

**Characterization and Evaluation of Caustic Wash Tank and  
Solvent Hold Tank Samples from MCU from August to  
September 2011 (SHT 221-226, SHT 314-319, SHT 1035-  
1040, and CWT 817-904)**

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August 2012

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Prepared for the U.S. Department of Energy Under  
Contract Number DE-AC09-08SR22470



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## EXECUTIVE SUMMARY

During processing of Salt Batches 3 and 4 in the Modular Caustic-Side Solvent Extraction Unit (MCU), the decontamination efficiency for cesium declined from historical values and from expectations based on laboratory testing. This report documents efforts to analyze samples of solvent and process solutions from MCU in an attempt to understand the cause of the reduced performance and to recommend mitigations.

CWT Solutions from MCU from the time period of variable decontamination factor (DF) performance which covers from April 2011 to September 2011 (during processing of Salt Batch 4) were examined for impurities using chromatography and spectroscopy. The results indicate that impurities were found to be of two types: aromatic containing impurities most likely from Modifier degradation and aliphatic type impurities most likely from Isopar<sup>®</sup> L and tri-*n*-octylamine (TOA) degradation. Caustic washing the Solvent Hold Tank (SHT) solution with 1M NaOH improved its extraction ability as determined from <sup>22</sup>Na uptake tests. Evidence from this work showed that pH variance in the aqueous solutions within the range of 1M nitric acid to 1.91M NaOH that contacted the solvent samples does not influence the analytical determination of the TOA concentration by GC-MS.

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## ACRONYMS

BOBCalixC6	Calix[4]arene-bis( <i>tert</i> -octylbenzene-crown-6)
cm <sup>-1</sup>	wavenumbers (=1/λ or the reciprocal of wavelength in centimeters)
Cs-7SB	1-(2,2,3,3-Tetrafluoropropoxy)-3-(4- <i>sec</i> -butylphenoxy)-2-propanol
DF	decontamination factor
FTIR	Fourier-Transformed Infrared Spectroscopy
FTNMR	Fourier Transformed Nuclear Magnetic Resonance
GC	Gas Chromatography
GC-MS	Gas Chromatographic hyphenated to a Mass Spectrometer (Ionization Quadrupole Electron multiplier combo)
HLW	High Level Waste
HPLC	High Performance Liquid Chromatography
Isopar <sup>®</sup> L	branched average 12-carbon atoms aliphatic
MCU	Modular Caustic-Side Solvent Extraction Unit
PPM	part per million
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
SVOA	Semivolatile Organic Analysis
TOA	tri- <i>n</i> -octylamine
VOA	Volatile Organic Analysis

## 1.0 Introduction

Since 2008, the MCU facility at the Savannah River Site (SRS) has processed nearly three million gallons of supernate using immiscible liquid-liquid extraction technology via centrifugal contactors. The organic liquid that removes cesium from supernate consists of four chemical components which include an extractant (BOBCalixC6)<sup>i</sup>, a Modifier (Cs-7SB)<sup>ii</sup> to increase the solubility of the extractant in the diluent (Isopar<sup>®</sup> L), and a third phase suppressor tri-n-octyl amine. This solvent (blend) sequentially contacts High Level Waste supernate to extract the cesium, scrub solution (50 mM nitric acid), strip solution (1 mM nitric acid into which the cesium distributes extensively), and caustic wash (0.3 M NaOH).

MCU is designed to achieve a minimal cesium decontamination factor (DF) of 12. Early facility operations achieved DF values in excess of 200 but during processing of Salt Batch 3, and throughput Salt Batch 4, the removal efficiency showed modest declines.<sup>1</sup> A panel of solvent extraction experts reviewed the processing behavior and offered thoughts on causes and mitigations.<sup>2</sup>

To understand the performance variability, samples from both the Caustic Wash Tank (CWT) and the Solvent Hold Tank (SHT) were collected from the time period when performance degraded (August through September 2011) and analyzed for the presence of impurities.

## 2.0 Experimental

The ability to detect organic impurities by direct analysis of the solvent samples is hampered by the relatively low concentration of impurities. The multi-component solvent has spectroscopic features that “crowd” or “hide” the features of any impurities. The caustic wash operation is intended to remove such adverse impurities. Although distribution into the caustic wash may be poor for those impurities that are adversely impacting cesium mass transfer performance, obtaining spectroscopic signatures of these species may be easier than in the complex solvent matrix. Also, although the organic impurities may be present in trace concentrations, extraction of those impurities from the CWT samples by using a high aqueous to organic extractant ratio will help concentrate the impurities. Hence, Savannah River National Laboratory (SRNL) pursued analyses of both the SHT and the CWT samples.

Communication with MCU personnel identified that CWT samples from August through the first week of September 2011 correlated with periods of low cesium removal efficiencies. Personnel combined several samples into a blend to simplify efforts to identify any accumulated component that may be detrimental to the process. Thus, all samples listed in this work represent blends of samples sent to the laboratory for analytical and performance confirmation.

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<sup>i</sup> BOCCalixC6 is an informal designation for calix[4]aren-bis(*tert*-octybenzo-crown-6).

<sup>ii</sup> Modifier, or Cs-7SB, are informal designations for 1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol.



Samples from the CWT (aqueous) were characterized by spectroscopic and chromatography methods. Chromatography methods used included gas chromatography (GC) for both volatile organic analyses (VOA) and semi-volatile organic analyses (SVOA) (of low molecular weight organics) as well as High Performance Liquid Chromatography (HPLC) for organic compounds containing aromatic rings (that have UV-active substituents). Spectroscopic methods included Fourier Transformed Infrared (FTIR) selected for its high sensitivity and Fourier Transformed Nuclear Magnetic Resonance (FTNMR) selected for its high resolution. Both spectroscopic methods can detect organic molecules regardless of their molecular weight. Some CWT samples were contacted with hexane (for removing hydrocarbons) and dichloromethane (for removing everything except water) and then analyzed by FTIR and the chromatographic methods. (These extracting solvents are compatible with the chromatographic methods.)

Similarly, organic samples from the SHT were characterized by spectroscopic and chromatographic analysis. In addition, some solvent samples were evaluated for extraction performance and in this study the ability to extract a cesium competitor, sodium, was measured. Some SHT samples were purged with in-house nitrogen gas (99 vol %) to remove Isopar<sup>®</sup> L which account for 69.1 wt % of the sample mass before characterization. These concentrated samples were also characterized by spectroscopic and chromatographic methods as shown in Table 1. For NMR analysis samples were not doped with known calibrating compounds like tetramethyl silane or deuterated chloroform due to their high vapor pressure and the concern of radioactive material removal by this mechanism.

A summary of the sample treatment and analysis is listed in Table 1. As noted in Table 1, a portion of sample SHT-11-1035-1040 (aggregate of samples from 1035 to 1040) was washed with 1M NaOH to evaluate the effect of washing for impurity removal on the exchange ability of this solvent with sodium. For evaluating the effect of TOA ionicity on the accuracy of SVOA, a previously qualified solvent (S2-D2-YesBOB-T-WI<sup>3</sup>) was doped with additional TOA to raise its levels to 6 and 10 mM, levels above the limit of quantification (LOQ) of the SVOA method, and contacted with solutions of various pH (e.g., 1M NaOH, 1 mM nitric acid, 1M nitric acid and 1.91M [OH] salt simulant). CWT samples from the time period of low DF (August 2011) and from November 2011 (when DF performance recovered) were analyzed for comparison. Chromatographic and NMR analysis requires approximately 1 mL of sample while approximately 0.1 mL of sample was required for FTIR analysis.

Caustic washing was performed identical to established Extraction, Scrub, and Strip protocol. This includes contacting a 3:1 volume ratio (for extraction and 1:5 for scrub and strip) aqueous solution to organic solution in a Teflon<sup>™</sup> separatory funnel. The funnel was shaken, end over end (approximately one bottle was turned over completely once per second) for five minutes, the temperature was recorded, and then the funnel was placed vertically for 24 hours after which the aqueous layer was separated from the organic layer.

**Table 1. The treatment protocol conducted on samples from CWT and SHT as well as the analytical methods used to analyze them.**

Sample	Treatment	Analysis
CWT-11 817-904 1119-1120	None	SVOA and FTIR
	Evaporated (for FTIR only)	
SHT-11 314-319 221-226	Evaporated	FTIR, NMR, HPLC, SVOA
SHT-11 1035-1040	Washed with 1M NaOH, separated into two portions: one portion extracted with dichloromethane, and the other portion extracted with hexane	SVOA, HPLC, and FTIR
S2-D2-YesBOB-T-WI-2005	Added TOA and washed with 50 mM NaOH, 1M HNO <sub>3</sub> , 1mM HNO <sub>3</sub> , and 2M NaOH	Oil: SVOA
SHT-11- 1035 to 1040 and 1M OH Washed SHT-11-1035 to 1040	Spiked salt simulant with <sup>22</sup> Na and contacted with CSSX solvent	Gamma spectroscopy

### 3.0 Results and Discussion

#### 3.1 Impurity Analysis of CWT Samples

The extracts from hexane and dichloromethane solvents contact with CWT samples were submitted to SVOA analysis. At the quantitative level (~8 times the standard error deviation of the method), no impurities were found. However, at the qualitative level (i.e., at lower concentrations), a residue fingerprint was detected at the mass spectroscopy instrument. That fingerprint was compared to the fingerprints of known chemicals compounds and the database software identified and listed those compounds whose fingerprints matched at least 75% of the residue fingerprint. These chemicals are listed in Table 2.

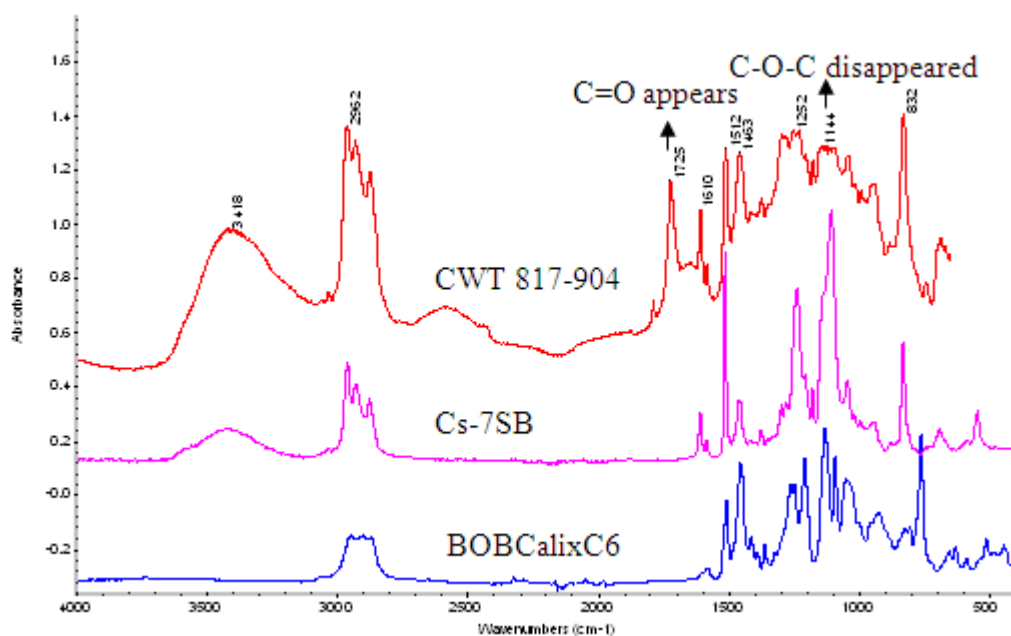
**Table 2. Chemicals found by SVOA that at least matched 75% of the impurities fingerprints (in mg/L) found in CWT 817-904 and 1119-1120. A molecular representation of these compounds is provided in Appendix A. Numbers in parenthesis represent the percent ratio of the standard deviation to the average value (%RSD)**

Sample	Component	Hexane Extraction*	Dichloromethane Extraction*
<b>CWT 817-904</b>	alpha-methylstyrene	0.052 (43.5%)	0.026 (69 %)
	acetophenone	0.017 (42 %)	-
	alpha*alpha-dimethylbenzenemethanol	0.26 (76 %)	0.074 (49 %)
	Modifier	0.13 (22 %)	-
<b>CWT 1119-1120</b>	2*4*4-trimethyl-1-hexene	0.073	-
	alpha-methylstyrene	0.035	-
	acetophenone	0.031	-
	1-indanone	0.063	-
	alpha*alpha-dimethylbenzenemethanol	0.25	0.07
	Modifier	1.2	0.12
	phthalic anhydride	-	0.027
phenol	-	0.051	

**\*LOD (level of detection)= 0.2 mg/L**

As can be seen in Table 2, the chemicals contains at least one aromatic group with substitution containing either alcohols or aliphatics (chain or cyclic). This common chemical structure may indicate the impurity originates from degraded MCU components (which contain aromatics) or from the salt batch (supernate) since some of these compounds have been found throughout the tank farm. It is more unlikely that these impurities originate from the chemical feedstock, NaOH, to the CWT. Manufacturers typically remove all of the organic material when making sodium chloride via solar concentration. Sodium chloride is used in the NaOH synthesis. Presence of these impurities in the process water used to dilute the sodium hydroxide also seems unlikely. SRNL recommends investigating the chemicals (listed in Table 2) for their possible interference in mass transfer.

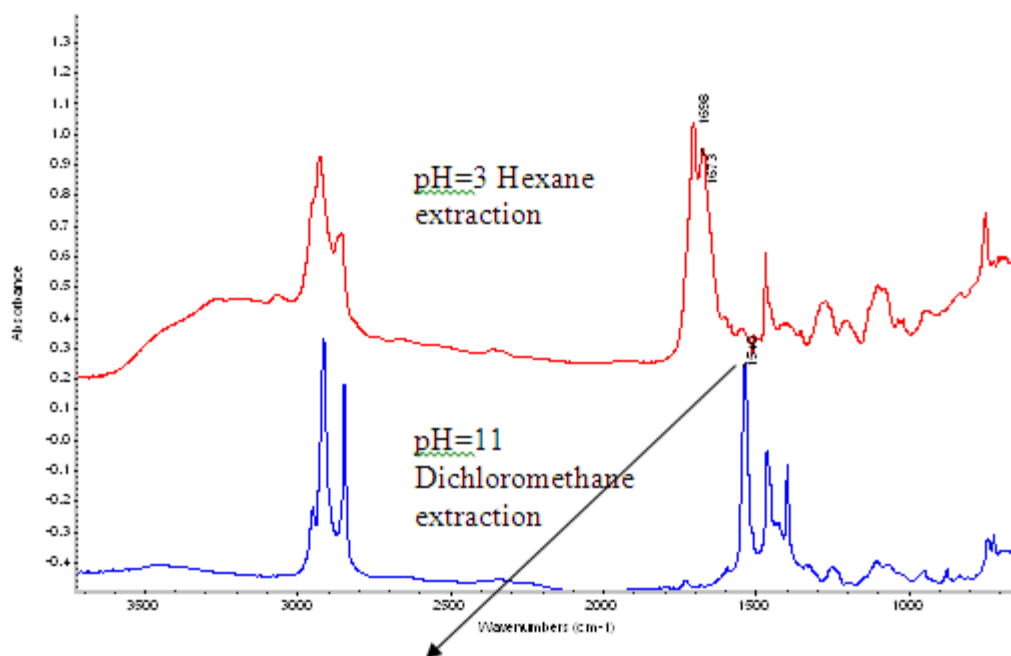
A portion of sample CWT 817-904 was allowed to dry by purging with inhouse nitrogen gas until gravimetric steady state was achieved and the residue analyzed by FTIR. The FTIR of the dried material is shown in Figure 1. Also shown in Figure 1 is the spectrum of the Modifier and the extractant. As can be seen in Figure 1, the spectrum of the residue is similar to that of the Modifier except that it is missing both the C-O-C and C-F's group of the Modifier and it contains a carbonyl group. This indicates this residue contains Modifier that was attacked (oxidized) at the C-O-C linkage to the C-F group and spawning a carbonyl at that spot. The tail of the Modifier has been cut short giving rise to a perfluoronoate chain.



**Figure 1. Impurity found in aggregate CWT 817-904 when the sample was Evaporated (no C-F vibrations is seen).**

It is possible that this residue has an aryl ester or an aliphatic aldehyde as denoted by the  $1725\text{ cm}^{-1}$  peak attached to it. Based upon the strength of the FTIR peak and the concentration step (Isopar<sup>®</sup>L evaporation) indicate the impurity concentration is approximately 23 ppm in the starting solvent. A previous laboratory study<sup>4</sup> showed that after three years of equivalent gamma irradiation (8 Mrad/year) less than 1% and 3% of the extractant and Modifier degraded respectively (9 ppm and 890 ppm respectively). Portions of the solvent inventory has been in operation for at least four years (since March 2008) and given that the solvent was exposed to caustic and acidic solution that could have washed out some radiation-generated impurities, the level determined in this study (23 ppm) is well within the levels found in reference 4.

When the extracts of CWT 817-904 from contacting hexane and dichloromethane were analyzed by FTIR (after allowing these solvents to evaporate before examination), two additional molecules were identified as shown in Figure 2.

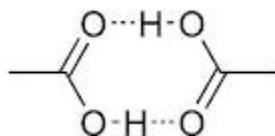


Metal	C=O stretch (cm <sup>-1</sup> )	
Li+	1581	1557
Na+	1559	
K+	1561	
Mg <sup>2+</sup>	1566	
Ca <sup>2+</sup>	1557	1541
Sr <sup>2+</sup>	1514	
Cd <sup>2+</sup>	1528	
Co <sup>2+</sup>	1528	
Al <sup>3+</sup>	1589	
Pb <sup>2+</sup>	1513	
Zn <sup>2+</sup>	1539	

- Aliphatic carboxylate is complexed to a metal heavier than potassium

**Figure 2. Top: dichloromethane extract from CWT 817-904 aggregate. Bottom: hexane extract from CWT 817-904 aggregate. Bottom spectrum has features similar to a metal-complexing C<sub>10</sub> length molecule.**

As can be seen from Figure 2, extraction with hexane revealed a straight chain molecule that resembles dimeric carboxylic acid.



This assignment is based on the carbonyl stretch at  $1696\text{ cm}^{-1}$  and  $1673\text{ cm}^{-1}$  and the broad peak at  $3200\text{ cm}^{-1}$ .

Analysis of the extraction with dichloromethane revealed another straight chain hydrocarbon at least 10 carbon atoms long as noted in the split of the  $720\text{ cm}^{-1}$  peak and also noted in the strong peaks at  $2926$  and  $2860\text{ cm}^{-1}$  due to the stretching of the  $\text{CH}_2$  group. At the other end of this molecule there is a carboxylate group as noted by the  $1540\text{ cm}^{-1}$  peak. The low frequency of this peak indicates that the carboxylate is likely complexed to a metal. As shown in the table insert in Figure 2, this frequency indicates the metal is not sodium, potassium, calcium (which has a doublet as indicated by the two numbers), or aluminum. The peak is broad when complexed to a 2+ or higher valence metal. It is likely this metal is heavier than potassium but is not a transition metal.

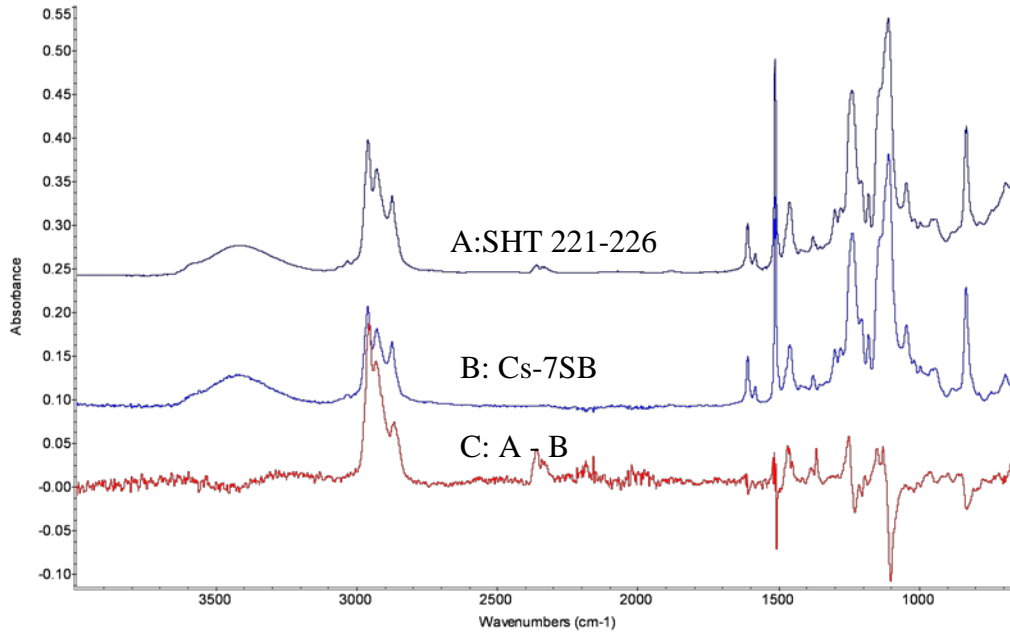
Analysis of the extracts from CWT 1119-1120 or the evaporated residue from CWT 1119-1120 did not reveal any organic molecule. Only sulfates and carbonates were seen in this sample.

### 3.2 Impurity Analysis of the SHT Samples

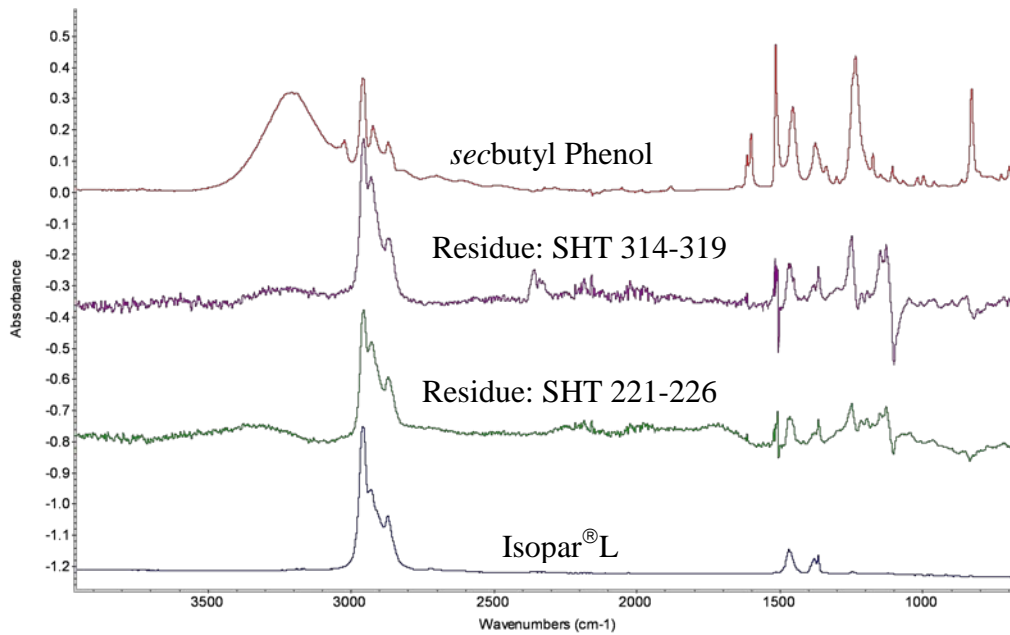
Samples from SHT 221-226 and SHT 314-319 were purged with nitrogen gas until no further gravimetric loss was observed. The evaporated samples were then analyzed by FTIR. Figures 3 and 4 show the raw spectra of the SHT samples, that of Cs-7SB (or Modifier), and the difference spectrum between SHT and the Modifier. As can be seen from Figure 4, which shows the reference spectra of *sec*butyl phenol and of Isopar<sup>®</sup> L for identification, the difference spectra from both SHT samples are similar and the remaining FTIR peaks may be explained as residual Isopar<sup>®</sup> L with very a small amount of *sec*butyl phenol which has been previously reported as a major impurity when CSSX is exposed to high nitrite solutions.<sup>5</sup>

Detection of impurities is made difficult with the large concentration of Modifier left in the evaporated sample. It is more craft than science to interpret the residuals from a subtraction spectrum as this mathematical treatment introduces effects and variability to the final result. Nevertheless, the difference spectra obtained in Figures 3 and 4 contain clear and strong residual peaks for easy identification.

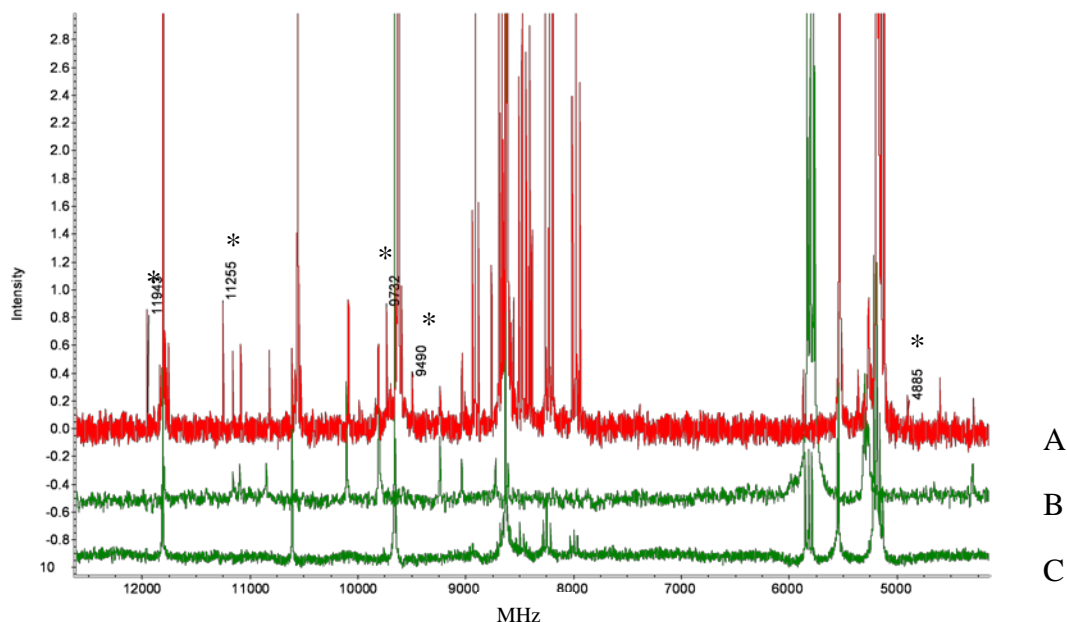
Because of this difficulty, we tried to conduct  $^{13}\text{C}$  NMR but equipment limitations have postponed that collection. Instead, Figure 5 shows the  $^{13}\text{C}$  NMR of the SHT from April 2011 collection prior to the equipment issues. The April 2011 was selected since prior analyses of this sample suggested potential damage to the solvent and since decline in cesium stripping efficiency was suggested in earlier tests.<sup>6</sup>



**Figure 3. Concentrated SHT 221-226 compared to Modifier. The difference spectrum shows residual Isopar® L in the 221-226 aggregate.**



**Figure 4. A comparison between the difference spectrum obtained from evaporated SHT 221-226 and SHT 314-319 with the spectrum of sec-butyl phenol and Isopar® L indicating *secbutyl phenol* is present.**

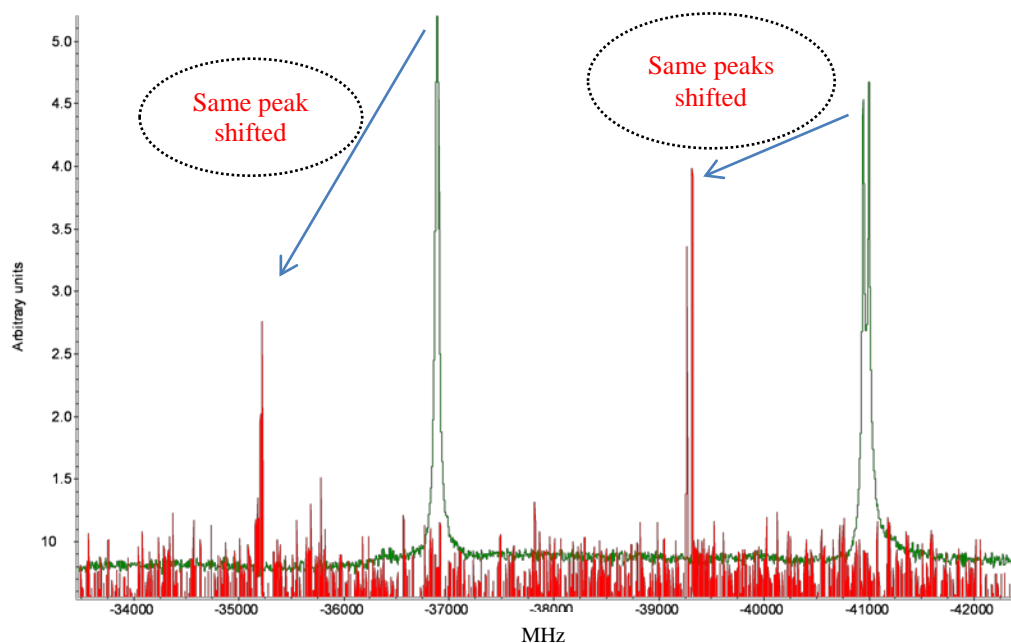


**Figure 5. The  $^{13}\text{C}$  NMR spectrum of the April 2011 SHT sample (A = SHT April 2011, B = BOBCalixC6, and C = Cs-7SB). The labeled peaks in the SHT April 11 spectrum (red curve) are new peaks not seen in the Modifier or in the extractant. These peaks are at 158, 149, 129, 125.5, and 64.6 ppm respectively indicated by the “\*” symbol.**

Figure 5 also shows the  $^{13}\text{C}$ NMR of Cs-7SB and BOBCalixC6 as references. As can be seen from Figure 5, five new peaks were detected: four of them are associated with substituted aromatic ring and the fifth peak at 64.6 is due to ethers. Thus, the impurity is a para- or mono-substituted aromatic containing an ether group. This set of chemical groups is common to the CSSX components and to the chemicals found in the SVOA analysis of the CWT samples. This data again reinforces the suspicion that low molecular weight oxidized aromatics is common to the impurities. Notice that no peak is seen in this spectrum at 165 ppm or higher which would result from a carbonyl as found in the extracted CWT by FTIR.

The fluorine NMR spectra for the SHT April 2011 sample and the Modifier is shown in Figure 6. As can be seen in Figure 6, the number of peaks is the same in both samples and there is a constant horizontal shift in the SHT spectrum due to the fact that no calibration chemical was added to the SHT sample. Qualitatively, the right most peak is a doublet and the most left peak is a singlet and both shifted by the same amount from the same corresponding peaks in the Modifier. Therefore, the impurity observed in the carbon NMR does not contain a fluorine atom in it.





**Figure 6.** The fluorine NMR spectra of the SHT sample from April 2011 (red noisy curve) and that of the Modifier (green curve). The SHT curve is horizontally shifted since it was not spiked in with a calibration marker but the shift is the same for both observable peaks relative to corresponding peaks in the Modifier. Note:  $^{19}\text{F}$  shifts are obtained by dividing the X-axis by 282.

### 3.3 The effect of caustic wash on SHT-11-1035-1040 Aggregate

The effect of washing SHT-11-1035-1040<sup>iii</sup> is shown in Figure 7. Also shown in Figure 7 is the optical appearance (color) of the sample as a result of caustic wash. Inspection of Figure 7 shows leaching of Modifier (as noted by the increase in the Isopar<sup>®</sup> L peaks) and the physical picture of the sample (small insert in Figure 7) shows a noticeable color change. We believe the caustic wash removed impurities associated with the Modifier.

We also conducted a  $^{22}\text{Na}$  exchange between as-received and washed SHT-11-1035-1040 with salt simulant containing  $^{22}\text{Na}$  to determine the level of sodium capacity and infer the effect of caustic washing SHT-11-1035-1040 with 1M caustic. The data from the test is shown in Fig. 8. As can be seen from Fig. 8, the ability to absorb sodium by SHT-11-1035-1040 is comparable to that reported earlier.<sup>7</sup> After caustic washing the SHT-11-1035-1040 the sodium uptake remained the same. Therefore, this solvent is performing as expected and the possible loss in DF may be attributed to operational variations at MCU.

<sup>iii</sup> SHT-11-1035-1040 is a designator for an aggregate sample composed of samples SHT-11-1035, SHT-11-1036, SHT-11-1037, SHT-11-1038, SHT-11-1039 and SHT-11-1040.

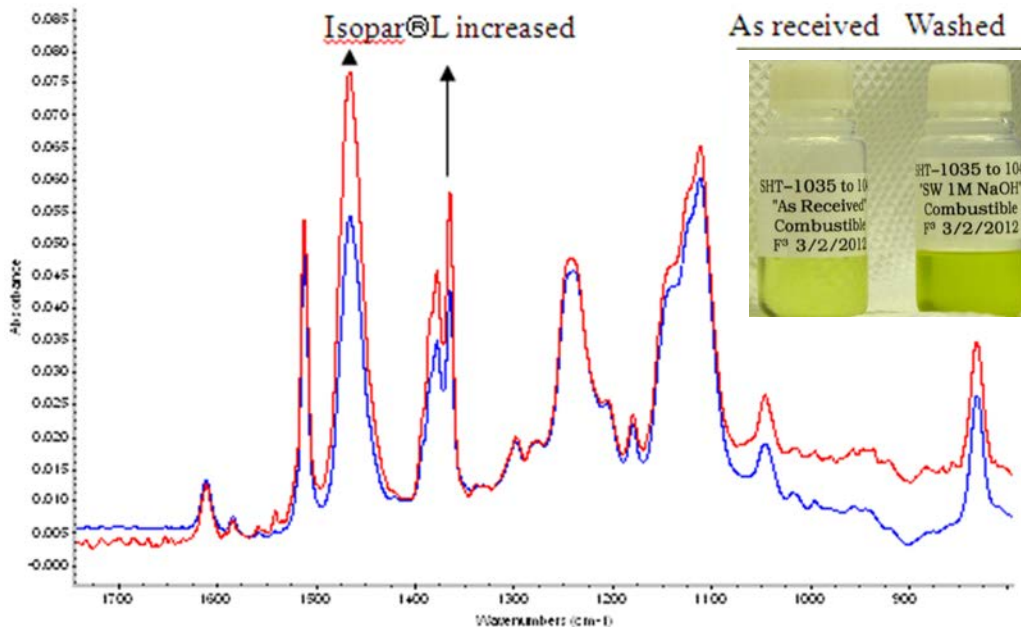


Figure 7. The effect of washing SHT-11- 1035-1040 aggregate with 1M NaOH: It appeared washing leached Modifier.

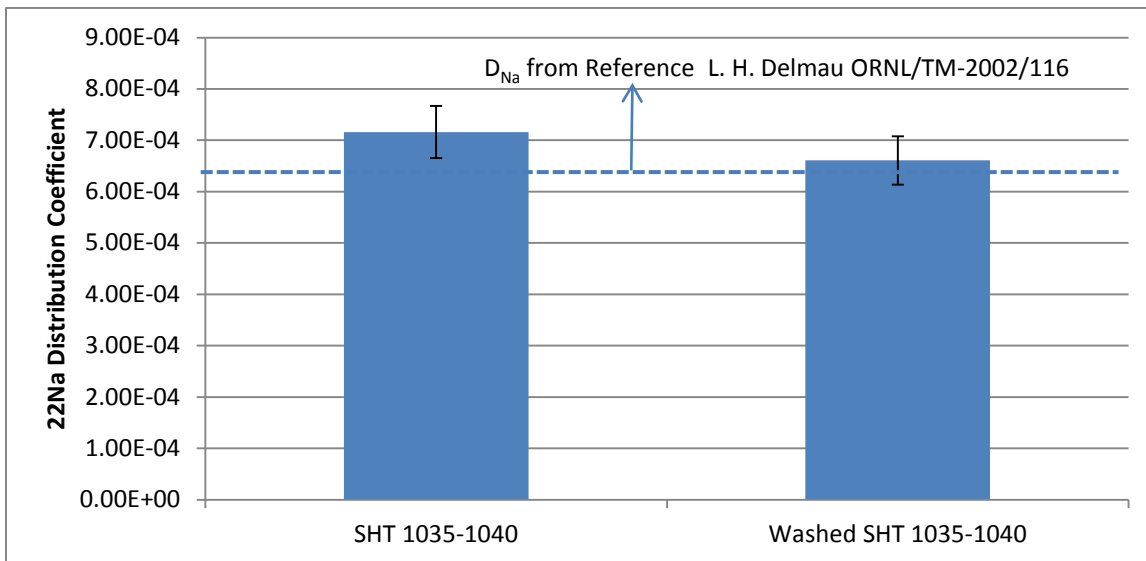


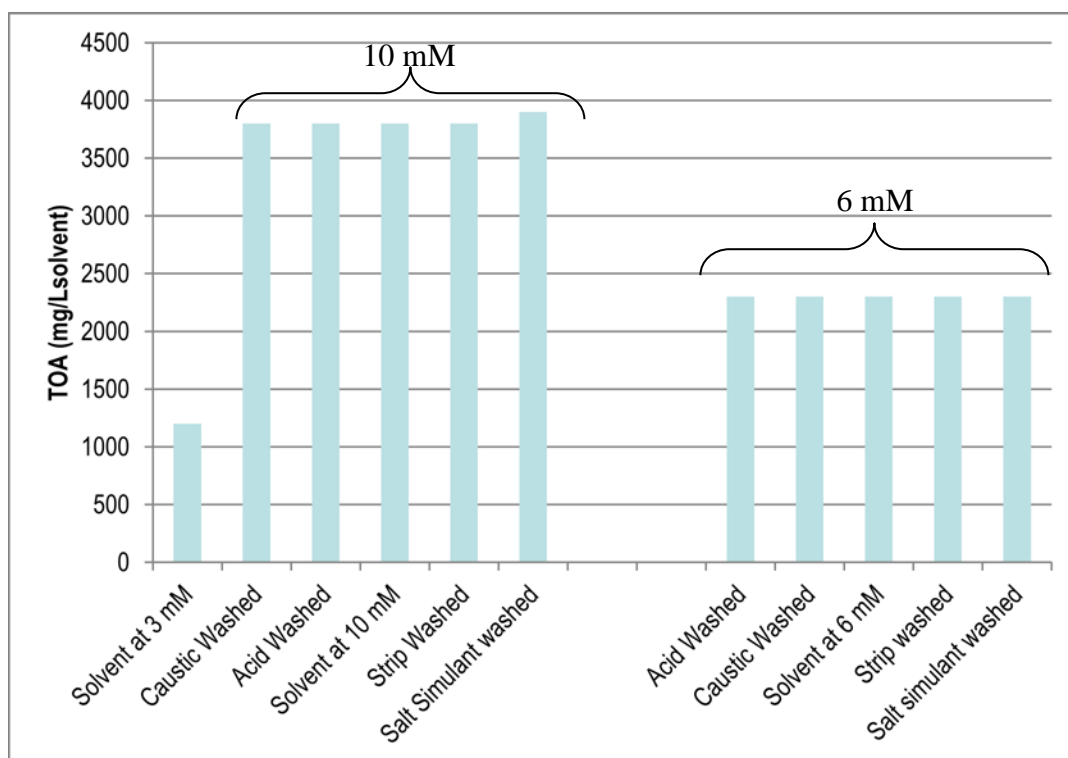
Figure. 8 The effect of caustic washing (1M NaOH) on the sodium extraction of SHT-11-1035-1040. In this test, <sup>22</sup>Na was used as the radiotracer.

### 3.4 The Effect of pH on TOA Partitioning from CSSX Solvent to Aqueous Solutions

During the past two years of MCU operation, the concentration of TOA in MCU has declined to lower concentrations than in prior operations. Recently, Moyer suggested that the low TOA measurements may be an analytical artifact due to variation in the

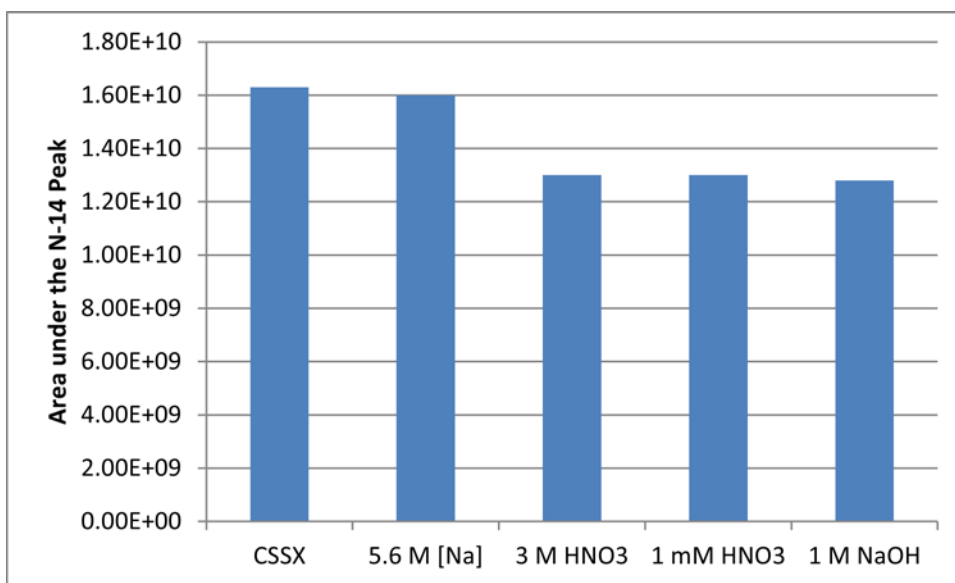
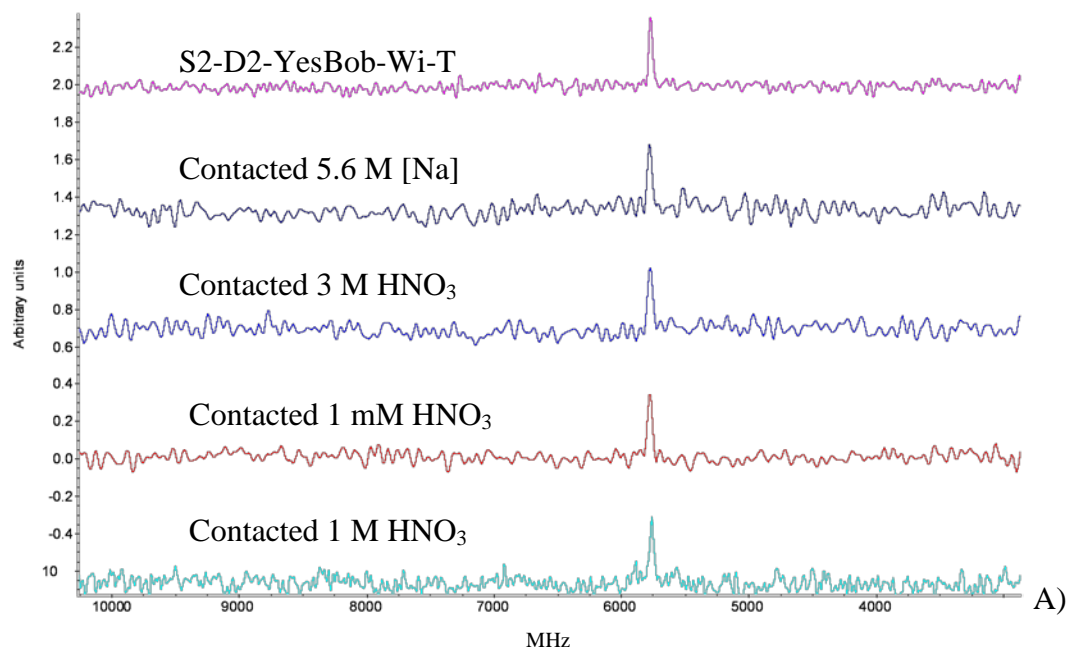
protonation of TOA with solvent samples exposed at differing pH.<sup>2</sup> To investigate that suspicion, personnel took control solvent CSSX (S2-D2-YesBob-WI-T) and added sufficient TOA to raise its level from the nominal value of 3 mM to 6 mM and 10 mM so that data will be above the LOQ of the SVOA method. Using this TOA loaded CSSX, contact tests were conducted with aqueous solutions of different pH that included caustic salt solution ([OH]=1.91M), acid wash (1 M nitric acid), 1 mM nitric strip acid, and caustic wash (1M NaOH). After the contact tests (using the same protocol as use for Extraction-Scrub-Strip tests for qualifying CSSX solvents) the organic was analyzed by SVOA (GC-MS) method and the results are shown in Figure 9.

As can be seen in Figure 9, pH had no effect on the concentration level of TOA in CSSX. The varying pH – and implied protonation variances – did not impact the measured TOA concentrations or its partitioning to the various aqueous solutions. Also shown in Figure 9 is the SVOA results for CSSX containing 3 mM TOA as a quality control check.



**Figure 9. SVOA results from CSSX solvent containing two different levels of TOA (6 and 10 mM) that contacted solutions with different pH values.**

To further verify the SVOA results, the same samples were analyzed by nitrogen NMR. The results are shown in Figure 10. As can be seen in Figure 10, both the raw data (which shows only one nitrogen peak area as expected) and the integral peak area (bottom insert of Figure 10) shows that TOA did not partition into the aqueous solutions examined here and that the values measured by the SVOA method were not affected by the ionization of TOA (pKa ~ 4.2).



B)

**Figure 10.** The  $^{14}\text{N}$  NMR of the samples analyzed by SVOA (6 mM). Figure A) shows the raw data (X-axis is shown in Hz units) and Fig. B) shows the area under the peak.

#### 4.0 Conclusions

Solutions from MCU from about the time period of variable DF performance which covers from April 2011 to September 2011 (during processing of Salt Batch 4) were examined for impurities using chromatography and spectroscopy. The result indicates that impurities were found to be of two types: aromatic containing impurities most likely from Modifier degradation and aliphatic type impurities most likely from Isopar<sup>®</sup> L and TOA degradation. The degradation is believed to be due to radiation rather than solution chemistry changes. Caustic washing the suspected SHT solution with 1M NaOH improved its extraction ability as determined from <sup>22</sup>Na uptake tests. Evidence from this work showed that pH variance in the aqueous solutions within the range of 1M nitric acid to 1.91M NaOH that contacted the solvent samples does not influence the analytical determination of the TOA concentration by GC-MS.

#### 5.0 Recommendations, Path Forward or Future Work

The analyses identified several chemicals as representative of the organic impurities that are present in the aging solvent within MCU. SRNL recommends testing the impact of trace concentrations of these impurities (with the exception of secbutylphenol whose impact was previously investigated) on the cesium mass transfer performance for fresh solvent. Such a study would qualitatively compare the relative impact on cesium mass transfer to the declines observed in MCU.

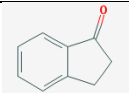
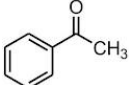
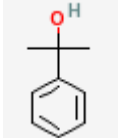
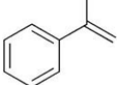
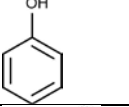
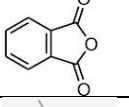

SRNL also recommends performing partitioning tests for the identified impurities into various wash solutions and acids to understand removal efficiency of the impurities from the process.

SRNL recommends tests that irradiate a solvent to exposures near the four year MCU history and examining that solvent for similar chemical and performance features by means used in the current study.

SRNL also recommends conducting organic impurities extraction from Tank 49H salt solution feed and corresponding analytical analysis.

## 6.0 Appendix A: Molecular representation of the impurities found in CWT 817-904 and 1119-1120 by SVOA

**Table 1A. Impurities found in CWT-11-817 to 904 and CWT-11-1119 to 1120 by SVOA**

Compound	Molecular representation
1-Indanone	
Acetophenone	
Dimethylbenzyl alcohol	
Alpha methyl styrene	
Phenol	
phthalic anhydride	
2,4,4 trimethyl-4-hexene	

## 7.0 References

<sup>1</sup> T. E. Laupa, "Response to Path Forward for the Degradation of the Cs-137 Decontamination Factor at the MCU," X-ESR-H-00362, September 29, 2011.

<sup>2</sup> B. A. Moyer, "Recommendations Related to MCU DF Performance," August 29, 2011.

<sup>3</sup> K. Adu-Wusu, D. D. Walker, T. L. White, and S. L. Crump, "Preparation of Caustic-side Solvent Extraction (cssx) Solvent with Bobcalixc6 for Wright Industries -component Amounts, Analytical and Quality Assurance Results," SRNL-WPT-2005-00134, 12/2005.

<sup>4</sup> C. L. Crawford, F. F. Fondeur, R. A. Peterson, and T. L. White, "Radiation Stability of Calixarene Based Solvent System," WSRC-TR-98-00371, October 1998.

<sup>5</sup> P. V. Bonnesen, F. V. Sloop, Jr., and N. L. Engle, "Stability of the Caustic-Side Solvent Extraction (CSSX) Process Solvent: Effect of High nitrite on Solvent Nitration," ORNL/TM-2002/115, July 2002

<sup>6</sup> T. B. Peters, A. L. Washington, II, F. F. Fondeur, and S. D. Fink, "Supplemental Analyses for Solvent Hold Tank Samples MCU-11-314, MCU-11-315, MCU-11-316, MCU-11-317, MCU-11-318 and MCU-11-319," SRNL-STI-2011-00591, September 2011.

<sup>7</sup> L. H. Delmau, D. A. Bostick, T. J. Haverlock, and B. A. Moyer, "Caustic-Side Solvent Extraction: Extended Equilibrium Modeling of Cesium and Potassium Distribution Behavior," ORNL/TM-2002/116.