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Characterization of DWPF Recycle Condensate Tank Materials

C. J. Bannochie

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EXECUTIVE SUMMARY

A Defense Waste Processing Facility (DWPF) Recycle Condensate Tank (RCT) sample was delivered to the Savannah River National Laboratory (SRNL) for characterization with particular interest in the concentration of I-129, U-233, U-235, total U, and total Pu. Since a portion of Salt Batch 8 will contain DWPF recycle materials, the concentration of I-129 is important to undertand for salt batch planning purposes. The chemical and physical characterizations are also needed as input to the interpretation of future work aimed at determining the propensity of the RCT material to foam, and methods to remediate any foaming potential. According to DWPF the Tank Farm 2H evaporator has experienced foaming while processing DWPF recycle materials. The characterization work on the RCT samples has been completed and is reported here.

The composition of the Sludge Batch 8 (SB8) RCT material is largely a low base solution of 0.2M $NaNO_2$ and 0.1M $NaNO_3$ with a small amount of formate present. Insoluble solids comprise only 0.05 wt.% of the slurry. The solids appear to be largely sludge-like solids based on elemental composition and SEM-EDS analysis. The sample contains an elevated concentration of I-129 (38x) and substantial 59% fraction of Tc-99, as compared to the incoming SB8 Tank 40 feed material. The Hg concentration is 5x, when compared to Fe, of that expected based on sludge carryover. The total U and Pu concentrations are reduced significantly, 0.536 wt.% TS and 2.42E-03 wt.% TS, respectively, with the fissile components, U-233, U-235, Pu-239, and Pu-241, an order of magnitude lower in concentration than those in the SB8 Tank 40 DWPF feed material.

This report will be revised to include the foaming study requested in the TTR and outlined in the TTQAP when that work is concluded.

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LIST OF ABBREVIATIONS

AD	Analytical Development
AR	Aqua Regia (Digestion)
BSD	Backscattered Electron Detector
CSEM	Contained Scanning Electron Microscopy
CVAA	Cold Vapor Atomic Absorption
DI	Deionized
dpm	Disintegrations Per Minute
DWPF	Defense Waste Processing Facility
EDS	Energy Dispersive Spectroscopy
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
PF	Peroxide Fusion (Digestion)
RCT	Recycle Condensate Tank
RSD	Relative Standard Deviation
SB8	Sludge Batch 8
SE	Secondary Electron (Detector)
SEM	Scanning Electron Microscopy
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TICTOC	Total Inorganic Carbon – Total Organic Carbon
TS	Total (Dried) Solids
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request

1.0 Introduction

A Defense Waste Processing Facility (DWPF) Recycle Condensate Tank (RCT) sample was delivered to the Savannah River National Laboratory (SRNL) for characterization with particular interest in the concentrations of I-129, U-233, U-235, total U, and total Pu. Since a portion of Salt Batch 8 will contain DWPF recycle materials, the concentration of I-129 is important to understand for salt batch planning purposes. The chemical and physical characterizations are also needed as input to the interpretation of future work aimed at determining the propensity of the RCT material to foam, and methods to remediate any foaming potential. The Tank Farm 2H evaporator has experienced foaming while processing DWPF recycle materials. This work was requested in a Technical Task Request (TTR)[1], and a Task Technical and Quality Assurance Plan (TTQAP)[2] was prepared. The characterization work on the RCT samples has been completed and is reported here.

2.0 Experimental Procedure

2.1 Sample Receipt and Consolidation

Five, 200 mL doorstops were received at SRNL on July 25, 2014. The doorstops were combined into a single 1-L wide mouth, high density, polyethylene storage bottle. One doorstop was extremely full, so a small portion was lost upon transfer to the composite bottle. The mass of transferred material was 919.14 g. The slurry was allowed to settle overnight and upon doing so it developed approximately ⅓ – ½ inch of black solids. Rinsing the doorstops with supernatant liquid did not increase the amount of consolidated material, i.e., initial transfers were complete.

2.2 Analytical Preparations

Weight percent solids[3] and density[4] were measured on both the slurry and supernate. The supernate was collected by filtering slurry through a 0.5µm filter cup.

Approximately 5 g of supernate was diluted to 50 mL in a volumetric flask with 1M nitric acid and subsamples submitted to Analytical Development (AD) for inductively coupled plasma – atomic emission spectroscopy (ICP-AES). Approximately 2 g of slurry was diluted to 50 mL in a volumetric flask with deionized water (DI H_2O), with subsamples submitted for total inorganic carbon – total organic carbon (TICTOC) and total base/free OH^-/other bases analyses. Separately, approximately 5 g of slurry was diluted to 50 mL with DI H_2O, filtered through a 0.5 µm filter cup, and subsamples submitted for ion chromatography (IC) anion analysis.

Slurry was subjected to three separate digestions: aqua regia (AR),[5] peroxide fusion (PF),[6] and I-129 special preparation.[7] The AR and PF digestions target 0.25 g of total solids diluted to 100 mL while the I-129 preparation used 0.13 g of total solids diluted to 36.5 mL. The AR digestions were submitted for elemental analysis by ICP-AES and inductively coupled plasma – mass spectrometry (ICP-MS), and Hg analysis by cold vapor atomic absorption (CVAA) spectroscopy. The PF digestions were submitted for elemental analysis by ICP-AES, Pu-238/241, and U-233/234/235/236. The radiochemical separations and counting methods have been described elsewhere in great detail.[7]

Solids collected and air dried following the preparation of supernate were sampled twice and submitted to AD for scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS). All data has been recorded in the SRNL E-Notebook system.[8,9]

2.3 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.

3.0 Results and Discussion

The analytical results for the analyses conducted on the SB8 RCT sample appear in the following tables. Table 3-1 provides the weight percent solids[10] and density information collected on the sample. The insoluble solids settle rapidly, but comprise a small portion of the total solids present. Due to the small amount of insoluble solids the value given is at best an approximation due to limitation in the measurement of these trace insoluble solids levels. The closeness of the slurry and supernate densities is consistent with a low insoluble solids content. When settled they appear black, and comprise a layer of about ⅓ to ½ an inch at the bottom of a 1 L poly bottle.

Table 3-1 Weight Percent Solids and Density for the SB8 RCT Sample [Number of Replicates Included in the Average]

Property	SB8 RCT	%RSD*
Slurry Density (g/mL)	1.03 [4]	0.97
Supernate Density (g/mL)	1.02 [4]	0.26
Total Solids (Wt.% in Slurry)	3.03 [4]	4.25
Dissolved Solids[a] (Wt.% in Supernate)	2.98 [4]	2.60
Insoluble Solids (Wt.% in Slurry)	0.050	NA
Soluble Solids[b] (Wt.% in Slurry)	2.98	NA

NA ≡ not applicable
* Parenthetical %RSD values are relative to the true calculated averages of the quantities in the table, while the average values reported have been rounded off to a reasonable number of significant figures.
[a] Also known as Uncorrected Soluble Solids
[b] Also known as Corrected Soluble Solids

Table 3-2 provides the results from the supernate analyses conducted on the SB8 RCT sample for the analytes listed in column 1 by the method listed in column 6. Columns 2 and 3 provide the results on a supernate basis in moles/L (M), while columns 4 and 5 provide the results on a slurry basis in mg/kg. The IC data was obtained from weighted dilutions of slurry which were then filtered prior to analysis. The ICP-AES results were obtained from supernate dilutions into acid, as previously described in Section 2.2. The sample is mostly comprised of sodium nitrate and nitrite salts along with some measurable formate. The nitrite to nitrate ratio is more than 2X on a molar basis. The measurable metal ions, i.e. those above the detection limits, are provided in the Table 3-2. The detection limits are dependent upon the dilutions submitted. Sodium dominates, with measurable levels of other metal ions in the following order of abundance, but all orders of magnitude below the concentration of Na:

$$Al > S > Si > U > B > P > Li > Ca \approx Cr$$

Table 3-2 Supernate Analyses for SB8 RCT Sample [Number of Samples Included in Average]

1	2	3	4	5	6
Analyte	SB8 RCT (%RSD*) Mol/L super. Wt'd Dil. Slurry	SB8 RCT (%RSD*) Mol/L super. Wt'd Dil. Super.	SB8 RCT (%RSD*) mg/kg slurry Wt'd Dil. Slurry	SB8 RCT (%RSD*) mg/kg slurry Wt'd Dil. Super.	Method
NO_3^-	0.0855 (1.7) [3]	NA	5170 (1.7) [3]	NA	IC
NO_2^-	0.186 (0.69) [3]	NA	8340 (0.69) [3]	NA	IC
SO_4^{2-}	<0.00103	NA	<95.5	NA	IC
PO_4^{3-}	<0.00104	NA	<95.5	NA	IC
Br^-	<0.00124	NA	<95.5	NA	IC
Cl^-	<0.00279	NA	<95.5	NA	IC
CHO_2^-	0.0179 (0.82) [3]	NA	787 (0.82) [3]	NA	IC
$C_2O_4^{2-}$	<0.00112	NA	<95.5	NA	IC
F^-	<0.00520	NA	<95.5	NA	IC
Al	NA	0.00194 (2.2) [4]	NA	50.9 (2.2) [4]	ICP-AES
B	NA	0.00140 (2.3) [4]	NA	14.8 (2.3) [4]	ICP-AES
Ca	NA	0.0000141 (11) [4]	NA	0.552 (11) [4]	ICP-AES
Cr	NA	0.0000102 (8.8) [4]	NA	0.520 (8.8) [4]	ICP-AES
Li	NA	0.000330 (2.7) [4]	NA	2.23 (2.7) [4]	ICP-AES
Na	NA	0.401 (4.4) [4]	NA	8980 (4.4) [4]	ICP-AES
P	NA	0.000151 (1.0) [4]	NA	4.55 (1.0) [4]	ICP-AES
S	NA	0.00133 (9.4) [4]	NA	41.6 (9.4) [4]	ICP-AES
Si	NA	0.00135 (1.7) [4]	NA	37.1 (1.7) [4]	ICP-AES
U	NA	0.000131 (1.9) [4]	NA	30.4 (1.9) [4]	ICP-AES

NA ≡ not measured
* Parenthetical %RSD values are relative to the true calculated averages of the quantities in the table, while the average values reported have been rounded off to a reasonable number of significant figures.

Table 3-3 provides the TICTOC results for the SB8 RCT sample. There were equal amounts of inorganic and organic carbon based on the results. However, the sample blank had the unusual situation of having about 80% of the inorganic carbon found for the sample; there is currently no explanation for this observation. The dilutions submitted for base analysis (total base, free hydroxide, and other base excluding carbonate) gave no measurable readings, so it would appear that the sample is not highly caustic.

Table 3-3 Carbon Analysis for SB8 RCT Sample [Number of Samples Included in Average] (mg C/kg slurry)

Analyte	Slurry Wt'd Dilution SB8 RCT (%RSD*)
Total Inorganic Carbon	238 (5.4) [4]
Total Organic Carbon	235 (5.4) [4]
Total Carbon	474 (4.5) [4]

Table 3-4 provides the elemental composition of the SB8 RCT material based upon the total digestions performed by AR and PF. The analyses were obtained from ICP-AES analysis unless indicated otherwise in the table footnotes. Once again Na dominates at nearly 31 wt.% of total solids. The relative abundance of elements is:

$$Na >> Fe > Hg \approx Al > Mn > U > Si > Ni > Ca > S > Th \qquad \text{SB8 RCT}$$

The ratio of U to Th is roughly 5:1 in the RCT sample which is close to the 4.5:1 found for Tank 40 SB8 material.[11] The composition is reminiscent of dilute sludge components with an increased ratio of Hg to the other elements:

$$Fe > Na > Al > Mn > U > Hg > Ni > Si > Ca > Th \qquad \text{SB8 Tank 40}$$

Table 3-4 Elemental Concentration in SB8 RCT Sample in Wt. % of Total Dried Solids (%RSD) [Number of Samples Included in Average]**

Element	SB8 RCT	Element	SB8 RCT
Al	1.09 (2.4) [3]	Mn	0.802 (5.3) [6]
B	0.0624 (5.7) [6]	Mo	<0.016
Ba	0.0113 (5.3) [6]	Na	30.9 (2.9) [3]
Be	<0.00013	Ni	0.251 (10) [6]
Ca	0.163 (9.2) [3]	P	0.0322 (6.4) [3]
Cd‡	0.00269 (6.7) [3]	Pb‡	0.00506 (4.7) [3]
Ce‡‡	0.0313 (2.6) [3]	S	0.148 (1.4) [3]
Co	0.00137 (1.0) [3]	Sb	<0.038
Cr	0.0205 (7.2) [3]	Si	0.363 (11) [3]
Cu	0.0204 (4.1) [3]	Sn	<0.076
Fe	2.27 (7.0) [6]	Sr	0.00518 (4.1) [3]
Gd‡	0.0121 (5.4) [3]	Th‡‡	0.106 (5.8) [3]
Hg^	1.20 (2.1) [3]	Ti	0.00186 (6.8) [3]
K	0.0959 (8.6) [3]	U‡‡	0.536 (3.0) [3]
La‡	0.00764 (5.7) [3]	V	<0.00028
Li	0.0104 (4.4) [3]	Zn	0.00507 (2.7) [3]
Mg	0.0362 (6.6) [6]	Zr‡‡‡	0.0329 (12) [3]

* ICP-AES data unless specified otherwise. ^ Calculated from CV-AA data.
** Parenthetical %RSD values are relative to the true calculated averages of the quantities in the table, while the average values reported have been rounded off to a reasonable number of significant figures.
‡ Calculated from MS data for Cd: Cd-112, Cd-114; La-139; Gd: Gd-155, Gd-156, Gd-157, Gd-158, Gd-160; Pb: Pb-206, Pb-207, Pb-208; and Th-232; respectively.
‡‡ Calculated from the sum of MS data for Ce: Ce-140 and Ce-142; U: U-233, U-234, U-235, U-236, and U-238.
‡‡‡ Zr may be biased low based upon the value obtained for the ARG standard.

The concentration of Hg is lower than that found for the incoming SB8 Tank 40 feed, 1.86 wt.% TS, but still significant considering the DWPF processing to remove it that has occurred. The ratio of Hg:Fe in

SB8 is 0.11, while in the RCT material it is 0.53, or five times as much as would be expected from sludge carryover.

Table 3-5 gives the activities, expressed as µCi/g total dried solids (TS), and concentrations, expressed as wt.% TS, of select radionuclides[1] found in the SB8 RCT sample. If the activities provided for U in the table are converted to a wt.% TS basis with the specific activities of each isotope[12] and summed, the value is 0.535 wt.% TS, essentially the same value determined for U in Table 3-4. Since both measurements came from separate digestions, i.e. those in Table 3-5 from PF and those in Table 3-4 from AR, and were analyzed by different instruments, this agreement significantly improves the reliability of this data.

In a similar manner, if the specific activities of the Pu isotopes are employed to convert the Pu isotope activities to the mass of each Pu isotope and then summed, the total Pu is not more than 2.42E-03 wt.% TS – the uncertainty arises from the detection limit value for Pu-242.

The concentration of I-129 is increased about 38x over that found for the incoming SB8 Tank 40 feed to DWPF (1.15E-03 wt.% TS).[7] This would seem to point to a significant portion not being incorporated into the glass. The concentration of Tc-99 decreased over that found in SB8 Tank 40 feed (2.02E-03 wt.% TS)[7], but a substantial 59% fraction on a wt.% TS basis is in the RCT material and this appears to be mostly soluble since there was little difference between the digestion replicates for this isotope as compared to species expected to be in the insoluble solids. The U-233, U-235, Pu-239, and Pu-241 fissile component concentrations are an order of magnitude lower than those found for SB8 Tank 40 feed[7]. The values reported previously were 6.20E-04, 2.50E-02, 1.25E-02, 4.19E-05 wt.% TS, respectively.

Table 3-5 Activities of Select Radionuclides for the SB8 RCT Sample in µCi/g of Total Dried Solids

Radionuclide	Specific Activity (Ci/g)	Wt.% of Total Solids	Activity (µCi/g TS)	%RSD*	Replicates	Method
Tc-99	1.695E-02	1.20E-03	2.03E-01	2.7	4	ICP-MS
I-129	1.765E-04	4.32E-02	7.62E-02	11	4	I-129
U-233	9.680E-03	8.19E-05	7.93E-03	6.0	3	U – ICP-MS
U-234	6.248E-03	9.73E-05	6.08E-03	7.8	3	U – ICP-MS
U-235	2.161E-06	3.49E-03	7.54E-05	5.4	3	U – ICP-MS
U-236	6.469E-05	2.27E-04	1.47E-04	5.7	3	U – ICP-MS
U-238	3.362E-07	5.31E-01	1.79E-03	5.2	3	U – ICP-MS
Pu-238	1.712E+01	1.21E-04	2.08E+01	8.2	3	Pu-238/-241
Pu-239	6.216E-02	1.72E-03	1.07E+00	5.7	3	ICP-MS
Pu-240	2.279E-01	1.13E-04	2.58E-01	NA	NA	Calculated from Pu-238/-241
Pu-241	1.030E+02	4.36E-06	4.49E+00	7.9	3	Pu-238/-241
Pu-242	3.818E-03	<4.57E-04	<1.74E-02	NA	3	ICP-MS

* Values in the %RSD column are relative to the true calculated averages of the quantities in the table, while the average values reported have been rounded off to a reasonable number of significant figures.

Solids obtained during the collection of supernate were air dried and analyzed by SEM-EDS. Figure 3-1 provides an example of the SEM images that were obtained from these air dried solids.

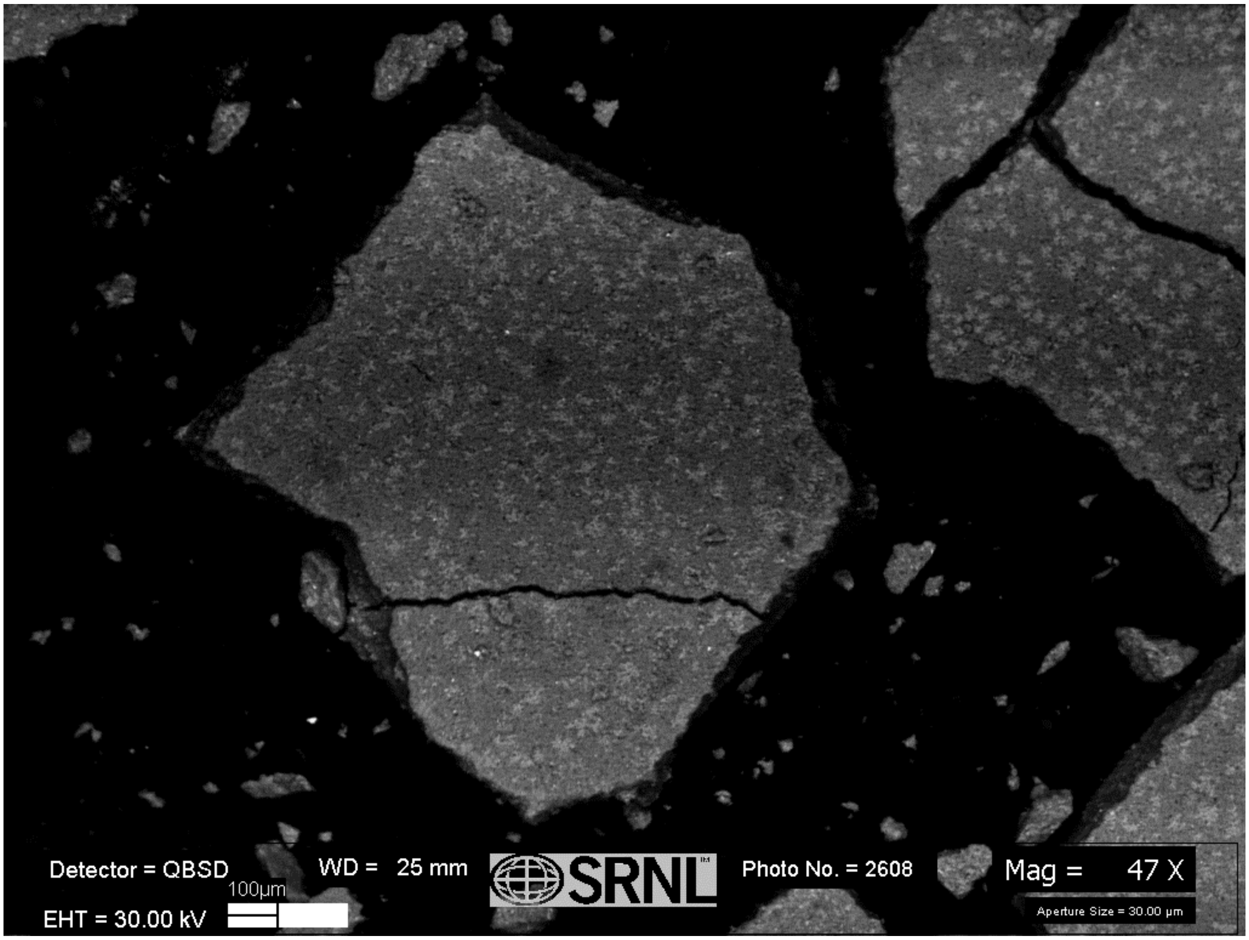

Figure 3-1 SEM Image 2608 of SB8 RCT Solids

The image shows a solid surface that is mottled with lighter and darker areas. As will be shown later, the EDS spectral analysis showed that the lighter areas contained U, as well as other elements, while the darker areas did not contain U.

Figure 3-2 shows two raster areas analyzed by EDS and marked “1” and “2”. Both areas gave the same analysis, which is shown in Figure 3-3 for area 1, and represents a characteristic overall analysis for both samples that were examined by SEM.

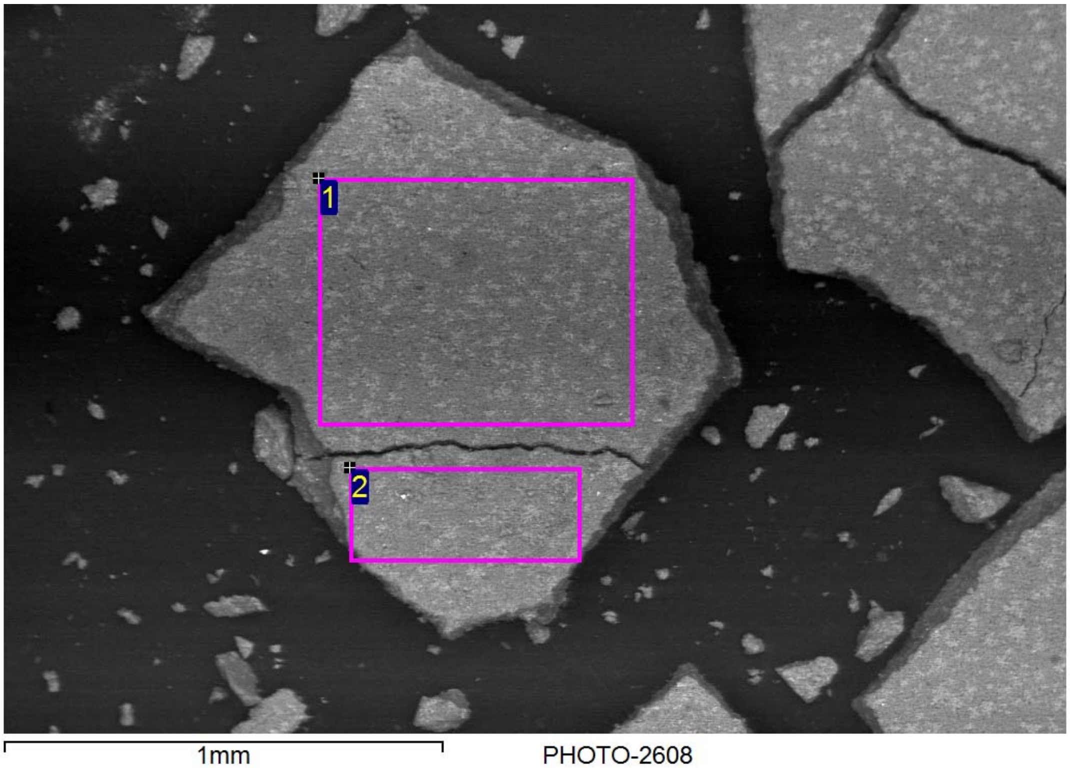

Figure 3-2 SEM Image 2608 Showing Areas of Raster Scan for EDS Analysis

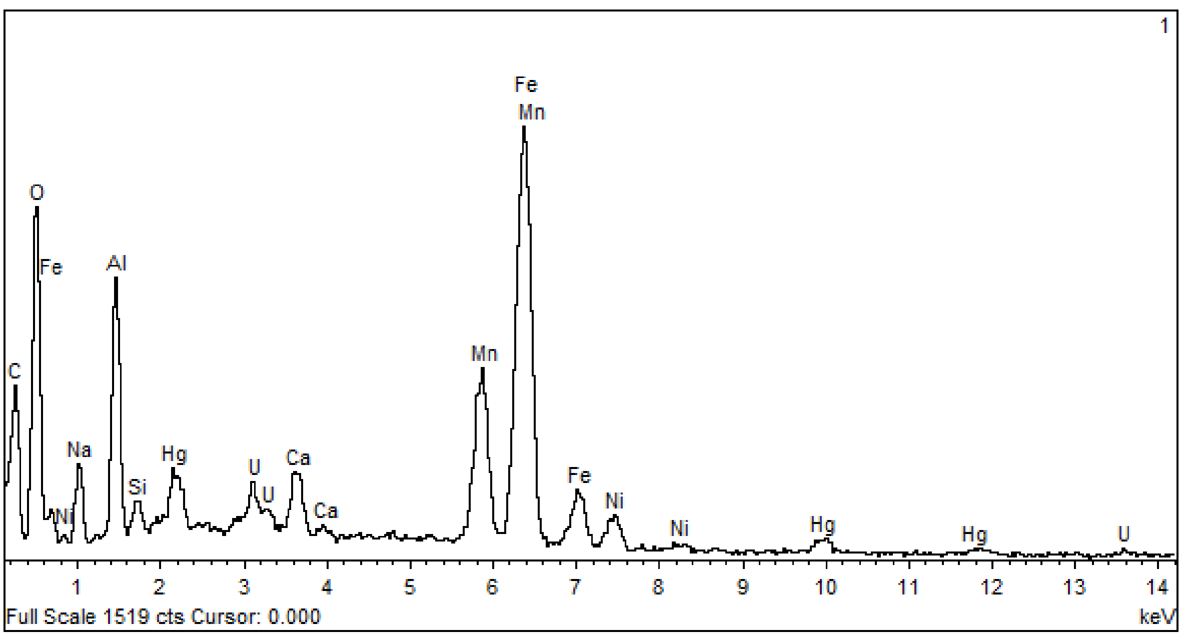

Figure 3-3 EDS Spectra for Raster Area 1 in Image 2608

The composition of elements matches that listed above as determined from ICP-AES, ICP-MS, and CVAA analysis of the digested material (Table 3-4). There is less Na since that is largely in solution, but Hg, Al, Mn, U, Si, Ni, and Ca are clearly present. Closer examination of individual areas reveals the S and Th components.

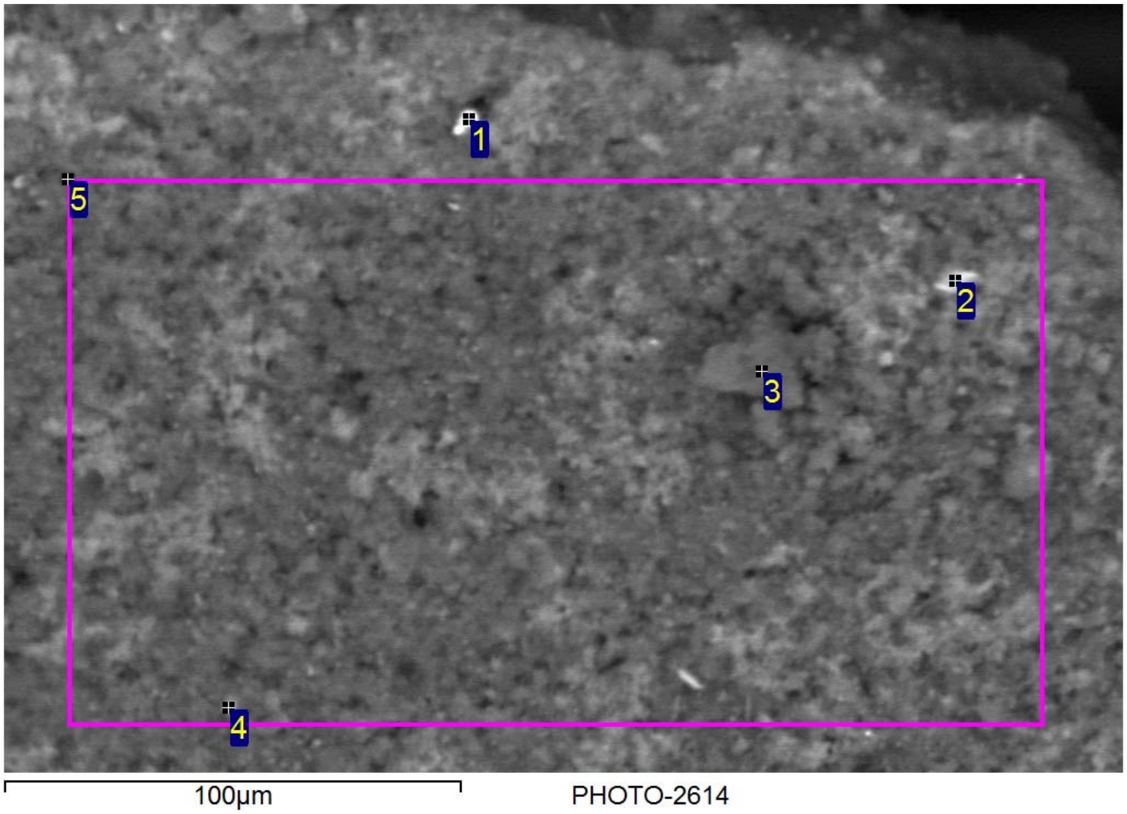

Figure 3-4 SEM Image 2614 Showing Raster Area and Select Spots for EDS Analysis

The EDS spectra of Spot 1 and 4 is shown in Figure 3-5. Spot 1 clearly shows the presence of Hg along with the other sludge components. There is likely some elemental Hg or Hg compounds present in the RCT solids. Spot 4 indicates the presence of Th distinct from the signal for U which does not show up at this spot. The spectrum for Spot 2 is not given since it was similar to that shown for Spot 4. Overall, as has been stated, there is about a 5:1 ratio of U:Th, but the Th can be selectively found with the SEM analysis.

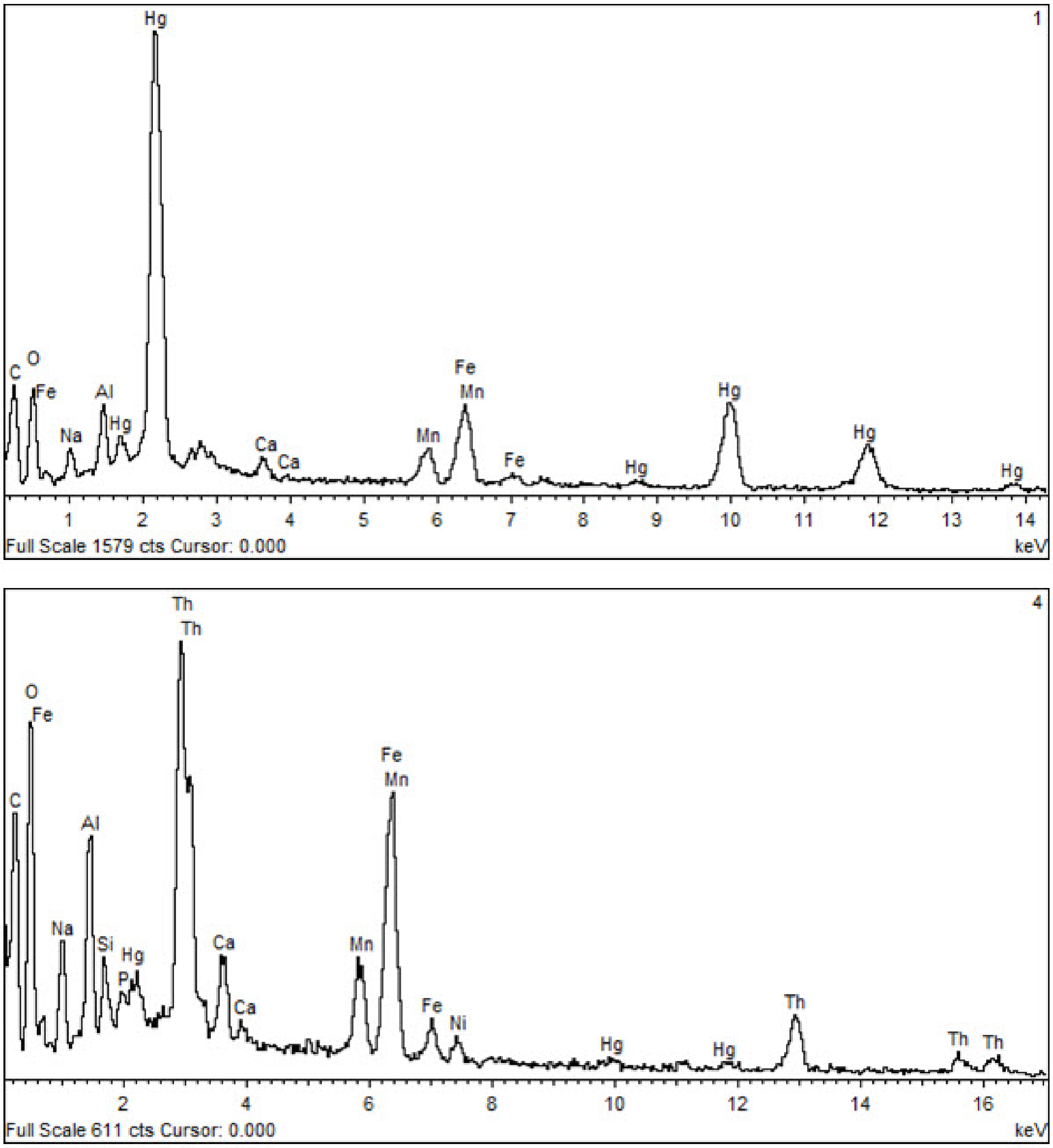

Figure 3-5 EDS Spectra of Spots 1 and 4 in Image 2614 of SB8 RCT Solids

Figure 3-6 provides the EDS spectra obtained for Spot 3 and Raster Area 5 in Figure 3-4. Both Spot 3 and Raster Area 5 show the usual sludge elemental components: Al, Si, Hg, Ca, Mn, Fe, Ni, and Th. Note, the keV window size is larger for the raster area, going from 0 – 14 keV, rather than 0 – 10 keV, so this makes the spectra appear slightly different. Additionally, the U signal at 3.0 – 3.4 keV is not labeled for Spot 3. The smaller raster area spectra collected here is nearly identical to that shown in Figure 3-3.

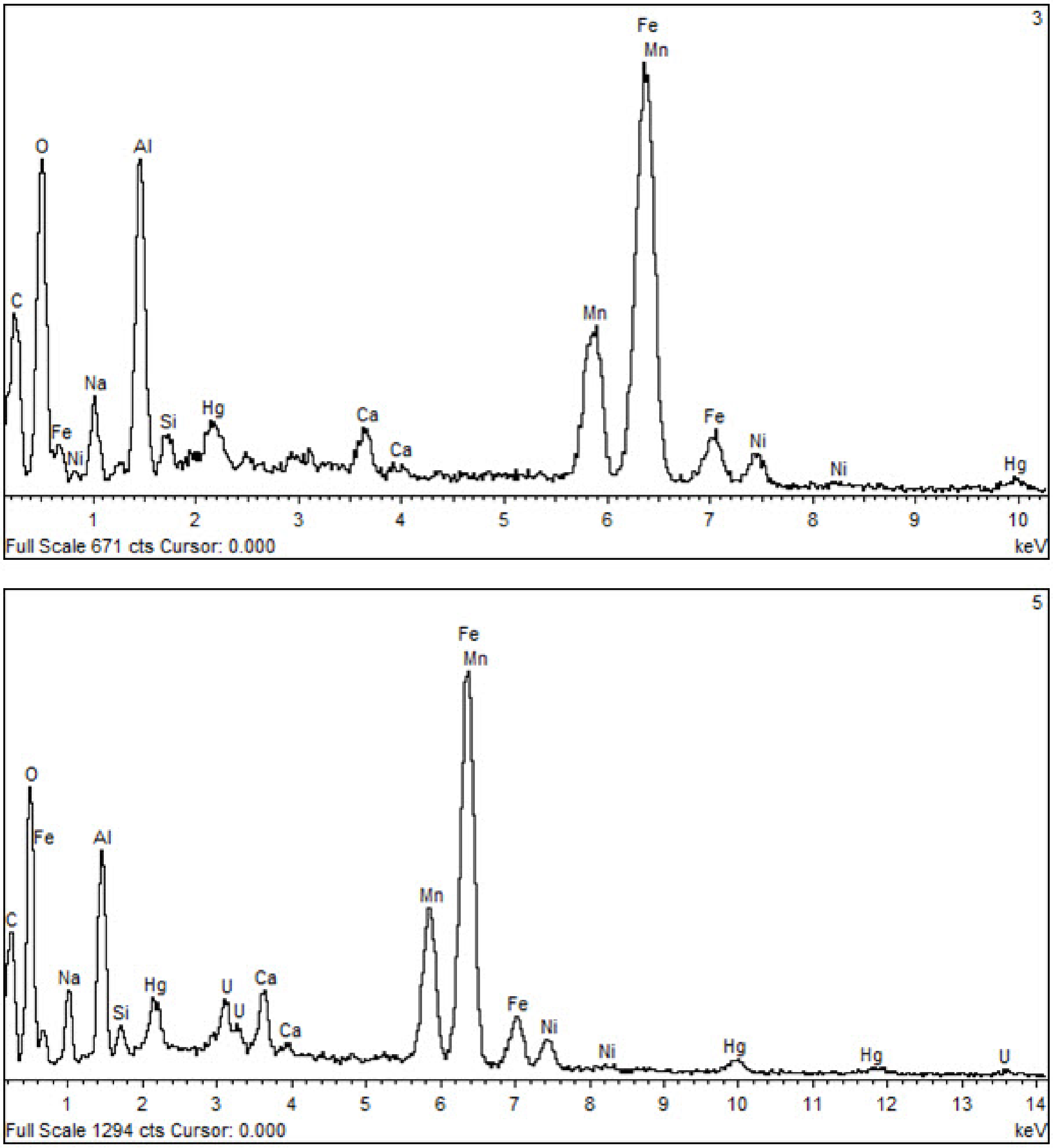

Figure 3-6 EDS Spectra of Spot 3 and Raster Area 5 in Image 2614 of SB8 RCT Solids

4.0 Conclusions

The composition of the SB8 RCT material is largely a low base solution of 0.2M $NaNO_2$ and 0.1M $NaNO_3$ with a small amount of formate present. Insoluble solids comprise only 0.05 wt.% of the slurry. The solids appear to be largely sludge-like solids based on elemental composition and SEM-EDS analysis. The sample contains an elevated concentration of I-129 (38x) and substantial 59% fraction of Tc-99, as compared to the incoming SB8 Tank 40 feed material. The Hg concentration is 5x, when compared to Fe, of that expected based on sludge carryover. The total U and Pu concentrations are reduced significantly, 0.536 wt.% TS and 2.42E-03 wt.% TS, respectively, with the fissile components, U-233, U-235, Pu-239, and Pu-241, an order of magnitude lower in concentration than those in the SB8 Tank 40 DWPF feed material.

5.0 Future Work

This report will be revised to include the foaming study requested in the TTR[1] and outlined in the TTQAP[2] when that work is concluded.

6.0 References

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T. B. Edwards, 999-W
K. M. Fox, 999-W
F. C. Johnson, 999-W
D. P. Lambert, 999-W
J. D. Newell, 999-W
D. K. Peeler, 999-W
M. E Stone, 999-W
J. R. Zamecnik, 999-W
J. W. Amoroso, 999-W

J. M. Bricker, 704-S
T. L. Fellinger, 766-H
E. J. Freed, 704-S
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. W. Holtzscheiter, 766-H
J. F. Iaukea, 704-27S
P. A. Lavigne, 704-27S
J. W. Ray, 704-27S
M. A. Rios-Armstrong, 766-H
H. B. Shah, 766-H
M. A. Sudduth, 704-27S
S. A. Utlak, 704-27S
J. R. Vitali, 704-30S

J. S. Contardi, 704-56H
V. Jain, 766-H
J. N. Leita, 704-56H
S. P. Simner, 705-1C

P. R. Jackson, DOE-SR, 703-46A
J. A. Crenshaw, 703-46A

D. J. Martin, 766-H
A. W. Wiggins, 705-1C