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Extraction-Scrub-Strip Test Results from the Interim Salt Disposition Program Macrobatch 9 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) 9 for the Interim Salt Disposition Program (ISDP). The Salt Batch 9 characterization results were previously reported.[i,ii]

An Extraction-Scrub-Strip (ESS) test was performed to determine cesium distribution ratios ($D_{(Cs)}$) and cesium concentration in the strip effluent 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 blend solvent prepared by SRNL that mimics the solvent composition 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 52.4. This value is consistent with results from previous salt batch ESS tests using similar solvent formulations. This compares well against the predicted value of 56.5 from a recently created $D_{(Cs)}$ model.

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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
SRNL	Savannah River National Laboratory
TOA	trioctylamine
TTQAP	Task Technical and Quality Assurance Plan

1.0 Introduction

This report provides $D_{(Cs)}$ and cesium concentration in the strip effluent 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) 9. Previous documents reported the chemical and radiological characterization required for qualification of the salt batch.[i,ii] This work was specified in a Technical Task Request[iii] and in a Task Technical and Quality Assurance Plan (TTQAP).[iv] Details of the work are contained in controlled laboratory notebooks.[v]

2.0 Experimental Procedure

For the ESS test, material from the Tank 21H composite (from samples HTF-21-15-106, -107, and -108) was used. The test used the same general protocol as used in previous macrobatch salt waste testing and is formalized in a SRNL manual.[vi] 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) Next Generation Solvent (NGS) blend. ♥ The scrub and strip solutions were 0.025 M NaOH and 0.01 M boric acid, respectively, and used an organic:aqueous volume ratio of 3.75:1.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 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

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 15.4 ºC to 19.7 ºC with an average temperature of 18.5 ºC. As a comparison, the results from the previous macrobatch qualification ESS test (using the same solvent) are displayed.[vii]

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

Material	Extraction	Scrub#1	Scrub#2	Strip#1	Strip#2	Strip#3
SB 9 NGS Blend	52.4	2.78	0.781	0.000266	0.000396	<0.0019
SB 8 NGS Blend	52.5	7.39	2.67	0.00226	0.00106	0.00671

♥ The NGS-MCU blend is 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, 0.5M Cs-7SB Modifier, 0.0015 M trioctylamine (TOA), 0.003 M TiDG, 0.0465 M MaxCalix, and the balance Isopar ™ L.

The current test shows the expected behaviors, with good overall performance. While the extraction step $D_{(Cs)}$ is slightly lower than that for Salt Batch 8, the strip step $D_{(Cs)}$ are better than typical. The variation in the scrub results is not unusual and should not be a concern. Any cesium that gets scrubbed out in the scrub stages is then refluxed back into the extraction contactors where it will be extracted.

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 solvent formulation this prediction was lacking. A new extraction stage $D_{(Cs)}$ predictor model has been created by SRNL,[viii] and this model predicts an extraction $D_{(Cs)}$ value of 56.5.

3.1.1 Strip Effluent and DSS Results

During, and at the end of the ESS test, the gamma activities and pH in the strip effluent and the DSS for a single extraction were measured (Table 2). As a point of comparison, the values for the Tank 21H Salt Batch 9 sample are also given in the table.

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

Sample	^{137}Cs (dpm/mL)	pH
Salt Batch 9 Feed	5.43E+08	14
DSS	1.82E+07	14
Strip Effluent #1	5.81E+09	8
Strip Effluent #2	7.87E+07	7
Strip Effluent #3	3.17E+06	7

The 1-σ analytical uncertainty on the ^{137}Cs activity is 5%. The ^{137}Cs results are typical, given the large increase in ^{137}Cs activity in the feed. 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. There is no unexpected behavior and there are no anticipated issues for cesium removal.

Appendix. Temperature Correction Factors for the ESS Tests

The actual MCU facility uses active temperature control to keep the extraction and scrub steps 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 solvent, and one set of coefficients for use in NGS type solvents with MaxCalix (NGS, cold blend, hot blend).

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 equation 1 of the applicable reference in Table 3.

Table 3. Temperature Coefficients

Step	MCU [ix]	NGS [x]
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

5.0 References

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[iv] T. B. Peters and D. H. Jones, "Task Technical and Quality Assurance Plan for Qualification of Salt Batches for Feed to ISDP", SRNL-RP-2015-00704, Rev. 0, September 2015.

[v] T. B. Peters, "Salt Batch 9 Qualification", ELN A4571-00084-23.

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