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Revision 0

TANK 11H ANALYTICAL RESULTS AS INPUT TO DWPF SLUDGE BATCH 4

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September 2004

Immobilization Technology Section
Savannah River National Laboratory
Aiken, SC 29808

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EXECUTIVE SUMMARY

SRNL was requested by DWPF to conduct analyses on dissolved samples of Tank 11H material in preparation for DWPF processing[a]. Two separate samples of Tank 11H were pulled during Tank Farm slurry and transfer operations. These samples have been designated Tank 11 – Sample 1 and Tank 11 – Sample 2. Aliquots of each slurry sample were digested in HNO_3/HF and analyzed by inductively coupled plasma – atomic emission spectroscopy, inductively coupled plasma – mass spectrometry, and cold vapor – atomic absorption spectroscopy.

The following conclusions can be drawn based upon the analyses of the two Tank 11 samples received by the Savannah River National Laboratory:

- The concentration of sulfur in Sample 2 is high and 99% of the sulfur is soluble. The increase in soluble sulfur is likely due to the decrease in NaOH concentration. The significance of this increase cannot be determined at this point. The second transfer from Tank 11 needs to occur and volume estimates made.
- The lower than anticipated mercury values tend to support the suspicion that all of the sludge solids in Tank 11 were not suspended at the time the second Tank 11 sample was pulled for analysis. The amount of mercury present in SB4 will impact the acid addition needs during SRAT processing.
- While still low relative to SB2 and SB3, the level of U is higher than forecast by the Waste Characterization System (WCS).
- Noble metal concentrations in these two samples are not as high as previously anticipated based upon earlier direct measurements of the noble metals[b] and WCS predicted La values in unmixed Tank 11 solids. It may be that not all of the solids were suspended at the time the second Tank 11 sample was pulled for analysis. The noble metal content will have a direct impact on the amount of hydrogen produced during SRAT processing.

Once all bulk waste removal operations from Tank 11 to Tank 51 have been completed, it is recommended that a sample be taken from Tank 51. The analysis of this sample will provide better projections of SB4 content and allow decisions to be made on washing as well as simulant and glass studies in support of qualification for DWPF processing.

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[a] Washburn, F. A. *Analysis of Tank 11 Material in Preparation of Sludge Batch 4*, HLW/DWPF/TTR-04-0016, 6/22/04.

[b] Coleman, C. J., Kinard, W. F., Bibler, N. E., Bickford, D. F., and Ramsey, W. G. *Basic Data Report: Determination of Noble Metals in Tank 51, 4, 11, and 15 High-Level Sludges*, WSRC-TR-91-396, Savannah River Site, Aiken, SC 29808 (1991)

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LIST OF ACRONYMS

ADS	Analytical Development Section
ASP	Analytical Study Plan
CV-AA	Cold Vapor – Atomic Absorption Spectroscopy
DWPF	Defense Waste Processing Facility
H&F	H-and F-Area
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
L	Liter
M	Molar
NA	Not Available (e.g. Not Measured)
RSD	Relative Standard Deviation
SB2	Sludge Batch 2
SB3	Sludge Batch 3
SB4	Sludge Batch 4
SRNL	Savannah River National Laboratory
Std. Dev.	Standard Deviation
TTQAP	Task Technical and Quality Assurance Plan
TTR	Task Technical Request
WCS	Waste Characterization System
wt %	Weight percent

1.0 INTRODUCTION AND BACKGROUND

The Tank Farm has begun slurry and transfer operations in support of Sludge Batch 4 (SB4) preparations. Several tanks including Tanks 4F, 5F, 6F, 7F and 8F and 11H are currently planned to comprise SB4. Two samples of Tank 11H, the first tank to be transferred into Tank 51for SB4, were pulled and taken to the Savannah River National Laboratory (SRNL) Shielded Cells Facility for analyses required to determine if the materials were suitable for transfer to Tank 51H in preparation for SB4[1,2,3,4]. The first sample was taken in May 2004 and the second in July 2004. In addition to corrosion chemistry analyses and counting data performed for Waste Processing Technology (WPT) personnel, the samples were analyzed for elemental composition including fission products and actinides.

SRNL was requested by DWPF via Technical Task Request (TTR) HLW/DWPF/TTR-04-0016 to conduct analyses on dissolved samples of Tank 11H material in preparation for DWPF processing[5]. The data reported here is evaluated for potential impacts on DWPF processing. The sample preparation work is governed by a Task Technical and Quality Assurance Plan (TTQAP)[6] prepared by WPT personnel and two Analytical Study Plans (ASP)[7,8], one for each sample of Tank 11 material analyzed. A separate TTQAP was not required for this work per the TTR.

According to H-& F-Area Process Support personnel Tank 11H initially contained 52" of settled sludge with a total volume/level of 98 – 105" (2710 gallons/inch). The contents of the tank were slurried and a sample drawn on May 10, 2004 for SRNL analysis. This sample is designated as Tank 11 – Sample 1. After some additional mixing, the contents were transferred to Tank 51H from June 18-21, leaving a level of approximately 25.5" in Tank 11H, but with some mounds of sludge above this level. Inhibited water was added to Tank 11 to bring the level back to 105". The contents of the tank were mixed (slurried) a second time and a sample drawn on July 29, 2004 for SRNL analysis. This sample is designated as Tank 11 – Sample 2. As of September 7, the contents of Tank 11H, following the second series of slurry operations, have not been transferred to Tank 51H. Hence, the size of the heel which will remain in Tank 11H, theoretically as little as 3" if all of the settled sludge is suspended, is still unknown. Table 1-1 summarizes the sampling dates and sample designations for the two samples received by SRNL.

Table 1-1 Tank 11 Sampling Dates and Designations

Sample Date	Sample Designation
May 10, 2004	Tank 11 – Sample 1
July 29, 2004	Tank 11 – Sample 2

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2.0 APPROACH AND RESULTS

2.1 Tank 11 – Sample 1

Tank 11 – Sample 1 was comprised of three dip samples designated HTF-04-10, HTF-04-11, and HTF-04-12 which were composited[1]. Three aliquots of the slurry were digested in HNO_3/HF for two hours at 115°C and diluted to 1:250 with deionized water. Each of these digestions along with a blank was then diluted 1:3 with deionized water and submitted to the Analytical Development Section (ADS) for radioactive inductively couple plasma – mass spectrometry (ICP-MS) analysis for masses 81-209 and 230-252. A separate set of equivalent dilutions were submitted to ADS for radioactive inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analysis along with a diluted ICP-AES solution standard.

Results documented by Swingle for sample weight percent solids and density (Table 2-1), corrosion chemistry (Table 2-2), and counting (Table 2-3)[1,2] have been included in the summary tables below. Anions were determined by ion chromatography (IC) and Na and K by ICP-AES, both on a sample of diluted supernate. Table 2-4 and Table 2-5 provide the ICP-AES and ICP-MS data collected in this work.

2.2 Tank 11 – Sample 2

The second sample of Tank 11 sludge slurry was also comprised of three dip samples designated HTF-04-26, HTF-04-027, and HTF-04-028 which were composited[3]. Three separate aliquots of the slurry were digested in HNO_3/HF for two hours at 115°C and diluted to 1:100 with deionized water. Each of these digestions along with a blank was then diluted 1:2 with deionized water and submitted to ADS for ICP-MS analysis for masses 81-209 and 230-252. Separate sets of equivalent dilutions were submitted to ADS for ICP-AES and cold vapor atomic absorption (CV-AA) analyses. The latter technique is used for Hg determination.

Results documented by Oji for sample weight percent solids and density (Table 2-1), corrosion chemistry (Table 2-2) and counting (Table 2-3)[3,4] have been included in the summary tables below. Anions were determined by ion chromatography (IC) and Na and K by ICP-AES, both on a sample of diluted supernate. Table 2-4 and Table 2-5 provide the ICP-AES, ICP-MS and CV-AA data collected in this work.

Table 2-1. Weight Percent Solids and Density for Tank 11 Samples

Property	Tank 11 – Sample 1[1] (Std. Dev.)	Tank 11 – Sample 2[3] (Std. Dev.)
Slurry Density	1.53 (0.02)	1.15 (0.01)
Supernate Density	1.43 (0.02)	1.12 (0.01)
Wt % Total Solids	52.4 (0.1)	19.4 (0.02)
Wt % Dissolved Solids	48.1 (0.01)	14.6 (0.3)
Wt % Insoluble Solids	8.31	5.68
Wt % Soluble Solids	44.1	13.8

Table 2-2. Corrosion Chemistry Analyses for Tank 11 Samples (Molar, except pH)

Analyte	Tank 11 – Sample 1[2] (Std. Dev.)	Tank 11 – Sample 2[4] (Std. Dev.)
Free OH^-	0.665 (0.079)	0.178 (0.010)
NO_3^-	3.36 (0.04)	0.744 (0.159)
NO_2^-	3.24 (0.04)	0.748 (0.159)
SO_4^{2-}	0.079 (0.002)	0.202 (0.010)
Cl^-	0.0061 (0.00002)	<0.001
CO_3^{2-}	0.170 (0.017)	<0.002
PO_4^{3-}	0.001 (0.000002)	<0.003
AlO_2^{2-}	0.382 (0.002)	0.0478 (0.0002)
$C_2O_4^{2-}$	0.0030 (0.0001)	<0.004
F^-	<0.001	<0.003
Na^+	9.32 (0.12)	2.09 (0.01)
K^+	0.0252 (0.0015)	0.00433 (0.00057)
pH	13.8	13.3

Table 2-3. Activity of Tank 11 Samples (Ci/L)

	Tank 11 – Sample 1[1,2] (Std. Dev.)	Tank 11 – Sample 2[3,4] (Std. Dev.)
Gross Alpha (slurry)	0.108 (0.007)	<0.02
Gross Gamma (filtrate)	0.316 (0.0004)	0.0490 (0.0015)

Table 2-4. Elemental Concentrations in Tank 11 Samples in Wt % of Total Solids (Std. Dev., %RSD)

Element	Tank 11 – Sample 1	Tank 11 – Sample 2	Element	Tank 11 – Sample 1	Tank 11 – Sample 2
Al	7.50 (0.10, 1.3)	8.33 (0.63, 7.5)	Mo	0.0143 (0.0006, 3.9)	0.0311 (*)
B	<0.004	<0.005	Na	26.4 (0.4, 1.7)	23.7 (1.3, 5.6)
Ba	0.0193 (0.0003, 1.5)	0.0201 (0.0038, 19)	Ni	0.224 (0.002, 0.9)	0.365 (0.011, 3.0)
Ca	0.354 (0.016, 4.6)	0.289 (0.017, 5.8)	P	0.0908 (0.0033, 3.7)	0.0620 (0.0113, 18)
Cd	<0.003	<0.002	Pb	<0.08	<0.03
Ce	0.0277 (0.0024, 8.5)	0.0824 (*)	S	0.409 (0.006, 1.5)	2.85 (0.06, 2.0)
Cr	0.0484 (0.0012, 2.5)	0.0327 (0.0034, 10.6)	Sb	0.0247 (0.0007, 2.7)	0.0350 (0.0091, 26)
Cu	0.0112 (0.0003, 3.1)	0.0151 (0.0006, 3.7)	Si	0.120 (0.004, 3.2)	1.2 (0.6, 49)
Fe	3.12 (0.04, 1.2)	2.26 (0.11, 4.8)	Sn	<0.05	<0.06
Gd	0.00241 (0.00008, 3.4)	0.00895 (*)	Sr	0.0910 (0.0048, 5.3)	0.0728 (0.0057, 7.9)
Hg	0.257† (0.006, 2.2)	0.978^ (0.023, 2.3)	Ti	0.00263 (0.00008, 3.1)	0.00539 (0.00031, 5.8)
K	<1	NA	U	0.00862‡ (0.00019, 2.2)	0.900 (0.048, 5.4)
La	0.0106 (0.0012, 12)	0.0280 (*)	V	<0.004	NA
Li	<0.005	<0.02	Zn	0.0143 (0.0004, 2.6)	0.0134 (0.0014, 10)
Mg	0.0720 (0.0009, 1.2)	0.0535 (0.0024, 4.5)	Zr	0.0437 (0.0007, 1.6)	0.0551 (0.0024, 4.4)
Mn	0.758 (0.009, 1.2)	0.503 (0.017, 3.3)			

† Calculated from MS data for Hg-196, -198, -199, -200, -201. -202, -204)

‡ Calculated from MS data for U-238

* Calculated from a single datum point, other data less than detection limit

^ Calculated from CV-AA data

NA ≡ not measured

There was good agreement between the Hg value determined in the second sample by CV-AA (0.978 wt %) and that determined from ICP-MS data for Hg-196, -198, -199, -200, -201, -202, and -204 (1.00 wt % (Std. Dev. 0.06, %RSD 5.8)). The second sample had lower concentrations of Ca, Cr, Fe, Mg, Mn, and P than the first sample. There were increases in the observed concentrations of Ce, Gd, Hg, La, Mo, Ni, S, Si, Ti, U, and Zr.

There was a seven-fold increase in the concentration of sulfur. The reason for this increase will be discussed in Section 3.

The actinide concentrations are given in Table 2-5. The U-238 concentration changed by over two orders of magnitude between the first and second sampling. The first sample was enriched to 10% U-235 while the second sample was only 0.8%, close to natural U. There was also an order of magnitude more Th-232 in the second Tank 11 sample.

Table 2-5. Actinide Concentrations in Tank 11 Samples in Wt % of Total Solids (Std. Dev., %RSD)

Isotope	Tank 11 – Sample 1	Tank 11 – Sample 2
Th-232	0.00370 (0.00018, 4.9)	0.0404 (0.0148, 37)
U-233	0.0000280 (0.0000004, 1.3)	0.000241 (0.000048, 20)
U-234	0.000380 (0.000005, 1.3)	0.000332 (0.000018, 5.5)
U-235	0.00104 (0.00002, 2.1)	0.00865 (0.00011, 1.3)
U-236	0.000449 (0.000008, 1.8)	0.000588 (0.000038, 6.5)
Np-237	0.000401 (0.000017, 4.1)	0.000387 (0.000034, 8.8)
U-238	0.00862 (0.00019, 2.2)	1.07 (0.01, 1.0)
Pu-239	0.00269 (0.00011, 4.1)	0.00235 (0.00005, 2.2)
Pu-240	0.000515 (0.000013, 2.5)	0.000384 (0.000029, 7.6)
Am-241	0.000294 (0.000010, 3.5)	0.000171 (0.000012, 6.8)
Pu-242	0.0000577 (0.0000033, 5.7)	0.0000337 (0.0000031, 9.2)

The fission product noble metal and silver concentrations are given in Table 2-6. The values were calculated from ICP-MS data using a spreadsheet developed by Ned Bibler (SRNL) which uses the fission yield to account for the mass contribution from isotopes in the tank that could not be measured because isotopes of Cd interfere.

Table 2-6. Noble Metal Fission Products and Silver Concentrations in Tank 11 Samples in Wt % of Total Solids

Element	Tank 11 – Sample 1	Tank 11 – Sample 2
Ag (-107, -109)	0.000409	0.00103
Pd (-105, -106, -107, -108, -109)	0.000670	0.000315
Rh (-103)	0.00434	0.00388
Ru (-101, -102, -104)	0.0194	0.0207

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3.0 DISCUSSION

There were significant differences between the first and second Tank 11H samples. These differences are not unexpected considering the known stratification of sludge layers in the tanks and the observation that Tank 11H still had at least 25.5" of settled solids and observed "mounds" of solids following the first transfer to Tank 51H.

The most notable observation from Tank 11 – Sample 2 is the elevated concentration of S. Based upon the measured sulfate value of 0.202 M[4] in the supernate sample a concentration of 2.81 wt % S was calculated on a total solids basis. The value determined for total S in the digested sludge slurry was 2.85 wt % S, indicating 99% of the S is soluble. The excellent agreement between the ICP-AES and IC values gives us a high degree of confidence in these data. The S content can be expected to decrease sharply when the supernate for the combined sludges is decanted during sludge washing. The sharp increase in S between the first two samples is not completely unexpected. Hobbs and Karraker[9] have shown in synthetic waste supernate that the concentration of Na_2SO_4 increases as the NaOH concentration decreases. The initial sample was 9.3M Na with measured free hydroxide of 0.66M, whereas the second sample was 2.1M Na with measured free hydroxide of 0.18M. Correspondingly, the soluble sulfate concentration increased from 0.08M to 0.20M.

Tank 11 was expected to contribute significant levels of Hg. Early reports[10,11] on a core sample of Tank 11 indicated on the order of 4 – 4.5 wt% Hg. In the slurried Tank 11 – Sample 2, the CV-AA measured Hg value was 0.98 wt % and that from ICP-MS was 1.00 wt %, excellent agreement for the two independent measurements on separate sets of three samples. Following the first transfer from Tank 11 to Tank 51, mounds of sludge solids were observed in Tank 11. The second transfer following slurry operations in Tank 11 has not yet occurred, so it is unclear as to whether or not the settled solids have been completely suspended and the second sample was a representative sample of the total solids. The previous results for Hg when compared with the current results would suggest that not all of the settled solids present in Tank 11 have yet been suspended.

Table 3-1 summarizes the Tank 11 and SB4 projected elemental composition (non-zero values) based upon Waste Characterization System (WCS) data washed to 1.0M Na.[12]

Table 3-1. WCS Estimated Sludge Weight Percent Elementals for Tank 11 and SB4

Element	Tank 11	SB4	Element	Tank 11	SB4
Al	20.21	10.83	Mn	2.5	4.92
Ba	0.17	0.27	Na	9.89	9.33
Ca	2.22	1.63	Ni	0.31	6.53
Ce	0.39	0.27	Pb	0.16	0.12
Cr	0.21	0.24	Si	3.41	1.69
Cu	0.06	0.08	Th	0.13	0.06
Fe	17.37	19.71	U	0.2	10.75
K	0.16	0.14	Zn	0.05	0.12
La	0.15	0.11	Zr	0.39	0.37
Mg	0.27	0.21			

WCS levels of La found in Table 3-1, a fission product like the noble metals, pointed to an expected elevation in the noble metal concentrations for Tank 11. The lower La values, 0.011 wt% and 0.028 wt%, respectively for samples 1 and 2, support the expectation that the noble metal concentrations measured will be lower than anticipated. It may also suggest that not all of the noble metals have yet been suspended in Tank 11.

While the estimate is washed to 1.0 M Na, the second Tank 11 sample analyzed here had not been decanted of soluble solids and was still at the elevated Na levels noted above. With this in mind the ratio of forecast Fe to the remaining elements has been calculated in Table 3-2. Also calculated are the ratio of actual Fe to the same set of elements for which there was a WCS forecast. Elements have been excluded from the table if they were either forecast but not measured for Tank 11 – Sample 2 (Cs, Nb, Y), measured in the sample but not forecast (Cd, Gd, Hg, P, S, Sb, Sn, Sr), or had measured values less than the instrumental detection limit (B, Cd, Li, Sn). For comparison, Table 3-2 also gives the ratios based upon two earlier characterizations of a single, archived sample of unmixed Tank 11 solids.

Table 3-2. Comparison of WCS Forecast and Actual Iron Ratios for Tank 11 Analyses

Element	Forecast Ratio Fe:Element	Sample 2 Ratio Fe:Element	Sample 1 Ratio Fe:Element	1998 Ratio[10] Fe:Element	1991 Ratio[11] Fe:Element
Al	1.2	3.7	2.4	6.9	6.6
Ba	0.01	0.01	0.01	0.04	NA
Ca	0.1	0.1	0.1	0.1	0.04
Ce	0.02	0.04	0.01	0.2	NA
Cr	0.01	0.01	0.02	0.03	0.01
Cu	0.003	0.007	0.004	0.05	0.01
Fe	1.0	1.0	1.0	1.0	1.0
K	0.01	0.03	0.04	NA	0
La	0.01	0.01	0.003	0.1	NA
Mg	0.02	0.02	0.02	0.04	0.1
Mn	0.1	0.2	0.2	0.5	0.5
Mo	0.00	0.01	0.005	0.03	NA
Na	0.6	10	8.5	2.6	2.2
Ni	0.02	0.2	0.1	0.3	0.2
Pb	0.01	NA	NA	0.2	NA
Si	0.2	0.5	0.04	0.2	0.1
Th	0.01	0.02	0.001	NA	0.001
U	0.01	0.4	0.003	NA	0.02
Zn	0.003	0.006	0.005	0.007	NA
Zr	0.02	0.02	0.01	NA	NA

As expected, soluble species such as K and Na have actual ratios to Fe which are greater than anticipated for the final washed sludge. Note that the increased ratios of the insoluble species Al, Ni, and U would not be expected to change enough upon washing to come into line with the WCS based forecast. The Ni ratio is of the same magnitude as that found in the earlier characterizations, while the U ratio is an order of magnitude higher.

A comparison of the fission yield ratios for Ru:Rh, Ru:Pd, and Ru:Ag with those measured for various Tank 11 samples is provided in Table 3-3. The ratios are based upon Ru due to its relatively high concentration in the sludge as compared with the other noble metals. The Ru:Rh ratios agree rather well across the various samples, while the Ru:Pd and Ru:Ag ratios differ significantly from the fission yield ratios.

There was a significant increase in the amount of measured Ag in the second Tank 11 sample, but the fission product noble metal concentrations were generally nearly the same or lower than observed in the first sample. This observed difference in the noble metal behavior is not unexpected when one considers their sources. The Ag is natural Ag, originating from Ag saddles used in the dissolvers to scavenge radioactive iodine, while the other noble metals are fission products of U-235. The Ag and the fission product noble metals relative concentrations are not linked to one another.

Some Pd may have been transferred to salt tanks possibly due to its minor solubility in caustic[13]. Therefore, the increased ratio of Ru:Pd seen in Table 3-3 is not unexpected.

Table 3-3. Fission Yield Ratios and Measured Noble Metal Ratios in Various Tank 11 Samples

Ratio	Fission Yield	Sample 2	Sample 1	98 Analysis[10]	91 Analysis[11]
Ru:Rh	3.4	5.3	4.5	4.8	4.5
Ru:Pd	6.7	66	29	33	20
Ru:Ag	366	20	47	2.0	141

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4.0 CONCLUSIONS

The following conclusions can be drawn from the data collected on the two samples of Tank 11 received by SRNL:

- The concentration of sulfur in Sample 2 is high and 99% of the sulfur is soluble. The increase in soluble sulfur is likely due to the decrease in NaOH concentration. The significance of this increase cannot be determined at this point. The second transfer from Tank 11 needs to occur and volume estimates made.
- The lower than anticipated mercury values tend to support the suspicion that all of the sludge solids in Tank 11 were not suspended at the time the second Tank 11 sample was pulled for analysis. The amount of mercury present in SB4 will impact the acid addition needs during SRAT processing.
- While still low relative to SB2 and SB3, the level of U is higher than forecast by WCS.
- Noble metal concentrations in these two samples are not as high as previously anticipated based upon earlier direct measurements of the noble metals[11] and WCS predicted La values in unmixed Tank 11 solids. It may be that not all of the solids were suspended at the time the second Tank 11 sample was pulled for analysis. The noble metal content will have a direct impact on the amount of hydrogen produced during SRAT processing.

5.0 RECOMMENDATIONS/PATH FORWARD

Once all bulk waste transfer operations from Tank 11 to Tank 51 have been completed, it is recommended that a sample be taken from Tank 51. The analysis of this sample will provide better projections of SB4 content and allow decisions to be made on washing as well as simulant and glass studies in support of qualification for DWPF processing.

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