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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).

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# **Characterization of Infrequent Samples from the Concentration, Storage, and Transfer Facility: H-Area Diversion Box 7 (HDB-7) Sump Sample: January 2022 Samples**

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March, 2022

SRNL-STI-2022-00106, Revision 0

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**Printed in the United States of America**

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

**Keywords:** *Tank Farm*

**Retention:** *Permanent*

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Savannah River National Laboratory is operated by  
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## **ACKNOWLEDGEMENTS**

The author acknowledges assistance in the completion of sample transfers and handling tasks by Shirley Mccollum, and Scott Mcdonald and the completion of sample analyses tasks by David DiPrete and Sonia Dyer.

## EXECUTIVE SUMMARY

Savannah River Remediation Engineering (SRR-E) requested that the Savannah River National Laboratory (SRNL) analyze the Concentration, Storage, and Transfer Facility (CSTF) samples from the following Tank Farm areas: the sump encasement, catch tank, drain cell, and waste tank annulus. In general, these CSTF samples will be analyzed on an infrequent basis and analyses will include detection for total beta/gamma, total alpha activities, density, free hydroxide, and pH measurements.

This report presents characterization results for the H-Area diversion box 7 sump (HDB-7) 07 January 2022 and 18 January 2022 samples. The samples were clear and colorless and free from any solids. The results are measurements for total gamma, total alpha, total beta, density, free hydroxide, and pH.

These analyses were performed in triplicate. A summary of the average analytical results for the two HDB-7 samples includes the following.

The directly measured pH for the HDB-7 January 07, 2022 “as-received” sample was 7.9, the calculated free hydroxide concentration was  $7.15\text{E-}07$  M, and the density was  $1.00$  g/mL. The total alpha activity for the HDB-7 January 07, 2022 sample is reported as a less than value (upper limits) either because of possible spectral interferences or because there is not much alpha activity in the sample. Thus, the total alpha activity averaged  $<2.15\text{E+}02$  dpm/mL. This value is less than  $4.83\text{E+}03$  dpm/mL, which is the procedural limit for non-waste determination.<sup>i</sup> The total beta activity in the HDB-7 January 07, 2022 sample is above the instrument detection limits and averaged  $3.29\text{E+}04$  dpm/mL. The average measured cesium-137 activity in the HDB-7 January 07, 2022 sample is  $2.57\text{E+}04$  dpm/mL. The corresponding Ba-137m activity, calculated as 94.6% of the Cs-137 values, is  $2.43\text{E+}04$  dpm/mL. The total empirical activity of the beta and gamma emitting activities equals  $5.72\text{E+}04$  dpm/mL. This value is less than  $8.69\text{E+}05$  dpm/mL, which is the procedural limit for non-waste determination.<sup>i</sup>

The directly measured pH for the HDB-7 January 18, 2022 “as-received” sample was 8.0, the calculated free hydroxide concentration was  $1.03\text{E-}06$  M, and the density was  $1.00$  g/mL. The total alpha activity for the HDB-7 January 18, 2022 sample is reported as a less than value (upper limits) either because of possible spectral interferences or because there is not much alpha activity in the sample. Thus, the total alpha activity averaged  $<1.98\text{E+}02$  dpm/mL. This value is less than  $4.83\text{E+}03$  dpm/mL, which is the procedural limit for non-waste determination. The total beta activity in the HDB-7 January 18, 2022 sample is above the instrument detection limits and averaged  $5.36\text{E+}04$  dpm/mL. The average measured cesium-137 activity in the HDB-7 January 18, 2022 sample is  $4.34\text{E+}04$  dpm/mL. The corresponding Ba-137m activity, calculated as 94.6% of the Cs-137 values, is  $4.11\text{E+}04$  dpm/mL. The total empirical activity of the beta and gamma emitting activities equals  $9.47\text{E+}04$  dpm/mL. This value is less than  $8.69\text{E+}05$  dpm/mL, which is the procedural limit for non-waste determination.

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## **LIST OF ABBREVIATIONS**

CSTF	Concentration, Storage and Transfer Facility
HDB	H-Area Diversion Box
LSC	Liquid Scintillation Counting
MDA	Minimum Detectable Activity
PMP	Polymethyl pentene
SRNL	Savannah River National Laboratory
SRR-E	Savannah River Remediation-Engineering
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request

## 1.0 Introduction

On occasion, Savannah River Remediation Engineering (SRR-E) will request Savannah River National Laboratory (SRNL) to perform analysis on Concentration, Storage, and Transfer Facility (CSTF) samples originating from the sump encasement, catch tank, drain cell, or waste tank annulus per the Technical Task Request (TTR) or email. In January 2022, SRR-E sent SRNL two separate samples identified as HDB-7 from a diversion box sump. Following the specified TTR<sup>ii</sup>, Task Technical and Quality Assurance Plan (TTQAP)<sup>iii</sup>, and updated request by SRR-E through email, SRNL tested the samples for Total Alpha and Total Beta by liquid scintillation counting (LSC), Cs-137 by Gamma scan, density, and pH.

## 2.0 Experimental Procedure

The first H-Area diversion box sump HDB-7 sample was received on 07 January 2022 at SRNL. As the “as-received” sample radiation dose rate was 5 mrem/hr extremity and 1 mrem/hr skin and whole body, the container was moved to a radiological hood for inspection. Approximately 150 mL of sample was collected from the receipt vessel and was transferred into clear polymethyl pentene (PMP) beakers for visual inspection.

The sample appearance was clear and colorless with no visual solids. Aliquots of the sample were directly transferred into shielded bottles and submitted in triplicate preparations each for total gamma/beta/alpha and pH analyses.

The density of the sample was measured in triplicate by recording the weight of 2 mL portions of sample on a balance at 18.2 °C. The sub-samples used for density determination were combined with the original sample and stored in a plastic bottle.

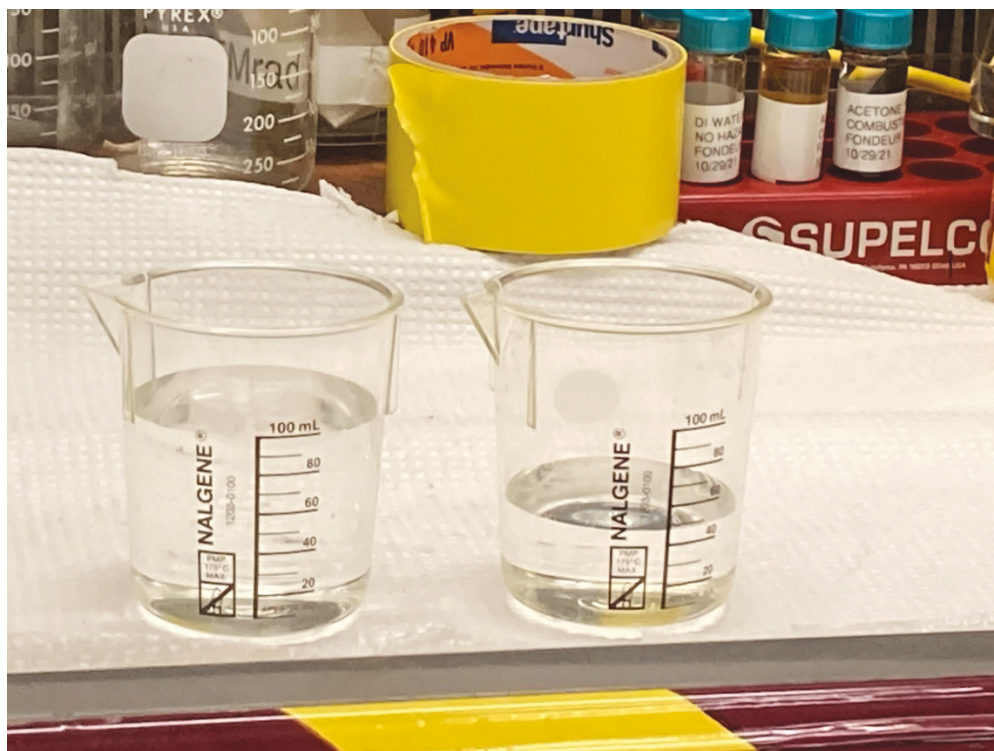
The same procedure was repeated for the second HDB-7 sample received on 18 January 2022 at SRNL. This sample had a volume of approximately 225 mL and was also clear and colorless with no visual solids. During density determination, the temperature was 19.1 °C.

### 2.1 Quality Assurance

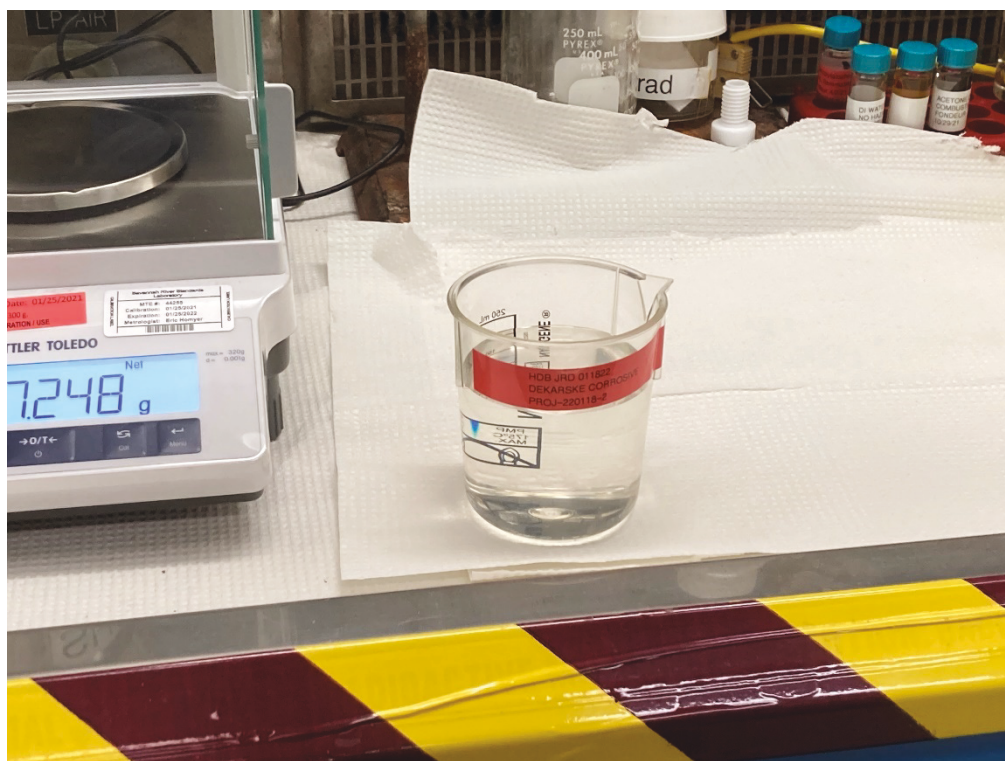
Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60<sup>iv</sup>. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2<sup>v</sup>. This review meets the acceptance criteria to comply with the TTR<sup>ii</sup> requesting this work with a functional classification of Safety Class and per guidance in the TTQAP.<sup>iii</sup> Data are recorded in the electronic laboratory notebook system as Experiment ID M0869-00537-01<sup>vi</sup> and M0869-00537-02<sup>vii</sup>.

## 3.0 Results and Discussion

Photographs of the HDB-7 samples are provided in Figure 3-1 and Figure 3-2. The HDB-7 07 January 2022 sample was split into two portions for inspection. Separate samples were submitted in triplicate for analysis by each of the following methods: 1) radiochemical analysis (total gamma, total beta, and Cs-removed beta and alpha) and 2) pH. Free hydroxide was then calculated from the pH. Results are provided in Table 3-1 and Table 3-2.



**Figure 3-1. Photograph of the HDB-7 Sample received on 07 January 2022 Split Between Two PMP Beakers**



**Figure 3-2. Photograph of the HDB-7 Sample received on 18 January 2022**

**Table 3-1. Results for January 2022 HDB-7 sample received on 07 January 2022: Total Alpha, Total Beta, Total Gamma scan, Density, pH, and Free Hydroxide.**

Test	Replicate 1	Replicate 2	Replicate 3	Average	%RSD
Total Alpha (dpm/mL) (one sigma % uncertainty)	*<2.15E+02 (MDA)	*<2.13E+02 (MDA)	*<2.13E+02 (MDA)	*<2.15E+02 (MDA)	N/A
Cs-Removed Alpha (dpm/mL) (one sigma % uncertainty)	*<2.63E+01 (MDA)	*<2.60E+01 (MDA)	*<2.55E+01 (MDA)	*<2.63E+01 (MDA)	N/A
Total Beta (dpm/mL) (one sigma % uncertainty)	3.27E+04 (10%)	3.29E+04 (10%)	3.32E+04 (10%)	3.29E+04	0.76
Cs-Removed Beta (dpm/mL) (one sigma % uncertainty)	3.13E+02 (19%)	2.65E+02 (21%)	3.16E+02 (19%)	2.98E+02	9.60
Cs-137 (dpm/mL) (one sigma % uncertainty)	2.56E+04 (5.00%)	2.54E+04 (5.00%)	2.60E+04 (5.00%)	2.57E+04	1.19
**Ba-137m (dpm/mL)	2.42E+04	2.40E+04	2.46E+04	2.43E+04	1.19
**Sum of Total Beta and Ba-137m (dpm/mL)	5.69E+04	5.69E+04	5.78E+04	5.72E+04	0.88
pH (one sigma % uncertainty)	7.86	7.88	7.82	***7.85	***7.11
Free Hydroxide (M)	7.24E-07	7.59E-07	6.61E-07	7.15E-07	6.95
Density (g/mL)	0.994	0.997	1.001	0.997	0.36

\*Note: Results were below detectable limit for quantification by method, therefore result is upper limit based on sensitivity of the analysis method. Thus, the average value reported is the highest "<" result. All one sigma % uncertainties are as reported with the analytical methods. \*\*Note: Ba-137m activity is calculated as 94.6% of the Cs-137 value. \*\*\*Note: pH average and %RSD calculated by conversion of pH replicates to [H<sup>+</sup>].

**Table 3-2. Results for January 2022 HDB-7 sample received on 18 January 2022: Total Alpha, Total Beta, Total Gamma scan, Density, pH, and Free Hydroxide.**

Test	Replicate 1	Replicate 2	Replicate 3	Average	%RSD
Total Alpha (dpm/mL) (one sigma % uncertainty)	*<1.90E+02 (MDA)	*<1.95E+02 (MDA)	*<1.98E+02 (MDA)	*<1.98E+02 (MDA)	N/A
Cs-Removed Alpha (dpm/mL) (one sigma % uncertainty)	*<2.33E+01 (MDA)	*<2.35E+01 (MDA)	*<2.31E+01 (MDA)	*<2.35E+01 (MDA)	N/A
Total Beta (dpm/mL) (one sigma % uncertainty)	5.41E+04 (10%)	5.34E+04 (10%)	5.33E+04 (10%)	5.36E+04	0.81
Cs-Removed Beta (dpm/mL) (one sigma % uncertainty)	2.77E+02 (20%)	2.90E+02 (20%)	3.05E+02 (19%)	2.91E+02	4.82
Cs-137 (dpm/mL) (one sigma % uncertainty)	4.26E+04 (5.00%)	4.40E+04 (5.00%)	4.37E+04 (5.00%)	4.34E+04	1.70
**Ba-137m (dpm/mL)	4.03E+04	4.16E+04	4.13E+04	4.11E+04	1.70
**Sum of Total Beta and Ba-137m (dpm/mL)	9.44E+04	9.50E+04	9.46E+04	9.47E+04	0.33
pH (one sigma % uncertainty)	8.07 (10%)	7.91 (10%)	8.04 (10%)	***8.00	***20.41
Free Hydroxide (M)	1.17E-06	8.13E-07	1.10E-06	1.03E-06	18.53
Density (g/mL)	0.997	1.000	0.998	0.998	0.13

\*Note: Results were below detectable limit for quantification by method, therefore result is upper limit based on sensitivity of the analysis method. Thus, the average value reported is the highest "<" result. All one sigma % uncertainties are as reported with the analytical methods. \*\*Note: Ba-137m activity is

calculated as 94.6% of the Cs-137 value. \*\*\*Note: pH average and %RSD calculated by conversion of pH replicates to  $[H^+]$ .

#### **4.0 Conclusions**

The characterizations of the HDB-7 samples indicated low to moderate gamma and beta activity in the samples and no measurable alpha activity. For the 07 January 2022 sample, the density was 1.00 g/mL, the pH was 7.9, and the free hydroxide was  $7.15E-07$  M, and in the 18 January 2022 sample, the density was 1.00 g/mL, the pH was 8.0, and the free hydroxide was  $1.03E-06$  M. Total alpha and total beta/gamma activity for both samples is less than the respective values of  $4.83E+03$  dpm/ml and  $8.69E+05$  dpm/mL for non-waste determination.

## 5.0 References

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- <sup>i</sup> “Tank Farm Transfer Control Program, Pump Tank Transfer Jet Control Program, and Waste Tank Chemical Cleaning Program”, WRSC-TR-2002-00403, Rev. 32, August 2021
- <sup>ii</sup> Technical Task Request, “Infrequent CSTF Samples”, X-TTR-H-0101, Rev. 1, June 2021.
- <sup>iii</sup> L. N. Oji, S. C. Lucatero, “Task Technical and Quality Assurance Plan for the Analysis of Infrequent Samples from the Concentration, Storage, and Transfer Facility”, SRNL-RP-2020-00565, Rev.1, July 2021.
- <sup>iv</sup> “Technical Reviews,” E7 Manual, Procedure 2.60, Rev. 18, 2019
- <sup>v</sup> “Savannah River National Laboratory Technical Report Design Check Guidelines”, WSRC-IM-2002-00011, Rev.2, 2004.
- <sup>vi</sup> J. R. Dekarske: ELN: M0869-00537-01 (Electronic Notebook (Production)); SRNL, Aiken, SC 29808 (2022).
- <sup>vii</sup> J. R. Dekarske: ELN: M0869-00537-02 (Electronic Notebook (Production)); SRNL, Aiken, SC 29808 (2022).

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