

Contract No:

This document was prepared in conjunction with work accomplished under Contract No. 89303321CEM000080 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

- 1) warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
- 2) representation that such use or results of such use would not infringe privately owned rights; or
- 3) endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.



**Savannah River
National Laboratory®**

A U.S. DEPARTMENT OF ENERGY NATIONAL LAB • SAVANNAH RIVER SITE • AIKEN, SC • USA

Summary of MCU Flush Sample Results

T. B. Peters

October 2021

SRNL-STI-2021-00460, Revision 0

SRNL.DOE.GOV

DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *MCU*

Retention: *Varies*

Summary of MCU Flush Sample Results

T. B. Peters

October 2021

Savannah River National Laboratory is operated by
Battelle Savannah River Alliance for the U.S. Department
of Energy under Contract No. 89303321CEM000080.



REVIEWS AND APPROVALS

AUTHORS:

T. B. Peters, Chemical Flowsheet Development	Date
--	------

TECHNICAL REVIEW:

M. S. Hay, Chemical Flowsheet Development, Reviewed per E7 2.60	Date
---	------

APPROVAL:

G. A. Morgan, Manager Chemical Flowsheet Development	Date
---	------

F. M. Pennebaker, Director Chemical Processing	Date
---	------

K. Fernandez MCU & Salt/Sludge	Date
-----------------------------------	------

EXECUTIVE SUMMARY

Starting in May 2021, samples from the Modular Caustic-Side Solvent Extraction Unit (MCU) were sent to the Savannah River National Laboratory (SRNL) for analysis. Samples resulting from flushes of the Strip Effluent Hold Tank (SEHT) and the Decontaminated Salt Solution Hold Tank (DSSHT) were sent on multiple dates. Over the range of samples, all had less than detectable concentrations of Isopar-L[™], and there was an overall decline in ¹³⁷Cs activity.

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF ABBREVIATIONS	viii
1.0 Introduction	1
2.0 Background	1
3.0 Experimental Procedure	1
4.0 Results and Discussion	3
5.0 Conclusions	3
6.0 References	4

LIST OF TABLES

Table 1. Initial Combined DSSHT and SEHT Sample Results	2
Table 2. Corrected Combined DSSHT and SEHT Sample Results	3

LIST OF ABBREVIATIONS

CDT	Contactactor Drain Tank
DIW	De-Ionized Water
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
DSSHA	Decontaminated Salt Solution Hydraulic Accumulator
MCU	Modular Caustic-Side Solvent Extraction Unit
SED	Strip Effluent Decanter
SEHT	Strip Effluent Hold Tank
SRNL	Savannah River National Laboratory
SVOA	Semi-Volatile Organic Analysis
SSFT	Salt Solution Feed Tank
SSRT	Salt Solution Receipt Tank

1.0 Introduction

The Modular Caustic-Side Solvent Extraction Unit (MCU) stopped operations on May 22, 2019. As part of a plan to flush the MCU system to support placing MCU in a suspended operations state, samples were sent to the Savannah River National Laboratory (SRNL) for analysis for both Isopar-L TM content, as well as ¹³⁷Cs content.

2.0 Background

Samples received from MCU were the result of flushes of various parts of the system, but ultimately described as either SEHT or DSSHT flushes. The sample results have been previously reported in separate documents.^{1,2,3,4}

The first SEHT contents from which the sample was pulled on 5/6/21 consisted of material from flushing and draining the Strip Effluent Decanter (SED) as part of MCU de-inventory/flushing activities. The flush was performed with strip acid (0.01 M boric acid).

The first DSSHT sample contents (pulled 5/27/21) were deionized water (DIW) flushes of the Salt Solution Receipt Tank (SSRT) 1 & 2, and Salt Solution Feed Tank (SSFT) which were originally at heel. The contents were transferred through the Contactor Drain Tank (CDT) to the DSSHT. Included in the DSSHT was drainback from the last SEHT to Tank 50 transfer (the shared transfer line drains back to the DSSHT).

The second SEHT sample (pulled 5/28/21) contents were the heel from the 1st SEHT flush batch (Strip Effluent Decanter draining and flushing with 0.01 M boric acid) and DIW added directly to SEHT as part of the MCU de-inventory/flushing activities.

The second set of DSSHT sample contents were pulled on 6/25/21 and included heel from the first DSSHT flush batch, DIW backflush of the DSS (Decontaminated Salt Solution) Coalescer, DIW flushes of the DSS Hydraulic Accumulator (DSSHA), DIW flush of the CDT, and DSS Decanter drain material. The DIW flushes from the coalescer, DSSHA, and CDT were pumped through the DSS Decanter to allow for flushing of the DSS Decanter.

The third set of DSSHT samples was pulled from the DSSHT on 7/15/21. The DSSHT contents included heel from the second DSSHT flush batch, and a DIW flush of the DSSHT itself.

3.0 Experimental Procedure

Samples were received in 15 mL p-nut vials. Each of them contained a single phase of material with no obvious discoloration or second phase. Of each set of samples (2 – 3 per set), one was sent in its entirety for the semi-volatile organic analysis (SVOA) (Isopar-L TM). An aliquot from a second vial was used for gamma scan (¹³⁷Cs), and any remaining samples or vials set aside for later use. The results are reported in Table 1.

Table 1. Initial Combined DSSHT and SEHT Sample Results

Sample ID	Type	Isopar-L (mg/L)	Cs-137 (pCi/mL)
MCU-21-1, 2, 3	SEHT	<7	2.13E+08 (5.0%)
MCU-21-4, 5, 6	DSSHT	<7	5.63E+07 (5.0%)
MCU-21-7, 8, 9	SEHT	<7	4.13E+06 (5.0%)
MCU-21-10, 11, 12	DSSHT	<7	6.31E+03 (8.2%)
MCU-13, 14	DSSHT	<7	4.13E+04 (7.1%)

However, upon review of the gamma scan data, and a comparison to the estimated values based upon process knowledge, the SRR customer proposed that the DSSHT (MCU-21-4/5/6) and SEHT (MCU-21-7/8/9) values were in fact switched with each other. In order to clarify this, a second DSSHT sample was analyzed via gamma scan, this time from sample MCU-21-6. The result was 5.99E+06 pCi/mL with an analytical uncertainty (1- σ) of 5%. This sample was much closer to the previous SEHT sample result, and matched customer expectations. SEHT flush volumes were provided by SRR engineering:

“SEHT 1st batch flush (MCU-21-1/2/3) sample was pulled on 5/6/21 (and gamma scan) results show 2.13E8 pCi/mL, with an SEHT volume of ~ 417 gal. This batch was transferred on 5/20/21 leaving ~ 140 gallons left in SEHT (still at the original concentration). (SRR) then added DIW directly into the tank to a level of ~640 gallons on 5/28/21 and pulled the second SEHT sample the same day after adding the DIW water.”

This description of SEHT flush volumes correspond to a dilution of ~ 4.6x from the 5/6/21 sample data, a customer expectation of a SEHT result ~ 4.7E+07 pCi/mL from volume dilution estimate. From the above argument it was determined that at some point a sample swap had occurred. The value for reporting the DSSHT result was selected from the second analysis, sample MCU-21-6. Therefore, the corrected gamma scan values are as reported in Table 2.

Upon review of the gamma scan data, and comparison to the estimated values based upon process knowledge, the SRR customer noted the DSSHT gamma scan from MCU-21-11 was lower than expected. This is likely due to not fully recirculating the DSSHT contents prior to sampling. Following the DSSHT first batch transfer, a heel of approximately 256 gallons remained in the tank. As part of compiling the second DSSHT batch, the volume was increased to approximately 1783 gallons with DIW flush material and samples were pulled. The first DSSHT batch had a reported ¹³⁷Cs rate of 5.99E+06 pCi/mL. With a dilution factor of 6.96 (1783 gal/256 gal) from the 5/27/21 samples, the expected ¹³⁷Cs rate for the second batch would be approximately 8.61E+05 pCi/mL. Despite the discrepancy in the ¹³⁷Cs rate, both the estimated and measured ¹³⁷Cs rates are well below the Tank 50 Waste Acceptance Criteria limit.

A further comparison to the estimated values based upon process knowledge, the SRR customer noted the DSSHT gamma scan from MCU-21-14 is as expected. The ^{137}Cs rate for the second batch was expected to be approximately $8.61\text{E}+05$ pCi/mL though sample results showed $6.31\text{E}+03$ pCi/mL (reference SRNL-L3100-2021-00020). For the third DSSHT flush, with a dilution factor of 13.7 (3,801 gal/278 gal) from the 6/25/21 expected sample results ($8.61\text{E}+05$ pCi/mL), the expected ^{137}Cs rate for the third batch would be approximately $6.28\text{E}+04$ pCi/mL. Similarly, if using the reported 6/25/21 sample results ($6.31\text{E}+03$ pCi/mL), the expected ^{137}Cs rate for the third batch would be approximately $4.61\text{E}+02$ pCi/mL. From this comparison, the third DSSHT batch reported result in Table 1 more closely follows the dilution expected from the second DSSHT batch ^{137}Cs rates rather than the second batch reported sample results.

Table 2. Corrected Combined DSSHT and SEHT Sample Results

Sample ID	Type	Isopar-L (mg/L)	Cs-137 (pCi/mL)
MCU-21-1, 2, 3	SEHT	<7	$2.13\text{E}+08$ (5.0%)
MCU-21-4, 5, 6	DSSHT	<7	$5.99\text{E}+06$ (5.0%)
MCU-21-7, 8, 9	SEHT	<7	$5.63\text{E}+07$ (5.0%)
MCU-21-10, 11, 12	DSSHT	<7	$6.31\text{E}+03$ (8.2%)
MCU-13, 14	DSSHT	<7	$4.13\text{E}+04$ (7.1%)

The Isopar-L TM uncertainty for samples above detection limit is 20%.

4.0 Results and Discussion

Over the series of SEHT and DSSHT samples, the Isopar L TM was always noted to be at less than detectable levels. Furthermore, the ^{137}Cs levels for both sets of samples also declined overall.

5.0 Conclusions

The campaign to flush the SEHT and DSSHT systems has sufficiently de-inventoried the Isopar-L TM and enough ^{137}Cs to place MCU in a suspended operations state.

6.0 References

1. T. B. Peters, “Results from First MCU Flush SEHT Sample”, SRNL-L3100-2021-00013, May 2021.
2. T. B. Peters, “Results from First MCU Flush DSSHT Sample and the Second SEHT Flush Sample”, SRNL-L3100-2021-00019, June 2021.
3. T. B. Peters, “Results from Second MCU Flush DSSHT Sample”, SRNL-L3100-2021-00020, July 2021.
4. T. B. Peters, “Results from the Third MCU Flush DSSHT Sample”, SRNL-L3100-2021-00024, August 2021.

Distribution:

Kevin.Brotherton@srs.gov
alex.cozzi@srnl.doe.gov
Richard.Edwards@srs.gov
brent.gifford@srs.gov
connie.herman@srnl.doe.gov
Thomas.Huff@srs.gov
Vijay.Jain@srs.gov
john.mayer@srnl.doe.gov
daniel.mccabe@srnl.doe.gov
ryan.mcnew@srs.gov
gregg.morgan@srnl.doe.gov
frank.pennebaker@srnl.doe.gov
william.ramsey@SRNL.DOE.gov
michael.stone@srnl.doe.gov
boyd.wiedenman@srnl.doe.gov
jeffrey.crenshaw@srs.gov
kenneth.fernandez@srs.gov
thomas.peters@srnl.doe.gov
charles.nash@srnl.doe.gov
kathryn.taylor-pashow@srnl.doe.gov
daniel.jones@srnl.doe.gov
fernando.fondeur@srnl.doe.gov
Eric.Barrowclough@srs.gov
Celia.Aponte@SRS.gov
John.Occhipinti@SRS.gov
John.Iaukea@SRS.gov