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Results of the Solvent Hold Tank Disposal Sample

T. B. Peters

F. F. Fondeur

JUNE 2020

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EXECUTIVE SUMMARY

Multiple samples from the Solvent Hold Tank (SHT) at the Modular Caustic-Side Solvent Extraction Unit (MCU) were delivered to SRNL and composited for comprehensive analysis in support of a disposal pathway for this organic liquid. A suite of radiochemical and non-radiochemical analyses show that the solvent composition is as expected with respect to the solvent components. The radiochemical analyses confirm the assumption that very few radioactive species are associated with the solvent (other than cesium) and those that have measurable values are typically a few tens of pCi/mL in activity concentration. The solvent has a total activity of $\leq 1.99\text{E-}08$ Ci/g, or $\leq 6.27\text{E-}05$ Ci/gal. The cesium in the solvent has an activity of $\leq 1.76\text{E-}08$ Ci/g, or $\leq 5.55\text{E-}05$ Ci/gal.

For the non-radiochemical analyses, the results are as anticipated. The solvent shows no or minimal propensity to take up Resource Conservation Recovery Act (RCRA) metals, except for mercury, likely due to uptake of methyl-mercury compounds known to be in the feed stream.

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LIST OF ABBREVIATIONS

AD	Analytical Development
DMA	Direct Mercury Analysis
EAV	E-Area Vault
FTIR	Fourier Transform InfraRed
HPLC	High-Performance Liquid Chromatography
IC-A	Ion Chromatography – Anions
ICPES	Inductively-Coupled Plasma Emission Spectroscopy
ICPMS	Inductively-Coupled Plasma Mass Spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
NMR	Nuclear Magnetic Resonance
RCRA	Resource Conservation Recovery Act
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SWPF	Salt Waste Processing Facility
TGA	Thermogravimetric Analysis
TiDG	tris(isodecyl)Guanidine
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance Plan
WAC	Waste Acceptance Criteria

1.0 Introduction

As part of the MCU facility decommissioning, the piping and various hold tanks must be flushed and sampled. Savannah River Remediation (SRR) developed a plan for the layup of the MCU facility.ⁱ The SHT contains approximately 200 gallons of the solvent used for cesium removal. Samples were provided to characterize the Next Generation Solvent Blend from the MCU SHT, a requirement of the site-wide procedure manual 1S, Chapter 3, “Waste Characterization.”. The methods listed in references [1] and [5] support the characterizations required by the Waste Management Program which include: E-Area Vault (EAV) Suite radionuclides (e.g., Cs-135), Gamma scan (e.g., Cs-137/Ba-137m), Total Beta, Total Alpha, Resource Conservation and Recovery Act Metals (e.g., Hg), next generation solvent components (e.g., Isopar-L), and select properties of the solvent (e.g., density).

1.1 Quality Assurance

This work was performed under a Technical Task Request (TTR).ⁱⁱ The recorded data, analysis, and conclusions satisfy the Safety Significant requirements in the Task Technical and Quality Assurance Plan (TTQAP) associated with this TTR.ⁱⁱⁱ

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.^{iv}

2.0 Analysis

A set of 17 p-nut vials from the SHT was delivered on January 21, 2020. A set of 11 p-nut vials from the SHT was delivered on January 23, 2020. The samples (MCU-20-1 through -28) were combined into a single composite before analysis. No dilution or alteration of the samples occurred outside of analysis. For some analyses sample digestion was required. In those cases, the dilution from digestion has been applied to the result.

2.1 Radiochemical Analyses

An extensive set of radiochemical analyses were required for this sample. The results are reported in Table 1. Values in parentheses are the analytical uncertainty.

The Ci/g and Ci/gal terms are calculated values from the sums of all unique isotope measurements. For these calculations, the less-than values are treated as detected concentrations.

Table 1. Radiochemical Analyses

Analyte	Result (pCi/mL)	Analyte	Result (pCi/mL)
²⁴¹ Am	<6.04E-01	⁹⁹ Tc	4.28E+00 (15%)
^{242m} Am	<5.41E-01	³ H	<1.18E+02
²⁴³ Am	<3.51E-01	²² Na	< 1.08E+00
²⁴² Cm	<4.49E-01	⁴⁰ K	< 2.72E+01
²⁴³ Cm	<1.05E+00	⁵⁴ Mn	< 1.62E+00
²⁴⁴ Cm	<2.40E-01	⁵⁷ Co	< 1.23E+00
²⁴⁵ Cm	<8.65E-01	⁵⁸ Co	< 1.56E+00
²⁴⁷ Cm	<1.02E+00	⁶⁵ Zn	< 3.62E+00
²⁴⁹ Cf	<1.06E+00	⁸⁸ Y	< 1.59E+00
²⁵¹ Cf	<9.78E-01	⁹⁵ Zr	< 2.74E+00
¹⁴ C	<1.14E+01	¹⁰³ Ru	< 1.53E+00
¹³⁴ Cs	<7.57E+00	¹¹³ Sn	< 2.06E+00
¹³⁵ Cs	<6.80E-02	¹²⁴ Sb	< 1.52E+00
¹³⁷ Cs	1.46E+04 (5.0%)	¹³³ Ba	< 2.18E+00
⁶⁰ Co	<1.70E+00	¹⁴¹ Ce	< 2.20E+00
¹⁰⁶ Ru	<9.78E+00	¹⁴⁴ Pm	< 1.46E+00
¹²⁵ Sb	<4.64E+00	¹⁴⁶ Pm	< 2.10E+00
¹²⁶ Sb	<1.58E+00	¹⁵² Eu	< 3.71E+00
¹²⁶ Sn	<3.98E+00	²⁰⁸ Tl	< 1.12E+00
¹⁴⁴ Ce	<9.55E+00	²¹² Bi	< 2.39E+01
¹⁵⁴ Eu	<2.59E+00	²¹² Pb	< 2.37E+00
¹⁵⁵ Eu	<4.55E+00	²¹⁴ Bi	< 3.21E+00
²²⁶ Ra	<2.94E+01	²²⁸ Ac	< 5.72E+00
²³⁷ Np	<1.21E+01	²³³ Pa	< 3.66E+00
¹²⁹ I	1.45E+01 (20%)	²³⁹ Np	< 2.65E+00
Total alpha (Cs-R) [∞]	9.06E+00 (19%)	⁷⁹ Se	<3.84E+00
Total Beta	1.79E+04 (10%)	²³² U	<1.40E-01
⁹⁴ Nb	<3.03E-01	²³³ U	<6.50E+02
⁵⁹ Ni	<1.82E+02	²³⁴ U	<4.20E+02
⁶³ Ni	<7.30E+00	²³⁵ U	<1.45E-01
¹⁴⁷ Pm	<1.02E+01	²³⁶ U	<4.34E+00
¹⁵¹ Sm	<1.27E+01	²³⁸ U	2.45E-02 (20%)
²³⁸ Pu	8.29E+00 (12%)		
^{239/40} Pu	1.84E-01 (104%)	Ci/g total	≤1.99E-08 Ci/g
²⁴¹ Pu	5.41E+00 (32%)	Ci/gal	≤6.27E-05 Ci/gal
²⁴² Pu	<2.57E+02		
⁹⁰ Sr	<6.40E+00		

[∞] In order to avoid historically noted interference and high biases with this alpha measurement, the cesium was removed beforehand.

2.2 Non-Radiochemical Analyses

A variety of non-radiochemical analyses were also required: Ion Chromatography-Anions (IC-A) [fluoride, chloride, bromide, iodide], Inductively Coupled Plasma Emission Spectroscopy (ICPES) [Ag, Ba, Cd, Cr], Inductively Coupled Plasma Mass Spectroscopy (ICPMS) [As, Pb, Se], Thermo Gravimetric Analysis (TGA) [%ash], Direct Mercury Analysis (DMA) [total Hg], Nuclear Magnetic Resonance (NMR) [%water, Isopar-L, Modifier, MAXCalix] and High-Performance Liquid Chromatography (HPLC) [Modifier, BOBCalix, MAXCalix]. The heat of combustion is a calculated value. The results are reported in Table 2. Some of these results have been previously reported in a monthly solvent quality sample.^v For some analyses sample digestion was required. In those cases, the dilution from digestion has been applied to the result. Values in parentheses are the analytical uncertainty.

Table 2. Non-Radiochemical Analyses

Analyte	Result (mg/L)	Analyte	Result
Total Hg	24.3 (20%)	Se	<6.73E-02 mg/L
F	<10	%Water	0.01 moles/L (2.5%)
Cl	<10	% Ash	2.11 wt % remaining (0.4%)
Br	<10	Heat of Combustion	3.46E+04 kJ/L
I	<10	Density	0.834 g/mL (3%)
Ag	<1.17	Isopar-L	6.10E+05 mg/L (8%)
As	<6.73E-02	Modifier	1.73E+05 mg/L (6%)
Ba	5.94 (10%)	TiDG	7.90E+02 mg/L (10%)
Cd	<0.834	MAXCalix	4.80E+04 mg/L (10%)
Cr	<1.92	BOBCalixC6	1200 mg/L (10%)
Pb	0.348 (20%)	TOA	210 mg/L (16%)

3.0 Conclusions

A sample from the SHT was delivered to SRNL for comprehensive analysis in support of a disposal pathway for this organic liquid. A suite of radiochemical and non-radiochemical analyses show that the solvent composition is as expected with respect to the solvent components. The radiochemical analyses confirm the assumption that other than cesium, very few radioactive species are associated with the solvent and those that have measurable values are typically a few tens of pCi/mL in activity concentration. The solvent has an activity of $\leq 1.99\text{E-}08$ Ci/g, or $\leq 6.27\text{E-}05$ Ci/gal. The cesium in the solvent has an activity of $\leq 1.76\text{E-}08$ Ci/g, or $\leq 5.55\text{E-}05$ Ci/gal.

For the non-radiochemical analyses, the results are as anticipated. The solvent shows no or minimal propensity to take up RCRA metals (is not RCRA-characteristic), except for mercury, likely due to uptake of methyl-mercury compounds known to be in the feed stream.

4.0 References

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- ⁱ Jorge Medina, “Sample Plan for the Second Phase of the Strategy for the Interim Lay-Up of MCU During Initial SWPF Operation”, X-ESR-H-01003, Rev.0, November 2019
- ⁱⁱ Q. L. Nguyen, TTR “Technical Task Request”, X-TTR-H-00026, Rev. 0, July 2013
- ⁱⁱⁱ T. B. Peters, A. L. Washington, II, F. F. Fondeur, “Task Technical and Quality Assurance Plan for Routine Samples in Support of ARP and MCU”, SRNL-RP-2013-00536, Rev.2, January 2019
- ^{iv} Savannah River National Laboratory, “Technical Report Design Check Guidelines”, WSRC-IM-2002-00011, Rev. 2, August 2004
- ^v F. F. Fondeur, “Solvent Hold Tank Results for MCU-20-1-28: January 2020 Monthly Sample”, SRNL-L3160-2020-00002, Rev.0, March 2020.