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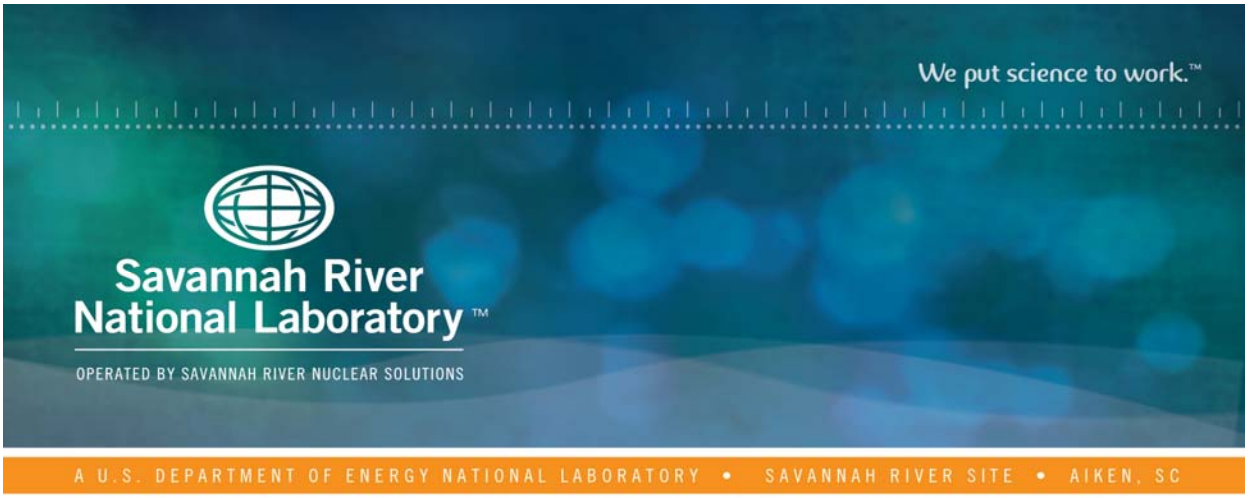

Extraction, Scrub, and Strip Test Results from the Interim Salt Disposition Program Macrobatch 11 Tank 21H Qualification Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Macrobatch (Salt Batch) 11 for the Interim Salt Disposition Program (ISDP). The Salt Batch 11 characterization results were previously reported.[i,ii]

An Extraction, Scrub, and Strip (ESS) test was performed to determine cesium distribution ratios ($D_{(Cs)}$) and cesium concentration in the strip effluent (SE) and decontaminated salt solution (DSS) streams; this data will be used by Tank Farm Engineering to project a cesium decontamination factor (DF). This test used actual Tank 21H material, and a sample of the Next Generation Solvent (NGS) Blend solvent currently being used at the Modular Caustic-Side Solvent Extraction Unit (MCU).

The ESS test showed acceptable performance with an extraction $D_{(Cs)}$ value of 117. This value is consistent with results from previous salt batch ESS tests using similar solvent formulations. This cesium removal efficiency is better than the predicted value of 50.8 from a recently created $D_{(Cs)}$ model.[iii]

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LIST OF ABBREVIATIONS

$D_{(Cs)}$	Distribution Ratio for Cesium
DF	Decontamination Factor
DSS	Decontaminated Salt Solution
ESS	Extraction, Scrub, Strip
ISDP	Interim Salt Disposition Program
MCU	Modular Caustic Side Solvent Extraction Unit
NGS	Next Generation Solvent
SE	Strip Effluent
SRNL	Savannah River National Laboratory
TOA	Trioctylamine
TTQAP	Task Technical and Quality Assurance Plan

1.0 Introduction

This report provides distribution ratios for cesium ($D_{(Cs)}$) and cesium concentration in the SE and DSS streams obtained from performance of an ESS test using the Tank 21H qualification sample. This data will be used by Tank Farm Engineering to project a cesium DF for ISDP Macrobatch (Salt Batch) 11. Previous documents reported the chemical and radiological characterization required for qualification of the salt batch. This work was specified in a Technical Task Request[iv] as Task 5 and in a Task Technical and Quality Assurance Plan (TTQAP).[v] This task is not required for salt batch qualification unless use of a different solvent is implemented at MCU; data/observations from this demonstration will be used for process knowledge. Details of the work are contained in a controlled laboratory notebook.[vi]

2.0 Experimental Procedure

For the ESS test, material from the Tank 21H composite (samples HTF-21-17-70, -71, and -72) was used. The test used the same general protocol as used in previous macrobatch salt waste testing and is formalized in an SRNL manual.[vii] The test used a nominal starting volume of 80 mL of aqueous salt solution feed and 20 mL (4:1 aqueous:organic volume ratio) of freshly sampled (from MCU) NGS blend.♥ The scrub and strip solutions were 0.025 M NaOH and 0.01 M boric acid, respectively, and an organic:aqueous volume ratio of 3.75:1 was used.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.[viii] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[ix]

3.0 Results and Discussion

Table 1 shows the results from the ESS test, corrected to the normal process operating temperatures (i.e., 23 ºC for extraction and scrub and 33 ºC for strip). For these tests, the temperature correction factors for the NGS solvent were used (see Appendix).

The temperature in the Shielded Cells during the ESS test ranged from 18 ºC to 22 ºC with an average temperature of 20.3 ºC. As a comparison, the results from the previous macrobatch qualification ESS test (using the NGS Blend) are displayed.[x]

♥ The NGS-MCU blend was initially a 50/50 volume % blend of MCU solvent and a prepared mixture of compounds, that once mixed, gives a nominal composition as follows: 0.0035 M BOBCalixC6 [Calix[4]arenebis (*tert*-octylbenzo-crown-6)], 0.5M Cs-7SB Modifier [1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-secbutylphenoxy)-2-propanol], 0.0015 M trioctylamine (TOA), 0.003 M TiDG [*N,N',N"*–tris(3,7-dimethyloctyl)guanidine], 0.0465 M MaxCalix [1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arenebenzocrown-6], and the balance Isopar ™ L. Since the material was prepared in 2013, the TOA and BOBCalixC6 have been depleting with no further trims of these two materials. As a result the nominal concentration of the MaxCalix has increased from the original 46.5 mM.

Table 1. Cesium Distribution Ratios ($D_{(Cs)}$) for the ESS Tests

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
SB 11 NGS Blend	117	4.80	3.70	0.000516	0.000783	0.0117
SB 10 NGS Blend	110	12.1	13.1	0.00760	0.00263	0.00810

The current test shows the expected behaviors, with good overall performance. The extraction step $D_{(Cs)}$ is higher than predicted, but equivalent to that for Salt Batch 10. The first and second strip step $D_{(Cs)}$ for SB11 are better than the previous SB10 ESS test, while the third strip step $D_{(Cs)}$ is worse; although, the average of the strip step $D_{(Cs)}$ for Salt Batch 10 (0.00611) is about the same as for Salt Batch 11 (0.00433). The relatively high scrub results continue to indicate retention of ^{137}Cs in the organic phase during scrubbing which is a desired effect [⊗] and is likely correlated with the high pH values in the used scrub solution (see Table 2). The high pH values in the scrub may be due to aqueous carryover from the extraction step.

In past years when MCU was using an older solvent formulation, SRNL had an extraction stage $D_{(Cs)}$ predictor model which allowed SRNL to get an early indication of possible extraction problems. With the new NGS formulation this prediction was lacking. A new extraction stage $D_{(Cs)}$ predictor model has been created by SRNL,[iii] and this model predicts an extraction $D_{(Cs)}$ value of 50.8, which is not as high as the experimental value. SRNL is investigating the possible reasons for improved cesium removal compared to what was predicted.

3.1.1 Scrub Effluent, SE and DSS Results

During, and at the end of the ESS test, the gamma activities and pH in the scrub effluent, SE and the DSS for a single extraction were measured (see Table 2).

Table 2. Scrub Effluent, Strip Effluent and DSS ^{137}Cs Results

Sample	^{137}Cs (dpm/mL)	pH
Salt Batch 11 Feed	3.42E+08	14
DSS	7.60E+06	14
Scrub Effluent #1	2.08E+08	14
Scrub Effluent #2	2.47E+08	12
Strip Effluent #1	3.42E+09	7
Strip Effluent #2	4.10E+07	5
Strip Effluent #3	2.09E+06	5

[⊗] Any cesium that gets scrubbed out in the scrub stages proceeds forward into the extraction contactors where it will be extracted.

The 1-σ analytical uncertainty on the ^{137}Cs activity is 5%. The ^{137}Cs results are typical, given the high ^{137}Cs activity in the feed (3.42E+08 dpm/mL). The analytical uncertainty is ±1 pH unit for the pH measurement performed with colorimetric strips. The pH results from the test are typical.

4.0 Conclusions

Results of the ESS test are typical of the salt batch feeds and the solvent in use, other than a higher than anticipated extraction step result. There are no anticipated issues for cesium removal while SRNL is investigating the possible reasons for improved cesium removal compared to what was predicted.

Appendix. Temperature Correction Factors for the ESS Tests

The actual MCU facility uses active temperature control to keep the extraction step at 23 °C, and the strip steps at 33 °C. However, the ESS tests do not have active temperature control. During each step of an ESS test, the calculated distribution values must be corrected for temperature. The general formula for temperature correction is as follows:

correction factor = EXP((**COEF**/0.0083144)*((1/**TEMP**)-(1/(**STEP**)))) (Eqn. 1)

where "COEF" is the particular temperature coefficient for the step in question, the "TEMP" is the ambient temperature, in Kelvin, and "STEP" is 296.15 for extraction and scrub and 306.15 for strip steps. There is one set of coefficients for the MCU BOBCalixC6-based solvent, and one set of coefficients for use in NGS type solvents with MaxCalix.

Table 3 lists the temperature coefficients for each step in an ESS test. The coefficients for the NGS solvent are derived from the van't Hoff formalism in Eqn. 1 of the applicable reference in Table 3.

Table 3. Temperature Coefficients

Step	MCU(BOBCalix) [xi]	NGS [xii]
Extraction	-47.95	-90.12
Scrub#1	-86.82	-115.5
Scrub#2	-74.24	-91.40
Strip#1	-79.36	-80.18
Strip#2	-82.94	-143.4
Strip#3	-82.49	-65.63

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