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Characterization Results for the April 2022 Tank Farm 2H Evaporator Overhead Sample

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June, 2022

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EXECUTIVE SUMMARY

On an annual basis, Savannah River Mission Completion (SRMC) provides 2H and 3H evaporator overhead samples to Savannah River National Lab (SRNL) to be analyzed per Section 5.2 of the Effluent Treatment Project (ETP) Waste Compliance Plan (WCP)ⁱ and the Waste Acceptance Criteria (WAC)ⁱⁱ.

This report presents characterization results for the April 2022 2H evaporator overhead sample. The sample was clear and colorless with no visible solids. The results provide measurements for cesium-137 (¹³⁷Cs), strontium-90 (⁹⁰Sr), and iodine-129 (¹²⁹I) with the radionuclide concentration limits specified by the WAC.

These analyses were performed in duplicate. A summary of the analytical results for the 2H evaporator overhead sample includes the following.

The measured cesium-137 activity in the 2H evaporator overhead sample is 4.18E+02 dpm/mL which is above the ETP WAC limit of 3.28E+02 dpm/mL.

The strontium-90 activity in the 2H evaporator overhead sample is <2.05E+00 dpm/mL. This is below the ETP WAC limit of 1.76E+02 dpm/mL.

The iodine-129 activity in the 2H evaporator overhead sample is <2.14E-02 dpm/mL. This is below the ETP WAC limit of 1.00E+00 dpm/mL.

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LIST OF ABBREVIATIONS

ETF Effluent Treatment Facility
ETP Effluent Treatment Project
HPGe High Purity Germanium

MDA Minimum Detectable Activity

ND Not Detected

SRMC Savannah River Mission Completion
SRNL Savannah River National Laboratory

TTQAP Task Technical and Quality Assurance Plan

TTR Technical Task Request
WAC Waste Acceptance Criteria
WCP Waste Compliance Plan

1.0 Introduction

To minimize and reduce the large amount of high-level liquid waste volume at the Savannah River Site (SRS), the 2H and 3H evaporators were constructed and began operations in H Area in 1982 and 2000, respectively. The evaporation process is performed through boiling the liquid waste in the evaporator cell, cooling and condensing the overhead vapors in the condenser cell, followed by collecting the condensate in the overheads cell. The low-level liquid waste is further treated at the Effluent Treatment Facility (ETF) prior to release into the environment.

On an annual basis, Savannah River Mission Completion (SRMC) provides 2H and 3H evaporator overhead samples to Savannah River National Laboratory (SRNL) for select radionuclide (137Cs, 90Sr, and 129I) characterizations to ensure that the Effluent Treatment Project (ETP) Waste Acceptance Criteria (WAC) for these radionuclides are met as specified in Section 5.2 of the ETP Waste Compliance Plan (WCP)i and the WACii. In this report, following the specified Technical Task Request (TTR)iii and Task Technical and Quality Assurance Plan (TTQAP)iv, the April 2022 2H evaporator overhead sample was analyzed for cesium-137 (137Cs), strontium-90 (90Sr), and iodine-129 (129I) with the radionuclide concentration limits specified by the WAC.

2.0 Experimental Procedure

Two 250 mL containers holding the 2H evaporator overhead sample were received on April 20, 2022 at SRNL. Since the "as-received" sample radiation dose rate was low [extremity, skin, and whole body were below instrument detection limit (ND)], the containers were moved to a radiological hood for inspection. Approximately 250 mL of sample (total of 500 mL of sample) was collected from each receipt vessel and transferred into a clear plastic beaker for visual inspection. Two distilled/deionized water blank samples were prepared in parallel at SRNL with the 2H evaporator overhead sample inspection to evaluate against sample contamination during inspection.

Two 2H evaporator overhead sample replicates and two distilled/deionized water blank samples were submitted for: 1) gamma spectroscopy (¹³⁷Cs) and 2) chemical separations followed by beta counting (¹²⁹I and ⁹⁰Sr).

Cs-137 concentrations were determined by gamma spectrometry. A 50 milliliter aliquot of sample was analyzed directly for analysis periods of at least 4 hours. The sample was analyzed by shielded, high purity germanium (HPGe) gamma spectrometers. I-129 and Sr-90 were determined by radiochemistry methods. These analytical methods involved separation techniques that enabled radionuclides at low concentrations to be measured more accurately and with lower detection limits. The techniques and methodology for these separations will be summarized here.

Sr-90 Method: A 20 milliliter aliquot of each sample was spiked with a stable Sr carrier, and a stable Ce carrier. The Sr carrier was used for separation yielding purposes and the Ce carrier was used to enhance the separation rates of undesirable isotopes such as Y-90, the lanthanides or the actinides. The spiked sample aliquot was acidified with nitric acid, evaporated to dryness and re-dissolved in 8N nitric acid. The Sr in the sample was then extracted using a commercially available Sr extraction resin. This resin also extracts some of the Pu under the conditions used to extract the Sr. The Pu was washed from the resin using an oxalic acid/nitric acid mixture. The Sr was eluted from the resin, and the resulting solution concentrated. A portion of the purified Sr solution was neutron activated in a Cf-252 neutron activation facility at SRNL to determine the total Sr and in order to calculate the fraction of Sr isolated by the procedure. A second portion of each of the Sr fractions was stored for five to seven days to allow Y-90 to grow in. Each fraction was then counted by liquid scintillation analysis using a Low-Level Perkin Elmer Tri-Carb Liquid scintillation counter to determine the Y-90 activity in a high energy beta window free of interferences from Sr-90 or any residual beta interferences from isotopes such as Cs-137. The Sr-90 beta activity in each case was

calculated from the Y-90 activity. The yields of the stable Sr carriers were applied to the Sr-90 beta activity results to determine Sr-90 activities in the original aliquots of the solutions.

I-129 Method: A 50 milliliter aliquot of sample was spiked with a known amount of stable KI to act as an iodine tracer/carrier. The sample was acidified with nitric acid. The sample was decontaminated with a resin treatment to enhance removal of the actinide elements. The iodine in the sample was then reduced to iodide. The solution was then treated with AgNO₃ in order to precipitate the iodide ion as AgI. The precipitate was analyzed by low energy photon spectrometry to determine the amount of I-129 present. I-129 is detected by its characteristic gamma and x-ray emissions. The precipitate was then neutron activated in a Cf-252 neutron source at SRNL to determine the total amount of iodine present in order to calculate the recovery of I-129 in the radiochemical separation.

3.0 Results and Discussion

A photograph of the "as-received" 2H evaporator overhead samples in two 250 mL capacity plastic containers is provided in Figure 3-1. The samples appearance were clear and colorless with no visible solids.

The analytical results for the characterization of the 2H evaporator overhead and the water blanks are provided in Table 3-1. The ¹³⁷Cs activity in the 2H evaporator overhead is above the ETP WAC limit of 3.28E+02 dpm/mL. This analytical result for ¹³⁷Cs is unlike the previous (2020 sample) 2H evaporator overhead sample analytical result for ¹³⁷Cs, which was not above the WAC limit. Historical results for 2H evaporator overhead samples are provided in Table 3-2, and a survey of the historical results trends demonstrates that ¹³⁷Cs activity is generally close to the ETP WAC limit.



Figure 3-1. Photograph of the 2H Evaporator Overhead Samples in Plastic Beakers

Table 3-1. Average of Duplicate Results for 2022 2H Evaporator Overhead and Blank samples: 137 Cs, 129 I, and 90 Sr (April 2022).

Analyte	Activity (dpm/mL) (2H Evaporator Overhead)	Activity (pCi/mL) (2H Evaporator Overhead)	Blank Sample (dpm/mL)	ETP WAC limits (dpm/mL) ⁱⁱ
¹³⁷ Cs	$4.18E+02 (\%RSD: 0.34\%)^{\alpha}$	1.88E+02	$<1.66E-01 (MDA)^{\beta}$	3.28E+02
⁹⁰ Sr	$<2.05E+00 (MDA)^{\beta}$	<9.23E-01 ^β	$<1.91E+00 (MDA)^{\beta}$	1.76E+02
¹²⁹ I	$<2.14E-02 (MDA)^{\beta}$	$<9.64E-03^{\beta}$	$<2.43E-02 (MDA)^{\beta}$	1.00E+00

^α One sigma % uncertainty of 5.00% for analytical method.

 $^{^{\}beta}$ Minimum result is reported.

Table 3-2. Historical Analytical Results for 2H Evaporator Overhead Samples: ¹³⁷Cs, ¹²⁹I, and ⁹⁰Sr.

Analyte	¹³⁷ Cs, dpm/mL	⁹⁰ Sr, dpm/mL	¹²⁹ I, dpm/mL
ETP WAC limits ⁱⁱ	3.28E+02	1.76E+02	1.00E+00
April 2022 2H Evaporator Overhead Sample	4.18E+02	<2.05E+00	<2.14E-02
2020 2H Evaporator Overhead Sample ^v	1.58E+02	<9.49E+00	<2.32E-01
2018 2H Evaporator Overhead Sample ^{vi}	9.13E+01	<4.99E+00	<2.74E-02
2017 2H Evaporator Overhead Sample ^{vii}	6.97E+01	<5.35E+01	<6.66E-01
2016 2H Evaporator Overhead Sample viii	7.04E+01	<1.00E+01	5.83E-02
2014 2H Evaporator Overhead Sample ix	5.80E+01	<8.17E+00	<8.02E-02

4.0 Conclusions

The April 2022 2H evaporator overhead sample characterization result for ¹³⁷Cs activity (4.18E+02 dpm/mL) was above the ETP WAC limit of 3.28E+02 dpm/mL. The ¹²⁹I activity (<2.14E-02 dpm/mL) and ⁹⁰Sr activity (<2.05E+00 dpm/mL) in the sample were below the ETP WAC limits of 1.00E+00 dpm/mL and 1.76E+02 dpm/mL, respectively.

5.0 Quality Assurance

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°. This review, a design verification done by document review, meets the acceptance criteria to comply with the TTRⁱⁱⁱ requesting this work with a functional classification of Safety Class and per guidance in the TTQAP^{iv}. Data are recorded in the electronic laboratory notebook system as Experiment ID M0869-00537-08. **ii

6.0 References

ⁱ S. Campbell, "F/H Tank Farms Waste Compliance Plan for Transfers to the Effluent Treatment Project", X-WCP-H-00013, Rev. 8, June 2016.

ii "F/H Effluent Treatment Facility Waste Acceptance Criteria", X-SD-H-00009, Rev. 7, December 2020.

- vii T.T. Truong, J.C. Nicholson, "Characterization Results for the January 2017 H-Tank Farm 2H Evaporator Overhead Sample", SRNL-STI-2017-00166, Rev. 0, 2017.
- viii J.C. Nicholson, "Characterization Results for the March 2016 H-Tank Farm 2H Evaporator Overhead Samples", SRNL-STI-2016-00253, Rev. 1, 2016.
- ^{ix} A.L. Washington, II, "Characterization Results for the 2014 HTF 3H & 2H Evaporator Overhead Samples", SRNL-STI-2015-00198, Rev. 0, 2015.
- x "Technical Reviews," E7 Manual, Procedure 2.60, Rev. 18, 2019.
- xi "Savannah River National Laboratory Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Rev.2, 2004.
- xii J.R. Dekarske: ELN: M0869-00537-08 (Electronic Notebook (Production)); SRNL, Aiken, SC 29808 (2022).

iii Technical Task Request, "2020 Annual Overheads Sample Analysis for 2H/3H Evaporators", X-TTR-H-00115, Rev. 0, December 2020.

iv S.C. Lucatero, "Task Technical and Quality Assurance Plan for the Annual Overheads Sample Analysis for 2H/3H Evaporators", SRNL-RP-2021-00045, Rev. 0, February 2021.

^v S.C. Lucatero, "Characterization Results for the 2020 Tank Farm 2H and 3H Evaporator Overhead Samples", SRNL-STI-2021-00076, Rev. 0, 2021.

vi A.L. Washington, II, "Characterization Results for the July 2018 H-Tank Farm 2H Evaporator Overhead Samples", SRNL-STI-2019-00069, Rev. 1, 2019.

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