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# Results from the Salt Disposition Project (SDP) Next Generation Solvent (NGS) Demonstration Plan

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## EXECUTIVE SUMMARY

Strip Effluent Hold Tank (SEHT), Decontaminated Salt Solution Hold Tank (DSSHT), Caustic Wash Tank (CWT) and Solvent Hold Tank (SHT) samples were taken throughout the Next Generation Solvent (NGS) Demonstration Plan. These samples were analyzed and the results are reported.

- SHT: The solvent behaved as expected, with no bulk changes in the composition over time, with the exception of the TOA and TiDG. The TiDG depletion is higher than expected, and consideration must be taken on the required rate of replenishment. Monthly sampling of the SHT is warranted. If possible, additional SHT samples for TiDG analysis (only) would help SRNL refine the TiDG degradation model.
- CWT: The CWT samples show the expected behavior in terms of bulk chemistry. The  $^{137}\text{Cs}$  deposited into the CWT varies somewhat, but generally appears to be lower than during operations with the BOBCalix solvent. While a few minor organic components were noted to be present in the Preliminary sample, at this time these are thought to be artifacts of the sample preparation or may be due to the preceding solvent superwash.
- DSSHT: The DSSHT samples show the predicted bulk chemistry, although they point towards significant dilution at the front end of the Demonstration. The  $^{137}\text{Cs}$  levels in the DSSHT are much lower than during the BOBCalix operations, which is the expected observation.
- SEHT: The SEHT samples represent the most different output of all four of the outputs from MCU. While the bulk chemistry is as expected, something is causing the pH of the SEHT to be higher than what would be predicted from a pure stream of 0.01 M boric acid. There are several possible different reasons for this, and SRNL is in the process of investigating. Other than the pH issue, the SEHT is as predicted.

In summary, the NGS Demonstration Plan samples indicate that the MCU system, with the Blend Solvent, is operating as expected. The only issue of concern regards the pH of the SEHT, and SRNL is in the process of investigating this. SRNL results support the transition to routine operations.

## TABLE OF CONTENTS

LIST OF TABLES .....	vii
LIST OF FIGURES .....	vii
1.0 Introduction.....	1
2.0 Experimental Procedure.....	3
2.1 Quality Assurance .....	4
3.0 Results and Discussion .....	4
3.1 SEHT Sample Results .....	4
3.1.1 F/H Lab Outage .....	5
3.1.2 Density Measurements.....	5
3.1.3 Considerations of pH Impacts on DWPF .....	6
3.1.4 Considerations of Sodium Impacts on DWPF .....	7
3.2 DSSHT Sample Results.....	8
3.3 CWT Sample Results .....	9
3.4 SHT Sample Results.....	12
3.4.1 Consideration of the TiDG Depletion Rate .....	16
3.4.2 <sup>137</sup> Cs in the SHT samples .....	17
3.5 Strip Feed Sample Results.....	18
3.6 Scrub Feed Sample Results .....	19
3.7 Comparison between F/H and SRNL Sample Results .....	19
3.8 Brief Discussion of the Decontamination and Concentration Factors .....	21
4.0 Conclusions.....	22
5.0 Path Forward.....	23

## LIST OF TABLES

Table 1. Nominal Component Concentration in the Three Solvents .....	1
Table 2. Analyses on Each Type of Sample .....	2
Table 3. Operating Conditions for Different Samples .....	3
Table 4. Radiochemical Results for the SEHT Samples .....	4
Table 5. Isopar™ L Results From SEHT Samples .....	5
Table 6. Density Measurements of the SEHT Samples .....	5
Table 7. pH Results for Demonstration Plan SEHT Samples .....	6
Table 8. TIC/TOC Measurements of Selected SEHT Samples .....	6
Table 9. Average Sodium Content of Previous Salt Batch 4, 5, and 6 .....	7
Table 10. Radiochemical Results for the DSSHT Samples .....	8
Table 11. Selected Non-Radiochemical Results for DSSHT Samples .....	9
Table 12. <sup>137</sup> Cs Results for the CWT samples .....	10
Table 13. Selected Non-Radiochemical Analyses on CWT Samples .....	11
Table 14. SVOA Analysis of the Preliminary CWT Sample .....	12
Table 15. All Analyses for SHT Samples .....	14
Table 16. TiDG Loss Rates .....	17
Table 17. <sup>137</sup> Cs in the SHT Samples .....	17
Table 18. Results of Analyses of Microbatch 5 and 6a Strip Feed .....	18
Table 19. Results of Analyses of Microbatch 6c Scrub Feed .....	19
Table 20. Comparison of <sup>137</sup> Cs Results between SRNL and F/H Lab .....	20
Table 21. Free Hydroxide Results for Duplicate SRNL and F/H Samples .....	20
Table 22. DF and CF Values for Demonstration Plan Samples .....	21

## LIST OF FIGURES

Figure 1. Concentration Factor over the Range of Samples .....	22
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## LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CF	Concentration Factor
CWT	Caustic Wash Tank
DF	Decontamination Factor
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICPES	Inductively-Coupled Plasma Emission Spectroscopy
ICPMS	Inductively-Coupled Plasma Mass Spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
MST	monosodium titanate
PuTTa	Plutonium thenoyl trifluoroacetone scintillation
RSD	Relative standard deviation
SE	Strip Effluent
SEHT	Strip Effluent Hold Tank
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon
TOA	trioctylamine
TiDG	N, N', N''- tris(isotridecyl)guanidine

## 1.0 Introduction

The MCU process previously operated with a four component solvent which removes cesium from clarified salt solution. The previous solvent, designated BOBCalixC6 Based Solvent (or “BOB Solvent”), is comprised of BOBCalixC6 (extractant), trioctylamine (TOA) (suppressor), Cs-7SB (modifier), and Isopar™ L (diluent). NGS utilizes a new extractant and suppressor in MCU; it is a four component mixture comprised of MaxCalix (extractant), N, N', N''- tris(isotridecyl)guanidine (TiDG, the suppressor), Cs-7SB (modifier), and Isopar™ L (diluent). The NGS solvent components were introduced to the MCU system in a “concentrate” and blended with the residual volume (95 gallons) of BOBCalixC6 Based Solvent to create the NGS Blend.<sup>i</sup>

**Table 1. Nominal Component Concentration in the Three Solvents**

Component	Nominal component concentration in the three Solvents (mg/L)		
	BOB Solvent	NGS Blend	NGS
BOBCalix	8050	4030	NA
MAXCalix	NA	44400	47800
Modifier	254000	169000	169000
TOA	1060	530	NA
TiDG	NA	1550	1550
Isopar™ L	balance	balance	balance

After NGS implementation with the NGS Blend, all solvent additions will consist of only NGS constituents. Therefore, the operating solvent will gradually approach the final “NGS” concentrations indicated in Table 1. The NGS Blend composition in Table 1 is a projected estimate that assumes the residual BOBCalix solvent was mixed with an equal volume of concentrate. The actual component concentrations in the Blend solvent depends on the volume of the operating heel of solvent at time of transition and thus, may vary slightly from the estimated composition.

In order to transition from the BOB Solvent to the NGS Blend, the MCU facility is undergoing a period of deliberate operations deemed the NGS Demonstration. A Preliminary sample and six micro-batches (1, 2a/2, 3, 4, 5, and 6a/6b/6c) of clarified salt solution were processed during the Demonstration Period. Each microbatch was operated at a prescribed flow rate until the DSSHT was full. During the Demonstration Period the MCU was operated under a sample-and-hold strategy. After each microbatch, samples were taken from key process vessels; the Strip Effluent Hold Tank (SEHT), the Decontaminated Salt Solution Hold Tank (DSSHT), the Solvent Hold Tank (SHT) and the Caustic Wash Tank (CWT). In addition, samples from the scrub feed tank (0.025 M NaOH) and strip feed tank (0.01 M boric acid) were analyzed as needed. The analyses performed on each sample varied depending on the particular sample (see Table 2).<sup>ii</sup> Some samples were sent to SRNL and some were sent to F/H lab. Results from the F/H

lab samples are not generally reported in this document, unless otherwise required for comparison.

**Table 2. Analyses on Each Type of Sample**

Sample	Types of Analyses for Each Sample			
	<b>SEHT</b>	<b>DSSHT</b>	<b>CWT</b>	<b>SHT</b>
Preliminary	<i><sup>137</sup>Cs, PuTTa, <sup>90</sup>Sr, ICPMS, SVOA, density, pH</i>	<i><sup>137</sup>Cs, PuTTa, <sup>90</sup>Sr, ICPMS, SVOA, Free OH, density</i>	<sup>137</sup> Cs, SVOA, HPLC, pH	solvent, <sup>137</sup> Cs, density
Microbatch 1	<i><sup>137</sup>Cs, PuTTa, <sup>90</sup>Sr, ICPES, SVOA, density, pH</i>	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH, ICPES, SVOA, density	solvent, <sup>137</sup> Cs, density
Microbatch 2 (Received two sets of samples, deemed 2a and 2)	<i><sup>137</sup>Cs, ICPES, SVOA, density, pH</i>	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH, ICPES, SVOA, density	solvent, <sup>137</sup> Cs, density
Microbatch 3	<i><sup>137</sup>Cs, ICPES, SVOA, density, pH</i>	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH, ICPES, SVOA, density	solvent, <sup>137</sup> Cs, density
Microbatch 4	<i><sup>137</sup>Cs, ICPES, SVOA, density, pH</i>	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH, ICPES, SVOA, density, Free OH	solvent, <sup>137</sup> Cs, density
Microbatch 5	<sup>137</sup> Cs, ICPES, SVOA, density, pH, TIC/TOC, carbonate	<sup>137</sup> Cs, SVOA, Free OH, density	<sup>137</sup> Cs, pH, ICPES, SVOA, density, Free OH	solvent, <sup>137</sup> Cs, density
Microbatch 6a	<sup>137</sup> Cs, ICPES, SVOA, density, TIC/TOC, carbonate, pH	<sup>137</sup> Cs, SVOA, Free OH, density	No sample	solvent, <sup>137</sup> Cs, density
Microbatch 6b	<sup>137</sup> Cs, ICPES, SVOA, density, TIC/TOC, carbonate, pH	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH	solvent, <sup>137</sup> Cs, density
Microbatch 6c	<i><sup>137</sup>Cs, ICPES, SVOA, density, TIC/TOC, carbonate, pH</i>	<i><sup>137</sup>Cs, SVOA, Free OH, density</i>	<sup>137</sup> Cs, pH	solvent, <sup>137</sup> Cs, density

Samples in italic were run at F/H and not necessarily reported in this document. “Solvent” type analysis means separate analyses were run for Isopar™ (Semi-Volatile Organic Analysis - SVOA), Modifier (High performance Liquid Chromatography - HPLC, SVOA), Trioctylamine - TOA, BOB/MaxCalix (HPLC) and TiDG (titration).

In switching to the NGS, MCU also implemented the NGS flow sheet, which changed some process chemistry and operating parameters. Therefore, the hydraulic stability and the solvent efficiency was evaluated at the flow rates seen in Table 3.

**Table 3. Operating Conditions for Different Samples**

<b>Sample</b>	<b>Salt Solution Feed Rate (gpm)</b>
Preliminary	NA
Microbatch 1	4
Microbatch 2a/2	6
Microbatch 3	8
Microbatch 4	7.5
Microbatch 5	8.5
Microbatch 6a	6
Microbatch 6b	5
Microbatch 6c	8.5

During Microbatch 2, a process upset caused by an extraction contactor spinning in reverse caused MCU to be shutdown prematurely. A complete set of process vessel samples (i.e. “microbatch 2 samples”) were pulled to confirm the integrity of the corresponding tank material. Upon correction of the mechanical issue MCU was restarted to finish the microbatch 2 run and then resampled per standard NGS Demonstration requirements. During Microbatch 5 and Microbatch 6a additional analysis was requested on the DSSHT and SEHT samples as a results of an unexpected outage at F/H lab.

MCU was required to complete five micro-batches at prescribed flow rates per the NGS Demonstration Plan. Due to mechanical issues experienced during Microbatch 2, MCU was expected to stop sampling after the sixth microbatch. However, due to the increased strip feed flow rate issue experienced in Microbatch 6, coupled with the required startup time to reach the last prescribed flow rate (8.5 GPM), the decision was made to restart the micro-batch with only the DSSHT heel. The complete working volume of the DSSHT would allow for more sufficient demonstration time at 8.5 GPM; therefore, this series was labeled as Microbatch 6a/6b/6c.

## **2.0 Experimental Procedure**

The samples arriving at SRNL were contained in 10-mL P-nut vials. SEHT samples were delivered in doorstops for shielding purposes. SHT samples were delivered in a new sample holder. The CWT and DSSHT samples were delivered in thief holders.

Samples of the scrub and strip feeds were delivered in 250 mL poly bottles. All samples were sent for analysis without dilution or filtration. pH measurements were provided by a pH probe, which was calibrated at pH 4, 7, and 10 before use. Density measurements used a calibrated 2 mL density tube.

### 2.1 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

### 3.1 SEHT Sample Results

Most of the SEHT sample analyses were performed at F/H lab. SRNL SEHT analytical results are reported in Tables 4 – 6. The  $^{137}\text{Cs}$  activity and the Isopar L concentration are especially of interest in demonstrating NGS processability. The pH probe and density measurements were performed in the Shielded Cells. For those SE samples sent for SVOA analysis, the samples were prepared in the Shielded Cells as described in section 3.1.1.

The  $^{137}\text{Cs}$  activity and other radioisotope results are reported in Table 4.

**Table 4. Radiochemical Results for the SEHT Samples**

Sample	dpm/mL		
	$^{137}\text{Cs}$	$^{238}\text{Pu}$	$^{90}\text{Sr}$
Prelim	NA	<6.74	5.35E+01 (31.1%)
Microbatch 1	1.55E+08	<6.07	6.49E+01 (25.7%)
Microbatch 2a/2	NA	NA	NA
Microbatch 3	NA	NA	NA
Microbatch 4	NA	NA	NA
Microbatch 5	1.74E+09	NA	NA
Microbatch 6a	1.65E+09	NA	NA
Microbatch 6b	1.09E+09	NA	NA
Microbatch 6c	NA	NA	NA
SB6 Feed	1.30E+08	2.91E+04	4.14E+05

The analytical uncertainty on the  $^{137}\text{Cs}$  samples is 5%. Otherwise, the analytical uncertainty is listed in parentheses next to the reported value.

The Pu results are very comparable to previous Salt Batch 6 routine samples.<sup>iii</sup> This is not surprising given that the change in solvent should have no effect on the Pu in the SE. The prelim <sup>90</sup>Sr value is previously reported,<sup>iii</sup> and the microbatch 1 sample is comparable. The <sup>137</sup>Cs is also roughly comparable to previously reported values, which may seem surprising given the expected increased performance in <sup>137</sup>Cs removal of the NGS Blend solvent. However, the reader must recall that the increased performance (decontamination factor) is manifested in the further decline in the DSSHT <sup>137</sup>Cs values (compared to the BOBCalix solvent). Those values are numerically small compared to the large SEHT <sup>137</sup>Cs values, and so a large increase in the SEHT <sup>137</sup>Cs values was not predicted.

### 3.1.1 F/H Lab Outage

At two points during the Demonstration Plan, F/H lab experienced an outage. SRNL was able to analyze the SEHT samples during that time, which would normally have been sent to F/H lab. In the shielded cells, the samples were prepared by using 3 mL of hexane to extract the organic compounds from the 10 mL samples. These 3 mL SEHT extracts were analyzed for Isopar™ L content. The results are reported in Table 5.

**Table 5. Isopar™ L Results From SEHT Samples**

Sample	Isopar™ L (mg/L)
Microbatch 5	<7
Microbatch 6a	<7

In both cases, less than detectable amounts of Isopar™ L were measured.

### 3.1.2 Density Measurements

The densities of selected SEHT samples were measured. See Table 6.

**Table 6. Density Measurements of the SEHT Samples**

Sample	Density (g/mL)
Microbatch 5	1.00 (25 °C)
Microbatch 6a	0.994 (13.4 °C)
Microbatch 6b	0.964 (19 °C)
Microbatch 6c	0.992 (15.1 °C)

The density of pure 0.01 M boric acid should be ~1.00 g/mL. The measured deviations from 1.00 are indicative of the difficulty in measuring density in the shielded cells.

3.1.3 *Considerations of pH Impacts on DWPF*

The SE is required to have a pH between 2 and 8.<sup>iv</sup> However, starting with the Microbatch 4 samples, all the samples were measured to have a pH higher than 8, with one as high as 8.7. See Table 7.

**Table 7. pH Results for Demonstration Plan SEHT Samples**

sample	pH
Microbatch 1	6.8
Microbatch 2	7.7
Microbatch 3	6.4
Microbatch 4	8.3
Microbatch 5	8.7
Microbatch 6a	8.6
Microbatch 6b	8.3
Microbatch 6c	8.2

The uncertainty of the SRNL pH probe is 0.1 pH units and 1.6% for the F/H lab probe.

The pH values were measured at F/H lab (except for Microbatch 5), but reported here for continuity. F/H lab used a pH probe for the measurement. In addition, SRNL measured the pH of the Microbatch 6c sample by pH probe and also found the results to be 8.3.

This triggered additional work scope at SRNL in order to determine the reason for the elevated pH results, and the effect it would have on DWPF. First, SRNL studied the pH impacts and issued two white papers on the subject.<sup>v,vi</sup> Second, SRNL is following a task technical request<sup>vii</sup> to cover future experiments that investigate this issue. Finally, the customer and SRNL agreed to additional sample analyses of selected SEHT microbatch samples coming to SRNL. Microbatch 5, 6a, 6b and 6c SEHT samples were analyzed by Total Inorganic Carbon/Total Organic Carbon (TIC/TOC). See Table 8.

**Table 8. TIC/TOC Measurements of Selected SEHT Samples**

Sample	Total Inorganic Carbon (mg/L)	Total Organic Carbon (mg/L)
Microbatch 5	26.3	47.1
Microbatch 6a	21.6	49.6
Microbatch 6b	27.8	50.5
Microbatch 6c	34.4	50.6

The analytical uncertainties of the TIC/TOC measurements are 10%. The Microbatch 5 result was previously reported.<sup>viii</sup>

The TIC result is considered to correspond to carbonate, and once the TIC results are converted to carbonate, would result in 0.0022, 0.0018, 0.0023 and 0.0029 M of carbonate, respectively for Microbatch 5, 6a, 6b, and 6c. This is perhaps coincidentally the same amount of base estimated to be required to titrate a solution of 0.01 M boric acid to a pH of ~8.7.<sup>viii</sup> The organic carbon could correspond to formate or oxalate or actual organic material.

Without corroboration with experimental data, it is difficult to say what precisely is causing the pH to be elevated past the theoretical value for 0.01 M boric acid (5.7), a more in-depth discussion is provided in one of the previously completed white papers.<sup>v</sup>

The Microbatch 5 and 6c Strip Feed samples were analyzed, and this is reported in a later section (see section 3.5). As a result of all of the additional work, the DWPF WAC was modified to accept a pH range of 2 to 11.<sup>ix</sup>

#### 3.1.4 Considerations of Sodium Impacts on DWPF

While the revision to the DWPF WAC provided relief of the elevated pH issues in the SEHT, the latest revision added a sodium concentration requirement. The SEHT samples must now contain  $\leq 265$  mg/L of sodium. While this sodium content issue is addressed in depth in the previously mentioned white paper,<sup>v</sup> it is still relevant to mention here since sodium results could be indicators of NGS process performance.

An examination of the last three Salt Batches (previous processing campaigns using the BOBCalix Solvent) SEHT samples show a consistent pattern of sodium content (Table 9).

**Table 9. Average Sodium Content of Previous Salt Batch 4, 5, and 6 SEHT Samples**

Salt Batch	Average Na Concentration (mg/L)
4	47.3
5	61.7
6	55.6

The analytical uncertainty on the sodium measurements are 10%. For the Demonstration Plan samples, Microbatch 5, 6a, 6b and 6c SEHT samples were analyzed for sodium content.\* The average of all four microbatch results is 44.0 mg/L. Even with the change

\* Microbatch 1 was also analyzed for sodium content, but was discarded from this consideration due to the fact that this early in the demonstration plan, the processed feed was still heavily compromised by dilution.

from the old solvent (Salt Batch 4, 5, 6) to the new Blend solvent, there is little change in the sodium content. This implies that the increase on calixarene does not affect sodium uptake. Although it may be a coincidence, the sodium concentration in the SE (~45 mg/L, or ~0.002 M) is approximately the concentration of the carbonate in the same samples.

### 3.2 DSSHT Sample Results

While F/H lab performed most of the analyses on the DSSHT sample, SRNL performed several types of analyses on various samples. The most important analytes of the DSSHT samples were the  $^{137}\text{Cs}$ ,  $^{238}\text{Pu}$ , and  $^{90}\text{Sr}$  activity and the Isopar™ L concentration.

The radioisotope results are reported in Table 10.

**Table 10. Radiochemical Results for the DSSHT Samples**

Sample	dpm/mL		
	$^{137}\text{Cs}$	$^{238}\text{Pu}$	$^{90}\text{Sr}$
Prelim	NA	6.09E+02 (5.52%)	5.37E+03 (14.1 %)
Microbatch 1	1.35E+06	2.98E+03 (6.00%)	3.89E+03 (9.56%)
Microbatch 2a/2	NA	NA	NA
Microbatch 3	NA	NA	NA
Microbatch 4	NA	NA	NA
Microbatch 5	7.17E+04	NA	NA
Microbatch 6a	8.47E+04	NA	NA
Microbatch 6b	NA	NA	NA
Microbatch 6c	NA	NA	NA
SB6 Feed	1.30E+08	2.91E+04	4.14E+05

The analytical uncertainty on the  $^{137}\text{Cs}$  measurements is 5%. Otherwise, the analytical uncertainty is listed in parentheses next to the reported value.

The few  $^{238}\text{Pu}$  and  $^{90}\text{Sr}$  results for the DSSHT samples are similar to those previously reported results from routine Salt Batch 6 samples.<sup>iii</sup> The  $^{137}\text{Cs}$  results show an overall decline from the beginning to the end. This is an indication that the Blend Solvent is performing in the expected manner. The higher extractant concentration is expressing itself as a lower  $^{137}\text{Cs}$  activity in the DSSHT samples.

Across the spectrum of samples in the Demonstration Plan, various analyses were performed. Inductively Coupled Plasma Emission Spectroscopy (ICPES) analysis was performed on the Preliminary sample. An Ion Chromatography (IC) Anions analysis was

performed on a Microbatch 1 sample. SVOA, density and Free Hydroxide measurements were performed on the Microbatch 5 and 6a samples. See Table 11.

**Table 11. Selected Non-Radiochemical Results for DSSHT Samples**

Analyte	Prelim	Microbatch 1	Microbatch 5	Microbatch 6a
Sodium (mg/L)	105000	NA	NA	NA
Isopar™ L (mg/L)	NA	NA	<7	<7
Nitrate (mg/L)	NA	68900	NA	NA
Nitrite (mg/L)	NA	11000	NA	NA
Density (g/mL)	NA	NA	1.25	1.25
Free OH (M)	NA	NA	2.07	1.96

The sodium, nitrate, nitrite, and Free OH measurements have an analytical uncertainty of 10%. The Isopar™ L measurement has an analytical uncertainty of 20%. Density results from the average of replicate volumetric trials typically have a percentage standard deviation of <1% between each value and the average.

The SVOA, density and Free Hydroxide measurements on the Microbatch 5 and 6a samples were performed as part of the F/H lab outage mentioned in section 3.1.1. All of these results were as expected. SVOA was expected to be less than detectable, the density was expected to be ~1.25 g/mL, and the Free Hydroxide was expected to be ~2 M.

The other analyses were performed in order to examine the relative dilution the feed was experiencing. For example, the Preliminary sample gave a sodium result of 105000 mg/L. The Macrobatch 6 feed was an average of 151000 mg/L, indicating a significant dilution (~30% dilution). For the Microbatch 1 sample, both the measured nitrate and nitrite were measured to be ~50% of the Macrobatch 6 feed value. Early on in the demonstration plan, significant dilution of the feed material was occurring as a result of residual flush materials present in the hold tanks and decanters prior to the start of the NGS Demonstration.

### 3.3 CWT Sample Results

In theory, the caustic wash contactors serve to clean the solvent of any degradation products, such as *sec*-butyl-phenol. In reality, they also function as a scrub contactor, as the caustic wash (0.03 M NaOH) is very similar to the caustic scrub (0.025 M). This also means that the stripped solvent, will deposit some <sup>137</sup>Cs activity into the spent caustic wash. The extent of the deposition of activity into the Caustic Wash Tank (CWT) has varied over the years. Previous CWT samples have varied from ~1E+03 dpm/mL to

$\sim 1\text{E}+06$  dpm/mL<sup>x</sup> of  $^{137}\text{Cs}$  activity. While microbatch sample 6a was not delivered to SRNL, the others were analyzed for  $^{137}\text{Cs}$  content. The results are reported in Table 12.

Other than the Preliminary sample, the  $^{137}\text{Cs}$  activities were all lower than previous CWT samples for the BOBCalix solvent. This could be an indication that the caustic wash is poorer at removing activity from the solvent, or that the stripped solvent has less activity to transfer to the caustic wash to begin with. Since the caustic scrub in the NGS or the Blend solvent in SRNL ESS testing removed non-trivial amounts of  $^{137}\text{Cs}$ , the latter reason is more probable.<sup>xi</sup>

**Table 12.  $^{137}\text{Cs}$  Results for the CWT samples**

Sample	$^{137}\text{Cs}$ (dpm/mL)
Prelim	5.28E+04
Microbatch 1	6.62E+02
Microbatch 2	7.67E+02
Microbatch 3	8.60E+02
Microbatch 4	1.22E+02 (8.46%)
Microbatch 5	5.65E+02
Microbatch 6a	NA
Microbatch 6b	4.24E+02
Microbatch 6c	7.80E+02

The analytical uncertainty for all samples is 5%, unless otherwise noted in parentheses next to the reported value.

In addition to the  $^{137}\text{Cs}$  measurements, several other types of measurements were performed on various CWT samples, with the goal of monitoring the quality of the caustic wash. See Table 13. In these samples, pH was measured by pH paper and not pH probe. No organic or gel layers were seen during visual inspection of the CWT samples.

**Table 13. Selected Non-Radiochemical Analyses on CWT Samples**

<b>Sample</b>	<b>Na (mg/L)</b>	<b>B (mg/L)</b>	<b>Modifier (mg/L)</b>	<b>Density (g/mL)</b>	<b>pH</b>	<b>Free OH (M)</b>
Prelim	NA	NA	<10	NA	14	NA
Microbatch 1	NA	NA	NA	NA	12	NA
Microbatch 2	NA	NA	NA	NA	12	NA
Microbatch 3	NA	NA	NA	NA	12	NA
Microbatch 4	833	1.28	NA	1.00	12	0.0209
Microbatch 5	NA	NA	NA	NA	12	NA
Microbatch 6a	NA	NA	NA	NA	12	NA
Microbatch 6b	NA	NA	NA	NA	12	NA
Microbatch 6c	NA	NA	NA	NA	12	NA

The analytical uncertainty on the sodium and Free OH measurements are 10%. The pH paper measurements are typically good for  $\pm 1$  pH unit, although discrepancies between pH paper and pH probe measurements have been noted. Density results from the average of replicate volumetric trials typically have a percentage standard deviation of <1% between each value and the average. It is important to note that the MCU facility was expected to completely replenish the CWT between microbatches.

In theory, the CWT samples would have a starting sodium concentration of 690 mg/L, a free hydroxide concentration of 0.03 M (0.03 M NaOH), a density of  $\sim 1.00$ , and a pH of  $\sim 12$ . As a whole the results indicate that the caustic wash is somewhat depleted in free hydroxide and slightly elevated in sodium, possibly from deposition of sodium from the solvent. Other than the Prelim sample pH of 14, the pH results are as expected. The high pH noted in the Prelim sample may be related to the superwash of the solvent in the wash contactors. The superwash used 0.3 M NaOH, and residual material 0.3 M NaOH would increase the pH.

The Modifier analysis (by HPLC) was a check to ensure there was no selective depletion of this material from the solvent.

In addition to the HPLC analysis of a CWT sample, a SVOA analysis was performed on the Preliminary CWT sample as a general scan for any organics. See Table 14.

**Table 14. SVOA Analysis of the Preliminary CWT Sample**

Analyte	Concentration (mg/L)
Modifier	5.9
<i>sec</i> -butyl phenol	2.4
<i>α, α</i> -dimethylbenzyl alcohol	3.3
<i>α</i> -methyl styrene	0.84

The analytical uncertainty for all results is 20%.

The presence of small concentrations of Modifier, or its decomposition product *sec*-butyl phenol are not matters for concern. The Modifier is known to have a limited (~25 mg/L) solubility in the previous strip acid (0.001 M nitric acid), so it is not surprising a small concentration is evident in the CWT. SRNL does not normally note the presence of the Modifier or the *sec*-butyl phenol, and the presence of these materials are possibly due to the preceding solvent superwash.

The other two compounds, *α, α*-dimethylbenzyl alcohol and *α*-methyl styrene were seen at very low concentrations and low spectrum match factors. The latter two compounds (red text in Table 14) have no apparent relationship to the MCU solvent or its possible breakdown products.<sup>xii,xiii</sup> Previous documents from SRNL have analyzed CWT samples, or caustic feed, and found similar types of compounds.<sup>xiv,xv</sup> More recently, a Tank 50H SVOA sample noted the presence of a number of organic compounds at the 6-7 mg/L range. After further investigation (which is still ongoing), it is likely that the presence of those compounds were an artifact of sample preparation in the SVOA analyses.<sup>xvi</sup>

### 3.4 SHT Sample Results

The SHT samples comprised the bulk of the analyses performed at SRNL. SVOA (Isopar™ L, Modifier), HPLC (Modifier, BOBCalix, MAXCalix), TOA (TOA), NMR (Isopar™ L, Modifier, MAXCalix, TiDG), FTIR (Isopar™ L, Modifier), TiDG titration (TiDG) and density were all the methods used, providing at least 2 different measurements in most cases. Please note that while SRNL has confidence in the NMR results, this method is still in development.

The results of all the measurements are reported in Table 15. In addition, the target concentrations of the components and the density from a reference sample are provided for comparison. In the case of the TiDG titration result, the analysis requires the TiDG to be in the freebase form. In order to validly compare to the reference material, which is prepared as the TiDG•HCl salt, all the TiDG titration results are converted to the TiDG•HCl molecular weight.

The Microbatch 2a FTIR and NMR results noted the presence of an unusual amount of water, which is likely to increase the effective analytical uncertainty for those two measurements. The high water content is most likely related to sample preparation and not a reflection of a process upset.

The density results show a larger than predicted variation in certain samples. This is considered to be due to recent difficulties in density sample preparation. SRNL is taking steps to improve the density measurements.

**Table 15. All Analyses for SHT Samples**

microbatch Sample	g/mL density	HPLC		SVOA		FTIR		NMR		SVOA		FTIR		NMR		HPLC		titration		NMR		TOA		titration		HPLC	
		mg/L Modifier	mg/L Isopar	mg/L Isopar	mg/L Isopar	mg/L Isopar	mg/L Isopar	mg/L Isopar	mg/L MAXCalix	mg/L MAXCalix	mg/L TIDG	mg/L TIDG	mg/L TIDG	mg/L TOA	mg/L TOA	mg/L TOA	mg/L TOA	mg/L BOBCalix	mg/L BOBCalix								
Prelim	0.8445	156000	130000	155000	168000	650000	628000	616000	48000	43300	1690	1420	570	624	3860												
1	0.834	160000	120000	177000	161000	660000	606000	620000	44200	42800	1540	1170	600	610	3750												
2a	0.833	165000	160000	173000	161000	620000	594000	569000	NA	41300	1280	667	540	465	NA												
2	0.834	162000	160000	174000	163000	590000	609000	614000	41900	41800	1400	1080	530	485	3820												
3	0.846	155000	160000	170000	158000	580000	613000	626000	40300	40400	1260	1080	560	473	3770												
4	0.8175	159000	160000	159000	158000	560000	624000	620000	40900	40800	1270	1000	580	467	3790												
5	0.8325	158000	170000	152000	156000	570000	632000	591000	39000	40700	1260	917	610	465	3600												
6a	0.815	150000	180000	136000	153000	580000	647000	609000	40000	41600	1120	917	270	469	3700												
6b	0.817	157000	160000	160000	158000	550000	623000	615000	41000	43300	1090	917	320	448	4200												
6c	0.8325	151000	170000	156000	157000	570000	617000	605000	39000	45200	1030	1040	340	436	3600												
reference	0.8335		167000			616000			44000		1530		530	4030													
	Uncertainty	10%	20%	13%	8%	20%	8%	4%	10%	11%	10%	15%	20%	16%	10%												
	high	184000	201000	189000	181000	739000	665000	641000	48300	48700	1690	1770	636	615	4430												
	low	151000	134000	146000	154000	493000	567000	592000	39500	39100	1390	1310	424	445	3620												

The analytical uncertainty of each measurement is listed below each column. The analytical uncertainties vary between 4 and 20%, depending on the particular method. In addition, two more rows of values are listed, below the analytical uncertainties. The “high” and “low” values are derived from the reference values, with the associated analytical uncertainty factored in, to generate a range. For example, for a reference Modifier value of 167000 mg/L, a HPLC measurement gives a range of 151000 to 184000 mg/L. In this case, a HPLC result that lies within this range, is within the analytical uncertainty of the reference value, and can be declared to be statistically the same as the reference value. In other words, that analysis indicates the analyte is at nominal concentration.

With such a large set of data, it is more productive to examine the data set for trends rather than attempting to deconvolute each analysis against all the others. It is better to examine the individual results of each analysis and compare to the “high”-“low” range of values discussed in the paragraph above. From these comparisons, we can derive the following conclusions:

- The TiDG is being depleted at an appreciable rate. The Microbatch 6c sample indicates a TiDG concentration ~65% of the reference value. This is similar to the pattern of depletion for TOA that has been observed with the BOBCalix solvent operations.<sup>xvii</sup> However, in the same set of samples, the TOA has only barely depleted to a statistically significant degree; ~84% of the reference value (from the titration data). From this comparison it would appear that the TiDG could deplete at a faster rate than the TOA did in previous operations. Recent ORNL studies corroborate this general behavior.<sup>xviii</sup>
- Both the MAXCalix and BOBCalix are within the expected range of concentrations, with the exception of four results (highlighted in Table 15), which case both values are just lower (~11%) than the “low” value in the range. As all of these results are from the HPLC, it is possible that method is showing a slight low bias on the analyses.
- Of the 40 Modifier analyses, only five samples results (highlighted in Table 15) are statistically different (lower than) from the reference value. Furthermore, three of those five corresponded to a single sample (6a).
- Of the 30 Isopar™ L analyses, only two sample results from the NMR (highlighted in Table 15) are statistically different from the reference value. This is probably due to the very small analytical uncertainty (4%) of the NMR, or due to small variations from the addition of Isopar™ L at several points during the Demonstration run.

Given the relatively small amount of time and material that was processed, the solvent should not undergo any large changes in composition, with the exception of the Isopar™ L, TOA and TiDG, which are anticipated to deplete more rapidly. From the data set as a whole, we can say that the solvent is at nominal composition and has held up

well so far to processing/radiolysis decay, with the excepting TOA and TiDG. There are no real discernable trends in the solvent. Microbatch 5 and 6a samples have a slight low bias in some of their measurements, and this is likely due to sample preparation as opposed to actual changes in the solvent.

Upon consideration, the 20% analytical uncertainty of the SVOA measurement gives limited utility to this analytical method. Given the large “acceptable” value the SVOA and the availability of alternate methods, consideration should be given to no longer relying on SVOA. However, SVOA should still be used to examine samples for trace organic impurities on a quarterly basis.

#### 3.4.1 *Consideration of the TiDG Depletion Rate*

Of all the components in the solvent, only the TOA and the TiDG show accelerated depletion.<sup>¶</sup> For the TOA, the depletion is well known and has been addressed during processing. In the NGS or Blend solvent, the TOA is essentially a bystander, and there are no plans to add further TOA to the MCU system. In the case of the TiDG, the depletion must be considered. In order to forecast the demand for further TiDG additions to the system, the depletion rates are calculated below.

The depletion rate can be examined in two different ways. First, the rate can be on a time basis; mg/L/day. The depletion rate on a volume of processed material (mg/L/gallon of feed) is also a useful rate to consider. Examination of both derived rates can potentially provide insight into the reason for the TiDG depletion. For example, if the TiDG is decomposing due to a chemical reaction with the salt solution feed or partitioning into the aqueous phases during processing, then the rate in [mg/L/gallon of feed] is the more relevant rate. On the other hand, if the TiDG is chemically decomposing over time, then the rate in [mg/L/day] is more relevant.

From the MCU data set, it is possible to extract both of the cumulative loss rates (Table 16). At this time, no attempt has been made to factor in the effect of temperature, so these rates are not refined. SRNL is in the process of further analyzing the data to forecast the TiDG demand.

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<sup>¶</sup> Isopar is known to evaporate; therefore, the MCU facility evaluates the need for an Isopar addition monthly.

**Table 16. TiDG Loss Rates**

Sample	TiDG*HCl (mg/L)	Loss (mg/L/day)	Loss (mg/L/gal)
Prelim	1690	NA	NA
Microbatch 1	1540	28	0.031
Microbatch 2a	1280	40	0.042
Microbatch 2	1400	18	0.030
Microbatch 3	1260	17	0.030
Microbatch 4	1270	14	0.023
Microbatch 5	1260	12	0.019
Microbatch 6a	1120	12	0.022
Microbatch 6b	1090	11	0.021
Microbatch 6c	1030	11	0.020

3.4.2 <sup>137</sup>Cs in the SHT samples

The SHT samples were also analyzed for <sup>137</sup>Cs content. This gives an indirect indication of stripping performance. See Table 17.

**Table 17. <sup>137</sup>Cs in the SHT Samples**

Sample	<sup>137</sup> Cs (dpm/mL)
Prelim	6.85E+05
Microbatch 1	3.44E+04
Microbatch 2a	1.18E+05
Microbatch 2	3.54E+04
Microbatch 3	2.56E+04
Microbatch 4	3.52E+04
Microbatch 5	3.24E+04
Microbatch 6a	2.28E+04
Microbatch 6b	2.66E+04
Microbatch 6c	3.31E+04

The analytical uncertainty is 5% for each sample.

Over the length of the Demonstration, the <sup>137</sup>Cs in the SHT samples is down to about an order of magnitude less than the previous SHT samples during the use of the BOBCalix solvent.<sup>xix</sup> The Macrobatch 2a sample result seems out of the pattern and this may be due

to the mechanical issue experienced during that microbatch. As the calix concentration in the Blend Solvent is 7 fold higher than with the BOBCalix solvent, this means that the stripping performance is much improved over the BOBCalix solvent.

### 3.5 Strip Feed Sample Results

With the observation of elevated pH values in the SEHT samples (section 3.1.3), two samples (Microbatch 5, 6c) of the strip acid feed were analyzed. ICPES, TIC/TOC and pH were measured for both samples. The results are reported in Table 18.

**Table 18. Results of Analyses of Microbatch 5 and 6a Strip Feed**

Sample	B (mg/L)	TIC (mg/L)	TOC (mg/L)	pH
Microbatch 5	104	11.9	<10	6.3
Microbatch 6c	99.7	6.20	<10	5.9

The analytical uncertainty for the boron and TIC/TOC results are 10%. The uncertainty of the pH probe is 0.1 pH units.

The boron result is a measure of the boric acid concentration, which for a 0.01 M boric acid solution corresponds to 108 mg/L of boron. As with the SEHT sample, the TIC result is assumed to correspond to carbonate. These sample results then give 59.6 and 31 mg/L of carbonate respectively.

The pH results can be compared to the Boron results. The Boron results indicate the boron is as expected, implying the boric acid was prepared to the specified concentration. On the other hand, when the pH values are converted to boric acid concentrations, concentrations of 0.3 mM and 0.2 mM are calculated for Microbatch 5 and 6c, respectively. This in turn implies one of several conditions:

- the pH measurement is biased high in these solutions
- the boric acid was not prepared as a pure acid
- there is some impurity present that is biasing the pH measurement high

The pH probes are calibrated at 3 pH values before use (4, 7, 10) so the first condition is unlikely. The ICPES of the strip feeds show less than detectable concentrations of the common cations (Na, K, Ca) that would indicate an impurity; making the second condition unlikely.

The only plausible reason for the high pH in the strip feed is the inclusion of a very small amount of bicarbonate/carbonate impurity in the feed, which will drive the pH upwards. This may be further counter-balanced by a very small quantity of absorbed CO<sub>2</sub>, which

would drive the pH down. The OLI model suggests that the small quantity of TIC (which can be any or both of  $\text{CO}_2$  or  $\text{HCO}_3^-$ ) could theoretically provide the measured pH result. SRNL recommends, as part of future work, monitoring the pH a solution of boric acid purged with  $\text{CO}_2$  to examine this theory.<sup>vii</sup>

### 3.6 Scrub Feed Sample Results

With the observation of elevated pH values in the SEHT samples (section 3.1.3), one sample (Microbatch 6c) of the scrub caustic feed was analyzed. ICPEs, TIC/TOC and pH were measured for the sample. The results are reported in Table 19.

**Table 19. Results of Analyses of Microbatch 6c Scrub Feed**

Sample	Na (mg/L)	TIC (mg/L)	TOC (mg/L)	pH
Microbatch 6c	610	122	<4	11.6

The analytical uncertainty for the sodium and TIC/TOC results are 10%. The uncertainty of the pH probe is 0.1 pH units.

The pH result, when converted to Free Hydroxide, gives a value of 4 mM, where in theory it should be 25 mM. The sodium result of 610 mg/L converts to 0.0265 M, which is where 0.025 M NaOH should be. The scrub feed is subject to  $\text{CO}_2$  uptake from the atmosphere, which will convert hydroxide into bicarbonate. If further hydroxide is still available, this can react with the bicarbonate to produce carbonate. SRNL performed a run of the OLI model of a 0.0265 M NaOH solution, with enough  $\text{CO}_2$  to account for the TIC result. OLI predicts a pH of 11.7, which is very close to the measured 11.6 value.

### 3.7 Comparison between F/H and SRNL Sample Results

In a selected number of cases, both F/H lab and SRNL have performed analyses on the same Microbatch sample. It is appropriate to compare these results to ensure neither lab is suffering from a particular bias. The largest body of data for comparison is the  $^{137}\text{Cs}$  data. Samples of the CWT, DSSHT and SEHT were sent to both SRNL and F/H lab, leading to 12 sample overlaps. See Table 20. “mb” is shorthand for “Microbatch”

**Table 20. Comparison of <sup>137</sup>Cs Results between SRNL and F/H Lab**

<b>Sample</b>	<sup>137</sup> Cs (SRNL) (dpm/mL)	<sup>137</sup> Cs (F/H) (dpm/mL)
CWT mb1	6.62E+02	1.24E+03 (18.3%)
CWT mb2	7.67E+02	1.35E+03 (25.5%)
CWT mb3	8.60E+02	1.13E+03 (39.2%)
CWT mb4	1.22E+02	8.94E+02 (33.9%)
CWT mb5	5.65E+02	1.10E+03 (32.8%)
CWT mb6b	4.24E+02	6.72E+02 (8.62%)
CWT mb6c	7.80E+02	7.95E+02 (20.6%)
DSSHT mb1	1.35E+06	1.21E+06
SEHT mb1	1.55E+08	3.30E+08
SEHT mb6b	1.09E+09	7.51E+08

The analytical uncertainty for F/H lab is 12%, where as SRNL results are 5%, except for the SRNL CWT mb4 result, which is 8.46%. The F/H results for the CWT samples are averages of multiple sample results. For these results the value in parentheses are the relative standard deviation (RSD).

In the most recent comparison of past SRNL and F/H lab results,<sup>iii</sup> it was found that the DSSHT results differed by an average of ~7% and the SEHT results differed by an average of ~11%. Given this history of overall corroboration, it is surprising to see such a lack of corroboration in these comparisons. However, an examination of the CWT results from F/H lab provided some insight. The F/H lab CWT sample results are averages of multiple samples pulled within each microbatch. The large relative standard deviation indicates that even during a single microbatch, there are wide swings in the sample results. This is probably due to the system not being at equilibrium, and therefore, there is a high degree of time-dependence on when the sample is pulled.

Other than the <sup>137</sup>Cs results, there was a single Free Hydroxide measurement. See Table 21.

**Table 21. Free Hydroxide Results for Duplicate SRNL and F/H Samples**

<b>Sample</b>	<b>Free OH (SRNL) M</b>	<b>Free OH (F/H) M</b>
CWT microbatch4	0.0209	0.0244

The analytical uncertainty for the Free OH is 8% for F/H lab and 10% for SRNL. The crosscheck of the Free Hydroxide shows that both labs are giving the same effective results.

### 3.8 Brief Discussion of the Decontamination and Concentration Factors

While a detailed examination of the cesium removal performance is beyond the scope of this document, it is appropriate to briefly discuss some of the more obvious results. The Decontamination Factor (DF) is defined as the  $^{137}\text{Cs}$  activity in the Tank 49H feed solution, divided by the  $^{137}\text{Cs}$  activity in the DSSHT sample. The Concentration Factor (CF) is defined as the  $^{137}\text{Cs}$  activity in the SEHT sample divided by the  $^{137}\text{Cs}$  activity in the Tank 49H feed solution. The higher the DF, the greater the decontamination in the DSSHT sample. This value has no upper bound. The CF has a theoretical maximum of 15. Table 22 shows the calculated DF and CF values, using the  $^{137}\text{Cs}$  activities from each sample, as well as the Tank 49H feed value (1.30E+08 dpm/mL). For the sake of consistency, in cases where there are  $^{137}\text{Cs}$  results from both SRNL and F/H lab, the F/H value is used.

**Table 22. DF and CF Values for Demonstration Plan Samples**

microbatch	dpm/mL		DF	CF
	DSSHT	SEHT		
prelim	9.68E+05	6.28E+07	1.34E+02	4.83E-01
1	1.21E+06	3.30E+08	1.08E+02	2.54E+00
2	5.73E+05	8.39E+08	2.27E+02	6.45E+00
3	8.27E+04	1.15E+09	1.57E+03	8.84E+00
4	9.44E+04	1.34E+09	1.38E+03	1.03E+01
5	7.10E+04	1.74E+09	1.81E+03	1.34E+01
6a	8.45E+04	1.65E+09	1.53E+03	1.27E+01
6b	7.68E+04	7.51E+08	1.69E+03	5.78E+00
6c	7.68E+04	8.04E+08	1.69E+03	6.18E+00

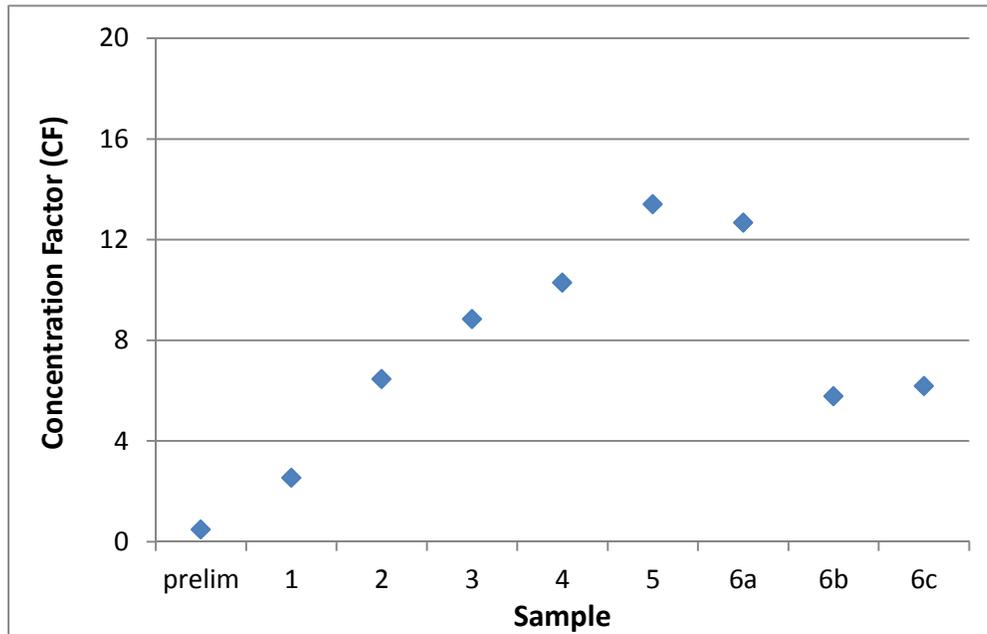
The analytical uncertainty on each  $^{137}\text{Cs}$  measurement is 12%.

Given that the process started with residual flush material, which was worked off over time, the DF and CF should only be considered as a starting point.

The DF and CF value show a general trend – as the Demonstration continues, the DF values increase, to a maximum of ~1700. The CF attains a maximum of 13.4, but then declines. See Figure 1. The decline is a result of excess strip acid being added to the

SEHT and SE decanter due the failure of the strip feed flow meter at the end of Microbatch 6a and continuing into Microbatches 6b and 6c.

**Figure 1. Concentration Factor over the Range of Samples**



#### 4.0 Conclusions

SRNL and F/H lab analyzed a large collection of samples throughout the Demonstration Plan to provide evidence as to the processability of the Blend Solvent at MCU. Samples of each of the outputs from MCU (SHT, CWT, DSSHT, and SEHT) were analyzed by multiple methods. From these analyses the following conclusions can be made on each of the outputs.

- SHT: The solvent behaved as expected, with no bulk changes in the composition over time, with the exception of the TOA and TiDG. The TiDG depletion is higher than expected, and consideration must be taken on the required rate of replenishment. Monthly sampling of the SHT is warranted. If possible, additional SHT samples for TiDG analysis (only) would help SRNL refine the TiDG degradation model.
- CWT: The CWT samples show the expected behavior in terms of bulk chemistry. The <sup>137</sup>Cs deposited into the CWT varies somewhat, but generally appears to be

lower than during operations with the BOBCalix solvent. While a few minor organic components were noted to be present in the Preliminary sample, at this time these are thought to be artifacts of the sample preparation or may be due to the preceding solvent superwash.

- DSSHT: The DSSHT samples show the predicted bulk chemistry, although they point towards significant dilution at the front end of the Demonstration. The  $^{137}\text{Cs}$  levels in the DSSHT are much lower than during the BOBCalix operations, which is the expected observation.
- SEHT: The SEHT samples represent the most different output of all four of the outputs from MCU. While the bulk chemistry is as expected, something is causing the pH of the SEHT to be higher than what would be predicted from a pure stream of 0.01 M boric acid. There are several possible different reasons for this, and SRNL is in the process of investigating. Other than the pH issue, the SEHT is as predicted.

The DF and CF values are generally showing the expected trends (increased removal). The data set is limited, and as time progresses in Sample and Send mode operations, additional information will become available to make a longer term prediction of the ultimate cesium decontamination.

SRNL also performed an abbreviated set of analyses on the scrub and strip feed solutions. These were shown to be as expected, although there is evidence that both the strip feed and scrub feed contain small quantities of carbonate or bicarbonate.

Taken as a whole, the Demonstration Plan samples indicate that the MCU system, with the Blend Solvent, is operating as expected with the exception of the SEHT pH trending higher than expected, with minor issues as previously noted. SRNL is in the process of investigating the cause of the elevated pH.

## **5.0 Path Forward**

Once the Demonstration Plan is complete, MCU will be operating in a Sample and Send mode of operations in which MCU operations is expected to pull monthly SHT, CWT, SEHT and DSSHT samples for SRNL. Ideally these samples should all be pulled at the same time, or as close as possible. This will enable SRNL to provide the most accurate analysis of MCU operations.

In regards to cesium removal, the data set is limited, and as time progresses in normal Sample and Send mode operations, enough information will be available to make a longer term prediction of ultimate cesium decontamination.

The most important consideration at this time is the depletion of the TiDG. The depletion should be carefully monitored to ensure adequate stocks of this material are available for addition to MCU.

Further SRNL suggests the following lessons are related to the analysis of the SHT samples. Based upon the large matrix of SHT sample results, SRNL recommends the following:

- Move density measurements to outside of the cells where the best precision can be obtained.
- Delete the SVOA measurement of Isopar™ L and Modifier. Instead, establish the Isopar™ L and Modifier relationship to density. The nominally more accurate density measurement will provide a better understanding of the concentration of these 2 largest components of the solvent.
- Delete the TOA measurement as there will be no further additions.
- Delete the BOBCalix measurement as it is no longer of concern.
- Improve and qualify the FTIR method for general analytical use. This method show great promise in SHT analyses.
- Analyze CWT samples with NMR to detect decomposition products.
- SHT samples should be taken at monthly intervals. If possible, additional SHT samples for TiDG analysis (only) would help SRNL refine the TiDG degradation model.

## 6.0 References

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**Distribution:**

S. L. Marra, 773-A  
F. M. Pennebaker 773-42A  
T. B. Brown, 773-A  
S. D. Fink, 773-A  
C. C. Herman, 773-A  
E. N. Hoffman, 999-W  
D. H. McGuire, 999-W  
W. R. Wilmarth, 773-A  
Records Administration (EDWS)

E. A. Brass, 249-8H  
T.E. Smith, 241-152H  
C. J. Scherman, 241-152H  
Q. L. Nguyen, 241-152H  
E. J. Freed, 704-S  
A. G. Garrison, 248-9H  
B. A. Gifford, 704-56H  
T. A. Le, 766-H  
D. J. Martin, 241-152H  
A. R. Shafer, 704-27S  
R. H. Spires, 248-8H

C. A. Nash, 773-42A

P. R. Jackson, DOE-SR, 703-46A