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Solvent Hold Tank Sample Results for MCU-11-1452, MCU-11-1453, MCU-11-1454, MCU-11-1455, MCU-11-1456 and MCU-11-1457

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed solvent samples from Modular Caustic-Side Solvent Extraction Unit (MCU) in support of continuing operations. A quarterly analysis of the solvent is required to maintain solvent composition within specifications. Analytical results of the analyses of Solvent Hold Tank (SHT) samples MCU-11-1452, MCU-11-1453, MCU-11-1454, MCU-11-1455, MCU-11-1456 and MCU-11-1457 are reported.

The results show that the solvent at MCU does not require an Isopar® L addition, but it will require addition of trioctylamine.

LIST OF ABBREVIATIONS

FTIR – Fourier transform infra-red spectroscopy
HPLC – High Performance Liquid Chromatography
ISDP – Integrated Salt Disposition Project
RSD – residual standard deviation
SHT – Solvent Hold Tank
SRNL – Savannah River National Laboratory
SVOA – Semi Volatile Organic Analysis
TOA - trioctylamine

1.0 Introduction

Solvent Hold Tank (SHT) samples are sent to Savannah River National Laboratory (SRNL) to examine solvent composition changes over time.[1] On December 5, 2011, Operations personnel delivered six samples from the SHT (MCU-11-1452 through -1457) for analysis. These samples are intended to verify that the solvent is within the specified composition range. The results from the analyses are presented in this document.

2.0 Experimental Procedure

Samples were received in p-nut vials containing ~10 mL each. Once taken into the Shielded Cells, the samples were combined. Samples were removed for analysis by density, semi-volatile organic analysis (SVOA), high performance liquid chromatography (HPLC), and Fourier-Transform Infra-Red spectroscopy (FTIR).

Details for the work are contained in a controlled laboratory notebook.[2]

3.0 Results and Discussion

Each of the six p-nut vials contained a single phase, with no apparent solids contamination or cloudiness. Table 1 contains the results of the analyses for the combined samples.

A duplicate density measurement of the organic phase gave a result of 0.844 g/mL (1.2% residual standard deviation - RSD). Using the density as a starting point, we know that the Isopar® L should be slightly higher than nominal and the other components should be slightly lower than nominal.

The results as a whole are internally consistent. All measurements indicate Isopar® L higher than nominal, and Modifier lower than nominal. The extractant result is higher than expected – given the other results, the extractant concentration should be under nominal values. Using the measured density as well as the Isopar® L and Modifier concentrations from the FTIR results, we calculate an extractant concentration of 6888 mg/L. This value is outside the analytical uncertainty of the reported HPLC value. Given the other results, this most likely indicates that the HPLC extractant result was biased high.

When compared to the MCU density target of 0.845 g/mL, there is no need to add an Isopar® L trim.[♣] However, it is advisable to add sufficient trioctylamine (TOA) to return

[♣] Note that while freshly prepared MCU solvent has a target density of 0.852 g/mL, the MCU facility targets tries to maintain the solvent inventory at 0.845 g/mL in order to prevent over-evaporation.

the solvent composition to within specifications as that component has declined to about 64% the concentration since the last analysis. The TOA measurement was performed twice, so the result is not an analytical aberration. TOA has not been added to the system since the previous quarterly sample in October 2011.

Table 1. Sample Results for MCU-11-1452/1453/1454/1455/1456/1457 Composite

Analysis	Method	LIMS #	Result (mg/L)[#]	Nominal[*] Result (mg/L)	% of (Result ÷ Nominal Result)
Isopar® L	SVOA	300295714	665,000	589,000	113%
Isopar® L	FTIR	NA	610,000	589,000	104%
Isopar® L	Density [ϒ]	NA	600,000	589,000	102%
average	all	NA	625,000	589,000	106%
Modifier	SVOA	300295714	230,000	254,000	90.6%
Modifier	HPLC	300295714	234,000	254,000	92.1%
Modifier	FTIR	NA	221,000	254,000	87.0%
Modifier	Density [ϒ]	NA	235,000	254,000	92.5%
average	all	NA	230,000	254,000	90.6%
trioctylamine	SVOA	300295714	655	1,020	64.2%
Extractant	HPLC	300295714	8,400	8,000	105%
Density	Direct measurement	NA	0.844	0.852 g/mL	0.991%

[#] Analytical uncertainty is 20% for SVOA and 10% for HPLC. FTIR analytical uncertainty is 15% for Isopar® L and 10% for Modifier. Density results from the average of replicate volumetric trials typically have a percentage standard deviation of <1% between each value and the average.

[*] Nominal value is the expected value for freshly prepared solvent with a target density = 0.852 g/mL.[3]

NA = not applicable

[ϒ] We can estimate the Isopar® L and Modifier concentrations by knowing the densities of the individual components and using the Microsoft Excel goal seek function to assess a range of Isopar® L, Modifier and TOA compositions to arrive at the measured density.

4.0 Conclusions

As with the previous solvent sample results,[4] these analyses indicate that the solvent does not require Isopar® L trimming at this time. However, addition of TOA is warranted. These findings indicate that the new protocols for solvent monitoring and control are yielding favorable results. Nevertheless, the deviation in the TOA concentration since the last analysis indicates continued periodic (i.e., quarterly) monitoring is recommended.

5.0 References

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