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Analysis of the Tank 5F Final Characterization Samples-2011 (U)

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DISCLAIMER

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LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
1.0	Updated the ICPES elemental values reported for Tank 5F composite sample number 3, run 2, in Table 7 page 23. Revision of the report was necessary to correct for elemental metal concentration transcription error affecting original data.	**8/03/12**
1.0	Added AD sample tracking numbers (LIMS #) on pages 33 and 34 as appendix A-0 and cited appendix A-0 in the result section of the report in paragraph 1.	**8/03/12**
1.0	Re-oriented Figure 2 and modified it to change the sampling notation on the southeast corner consistent with the revision to the TTR.	**8/03/12**
1.0	Typo in appendix B was corrected.	**8/03/12**
1.0	Document formatting was performed in different sections of the report.	**8/03/12**
1.0	Updated references to the following document recent versions: (1) Technical Task Request number HLE-TTR-2010-004, Rev 7. (2) Task Technical and Quality Assurance Plan for the Analysis of the Tank 5F and Tank 6F Final Characterization Samples-SRNL-RP-2010-01695, Rev. 1 and (3) Tank 5 Sampling and Analysis Plan-SRR-LWE-2010-00285, Rev. 1, Nov. 15, 2010.	**8/03/12**

EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) was requested by SRR to provide sample preparation and analysis of the Tank 5F final characterization samples to determine the residual tank inventory prior to grouting. Two types of samples were collected and delivered to SRNL: floor samples across the tank and subsurface samples from mounds near risers 1 and 5 of Tank 5F. These samples were taken from Tank 5F between January and March 2011. These samples from individual locations in the tank (nine floor samples and six mound Tank 5F samples) were each homogenized and combined in a given proportion into 3 distinct composite samples to mimic the average composition in the entire tank. These Tank 5F composite samples were analyzed for radiological, chemical and elemental components. Additional measurements performed on the Tank 5F composite samples include bulk density and water leaching of the solids to account for water soluble species. With analyses for certain challenging radionuclides as the exception, all composite Tank 5F samples were analyzed and reported in triplicate.

The target detection limits for isotopes analyzed were based on customer desired detection limits as specified in the technical task request documents. SRNL developed new methodologies to meet these target detection limits and provide data for the extensive suite of components. While many of the target detection limits were met for the species characterized for Tank 5F, as specified in the technical task request, some were not met. In a few cases, the relatively high levels of radioactive species of the same element or a chemically similar element precluded the ability to measure some isotopes to low levels. The Technical Task Request allows that while the analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes. The isotopes whose detection limits were not met in all cases included the following: Al-26, Sn-126, Sb-126, Sb-126m, Eu-152 and Cf-249. SRNL, in conjunction with the plant customer, reviewed all these cases and determined that the impacts were negligible.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARG	Analyzed Reference Glass
AQR	Aqua Regia Digestions
DL	Detection limit: As used in mass spectrophotometer analyses or ICP-ES; three times the standard deviation of the blank measurements.
ICP-ES	Inductively Coupled Plasma–Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma–Mass Spectroscopy
LWO	Liquid Waste Operations
MDA	Minimum Detectable Activity: Minimum detectable activity is the value above which instrument signal can be considered real.
PF	Sodium Peroxide/Hydroxide Fusions
PMP	Polymethyl Pentane
PuTTA	Plutonium thenoyltrifluoro-acetone
UL	Upper limit: Activity observed but biased high due to spectral interference or blank contamination.
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request

1.0 INTRODUCTION

Savannah River Remediation (SRR) is preparing Tank 5F for closure. The Savannah River National Laboratory (SRNL) was requested by SRR to provide sample preparation and analysis of the Tank 5F final characterization samples to determine the residual tank inventory prior to grouting. In all, nine floor samples and six mound Tank 5F samples were provided by SRR. A photo image of some of the "as-received" Tank 5F samples is shown in Figure 1 (three mound samples and six floor samples). These Tank 5F samples were taken between January and March 2011 and made available to SRNL.

The types of Tank 5F samples collected and delivered to SRNL for analysis were floor samples across the tank and subsurface samples from mounds near risers 1 and 5. These two types of samples formed the basis for designing the three Tank 5F composite materials (Tank 5F- Composite sample # 1, Tank 5F- Composite sample # 2 and Tank 5F- Composite sample # 3). The volume of residual material in each of the Tank 5F region was obtained by SRR and this information was used to estimate the strata volumes in the tank. These strata volumes were converted into volumetric proportions, and subsequently to the mass of residual material to be obtained from each primary sample for each composite sample[1] Thus; each Tank 5F composite sample was derived from five individual Tank 5F materials as shown in Table 1.

The Tank 5F samples were analyzed in accordance with Technical Task Request number HLE-TTR-2010-004, Rev 7, and Task Technical and Quality Assurance Plan for the Analysis of the Tank 5F and Tank 6F Final Characterization Samples-SRNL-RP-2010-01695, Rev. 1 and Tank 5 Sampling and Analysis Plan-SRR-LWE-2010-00285, Rev. 1, November 15, 2010.

[1] B. Dean, "Tank 5 Composite Sample Volumetric Proportions," SRR-CWDA-2011-00067, Rev.1 April 20, 2011.

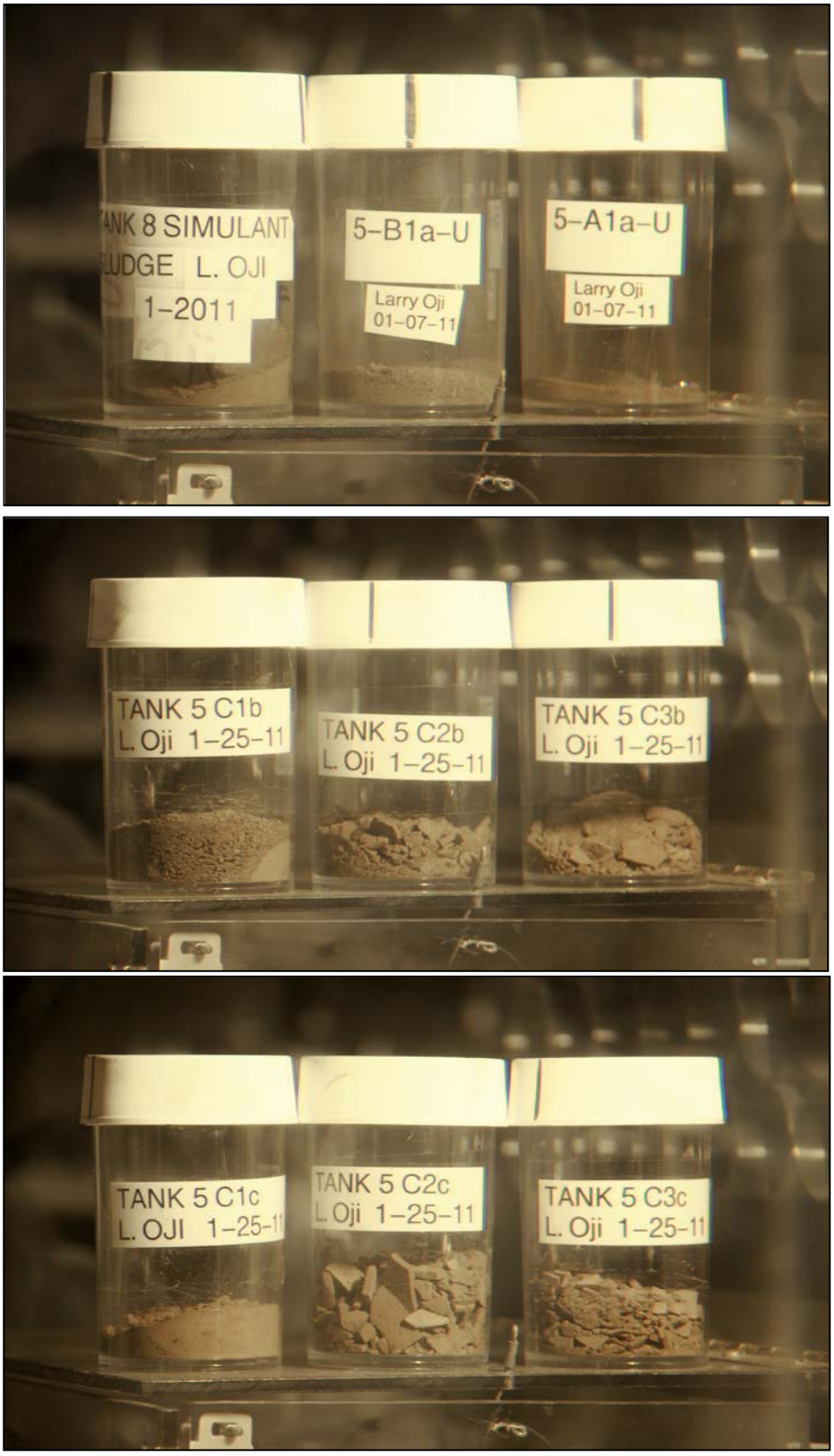

Figure 1. Photo images of "as-received" Tank 5F samples.

2.0 SAMPLING AND SAMPLE PREPARATION FOR CHARACTERIZATION

Tank 5F samples provided by SRR for characterization came from various locations in Tank 5F as shown in Figure 2 below [HLE-TTR-2010-004].

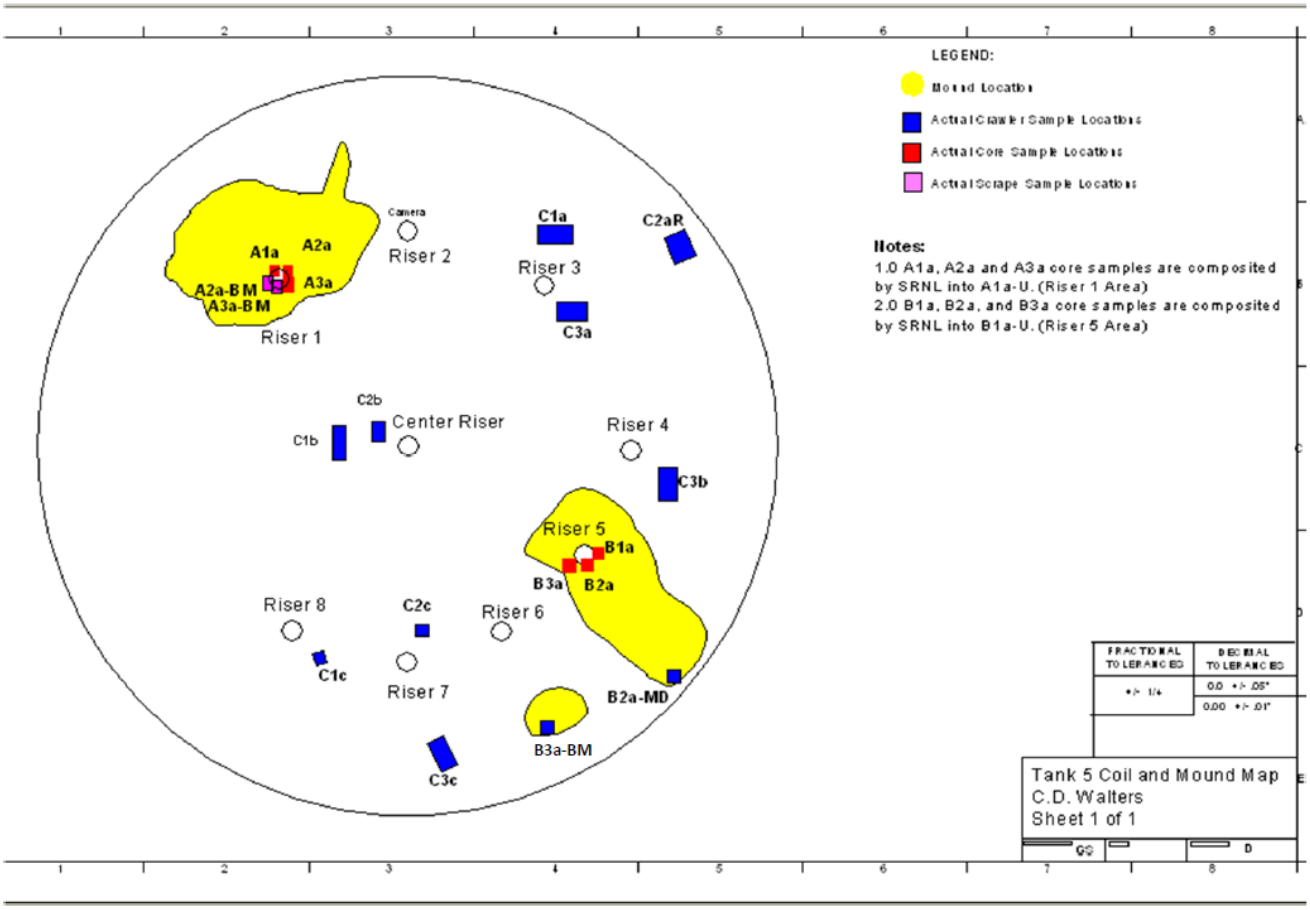

Figure 2. Tank 5F sample locations.

The last batch of Tank 5F sample containment bag received in March of 2011, when opened at SRNL shielded cell, was found to contain designated Tank 5F sample holder (5-B3a-BM) which was empty. However, some materials were found inside the secondary containment bag. The other sample container in the same containment bag (5-B2a-MD) contained some amount of Tank 5F materials. This information was brought to the attention of SRR. After their reviews, SRR concluded that both samples came from the same region of Tank 5F and issued a justification and approval for SRNL to use the material found in the secondary containment bag as sample material 5-B3a-BM (B. Dean, "Tank 5F samples 5-B2a-MD and 5-B3a-BM Justification" SRR-CWDA-2011-00100 Rev. 0, May19, 2011).

These Tank 5F samples were fairly dry and needed no further air drying in the shielded cells before preparation for compositing. The individual "as-received" materials were weighed and their "as-received" dry bulk densities determined prior to preparing each sample for

characterizations. Each Tank 5F sample was homogenized to promote particle size reduction due to the presence of chunks of solids. Homogenizing each sample involved grinding with a mortar and pestle and then passing the powder through a sieve pan with 850 micron openings (mesh 18). Samples which did not go through the sieve were ground with mortar and pestle until they were small enough to go through the sieve. The bulk density of each homogenized sample was determined followed by the blending of proportional amounts of the samples by weight to form three composite Tank 5F samples (See Table 1). The bulk density of each of the three composite samples was then determined by the process described in Appendix B. A reference simulant sludge sample, based on Tank 8 sample chemistry [See Appendix A-1], was air-dried in a clean laboratory and the resulting sludge cake ground and homogenized with a mortar and pestle. The bulk density of this reference Tank 8 sludge was determined both inside the shielded cell along with the Tank 5F samples and outside the cell in a clean laboratory hood. These Tank 8 sludge simulant bulk density values were used to verify how well the reference bulk densities could be reproduced both inside (using remote handling via manipulators) and outside of the shielded cell.

SRR determined the composite sample volumetric percent as shown in Table 1 [B. Dean, "Tank 5 Composite Sample Volumetric Proportions," SRR-CWDA-2011-00067, Rev.1 April 20, 2011]. These individual sample proportional location volumes, along with the homogenized sample bulk densities (Table 2), were used to calculate each sample's mass per composite volume and totaled. The total provided the composite density and the required weight from each of the fifteen Tank 5F sample material to make the three 70-gram composite Tank 5F samples as shown in Table 3. The Tank 5F sample with sample identification number **5-A1a-U,** designated for sample composition # 1 as shown in Tables 1 and 3, did not have sufficient material for composition to meet the 70 g requirement. The most that could be proportionally composited using available amount of sample **5-A1a-U** (15.96 g) was 29.8 g. The weight percent solid of the three Tank 5F composite materials was also determined as shown in Table 4. All bulk density data for the "as-received", homogenized and composited Tank 5F samples are presented in Appendices A-4 and A-5. Appendix A-6 shows the amount of discrete and composite Tank 5F samples left after all samples digestions and analyses. The weight percent solid determination method is described in Appendix B.

Table 1. Composite Samples Volumetric Distribution

	Composite Samples					
Composite sample number	**# 1**		**# 2**		**# 3**	
Riser 1	50%	5-A1a-U	55%	5-A2a-BM	44%	5-A3a-BM
Riser 5	42%	5-B2a-MD	38%	5-B3a-BM	43%	5-B1a-U
Remainder of Tank	2.67%	5-C1a	2.33%	5-C1b	4.33%	5-C1c
	2.67%	5-C2b	2.33%	5-C2c	4.33%	5-C2a
	2.67%	5-C3c	2.33%	5-C3a	4.33%	5-C3b
Total	100%		100%		100%	

Table 2. Tank 5F Sample Bulk Densities

Tank 5F Sample ID	"As-received" Sample Density Average, g/mL	"Homogenized" Sample Density Average, g/mL	Composite Sample Density Average, g/mL	Comments
5-C1a	**0.91***	**1.04** ±*0.04*	NA	Limited amount of sample
5-C1b	**1.21** ± *0.09*	**1.11** ±*0.01*	NA	
5-C1c	**0.97** ±*0.10*	**0.95** ±*0.07*	NA	Flaky and chunky "As-received" sample
5-C2a	**0.85** ±*0.02*	**1.02** ±*0.04*	NA	
5-C2b	**0.87** ± *0.06*	**0.97** ±*0.03*	NA	Flaky and chunky "As-received" sample
5-C2c	**0.84** ±*0.07*	**0.93** ±*0.04*	NA	Flaky and chunky "As-received" sample
5-C3a	**1.05** ±*0.01*	**1.11** ±*0.04*	NA	
5-C3b	**1.17** ± *0.05*	**1.03** ±*0.06*	NA	
5-C3c	**0.85** ±*0.04*	**0.96** ±*0.03*	NA	Flaky and chunky "As-received" sample
5-A1a-U	**1.45** ±*0.01*	**1.43** ±*0.02*	NA	
5 -B1a-U	**1.47** ±*0.01*	**1.44** ±*0.01*	NA	
5-A2a-BM	**1.22** ±*0.10*	**1.25** ±*0.01*	NA	
5 -A3a-BM	**1.09** ±*0.02*	**1.05** ±*0.00*	NA	
5 –B2a-MD	**1.33** ±*0.03*	**1.16** ±*0.03*	NA	
5 –B3a-BM	**1.05** ±*0.09*	**1.24** ±*0.03*	NA	
Reference Tank 8 Simulant	NA	**1.45** ±*0.04*		Value is within 10 % of out of cell determination (1.60 ±*0.03 g/mL)*
Composite sample #1	NA	NA	**1.41** ±*0.01*	
Composite sample #2	NA	NA	**1.34** ±*0.03*	
Composite sample #3	NA	NA	**1.31** ±*0.01*	

*The **"**As-received" density values are suspect and may in some cases have large uncertainty values. Problems were encountered in determining the volumes of these samples in calibrated PMP beakers used. Most of the samples contained large chunky pieces, which made it difficult to determine acceptable sample volumes.

Table 3. Tank 5F sample compositions for Composite samples 1, 2 and 3.

Composite Sample IDs	Material Available, g	Proportional Sample location volume, %	As-received density, g/mL	Wt. Fraction	Required Wt. of material to make 70 g composite	Optional for composite Sample #1**	Amount Weighed,g
Composite Sample #1							
5-A1a-U	15.962	50	1.45	0.54	37.49	15.96	15.96
5-B2a-MD	75.784	42	1.33	0.41	28.88	12.26	12.310
5-C1a	21.832	2.67	0.91	0.02	1.26	0.53	0.530
5-C2b	49.964	2.67	0.87	0.02	1.20	0.51	0.510
5-C3c	61.402	2.67	0.85	0.02	1.17	0.50	0.530
Mass sum		100	1.35	1.0	70.00	29.8	29.84
Composite Sample #2							
5-A2a-BM	278.324	55	1.22	0.59	41.12		41.174
5-B3a-BM	156.933	38	1.05	0.35	24.45		24.412
5-C1b	70.309	2.33	1.21	0.02	1.73		1.734
5-C2c	78.696	2.33	0.84	0.02	1.20		1.218
5-C3a	98.893	2.33	1.05	0.02	1.50		1.521
Mass sum		100	1.14	1.0	70.00		70.006
Composite Sample #3							
5-A3a-BM	200.41	44	1.09	0.39	27.05		27.008
5-B1a-U	*39.596*	43	1.47	0.51	35.65		35.654
5-C1c	43.266	4.33	0.97	0.03	2.37		2.373
5-C2a	47.696	4.33	0.85	0.03	2.08		2.080
5-C3b	54.742	4.33	1.17	0.04	2.86		2.867
Mass sum		100	1.42	1.0	70.00		70.005

**Sample highlighted in red (5-A1a-U) from sample composition #1 did not have sufficient material for composition to meet the 70 g requirement. The most that could be composited using sample 5-A1a-U for composite sample #1 was 29.8 g. See column 7 of Table above.

Table 4. Weight Percent Solids for Tank 5F Composite Samples

Tank 5F Sample ID	Wt% solids	Comments
Tank 5F Composite No. 1	95.7 ± 0.2	
Tank 5F Composite No. 2	96.7 ± 0.3	
Tank 5F Composite No. 3	96.4 ± 0.4	
Tank 8 Simulant sludge	89.2 ± 0.4	
5% Reference NaCl Salt solution	4.80 ± 0.1	Reference target weight percent NaCl solid = 4.9%

Because of the inherent risk of cross-contamination of these samples in the shielded cells environment, certain actions were taken to minimize the risks. Actions taken to control cross-contaminations in the cell included wiping down the cell (cell decontaminated), covering the cell floor, and changing manipulator fingers prior to initiating work. Additionally, blanks were processed with samples to evaluate potential issues from methods.

2.1 Blank Evaluations and Reference Materials

Two types of reference matrices were used during the characterization of Tank 5F samples. The first reference material was an analyzed reference glass (ARG) which was stored outside the shielded cells but processed in the shielded cells along with the samples during sample preparations. The second was a dried Tank 8 simulant sludge, which was exposed to the shielded cell radiological environment in which the Tank 5F radionuclide material was processed prior to analysis. The elemental chemical composition of the Tank 8 simulant sludge and analyzed reference glass are presented in Appendices A-1 and A-2. Distilled and de-ionized water was used as the liquid reagent media and blanks in all cell digestion cases. The absence of radionuclides in these reference materials allowed the materials to additionally be utilized as blanks for radiochemical analyses.

Prior to the processing of the Tank 5F samples, which normally involved the opening of selected samples to be blended together, two in-cell reference Tank 8 simulant sludge samples in 250-mL capacity poly-bottles were placed at strategic locations in the shielded cell. Each simulant sludge reference sample container contained about 20 grams of Tank 8 simulant sludge. The containers were opened when the Tank 5F samples were being processed or air dried and closed at the end of each day of work in the cell. At the end of each Tank 5F sample preparations or digestion (aqua regia and peroxide fusion digestions), the Tank 8 simulant sludge reference material was also prepared in a manner similar to that for the preparation of Tank 5F samples and submitted for the same analyses as the actual samples from Tank 5F. Additional analytical blanks were run for each of the methods.by Analytical Development.

2.2 Leaching Characterization of Tank 5F Solids

Known amounts of homogenized Tank 5F composite solids were leached with distilled and de-ionized water in triplicate. An average of 1.17 ± 0.06 grams of the composite solids was leached with an average of 50.01 ± 0.01 grams of distilled and de-ionized water. In this process each solid fraction was thoroughly mixed with the given amount of distilled and de-ionized water, and the mixture was hand agitated (shielded cell manipulator) for a total of about five minutes and left to stand for another 24 hours before another agitation and filtering of the mixture using a 0.45 micron Nalgene filter unit. The filtrate from the mixture was analyzed in triplicate for anion components as required. Thus, only surface-bound and water soluble constituents are assumed to be accounted for in the leachate analyses.

3.0 RESULTS

Appendix A-0 contains the SRNL Analytical Development Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report.

Details of most of the analytical methodologies including weight percent solids and density determinations applied in Tank 5F sample characterizations are summarized in Appendix B. It is worth pointing out that many digestion methods were performed in the shielded cells prior to taking representative sample aliquots out of the cells for analyses. These digestions and other methods in the cells included digestions for all the "challenging" radionuclide analyses, aqua regia digestions, peroxide fusion digestions and digestions for Th-229/230 analyses. Additionally, new methods were developed, specifically tailored for the presence of oxalic acid and other components, for characterization of Tank 5F radionuclides. These methods are summarized in Appendix B.

In the Tank 5F composite sample characterization results presented below, values preceded by "<" (less than sign) indicate values were below minimum detection limits, and values proceeded by "≤" (less than or equal to sign) indicate that for replicates, at least one of the analysis values was above the instrument or method detection limit. Thus, where replicate analyses were both above and below the detection limit, the average of all replicates above and below the detection limit is given and a "≤ " sign precedes the average value. The standard deviation values were calculated only for values that were above the detection limits. The minimum detectable activity (MDA) is defined as the value above which instrument signal can be considered real and the upper limit (UL) is defined as activity observed but biased high due to spectral interference or blank contamination. The detection limit (DL) as used in mass spectrophotometer or ICP-ES analyses is equivalent to three times the standard deviation of the blank measurements.

The one sigma percent uncertainty for each major radionuclide, as reported in the tables, is based on the pooled estimate derived from the individual uncertainties for each replicate measurement for that radionuclide [SQRT((SUMSQ(x_i)/n))], where n is the number of replicates and x_i is the individual uncertainty associated with each radionuclide for each run. Here it is assumed that the radio-analytical processes, be it counting or other techniques, are of the same precision for each individual measurement.

Occasionally, situations were encountered where the samples prepared and analyzed in triplicate gave mixed results with one or two of the triplicate analyses results being less than the MDA. In these cases, the reporting of the one sigma percent uncertainty is presented in a different format. In this situation, the individual percent uncertainty associated with each run for that radionuclide is reported along with MDA or upper limit values as indicated by the analytical method. For example, under the one sigma percent uncertainty column for a radionuclide in Table 12, the 8.0/MDA designation implies that the one sigma percent uncertainty for I-129 in runs 1 and 3 of the analyses is reported with values above the detection limit and thus has a pooled one sigma percent uncertainty of 8 percent. The measurement which was below the detection limit [I-129, run 2; with no percent uncertainty value assigned)] is assigned MDA, DL or upper limit designation. In some cases, especially with the radionuclides (See Th-229/230 in Table 11), the one sigma measurement uncertainty

may be higher than 100%. In such a situation, the reported value is barely above the magnitude of the minimum detection activity (MDA) for that radionuclide, which leads to a higher uncertainty in the analytical result.

To verify the absence of sample contamination during processing, analytical blank (reagent blanks and Tank 8 simulant sludge) results were compared to sample analytical results (See second column blank results for most radionuclide as presented in each Table). The blank analytical results for the different species of interest are all well below the sample analytical results or below a measureable limit. Thus, there were no measurable cross contamination issues either from the environment of the shielded cell staging and operation areas or the reagents used in sample preparations.

The reporting units for all radionuclides including PF and AQR digestion analytical results are presented per gram of composite Tank 5F sample. These composite Tank 5F samples were digested and characterized without further processing such as drying. Correction for water content as determined by sub-sample drying at 110 ºC, if required (original "as received" basis to dry basis), can be accomplished through the use of the dry solid weight percent (wt %) values as shown in Table 4 for each composite sample. For example, μCi/g dried solids = [x μCi/g of "as-received solids * (100 g of "as-received solids)/95.7g dried solids]; using composite sample 1 in Table 4.

The one sigma measurement uncertainty value for all of the anions and transition metals reported here is 20 percent. Leaching results are presented per gram of the "homogenized and composite" Tank 5F composite samples.

Tables 5-7 contain inorganic constituent analytical results for the three composite Tank 5F samples, while Tables 8-10 show the water soluble anion constituents for the Tank 5F composite sample. Tables 11-13 contain the analytical results for the standard radiological constituents for the three composite Tank 5F samples, while Tables 14-16 show the analytical results for the "challenging" radionuclide constituents.

3.1 Data Quality and Presentations for Routine Radionuclide Constituents

The ICP-MS results are given for each atomic mass and, in most cases each mass number represents only one isotope. An example of an exception is mass 238, since both uranium and plutonium are represented by this mass number. However, since the mass contribution of U-238 is significantly greater than that of Pu-238, the 238 signal is used to quantify U-238, not Pu-238. For this reason, Pu-238 was determined by PUTTA (chemical separation coupled with alpha spectroscopy). See Appendix B for summaries of the methods. In cases where ICP-MS and radiochemistry data give similar results for a species, radiochemistry is typically selected due to better sensitivity and precision.

While many of the minimum detection limits (MDL), as specified in the TTR and TTQAP were met for the species characterized for Tank 5F composite samples, some were not met. In a number of cases, the relatively high levels of radioactive species of the same element or a chemically similar element precluded the ability to measure an isotope to lower levels. For example, the high activities of Am-241 and Cm-244 in the sample matrix raised the alpha

spectroscopy instrumental backgrounds for Cm-243, having a detrimental effect on the detection limit for that isotope. The 2.6 year half-life Pm-147 co-extracts with the 90 year half-life Sm-151. Both have overlapping beta spectra, with slightly higher continuum beta end-point energy for Pm-147. The Sm-151 levels in these composite Tank 5F samples were relatively high, substantially raising the detection limit achievable for Pm-147. A number of gamma emitting radionuclides were analyzed using a Cs-137 removed gamma analysis; Cesium-137 was expected to be the main contributor to background levels which would lower the sensitivity of the gamma analysis for other species. While that was true, the samples also contained significant quantities of other gamma emitting isotopes (i.e. Co-60, Eu-154, Eu-155, etc…) which raised the background and thus the detection limits for gamma emitting species not observed (i.e. Nb-94, Sb-125, etc…).

For the Th-229/Th-230 analyses, the Tank 5F Th-230 sample activities were substantially higher than the activities of the Th-229 tracer used to trace the analyses. This resulted in a large uncertainty in the measurement of the tracer recovery, which translated into a large uncertainty in the measurement of the Th-229 and Th-230 activities present in the samples.

3.2 Data Quality and Presentations for Elemental Constituents

The reference materials for the elemental analyses results presented in Tables 5-7 were ARG and dried Tank 8 simulant sludge. Appendices A-1 and A-2 contain the elemental analytical results for the two reference matrices in comparison to the known values for these reference materials[2].

A comparison of the laboratory results for the cations present in the simulant sludge, with potassium being the exception, shows that our laboratory results are not significantly different from the known reference values for these cations. The percent relative deviation (%RD defined as [difference/mean]*100) for each of the 16 constituent cations of this simulant sludge material is less than 12%. Only potassium analysis had a %RD greater than 25%. Similarly, looking at the analytical results for the 17 elemental constituents of the ARG reference sample [Appendix A-2], only strontium and zinc analyses result are above 15% RD.

The one liter reference simulant sludge sample used in these analyses came from batch simulant preparation process in which the sludge sample is prepared in 200 liter batches. Thus, using only one liter from this batch sample may not be representative sampling. Hence, the difference in potassium concentration measured in comparison to the expected nominal value may be attributed to representative subsampling. Overall, the laboratory analytical results from these two reference materials for the constituent elements are quite comparable to the expected or known concentrations.

[2] C. J Coleman, R. A. Dewberry, M. F. Bryant and J.J Gemmill," SRL's performance in Round Robin #6-Analyses of Simulated Defense Waste Glass', WSRC-TR-91-187, Rev. 0, May 31, 1999, D. Koopman,"Tank 8, Drum1, Sludge simulant, SRTC mobile Lab. ID # 20000616," July 26, 2000 and M. R. Poirier,"Tank 40 and Tank 8 sludge feed simulants" SRTC-WHM-2000, Rev.2, June 2000.

To compare Tank 5F composite sample elemental analytical results by inductively coupled plasma-mass spectrophotometer (ICP-MS) with result by inductively coupled plasma-emission spectroscopy (ICP-ES) [data presented here for the elemental composition are based solely on ICP-ES], the concentration of select cations (natural Cd, Ba and La) were calculated from ICP-MS information and the resulting concentration values compared with the ICP-ES corresponding results presented in this report. Typical calculations are shown in Appendix A-2 for cadmium, barium and lanthanum. The average percent relative deviation between ICP-MS and ICP-ES analytical results for La, Cd and Ba are, respectively, 4.9, 29 and 9.9 %. These comparison results are summarized in Appendix A-2 and show that ICP-ES analytical results are the same order of magnitude as the ICP-MS data for these select cations.

3.3 Data Quality and Presentations for Non-Routine Radionuclide Analytes.

Some radiochemical analyzes are considered "challenging" when dealing with radionuclides that are not present in easily measurable concentrations with existing standard methods and thus require new method development to meet the low detection limit requirements. The analytical results for some of the "challenging" to analyze isotopes, (Al-26, Cl-36, K-40, Nb-94, Sn-126, Sb-126 and Sb-126m, Pd-107, Eu-152, Pt-193, Ra-226 and Ac-227), are presented slightly differently. Since analyses for these challenging analytes are confirmatory in nature, special emphasis was placed on achieving these target detection limits for at least one composite sample. Thus, only one replicate per sample is required for these analytes and for a given analyte, if all sample analysis results were below detection limits, then only the lowest detection level obtained was reported as summarized in Tables 14-16.

In some instances the analyses were not performed and reported for all three composite Tank 5F samples in triplicate, partly because of challenging analytical separation issues associated with the different methods developed for these analytes (specific methods are provided in Appendix B). Some of the development methods evaluated were unsuccessful even with extra distillation/separation and longer counting efforts. Thus, the customer specified detection limits for these challenging radionuclide species were not consistently met in the three Tank 5F composite samples as presented in detail in Appendix A-3.

The customer specified detection limits for Al-26, Eu-152, Sn-126, Sb-126, and Sb-126m were **not** met in any of the three Tank 5F composite samples as summarized in Tables 14-16. Analyses results obtained for the three Tank 5F composite samples for Cl-36 and Ac-227 were all upper limit values. The presence of high concentrations of stable metals and radionuclides described in section 3.1 created a challenging matrix, which limited the ability of the methods to achieve minimum detection limits as presented in Tables 14-16. These results were reviewed with the customer, who accepted the given results as presented [SRR-CWDA-2012-00013].

The detection limits for Nb-94 and Pt-193 were met in the analyses of Tank 5F composite sample No. 1, while the detection limits obtained for Ra-226, Ac-227, Cl-36 and K-40 for Tank 5F composite sample No. 1 were about the same order of magnitude as the customer desired detection limits of 10^{-3} uCi/g (Table 14). Analytical results for Tank 5F composite sample No. 2, shows that the detection limits for Nb-94, Pt-193, Ra-226, and Ac-227 were

met, while analytical results for Cl-36 and K-40 were about the same order of magnitude as the customer desired detection limits. The analytical results for Tank 5F composite sample No. 3 shows that the customer specified detection limits, as summarized in Table 16, were met for K-40, Nb-94 and Ra-226, while the detection limits for Cl-36, Pt-193 and Ac-227 were about the same order of magnitude as the customer desired detection limits. Meanwhile, a review of blank analytical data shows that analyses performed for Cl-36 blanks in the Tank 5F composite samples show evidence of beta activity. This is an indication that counts from the composite samples for Cl-36 could have been due to beta cross contamination caused by sample manipulation. Thus, it can be concluded that the limited measured activity assigned to Cl-36 may not be due to the presence of Cl-36 in all three composite Tank 5F samples.

The Pu-241 liquid scintillation analyses had traces of high energy beta activity present in addition to the Pu-241 beta. As a consequence, the Pu-241 values reported are flagged as upper limits as the Pu-241 results had a small positive bias (about 10%) resulting from spill-over of the higher energy beta emitter's activity into the Pu-241 lower energy beta window.
Where the use of tracers showed extremely poor or no recovery at all, as in the case of using stable selenium as a carrier with the blank for selenium-79 analyses, no results are presented in the Tables, but instead a "no yield" is indicated. This is especially the case with blank and reference samples for routine radionuclide analytes and "challenging" analytes.

Table 5. Elemental Constituents in Tank 5F Composite Sample # 1, mg/g sample *

Analyte	Tank 5, Run1	Tank 5, Run 2	Tank 5, Run 3	Average	STDEV
Ag	2.01E-01	2.38E-01	2.73E-01	**2.37E-01**	*3.60E-02*
Al	4.06E+00	4.42E+00	8.70E+00	**5.73E+00**	*2.58E+00*
B	1.40E+00	1.30E+00	1.20E+00	**1.30E+00**	*1.00E-01*
Ba	1.74E+00	1.97E+00	2.07E+00	**1.93E+00**	*1.69E-01*
Be	1.30E-02	1.21E-02	1.12E-02	**1.21E-02**	*9.00E-04*
Ca	1.43E+00	1.38E+00	1.43E+00	**1.41E+00**	*2.89E-02*
Cd	8.13E-02	9.16E-02	9.69E-02	**8.99E-02**	*7.93E-03*
Ce	1.97E+00	2.59E+00	3.06E+00	**2.54E+00**	*5.47E-01*
Co	1.87E-01	2.12E-01	2.39E-01	**2.13E-01**	*2.60E-02*
Cr	1.39E+00	1.04E+00	9.68E-01	**1.13E+00**	*2.26E-01*
Cu	6.84E-01	7.16E-01	7.60E-01	**7.20E-01**	*3.82E-02*
Fe	5.23E+02	4.88E+02	4.52E+02	**4.88E+02**	*3.55E+01*
Gd	1.93E-01	1.93E-01	1.93E-01	**1.93E-01**	*0.00E+00*
K	2.94E-01	3.68E-01	3.64E-01	**3.42E-01**	*4.16E-02*
La	1.33E+00	1.38E+00	1.45E+00	**1.39E+00**	*6.03E-02*
Li	4.00E-01	5.30E-01	6.09E-01	**5.13E-01**	*1.06E-01*
Mg	4.54E-01	4.88E-01	5.27E-01	**4.90E-01**	*3.65E-02*
Mn	3.11E+01	3.43E+01	3.80E+01	**3.45E+01**	*3.45E+00*
Mo	4.87E-02	5.10E-02	5.06E-02	**5.01E-02**	*1.23E-03*
Na	3.60E+00	3.55E+00	3.61E+00	**3.59E+00**	*3.21E-02*
Ni	4.54E+01	5.63E+01	6.34E+01	**5.50E+01**	*9.07E+00*
P	2.26E-01	2.11E-01	2.65E-01	**2.34E-01**	*2.79E-02*
Pb	3.88E+00	3.70E+00	3.49E+00	**3.69E+00**	*1.95E-01*
S	<2.70E-01	<3.06E-01	5.13E-01	**≤3.63E-01**	
Sb	<7.67E-01	<8.68E-01	<7.80E-01	**<8.05E-01**	
Si	1.05E+00	1.05E+00	6.04E-01	**9.01E-01**	*2.57E-01*
Sn	4.37E-02	5.06E-02	3.18E-02	**4.20E-02**	*9.51E-03*
Sr	4.11E-01	3.81E-01	3.65E-01	**3.86E-01**	*2.34E-02*
Th	<3.90E-01	<4.40E-01	<3.90E-01	**<4.04E-01**	
Ti	2.22E-01	2.55E-01	2.79E-01	**2.52E-01**	*2.86E-02*
U	7.14E+00	8.13E+00	9.23E+00	**8.17E+00**	*1.05E+00*
V	<3.40E-02	<3.80E-02	<3.40E-02	**<3.55E-02**	
Zn	3.70E-01	4.11E-01	5.12E-01	**4.31E-01**	*7.31E-02*
Zr	3.54E+00	4.43E+00	4.57E+00	**4.18E+00**	*5.59E-01*
Hg	1.84E+00	2.32E+00	2.55E+00	**2.24E+00**	*0.362E+00*
Se	<2.00E-02	<2.00E-02	<2.00E-02	**<2.00E-02**	
As	< 9.44E-03	<1.07E-02	<9.60E-03	**<9.91E-03**	

***The following color codes are used for the Table contents: Green for blank values, red for less than values, pink for less than or equal to values, and bold for averages. All subsequent tables have similar color code meanings.

Table 6. Elemental Constituents in Tank 5F Composite Sample # 2, mg/g sample

Analyte	Tank 5 Run 1	Tank 5 Run 2	Tank 5 Run 3	Average	STDEV
Ag	2.16E-01	2.15E-01	2.16E-01	**2.16E-01**	*5.77E-04*
Al	4.31E+00	4.45E+00	4.35E+00	**4.37E+00**	*7.21E-02*
B	1.4E+00	1.37E+00	1.35E+00	**1.37E+00**	*2.52E-02*
Ba	1.77E+00	1.86 E+00	1.83 E+00	**1.82E+00**	*4.58E-02*
Be	1.29E-02	1.27E-02	1.26E-02	**1.27E-02**	*1.53E-04*
Ca	1.55E+00	1.56E+00	1.55E+00	**1.55E+00**	*5.77E-03*
Cd	8.33E-02	8.73E-02	8.59E-02	**8.55E-02**	*2.03E-03*
Ce	1.92E+00	2.29E+00	2.18E+00	**2.13E+00**	*1.90E-01*
Co	1.86E-01	1.96E-01	1.95E-01	**1.92E-01**	*5.51E-03*
Cr	9.82E-01	9.98E-01	9.78E-01	**9.86E-01**	*1.06E-02*
Cu	6.42E-01	6.68E-01	6.59E-01	**6.56E-01**	*1.32E-02*
Fe	5.22 E+02	5.15 E+02	5.05E+02	**5.14E+02**	*8.54E+00*
Gd	1.93E-01	1.96E-01	1.95E-01	**1.95E-01**	*1.53E-03*
K	2.93E-01	2.47E-01	2.68E-01	**2.69E-01**	*2.30E-02*
La	1.21E+00	1.29E+00	1.26E+00	**1.25E+00**	*4.04E-02*
Li	4.15E-01	4.79E-01	4.55E-01	**4.50E-01**	*3.23E-02*
Mg	4.70E-01	4.87E-01	4.80E-01	**4.79E-01**	*8.54E-03*
Mn	3.05E+01	3.14E+01	3.18E+01	**3.12E+01**	*6.66E-01*
Mo	4.55E-02	4.42E-02	4.56E-02	**4.51E-02**	*7.81E-04*
Na	3.6E+00	3.71E+00	3.71E+00	**3.67E+00**	*6.35E-02*
Ni	4.63E+01	5.17E+01	4.99E+01	**4.93E+01**	*2.75E+00*
P	2.19E-01	3.21E-01	3.34E-01	**2.91E-01**	*6.30E-02*
Pb	3.85E+00	3.78E+00	3.77E+00	**3.80E+00**	*4.36E-02*
S	<3.00E-01	2.90E-01	<3.00E-01	**≤2.96E-01**	
Sb	<8.60E-01	<8.00E-01	<8.40E-01	**<8.40E-01**	
Si	1.24E+00	1.32E+00	9.13E-01	**1.16E+00**	*2.16E-01*
Sn	4.41E-02	4.08E-02	4.40E-02	**4.30E-02**	*1.88E-03*
Sr	4.11E-01	4.03E-01	4.01E-01	**4.06E-01**	*4.16E-03*
Th	<4.30E-01	<4.00E-01	<4.20E-01	**<4.20E-01**	
Ti	2.3E-01	2.45E-01	2.46E-01	**2.40E-01**	*8.96E-03*
U	7.97E+00	8.89E+00	9.43E+00	**8.76E+00**	*7.38E-01*
V	<3.80E-02	<3.60E-02	<3.70E-02	**<3.69E-02**	
Zn	3.62E-01	3.81E-01	3.80E-01	**3.74E-01**	*1.07E-02*
Zr	3.74E+00	4.07E+00	3.98E+00	**3.93E+00**	*1.71E-01*
Hg	1.92E+00	2.07E+00	1.98E+00	**1.99E+00**	*0.076*
Se	<2.10E-02	<2.00E-03	<2.00E-03	**< 8.0E-03**	
As	<1.06E-02	<9.91E-03	<1.04E-02	**<1.03E-02**	

Table 7. Elemental Constituents in Tank 5F- Composite Sample # 3, mg/g sample

Analyte	Tank 5 Run 1	Tank 5 Run 2	Tank 5 Run 3	Average	STDEV
Ag	2.82E-01	2.90E-01	2.95E-01	**2.89E-01**	*6.56E-03*
Al	4.58E+00	4.34E+00	4.59E+00	**4.50E+00**	*1.42E-01*
B	1.27E+00	1.25E+00	1.25E+00	**1.26E+00**	*1.15E-02*
Ba	2.1E+00	2.11E+00	2.1E+00	**2.10E+00**	*5.77E-03*
Be	1.16E-02	1.16E-02	1.17E-02	**1.16E-02**	*5.77E-05*
Ca	1.81E+00	1.45E+00	1.75E+00	**1.67E+00**	*1.93E-01*
Cd	9.28E-02	9.37E-02	9.56E-02	**9.40E-02**	*1.43E-03*
Ce	2.96E+00	3.00E+00	3.07E+00	**3.01E+00**	*5.57E-02*
Co	2.40E-01	2.41E-01	2.40E-01	**2.40E-01**	*5.77E-04*
Cr	1.03E+00	1.06E+00	1.06E+00	**1.05E+00**	*1.73E-02*
Cu	9.54E-01	6.92E-01	7.42E-01	**7.96E-01**	*1.39E-01*
Fe	4.66E+02	4.66E+02	4.65E+02	**4.66E+02**	*5.77E-01*
Gd	2.04E-01	2.01E-01	2.02E-01	**2.02E-01**	*1.53E-03*
K	3.39E-01	2.67E-01	4.40E-01	**2.83E-01**	*4.96E-02*
La	1.65E+00	1.63E+00	1.65E+00	**1.64E+00**	*1.15E-02*
Li	5.57E-01	5.78E-01	5.87E-01	**5.74E-01**	*1.54E-02*
Mg	5.15E-01	4.96E-01	5.10E-01	**5.07E-01**	*9.85E-03*
Mn	3.68E+01	3.54E+01	3.54E+01	**3.59E+01**	*8.08E-01*
Mo	4.57E-02	4.55E-02	4.49E-02	**4.54E-02**	*4.16E-04*
Na	4.23E+00	4.07E+00	4.11E+00	**4.14E+00**	*8.33E-02*
Ni	6.11E+01	6.25E+01	6.32E+01	**6.23E+01**	*1.07E+00*
P	2.95E-01	3.02E-01	2.94E-01	**2.97E-01**	*4.36E-03*
Pb	3.58E+00	3.60E+00	3.62E+00	**3.60E+00**	*2.00E-02*
S	3.62E-01	4.29E-01	3.25E-01	**3.72E-01**	*5.27E-02*
Sb	<8.00E-01	<8.45E-01	<8.50E-01	**<8.31E-01**	
Si	7.99E-01	1.86E+00	1.15E+00	**1.27E+00**	*5.41E-01*
Sn	3.70E-02	4.05E-02	4.12E-02	**3.96E-02**	*2.25E-03*
Sr	3.53E-01	3.47E-01	3.46E-01	**3.49E-01**	*3.79E-03*
Th	<4.00E-01	<4.24E-01	<4.30E-01	**<4.17E-01**	
Ti	2.54E-01	2.57E-01	2.66E-01	**2.59E-01**	*6.24E-03*
U	10.6E+00	9.79E+00	9.64E+00	**1.00E+01**	*5.16E-01*
V	<4.00E-02	<3.73E-02	<4.00E-02	**<3.66E-02**	
Zn	6.61E-01	4.43E-01	5.03E-01	**5.36E-01**	*1.13E-01*
Zr	4.58E+00	4.92E+00	4.93E+00	**4.81E+00**	*1.99E-01*
Hg	2.49E+00	2.53E+00	2.61E+00	**2.54E+00**	*6.10E-02*
Se	<2.00E-02	<2.10E-02	<2.10E-02	**<.2.0E-02**	
As	<9.79E-03	<1.04E-02	<1.05E-02	**<1.01E-02**	

Table 8. Anions Leached per gram of Tank 5F- Composite Sample #1

Analyte	Run-1	Run-2	Run-3	Average	Std. Dev.	Unit
F^{-1}	< 0.046	< 0.040	< 0.037	**< 0.041**		*mg/g*
Cl^{-1}	0.046	0.040	0.037	**0.041**	*0.00*	*mg/g*
NO_3^{-1}	0.23	0.32	0.075	**0.21**	*0.12*	*mg/g*
NO_2^{-1}	0.046	0.079	< 0.037	**≤0.054**		*mg/g*
SO_4^{-2}	0.27	0.24	0.22	**0.25**	*0.03*	*mg/g*
$C_2O_4^{-2}$	1.96	1.87	1.90	**1.91**	*0.05*	*mg/g*
PO_4^{-3}	< 0.046	< 0.040	< 0.037	**< 0.041**		*mg/g*
CHO_2^{-1}	0.59	0.56	0.52	**0.56**	*0.04*	*mg/g*

Table 9. Anions Leached per gram of Tank 5F- Composite Sample #2

Analyte	Run-1	Run-2	Run-3	Average	Std. Dev.	Unit
F^{-1}	< 0.036	< 0.045	< 0.049	**< 0.043**		*mg/g*
Cl^{-1}	<0.036	0.045	0.049	**≤0.043**		*mg/g*
NO_3^{-1}	0.072	0.045	0.049	**0.055**	*0.01*	*mg/g*
NO_2^{-1}	< 0.036	<0.045	<0.049	**<0.043**		*mg/g*
SO_4^{-2}	0.14	0.13	0.15	**0.14**	*0.01*	*mg/g*
$C_2O_4^{-2}$	2.24	2.42	2.30	**2.32**	*0.09*	*mg/g*
PO_4^{-3}	< 0.036	< 0.045	< 0.049	**< 0.043**		*mg/g*
CHO_2^{-1}	0.22	0.22	0.24	**0.23**	*0.01*	*mg/g*

Table 10. Anions Leached per gram of Tank 5F- Composite Sample #3

Analyte	Run-1	Run-2	Run-3	Average	Std. Dev.	Unit
F^{-1}	< 0.044	< 0.044	< 0.048	**< 0.045**		*mg/g*
Cl^{-1}	< 0.044	< 0.044	< 0.048	**< 0.045**		*mg/g*
NO_3^{-1}	0.088	0.089	0.095	**0.091**	*0.00*	*mg/g*
NO_2^{-1}	< 0.044	< 0.044	< 0.048	**< 0.045**		*mg/g*
SO_4^{-2}	0.31	0.31	0.33	**0.32**	*0.01*	*mg/g*
$C_2O_4^{-2}$	2.65	2.66	2.86	**2.72**	*0.12*	*mg/g*
PO_4^{-3}	< 0.044	< 0.044	< 0.048	**< 0.045**		*mg/g*
CHO_2^{-1}	0.40	0.40	0.38	**0.39**	*0.01*	*mg/g*

Table 11. Radiological Constituents for Tank 5F Composite Sample #1, µCi/g.

Analytes	Blank	Run 1,	Run 2	Run 3	Average	Stdev.	One Sigma %Uncert.	Targeted Minimum Detection Limits
Gross alpha	<4.92E+0	<3.23E+02	<4.91E+02	<4.20E+02	**<4.12E+02**		MDA	NA
Non-volatile beta	<1.09E+01	2.95E+04	2.98E+04	3.05E+04	**3.00E+04**	*5.19E+02*	10	NA
H-3	<9.06E-03	<8.65E-03	<1.01E-02	<9.09E-03	**<9.28E-03**		MDA	1.0E-01
C-14	<7.84E-04	<8.33E-04	<8.51E-04	<7.39E-04	**<8.08E-04**		MDA	1.0E-01
Ni-59	<9.23E-03	4.73E+00	7.84E+00	6.67E+00	**6.41E+00**	*1.57E+00*	10	9.0E-02
Ni-63	<8.56E-02	2.65E+02	3.01E+02	3.50E+02	**3.05E+02**	*4.25E+01*	20	1.0E-01
Co-60	<1.33E-02	7.25E+00	6.62E+00	6.22E+00	**6.70E+00**	*5.22E-01*	5	1.0E-03
Se-79	No yield	1.33E-02	8.29E-03	1.42E-02	**1.19E-02**	*3.19E-03*	31	1.0E-03
Sr-90	<7.43E+0	1.21E+04	1.19E+04	1.23E+04	**1.21E+04**	*2.03E+02*	6	1.0E-03
Y-90	<7.43E+0	1.21E+04	1.19E+04	1.23E+04	**1.21E+04**	*2.03E+02*	6	1.0E-03
Zr-93	2.08E-02	3.15E+00	3.18E+00	2.49E+00	**2.94E+00**	*0.39*	20	1.0E-03
Tc-99	<9.86E-05	1.02E-02	7.66E-03	8.47E-03	**8.78E-03**	*1.31E-03*	6	1.0E-03
Pd-107**	<3.38E-04	2.50E-03	2.51E-03	2.81E-03	**2.61E-03**	*1.76E-04*	20	1.0E-03
I-129	<2.31E-06	1.10E-04	1.98E-04	1.64E-04	**1.57E-04**	*4.41E-05*	14	1.0E-04
Cs-135	8.10E-06	2.34E-03	1.86E-03	1.67E-03	**1.96E-03**	*3.45E-04*	20	5.0E-02
Cs-137	<3.23E-02	4.47E+02	3.71E+02	3.49E+02	**3.89E+02**	*5.14E+01*	5	1.0E-03
Ba-137m	<3.06E-02	4.23E+02	3.51E+02	3.30E+02	**3.68E+02**	*5.14E+01*	5	1.0E-03
Pm-147	<3.84E+00	<4.59E+02	<5.45E+02	<5.81E+02	**<6.38E+02**			None
Sm-151	<3.46E+0	7.16E+02	7.25E+02	8.02E+02	**7.48E+02**	*4.70E+01*	20	3.0E+00
Eu-154	<2.06E-02	3.00E+01	2.87E+01	2.85E+01	**2.91E+01**	*7.92E-01*	5	1.0E-03
Eu-155	<4.00E-02	3.77E+00	5.32E+00	4.32E+00	**4.47E+00**	*7.82E-01*	11	None
Pt-193	<8.12E-04	<6.37E-04	<6.95E-04	<4.91E-04	**<6.08E-04**		MDA	1.0E-03
Th-229**	No yield	2.95E-06	4.10E-06	2.38E-05	**1.03E-05**	*1.17E-05*	151	1.0E-03
Th-230**	No yield	1.63E-03	1.56E-03	2.16E-03	**1.78E-03**	*3.27E-04*	151	1.0E-03
Pa-231**	<1.44E-03	<1.02E-03	<2.14E-04	<1.41E-04	**<4.58E-04**		DL	1.0E-03
U-232	No yield	<1.82E-05	<1.94E-05	<1.23E-05	**<1.66E-05**		DL	1.0E-03
U-233	<2.05E-03	<2.62E-04	<2.64E-04	<2.48E-04	**<2.58E-04**		DL	1.0E-03
U-234	<1.32E-03	4.27E-03	4.08E-03	3.54E-03	**3.96E-03**	*3.79E-04*	20	1.0E-03
U-235	<4.60E-07	1.80E-04	1.78E-04	1.60E-04	**1.73E-04**	*1.10E-05*	20	1.0E-04
U-236	<1.38E-05	2.25E-04	2.24E-04	1.95E-04	**2.15E-04**	*1.70E-05*	20	1.0E-03
U-238	8.34E-07	4.19E-03	4.10E-03	3.65E-03	**3.98E-03**	*2.89E-04*	20	1.0E-03
Np-237	<1.72E-04	2.41E-02	2.51E-02	1.12E-02	**2.01E-02**	*7.75E-03*	26	1.0E-03
Pu-238	6.80E-02	2.89E+00	2.51E+00	2.28E+00	**2.56E+00**	*3.09E-01*	7	1.0E-03
Pu-239	<3.61E-02	8.80E+00	8.47E+00	7.25E+00	**8.17E+00**	*8.16E-01*	20	1.0E-03
Pu-240	<1.32E-01	2.05E+00	1.97E+00	1.68E+00	**1.90E+00**	*1.95E-01*	20	1.0E-03
Pu-239/240	<4.55E-02	1.09E+01	1.05E+01	8.92E+00	**1.01E+01**	*1.02E+00*	5	None

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes.

Table 11-continued. Radiological Constituents for Tank 5F Composite Sample #1 Continued, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	Stdev	One Sigma %Uncert.	Targeted Minimum Detection Limits
Pu-241	<1.05E-02	<1.19E+01	<1.19E+01	<1.30E+01	**<1.23E+01**		Upper Limit	1.0E-03
Pu-242	<2.29E-03	3.97E-04	4.24E-04	3.41E-04	**3.87E-04**	*4.23E-05*	20	1.0E-03
Pu-244	<1.07E-05	<8.36E-07	<1.05E-06	<9.74E-07	**<9.53E-07**		DL	1.30E-04
Am-241	<7.39E-02	7.30E+01	6.53E+01	6.40E+01	**6.74E+01**	*4.86E+00*	5	1.0E-03
Am-242m	<1.01E-03	1.77E-01	1.70E-01	1.41E-01	**1.63E-01**	*1.92E-02*	24	1.0E-03
Am-243	<5.72E-04	5.45E-01	5.09E-01	4.77E-01	**5.11E-01**	*3.38E-02*	6.5	1.0E-03
Cm-242	<8.33E-04	1.47E-01	1.41E-01	1.17E-01	**1.35E-01**	*3.05E-01*	24.	None
Cm-243	<4.64E-04	<7.52E-02	<5.77E-02	<4.36E-01	**<1.90E-01**		Upper limit	2.0E-02
Cm-244	<7.93E-03	3.32E+00	2.98E+00	3.59E+00	**3.29E+00**	*1.59E-02*	11.4	1.0E-03
Cm-245	<8.11E-06	<1.45E-03	<1.35E-03	<7.03E-04	**<1.17E-03**		Upper limit	2.0E-02
Cm-246	<1.68E-05	<2.09E-03	<2.39E-03	<1.24E-03	**<1.91E-03**		Upper limit	2.0E-02
Cm-247	<4.82E-09	<4.68E-07	<3.67E-07	<1.47E-07	**<3.27E-07**		Upper limit	1.3E-04
Bk-247	<5.36E-05	<5.23E-03	<4.09E-03	<1.64E-03	**<3.65E-03**		Upper limit	None
Cm-248	<5.90E-07	<5.72E-05	<3.43E-05	<1.45E-05	**<3.53E-05**		Upper limit	1.30E-04
Cf-249	<1.05E-04	<1.78E-02	<1.36E-02	<7.39E-03	**<1.29E-02**		MDA	5.0E-03
Cf-250	<1.22E-05	<8.29E-04	<7.61E-04	<2.24E-04	**<6.05E-04**		Upper limit	None
Cf-251	<2.28E-04	<4.82E-02	<3.66E-02	<1.99E-02	**<3.49E-02**		MDA	None
Cf-252	<8.33E-04	<1.47E-01	<1.41E-01	<1.17E-01	**<1.35E-01**		Upper limit	None

Table 12. Radiological Constituents for Tank 5F Composite Sample #2, μCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	Stdev	One Sigma %Uncert.	Targeted Minimum Detection Limits
Gross alpha	<2.34E+01	<3.09E+02	<3.48E+02	<1.07E+03	**<5.77E+02**		MDA	None
Non-volatile beta	<4.40E+01	3.24E+04	2.98E+04	3.12E+04	**3.12E+04**	*1.31E+03*	10	None
H-3	<9.06E-03	<1.00E-02	<8.37E-03	<1.72E-02	**<1.19E-02**		Upper limit	1.0E-01
C-14	<7.84E-04	<7.39E-04	<1.58E-03	<1.58E-03	**<1.30E-03**		MDA	1.0E-01
Ni-59	<9.23E-03	5.18E+00	5.72E+00	4.50E+00	**5.14E+00**	*6.09E-01*	10	9.0E-02
Ni-63	<8.56E-02	2.18E+02	2.33E+02	2.10E+02	**2.20E+02**	*1.19E+01*	20	1.0E-01
Co-60	<1.33E-02	6.85E+00	6.04E+00	6.94E+00	**6.61E+00**	*4.96E-01*	5	None
Se-79	No yield	5.86E-03	3.17E-03	1.85E-02	**9.17E-03**	*8.17E-03*	49	1.0E-03
Sr-90	<7.43E+0	1.28E+04	1.25E+04	1.23E+04	**1.26E+04**	*2.72E+02*	6	1.0E-03
Y-90	<7.43E+0	1.28E+04	1.25E+04	1.23E+04	**1.26E+04**	*2.72E+02*	6	1.0E-03
Zr-93	2.08E-02	3.12E+00	2.70E+00	2.83E+00	**2.88E+00**	*0.22*	20	1.0E-03
Tc-99	<9.86E-05	1.06E-02	9.64E-03	1.26E-02	**1.09E-02**	*1.49E-03*	6.7	1.0E-03
Pd-107**	<3.38E-04	6.12E-03	2.21E-03	8.04E-03	**5.46E-03**	*2.97E-03*	20	1.0E-03
I-129	<2.31E-06	1.36E-04	<3.49E-03	1.18E-03	**≤1.60E-03**		8.0/MDA	1.0E-04
Cs-135	8.10E-06	2.58E-03	1.70E-03	2.23E-03	**2.17E-03**	*4.43E-04*	20	5.0E-02
Cs-137	<3.23E-02	4.55E+02	3.53E+02	4.35E+02	**4.14E+02**	*5.42E+01*	5	1.0E-03
Ba-137m	<3.06E-02	4.30E+02	3.34E+02	4.12E+02	**3.92E+02**	*5.42E+01*	5	1.0E-03
Pm-147	<3.84E+00	<5.95E+02	<5.36E+02	<5.63E+02	**<6.58E+02**			None
Sm-151	<3.46E+0	7.88E+02	7.07E+02	7.57E+02	**7.51E+02**	*4.09E+01*	20	3.0E+00
Eu-154	<2.06E-02	2.98E+01	2.73E+01	2.93E+01	**2.88E+01**	*1.35E+00*	5	1.0E-03
Eu-155	<4.00E-02	3.79E+00	4.36E+00	4.33E+00	**4.16E+00**	*3.18E-01*	13	None
Th-229**	No yield	3.77E-06	6.67E-06	8.11E-06	**6.18E-06**	*2.21E-06*	202	1.0E-03
Th-230**	No yield	5.99E-04	2.31E-03	3.50E-04	**1.09E-03**	*1.06E-03*	202	1.0E-03
Pa-231**	<1.44E-03	<2.35E-04	<2.77E-04	<2.53E-04	**<2.55E-04**		DL	1.0E-03
U-232	No yield	<1.59E-05	<2.72E-05	<3.47E-05	**<2.59E-05**		DL	1.0E-03
U-233	<2.05E-03	<2.52E-04	<2.56E-04	<7.58E-03	**<2.70E-03**		DL	1.0E-03
U-234	<1.32E-03	5.33E-03	4.32E-03	<4.89E-03	**≤4.85E-03**		20/MDA	1.0E-03
U-235	<4.60E-07	2.33E-04	1.87E-04	2.25E-04	**2.15E-04**	*2.46E-05*	20	1.0E-04
U-236	<1.38E-05	2.85E-04	2.29E-04	2.70E-04	**2.61E-04**	*2.90E-05*	20	1.0E-03
U-238	8.34E-07	5.33E-03	4.31E-03	5.29E-03	**4.98E-03**	*5.78E-04*	20	1.0E-03
Np-237	<1.72E-04	1.19E-02	2.66E-02	3.22E-02	**2.36E-02**	*1.05E-02*	27.7	1.0E-03
Pu-238	6.80E-02	2.40E+00	2.24E+00	2.58E+00	**2.41E+00**	*1.71E-01*	7.8	1.0E-03
Pu-239	<1.32E-01	8.12E+00	6.21E+00	8.83E+00	**7.72E+00**	*1.36E+00*	20	1.0E-03
Pu-240	<4.55E-02	1.88E+00	1.47E+00	2.08E+00	**1.81E+00**	*3.11E-01*	20	1.0E-03
Pu-239/240	4.55E-02	1.00E+01	7.66E+00	1.09E+01	**9.52E+00**	*1.67E+00*	5	None

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes. .

Table 12-continued. Radiological Constituents for Tank 5F Composite Sample #2, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	Stdev	One Sigma %Uncertainty	Targeted Minimum Detection Limits
Pu-241	<4.33E-02	<1.37E+01	<8.51E+00	<8.69E+0	**<1.03E+01**		Upper Limit	1.0E-03
Pu-242	<2.29E-03	3.32E-04	2.70E-04	3.95E-04	**3.32E-04**	*6.25E-05*	20	1.0E-03
Pu-244	<1.07E-05	<1.17E-06	<7.43E-07	<9.46E-07	**<9.53E-07**		DL	1.30E-04
Am-241	<7.39E-02	7.12E+01	6.35E+01	7.16E+01	**6.88E+01**	*4.56E+00*	5	1.0E-03
Am-242m	<1.01E-03	1.90E-01	1.27E-01	1.73E-01	**1.63E-01**	*3.27E-02*	20	1.0E-03
Am-243	<5.72E-04	5.54E-01	4.77E-01	5.72E-01	**5.35E-01**	*5.02E-02*	6	1.0E-03
Cm-242	<8.33E-04	1.57E-01	1.05E-01	1.43E-01	**1.35E-01**	*2.71E-02*	20.02	None
Cm-243	<4.64E-04	<5.36E-01	<4.64E-01	<3.31E-01	**<4.44E-01**		Upper Limit	2.0E-02
Cm-244	<7.93E-03	2.93E+00	2.57E+00	2.88E+00	**2.79E+00**	*1.98E-01*	10.9	1.0E-03
Cm-245	<8.11E-06	<7.70E-04	<6.22E-04	<5.63E-04	**<6.52E-04**		Upper Limit	2.0E-02
Cm-246	<1.68E-05	<1.98E-03	<1.04E-03	<1.11E-03	**<1.38E-03**		Upper Limit	None
Cm-247	<4.82E-09	<2.81E-07	<1.74E-07	<1.03E-07	**<1.86E-07**		Upper Limit	1.3E-04
Bk-247	<5.36E-05	<3.13E-03	<1.94E-03	<1.15E-03	**<2.07E-03**		Upper Limit	None
Cm-248	<5.90E-07	<3.96E-05	<1.93E-05	<1.32E-05	**<2.40E-05**		Upper Limit	1.30E-04
Cf-249	<1.05E-04	<1.19E-02	<1.13E-02	<7.75E-03	**<1.03E-02**		MDA	5.0E-03
Cf-250	<1.22E-05	<3.46E-04	<3.68E-04	<1.52E-04	**<2.89E-04**		Upper Limit	None
Cf-251	<2.28E-04	<3.18E-02	<3.00E-02	<1.65E-02	**<2.61E-02**		MDA	None
Cf-252	<8.33E-04	<1.57E-01	<1.05E-01	<1.43E-01	**<1.35E-01**		Upper Limit	None

Table 13. Radiological Constituents for Tank 5F Composite Sample #3, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	Stdev	One Sigma %Uncert.	Targeted Minimum Detection Limits
Gross alpha	<2.34E+01	<2.09E+02	<6.58E+02	<3.68E+02	**<4.11E+02**		MDA	None
Non-volatile beta	<4.40E+01	2.85E+04	2.78E+04	2.76E+04	**2.80E+04**	*4.62E+02*	10	None
H-3	<9.06E-03	<9.28E-03	<9.53E-03	<1.83E-02	**<1.24E-02**		Upper limit	1.0E-01
C-14	<7.84E-04	<3.30E-03	<3.40E-03	<7.52E-04	**<2.48E-03**		MDA	1.0E-01
Ni-59	<9.23E-03	5.14E+00	3.82E+00	6.04E+00	**5.00E+00**	*1.11E+00*	10	9.0E-02
Ni-63	<8.56E-02	3.34E+02	4.64E+02	2.98E+02	**3.65E+02**	*8.73E+01*	20	1.0E-01
Co-60	<1.33E-02	7.16E+00	7.30E+00	6.89E+00	**7.12E+00**	*2.06E-01*	5	None
Se-79	No yield	5.05E-03	1.18E-02	1.18E-02	**9.56E-03**	*3.91E-03*	20	1.0E-03
Sr-90	<7.43E+0	1.17E+04	1.03E+04	1.25E+04	**1.15E+04**	*1.12E+03*	6	1.0E-03
Y-90	<7.43E+0	1.17E+04	1.03E+04	1.25E+04	**1.15E+04**	*1.12E+03*	6	1.0E-03
Zr-93	2.08E-02	3.07E+00	3.08E+00	3.25E+00	**3.13E+00**	*0.10*	20	1.0E-03
Tc-99	<9.86E-05	1.07E-02	9.10E-03	1.11E-02	**1.03E-02**	*1.05E-03*	6.4	1.0E-03
Pd-107**	<3.38E-04	3.46E-03	2.62E-03	3.55E-03	**3.21E-03**	*5.13E-04*	20	1.0E-03
I-129	<2.31E-06	2.50E-04	2.95E-04	1.38E-04	**2.28E-04**	*8.08E-05*	15	1.0E-04
Cs-135	8.10E-06	2.13E-03	2.44E-03	2.09E-03	**2.22E-03**	*1.92E-04*	20	5.0E-02
Cs-137	<3.23E-02	4.59E+02	4.77E+02	3.85E+02	**4.41E+02**	*4.92E+01*	5	1.0E-03
Ba-137m	<3.06E-02	4.34E+02	4.51E+02	3.64E+02	**4.17E+02**	*4.92E+01*	5	1.0E-03
Pm-147	<3.84E+00	<6.40E+02	<6.08E+02	<6.22E+02	**<7.31E+02**			None
Sm-151	<3.46E+0	8.51E+02	8.38E+02	8.29E+02	**8.39E+02**	*1.13E+01*	19.7	3.0E+00
Eu-154	<2.06E-02	3.15E+01	3.14E+01	3.22E+01	**3.17E+01**	*4.19E-01*	5.0	1.0E-03
Eu-155	<4.00E-02	7.12E+00	6.71E+00	5.95E+00	**6.59E+00**	*5.95E-01*	7.6	NA
Th-229**	No yield	<1.05E-06	1.22E-06	4.23E-05	**≤1.49E-05**		MDA/101	1.0E-03
Th-230**	No yield	5.72E-04	9.19E-04	3.88E-03	**1.79E-03**	*1.82E-03*	82.9	1.0E-03
Pa-231**	<1.44E-03	NA	<3.01E-04	<3.89E-04	**<3.45E-04**		DL	1.0E-03
U-232	No yield	<5.63E-06	<4.62E-06	<2.23E-05	**<1.09E-05**		DL	1.0E-03
U-233	<2.05E-03	<2.49E-04	<2.34E-04	<2.65E-04	**<2.49E-04**		DL	1.0E-03
U-234	<1.32E-03	4.67E-03	4.69E-03	4.30E-03	**4.55E-03**	*2.20E-04*	20	1.0E-03
U-235	<4.60E-07	2.09E-04	2.08E-04	1.96E-04	**2.04E-04**	*7.23E-06*	20	1.0E-04
U-236	<1.38E-05	2.59E-04	2.55E-04	2.36E-04	**2.50E-04**	*1.23E-05*	20	1.0E-03
U-238	8.34E-07	4.86E-03	4.82E-03	4.50E-03	**4.73E-03**	*1.97E-04*	20	1.0E-03
Np-237	<1.72E-04	2.78E-02	2.54E-02	2.34E-02	**2.55E-02**	*2.20E-03*	27.7	1.0E-03
Pu-238	6.80E-02	2.68E+00	3.07E+00	2.63E+00	**2.79E+00**	*2.44E-01*	7.6	1.0E-03
Pu-239	3.61E-02	8.80E+00	9.11E+00	8.42E+00	**8.78E+00**	*3.46E-01*	20	1.0E-03
Pu-240	<1.05E-02	2.06E+00	2.14E+00	1.97E+00	**2.06E+00**	*8.50E-02*	20	1.0E-03
Pu-239/240	4.55E-02	1.09E+01	1.13E+01	1.04E+01	**1.08E+01**	*4.28E-01*	5.7	NA

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes.
NA = Data not available.

Table 13-continued. Radiological Constituents for Tank 5F- Composite Sample #3, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	Stdev.	One Sigma %Uncert.	Targeted Minimum Detection Limits
Pu-241	<1.05E-02	<1.27E+01	<1.28E+01	<9.82E+00	**<1.18E+01**		Upper Limit	1.0E-03
Pu-242	<2.29E-03	4.28E-04	4.21E-04	3.83E-04	**4.11E-04**	*2.42E-05*	20	1.0E-03
Pu-244	<1.07E-05	<8.40E-07	<1.12E-06	<1.18E-06	**<1.05E-06**		DL	1.30E-04
Am-241	<7.39E-02	7.34E+01	7.70E+01	7.12E+01	**7.39E+01**	*2.95E+00*	5	1.0E-03
Am-242m	<1.01E-03	1.90E-01	1.42E-01	NA	**1.66E-01**	*3.38E-02*	22	1.0E-03
Am-243	<5.72E-04	5.54E-01	5.63E-01	NA	**5.59E-01**	*6.37E-03*	6.3	1.0E-03
Cm-242	<8.33E-04	1.57E-01	1.18E-01	NA	**1.37E-01**	*2.77E-02*	25.5	None
Cm-243	<4.64E-04	<5.68E-01	<5.00E-01	<1.40E+00	**<5.34E-01**		Upper Limit	2.0E-02
Cm-244	<7.93E-03	2.99E+00	3.04E+00	NA	**3.01E+00**	*3.50E-02*	12.2	1.0E-03
Cm-245	<8.11E-06	<1.50E-03	<7.34E-04	NA	**<1.11E-03**		Upper Limit	2.0E-02
Cm-246	<1.68E-05	<3.22E-03	<1.24E-03	NA	**<2.23E-03**		Upper Limit	None
Cm-247	<4.82E-09	<5.41E-07	<1.01E-07	NA	**<3.21E-07**		Upper Limit	1.3E-04
Bk-247	<5.36E-05	<5.99E-03	<1.13E-03	NA	**<3.56E-03**		Upper Limit	None
Cm-248	<5.90E-07	<5.36E-05	<1.39E-05	NA	**<3.38E-05**		Upper Limit	1.30E-04
Cf-249	<1.05E-04	<1.88E-02	<7.93E-03	NA	**1.34E-02**		MDA	5.0E-03
Cf-250	<1.22E-05	<1.02E-03	<2.23E-04	NA	**6.23E-04**		Upper Limit	None
Cf-251	<2.28E-04	<5.05E-02	<2.11E-02	NA	**3.58E-02**		MDA	None
Cf-252	<8.33E-04	<1.57E-01	<1.18E-01	NA	**1.37E-01**		Upper Limit	None

Table 14. Tank 5F Composite Sample #1: Challenging Radiological Constituents, µCi/g.

Analytes	Blank	Tank 5F Composite #1	One Sigma %Uncertainty	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<1.29E-02	MDA	1.0E-03
Cl-36	<2.43E-02	<2.87E-03	Upper limit	1.0E-03
K-40	<5.54E-04	<5.14E-03	MDA	1.0E-03
Nb-94	<5.33E-03	<2.87E-03	MDA	3.0E-03
Sn-126	<3.57E-02	<8.06E-01	MDA	1.0E-03
Sb-126	<1.14E-02	<1.09E-01	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.09E-01	MDA	1.0E-03
Eu-152	<2.95E-02	<2.11E-01	MDA	7.0E-03
Pt-193	<8.12E-04	<4.91E-04	MDA	1.0E-03
Ra-226**	No yield	< 9.78E-03	MDA	5.0E-03
Ac-227**	No yield	<5.23E-04	Upper limit	1.30E-04

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes.

Table 15. Tank 5F Composite Sample #2: Challenging Radiological Constituents, µCi/g.

Analytes	Blank	Tank 5 F Composite # 2	One Sigma %Uncertainty	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<1.31E-02	MDA	1.0E-03
Cl-36	<2.43E-02	<5.05E-03	Upper limit	1.0E-03
K-40	<5.54E-04	<3.71E-03	MDA	1.0E-03
Nb-94	<5.33E-03	<2.11E-03	MDA	3.0E-03
Sn-126	<3.57E-02	<8.51E-01	MDA	1.0E-03
Sb-126	<1.14E-02	<1.04E-01	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.04E-01	MDA	1.0E-03
Eu-152	<2.95E-02	<1.47E-01	MDA	7.0E-03
Pt-193	<8.12E-04	<4.24E-04	MDA	1.0E-03
Ra-226**	No yield	<6.81E-04	MDA	5.0E-03
Ac-227**	No yield	<9.50E-05	Upper limit	1.30E-04

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes.

Table 16. Tank 5F Composite Sample #3: Challenging Radiological Constituents, µCi/g.

Analytes	Blank	Tank 5F Composite No. 3	One Sigma %Uncert.	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<1.94E-02	MDA	1.0E-03
Cl-36	<2.43E-02	<8.42E-03	Upper limit	1.0E-03
K-40	<5.54E-04	<8.24E-04	MDA	1.0E-03
Nb-94	<5.33E-03	<1.14E-03	MDA	3.0E-03
Sn-126	<3.57E-02	<1.18E+00	MDA	1.0E-03
Sb-126	<1.14E-02	<1.50E-01	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.50E-01	MDA	1.0E-03
Eu-152	<2.95E-02	<2.11E-01	MDA	7.0E-03
Pt-193	<8.12E-04	<1.56E-03	MDA	1.0E-03
Ra-226**	No yield	<2.44E-03	MDA	5.0E-03
Ac-227**	No yield	<1.65E-04	Upper limit	1.30E-04

** While analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes.

4.0 CONCLUSIONS

Tank 5F composite samples were analyzed for radiological, elemental and chemical constituents. Where analytical methods yielded additional contaminants other than those requested by the customer, these results are also reported.

The target detection limits for isotopes analyzed were based on customer desired detection limits as specified in the technical task request documents. While many of the target detection limits, as specified in the technical task request and task technical and quality assurance plans were met for the species characterized for Tank 5F, some were not met. In a number of cases, the relatively high levels of radioactive species of the same element or a chemically similar element precluded the ability to measure some isotopes to low levels. The isotopes whose minimum detection limits were not met in all cases included the following: Al-26, Sn-126, Sb-126, Sb-126m, Eu-152 and Cf-249. The TTR allows that while the analyses of these isotopes is needed, meeting the detection limits for these isotopes is a lower priority than meeting detection limits for the other specified isotopes. However, SRNL, in conjunction with the customer, reviewed the few cases where the detection limit goals were not met and determined that the impacts were negligible. [SRR-CWDA-2012-00013]

5.0 QUALITY ASSURANCE

The Task Technical and Quality Assurance Plan details the planned activities and associated quality assurance implementing procedures for the characterization of Tank 5F (TTQAP, SRNL-RP-2010-01695, Rev. 1). Laboratory Notebook SRNL-NB-2011-00125, WSRC-NB-2001-00142 and various AD notebooks contain the experimental data. Other relevant QA documents include the Technical Task Request (HLE-TTR-2010-004, Rev 7), Tank 5 Sampling and Analysis Plan-SRR-LWE-2010-00285, Rev. 1, November 15, 2010 and Tank 5 Composite Sample Volumetric Proportions, SRR-CWDA-2011-00067, Rev.1 April 20, 2011.

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APPENDIX A-0: AD Tank 5F Characterization Tracking Numbers

Analytes	Method (s)	SRNL AD Tracking Number (LIMS)
Total Alpha	Rad Screen	300286107-300286121
Non-volatile Beta	Rad Screen	300286107-300286121
Al-26	GAMMA SPEC Cs REMOVED	300286107-300286120
Cl-36	Cl-36	300292253-300292262
K-40	K-40	300293878- 300293896
Sr-90	Sr-90	300286107-300286120
Pu-238	Pu-238/241	300286107-300286121
Pu-241	Pu-238/241	300286107-300286121
Cs-137	GAMMA SPEC	300286107-300286121
U-232	U-232	300286107-300286121
U-233	U-233, U-234, U-235, U-236	300286107-300286116
U-234	U-233, U-234, U-235, U-236	300286107-300286116
U-235	U-233, U-234, U-235, U-236	300286107-300286116
U-236	U-233, U-234, U-235, U-236	300286107-300286116
U-238	ICP-MS	300286107-300286116
Co-60	GAMMA SPEC Cs REMOVED	300286107-300286120
Sb-126	GAMMA SPEC Cs REMOVED	300286107-300286120
Sn-126	GAMMA SPEC Cs REMOVED	300286107-300286120
Eu-152	GAMMA SPEC Cs REMOVED	300286107-300286120
Eu-154	GAMMA SPEC Cs REMOVED	300286107-300286120
Eu-155	GAMMA SPEC Cs REMOVED	300286107-300286120
Am-241	Gamma Spec.	300286107-300286120
Pu-239	Pu-242/244	300286107-300286116
Pu-240	Pu-242/244	300286107-300286116
Pu-242	Pu-242/244	300286107-300286116
Pu-244	Pu-242/244	300286107-300286116
Pu-239/240	Pu-TTA	300286107-300286121
PM-147/ SM-151	Pm-147/Sm-151	300286107-300286121
Tritium	TRITIUM	300286123-300286135
Ni-59	Ni-59,63	300286123-300286135
Ni-63	Ni-59,63	300286123-300286135
Tc-99	Tc-99	300294396-300294405
I-129	I-129	300288706-300288716
Cs-135	Cs-135	300286107-300286120
Carbon-14	Carbon-14	300294468-300294482 300294509-300294521
Se-79	Se-79	300292711- 300292720
Zr-93	Zr-93	300286107-300286121
Pd-107	Pd-107	300291713- 300291722
Pt-193	Pt-193	300286107-300286120

APPENDIX A-0: AD Tank 5F Characterization Tracking Numbers-Continued

Analytes	Method (s)	SRNL AD Tracking Number (LIMS)
Nb-94	Nb-94	300286107-300286121
Am/Cm	Am/Cm	300290171-300290180
Ra-226	Ra-226	300294754- 300294773
Th-229/230	Th-229/230	300291231-300291248
Ac-227	Ac-227	300291231-300291248
Pa-231	Pa-231	300293293-300293311
Np-237	ICP-MS & Np-239 decay correction	300286107-300286121
Hg	CVAA Hg	300286123-300286135
Se	AASe	300286123-300286137
As	AASe	300286123-300286137
Cations	ICP-MS-PF digestions	300286107- 300286121
Cations	ICP-MS-AQR digestions	300286123-300286136
Cations	ICP-ES-AQR digestions	300286123-300286137
Cations	ICP-ES-PF digestions	300286107-300286120
Anions	IC- Leachate analysis	300287323-300287337

APPENDIX A-1: Chemical Composition of Reference Tank 8 Simulant

	Analytical Results for Tank 8 Simulant Sludge	Standard deviation	Nominal Recipe for Tank 8 Simulant Sludge	Percent Relative Deviation
	Average			%RD
Constituent	wt. %		wt. %	
Al	9.5	*0.06*	9.0	0.47
Ba	0.25	*0.00*	0.24	5.3
Ca	2.3	*0.01*	2.1	1.2
Cr	0.26	*0.00*	0.24	4.2
Cu	0.13	*0.01*	0.13	0.20
Fe	27	*0.20*	24	0.22
*K	0.038	0.00	0.005	38
Mg	0.13	*0.00*	0.12	2.5
Mn	2.8	*0.02*	2.7	1.9
*Na	6.1	0.06	7.4	4.8
*Ni	2.8	0.02	2.8	0.36
Pb	0.16	*0.01*	0.21	11
Si	0.88	*0.01*	0.76	0.28
Sr	0.093	*0.00*	0.09	3.7
Zn	0.31	*0.02*	0.27	8.8

* Aqua regia digestion data; all other data from Peroxide fusion. . Note that the moisture content of the Tank 8 simulant sludge (89.2%) was taken into account when calculating the weight percent values.

APPENDIX A-2: Chemical Composition of Analyzed Reference Glass

	Analytical Results for Reference Glass (ARG)	Standard deviation	Nominal Recipe for Reference Glass (ARG)	Percent Relative Deviation
	Average			%RD
Constituent	wt. %		wt. %	
Al	2.5	0.05	2.5	0.27
B	2.5	0.05	2.7	2.1
Ba	0.080	0.00	0.079	0.29
Ca	1.6	0.01	1.02	10
Cr	0.066	0.00	0.064	0.87
*Cu	0.0025	0.00	0.003	4.7
Fe	10	0.24	9.8	0.59
*K	2.3	0.08	2.3	0.52
Li	1.5	0.02	1.5	0.00
Mg	0.53	0.02	0.52	0.66
Mn	1.4	0.04	1.5	0.35
*Na	8.6	0.29	8.5	0.31
*Ni	0.83	0.04	0.83	0.06
*P	0.090	0.00	0.11	4.9
Si	23	0.97	22	0.62
Sr	0.010	0.00	0.003	27
Ti	0.63	0.07	0.69	2.4
Zn	0.046	0.00	0.016	24

* Aqua regia digestion data; all other data from Peroxide fusion.

Natural cadmium concentration by MS Tank 5F COMPOSITE 1

The main Cd isotopes used for calculations are masses 110, 111, 112, 114 and 105 (contribution from Pd-105).

1. ([Cd-110] – [(Pd-105)] *{fission yield for mass 110*110/fission yield for mass 105*105}])/ Cd-110 stable abundance of 12.49% or 0.1249.
2. Cd-111/0.128
3. Cd-112/0.2413
4. Cd-114/0.2873
5. Cd-116/0.0758

Values obtained through steps 1 and 5 above were averaged.

Typical Cd calculations for Tank 5F Composite 1-1

1. ([17.8] – [22.8)] *{0.025*110/0.96*105}])/0.1249.
   = [17.8-(22.8*0.0273)]/0.1249
   =137.53 ug/g or 0.1375 mg/g
2. 22.3/0.128 = 174.2 ug/g or 0.174 mg/g
3. 29.9/0.2413 = 123.9 ug/g or 0.1239 m/g
4. 21.1/0.2873 = 73.44 ug/g or 0.073 mg/g
5. 8.5/0.0758 = 112.14 or 0.1121 mg/g
6. Average of all values comes to 0.12
   Using the similar approach the calculations were performed for Tank 5 composite sample 2 and 3. Results are summarized in table below for Cd by MS and Cd by ICP-ES.

	300286107 COMPOSITE 1-1-PF, mg/g	300286108 COMPOSITE 1-2-PF, mg/g	300286109 COMPOSITE 1-3-PF, mg/g	Averages
Cd by ICP-MS	0.12	0.13	0.11	0.12
Cd by ICP-ES	0.081	0.092	0.097	0.090
%RD	39	34	13	**29**

The average percent relative deviation for cadmium concentrations based on ICP-MS and ICP-ES are about 29%.

Natural barium concentration by MS

Main stable Ba isotopes used for calculations are masses 136, 137 and 138

[Sum of MS signals from masses 136+137 +138]]

Tank 5F Composite No. 1, unit of ug/g sample
Run 1 14.4+297+1960 = 2,271.4 = 2.271 mg/g
Run 2 13.9 +301+1880 = 2,194.9= 2.195 mg/g
Run 3 0+ 261 + 1660 = 1921 = 1.921 mg/g

	300286107 COMPOSITE 1-1-PF, mg/g	300286108 COMPOSITE 1-2-PF, mg/g	300286109 COMPOSITE 1-3-PF, mg/g	Averages
Ba by ICP-MS	2.3	2.2	1.9	2.1
Ba by ICP-ES	1.7	2.0	2.1	1.9
%RD	26.5	10.8	7.5	**9.9**

The average percent relative deviation for barium concentrations based on ICP-MS and ICP-ES is over 9.9%.

Natural Lanthanum concentration by MS

Main stable La isotopes used for calculations is mass 139

[Sum of MS signals from masses 139]
Tank 5F Composite No. 1, unit of ug/g sample

Run 1 1540 ug/g = 1.540 mg/g
Run 2 1480 ug/g = 1.48 mg/g
Run 3 1370 = 1.370 mg/g

	300286107 COMPOSITE 1-1-PF, mg/g	300286108 COMPOSITE 1-2-PF, mg/g	300286109 COMPOSITE 1-3-PF, mg/g	Averages
La by ICP-MS	1.5	1.5	1.4	1.5
La by ICP-ES	1.3	1.4	1.5	1.4
%RD	15	7.0	5.7	**4.9**

The average percent relative deviation for lanthanum concentrations based on ICP-MS and ICP-ES is 4.9%.

APPENDIX A-3: Analytical Results for Challenging Radiological Constituents

Challenging Radiological Constituents for Tank 5F Composite Sample #1, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	One Sigma %Uncert.	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<1.29E-02	<2.32E-02	<2.03E-02	**<1.88E-02**	MDA	1.0E-03
Cl-36	<2.43E-02	<3.76E-02	<7.21E-03	<2.87E-03	**<1.59E-02**	Upper limit	1.0E-03
K-40	<5.54E-04	<5.14E-03	<6.17E-03	<7.25E-03	**<6.19E-03**	MDA	1.0E-03
Nb-94	<5.33E-03	no yield	<2.87E-03	<4.85E-02	**<2.57E-02**	MDA	3.0E-03
Sn-126	<3.57E-02	<8.74E-01	<8.06E-01	<1.23E+00	**<9.68E-01**	MDA	1.0E-03
Sb-126	<1.14E-02	<1.09E-01	<1.58E-01	<1.50E-01	**<1.39E-01**	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.09E-01	<1.58E-01	<1.50E-01	**<1.39E-01**	MDA	1.0E-03
Eu-152	<2.95E-02	<2.23E-01	<2.11E-01	<2.20E-01	**<2.18E-01**	MDA	7.0E-03
Ra-226	No yield	No yield	No yield	< 9.78E-03	**<9.78E-03**	MDA	5.0E-03
Pt-193	<8.12E-04	<6.37E-04	<6.95E-04	<4.91E-04	**<6.08E-04**	MDA	1.0E-03
Ac-227	No yield	<5.23E-04	<4.73E-02	<7.07E-04	**<1.62E-02**	Upper limit	1.30E-04

Challenging Radiological Constituents for Tank 5F Composite Sample #2, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	One Sigma %Uncert.	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<1.73E-02	<1.31E-02	<1.87E-02	**<1.64E-02**	MDA	1.0E-03
Cl-36	<2.43E-02	<1.86E-02	<5.05E-03	<3.89E-02	**<2.09E-02**	Upper limit	1.0E-03
K-40	<5.54E-04	<3.71E-03	<4.00E-03	<4.86E-03	**<4.19E-03**	MDA	1.0E-03
Nb-94	<5.33E-03	No yield	<2.11E-03	<4.58E-02	**<2.40E-02**	MDA	3.0E-03
Sn-126	<3.57E-02	<1.27E+00	<8.51E-01	<1.24E+00	**<1.12E+00**	MDA	1.0E-03
Sb-126	<1.14E-02	<1.57E-01	<1.04E-01	<1.54E-01	**<1.38E-01**	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.57E-01	<1.04E-01	<1.54E-01	**<1.38E-01**	MDA	1.0E-03
Eu-152	<2.95E-02	<2.24E-01	<1.47E-01	<2.26E-01	**<1.99E-01**	MDA	7.0E-03
Pt-193	<8.12E-04	<1.78E-03	<2.74E-03	<4.24E-04	**<1.65E-03**	MDA	1.0E-03
Ra-226	No yield	<6.81E-04	<8.63E-04	No yield	**<7.72E-04**	MDA	5.0E-03
Ac-227	No yield	<1.34E-04	<6.08E-04	<9.50E-05	**<2.79E-04**	Upper limit	1.30E-04

Challenging Radiological Constituents for Tank 5F Composite Sample #3, µCi/g.

Analytes	Blank	Run 1	Run 2	Run 3	Average	One Sigma %Uncert.	Targeted Minimum Detection Limits
Al-26	<9.77E-03	<2.01E-02	<1.98E-02	<1.94E-02	**<1.98E-02**	MDA	1.0E-03
Cl-36	<2.43E-02	<1.22E-02	<8.42E-03	<1.81E-02	**<1.29E-02**	Upper limit	1.0E-03
K-40	<5.54E-04	<6.62E-03	<8.24E-04	<9.41E-04	**<2.80E-03**	MDA	1.0E-03
Nb-94	<5.33E-03	no yield	<1.14E-03	< 3.89E-02	**<2.00E-02**	MDA	3.0E-03
Sn-126	<3.57E-02	<1.18E+00	<1.18E+00	<1.18E+00	**<1.18E+00**	MDA	1.0E-03
Sb-126	<1.14E-02	<1.53E-01	<1.50E-01	<1.53E-01	**<1.52E-01**	MDA	1.0E-03
Sb-126m	<1.14E-02	<1.53E-01	<1.50E-01	<1.53E-01	**<1.52E-01**	MDA	1.0E-03
Eu-152	<2.95E-02	<2.23E-01	<2.11E-01	<2.20E-01	**<2.18E-01**	MDA	7.0E-03
Pt-193	<8.12E-04	<1.56E-03	<3.48E-03	<6.35E-03	**<3.80E-03**	MDA	1.0E-03
Ra-226	No yield	<2.44E-03	<9.01E-03	No yield	**<5.73E-03**	MDA	5.0E-03
Ac-227	No yield	<1.65E-04	<3.04E-04	<1.35E-03	**<6.05E-04**	Upper limit	1.30E-04

APPENDIX A-4: "As-received" and Composited Sample Bulk Densities, g/mL

Tank 5F Sample ID	Run 1	Run 2	Run 3	Run 4	"As-received" Sample Density Average	Comments
5-C1a	0.91	NA	NA	NA	**0.91**	Insufficient sample; Single run
5-C1b	1.296	1.275	1.133	1.134	**1.21** ± *0.09*	
5-C1c	1.017	1.081	0.876	0.890	**0.97** ±*0.10*	
5-C2a	0.83	0.87	NA	NA	**0.85** ±*0.02*	
5-C2b	0.831	0.960	0.854	0.848	**0.87** ± *0.06*	
5-C2c	0.773	0.930	0.816	0.836	**0.84** ±*0.07*	
5-C3a	1.04	1.06	NA	NA	**1.05** ±*0.01*	
5-C3b	1.211	1.227	1.128	1.128	**1.17** ± *0.05*	
5-C3c	0.896	0.796	0.857	0.867	**0.85** ±*0.04*	
5-A1a-U	1.46	1.46	1.45	NA	**1.45** ±*0.01*	
5 -B1a-U	1.48	1.47	1.45	NA	**1.47** ±*0.01*	
5-A2a-BM	1.14	1.29	NA	NA	**1.22** ±*0.10*	
5 -A3a-BM	1.08	1.11	NA	NA	**1.09** ±*0.02*	
5 –B2a-MD	1.336	1.357	1.297	1.336	**1.33** ±*0.03*	
5 –B3a-BM	1.094	0.956	1.112	NA	**1.05** ±*0.09*	
					Composite Sample Density Average	
Composite sample #1	1.40	1.40	1.43	NA	**1.41** ±*0.01*	
Composite sample #2	1.37	1.32	1.32	NA	**1.34** ±*0.03*	
Composite sample #3	1.30	1.31	1.32	NA	**1.31** ±*0.01*	

APPENDIX A-5: Tank 5F Homogenized Discrete Sample, Composited Sample Bulk Densities (g/mL) and Composited Sample weight percent solids

Tank 5F Sample ID	Run 1	Run 2	Run 3	Homogenized Sample Density Average, g/mL
5-C1a	1.08	1.00	1.04	**1.04** ±*0.04*
5-C1b	1.120	1.100	1.097	**1.11** ±*0.01*
5-C1c	0.981	0.999	0.868	**0.95** ±*0.07*
5-C2a	1.06	1.01	0.99	**1.02** ±*0.04*
5-C2b	0.993	0.972	0.939	**0.97** ±*0.03*
5-C2c	0.978	0.915	0.898	**0.93** ±*0.04*
5-C3a	1.13	1.13	1.06	**1.11** ±*0.04*
5-C3b	1.08	0.97	1.03	**1.03** ±*0.06*
5-C3c	0.98	0.93	0.97	**0.96** ±*0.03*
5-A1a-U	1.43	1.45	1.42	**1.43** ±*0.02*
5 -B1a-U	1.43	1.43	1.45	**1.44** ±*0.01*
5-A2a-BM	1.26	1.26	1.25	**1.25** ±*0.01*
5 -A3a-BM	1.05	1.04	1.04	**1.05** ±*0.00*
5 –B2a-MD	1.14	1.14	1.19	**1.16** ±*0.03*
5 –B3a-BM	1.21	1.26	1.26	**1.24** ±*0.03*
				Composite Sample Density Average
Composite sample #1	1.40	1.40	1.43	**1.41** ±*0.01*
Composite sample #2	1.37	1.32	1.32	**1.34** ±*0.03*
Composite sample #3	1.30	1.31	1.32	**1.31** ±*0.01*
				Composite Sample Wt%; Average
Composite sample #1	95.59	95.91	95.73	**95.7**±*0.2*
Composite sample #2	96.53	96.59	97.08	**96.7**±*0.3*
Composite sample #3	96.00	96.70	96.41	**96.4**±*0.4*

APPENDIX A-6: Tank 5F discrete and composite samples in stock.

	Residuals Tank 5F sample, g
COMPOSITE 1	No sample left in stock
5-A1a-U	No sample left in stock
5-B2a-MD	55
5-C1a	18
5-C2b	45
5-C3c	55
COMPOSITE 2	35
5-A2a-BM	230
5-B3a-BM	125
5-C1b	62
5-C2c	72
5-C3a	92
COMPOSITE 3	37
5-A3a-BM	168
5-B1a-U	2
5-C1c	35
5-C2a	40
5-C3b	45

APPENDIX B: Summary of Analytical Methods

Aqua Regia Digestions (AQR)

Samples were digested according to procedure L16.1, ADS-2226. In a typical digestion, ~0.5 g of Tank 5F composite sample was placed into a Teflon® digestion vessel. Then, 9 mL (hydrochloric acid) HCl, and 3 mL (nitric acid) HNO_3 were added to the Teflon® vessel. The Teflon® vessel was sealed and heated for a period of no more than 4 hours at 115 ºC. The sample was then cooled and diluted to 50 mL. Three samples, in total, from each composite sample were digested by aqua regia.

Sodium Peroxide/Hydroxide Fusions (PF)

Samples were digested according to procedure L16.1, ADS 2502. In a typical digestion, ~2 grams of composite Tank 5F sample was placed into a nickel (Ni) crucible with a known weight. The material in the crucible was dried until two consecutive weights were within ±0.02 grams. The remaining material in the crucible was fused at 675 ºC using a mixture of sodium peroxide (6.0 grams) and sodium hydroxide (4.0 grams). After the sample was cooled, water was added to dissolve the fused material and the solution was acidified by the addition of 25 mL HCl. The sample was diluted to 100 mL. Three samples, in total, from each composite sample were digested by sodium peroxide fusion.

Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-ES)

Samples are diluted as necessary to bring 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 is 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.

Ion Chromatography for Anions (IC-Anions)

For IC Anions, samples were diluted with a carbonate/bicarbonate diluent as necessary to bring analytes to within instrument calibration. A 3-point calibration curve is run daily on the instrument with concentrations of 10, 25 and 50 µg/mL.

Atomic Absorption Spectroscopy (AA)

Arsenic, selenium, and mercury are analyzed by AA. The mercury was determined using the cold vapor technique. Samples were diluted as necessary to bring analytes within the instrument calibration range. An instrument calibration is performed daily with a blank and two or three point standard. The standard is run for each element at the beginning of the day, after each five sample runs and at the end of the day.

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

Samples were run concurrently with a laboratory control standard (LCS) containing V, Co, As, Sr, Mo, Ru, Ag, Cd, Sb, Cs, Ba, La, Eu, Ho, Yb, Tl, Pb, Th, and U. This LCS 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:

Co mass 59
^{99}Tc mass 99. Subject to interference when Ru is present in the sample.
Ag mass 107, 109
Pb mass 206, 207,208
Sn mass 117, 118,120, 122, 124
^{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.

Gross Alpha/Gross Beta

The solid material was too concentrated to be analyzed directly. Aliquots of peroxide fusion dissolution were added to liquid scintillation cocktail and analyzed for gross alpha and gross beta activity using liquid scintillation analysis. Alpha/beta spillover was determined for each aliquot analyzed, and subsequently used for accurately determining alpha and beta activity, via the addition of a known amount of plutonium to an identical aliquot of each sample.

Other Specialty Separations and Preparations

Ni-59/63

Aliquots of dissolution from the aqua regia digestion were aliquoted and spiked with an elemental nickel carrier. The nickel species were extracted from the matrix using dimethylglyoxime (DMG) based extractant. Ni-59 concentrations were measured using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. Ni-63 concentrations were measured by liquid scintillation analysis. Elemental nickel carrier yields were measured by ICP-ES, and were used to correct the radioactive nickel species' analyses for any nickel losses from the radiochemical separations.

Cs-137/Cs-134

Aliquots of peroxide fusion dissolution and AQR were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure Cs-137 and Cs-134.

Sr-90

Aliquots of peroxide fusion and AQR dissolutions 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.

Pm-147/Sm-151

Aliquots of peroxide fusion and AQR dissolution were spiked with an elemental samarium carrier. The promethium and samarium species were extracted from the matrix using a combination of Octylphenyl-N,N-di-isobutyl carbamoylphosphine oxide/tri-n-butyl phosphate (one CMPO/TBP) and di(2-ethylhexyl) orthophosphoric acid (one HDEHP based). Sm-151 and Pm-147 concentrations were measured by liquid scintillation analysis. The matrix was high in Sm-151, but the short-lived Pm-147 component of the material had decayed below noise levels of the analysis. Elemental samarium carrier yields were measured by neutron activation analysis, and were used to correct the analyses for any samarium losses from the radiochemical separations. The separation was designed to extract both Sm and Pm together; a Pm spike was run with the samples to confirm this.

Na-22, Al-26, Co-60, Nb-94, Rh-106, Ru-106, Sb-125, Sb-126, Sn-126, Sb-126m, Te-125m, Ce-144, Pr-144, Eu-152, Eu-154, Eu-155, Am-241, Ra-228, Ac-227

Aliquots of peroxide fusion were subjected to a Cs-removal process utilizing Bio Rad AMP-1 resin. The Cs-removed digestates were analyzed by coaxial high purity germanium spectrophotometers to measure the gamma-emitting radionuclides listed above.

Pu-238, 239/240, 241

Aliquots of peroxide fusion and AQR dissolutions 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 and Pu-239/240 using PIPs detectors. Each separation was traced based on the Pu-236 recovery. Aliquots of sample were also subjected to Cs-removal with Bio-Rad Ammonium Molybdophosphate (AMP) resin and extracted using TEVA columns (TEVA Brand name for one of Eichrom's resins). The Pu-containing extracts were measured by liquid scintillation analysis to determine Pu-241 concentration.

Am-241, 242m, 243, Cm-242, 243, 244, 245, 247, 248, Cf-249, 251, 252

Samples of composite Tank 5F materials were digested using a sodium peroxide fusion. Additionally, a matrix blank and matrix blank spiked with Am-241 and Cm-244 were prepared using Tank 8 simulated sludge. The americium, curium and californium species were extracted from aliquots of peroxide fusion using a CMPO/TBP based solid phase extractant and purified further with an HDEHP based solid phase extractant. Am-241, 243, Cm-243, 245, 247, Cf-249 and 251 concentrations were measured using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. Am-242m, Cm-242, 244, 248, Cf-252 concentrations were measured using passivated, implanted, planar silicon (PIPS) alpha spectrometers. Some of the Am, Cm and Cf isotopes were also measured using ICP-MS. Am-241 quantities had been measured from the cesium removed gamma analyses, all Am, Cm, and Cf results were traced with the Am-241 present in the sample matrix.

U-232

Aliquots of peroxide fusion were spiked with a U-233 radioactive tracer, additional aliquots were run through the method with no tracer added. Uranium was extracted from the matrix using two stages of a diamyl, amylphosphonate (DAAP)-based solid phase extraction and purified further via co-precipitation with cerium. U-232, U-233, and U-238 activities were measured using passivated, implanted, planar silicon (PIPS) alpha spectrometers. The Tank 5 samples were so high in U-234, the U-233 tracers (which have the same alpha energy as U-234) were swamped out. U-232/U-238 activity ratios were generated and were multiplied to U-238 activities measured with the ICP-MS to determine U-232 activities in the samples.

Cs-135 Separation for MS

Aliquots of dissolved material (alkali fusion digestion) were purified using a solvent-solvent caustic side solvent extraction-based (CSSX) extraction system. The purified Cs-containing aliquots were analyzed using ICP-MS to measure Cs-135/Cs-133 mass ratios. The Cs-133 and Ba-corrected Cs-135 ratios from the aliquots of separated material were used along with the associated Cs-133 ICP-MS result from the analysis of non-separated material to obtain a value for Cs-135. The Cs-135 result was then converted from ug/g to uCi/g using the specific activity of Cs-135.

Np-237

Aliquots of peroxide fusion dissolution were spiked with Np-239 and then purified with a quaternary amine based solid phase extraction. The purified aliquots were analyzed by low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers to yield the Np-239 recoveries and by the ICP-MS to measure Np-237. The Np-237 values were corrected with the decay-corrected Np-239 recoveries.

Tritium

Aliquots of dissolution from the aqua regia digestion were subjected to tritium separations via steam distillation, and aliquots of the tritium-containing distillate were analyzed by liquid scintillation analysis.

Se-79

Samples of composite Tank 5F materials were weighed out, spiked with an elemental selenium carrier and digested in concentrated acid. The selenium species were extracted from the matrix using a combination of resin decontamination, selenium metal precipitation, and TBP-based liquid-liquid extractions. The purified selenium products were analyzed by liquid scintillation to measure Se-79, and by neutron activation analysis to measure elemental selenium carrier yields. The selenium carrier yields were used to correct the Se-79 analyses for any selenium losses from the radiochemical separations.

Tc-99

Tank 5F composite samples were digested in a combination of concentrated nitric and hydrochloric acids. Several matrix blanks were prepared using Tank 8 simulated sludge spiked with a Tc-99 standard. The dissolutions were subjected to a number of resin

treatments to reduce dose prior to removal from the shielded cells. The treated samples were then spiked with Tc-99m and the technetium species were extracted from the matrix using an Aliquat-336 based solid phase extractant. Tc-99 concentrations were measured by liquid scintillation analysis. Tc-99m yields were measured with a NaI-well gamma spectrometer, and were used to correct the Tc-99 analyses for any technetium losses from the radiochemical separations. The average recovery of the Tc-99 spiked matrix blank was applied to the entire set of samples to correct for any losses from the decontamination steps used in the shielded cells.

Ra-226

Tank 5F composite samples were digested using a sodium peroxide fusion. Each replicate was prepared in duplicate with the duplicate containing a Ra-224 tracer. Additionally, a matrix blank and matrix spiked blank were prepared using Tank 8 simulated sludge. The Ra-226 was extracted from the matrix using a combination of resin decontamination and ion exchange. The purified Ra-226 was sealed in polypropylene tubes and stored for several daughter Rn-222 half-lives. The Ra-226 progeny daughter isotope Pb-214 was then analyzed for using a high purity germanium well gamma ray spectrophotometer and results were corrected for the tracer Ra-224 recoveries.

Pa-231

Tank 5F composite samples were digested using a sodium peroxide fusion. Each replicate was prepared in duplicate with the duplicate containing a Pa-233 tracer. Additionally, a matrix blank and matrix spiked blank were prepared using Tank 8 simulated sludge. The dissolutions were decontaminated with AMP and quaternary amine based resins. Protactinium species were then extracted from the matrix using a CMPO/TBP based extractant. Pa-233 tracer concentrations were measured using high purity germanium spectrometers to determine separation yields. Pa-231 was measured using the ICP-MS. The Pa-233 tracer yields were decay corrected and then used to correct the Pa-231 analyses for any losses from the radiochemical separations.

I-129

Tank 5F composite samples were dissolved in concentrated acid with an added KI carrier. A matrix blank and matrix blank containing an I-129 spike were also prepared using Tank 8 simulated sludge. Actinide and AMP resins were then added to the mixture to facilitate removal of interfering isotopes. Sodium sulfite is added to the material to reduce the iodine. Silver nitrate is added to the solution to precipitate the iodine as AgI, which is separated via filtration. The filtrate is analyzed for I-129 content using low energy photon/x-ray, thin-windowed, semi-planar, high purity germanium spectrometers. Elemental iodine yields were measured by neutron activation analysis, and were used to correct the I-129 analyses for any iodine losses from the radiochemical separation.

C-14

The solid material was used for the C-14 separation and analysis. The material was added to a mixture of sodium hydroxide, and sodium carbonate/sodium hydroxide. A series of oxidation and reduction steps designed to liberate C-14 containing carbon dioxide were

carried out, which selectively trapped the C-14 in a basic solution. Finally, C-14 containing carbon dioxide was captured in Carbosorb E and measured by liquid scintillation analysis.

Th-229/230, Ac-227

Tank 5F composite samples were digested using a sodium peroxide fusion. Each replicate was prepared in duplicate with the duplicate containing a Th-229 tracer. Additionally, a matrix blank and matrix spiked blank were prepared using Tank 8 simulated sludge. The matrix spiked blank contained both a Th-228 and Th-229 spike. Thorium was extracted from the matrix using two stages of a quaternary amine based solid phase extraction and purified further via co-precipitation with cerium. Th-227, Th-229 and Th-230 concentrations were measured using passivated, implanted, planar silicon (PIPS) alpha spectrometers. The Th-229 tracer yields were used to correct the various analytes analyses for any thorium losses from the radiochemical separations. Ac-227 activities were calculated from the Th-227 results

Cl-36

Tank 5F composite samples were dissolved in triplicate using a mixture of concentrated hydrochloric and nitric acids, along with a Tank 8 simulated sludge blank and a Cl-36 spiked simulated sludge blank. The samples were contacted with resins to reduce activity before removal from the shielded cells. The samples were then contacted with resins again to further decontaminate the samples, steam distilled, and finally the Cl-36 was precipitated as AgCl. The AgCl precipitate was counted for total beta using a gas flow proportional counter. The precipitate was then analyzed by NAA to determine Cl chemical yields to trace the separation.

K-40

Tank 5F composite samples were digested using a sodium peroxide fusion. The samples were subjected to a series of decontamination steps to remove Cs-137, Sr-90 as well as a number of trivalent, tetravalent and hexavalent radionuclides. The decontaminated solutions were then analyzed with high purity germanium detectors.

Pt-193

Aliquots of Tank 5F peroxide fusion dissolutions were spiked with stable Pt and then purified with a quaternary amine based solid phase extraction. The purified aliquots were analyzed by low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers to measure Pt-193m and by the ICP-ES to measure stable Pt recoveries. The Pt-193m values were corrected with the stable Pt recoveries.

Nb-94

Aliquots of peroxide fusion dissolution were spiked with Nb-95 and then purified with a anion exchange. The purified aliquots were analyzed by high purity germanium spectrometers to measure Nb-94 and to measure Nb-95 tracer recoveries. The Nb-94 values were corrected with the stable Nb-95 recoveries.

Pd-107

Tank 5F composite samples were dissolved in triplicate using hot aqua regia along with a Tank 8 simulated sludge blank and a Pd spiked simulated sludge blank. The samples were contacted with resins to reduce activity before removal from the shielded cells. Pd was extracted from the samples using a DMG based extractant. Pd-107 levels were measured using the ICP-MS, and the results were yielded from sample stable Pd recoveries as measured by the ICP-MS

Zr-93

Zr was extracted from aliquots of peroxide fusion dissolution. Zr-93 levels were measured using the ICP-MS, and the results were yielded from sample stable Zr recoveries as measured by the ICP-MS.

Weight Fraction Solids Measurement

The weight percent total solids in each Tank 5F sample were measured in the Shielded Cells using a conventional drying oven at 110 °C. An aliquot of each composite sample was placed in a container. The container was placed in the oven. The weights of the dried sample were checked periodically over 72 hours until two consecutive weights yielded comparable results. The weight fraction solid was calculated by dividing the dry weight of the sample by the initial weight of the sample. A 5% sodium chloride salt solution prepared by dissolving 5 grams of dried sodium chloride in distilled water was used as the reference matrix for weight percent determinations as described above.

Density Measurement

The bulk densities of the "as-received" granular Tank 5F solids were measured using a calibrated syringe tube assembly with graduation markings. With the syringe plunger removed, the syringe was loaded with Tank 5F solids with the help of a spatula to a level of about 2-mL. The plunger was then inserted into the syringe until the tip of the plunger touched the sample matrix, taking care not to excessively compress the sample. The plunger was then slightly pulled back and tapped to ensure the granular solid was uniformly distributed around the circumference of the tube. The volume of samples in the tube was read and the weight of the whole syringe assembly including the plunger was determined. A subtraction of the weight of the assembly without the granular samples provided the weight of the granular sample inside the syringe tube. Using the same syringe unit, the plunger was carefully removed and more granular sample was added to the syringe and the new volume and weight of the granular samples determined again. Using different syringe assemblies, this process was repeated three times for each Tank 5F sample.