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Results from the Interim Salt Disposition Program Macrobatch 10 Tank 21H Qualification Samples

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Macrobatch (Salt Batch) 10 for the Interim Salt Disposition Program (ISDP). 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 shows 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 qualification.
- The Eurofins Hg sample analysis results are all less than the WAC limits.

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 Salt Batch 10.

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LIST OF ABBREVIATIONS

AA	Atomic Absorption
AD	Analytical Development
CV-Hg	Cold Vapor Mercury
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 Thenoyl Trifluoroacetone
%RSD	Percent Relative Standard Deviation
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
Wt%	Weight percent

1.0 Introduction

This report provides the Tank 21H qualification sample results for ISDP Macrobatch (Salt Batch) 10. A previous document covered initial characterization which included results for a number of radiological and non-radiological analytes[i], these analyses are also included in this report for completeness. This work was specified in a TTR [ii] and in a Task Technical and Quality Assurance Plan (TTQAP).[iii] Details of the work are contained in controlled laboratory notebooks.[iv]

2.0 Experimental Procedure

Two 200 mL Tank 21H samples (HTF-21-16-104 and -105) and a single 1L Tank 21H sample (HTF-21-16-106) were collected and delivered to SRNL on November 21, 2016. The two 200 mL samples were surface samples and the 1L sample was a variable depth sample obtained approximately 62" from the bottom of the tank (transfer pump suction). The contents of Tank 21H were mixed at full speed for approximately 15 hours with one pump before the samples were collected; the samples were collected approximately 28 hours after pump shutdown. All the samples had the same visual appearance, clear solutions with no apparent solids.

A few mL of each sample were filtered using a 0.45 μm syringe filter and measured for density. This was repeated to provide duplicate density measurements for each sample. Averages of the individual results, with percent relative standard deviation (%RSD)[℘] in parentheses, are reported in Table 1. With Flowsheet Engineering concurrence, the contents of the three sample bottles were then combined and mixed, henceforth referred to as the composite sample. After combining and allowing the contents of the composite bottle to sit for ten days, it was found that a thin layer of fine off-white solids had settled to the bottom of the composite bottle.

Table 1. Sample Density Measurements (20.0ºC)

Sample	Measured Density (g/mL)
HTF-21-16-104	1.253 (0.73%)
HTF-21-16-105	1.257 (0.17%)
HTF-21-16-106	1.253 (0.34%)
Average	1.254 (0.20%)

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 Analytical Development (AD) for a battery 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.

[℘] %RSD is defined as the standard deviation of the array, divided by the average of the array, expressed in % terms.

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 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 20.0 ˚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
Ion Chromatography (IC) Cations	NH_4^+
IC Anions	F, Cl, formate, nitrite, nitrate, sulfate, phosphate, oxalate
Inductively Coupled Plasma Emission Spectroscopy (ICPES)	Ag, Al, Ba, Cd, Cr, Cu, K, Mn, Na, P, Pb, Si, Ti
Total Inorganic Carbon (TIC)	total inorganic carbon (carbonate)
Total Organic Carbon (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

Table 3. Radiological Analyses

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

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 to 209 and 230 to 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 Qualification 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 values shaded in green are calculated results.

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

Table 4. Non-Radiological Results of Tank 21H Analyses for Macrobatch 10[∫]

Analyte	Result (mg/L)	Analyte	Result (mg/L)
Ag	<1.53	U	<43.9[◊]
Al	6770 (0.84%)	V	<0.7
B	60.5 (0.23%)	Zn	1.79 (3.6%)
Ba	<0.116	Zr	<0.707
Be	<0.0483	F^-	<100
Ca	<1.24	Cl^-	722 (0.29%)
Cd	<1.5	Br^-	<500
Ce	<4.66	Formate	258 (0.27%)
Cr	52.0 (0.27%)	Nitrite	34600 (3.7%)
Cu	<5.46	Nitrate	92200 (2.0%)
Fe	<2.36	Phosphate	315 (1.4%)
Gd	<1.15	Sulfate	4350 (2.3%)
K	425 (4.7%)	Oxalate	420 (0.51%)
La	<1.17	TIC	3400 (0.0%)
Li	11.8 (1.2%)	TOC	238 (3.6%)
Mg	<0.283	pH	14
Mn	<0.21	Ammonium	<10
Mo	13.6 (1.0%)	NORPAR 13	<1
Na	6.07 (0.51%) M	Phenol	<1
Ni	<6.04	TPB	<5
P	134 (1.1%)	TBP	<1
Pb	<20.7	Propanol	<0.25
S	1690 (0.42%)	Butanol	<0.25
Sb	<42.7	Methanol	146
Si	21.7 (4.3%)	As	0.221 (9.3%)
Sn	<13.1	Hg (unfiltered)	112 (2.5%)
Sr	<0.0422	Hg (filtered)	101 (2.8%)
Th	<0.001[♦]	Se	0.275 (9.8%)
Ti	<8.96	Free Hydroxide	2.71 (0.26%) M

[∫] Several analytes listed in Table 4 were not requested in the TTR, but are included for completeness.
[◊] While the U value is provided by ICPES, a better total uranium value can be derived from the sum of the uranium isotopic results listed in Table 5 (12.2 mg/L).
[♦] Previously, a result of <9.93 mg/L from ICPES was reported. However, it was found that a lower detection limit of <0.001 mg/L was obtained from ICPMS.

The TIC and TOC results are in terms of mg/L of carbon. If we assume that the entire TIC result is carbonate, this translates to a carbonate concentration of 0.283 M. TIC results are reported in μg C/mL, so the TIC result is divided by 12,000 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 9, with typically <20% differences between the batches for the major components.

SRNL also notes the total Hg values are higher than in Salt Batch 9.[v] The sample filtration had no effect on the mercury concentration for SRNL's determination given that the results are statistically the same.

Per Table 4, the oxalate concentration is 420 mg/L, and the formate concentration is 258 mg/L. The oxalate result is converted to the equivalent carbon result of 114 mg/L. The formate result is converted to the equivalent carbon result of 69.1 mg/L. Subtracting these results from the TOC result gives a remainder of 54.9 mg/L of carbon. If it is assumed that all the remaining carbon is in the form of methanol, the calculated methanol result is 146 mg/L. This value is likely grossly conservative. No direct analytical method for methanol is available.

3.2 Tank 21H Qualification Results (radiological analytes)

The results of the radiological analysis in pCi/mL are listed in Table 5. The analytical uncertainty for ICPMS samples 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 10 (pCi/mL)

Analyte	Result	Analyte	Result
^{3}H	1.54E+03 (1.4%)	^{155}Eu	<4.36E+01
^{14}C	9.00E+02 (13%)	^{226}Ra	<9.18E+00
^{59}Ni	<2.12E+01	^{232}U	8.57E+00 (43%)
^{63}Ni	<1.76E+01	^{233}U	2.08E+01 (1.2%)
^{60}Co	<6.62E+00	^{234}U	8.75E+01 (1.1%)
^{90}Sr	3.06E+05 (13%)	^{235}U	6.80E-01 (0.94%)
^{90}Y	3.06E+05 (13%)	^{236}U	3.31E+00 (1.1%)
^{94}Nb	<7.88E+00	^{238}U	4.05E+00 (1.5%)
^{99}Tc	3.87E+04 (2.7%)	^{237}Np	4.34E+00 (3.1%)
^{106}Ru	<1.08E+02	^{238}Pu	1.83E+04 (19%)
^{106}Rh	<1.08E+02	^{239}Pu	7.81E+02 (2.8%)
^{125}Sb	2.51E+02 (0.63%)	^{240}Pu	3.16E+02 (4.6%)
^{125m}Te	2.51E+02 (0.63%)	$^{239/40}$Pu	8.37E+02 (11%)
^{126}Sn	3.69E+02 (0.52%)	^{241}Pu	5.63E+03 (17%)
^{129}I	2.57E+01 (4.5%)	^{242}Pu	<3.82E+00
^{134}Cs	<2.27E+04	^{244}Pu	<1.77E-02
^{135}Cs	4.91E+02 (0.0%)	^{241}Am	1.32E+00 (26%)
^{137}Cs	1.24E+08 (2.3%)	^{243}Am	<3.20E+00
^{137m}Ba	1.17E+08 (2.3%)	^{244}Cm	*4.95E-01 (120%)*
^{144}Ce	<1.45E+02	^{245}Cm	<8.24E+00
^{144}Pr	<1.45E+02	Total Alpha (w/o cesium)	<2.36E+04
^{147}Pm	<1.06E+02	Total Beta (w/cesium)	1.55E+08 (1.9%)
^{151}Sm	<4.30E+01	Total Beta (w/o cesium)	7.79E+05 (1.2%)
^{154}Eu	<2.11E+01	Total Gamma	1.17E+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.[vi,∩] ^{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 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 ^{137}Cs result is lower than Salt Batch 9 (2.44E+08 pCi/mL). The ^{137}Cs result for Salt

∩ 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.

Batch 10 is 1.24E+08 pCi/mL (0.47 Ci ^{137}Cs/gal). Other major radiochemical results are typical of previous salt batches.

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. Samples of the Tank 21H depth sample were sent to Eurofins Frontier Global Sciences, Inc for mercury speciation.

The 1L sample (HTF-21-16-106) 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.[vii]

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, unless specified below.

Prior to shipment, the cells diluted sample was diluted 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 January 17, 2017 and shipped the following day by next-day air to Eurofins where 12 samples were received on January 19, 2017. FedEx delayed the delivery of the four acid preserved samples in Memphis and were delivered to Eurofins on January 20, 2017. Since these were acid preserved, there was no loss of sample integrity caused by the delay. Eurofins reported the aqueous sample results in units of ng Hg / L sample on February 9, 2017 and the density measurements on February 10, 2017.

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 10) depth sample density of 1.253 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 dimethylHg 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 was measured at a low level above the RL in one Tank 21 replicate and this value is given in Table 6. Ethyl Hg was not measured above the detection or reporting limits.

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 reasonably high (81-82%) relative to some previous data sets. 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	157 (1.4%) [3]	Methyl Hg	33.8 (10%) [3]
Total Soluble Hg	104 (2.2%) [3]	Ethyl Hg	<1.8
Particulate Hg	48.1[ϒ]	Dimethyl Hg	0.0512 (1%)[⊕]
Elemental: Hg(0)	4.12 (2.6%) [3]	Species Fraction of Total Hg	81 – 82%
Ionic: Hg(I)+Hg(II)	40.1 (12%) [3]		

All of the mercury limits in the WAC (Total Hg, Elemental Hg, Methyl Hg and Ethyl Hg) are not challenged by the sample results from Eurofins. While there is a noticeable difference between the SRNL total Hg value (112 mg/L) and the Eurofins result (157 mg/L), the results overlap within the ±20% (1-σ) method uncertainty. Additionally, neither value challenges the WAC limits. The reason for the difference is being investigated in connection with recent Tank 50H data, which has shown a similar discrepancy. SRNL will note that the total Hg results are all greater than those for Salt Batch 9.

[ϒ] Uncertainty in the total Hg and total soluble 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.
[⊕] Based upon one value above the RL.

4.0 Conclusions and Recommendations

Savannah River National Laboratory (SRNL) analyzed samples from Tank 21H in support of qualification of Macrobatch (Salt Batch) 10 for the Interim Salt Disposition Program (ISDP). This document reports characterization data on the samples of Tank 21H and fulfills the requirements of Deliverable 3 of the Technical Task Request (TTR).[ii]

Results of the analyses of the Tank 21H Salt Batch 10 samples from this report indicate that the material does not display any unusual characteristics. The Pu and ^{90}Sr results indicate that a MST strike will not be needed for qualification.

The Eurofins sample results are all less than the WAC limits.[vii]

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 Salt Batch 10.

In order to better understand the solids present in the Tank 21H sample, SRNL recommends analyzing the solids for bulk chemical makeup.

5.0 References

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