CHARACTERIZATION OF TANK 18F WALL AND SCALE SAMPLES

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Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

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LIST OF ACRONYMS

ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
PUREX	Plutonium Uranium Reduction Extraction
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TTA	Thenoyltrifluoroacetone
XRD	X-ray Diffraction
AS	Alpha Spectroscopy
GS	Gamma Spectroscopy
CR/GS	Cesium Removal/Gamma Spectroscopy
S/ICP-MS	Separation/ Inductively Coupled Plasma-Mass Spectroscopy
LSC	Liquid Scintillation Counting
PuTTA	Plutonium by Thenoyltrifluoroacetone extraction
SGRS	Special Gamma Ray Spectrometry
PIPS	Passivated Implanted Planar Silicon
AMP	Aminomethylphosphonic acid
TEVA	Tetravalent actinide
DAAP	Diamyl amylphosphonate

SUMMARY

Samples from the wall of Tank 18F were obtained to determine the associated source term using a special wall sampling device. Two wall samples and a scale sample were obtained and characterized at the Savannah River National Laboratory (SRNL).

All the analyses of the Tank 18F wall and scale samples met the targeted detection limits. The upper wall samples show ~2X to 6X higher concentrations for U, Pu, and Np on an activity per surface area basis than the lower wall samples. On an activity per mass basis, the upper and lower wall samples show similar compositions for U and Pu. The Np activity is still ~2.5X higher in the upper wall sample on a per mass basis.

The scale sample contains 2-3X higher concentrations of U, Pu, and Sr-90 than the wall samples on an activity per mass basis. The plutonium isotopics differ for all three wall samples (upper, lower, and scale samples). The Pu-238 appears to increase as a proportion of total plutonium as you move up the tank wall from the lowest sample (scale sample) to the upper wall sample.

The elemental composition of the scale sample appears similar to other F-Area PUREX sludge compositions. The composition of the scale sample is markedly different than the material on the floor of Tank 18F. However, the scale sample shows elevated Mg and Ca concentrations relative to typical PUREX sludge as do the floor samples.

1.0 INTRODUCTION

Tank 18F has undergone several cleaning schemes. One of the post-cleaning analyses involves samples drilled from the inner vertical wall of the carbon steel liner of the tank. Tank 18F (a Type IV tank) is made of concrete with a carbon steel liner.

The objective of the work is therefore to analyze samples from the inner vertical wall of Tank 18F to evaluate the residual radionuclides which may have accumulated or embedded within several thousandth of an inch (mils) from the wall's surface due to corrosion of the carbon steel material. A special wall sampling device was fabricated and tested at Savannah River National Laboratory (SRNL) to facilitate sampling the carbon steel wall of the tank and recover the corrosion products/scale/adhered waste.[1]

The work was performed according to the "Task Technical Quality Assurance and Characterization Plan (TTQAP) for Tanks 18F and 19F Solid Fraction Analysis - 2008".[2] The Task Technical Quality Assurance and Characterization Plan was derived from "Technical Task Request (TTR) for Laboratory Analysis for Tanks 18 and 19 Closure Samples".[3]

2.0 SAMPLE DESCRIPTION, PREPARATION, AND ANALYSIS

2.1 SAMPLE DESCRIPTION

The Tank 18F wall samples were received at SRNL and moved to a radiological hood. Each sampling head was opened and photographed in a radiological hood. Figures 2.1 through 2.5 show examples of the holes drilled in the tank wall and the filter pads inside each sampler head used to capture the material drilled from the wall. Sample Tk 18-1 was obtained from two sampling events made in the tank wall ~17 ft above the bottom of the tank on 9-3-09. The extremity rate from the material on the filter shown in Figure 2.2 was ~2000 mrem/hr. Sample SP4 shown in Figure 2.4 was obtained from two sampling events ~10-12 ft above the tank floor on 9-24-09. The extremity rate for the material on the filter of SP4 was ~4000 mrem/hr. Sample Tk 18-2 was designated as the scale sample since it was obtained from areas on the tank wall covered with a thick layer of material. The scale sample was obtained from four sampling events ~6-7 ft above the tank floor on 9-2-09. The extremity rate for the material on the filter of sample Tk 18-2 was ~2000 mrem/hr. Table 2-1 summarizes the information on the three Tank 18F wall samples.

Figure 2.1 Photograph of Hole Drilled in Tank 18F Wall for Sample Tk 18-1

Figure 2.2 Photograph of Tank 18F Wall Sample Tk 18-1

Figure 2.3 Photograph of Hole Drilled in Tank 18F for Wall Sample SP4

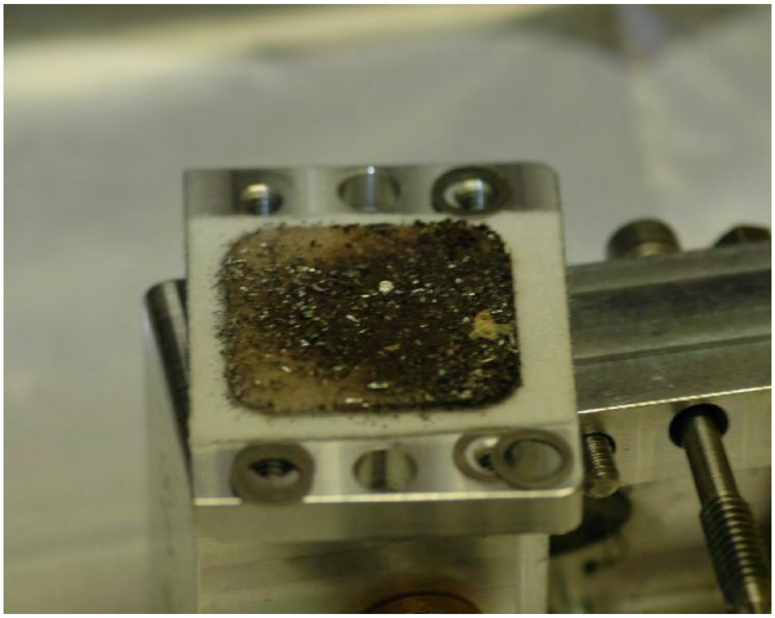

Figure 2.4 Photograph of Tank 18F Wall Sample SP4

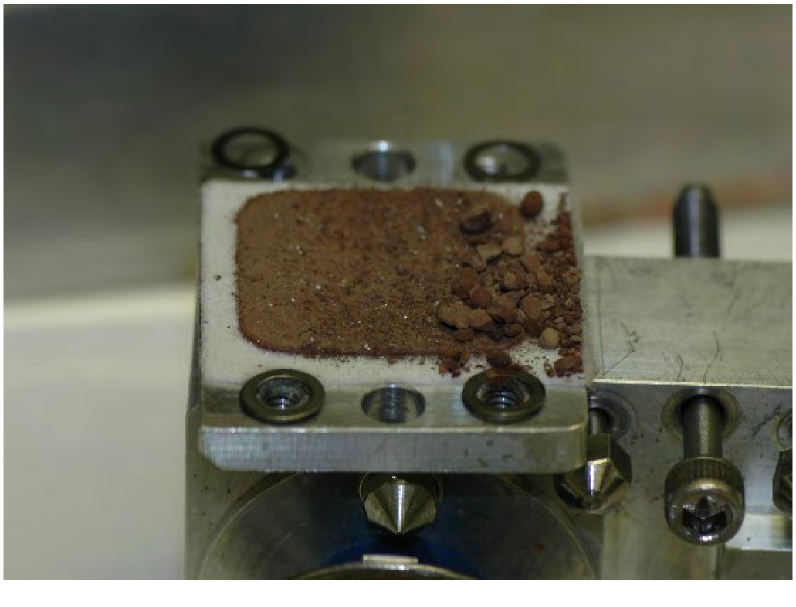

Figure 2.5 Photograph of Tank 18F Scale Sample Tk 18-2

Table 2-1. Characteristics of the drilled holes and sampled materials.

Sample Description	Height from Tank Floor, (ft)	Number of Holes Drilled	Estimated Surface Area (ft^2)	Mass of Drilled Material,[a] (g)	Radiation Rate, extremity (mrem/hr)
Tk 18-1 (Upper Wall Sample)	~17	2	1.36E-03	1.421	2000
SP4 (Lower Wall Sample)	~10-12	2	4.37E-04	0.1967	4000
Tk 18-2 (Scale Sample)	~6-7	5	Not Determined	1.1845	2000

[a] Obtained by subtracting average mass of typical filters from the mass of sample plus actual filter used.

2.2 SAMPLE PREPARATION AND ANALYSIS

Because of the inherent risk of cross-contamination when working in the shielded cells environment, and the expected lower dose rates of the samples, the sample preparations were conducted in a radiological hood in SRNL.

The two Tank 18F wall samples were digested according to procedure L16.1, ADS-2226. The sample plus filter from each wall sampler was transferred to a glass jar with a known weight. The exact weight of the filters used in each individual sampler was not known. Rather, the average weight of the filters used in the samplers was provided by the Engineering Development Lab which they determined to be 0.614 g. The average weight of the filters was subtracted from the weight of the sample plus filter. The subtracted weight of sample was 1.421 g for sample Tk 18-1 and 0.1967 g for SP4. In each case, the entire sample was subjected to an aqua regia digestion. The aqua regia digestions were performed using sealed Teflon® digestion vessels, 18 mL HCl, 6 mL HNO_3, 0.5 mL HF, and were heated for a period of no more than 4 hours at 115 ºC. The digestions were then cooled and diluted with de-ionized water to a final volume of 100 mL for sample Tk 18-1 and 50 mL for SP4. A blank sample was prepared concurrently with the Tank 18 wall samples. The blank sample contained only the acids used in digesting the samples and was diluted with de-ionized water to a final volume of 100 mL. By visual inspection, each of the samples dissolved completely. A tiny amount of oily residue was noticeable on the walls of each digestion vessel and was attributed to the lubricating grease used in the wall samplers.

The Tank 18 Scale sample (Tk 18-2) was subjected to two different digestion methods according to procedures L16.1, ADS-2226 (Aqua Regia Digestion) and ADS-2502 (Peroxide Fusion Digestion). The sample plus filter was transferred to a glass jar with a known weight. After subtracting the weight of the filter, the sample weight was 1.1845 g. A sample size of 0.2516 g was subjected to an aqua regia digestion. The digestion was performed using a sealed Teflon® digestion vessel, 9 mL HCl, 3 mL HNO_3, 0.5 mL HF, and was heated for a period of no more than 4 hours at 115 ºC. The sample was diluted to a final volume of 100 mL. A tiny amount of oily residue was noticeable on the walls of the digestion vessel and was attributed to the lubricating grease used in the wall samplers. A sample size of 0.2542 g was subjected to an alkali fusion digestion. The digestion was performed in a zirconium crucible using a mixture of 1.0 g NaOH and 1.5 g Na_2O_2. The mixture and sample were heated to 675 ºC for 10 minutes in a muffle furnace. The sample was then cooled, and dissolved in de-ionized water. Nitric acid (25 mL) was used to acidify the sample and finish rinsing any residue from the crucible. The sample was diluted to a final volume of 100 mL. A blank sample was prepared concurrently with the Tank 18 Scale sample for each digestion. The blank sample contained only the reagents used in digesting the samples and was diluted with de-ionized water to a final volume of 100 mL. By visual inspection, each of the samples dissolved completely.

Aliquots of the digested samples were sent to Analytical Development for analysis by the methods described in Appendix A.

3.0 RESULTS AND DISCUSSION

The results shown in the following tables are the product of a single digestion with no replicates and therefore, the sampling and analytical uncertainties cannot be determined. Additionally, the surface area values for the samples are estimated from photographs and the mass of sample obtained is based on an assumed weight for the filter media. For these reasons, the uncertainty associated with the reported values in the tables is likely to be quite high.

The abbreviations used for the analytical methods in the following data tables are listed below. More detail descriptions of the analytical methods are contained in the appendix (Section 7.0).

AS - Alpha Spectroscopy
GS - Gamma Spectroscopy
CR/GS – Cesium Removal/Gamma Spectroscopy
ICP-MS - Inductively Coupled Plasma-Mass Spectroscopy
S/ICP-MS – Separation/ Inductively Coupled Plasma-Mass Spectroscopy
ICP-ES - Inductively Coupled Plasma-Emission Spectroscopy
LSC - Liquid Scintillation Counting
PuTTA - Plutonium by Thenoyltrifluoroacetone extraction
SGRS - Special Gamma Ray Spectrometry for Np-237

3.1 TANK 18F WALL SAMPLES

For the Tank 18F Wall samples the primary objective was to dissolve all of the material contained in the sampler to determine the activity per unit area on the tank wall. The surface area estimates for each sample were obtained from a recent report on the fabrication and testing of the wall sampling device.[1] The estimated surface area of the upper tank wall sample (Tk 18-1) was 1.36E-03 ft^2 while the lower wall sample (SP4) was estimated at 4.37E-04 ft^2.

Table 3-1 shows the results from the analysis of the wall samples obtained from Tank 18F in terms of activity per unit surface area of the sample. The results from the analysis of the two wall samples indicate the upper wall sample generally shows ~2X to 6X higher concentrations for U, Pu, and Np as a function of surface area. The upper wall sample also shows ~50% higher Sr-90 and 25% lower Cs-137 than the lower wall sample. Some of the difference in the activity measured in the samples can be attributed to the larger mass of material obtained in the upper wall sampling event. The difference in surface area between the two samples is approximately a factor of 3, however the difference in the estimated mass of material from each sample differs by a factor of ~7.

Table 3-2 shows the same results on an activity per unit mass basis. On a mass basis, the difference in composition between the upper and lower wall samples largely disappears for the U and Pu. The differences of 20-40% fall within the expected sampling and analytical uncertainty. However, the Np is still ~2.5X higher in the upper wall sample and the lower wall sample shows higher fission product concentrations than the upper wall sample. The Cs-137 activity in the lower wall sample is an order of magnitude higher than the upper wall sample. The Pu isotopics differ slightly between the two wall samples. The plutonium in the upper wall sample has 5.9% Pu-238 while the lower samples has 2.8% Pu-238 on a mass basis.

3.2 TANK 18F SCALE SAMPLE

Table 3-2 also shows the results of the analysis of the scale sample obtained from Tank 18F (Tk 18-2). The results indicate the scale sample contains 2-3X higher concentrations of U, Pu, and Sr-90 than the wall samples. The scale sample also contains ~3X higher Am-241 and ~5X higher Co-60 than the wall samples.

Table 3-3 shows the average data again along with the results from the two digestion methods used to prepare the samples. The percent relative standard deviations show generally good agreement between the two digestion methods. The values in the table in units of µg/g can be converted to a wt% solids basis by dividing the values in the table by 1E+04. The elemental composition of the scale sample appears similar to other F-Area PUREX sludge compositions. The sample contains 26.0 wt% Fe, 10.3 wt% Al, 2.80 wt% Mg, 2.53 wt% Ca, 2.04 wt% U, 1.88 wt% Mn, and 1.13 wt% Si. This composition compares well with other F-Area sludge analyses, such as Tanks 5F and 8F,[4,5,6] although the Mg and Ca appear somewhat elevated in the scale sample. The composition for the scale sample is markedly different from the material on the floor of Tank 18F that is high in aluminum and low in iron.[7] However, the floor samples also show elevated Mg and Ca concentrations relative to typical PUREX sludge. The likely source of the elevated Mg and Ca concentrations in both the floor samples and the scale sample is the zeolite ion exchange material transferred from Tank 19F.[8]

The X-ray Diffraction (XRD) scan of the scale sample, shown in Figure 3.1, lends additional support to the scale being dried sludge versus corrosion products. Although the presence of hematite would be expected from corrosion of the steel tanks, the presence of gibbsite would not be expected. Based on the elemental composition of the scale sample (Table 3-3), the aluminuosilicates shown in the XRD make up only a small portion of the scale material since the silicon content of the sample is only 1.13 wt%.

The plutonium isotopics of the scale differ from both of the wall samples. The plutonium in the scale sample contains 0.5% Pu-238 versus the 5.9% and 2.8% Pu-238 of the upper and lower wall samples on a mass basis, respectively. Since the scale sample was obtained from lower on the tank wall than either of the wall samples, the Pu-238 appears to increase as a proportion of total plutonium as you move up the tank wall.

Table 3-1. Tank 18F Wall Sample Results per Unit Area

Isotope	Analytical Method	Wall Sample Target Detection Limit Ci/ft^2	Tk 18-1 (Upper Wall) Ci/ft^2	SP4 (Lower Wall) Ci/ft^2
U-233	S/ICP-MS	1.98E-05	1.93E-06	7.08E-07
U-234	S/ICP-MS	1.27E-05	9.13E-06	3.50E-06
U-235	S/ICP-MS	1.16E-08	1.11E-07	4.55E-08
U-236	S/ICP-MS	1.32E-07	4.17E-07	1.36E-07
U-238	ICP-MS	2.92E-07	2.57E-06	1.11E-06
Np-237	SGRS	1.82E-06	4.21E-06	6.38E-07
Pu-238	PuTTA	1.35E-05	5.70E-02	9.12E-03
Pu-239	S/ICP-MS	4.24E-04	3.03E-03	1.06E-03
Pu-240	S/ICP-MS	1.54E-03	8.09E-04	2.48E-04
Pu-241	PuTTA	1.76E-05	1.44E-02	3.47E-03
Pu-242	S/ICP-MS	2.64E-05	6.75E-07	1.73E-07
Pu-244	S/ICP-MS	Not Required	<4.3E-10	<1.9E-10
Cs-137	GS	No Target	7.65E-02	3.41E-01
Sr-90	LSC	Not Required	1.14E-01	7.67E-02
Co-60	GS	Not Required	2.47E-05	2.02E-05
Am-241	CR/GS	Not Required	2.53E-03	7.50E-04

Table 3-2. Tank 18F Wall and Scale Sample Results per Mass of Solids

Isotope	Analytical Method	Tk 18-1 (Upper Wall) Ci/g	SP4 (Lower Wall) Ci/g	Tk 18-2 Scale (Average) Ci/g
U-233	S/ICP-MS	1.85E-09	1.57E-09	2.03E-09
U-234	S/ICP-MS	8.74E-09	7.78E-09	8.14E-09
U-235	S/ICP-MS	1.06E-10	1.01E-10	2.79E-10
U-236	S/ICP-MS	3.99E-10	3.03E-10	3.32E-10
U-238	ICP-MS	2.46E-09	2.47E-09	6.84E-09
Np-237	SGRS	4.03E-09	1.42E-09	4.09E-09
Pu-238	PuTTA	5.45E-05	2.03E-05	1.51E-05
Pu-239	S/ICP-MS	2.90E-06	2.36E-06	9.58E-06
Pu-240	S/ICP-MS	7.74E-07	5.51E-07	2.16E-06
Pu-241	PuTTA	1.35E-05	6.87E-06	1.74E-05
Pu-242	S/ICP-MS	6.46E-10	3.84E-10	1.80E-09
Pu-244	S/ICP-MS	<4.1E-13	<4.2E-13	<7.7E-13
Cs-137	GS	7.32E-05	7.58E-04	8.72E-05
Sr-90	LSC	1.09E-04	1.70E-04	2.70E-04
Co-60	GS	2.36E-08	4.49E-08	1.12E-07
Am-241	CR/GS	2.43E-06	1.67E-06	7.70E-06

Table 3-3. Tank 18F Scale Sample Results per Mass of Solids

Isotope	Analytical Method	Tk 18-2 Scale Aqua Regia Ci/g	Tk 18-2 Scale Peroxide Fusion Ci/g	Average Ci/g	%RSD
U-233	S/ICP-MS	2.07E-09	1.99E-09	2.03E-09	3%
U-234	S/ICP-MS	8.51E-09	7.76E-09	8.14E-09	7%
U-235	S/ICP-MS	2.81E-10	2.78E-10	2.79E-10	1%
U-236	S/ICP-MS	3.43E-10	3.21E-10	3.32E-10	5%
U-238	ICP-MS	6.72E-09	6.96E-09	6.84E-09	2%
Np-237	SGRS	3.41E-09	4.76E-09	4.09E-09	23%
Pu-238	PuTTA	1.77E-05	1.25E-05	1.51E-05	24%
Pu-239	S/ICP-MS	9.26E-06	9.89E-06	9.58E-06	5%
Pu-240	S/ICP-MS	2.09E-06	2.23E-06	2.16E-06	4%
Pu-241	PuTTA	1.76E-05	1.72E-05	1.74E-05	1%
Pu-242	S/ICP-MS	1.77E-09	1.83E-09	1.80E-09	2%
Pu-244	S/ICP-MS	<7.7E-13	<2.6E-12	-	-
Cs-137	GS	9.77E-05	7.66E-05	8.72E-05	17%
Sr-90	LSC	2.17E-04	3.24E-04	2.70E-04	28%
Co-60	GS	1.04E-07	1.20E-07	1.12E-07	10%
Am-241	CR/GS	7.39E-06	8.02E-06	7.70E-06	6%

Table 3-3. Tank 18F Scale Sample Results per Mass of Solids (Continued)

Element	Analytical Method	Tk 18-2 Scale Aqua Regia µg/g	Tk 18-2 Scale Peroxide Fusion µg/g	Average µg/g	%RSD
Ag	ICP-ES	8.86E+01	<1.1E+02	-	-
Al	ICP-ES	9.98E+04	1.06E+05	1.03E+05	4%
B	ICP-ES	<3.8E+01	<3.6E+02	-	-
Ba	ICP-ES	6.78E+02	6.45E+02	6.62E+02	4%
Be	ICP-ES	<1.5E+01	<1.0E+01	-	-
Ca	ICP-ES	2.58E+04	2.47E+04	2.53E+04	3%
Cd	ICP-ES	3.15E+03	3.34E+03	3.25E+03	4%
Ce	ICP-ES	<8.2E+01	<3.8E+02	-	-
Cr	ICP-ES	1.60E+03	1.56E+03	1.58E+03	2%
Cu	ICP-ES	5.20E+02	4.04E+02	4.62E+02	18%
Fe	ICP-ES	2.83E+05	2.37E+05	2.60E+05	13%
Gd	ICP-ES	<6.0E+01	1.14E+02	-	-
K	ICP-ES	<1.1E+02	3.81E+03	-	-
La	ICP-ES	<4.8E+00	<4.8E+01	-	-
Li	ICP-ES	1.46E+02	1.43E+02	1.45E+02	1%
Mg	ICP-ES	2.65E+04	2.94E+04	2.80E+04	7%
Mn	ICP-ES	1.81E+04	1.94E+04	1.88E+04	5%
Mo	ICP-ES	<2.1E+01	<2.1E+02	-	-
Na	ICP-ES	1.35E+04	-	-	-
Ni	ICP-ES	2.06E+03	2.36E+03	2.21E+03	10%
P	ICP-ES	3.21E+03	1.28E+03	2.25E+03	61%
Pb	ICP-ES	8.12E+02	6.57E+02	7.35E+02	15%

Convert to a wt% solids basis by dividing the numbers in the table by 1E+04

Table 3-3. Tank 18F Scale Sample Results per Mass of Solids (Continued)

Element	Analytical Method	Tk 18-2 Scale Aqua Regia µg/g	Tk 18-2 Scale Peroxide Fusion µg/g	Average µg/g	%RSD
S	ICP-ES	<1.1E+03	<5.2E+03	-	-
Sb	ICP-ES	<1.0E+02	<1.0E+03	-	-
Si	ICP-ES	-	1.13E+04	1.13E+04	-
Sn	ICP-ES	6.24E+01	<5.0E+02	-	-
Sr	ICP-ES	3.42E+02	3.54E+02	3.48E+02	2%
Ti	ICP-ES	2.26E+02	2.10E+02	2.18E+02	5%
U	ICP-ES	1.99E+04	2.08E+04	2.04E+04	3%
V	ICP-ES	<3.3E+01	<4.8E+01	-	-
Zn	ICP-ES	5.55E+02	6.24E+02	5.90E+02	8%
Zr	ICP-ES	1.06E+02	-	-	-

Convert to a wt% solids basis by dividing the numbers in the table by 1E+04

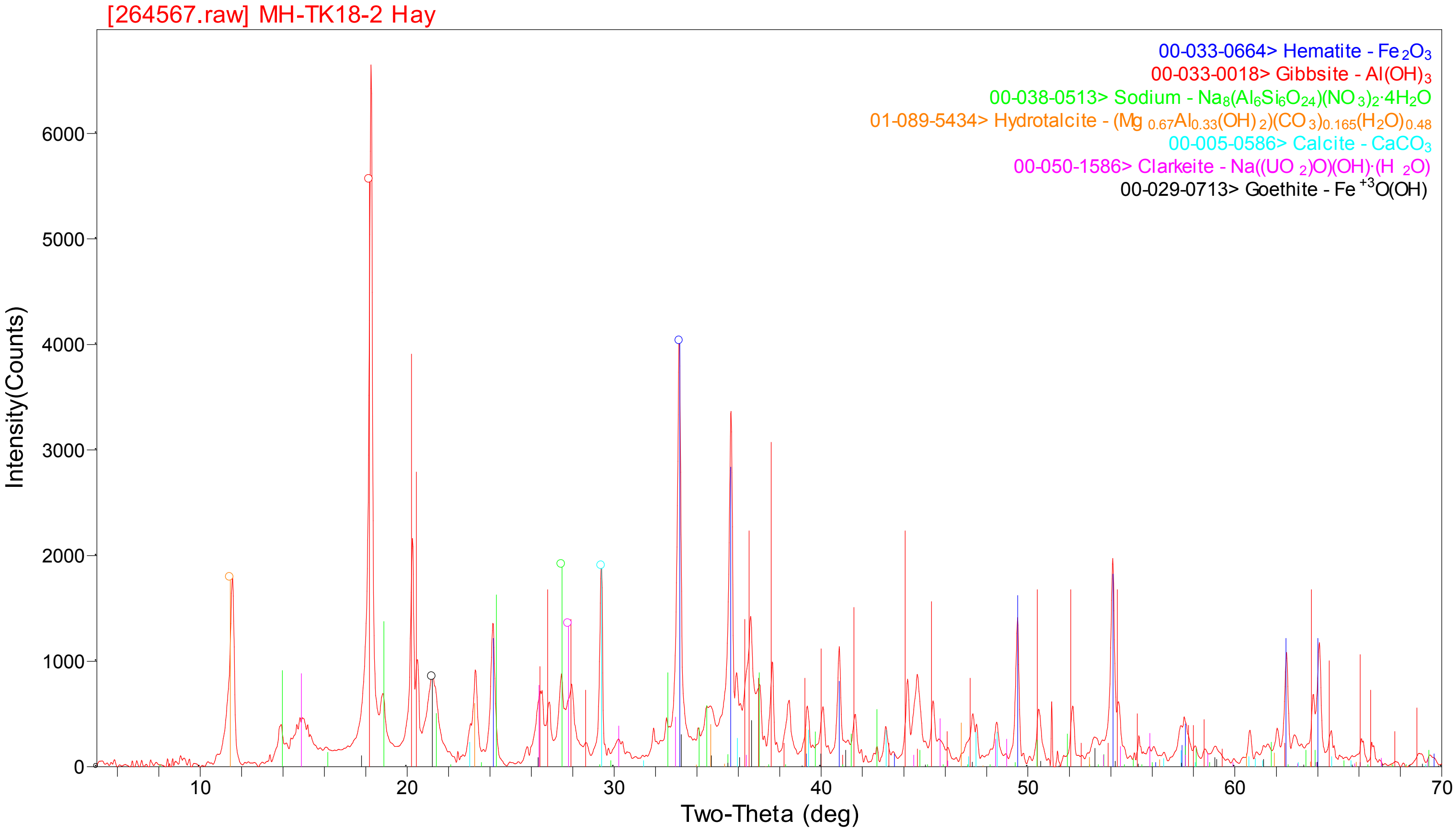

Figure 3.1 XRD of the Tank 18F Scale Sample

4.0 CONCLUSIONS

All the analyses of the Tank 18F wall and scale samples met the targeted detection limits. The upper wall samples show ~2X to 6X higher concentrations for U, Pu, and Np on an activity per surface area basis. On an activity per mass basis, the upper and lower wall samples show similar compositions for U and Pu. The Np activity is still ~2.5X higher in the upper wall sample on a per mass basis.

The scale sample contains 2-3X higher concentrations of U, Pu, and Sr-90 than the wall samples on an activity per mass basis. The plutonium isotopics differ for all three wall samples (upper, lower, and scale samples). The Pu-238 appears to increase as a proportion of total plutonium as you move up the tank wall from the lowest sample (scale sample) to the upper wall sample.

The elemental composition of the scale sample appears similar to other F-Area PUREX sludge compositions. The composition of the scale sample is markedly different than the material on the floor of Tank 18F. However, the scale sample shows elevated Mg and Ca concentrations relative to typical PUREX sludge as do the floor samples.

5.0 ACKNOWLEDGEMENTS

Many people in the organizations below at SRNL contributed to the successful completion of this work. Sincere thanks to all the people who provided assistance.

Environmental Management
Analytical Development
Engineering Development Laboratory
Radiological Protection

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7.0 APPENDIX A

SUMMARY DESCRIPTION OF ANALYTICAL METHODS

Cs-137/Co-60

Aliquots from the dissolutions of the solids from the sample were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure Cs-137 and Co-60.

Sr-90

Aliquots from the dissolutions of the solids from the sample were spiked with an elemental strontium carrier. The strontium species were extracted from the matrix using a crown-ether-based solid phase extractant. Sr-90 concentrations were measured by liquid scintillation analysis. Elemental strontium carrier yields were measured by neutron activation analysis, and were used to correct the Sr-90 analyses for any strontium losses from the radiochemical separations.

Pu-238, 239/240, 241

Aliquots from the dissolutions of the solids from the sample were spiked with Pu-236 tracer. The plutonium was extracted from the matrix using Thenoyltrifluoroacetone (TTA) following a series of oxidation-state adjustments. The TTA extracts were mounted on stainless steel counting plates and counted for Pu-238, Pu-239, and Pu-240 using Passivated Implanted Planar Silicon (PIPS) detectors. Each separation was traced based on the Pu-236 recovery. Aliquots of sample were also subjected to Cs-removal with Bio-Rad Aminomethylphosphonic acid (AMP) resin and extracted using tetravalent actinide (TEVA) columns. The Pu-containing extracts were measured by liquid scintillation analysis to determine Pu-241 concentration.

Am-241

Aliquots from the dissolutions of the solids from the sample were subjected to a Cs-removal process utilizing Bio Rad AMP-1 resin. The Cs-removed solution was analyzed by coaxial high purity germanium spectrophotometers to measure the gamma-emitting radionuclide.

Np-237

Aliquots from the dissolutions of the solids from the sample were analyzed by low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers.

U Separation for MS

To lower detection limits for U-233, U-234, and U-236 from the ICP-MS analyses on the dissolutions of the Tank 18 Wall and Scale samples, aliquots of the dissolutions were purified with a Diamyl amylphosphonate (DAAP) based solid phase extraction. The purified aliquots were analyzed by the ICP-MS to measure U-233/U-238, U-234/U-238, and U-236/U-238 mass ratios. Those ratios were applied to U-238 concentrations measured by the ICP-MS directly on aliquots of dissolution to quantify U-233, U-234, and U-236 concentrations.

Pu Separation for MS

To lower detection limits for Pu-242 and Pu-244 from the ICP-MS analyses on the dissolutions of the Tank 18 Wall and Scale samples, aliquots of dissolutions were purified with a quaternary amine based solid phase extraction. The purified aliquots were analyzed by the ICP-MS to measure Pu-242/Pu-239, and Pu-244/Pu-239 mass ratios. Those ratios were applied to Pu-239/240 concentrations measured by alpha counting directly on aliquots of dissolution to quantify Pu-242 and Pu-244 concentrations.

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

Samples were run concurrently with a laboratory control standard containing V, Co, As, Sr, Mo, Ru, Ag, Cd, Sb, Cs, Ba, La, Eu, Ho, Yb, Tl, Pb, Th, and U. This laboratory control standard provided a mass response covering most of the mass range of interest. The following describes the calculation of the analytes of interest from the mass values assuming sufficient concentrations to generate a mass response:

^{233}U	mass 233
^{234}U	mass 234
^{235}U	mass 235
^{236}U	mass 236
^{238}U	mass 238
^{237}Np	mass 237
^{239}Pu	mass 239
^{240}Pu	mass 240
^{242}Pu	mass 242. Assumes no ^{242}Cm present in sample.

Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES)

Aliquots from the dissolutions of the solids from the scale sample were diluted as necessary to bring the analytes within the instrument range. A scandium internal standard is added to all samples after dilution at a concentration of 2 mg/L. The instrument was calibrated daily with a blank and two standards (5 and 10 mg/L NIST traceable multi-element standards in dilute acid). Background and internal standard correction were applied to the results.