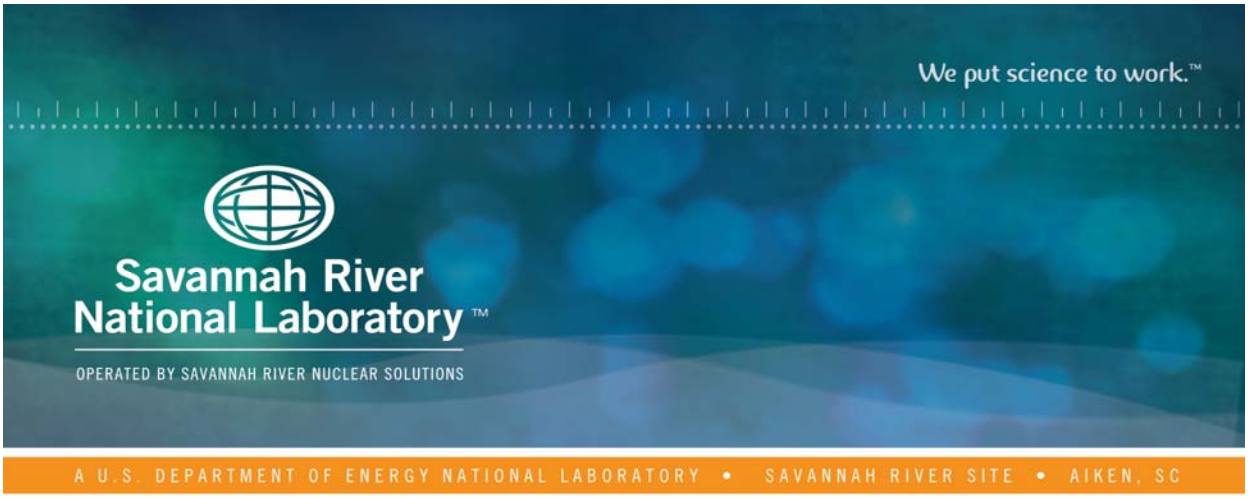

Sample Results from the Extraction, Scrub, and Strip Test for the Blended NGS Solvent

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EXECUTIVE SUMMARY

This report summarizes the results of the extraction, scrub, and strip testing for the September 2013 sampling of the Next Generation Solvent (NGS) Blended solvent from the Modular Caustic Side-Solvent Extraction Unit (MCU) Solvent Hold Tank. MCU is in the process of transitioning from the BOBCalixC6 solvent to the NGS Blend solvent. As part of that transition, MCU has intentionally created a blended solvent to be processed using the Salt Batch program. This sample represents the first sample received from that blended solvent. There were two ESS tests performed where NGS blended solvent performance was assessed using either the Tank 21 material utilized in the Salt Batch 7 analyses or a simulant waste material used in the V-5/V-10 contactor testing. This report tabulates the temperature corrected cesium distribution, or D_{Cs} values, step recovery percentage, and actual temperatures recorded during the experiment. This report also identifies the sample receipt date, preparation method, and analysis performed in the accumulation of the listed values. The calculated extraction D_{Cs} values using the Tank 21H material and simulant are 59.4 and 53.8, respectively. The D_{Cs} values for two scrub and three strip processes for the Tank 21 material are 4.58, 2.91, 0.00184, 0.0252, and 0.00575, respectively. The D-values for two scrub and three strip processes for the simulant are 3.47, 2.18, 0.00468, 0.00057, and 0.00572, respectively. These values are similar to previous measurements of Salt Batch 7 feed with lab-prepared blended solvent. These numbers are considered compatible to allow simulant testing to be completed in place of actual waste due to the limited availability of feed material.

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LIST OF ABBREVIATIONS

ADS	Analytical Development Section
ARP	Actinide Removal Process
DSS	Decontaminated Salt Solution
DWPF	Defense Waste Processing Facility
ELN	Electronic Laboratory Notebook
ESS	Extraction, Scrub, and Strip
MST	Monosodium Titanate
MCU	Modular Caustic Side-Solvent Extraction Unit
NGS	Next Generation Solvent
SB	Salt Batch
SRNL	Savannah River National Laboratory
TTQAP	Task Technical Quality Assurance Plan

1.0 Introduction

MCU has begun transitioning from the BOBCalixC6 solvent to NGS using a 1:1 blend strategy in August 2013. The early stages of this process involved several prerequisite tests as well as intentionally developing a blend material to ease the transition to the new solvent.[1] MCU is currently processing the NGS blended solvent through the Salt Batch program and has submitted preliminary for the NGS blended solvent program to SRNL for analysis. ARP/MCU has sent SRNL a processed sample of the NGS blended solvent without any additional cleaning for performance testing and verification of quality. A TTQAP has been developed to verify that the performance of the solvent prepared in MCU matches the performance measured in the lab with real waste and simulant solutions.[2] The samples were received on September 17, 2013 for ESS testing and subsequent ^{137}Cs determination, density, and solvent quality analysis. The density and solvent quality analysis were reported separately in SRNL-STI-2013-00661.

2.0 Experimental Procedure

The MCU solvent samples arrived at the SRNL on September 17, 2013 as part of the quarterly solvent hold tank sampling and the NGS Demonstration Plan preliminary sampling requirements. Six peanut vials of solvent labeled MCU 1403-1408 were delivered to SRNL and subsequently composited to one container. This composite sample was used as the organic feed for the ESS testing. The ^{137}Cs activity of the solvent was measured as 4.21E+05 dpm/mL.[3] The aqueous feed was provided by two separate sources. The real waste material was taken from unprocessed Tank 21 material obtained for the recently completed Salt Batch 7 report.[4] This sample was filtered to remove the insoluble solids present but not contacted with MST. A simulant waste was obtained from a current V-05/V-10 contactor test using a currently accepted recipe.[5] This simulant was subsequently spiked with ^{137}Cs to a concentration of 1.97E+06 dpm/mL (8.87E+05 pCi/mL) and further utilized without additional alteration.

Using this material, the researchers performed two ESS tests. All of the tests used the same general protocol as used in the previous Macrobatch testing.[4] The real waste test used a nominal starting volume of 80 mL of aqueous feed from Tank 21H and 20 mL of previously contacted MCU-13-1403-1408 blended solvent for extraction. In the simulant waste test, 80 mL of simulant material was combined with 20 mL of the previously contacted MCU blended solvent for extraction. For both tests, the scrub and strip solutions were 0.025 M NaOH and 0.01 M boric acid, respectively. Confirmation of pH was performed by pH strip paper. Organic and aqueous samples from each step including extraction, two scrubs, and three strips were removed and sent to ADS for gamma spectroscopy. Due to their low activity, the aqueous samples were submitted without dilution for analysis. However, the organic samples (~0.9 grams) were each diluted with approximately 4 mL of Isopar® L to reduce the external dose and maintain radiological hood limits in ADS. The organic dilution factors have been addressed to the D_{Cs} value calculations. A cell blank is not normally used in the radiochemical portion of this analysis and therefore is not addressed in this report.

3.0 Results and Discussion

There were two ESS tests performed to compare the solvent efficiency using a real waste material and a ^{137}Cs spiked simulant. First, we utilized a portion of the Tank 21H material remaining from the Salt Batch 7 analysis. The second test utilized a Cs spiked simulant from a current V-5/V-10

testing using a currently accepted waste recipe. Table 3-1 shows the results from the ESS tests. These results were temperature corrected to the normal process operating temperatures (i.e., 23 ºC for extraction and scrub and 33 ºC for stripping).[4] The temperature in the shielded cells during the ESS test ranged from 17.4 ºC to 21.5 ºC with an average temperature of 19.1 ºC. As a comparison, the results from the previous Salt Batch 7 Hot Blend Macrobatch qualification ESS test are displayed.[4]

Table 3-1. Results of Radiochemical Analysis, D_{Cs} Values

Material	Extraction D_{Cs}	Scrub#1 D_{Cs}	Scrub#2 D_{Cs}	Strip#1 D_{Cs}	Strip#2 D_{Cs}	Strip#3 D_{Cs}
Acceptable Range, BOBCalixC6 Solvent	>8 (50-70 expected)	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
Salt Batch 7 Hot blend[α]	58.6	2.32	2.58	0.00057	0.0026	0.011
NGS Blended Solvent with Tank 21H material	59.4	4.58	2.91	0.00184	0.0252[β]	0.00575
NGS Blended Solvent with V-05/V-10 simulant	53.8	3.47	2.18	0.00468	0.00057	0.00572[β]

[α] Data taken from Salt Batch 7 report: SRNL-STI-2013-00437, used MCU BOBCalixC6 solvent mixed with fresh NGS solvent.
[β]Data points are correct. Stripping in the blended solvent is not as consistent as the BOBCalixC6 solvent.

For the MCU solvent test, the extraction, scrub and strip data shown in Table 3-1 are mostly in the expected range for the NGS blended solvent program. The expected values for extraction have a range from 50-70 which fully supports the tabulated values. Additionally, the strip has continued to show improvement with lower D_{Cs} values than the prior baseline MCU solvent. Occasionally, the strip values will be closer to those seen in the old solvent as seen in strip 2 of the real test and strip 3 of the simulant test. This result is possibly attributed to the uncertainty in the step recovery from these steps. The scrub portion of the test has shown higher than expected values for ^{137}Cs remaining in the organic solvent. These higher values represent less ^{137}Cs being pulled out in the scrub steps decreasing the amount of activity being put back in the salt feed. The higher than normal scrub values that are established for the BOBCalixC6 solvent have also been shown previously in the Salt Batch 7 report. The scrub values are shown to exceed limits in every instance where the NGS blended solvent was used indicating that a change in the scrub limits be investigated. Since these limits were developed for the previous solvent, additional measurements may show the current values to become more typical.

Table 3-2. pH values for Both Real and Simulant ESS Test

Materials	Extraction	Scrub #1	Scrub #2	Strip #1	Strip #2	Strip #3
Real Waste	N/A	14	12	6	7	7
Simulant	N/A	14	13	7	5	6
Expected pH of cold chemical	N/A	12	12	5.6	5.6	5.6

Table 3-2 shows the pH measured from each of the scrub and strip steps of the ESS process. This measurement confirms that the correct material has been used in the each step. The pH in the scrub steps should be in the 12-14 range while the strips steps nominally have a pH in the 5-7

range. In the process, there is no mechanical carryover of scrub solution into the contactor during the ESS process giving some variation in the pH. However, all values measured fall within the expected range.

Table 3-3. Cesium activity in the solvent from the ESS process

ESS step	^{137}Cs activity (pCi/mL)			
	SB7 BOBCalixC6 Test	SB7 NGS Hot Blend Test	Real Waste Test	Simulant Test
Original	0.00E+00	6.17E+04	1.90E+05	1.90E+05
Extraction	9.46E+07	1.45E+08	1.72E+08	5.89E+06
Scrub #1	8.80E+07	1.30E+08	1.63E+08	5.48E+06
Scrub #2	5.16E+07	1.03E+08	1.53E+08	5.38E+06
Strip #1	1.64E+07	6.04E+05	4.41E+06	4.22E+05
Strip # 2	3.73E+06	3.27E+04	3.53E+06	1.56E+04
Strip #3	7.77E+05	5.94E+03	2.48E+05	1.56E+03

Table 3-3 shows the ^{137}Cs activity in the solvent in each step of the ESS process for the real waste and simulant tests using the NGS blended solvent and comparing that to the original BOBCalixC6 solvent utilized previously by MCU and the NGS Hot Blend Test both from the SB7 report. The generally consistent activity through the extraction and scrub steps are expected for this process proving that very minimal ^{137}Cs is removed in the scrub portion. The exponential decline in activity in the stripping process shows the intended removal of ^{137}Cs from the solvent as expected. The two order of magnitude disparity in the simulant versus the real waste correlates with the difference in the starting activity of the aqueous phase. The starting activities in the aqueous phase were 4.64E+07 pCi/mL for the real waste/SB7 tests and 8.87E+05 pCi/mL for the and simulant test. Additionally, the NGS blended solvent used in the real waste nearly doubles the activity of the Salt Batch 7 BOBCalixC6 test and also drops off to less than half the activity in the final strip. This is indicative of the better uptake and removal of ^{137}Cs by the NGS blended solvent even in this blended state.

3.1 Quality Assurance

This report was developed in accordance with the protocols identified in Task Technical and Quality Assurance Plan SRNL-RP-2013-00536.[2] The work was requested in HLW-DWPF-TTR-2013-0005 Rev. 0.[6]

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. The data from this experiment is contained in an electronic laboratory notebook (ELN).[7]

4.0 Conclusions

This report tabulates the temperature corrected D_{Cs} value[4], step recovery percentage, and actual temperatures recorded during the experiment. The calculated extraction D_{Cs} values for the Tank 21 material and simulant are 59.4 and 53.8, respectively. The D_{Cs} values for two scrub and three strip processes for the Tank 21H material are 4.58, 2.91, 0.00184, 0.0252, and 0.00575, respectively. The D_{Cs} values for two scrub and three strip processes for the simulant are 3.47, 2.18, 0.00468, 0.00057, and 0.00572, respectively. These numbers are compatible to allow simulant testing to be completed in place of actual waste.

5.0 References

[1] Smith, T.E. "Technical Evaluation of Mirco-Batchwise Next Generation Solvent Demonstration and Engineering Recommendations", X-ESR-H-00570, Rev. 0, September 2013.

[2] Peters, T.B, Washington, A.L., Fondeur, F.F. "Task Technical and Quality Assurance Plan for Routine Samples in Support of ARP and MCU", SRNL-RP-2013-00536, Rev. 0, September 2013.

[3] Fondeur, F.F., Taylor-Pashow, K.M.L. "Solvent Hold Tank Sample Results for MCU-13-1403/1404/1405/1406/1407/1408: Quarterly Sample from September 2013", SRNL-STI-2013-00661, November 2013.

[4] Peters, T.B., Washington, A.L. "Sample Results from the Interim Salt Disposition Program Macrobatch 7 Tank 21H Qualification Samples", SRNL-STI-2013-00437, July 2013.

[5] Herman, D.T., Duignan, M.R., Williams, M.R. Peters, T.B., Poirier, M.R., Fondeur, F.F. "Mass Transfer and Hydraulic Testing of the V-05 and V-10 Contactors with the Next Generation Solvent", SRNL-STI-2013-00413, July 2013.

[6] Ketusky, E. "Qualification of Salt Batch 6 with NGS", HLW-DWPF-TTR-2013-0005, Rev. 0, November 26, 2013.

[7] Electronic Laboratory Notebook, E5690-00077-02.

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