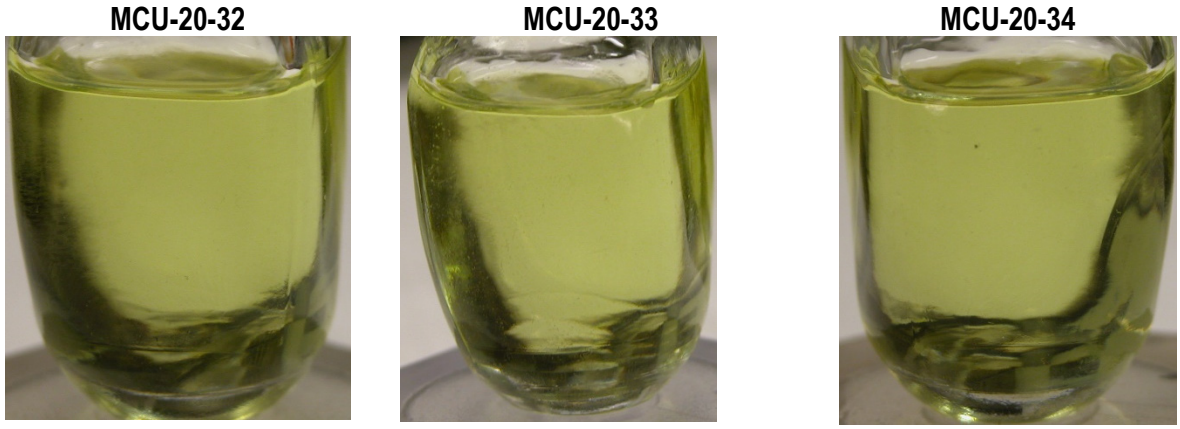

Figure 1. A picture of samples MCU-20-32, MCU-20-33, and MCU-20-34.

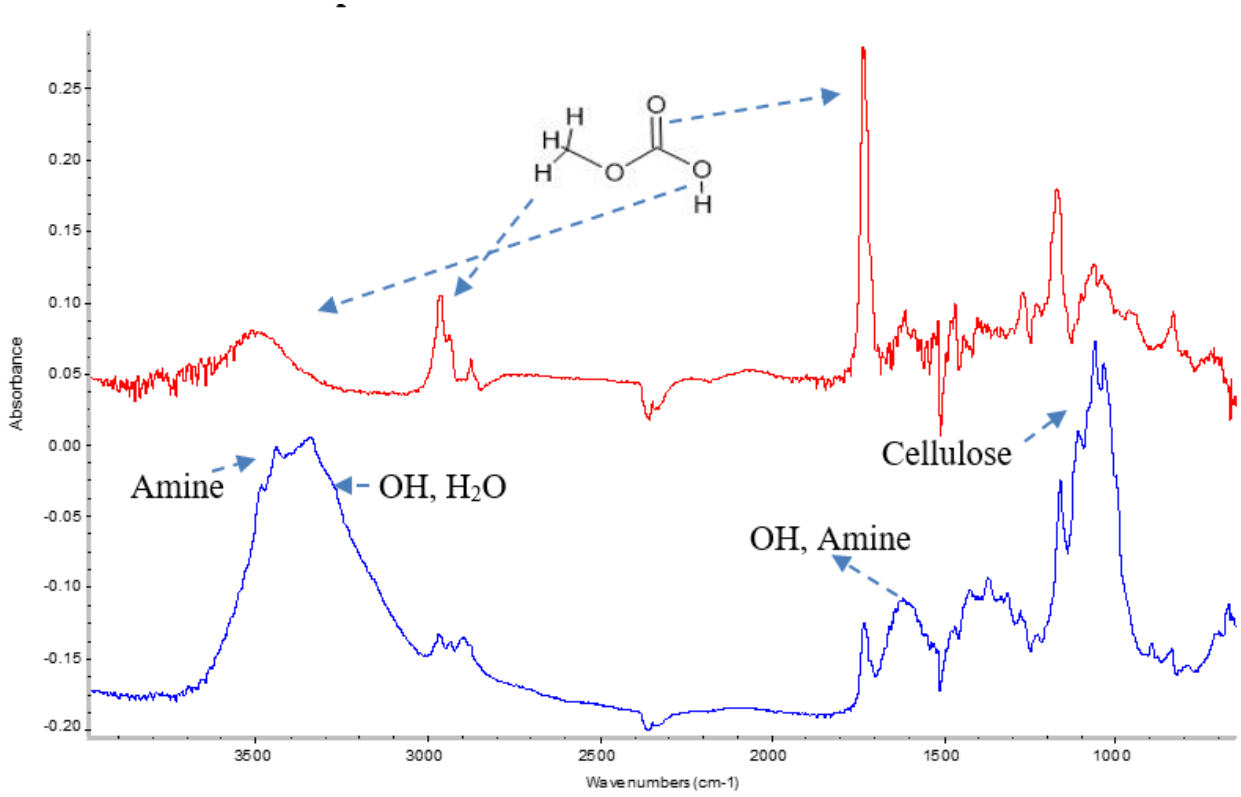

Figure 2. FTIR analysis of the debris found in MCU-20-32 and MCU-20-34.

Modifier Concentrations and Density Measurements

MCU ceased operations in May 22, 2019 (to support transfer line tie-in work for Salt Waste Process Facility or SWPF), and since then, a few SHT samples were sent to the laboratory for analysis. Based on this last sample (September 2020) results, both the density measurement and the Modifier concentration levels were high relative to past samples possibly due to Isopar™ L evaporation from the solvent but their corresponding error intervals included the nominal (0.830 mg/L at 25 °C in the case of the density measurement) and/or recommended value (in the case of the Modifier, the recommended level is 1.69E5 mg/L) [see Figure 3 and the tabulated data in Appendix A].[4] Although not seen in the measurements of this sample, past Modifier measurements indicated that the FTHNMR values had a slight positive bias relative to the HPLC values. (The HPLC method is the standard method for measuring the Modifier.) Using the average of the two methods minimizes any bias effect. The reported density measurements were obtained from triplicate measurements of the sample. The density was measured by the vibrations of a calibrated tube filled with the organic liquid and corrected for temperature using the CSSX temperature correction formula.[4] The uncertainty (one sigma) by this method is 3%.

Despite their high values, the observed density and Modifier values from the September 2020 sample were consistent with previous measurements (for example in January 2018). Both the density data and the Modifier concentration correlated strongly with each other as expected (see Figure 3).[4] Correspondingly, the Isopar™ L concentration (not shown) in the September 2020 sample was slightly below the Isopar™ L concentration of the baseline solvent (scratch made on September 2018). This finding is expected since the solvent density is largely a volume-weighed linear combination of the Modifier and Isopar™ L densities. Other physical measurements of the September 2020 SHT sample such as viscosity and surface tension were trending upwards relative to the baseline solvent measurements (see Figure 4), an indication of continued evaporation. No bias was detected in either the viscosity or surface tension measurements of the monthly solvent samples relative to the scratch baseline solvent. A summary of recent Modifier measurements is shown in Appendix A.

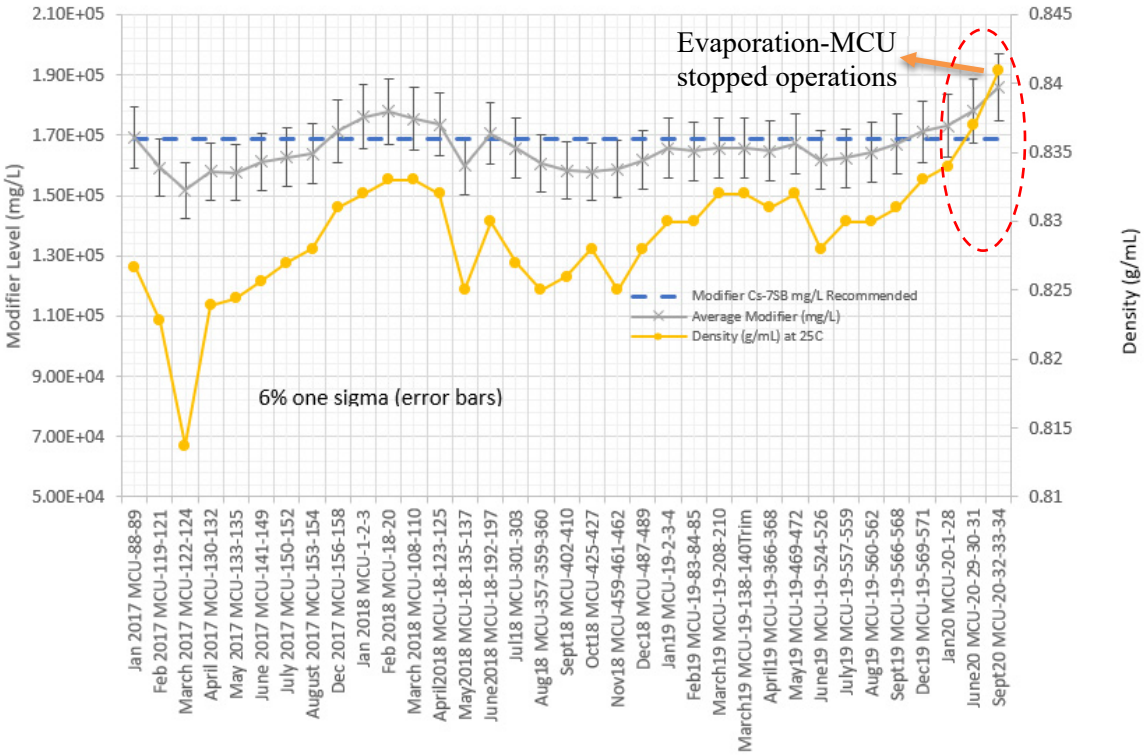

Figure 3. Modifier concentration in the solvent as measured by HPLC (one sigma is 10%).

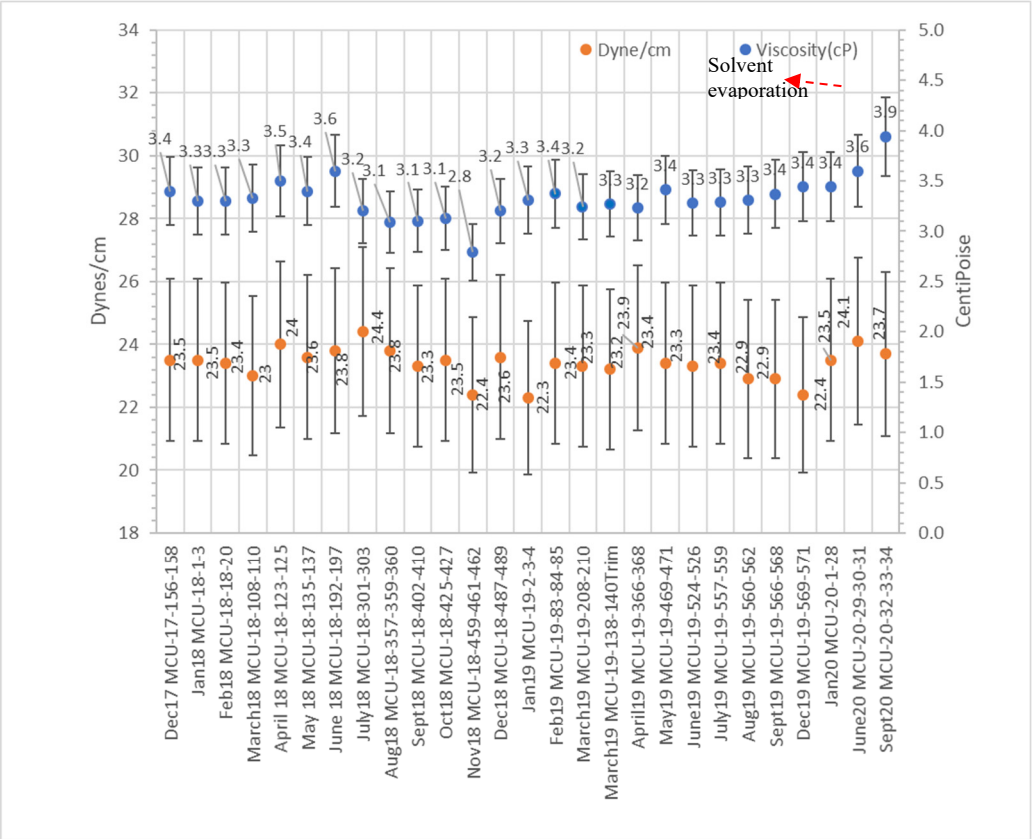

Figure 4. Viscosity and surface tension measurements of the last 27 SHT samples. The scratch blend measured a viscosity of 3 ± 0.3 cP and a surface tension of 23 ± 0.6 dynes/cm (at 25˚C).

Suppressor Concentrations

The average TiDG concentration of MCU-20-32-33-34 is shown in Figure 5. As can be seen in Figure 5, the TiDG concentration has remained steady at approximately 800 ± 30 mg/L since June 2019. Recall MCU stopped operations in May 2019. The TOA concentration appears to have remained steady at 188 ± 30 mg/L. Since May 2016, the TOA level range can be estimated by 188 ± 30 mg/L. Since MCU no longer adds TOA, the persistent detection of TOA at this concentration level is not expected. TOA concentration would be expected to have declined with processing time. We believe the TOA concentration persists because of possible and expected degradation rate 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, and therefore difficult to distinguish.[6]

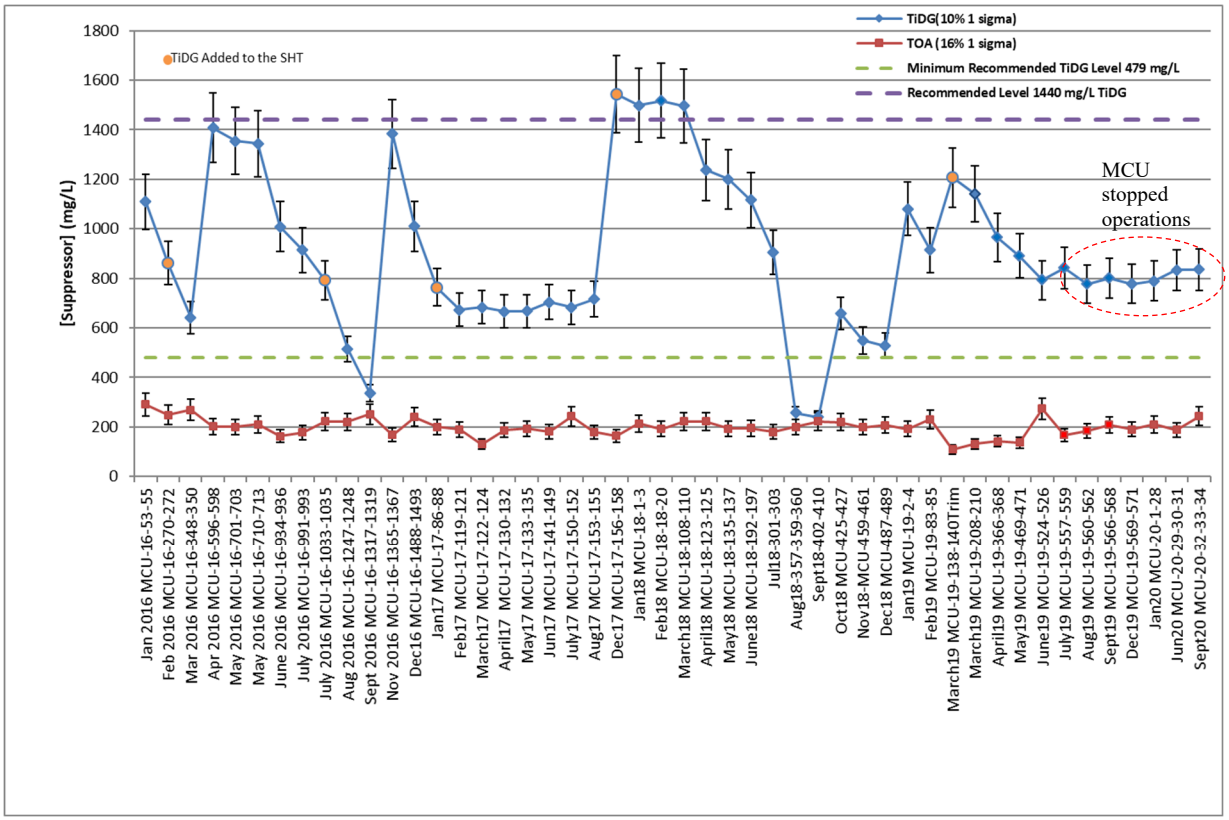

Figure 5. Suppressor concentration as measured by titration in the SHT samples since NGS implementation. The minimum recommended concentration is 479 mg/L for TiDG.

Extractant Concentrations

The calculated MaxCalix concentration for the September 2020 sample was 5.27 E4 mg/L. This measurement is consistent with the evaporation of Isopar™ L since it is part of an upward trend observed in the MaxCalix concentration (see Figure 6). Also note that a positive bias was detected in the FT-HNMR relative to the HPLC method that is minimized by using the average of the two methods. The residuals (difference the actual value and the corresponding recommended value) between the measured Modifier values and its recommended value correlates (correlation coefficient of 0.82) with the residuals from the MaxCalix measurements with its recommended value (see Figure 7). This indicates that both the Modifier and the MaxCalix are miscible and have similar physical-chemical behaviors. A summary of the recent MaxCalix concentration in the solvent is shown in Appendix A.

The residual concentration of BOBCalixC6 concentration was 1.11 E3 mg/L. Since no BOBCalixC6 was added to the SHT, the variability in the BOBCalixC6 concentration data with time is more reflective of the analytical uncertainty (the standard deviation of the BOBCalixC6 concentration since January 2018 is 9.3% which is similar to the 10% method of uncertainty reported by HPLC).

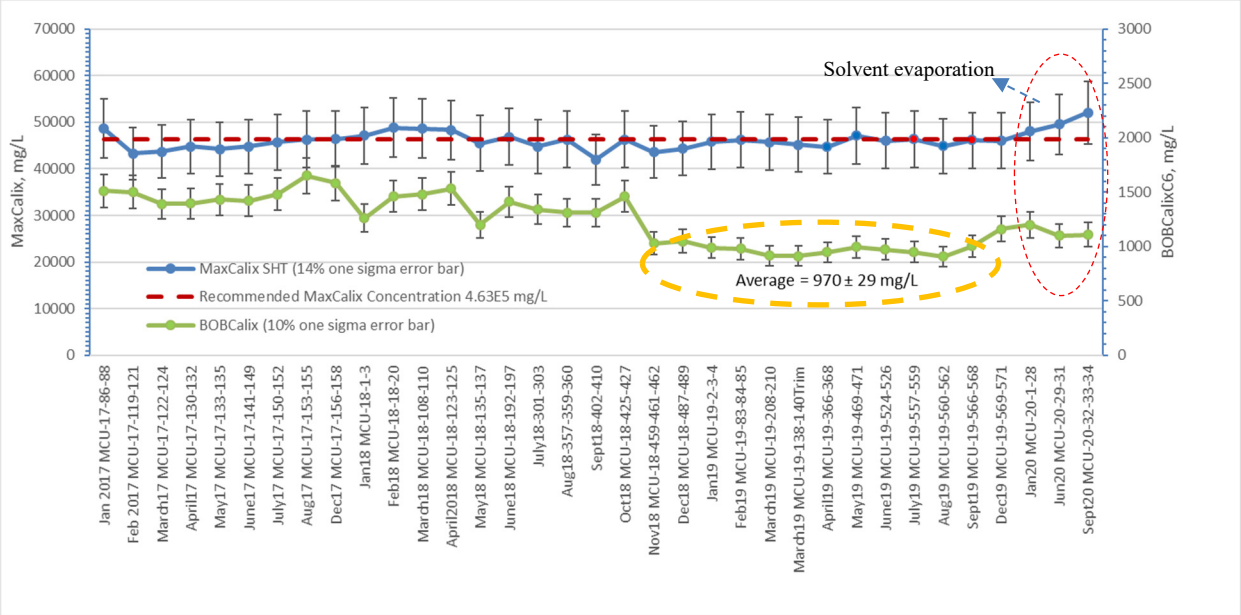

Figure 6. Average MaxCalix concentration from the average of the HPLC and FT-HNMR of recent samples since NGS implementation (46,000 mg/L is the nominal concentration).

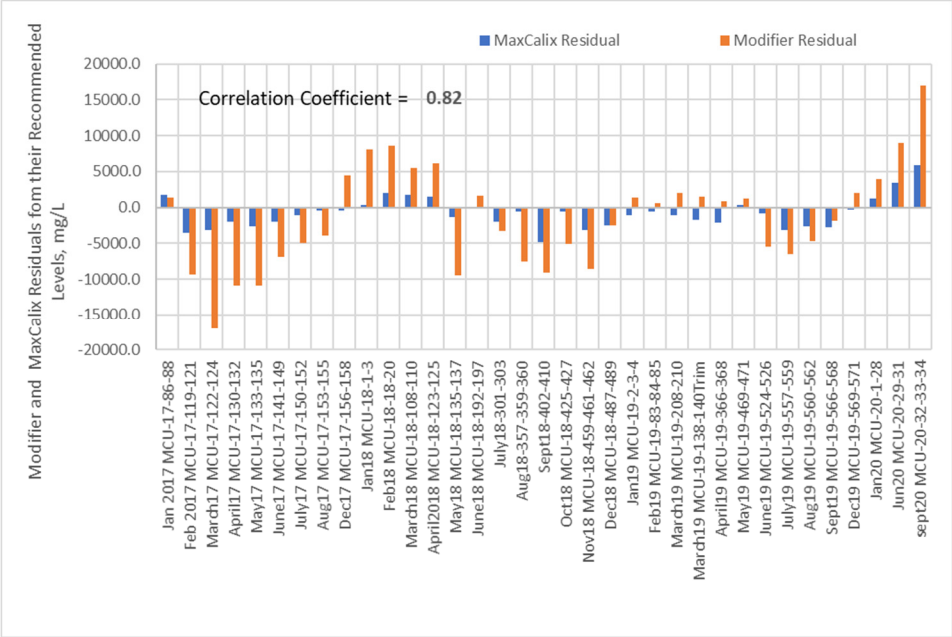

Figure 7. MaxCalix and Modifier residuals from their recommended levels

Gamma Measurements

The gamma measurements for the September 2020 SHT sample was 3.72 E4 dpm/mL and is shown in Figure 8 in relation to past measurements. The gamma measurements have been steadily increasing since MCU stopped operations possibly due to Isopar™ L evaporation.

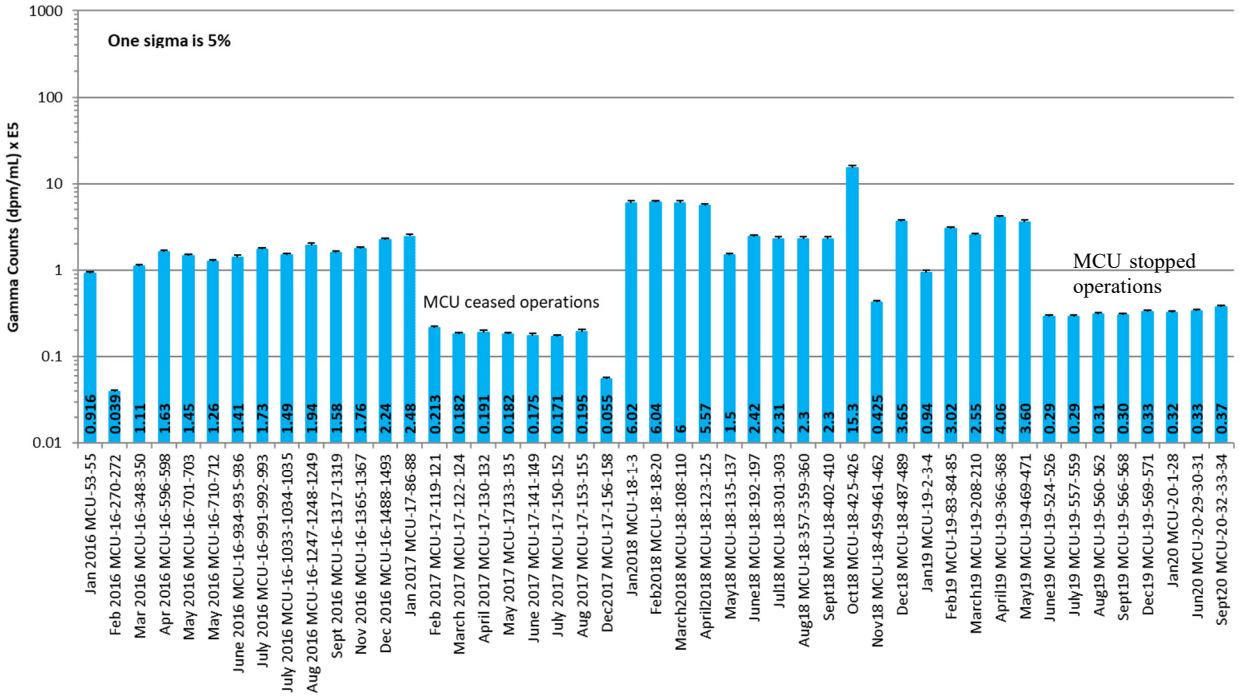

Figure 8. The gamma count of selected SHT samples.

Impurities

No organic impurities were observed in the September 2020 SHT sample from the SVOA and the FT-HNMR analysis.

However, another impurity being tracked in the SHT solvent is the concentration of mercury. A few mL of each sample was digested and analyzed for total mercury by the DMA method. The average mercury concentrations in the September 2020 SHT sample was 22 ± 4 ug/g (see Figure 9). The downward trend (opposite to the upward trend observed with the SHT component mentioned earlier) in the last four mercury measurements is possibly due to MCU being idle. During idle the solvent does not contact tank supernate, a source of mercury, and that the remaining mercury species in the solvent is either volatizing or sorbing on surfaces or precipitating.

The concentration of mercury observed in the December 2019, January 2020, June 2020, and September 2020 samples 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: solids were not observed in these samples) 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.[8,9] Based on the September 2020 SHT sample DMA mercury measurements, for 128 gallons of solvent (484.53 L), the solvent could contain up to 9 ± 2 g of mercury. A summary of recent mercury measurements of the solvent is listed in Appendix A.

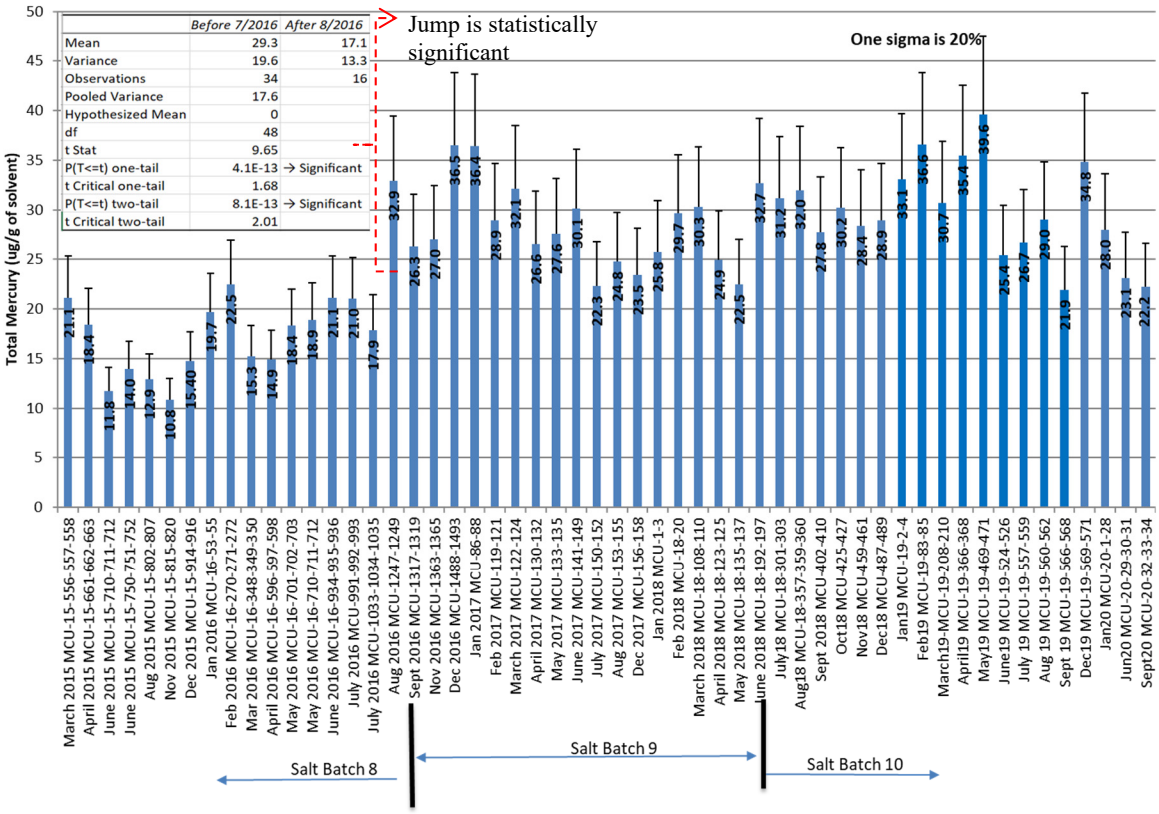

Figure 9. Total mercury in recent SHT samples (one sigma is 20%).

4.0 Conclusions

The final characterization of SHT solvent sample from September 2020 is reported and compared to past measurements. Most of the conclusions are based on the September 2020 SHT sample (MCU-20-32-33-34). Analyses of the September 2020 SHT sample results in relation to the past measurements indicated that the Modifier (Cs-7SB), the extractant (MaxCalix), the suppressor, and the gamma concentration were either steady or increasing due to Isopar™ L evaporation.

The Semi-Volatile Organic Analysis (SVOA) and FT-HNMR did not detect any organic impurities. Another impurity observed in the samples was mercury. Based on the September 2020 SHT sample, up to 22 ± 4 micrograms of mercury per gram of solvent (or 19 ± 4 mg/L) was detected.

The gamma concentration (~3.72 E4 dpm/mL) measured in the September 2020 SHT samples was consistent with previous values observed when MCU was idle (for example, between February 2017 and August 2017).

5.0 References

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Appendix A: SHT samples

Average Modifier concentration in the SHT samples

Sample	Average (mg/L)	HPLC (mg/L)	FT-HNMR (mg/L)	Density (mg/L)
Jan 2017 MCU-88-89	1.68E+05	1.65E+05	1.70E+05	1.70E+05
Feb 2017 MCU-119-121	1.53E+05	1.55E+05	1.51E+05	1.60E+05
March 2017 MCU-122-124	1.51E+05	1.50E+05	1.51E+05	1.52E+05
April 2017 MCU-130-132	1.59E+05	1.58E+05	1.59E+05	1.58E+05
May 2017 MCU-133-135	1.57E+05	1.56E+05	1.57E+05	1.58E+05
June 2017 MCU-141-149	1.58E+05	1.57E+05	1.59E+05	1.62E+05
July 2017 MCU-150-152	1.57E+05	1.56E+05	1.57E+05	1.64E+05
August 2017 MCU-153-154	1.59E+05	1.58E+05	1.59E+05	1.65E+05
Dec 2017 MCU-156-158	1.66E+05	1.63E+05	1.69E+05	1.73E+05
Jan 2018 MCU-1-2-3	1.73E+05	1.72E+05	1.73E+05	1.77E+05
Feb 2018 MCU-18-20	1.75E+05	1.77E+05	1.72E+05	1.78E+05
March 2018 MCU-108-110	1.75E+05	1.78E+05	1.72E+05	1.75E+05
April 2018 MCU-18-123-125	1.75E+05	1.66E+05	1.68E+05	1.75E+05
May 2018 MCU-18-135-137	1.60E+05	1.59E+05	1.53E+05	1.60E+05
June 2018 MCU-18-192-197	1.71E+05	1.68E+05	1.64E+05	1.71E+05
Jul 2018 MCU-301-303	1.63E+05	1.64E+05	1.61E+05	1.66E+05
Aug 2018 MCU-357-359-360	1.52E+05	1.54E+05	1.49E+05	1.62E+05
Sept 2018 MCU-402-410	1.50E+05	1.50E+05	1.49E+05	1.60E+05
Oct 2018 MCU-425-427	1.51E+05	1.40E+05	1.61E+05	1.62E+05
Nov 2018 MCU-459-461-462	1.55E+05	1.52E+05	1.58E+05	1.60E+05
Dec 2018 MCU-487-489	1.56E+05	1.47E+05	1.64E+05	1.65E+05
Jan 2019 MCU-19-2-3-4	1.59E+05	1.51E+05	1.67E+05	1.69E+05
Feb 2019 MCU-19-83-84-85	1.60E+05	1.50E+05	1.69E+05	1.68E+05
March 2019 MCU-19-208-210	1.54E+05	1.48E+05	1.59E+05	1.70E+05
March 2019 MCU-19-138-140Trim	1.52E+05	1.48E+05	1.55E+05	1.70E+05
April 2019 MCU-19-366-368	1.66E+05	1.47E+05	1.57E+05	1.69E+05
May 2019 MCU-19-469-472	1.68E+05	1.55E+05	1.59E+05	1.70E+05
June 2019 MCU-19-524-526	1.62E+05	1.55E+05	1.61E+05	1.63E+05
July 2019 MCU-19-557-559	1.63E+05	1.47E+05	1.57E+05	1.66E+05
Aug 2019 MCU-19-560-562	1.65E+05	1.52E+05	1.63E+05	1.67E+05
Sept 2019 MCU-19-566-568	1.67E+05	1.57E+05	1.61E+05	1.69E+05
Dec 2019 MCU-19-569-571	1.71E+05	1.65E+05	1.69E+05	1.72E+05
Jan 2020 MCU-20-1-28	1.73E+05	1.67E+05	1.71E+05	1.74E+05
Jun 2020 MCU-20-29-31	1.78E+05	1.76E+05	1.71E+05	1.79E+05
Sept 2020 MCU-20-32-33-34	1.87E+05	1.85E+05	1.86E+05	1.86E+05

Viscosity and surface tension of the SHT samples

SHT sample (Rheology)	Viscosity (cP)	Surface Tension (Dyne/cm)	Control Viscosity (cP)	Control Surface Tension (dyne/cm)
Dec 2017 MCU-17-156-158	3.4	23.5	3.1	22.4
Jan 2018 MCU-18-1-3	3.3	23.5	3.08	22.9
Feb 2018 MCU-18-18-20	3.3	23.4	3.08	23.44
March 2018 MCU-18-108-110	3.3	23	3.28	22.6
April 2018 MCU-18-123-125	3.5	24	3.47	22.9
May 2018 MCU-18-135-137	3.4	23.6	3.6	23.7
June 2018 MCU-18-192-197	3.6	23.8	3.6	23.6
July 2018 MCU-18-301-303	3.2	24.4	4	24
Aug 2018 MCU-18-357-359-360	3.1	23.8	3.16	24
Sept 2018 MCU-18-402-410	3.1	23.3	3.11	23.2
Oct 2018 MCU-18-425-427	3.1	23.5	3.15	23
Nov 2018 MCU-18-459-461-462	2.8	22.4	3.12	22.5
Dec 2018 MCU-18-487-489	3.2	23.6	3.17	23.8
Jan 2019 MCU-19-2-3-4	3.3	22.3	3.17	23.7
Feb 2019 MCU-19-83-84-85	3.4	23.4	3.2	23.2
March 2019 MCU-19-208-210	3.2	23.3	3.18	22.9
March 2019 MCU-19-138-140 Trim	3.3	23.2	3.21	23.4
April 2019 MCU-19-366-368	3.2	23.9	3.17	23.3
May 2019 MCU-19-469-471	3.4	23.4	3.19	22.9
June 2019 MCU-19-524-526	3.3	23.3	3.21	23.4
July 2019 MCU-19-557-559	3.3	23.4	3.19	23.4
Aug 2019 MCU-19-560-562	3.3	22.9	3.19	23.4
Sept 2019 MCU-19-566-568	3.4	22.9	3.2	22.9
Dec 2019 MCU-19-569-571	3.4	22.4	3.18	22.4
Jan 2020 MCU-20-1-28	3.4	23.5	3.16	23.4
Jun 2020 MCU-20-29-31	3.6	24.1	3.18	22.4
Sept 2020 MCU-20-32-33-34	3.9	23.7	3.26	23.4

Average MaxCalix concentration in the SHT samples

SHT Sample (MaxCalix)	HPLC (mg/L)	HNMR (mg/L)	Average (mg/L)
Jan 2017 MCU-17-86-88	4.82E+04	49300	48597
Feb 2017 MCU-17-119-121	4.30E+04	43700	43255
March 2017 MCU-17-122-124	4.33E+04	44300	43661
April 2017 MCU-17-130-132	4.41E+04	46000	44769
May 2017 MCU-17-133-135	4.39E+04	44700	44191
June 2017 MCU-17-141-149	4.42E+04	45800	44768
July 2017 MCU-17-150-152	4.58E+04	45600	45725
Aug 2017 MCU-17-153-155	4.65E+04	46000	46312
Dec 2017 MCU-17-156-158	4.61E+04	46900	46391
Jan 2018 MCU-18-1-3	4.64E+04	48300	47071
Feb 2018 MCU-18-18-20	4.83E+04	49600	48767
March 2018 MCU-18-108-110	4.89E+04	48100	48596
April 2018 MCU-18-123-125	4.84E+04	48100	48288
May 2018 MCU-18-135-137	4.63E+04	44200	45473
June 2018 MCU-18-192-197	4.67E+04	47100	46847
July 2018 MCU-301-303	4.50E+04	44400	44773
Aug 2018 MCU-357-359-360	45200	48400	46289
Sept 2018 MCU-402-410	4.28E+04	40700	41969
Oct 2018 MCU-18-425-427	4.52E+04	48400	46289
Nov 2018 MCU-18-459-461-462	4.18E+04	47600	43617
Dec 2018 MCU-18-487-489	4.24E+04	48500	44300
Jan 2019 MCU-19-2-3-4	44800	47600	45763
Feb 2019 MCU-19-83-84-85	44900	48800	46202
March 2019 MCU-19-208-210	4.42E+04	48800	45703
March 2019 MCU-19-138-140 Trim	4.34E+04	48800	45122
April 2019 MCU-19-366-368	4.22E+04	50700	44671
May 2019 MCU-19-469-471	4.45E+04	53300	47070
June 2019 MCU-19-524-526	4.32E+04	53300	46027
July 2019 MCU-19-557-559	4.36E+04	53300	46351
Aug 2019 MCU-19-560-562	4.41E+04	46200	44836
Sept 2019 MCU-19-566-568	4.40E+04	50700	46065
Dec 2019 MCU-19-569-571	4.53E+04	48800	46482
Jan 2020 MCU-20-1-28	4.61E+04	57700	49280
Jun 2020 MCU-20-29-31	4.84E+04	53900	50177
Sept 2020 MCU-20-32-33-34	5.10E+04	56000	52019

Mercury concentration measured by the DMA and XRF Methods

SHT Sample (mercury)	DMA (ug/g)	XRF (ug/g)
April 2018 MCU-18-123-125	24.40	25.44
May 2018 MCU-18-135-137	19.90	25.11
June 2018 MCU-18-192-197	33.20	32.15
July 2018 MCU-18-301-303	28.60	33.75
Aug 2018 MCU-18-357-359-360	26.20	37.79
Sept 2018 MCU-402-410	26.70	28.82
Oct 2018 MCU-425-427	28.00	32.4
Nov 2018 MCU-459-461	25.90	30.8
Dec 2018 MCU-487-489	25.80	32.0
Jan 2019 MCU-19-2-4	25.10	41.0
Feb 2019 MCU-19-83-85	38.50	34.61
March 2019-MCU-19-208-210	29.00	32.43
April 2019 MCU-19-366-368	33.70	37.16
May 2019 MCU-19-469-471	37.40	41.8
June 2019 MCU-19-524-526	25.40	NM
July 2019 MCU-19-557-559	26.70	NM
Aug 2019 MCU-19-560-562	29.00	NM
Sept 2019 MCU-19-566-568	21.90	NM
Dec 2019 MCU-19-569-571	34.80	NM
Jan 2020 MCU-20-1-28	28.00	NM
Jun 2020 MCU-20-29-31	23.10	NM
Sept 2020 MCU-20-32-34	22.20	NM

NM = Not Measured