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Results from the Interim Salt Disposition Program Macrobatch 11 Tank 21H Acceptance Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of verification of Macrobatch (Salt Batch) 11 for the Interim Salt Disposition Program (ISDP) for processing. This document reports characterization data on the samples of Tank 21H and fulfills the requirements of Deliverable 3 of the Technical Task Request (TTR). Extensive analysis of the samples show the following general characteristics:

- The density and color are typical of salt solution samples from Tank 21H.
- The bulk chemical composition (e.g., hydroxide, sodium, aluminum, nitrate, nitrite) is roughly similar to previous salt batch samples, with typical variations of <20%.
- The radionuclide concentrations are similar to previous salt batch samples.
- The plutonium and ^{90}Sr results indicate that a monosodium titanate (MST) strike will not be needed for ISDP processing.
- The Eurofins Hg sample analysis results are comparable to the previous salt batch. This report specifically notes that the concentration of methylmercury (MeHg) in SB11 is approximately double that found in SB10. However, it is not anticipated that the increase in MeHg between SB11 and SB10 would result in exceeding the current WAC limits, although the margin for SB11 grout may be decreased.

Further work will report the results of the Extraction-Scrub-Strip (ESS) testing (Task 5 of the TTR) using the Tank 21H material. Task 4 of the TTR (MST Strike) will not be completed for ISDP processing of Salt Batch 11. This report is revised to reflect changes in the reported methylmercury data per Eurofins Incident Report.[i]

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LIST OF ABBREVIATIONS

AA	Atomic Absorption
AD	Analytical Development
CV-Hg	Cold Vapor Mercury
DL	Detection Limits
ESS	Extraction-Scrub-Strip
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectroscopy
ICPMS	Inductively Coupled Plasma Mass Spectroscopy
ISDP	Interim Salt Disposition Program
MST	Monosodium Titanate
PuTTA	Plutonium Thenoyltrifluoroacetone
RL	Reporting Limits
%RSD	Percent Relative Standard Deviation
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SVOA	Semi Volatile Organic Analysis
TBP	Tributylphosphate
TIC/TOC	Total Inorganic Carbon-Total Organic Carbon
TPB	Tetraphenylborate
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VOA	Volatile Organic Analysis
WAC	Waste Acceptance Criteria

1.0 Introduction

This report provides the Tank 21H characterization sample results for Interim Salt Disposition Program (ISDP) Macrobatch (Salt Batch) 11. A previous document covered initial characterization which included results for a number of radiological and non-radiological analytes[ii], these analyses are also included in this report for completeness. This work was specified in a TTR [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

Two 200 mL Tank 21H samples (HTF-21-17-70 and -71) and a single 3L Tank 21H sample (HTF-21-17-72) were pulled and delivered to SRNL on July 31, 2017. The two 200 mL samples were pulled 1" from the surface and the 3L sample was a variable depth sample obtained approximately 62" from the bottom of the tank (transfer pump suction). Note that a 3L sample was obtained instead of the typical 1L due to a temporary shortage in 1L sample vials. Tank 21H was mixed at full speed for approximately 8.5 hours with two pumps before the samples were pulled; the samples were pulled approximately 27 days after pump shutdown. All the samples had the same visual appearance, clear solutions with no apparent solids.

The density of the filtered solution (using a 0.45 μm syringe filter) from each sample was measured twice and reported in Table 1. With Savannah River Remediation (SRR) concurrence, the contents of the three sample bottles were then combined (without filtering) and mixed. After compositing and allowing the contents of the composite bottle to sit for 10 days, it was found that a very fine layer of fine off-white solids had settled to the bottom of the composite bottle. Duplicate filtered samples (0.45 μm syringe filter) and one unfiltered sample, reported as Hg (unfiltered) in Table 4, were sent to Analytical Development (AD) for analysis. In the case of the unfiltered sample, a well-mixed sample from the composite bottle was removed for analysis with no filtration. None of the samples were diluted before delivery to AD.

Table 1. Sample Density Measurements (27.0ºC)

Sample	Measured Density (g/mL)
HTF-21-17-70	1.263 (1.37%)
HTF-21-17-71	1.271 (0.53%)
HTF-21-17-72	1.274 (0.53%)
Average (%Relative Standard Deviation)	1.269 (0.81%)

The analytical uncertainty is typically <1% (1-σ) for density measurements.

Material from the composite sample was filtered using 0.45 μm syringe filters, and the resulting filtrate was sent to AD for a variety of duplicate analyses. The one exception is the unfiltered Hg

result in Table 4. In this case, a well-mixed sample from the composite sample was removed for analysis with no filtration. None of the samples were diluted before delivery to AD.

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. Results from this report are not RW-0333P (enhanced quality assurance requirements) as per the TTR.

3.0 Results and Discussion

The tank samples were analyzed by AD using the listed non-radiological methods (Table 2) and radiological methods (Table 3). Analyses were performed in duplicate. Averages of the individual results, with %RSD in parentheses, are reported in Tables 4 and 5.

In a previous document,[I] density (at 27.0 ˚C), Inductively Coupled Plasma Emission Spectroscopy (ICPES), Ion Chromatography (IC), Total Inorganic Carbon/Total Organic Carbon (TIC/TOC), Hg (filtered and unfiltered), and Free Hydroxide were reported for the Tank 21H composite sample. These results (other than density which is reported in Table 2) are also reported here for completeness and are reported with other non-radiological results in Table 4. In that same document, ^{137}Cs, ^{238}Pu, $^{239/40}Pu$, ^{241}Pu, ^{90}Sr, and total alpha radiochemical results were reported for the Tank 21H composite sample. For completeness, those are included with the other radiochemical results in Table 5.

Table 2. Non-Radiological Analyses

Method	Analyte
IC Cations	NH_4^+
IC Anions	fluoride, chloride, formate, nitrite, nitrate, sulfate, phosphate, oxalate
ICPES	Ag, Al, Ba, Cd, Cr, Cu, K, Mn, Na, P, Pb, Si, Ti
TIC	total inorganic carbon (carbonate)
TOC	total organic carbon
Atomic Absorption (AA)-As	As
AA-Se	Se
Cold Vapor (CV)-Hg	Hg (total)[◊]
High Performance Liquid Chromatography (HPLC)	Tetraphenylborate (TPB)
Semi Volatile Organic Analysis (SVOA)	Tributylphosphate (TBP), phenol, NORPAR 13
Volatile Organic Analysis (VOA)	butanol, propanol
pH	pH
Titration	Free Hydroxide
Weight % Solids	Total Insoluble Solids

[◊] Elemental mercury, methylmercury and ethylmercury results will be provided by Eurofins and reported with the other results.

Table 3. Radiological Analyses

Method	Analyte
Tritium	^{3}H
^{14}C	^{14}C
Gamma Scan, Cs-removed⊕	^{60}Co, ^{106}Ru, ^{125}Sb, ^{126}Sn, ^{144}Ce, ^{154}Eu, ^{155}Eu
Individual radio count method for each isotope: ^{90}Sr, ^{94}Nb, ^{129}I, ^{99}Tc , ^{135}Cs, ^{226}Ra	^{90}Sr, ^{94}Nb, ^{129}I, ^{99}Tc , ^{135}Cs, ^{226}Ra
Gamma Scan	^{134}Cs, ^{137}Cs (from ^{137m}Ba)
^{232}U	^{232}U
PuTTA (Plutonium thenoyl-trifluoroacetone scintillation)	^{238}Pu, $^{239/40}Pu$
Am/Cm	^{241}Am, ^{243}Am, ^{244}Cm, ^{245}Cm
$^{59/63}Ni$	$^{59/63}Ni$
$^{147}Pm/^{151}Sm$	$^{147}Pm/^{151}Sm$
Inductively Coupled Plasma Mass Spectroscopy (ICPMS)	isotopes from mass number 81 - 209 and 230 - 252, including ^{233}U and above, ^{237}Np, ^{242}Pu, ^{244}Pu
Liquid Scintillation Counting	total alpha, total beta, ^{241}Pu

3.1 Tank 21H Characterization Results (non-radiological analytes)

Non-radiological results are listed in Table 4, except for the Eurofins Hg results. Results are in mg/L unless otherwise noted. The analytical uncertainties for all results are 10% (1-σ) except as noted. The analytical uncertainty for the pH measurement is typically 0.5 pH units. The analytical uncertainties for the As, Se, and Hg results are 20% (1-σ). Values in parentheses are %RSD. The

⊕ For these isotopes, the cesium must be removed in order to resolve these species.

values shaded in green are calculated results. Results in italic indicate a single real value as the other result was a less than the quantification limit.

Table 4. Non-Radiological Results of Tank 21H Analyses for Macrobatch 11[ʃ] (%RSD)

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<3.05	U	<35.1
Al	7020 (0.00%)	V	<1.40
B	57.7 (4.4%)	Zn	13.7 (6.7%)
Ba	<0.232	Zr	<1.41
Be	<0.097	F^-	<10
Ca	<2.47	Cl^-	473 (0.60%)
Cd	<2.99	Br^-	<10
Ce	<8.01	Formate	317 (0.45%)
Cr	60.9 (1.4%)	Nitrite	38500 (0.37%)
Cu	<10.9	Nitrate	102000 (0.70%)
Fe	<4.08	Phosphate	382 (1.1%)
Gd	<2.30	Sulfate	4720 (3.9%)
K	399 (3.0%)	Oxalate	413 (2.9%)
La	<1.80	TIC	3900 (0.73%)
Li	14.6 (4.8%)	TOC	296 (1.2%)
Mg	<0.434	pH	14
Mn	<0.420	Ammonium	<10
Mo	<17.2	NORPAR 13	<0.75
Na	149000 (6.2%)	Phenol	<10
Ni	<5.12	TPB	<5
P	*151*	TBP	<1
Pb	<41.5	Propanol	<0.25
S	<2600	Butanol	<0.25
Sb	<43.6	Methanol	263
Si	*21.6*	As	0.102 (7.7%)
Sn	<26.2	Hg (unfiltered)	1.19E+02 (1.8%)
Sr	<0.084	Hg (filtered)	9.42E+01 (4.7%)
Th	<2.37	Se	0.246 (1.2%)
Ti	<9.29	Free Hydroxide	2.79 (2.5%) M
		Wt% Solids	0.440 (68%)

[ʃ] Several analytes listed in Table 4 were not requested in the TTR, but are included for completeness.

The TIC and TOC results are in terms of mg of carbon/L. If we assume that the entire TIC result is carbonate, this translates to a carbonate concentration of 0.325 M. TIC results are reported in μg C/mL (equivalent to mg/L), so the TIC result is divided by 12,000 mg/mole to get the carbonate molarity.

The bulk chemical characteristics (e.g., hydroxide, aluminum, sodium, nitrate, nitrite, etc.) of this batch are roughly similar to that of Salt Batch 10, with typically <20% differences between the batches for the major components.

SRNL also notes the total Hg values are about the same as Salt Batch 10.[vi] The sample filtration had little effect on the mercury concentration for SRNL's determination given that the results are statistically the same.

Per Table 4, the oxalate concentration is 413 mg/L, and the formate concentration is 317 mg/L. If the oxalate result is converted to the equivalent carbon value, the result is 112 mg C/L. If the formate result is converted to the equivalent carbon value, the result is 85.0 mg C/L. Subtracting these results from the TOC result gives a remainder of 99 mg C/L. If it is assumed that all the remaining carbon is in the form of methanol, the calculated methanol concentration is 263 mg/L. This value is likely grossly conservative. No direct analytical method for methanol is available.

3.2 Tank 21H Characterization Results (radiological analytes)

The results of the radiological analysis in pCi/mL are listed in Table 5. The analytical uncertainty for ICPMS measurements is ±20% (1-σ). Other analytical methods have varying uncertainties, typically 5-15% (1-σ). Values in parentheses are the %RSD. The values shaded in green are calculated results. Results in italic indicate a single result, the other result being below the quantification limit. In the case of a single result, the value in parentheses is the one-sigma analytical (instrument) uncertainty.

Table 5. Radiological Results of Tank 21H Analyses for Macrobatch 11 (%RSD)

Analyte	Result (pCi/mL)	Analyte	Result (pCi/mL)
^{3}H	*1.31E+03 (15%)*	^{155}Eu	<4.77E+01
^{14}C	5.47E+02 (1.8%)	^{226}Ra	<9.45E+00
^{59}Ni	<5.31E+01	^{232}U	2.39E+00 (74%)
^{63}Ni	<5.13E+02	^{233}U	2.53E+01 (1.8%)
^{60}Co	<3.77E+00	^{234}U	1.13E+02 (0.3%)
^{90}Sr	2.61E+05 (11%)	^{235}U	4.97E-01 (0.6%)
^{90}Y	2.61E+05 (11%)	^{236}U	2.38E+00 (0.1%)
^{94}Nb	<3.50E+01	^{238}U	4.86E+00 (0.00%)
^{99}Tc	4.17E+04 (0.2%)	^{237}Np	3.65E+00 (0.4%)
^{106}Ru	<7.56E+01	^{238}Pu	2.98E+04 (2.4%)
^{106}Rh	<7.56E+01	^{239}Pu	8.55E+02 (1.5%)
^{125}Sb	8.96E+01 (6.4%)	^{240}Pu	<1.14E+03
^{125m}Te	8.96E+01 (6.4%)	$^{239/40}Pu$	1.07E+03 (5.0%)
^{126}Sn	3.78E+02 (0.2%)	^{241}Pu	9.43E+03 (3.0%)
^{129}I	2.88E+01 (2.3%)	^{242}Pu	<1.91E+01
^{134}Cs	<7.70E+04	^{244}Pu	<8.55E-02
^{135}Cs	5.31E+02 (3.6%)	^{241}Am	6.21E+00 (2.1%)
^{137}Cs	1.54E+08 (0.83%)	^{243}Am	<3.10E+00
^{137m}Ba	1.46E+08	^{244}Cm	*6.80E-01 (33%)*
^{144}Ce	<1.08E+02	^{245}Cm	<7.47E+00
^{144}Pr	<1.08E+02	Total Alpha (w/o cesium)	<3.88E+04
^{147}Pm	<5.67E+01	Total Beta (w/cesium)	1.64E+08 (0.2%)
^{151}Sm	<3.56E+01	Total Beta (w/o cesium)	8.62E+05 (0.4%)
^{154}Eu	<1.19E+01	Total Gamma	1.46E+08

^{90}Y is calculated as equal to the ^{90}Sr result. ^{106}Rh is calculated as equal to the ^{106}Ru result. ^{125m}Te is conservatively calculated as the ^{125}Sb result. ^{137m}Ba is calculated as 94.7% of the ^{137}Cs result.[vii,∩] ^{144}Pr is calculated as equal to the ^{144}Ce result.[ϒ] Total gamma is calculated as the sum of the ^{60}Co, ^{94}Nb, ^{106}Rh, ^{125}Sb, ^{125m}Te, ^{126}Sn, ^{134}Cs, ^{137m}Ba, ^{144}Ce, ^{144}Pr, ^{154}Eu, ^{155}Eu, ^{226}Ra, ^{235}U, ^{237}Np, ^{241}Am, ^{243}Am, and ^{245}Cm results. The ^{238}Pu, $^{239/40}Pu$, and ^{241}Pu results are from radio-counting, while the ^{239}Pu, ^{240}Pu, ^{242}Pu, and ^{244}Pu results are from ICPMS. The radiochemical $^{239/40}Pu$ result cannot distinguish between the ^{239}Pu and ^{240}Pu. However, if a specific 239/240 isotopic breakdown from the tank is used; the individual ^{239}Pu and ^{240}Pu values can be calculated from this method. The

∩ While the ^{137m}Ba result is calculated from the analytically provided ^{137}Cs result, in actuality the gamma of the ^{137m}Ba is measured and the ^{137}Cs is determined from that.

ϒ Nuclear decay transitions and values are generally taken from data from www.nndc.bnl.gov, NuDat 2.6.

total alpha result is from a sample with the cesium removed before analysis (failure to remove ^{137}Cs from the sample beforehand results in interference and a resulting higher minimum detection limit). The Salt Batch 11 ^{137}Cs result is higher than the Salt Batch 10 value (1.24E+08 pCi/mL, 0.47 Ci/gal). Other major radiochemical results are typical of previous salt batches, with some of the noticeable increases in Salt Batch 11 (as a percentage of SB10) for ^{238}Pu (163%), ^{241}Pu (167%), and ^{241}Am (470%).

3.3 Hg Speciation from Eurofins

At this time, SRNL does not possess the capability to measure Hg other than in the form of total Hg, methyl mercury and ethyl mercury. Samples of the Tank 21H depth sample were sent to Eurofins Frontier Global Sciences, Inc for mercury speciation.

The 3L sample (HTF-21-17-72) was recirculated in the sampler prior to placing a 1 mL aliquot into a 100 mL volumetric flask that was diluted to 100 mL with Eurofins supplied deionized H_2O. One portion of the diluted sample was placed into a 30 mL Teflon storage bottle with zero headspace and a second portion was placed into a 15 mL glass bottle with a Teflon lined cap. Each subsample was then removed from the cells and stored in the dark. The Tank 21H subsamples remained at ~4-6 °C until final dilutions were made.

Eurofins supplied deionized water and 250 mL clear and amber glass bottles. SRNL supplied the 1.2 mL concentrated HCl preservative. Triplicate samples were prepared for this shipment. Each replicate was analyzed for seven Hg species: total Hg, total soluble (dissolved) Hg, elemental Hg (Hg(0)), ionic (inorganic) Hg (Hg(I) and Hg(II)), methyl Hg (CH_3Hg-X, where X is a counter anion), ethyl Hg (CH_3CH_2-Hg-X, where X is a counter anion), and dimethyl Hg ($(CH_3)_2Hg$). The difference between the total Hg and total soluble Hg measurements gives the particulate Hg concentration after subtracting Hg(0), i.e. Hg adsorbed to the surface of particulate matter in the sample but without resolution of the specific adsorbed species. The analytes were determined from samples in four separate bottles: 1) methyl Hg and ethyl Hg; 2) dimethyl Hg; 3) total Hg and soluble total (dissolved) Hg; and 4) ionic Hg (Hg(I) & Hg(II)) and elemental Hg.

Prior to shipment, the cells diluted sample (1:100) was diluted further in a radiochemical hood with deionized water and preservative (preservative for bottle set #1 only) nominally 1:2500 by volume, for a total dilution of nominal 1:250,000. SRNL deionized water was employed as the blank. All containers were filled close to the maximum allowable volume to minimize headspace within the sealed samples. In total, 16 aqueous samples were prepared on September 5, 2017 of which 12 were shipped the following day by next-day air to Eurofins. Due to a FedEx transit delay, the samples were received on September 8, 2017. One cooler and one drum were over temperature, 8.1°C and 19.5°C, respectively. Since the drum content samples were acid preserved, there was no loss of sample integrity caused by the delay. It is not clear which of the remaining samples, that were not acid preserved, were outside the EPA method temperature parameters. Eurofins reported the aqueous sample results in units of ng Hg / L sample on September 22, 2017 and revised the methyl mercury values on April 11, 2018.

Table 6 provides the average concentrations of Hg species in the aqueous samples derived from Eurofins reported data corrected for dilutions performed by SRNL. A Tank 21H (Salt Batch 11) depth sample density of 1.269 g/mL (Table 1) was used in the calculations. All blanks were reported at the reporting limits, or 'RL' values. The RL values given by Eurofins are typically 1X to 7X higher than the associated detection limits, or 'DL' values. The RL values typically are associated with the 'quantification' limit for a given analyte and analytical method. There is a ±20% uncertainty that Eurofins reports in the measurement of total Hg and total soluble Hg, which are used to determine the particulate Hg value after subtracting Hg(0) for aqueous samples. The Hg(0) may be removed when the aqueous samples are filtered for total soluble Hg. The Hg(0) values reported were determined from the ionic Hg bottles (Set #4) because Eurofins has learned that analyzing the Hg(0) after sampling for dimethyl Hg leads to a significant loss of Hg(0) to the headspace created in the sample bottle. Eurofins purged the Hg(0) from the ionic Hg bottles prior to determining ionic Hg.

Dimethyl Hg and ethyl Hg were below the DL for all Tank 21 replicates so the RL values are given in Table 6.

The last column of Table 6 provides the percent of total Hg that the six species (particulate, elemental, ionic, methyl, ethyl, and dimethyl) represent. A range is provided to account for the uncertainty of the reporting limit values reported for various species. The recovery for the sample is high (92-94%). Table values in parentheses and brackets are the %RSD and number of replicates, respectively.

Table 6. Average Concentrations of various Hg species for Tank Samples expressed as mg Hg/L (ppm) (%RSD) [replicates]

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Total Hg	146 (1.1%) [3]	Methyl Hg	68.0 (4.2%) [3]
Dissolved Hg	131 (1.8%) [3]	Ethyl Hg	<1.9
Particulate Hg	14.0[r]	Dimethyl Hg	<0.054
Elemental: Hg(0)	1.47 (4.6%) [3]	Species Fraction of Total Hg	92-94%
Ionic: Hg(I)+Hg(II)	51.6 (4.3%) [3]		

While there is a noticeable difference between the SRNL total Hg (unfiltered) value (119 mg/L) and the Eurofins result (146 mg/L), the results overlap within the ±20% (1-σ) method uncertainty. SRNL will note that the total Hg results are approximately the same as for Salt Batch 10. There are some noticeable differences in the Salt Batch 11 distribution of mercury species. For

[r] Uncertainty in the total Hg and dissolved Hg measurements is ± 20% (1-σ), the particulate value is the difference of these two measured values after subtracting Hg(0) for the aqueous samples.

particulate, methyl-, dissolved-, and elemental mercury species, the SB11/SB10 concentration ratios are: 0.29, 2.01, 1.26, and 0.36, respectively.

4.0 Conclusions and Recommendations

SRNL analyzed samples from Tank 21H in support of acceptance of Macrobatch (Salt Batch) 11 for the ISDP. This document reports characterization data on the samples of Tank 21H and fulfills the requirements of Deliverable 3 of the TTR.[iii]

Results of the analyses of the Tank 21H Salt Batch 11 samples from this report indicate that the material does not display any unusual characteristics. The ^{137}Cs result is 0.583 Ci/gal and the sodium is 6.48 M. The Pu and ^{90}Sr results indicate that a MST strike will not be needed to verify batch acceptance for ISDP processing.

Further work will report the results of the ESS testing (Task 5 of the TTR) using the Tank 21H material. Task 4 of the TTR (MST Strike) will not be completed for ISDP processing of Salt Batch 11.

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