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Characterization of Tank 16H Annulus Samples

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EXECUTIVE SUMMARY

The closure of Tank 16H will require removal of material from the annulus of the tank. Samples from Tank 16H annulus were characterized and tested to provide information to evaluate various alternatives for removing the annulus waste.

The analysis found all four annulus samples to be composed mainly of Si, Na, and Al and lesser amounts of other elements. The XRD data indicate quartz (SiO_2) and sodium aluminum nitrate silicate hydrate ($Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$) as the predominant crystalline mineral phases in the samples. The XRD data also indicate the presence of crystalline sodium nitrate, sodium nitrite, gibbsite, hydrated sodium bicarbonate, and muscovite.

Based on the weight of solids remaining at the end of the test, the water leaching test results indicate approximately 20-35% of the solids dissolved after three contacts with an approximately 3:1 volume of water at 45 °C. The chemical analysis of the leachates and the XRD results of the remaining solids indicate sodium salts of nitrate, nitrite, sulfate, and possibly carbonate/bicarbonate make up the majority of the dissolved material. The majority of these salts were dissolved in the first water contact and simply diluted with each subsequent water contact. The water leaching removed large amounts of the uranium in two of the samples and ~1/3 of the ^{99}Tc from all four samples. Most of the other radionuclides analyzed showed low solubility in the water leaching test.

The preliminary data on the oxalic acid leaching test indicate the three acid contacts at 45 °C dissolved from ~34-47% of the solids. The somewhat higher dissolution found in the oxalic acid leaching test versus the water leaching test might be offset by the tendency of the oxalic acid solutions to take on a gel-like consistency. The filtered solids left behind after three oxalic acid contacts were sticky and formed large clumps after drying. These two observations could indicate potential processing difficulties with solutions and solids from oxalic acid leaching. The gel formation might be avoided by using larger volumes of the acid. Further testing would be recommended before using oxalic acid to dissolve the Tank 16H annulus waste to ensure no processing difficulties are encountered in the full scale process.

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LIST OF ABBREVIATIONS

AD	Analytical Development
BS	Beta Spectroscopy
GS	Gamma Spectroscopy
IC	Ion Chromatography
IE	Inductively Coupled Plasma-Emission Spectroscopy
IM	Inductively Coupled Plasma-Mass Spectrometry
SA	Separation/Alpha Spectroscopy
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TH	Titration for Free Hydroxide
TTR	Technical Task Request
XRD	X-Ray Diffraction

1.0 Introduction

The closure of Tank 16H will require removal of material from the annulus of the tank. Savannah River Remediation (SRR) requested that the Savannah River National Laboratory (SRNL) characterize samples from Tank 16H annulus to provide information to evaluate various alternatives for removing the annulus waste. The work was conducted as stated in the task plan[1] developed from the Technical Task Request (TTR).[2] This report documents the analysis of the initial as-received solids, the duct sample Cs-137 concentration, results of the water leachate analysis, and annulus cleaning recommendations.

A report on a previous analysis of samples from Tank 16H annulus contains a good description of the annulus waste and photographs of the annulus interior.[3]

2.0 Experimental

2.1 Sample Description

A total of four samples were obtained from the Tank 16H annulus. Table 2-1 provides the riser sampling location in the annulus and the dates the samples were received at SRNL. Two sampling locations required re-sampling to obtain sufficient material for the task. The initial plan was to obtain full depth core samples to evaluate any layering in the annulus waste. However, due to difficulties with obtaining intact core samples, a hole was augured at each sampling location and a vacuum device was used to collect the augured material from the hole. Figure 2-1 shows the vacuum device being unloaded in the SRNL shielded cells. Figures 2-2 through 2-6 provide photographs of the solids obtained from each of the four samples stored in glass jars (4" tall x 3 ¾" wide). The material obtained for each of the four samples appeared to be very dry and powdery. Some of the sample material contained larger clumps of solids. The color of the solids ranged from light grey to dark grey for the four samples as can be seen in the photographs.

2.2 Sample Preparation and Analysis

Small amounts of the as-received solids were removed from the cells and sent for analysis by X-ray Diffraction (XRD) to determine the crystalline mineral phases present in the samples. The sample material was not washed, dried or ground prior to sub-sampling. Duplicate sub-samples of each of the annulus samples were analyzed by XRD.

A portion of the solids from each of the four annulus samples were dried in an oven at 110 °C to constant weight to determine the weight percent total dried solids. The dried solids were prepared for analysis using two methods. An aqua regia digestion and a standard fusion with sodium peroxide followed by uptake in nitric acid were used to

dissolve the dried solids from the samples. The digested solids from each method were diluted to reduce activity and allow removal from the Shielded Cells. All sample preparations were conducted in duplicate. A digestion of a glass standard containing many of the elements found in tank samples was prepared concurrently with the sample digestions. Table 2-2 lists the composition of the Analytical Reference Glass-1 glass standard.[4] A reagent blank was prepared concurrently with the sample preparations consisting of the digestion reagents using the same manipulations and dilutions conducted on the sample.

An archived sample from a previous annulus sampling effort (HTF-16-06-104) was also prepared for analysis using a fusion with sodium peroxide followed by uptake in nitric acid.[3] This sample was submitted for a gamma scan to determine the ^{137}Cs concentration in the sample.

Bulk density measurements were made on the annulus samples by grinding portions of the samples in a mortar/pestle to visually uniform fine particles. The finely ground material was placed in a 10 mL syringe allowing determination of the sample volume and then weighed to determine the sample weight.

2.3 Water Leaching Test

A water leaching test was conducted on the Tank 16H annulus samples to determine the water solubility of the sample material. Approximately 50 grams of the as-received solids from each sample were contacted with 150 mL of de-ionized water for ~24 hours. The tests were conducted in a shaker oven maintained at 45 °C throughout the duration of the test. After ~24 hours with moderate agitation, the shaker was switched off and the solids allowed to settle. The liquid was decanted from the bottle as close to the settled solids interface as possible without disturbing the solids. Another 150 mL of de-ionized water was then added to the solids and the mixtures agitated at 45 °C for another 24 hours. A third water contact was conducted in the same manner.

All of the decanted solutions were filtered through a 0.45µm nylon filter to remove fines carried over during the decanting. Single aliquots of each decanted liquid from the water leaching test were removed without dilution and sent to Analytical Development (AD) for analysis.

The solids remaining from the water leaching test were filtered through a 0.45µm nylon filter and rinsed on the filter with three 30 mL portions of de-ionized water to remove the interstitial liquid. The solids were dried in a 110 °C oven overnight and prepared for analysis in the same manner as the as-received solids. Small portions of the dried solids were also submitted for analysis by XRD.

A leaching test using 4 wt% oxalic acid was conducted on the samples using the same overall process and ratios as the water leaching test.

Table 2-1. Sampling Locations of Tank 16H Annulus Samples

Sample ID	Sample Location	Date Received	Total Weight of Sample Received
HTF-16-N-1	N (North)	11/14/2011	162.1 g
HTF-16-S-1	S (South)*	9/13/2011	-
HTF-16-E-1	E (East)*	10/3/2011	-
HTF-16-W-1	W (West)	12/1/2011	234.8 g
HTF-16-S-2	S (South)	11/21/2011	208.8 g
HTF-16-E-2	E (East)	11/7/2011	144.4 g

* An insufficient quantity of material was obtained in the initial samples from the South and East risers, so additional samples were obtained from each of these locations.

Table 2-2. Composition of the Analytical Reference Glass-1 Standard.

Element	mg/kg in Glass
Al	2.50E+04
B	2.69E+04
Ba	7.90E+02
Ca	1.02E+04
Cr	6.40E+02
Cu	3.0E+01
Fe	9.79E+04
K	2.26E+04
Li	1.49E+04
Mg	5.2E+03

Element	mg/kg in Glass
Mn	1.46E+04
Na	8.52E+04
Ni	8.3E+03
P	1.1E+03
Si	2.24E+05
Sr	3.0E+01
Ti	6.9E+03
Zn	1.6E+02
Zr	9.6E+02

Figure 2-1 The Vacuum Sampling Device being Unloaded

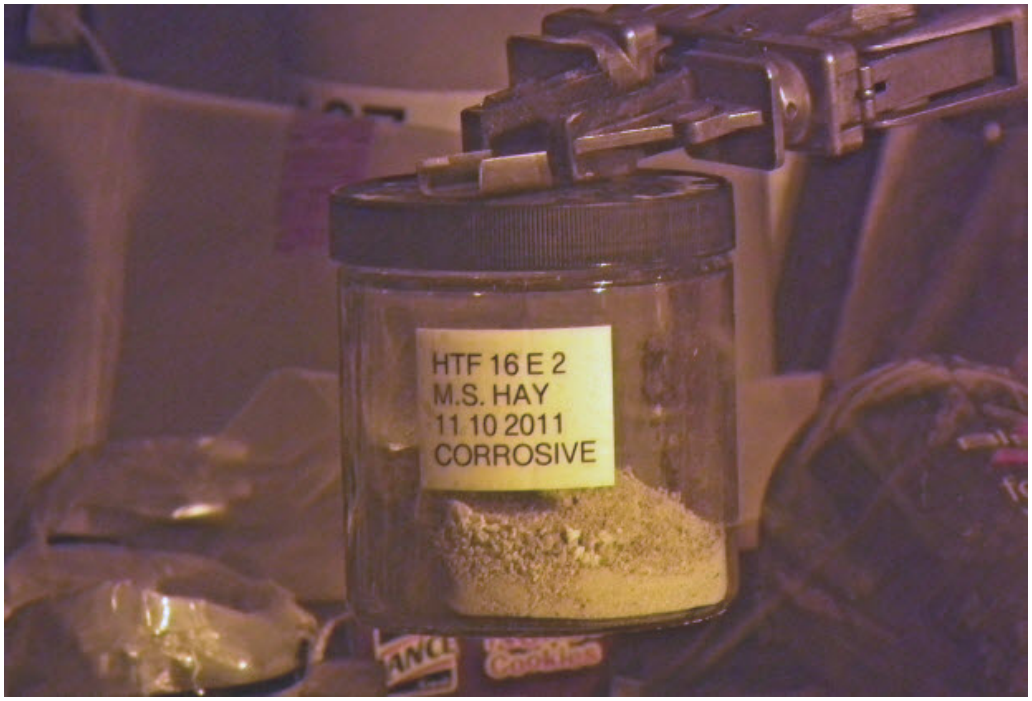

Figure 2-2 As-Received Tank 16H Annulus Sample HTF-16-E-2

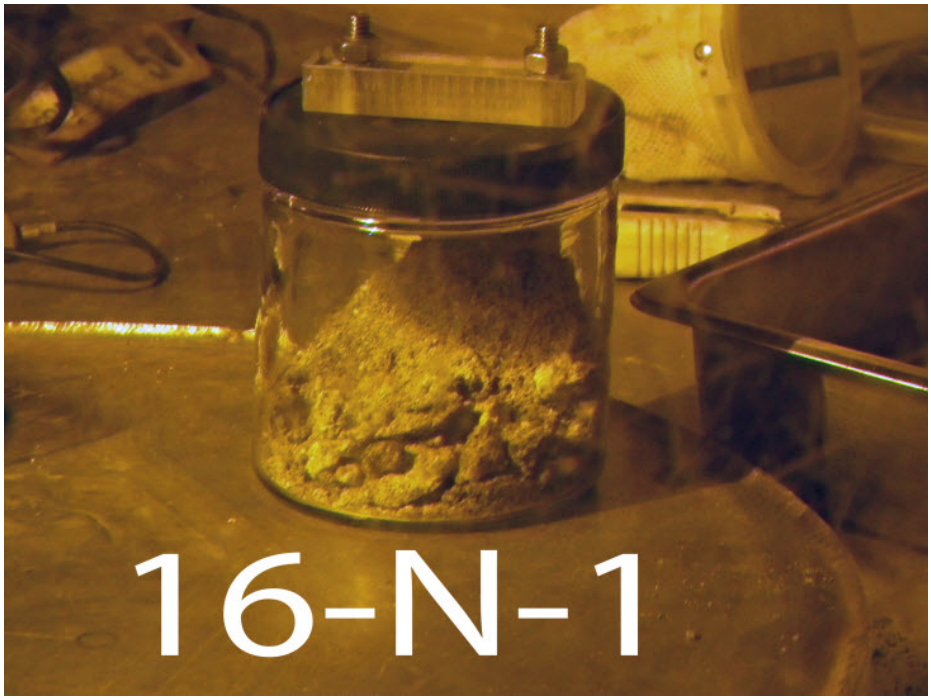

Figure 2-3 As-Received Tank 16H Annulus Sample HTF-16-N-1

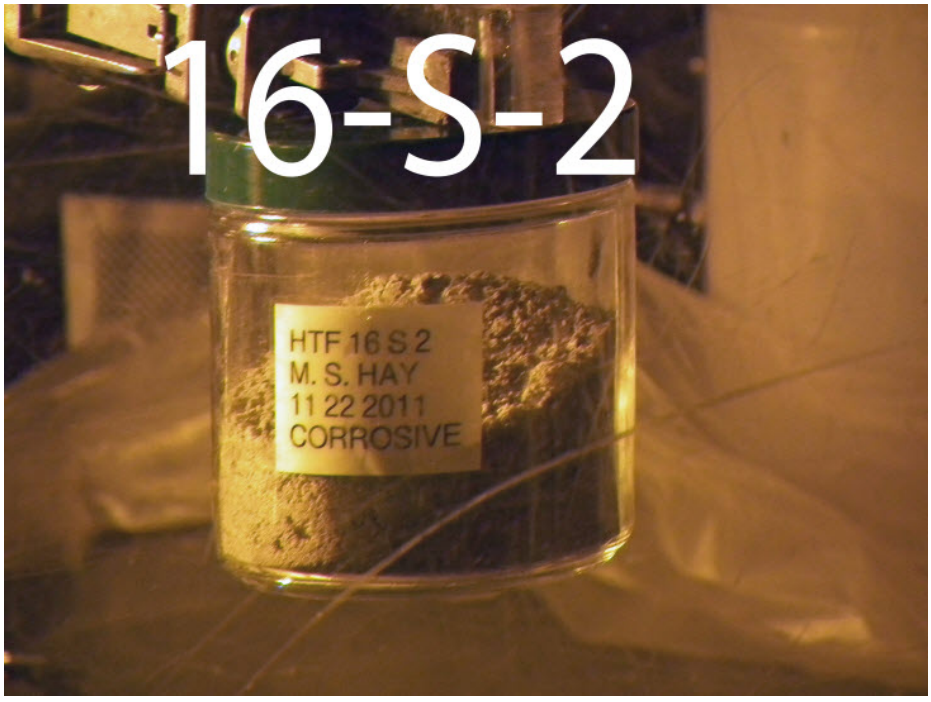

Figure 2-4 As-Received Tank 16H Annulus Sample HTF-16-S-2

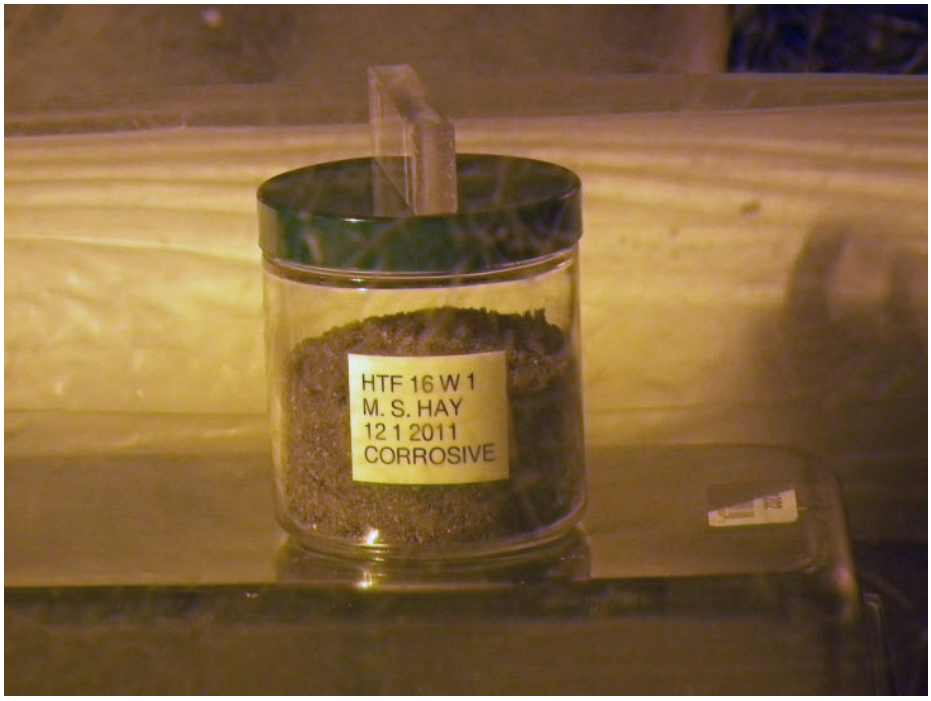

Figure 2-5 As-Received Tank 16H Annulus Sample HTF-16-W-1

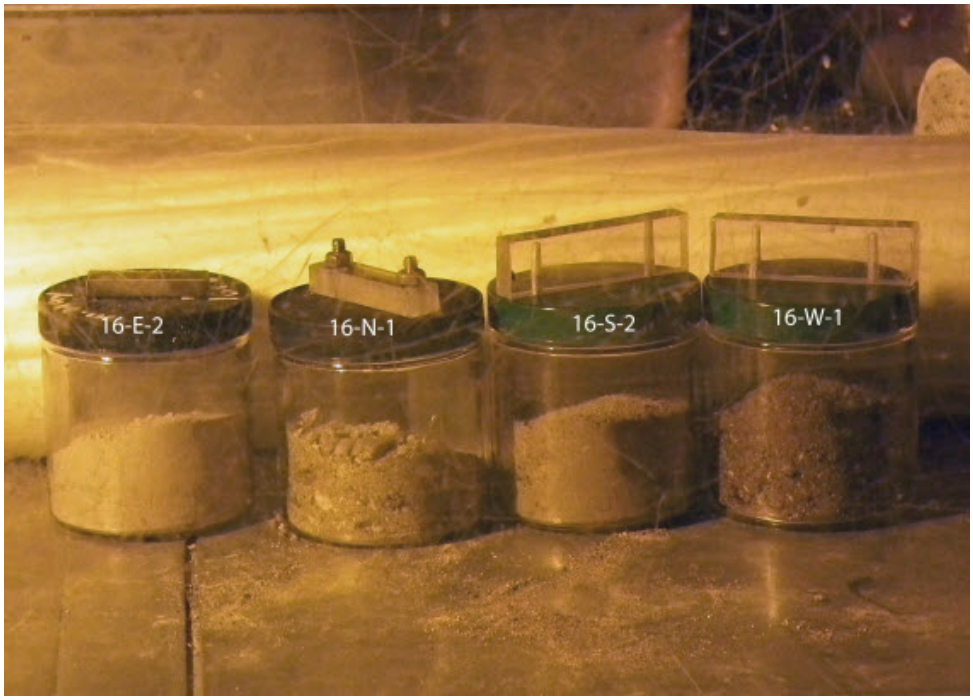

Figure 2-6 The Four As-Received Tank 16H Annulus Samples

3.0 Analytical Results and Discussion

3.1 General Information

Tables 3-2 through 3-7 provide analytical results on weight percent solids, bulk density, and the chemical composition of the as-received Tank 16H annulus samples. Table 3-8 and Figures 3-1 through 3-8 provide the results of the XRD analysis of the as-received samples. Tables 3-9 through 3-12 provide the chemical composition of the decanted leachates from the water leaching test. Figures 3-9 through 3-14 provide photographs of the water leaching tests. Analytical results for as-received samples use units of mg/kg and dpm/g of dried solids. Analytical results for water leachates use units of mg/L and dpm/mL. Values in italics were not used in the calculation of the average due to the unreliability of the values. The unreliability may be due to the presence of the analyte in the blank/standard or the unsuitability of the digestion method for that analyte. Additionally, the detection limits for the peroxide fusion data in the ICP-ES were generally much higher than for the aqua regia digestion and were not included in determining the less than value for the analyte. The results of the reagent blanks and glass standards prepared concurrently with the sample preparations can be found in the appendix in Table A-1.

For waste tank sample characterization, an uncertainty of approximately +/- 15% has been found to be the normal range for the combined sampling and analytical uncertainty.[5] For the Tank 16H samples, the percent relative standard deviation (%RSD) presented in the tables only includes the uncertainty associated with sub-sampling in the Shielded Cells and the uncertainty of the analytical method. It should be noted that the samples represent a small amount of material from four small areas in the tank annulus.

The column headings in the data tables indicate the dissolution method used to dissolve the samples (“Aqua Regia” and “Per Fusion”). The data tables also indicate the analytical method used to measure each analyte. Table 3-1 shows the abbreviations used for each analytical method:

Table 3-1. Abbreviations for Analytical Methods used in Data Tables

Analytical Method	Abbreviation in Tables
Inductively Coupled Plasma-Emission Spectroscopy	IE
Inductively Coupled Plasma-Mass Spectrometry	IM
Ion Chromatography	IC
Titration for Free Hydroxide	TH
Separation/Alpha Spectroscopy	SA
Gamma Spectroscopy	GS
Beta Spectroscopy	BS

3.2 As-Received Solids Results

Table 3-2 shows the results of the weight percent total dried solids and bulk density determinations. The samples contain very little water with the weight percent total dried solids ranging from 84.4% to 94.6%. The bulk density determination yielded values ranging from 1.00 g/mL to 1.14 g/mL. The bulk density measurement is greatly affected by how well the sample material packs into the volumetric device. The particle size of the material affects the packing characteristics of the material. The samples were ground to visually uniform fine particles, but additional grinding and a second bulk density measurement showed increasing density as the particle size decreased. The relationship of the bulk density measured on the small particles of the current samples to the density of the monolithic solid in the Tank 16H annulus remains uncertain.

Table 3-3 shows the results of the ^{137}Cs and ^{90}Sr analysis of the archived sample (HTF-16-06-104) from a previous annulus sampling effort.[3] This sample was obtained from inside the dehumidification duct in the annulus. The ^{137}Cs is somewhat lower and the ^{90}Sr somewhat higher in concentration than the four current samples from the annulus.

Table 3-4 through 3-7 show the analytical results of the four current samples from Tank 16 annulus. The results found Si, Na, Al, and Fe as the most abundant elements in all four samples. The heterogeneous nature of the material and difficulties in sub-sampling the solids may contribute to the higher %RSD's found in some of the results. For both digestion methods the results of the glass standards show close agreement with the expected concentrations and the reagent blanks show little contamination (see Table A-1).

Table 3-8 provides a summary of the XRD analysis of the samples including an estimate of the percentage of each mineral phase present. Figures 3-1 through 3-8 show the individual spectra for each sample in duplicate. The results indicate that all the samples

contain approximately 20-30% amorphous material (non-crystalline). The predominant crystalline mineral phase is either quartz (SiO_2) or sodium aluminum nitrate silicate hydrate ($Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$) in all of the samples. The XRD data also indicate the presence of crystalline sodium nitrate, sodium nitrite, gibbsite, hydrated sodium bicarbonate, and muscovite. All of the samples contain some percentage of sodium nitrate and sodium nitrite. Only two of the samples (HTF-16-N-1 and HTF-16-S-2) contain small percentages of hydrated sodium bicarbonate ($Na_3H(CO_3)_2.2H_2O$).

3.3 Water Leaching Test Results

Tables 3-9 through 3-12 provide the chemical composition of the decanted leachates from the water leaching test. All of the decanted leachates indicate the presence of soluble sodium salts of NO_2^-, NO_3^-, and SO_4^{2-} as the species of highest concentration. The anion/cation charge balance for the solutions analyzed from the first water decants show differences of ~20% to 40% with the sum of the anions being low. Most of the deficit is likely attributable to carbonate anions based on the XRD results indicating the presence of sodium bicarbonate in two of the samples and the large carbonate concentrations found in past Tank 16 annulus samples.[3,6,7] Table 3-13 shows the percentage of solids dissolved from each sample during the water leaching test.

Tables 3-9 through 3-12 also show two columns labeled “Decant 2/Decant 1” and “Decant 3/Decant 2”. The values in the columns represent the fraction from dividing the concentrations in the labeled decant solutions by one another. These columns provide an indication of how material is dissolving from the solids during each water contact in the water leaching test. Since the water leaching test decanted the first contact solution to approximately 50 mL and then added an additional 150 mL of water for the next contact, simple dilution should give a factor of 0.25 (i.e., 50/(50 +150) = 0.25) for the ratio of concentrations between contacts. In other words, if all of the material that will be dissolved was dissolved in the first water contact, the addition of water in second water contact merely serves to dilute the concentrations of species in solution from the first water contact. In that case, the ratio of the concentration in the first water contact to the concentration in the second water contact should be ~0.25. However, since the solutions were decanted to the level of the settled solids during the water leaching test, sometimes less and sometimes more than 50 mL of solution was left behind. Therefore, assuming the volume left after decanting was between 35-65 mL, a ratio of between ~0.2 to ~0.3 indicates simple dilution between water contacts. A value larger than ~0.3 indicates that material is still dissolving between each water contact. A value smaller than ~0.2 for the ratio of concentrations between water contacts may indicate precipitation of the analyte from solution.

Completing a clean separation of the liquid from the solids during the water leaching tests proved difficult due to the presence of fine particulates that did not settle within a 24 hour period. All of the decanted solutions required filtration to remove the fines. The amount of material removed by filtration was miniscule, barely coating the surface of the filter. Figure 3-9 shows a photograph of the settled solids just prior to decanting. The solids at

the bottom of the bottles settled within minutes of stopping agitation. The dark color of the liquid phase above the settled solids results from the suspended particulates. After filtration the decanted liquids were clear and nearly colorless to slightly yellow.

Figures 3-10 through 3-14 show the solids remaining after the water leaching test on each sample (beaker dimensions, 3 7/8" tall, 2" wide at base, 2 ¼" wide at top). The solids were more uniform in color after the water leaching test than the solids of the as-received samples. After drying, samples of the solids were sent for XRD analysis and dissolved for chemical analysis. Table 3-14 provides a summary of the XRD analysis and the estimated percentages of each mineral phase present in the samples. Figures 3-15 through 3-22 show the individual spectra for each sample in duplicate. The XRD results on the water leached solids show no crystalline sodium nitrate or hydrated sodium bicarbonate present and higher percentages of the quartz (SiO_2) or sodium aluminum nitrate silicate hydrate ($Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$) than in the as-received solids. Only the HTF-16-E-2 samples still contains a small percentage of crystalline sodium nitrate. The absence of these soluble to moderately soluble sodium salts in the samples after the water leaching test shows agreement with the analytical results for the decanted water leachate solutions.

Table 3-2. Weight Percent Solids and Bulk Density of the As-Received Tank 16H Annulus Samples

Sample ID	Wt% Total Dried Solids	Bulk Density (%RSD)* g/mL
HTF-16-E-2	94.6%	1.00 (2.4%)
HTF-16-N-1	89.9%	1.03 (13%)
HTF-16-S-2	90.6%	1.14 (9.7%)
HTF-16-W-1	84.4%	1.14 (9.2%)

* The bulk density listed in the table reflects the samples ground to a visually uniform size. Grinding the sample further resulted in higher density values. The reported value is the average of measurements from two grinding cycles.

Table 3-3. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-06-104 (Inside Duct Sample)

Analyte (Method)	Per Fusion Replicate 1 (dpm/g)	Per Fusion Replicate 2 (dpm/g)	Average (dpm/g)	%RSD
^{90}Sr (BS)	7.29E+09	1.69E+10	1.21E+10	56%
^{137}Cs (GS)	8.06E+08	7.61E+08	7.84E+08	4.1%

Table 3-4. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-E-2

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Ag (IE)	<1.13E+02	<1.14E+02	*<1.14E+03*	*<1.13E+03*	<1.14E+02	-
Al (IE)	6.75E+04	6.48E+04	6.85E+04	6.40E+04	6.62E+04	3.2%
B (IE)	<1.58E+01	<1.60E+01	*<1.13E+02*	*<1.12E+02*	<1.59E+01	-
Ba (IE)	6.60E+01	6.85E+01	5.71E+01	5.12E+01	6.07E+01	13%
Be (IE)	<4.71E+00	<4.76E+00	<6.35E+00	<6.30E+00	<5.53E+00	-
Ca (IE)	1.29E+04	1.50E+04	1.53E+04	1.98E+04	1.58E+04	18%
Cd (IE)	6.59E+00	6.59E+00	*<2.46E+01*	*<2.44E+01*	6.59E+00	0.0%
Ce (IE)	5.97E+01	<5.61E+01	<4.79E+02	<4.75E+02	<5.79E+01	-
Co (IE)	<7.61E+00	<7.70E+00	<6.75E+01	<6.69E+01	<7.66E+00	-
Cr (IE)	4.19E+02	3.84E+02	4.29E+02	4.81E+02	4.28E+02	9.4%
Cu (IE)	1.69E+02	2.15E+02	1.73E+02	1.58E+02	1.79E+02	14%
Fe (IE)	5.18E+04	7.26E+04	8.05E+04	5.66E+04	6.54E+04	21%
Gd (IE)	<1.69E+01	<1.71E+01	*<6.19E+01*	*<6.14E+01*	<1.70E+01	-
K (IE)	1.68E+03	1.59E+03	*3.75E+03*	*3.60E+03*	2.66E+03	44%
La (IE)	2.51E+01	2.65E+01	*<5.32E+01*	*<5.28E+01*	2.58E+01	3.8%
Li (IE)	1.58E+01	1.77E+01	*<1.16E+02*	*<1.15E+02*	1.68E+01	8.0%
Mg (IE)	1.57E+03	2.43E+03	1.64E+03	2.31E+03	1.99E+03	22%
Mn (IE)	3.93E+02	5.46E+02	7.98E+02	4.14E+02	5.38E+02	35%
Mo (IE)	3.49E+01	3.71E+01	*<2.13E+02*	*<2.11E+02*	3.60E+01	4.3%
Na (IE)	1.23E+05	1.18E+05	-	-	1.21E+05	2.9%
Ni (IE)	9.20E+01	9.18E+01	*<3.61E+02*	*<3.58E+02*	9.19E+01	0.2%
P (IE)	1.41E+02	1.34E+02	*<6.87E+02*	*<6.82E+02*	1.38E+02	3.6%
Pb (IE)	3.22E+02	4.14E+02	*<5.68E+02*	*<5.64E+02*	3.68E+02	18%
S (IE)	1.58E+03	1.30E+03	*<5.95E+03*	*<5.91E+03*	1.44E+03	14%
Sb (IE)	<1.00E+02	<1.01E+02	*<8.46E+02*	*<8.39E+02*	<1.01E+02	-
Si (IE)	-	-	1.73E+05	1.67E+05	1.70E+05	2.5%
Sn (IE)	<4.40E+01	<4.45E+01	*<4.45E+02*	*<4.42E+02*	<4.43E+01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-4. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-E-2 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Sr (IE)	4.69E+01	5.24E+01	*7.74E+01*	*8.35E+01*	4.97E+01	7.8%
Th (IE)	<4.30E+01	<4.35E+01	*<4.35E+02*	*<4.31E+02*	<4.33E+01	-
Ti (IE)	1.39E+02	1.34E+02	3.55E+02	3.16E+02	2.36E+02	49%
U (IE)	<2.56E+03	<2.59E+03	<2.59E+03	<2.57E+03	<2.58E+03	-
V (IE)	<3.69E+00	<3.73E+00	*<3.73E+01*	*<3.70E+01*	<3.71E+00	-
Zn (IE)	6.38E+02	6.06E+02	6.57E+02	6.47E+02	6.37E+02	3.5%
Zr (IE)	5.94E+01	6.25E+01	-	-	6.10E+01	3.6%
Hg (CV)	1.90E+03	2.31E+03	-	-	2.11E+03	14%
^{232}Th (IM)	2.51E+00	2.13E+00	3.16E+00	2.78E+00	2.64E+00	16%
^{233}U (IM)	<7.84E-01	<7.94E-01	<7.94E-01	<7.87E-01	<7.90E-01	-
^{234}U (IM)	<7.84E-01	<7.94E-01	<7.94E-01	<7.87E-01	<7.90E-01	-
^{235}U (IM)	9.07E+00	8.27E+00	1.01E+01	9.24E+00	9.17E+00	8.2%
^{236}U (IM)	9.80E-01	1.06E+00	1.38E+00	1.52E+00	1.23E+00	21%
^{238}U (IM)	2.57E+02	2.28E+02	2.59E+02	2.50E+02	2.48E+02	5.7%
U-total (IM)	2.67E+02	2.37E+02	2.70E+02	2.61E+02	2.59E+02	5.8%
^{90}Sr (BS)	5.91E+00	5.32E+00	4.44E+00	4.57E+00	5.06E+00	14%
^{137}Cs (GS)	1.06E+01	1.08E+01	1.12E+01	1.07E+01	1.08E+01	2.5%
^{238}Pu (SA)	1.12E-01	1.07E-01	1.14E-01	1.11E-01	1.11E-01	2.5%
^{99}Tc (IM)	1.15E+01	1.14E+01	1.29E+01	1.36E+01	1.23E+01	8.7%
^{237}Np (IM)	2.88E+00	2.43E+00	2.28E+00	2.76E+00	2.59E+00	11%
^{239}Pu (IM)	3.64E+00	4.11E+00	3.73E+00	3.90E+00	3.84E+00	5.4%
^{240}Pu (IM)	<7.84E-01	<7.94E-01	<1.19E+00	<1.18E+00	<9.87E-01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-4. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-E-2 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (dpm/g)	Aqua Regia Replicate 2 (dpm/g)	Per Fusion Replicate 1 (dpm/g)	Per Fusion Replicate 2 (dpm/g)	Average (dpm/g)	%RSD
^{90}Sr (BS)	1.80E+09	1.62E+09	1.35E+09	1.39E+09	1.54E+09	14%
^{137}Cs (GS)	2.04E+09	2.08E+09	2.16E+09	2.07E+09	2.09E+09	2.5%
^{238}Pu (SA)	4.25E+06	4.08E+06	4.33E+06	4.20E+06	4.22E+06	2.5%
$^{239/240}$Pu (SA)	7.23E+05	7.03E+05	7.41E+05	7.58E+05	7.31E+05	3.2%
^{99}Tc (IM)	4.34E+05	4.28E+05	4.85E+05	5.11E+05	4.65E+05	8.7%
^{233}U (IM)	<1.68E+04	<1.70E+04	<1.70E+04	<1.69E+04	<1.69E+04	-
^{234}U (IM)	<1.09E+04	<1.10E+04	<1.10E+04	<1.09E+04	<1.10E+04	-
^{235}U (IM)	4.35E+01	3.96E+01	4.85E+01	4.43E+01	4.40E+01	8.2%
^{236}U (IM)	1.41E+02	1.52E+02	1.98E+02	2.18E+02	1.77E+02	21%
^{237}Np (IM)	4.51E+03	3.80E+03	3.57E+03	4.32E+03	4.05E+03	11%
^{238}U (IM)	1.92E+02	1.70E+02	1.93E+02	1.86E+02	1.85E+02	5.7%
^{239}Pu (IM)	4.95E+05	5.59E+05	5.07E+05	5.31E+05	5.23E+05	5.4%
^{240}Pu (IM)	<3.93E+05	<3.97E+05	<5.96E+05	<5.92E+05	<4.95E+05	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-5. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-N-1

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Ag (IE)	<1.13E+02	<1.15E+02	*<1.13E+03*	*<1.13E+03*	<1.14E+02	-
Al (IE)	7.16E+04	7.05E+04	8.40E+04	6.47E+04	7.27E+04	11%
B (IE)	<1.58E+01	<1.62E+01	*<1.11E+02*	*<1.11E+02*	<1.60E+01	-
Ba (IE)	3.99E+01	2.96E+01	*<4.06E+01*	*<4.06E+01*	3.48E+01	21%
Be (IE)	<4.69E+00	<4.80E+00	<6.25E+00	<6.25E+00	<5.50E+00	-
Ca (IE)	1.98E+03	1.34E+04	5.12E+03	3.68E+03	6.05E+03	84%
Cd (IE)	4.30E+00	3.28E+00	*<2.42E+01*	*<2.42E+01*	3.79E+00	19%
Ce (IE)	<5.52E+01	<5.66E+01	*<4.71E+02*	*<4.71E+02*	<5.59E+01	-
Co (IE)	<7.58E+00	<7.76E+00	*<6.64E+01*	*<6.64E+01*	<7.67E+00	-
Cr (IE)	1.12E+02	7.80E+01	1.23E+02	*<7.97E+01*	1.04E+02	22%
Cu (IE)	2.06E+02	2.02E+02	1.39E+02	7.03E+01	1.54E+02	41%
Fe (IE)	1.11E+04	7.01E+03	7.21E+03	1.95E+04	1.12E+04	52%
Gd (IE)	<1.68E+01	<1.72E+01	*<6.09E+01*	*<6.09E+01*	<1.70E+01	-
K (IE)	9.27E+02	7.85E+02	*2.40E+03*	*2.11E+03*	1.56E+03	53%
La (IE)	1.45E+01	8.96E+00	*<5.23E+01*	*<5.23E+01*	1.17E+01	33%
Li (IE)	1.18E+01	1.35E+01	*<1.14E+02*	*<1.14E+02*	1.27E+01	10%
Mg (IE)	4.92E+02	8.75E+02	8.96E+02	7.93E+01	5.86E+02	66%
Mn (IE)	1.09E+02	1.28E+02	5.94E+01	1.02E+02	9.96E+01	29%
Mo (IE)	<2.09E+01	<2.14E+01	*<2.09E+02*	*<2.09E+02*	<2.12E+01	-
Na (IE)	1.22E+05	1.20E+05	-	-	1.21E+05	1.2%
Ni (IE)	<3.55E+01	<3.64E+01	*<3.55E+02*	*<3.55E+02*	<3.60E+01	-
P (IE)	<1.28E+02	<1.31E+02	*<6.77E+02*	*<6.77E+02*	<1.30E+02	-
Pb (IE)	2.93E+02	2.18E+02	*<5.59E+02*	*<5.59E+02*	2.56E+02	21%
S (IE)	1.36E+03	1.74E+03	*<5.86E+03*	*<5.86E+03*	1.55E+03	17%
Sb (IE)	<9.98E+01	<1.02E+02	*<8.33E+02*	*<8.33E+02*	<1.01E+02	-
Si (IE)	-	-	1.47E+05	2.75E+05	2.11E+05	43%
Sn (IE)	<4.38E+01	<4.49E+01	*<4.38E+02*	*<4.38E+02*	<4.44E+01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-5. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-N-1 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Sr (IE)	1.13E+01	1.59E+01	*3.79E+01*	*3.52E+01*	1.36E+01	24%
Th (IE)	<4.28E+01	<4.38E+01	*<4.28E+02*	*<4.28E+02*	<4.33E+01	-
Ti (IE)	2.18E+02	7.33E+01	8.71E+02	1.36E+02	3.25E+02	114%
U (IE)	<2.55E+03	<2.61E+03	<2.55E+03	<2.55E+03	<2.57E+03	-
V (IE)	1.18E+01	5.68E+00	*<3.67E+01*	*<3.67E+01*	8.74E+00	50%
Zn (IE)	8.29E+02	7.63E+03	1.90E+03	2.10E+02	2.64E+03	129%
Zr (IE)	2.42E+01	1.60E+01	-	-	2.01E+01	29%
Hg (CV)	1.22E+03	7.99E+02	-	-	1.01E+03	29%
^{232}Th (IM)	3.50E+00	1.77E+00	4.21E+00	1.20E+00	2.67E+00	53%
^{233}U (IM)	<7.81E-01	<8.00E-01	<7.81E-01	<7.81E-01	<7.86E-01	-
^{234}U (IM)	<7.81E-01	<8.00E-01	<7.81E-01	<7.81E-01	<7.86E-01	-
^{235}U (IM)	2.61E+00	1.68E+00	2.84E+00	1.63E+00	2.19E+00	29%
^{236}U (IM)	<7.81E-01	<8.00E-01	<7.81E-01	<7.81E-01	<7.86E-01	-
^{238}U (IM)	6.42E+01	4.90E+01	5.92E+01	3.03E+01	5.07E+01	30%
U-total (IM)	6.68E+01	5.07E+01	6.21E+01	3.19E+01	5.28E+01	29%
^{90}Sr (BS)	2.12E+00	1.22E+00	9.20E-01	5.39E-01	1.20E+00	56%
^{137}Cs (GS)	1.43E+01	1.23E+01	1.18E+01	1.01E+01	1.21E+01	14%
^{238}Pu (SA)	4.16E-02	3.03E-02	3.10E-02	2.15E-02	3.11E-02	26%
^{99}Tc (IM)	8.48E+00	5.80E+00	1.21E+01	4.36E+00	7.69E+00	44%
^{237}Np (IM)	<7.81E-01	<8.00E-01	9.57E-01	<7.81E-01	9.57E-01	-
^{239}Pu (IM)	1.52E+00	1.03E+00	1.13E+00	9.03E-01	1.15E+00	23%
^{240}Pu (IM)	<7.81E-01	<8.00E-01	<1.17E+00	<1.17E+00	<9.81E-01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-5. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-N-1 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (dpm/g)	Aqua Regia Replicate 2 (dpm/g)	Per Fusion Replicate 1 (dpm/g)	Per Fusion Replicate 2 (dpm/g)	Average (dpm/g)	%RSD
^{90}Sr (BS)	6.44E+08	3.70E+08	2.80E+08	1.64E+08	3.65E+08	56%
^{137}Cs (GS)	2.75E+09	2.36E+09	2.28E+09	1.94E+09	2.33E+09	14%
^{238}Pu (SA)	1.58E+06	1.15E+06	1.18E+06	8.17E+05	1.18E+06	26%
$^{239/240}$Pu (SA)	2.29E+05	1.61E+05	1.69E+05	1.26E+05	1.71E+05	25%
^{99}Tc (IM)	3.19E+05	2.18E+05	4.57E+05	1.64E+05	2.90E+05	44%
^{233}U (IM)	<1.67E+04	<1.71E+04	<1.67E+04	<1.67E+04	<1.68E+04	-
^{234}U (IM)	<1.09E+04	<1.11E+04	<1.09E+04	<1.09E+04	<1.09E+04	-
^{235}U (IM)	1.25E+01	8.04E+00	1.36E+01	7.81E+00	1.05E+01	29%
^{236}U (IM)	<1.12E+02	<1.15E+02	<1.12E+02	<1.12E+02	<1.13E+02	-
^{237}Np (IM)	<1.22E+03	<1.25E+03	1.50E+03	<1.22E+03	1.50E+03	-
^{238}U (IM)	4.79E+01	3.65E+01	4.42E+01	2.26E+01	3.78E+01	30%
^{239}Pu (IM)	2.07E+05	1.40E+05	1.54E+05	1.23E+05	1.56E+05	23%
^{240}Pu (IM)	<3.91E+05	<4.01E+05	<5.87E+05	<5.87E+05	<4.91E+05	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-6. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-S-2

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Ag (IE)	<1.12E+02	<1.13E+02	*<1.15E+03*	*<1.13E+03*	<1.13E+02	-
Al (IE)	3.01E+04	4.28E+04	4.50E+04	5.02E+04	4.20E+04	20%
B (IE)	<1.57E+01	*<1.59E+01*	<1.13E+02	<1.11E+02	<1.58E+01	-
Ba (IE)	7.46E+01	1.13E+02	5.66E+01	6.04E+01	7.62E+01	34%
Be (IE)	<4.67E+00	<4.72E+00	<6.37E+00	<6.27E+00	<5.51E+00	-
Ca (IE)	2.41E+03	6.14E+03	5.02E+03	5.12E+03	4.67E+03	34%
Cd (IE)	4.51E+00	4.09E+00	*<2.47E+01*	*<2.43E+01*	4.30E+00	6.9%
Ce (IE)	6.35E+01	6.85E+01	*<4.80E+02*	*<4.73E+02*	6.60E+01	5.4%
Co (IE)	<7.55E+00	<7.64E+00	*<6.77E+01*	*<6.67E+01*	<7.60E+00	-
Cr (IE)	1.49E+02	1.11E+02	1.43E+02	1.62E+02	1.41E+02	15%
Cu (IE)	1.17E+02	1.18E+02	1.17E+04	1.74E+02	3.03E+03	191%
Fe (IE)	2.29E+04	1.97E+04	2.20E+04	1.59E+04	2.01E+04	16%
Gd (IE)	<1.67E+01	<1.69E+01	*<6.22E+01*	*<6.12E+01*	<1.68E+01	-
K (IE)	1.04E+03	1.12E+03	*2.86E+03*	*3.38E+03*	2.10E+03	57%
La (IE)	2.73E+01	3.36E+01	*<5.34E+01*	*<5.25E+01*	3.05E+01	15%
Li (IE)	<1.14E+01	<1.15E+01	*<1.16E+02*	*<1.15E+02*	<1.15E+01	-
Mg (IE)	3.38E+03	1.89E+03	2.93E+03	2.06E+03	2.57E+03	28%
Mn (IE)	1.82E+02	1.56E+02	1.75E+02	1.53E+02	1.67E+02	8.5%
Mo (IE)	2.37E+01	2.22E+01	*<2.14E+02*	*<2.10E+02*	2.30E+01	4.6%
Na (IE)	1.74E+05	1.44E+05	-	-	1.59E+05	13%
Ni (IE)	6.77E+01	4.72E+01	*<3.63E+02*	*<3.57E+02*	5.75E+01	25%
P (IE)	<1.28E+02	<1.29E+02	*9.74E+02*	*<6.79E+02*	<1.29E+02	-
Pb (IE)	1.82E+03	4.64E+03	*6.18E+02*	*7.26E+02*	3.23E+03	62%
S (IE)	4.09E+03	6.91E+03	*<5.98E+03*	*<5.88E+03*	5.50E+03	36%
Sb (IE)	<9.94E+01	<1.01E+02	*<8.49E+02*	*<8.36E+02*	<1.00E+02	-
Si (IE)	-	-	1.68E+05	1.74E+05	1.71E+05	2.5%
Sn (IE)	<4.37E+01	<4.42E+01	*<4.47E+02*	*<4.40E+02*	<4.40E+01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-6. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-S-2 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Sr (IE)	2.50E+01	3.36E+01	*5.58E+01*	*5.57E+01*	2.93E+01	21%
Th (IE)	<4.26E+01	<4.31E+01	*<4.37E+02*	*<4.30E+02*	<4.29E+01	-
Ti (IE)	4.71E+01	5.77E+01	1.29E+02	1.34E+02	9.20E+01	50%
U (IE)	<2.54E+03	<2.57E+03	<2.60E+03	<2.56E+03	<2.57E+03	-
V (IE)	1.81E+01	8.27E+00	*<3.75E+01*	*<3.69E+01*	1.32E+01	53%
Zn (IE)	2.87E+02	3.16E+02	3.71E+02	3.49E+02	3.31E+02	11%
Zr (IE)	6.68E+01	9.16E+01	-	-	7.92E+01	22%
Hg (CV)	3.82E+03	3.10E+03	-	-	3.46E+03	15%
^{232}Th (IM)	*<1.56E+00*	1.90E+00	2.45E+00	2.82E+00	2.39E+00	19%
^{233}U (IM)	<7.78E-01	<7.87E-01	<7.97E-01	<7.84E-01	<7.87E-01	-
^{234}U (IM)	<7.78E-01	<7.87E-01	<7.97E-01	<7.84E-01	<7.87E-01	-
^{235}U (IM)	3.29E+00	4.84E+00	5.93E+00	6.22E+00	5.07E+00	26%
^{236}U (IM)	<7.78E-01	<7.87E-01	<7.97E-01	<7.84E-01	<7.87E-01	-
^{238}U (IM)	9.02E+01	1.11E+02	1.42E+02	1.39E+02	1.20E+02	20%
U-total (IM)	9.34E+01	1.16E+02	1.47E+02	1.45E+02	1.25E+02	21%
^{90}Sr (BS)	5.36E+00	5.72E+00	4.11E+00	5.19E+00	5.09E+00	14%
^{137}Cs (GS)	6.59E+00	8.72E+00	8.88E+00	9.66E+00	8.46E+00	15%
^{238}Pu (SA)	5.97E-02	7.08E-02	7.66E-02	7.87E-02	7.14E-02	12%
^{99}Tc (IM)	1.13E+01	1.39E+01	1.62E+01	1.65E+01	1.45E+01	17%
^{237}Np (IM)	1.09E+00	1.62E+00	2.34E+00	1.77E+00	1.71E+00	30%
^{239}Pu (IM)	1.77E+00	2.61E+00	2.81E+00	2.36E+00	2.39E+00	19%
^{240}Pu (IM)	<7.78E-01	<7.87E-01	<1.20E+00	<1.18E+00	<9.84E-01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-6. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-S-2 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (dpm/g)	Aqua Regia Replicate 2 (dpm/g)	Per Fusion Replicate 1 (dpm/g)	Per Fusion Replicate 2 (dpm/g)	Average (dpm/g)	%RSD
^{90}Sr (BS)	1.63E+09	1.74E+09	1.25E+09	1.58E+09	1.55E+09	14%
^{137}Cs (GS)	1.27E+09	1.68E+09	1.71E+09	1.86E+09	1.63E+09	15%
^{238}Pu (SA)	2.27E+06	2.69E+06	2.91E+06	2.99E+06	2.72E+06	12%
$^{239/240}$Pu (SA)	3.59E+05	4.36E+05	4.95E+05	5.11E+05	4.50E+05	15%
^{99}Tc (IM)	4.26E+05	5.22E+05	6.11E+05	6.20E+05	5.45E+05	17%
^{233}U (IM)	<1.67E+04	<1.69E+04	<1.71E+04	<1.68E+04	<1.68E+04	-
^{234}U (IM)	<1.08E+04	<1.09E+04	<1.11E+04	<1.09E+04	<1.09E+04	-
^{235}U (IM)	1.58E+01	2.32E+01	2.84E+01	2.98E+01	2.43E+01	26%
^{236}U (IM)	<1.12E+02	<1.13E+02	<1.14E+02	<1.13E+02	<1.13E+02	-
^{237}Np (IM)	1.71E+03	2.53E+03	3.66E+03	2.77E+03	2.67E+03	30%
^{238}U (IM)	6.72E+01	8.28E+01	1.06E+02	1.04E+02	8.98E+01	20%
^{239}Pu (IM)	2.41E+05	3.55E+05	3.82E+05	3.21E+05	3.25E+05	19%
^{240}Pu (IM)	<3.90E+05	<3.94E+05	<5.99E+05	<5.89E+05	<4.93E+05	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-7. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-W-1

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Ag (IE)	<1.13E+02	<1.17E+02	*<1.07E+03*	*<1.05E+03*	<1.15E+02	-
Al (IE)	8.61E+04	8.59E+04	8.40E+04	7.80E+04	8.35E+04	4.5%
B (IE)	<1.58E+01	<1.64E+01	*<1.06E+02*	*<1.04E+02*	<1.61E+01	-
Ba (IE)	1.38E+02	1.28E+02	8.62E+01	1.07E+02	1.15E+02	20%
Be (IE)	<4.69E+00	<4.88E+00	<5.95E+00	<5.84E+00	<5.34E+00	-
Ca (IE)	1.04E+03	1.83E+03	3.37E+03	3.34E+03	1.44E+03	39%
Cd (IE)	7.66E+00	7.80E+00	*<2.30E+01*	*<2.26E+01*	7.73E+00	1.3%
Ce (IE)	1.88E+02	1.79E+02	*<4.48E+02*	*<4.40E+02*	1.84E+02	3.5%
Co (IE)	9.76E+00	<7.89E+00	*<6.32E+01*	*<6.20E+01*	9.76E+00	-
Cr (IE)	1.18E+02	1.37E+02	1.24E+02	1.20E+02	1.25E+02	6.8%
Cu (IE)	1.51E+02	1.38E+02	8.18E+01	1.14E+02	1.21E+02	25%
Fe (IE)	5.06E+04	3.63E+04	2.72E+04	2.90E+04	3.58E+04	30%
Gd (IE)	<1.68E+01	<1.75E+01	*<5.80E+01*	*<5.69E+01*	<1.72E+01	-
K (IE)	1.67E+03	1.64E+03	*3.77E+03*	*3.34E+03*	2.61E+03	43%
La (IE)	8.73E+01	7.97E+01	*2.75E+02*	*6.50E+01*	8.35E+01	6.4%
Li (IE)	1.51E+01	1.13E+01	*<1.09E+02*	*<1.07E+02*	1.32E+01	20%
Mg (IE)	2.96E+02	5.83E+02	2.57E+02	2.26E+02	3.41E+02	48%
Mn (IE)	3.50E+02	3.03E+02	3.13E+02	3.13E+02	3.20E+02	6.5%
Mo (IE)	3.63E+01	3.68E+01	*<1.99E+02*	*<1.96E+02*	3.66E+01	1.0%
Na (IE)	1.74E+05	1.83E+05	-	-	1.79E+05	3.6%
Ni (IE)	1.05E+02	1.25E+02	*<3.38E+02*	*<3.32E+02*	1.15E+02	12%
P (IE)	<1.28E+02	<1.33E+02	*<6.44E+02*	*<6.32E+02*	<1.31E+02	-
Pb (IE)	8.67E+02	5.76E+02	*6.20E+02*	*6.89E+02*	7.22E+02	29%
S (IE)	1.38E+03	1.46E+03	*<5.58E+03*	*<5.47E+03*	1.42E+03	4.0%
Sb (IE)	<9.98E+01	<1.04E+02	*<7.93E+02*	*<7.78E+02*	<1.02E+02	-
Si (IE)	-	-	1.19E+05	1.14E+05	1.17E+05	3.0%
Sn (IE)	<4.38E+01	<4.56E+01	*<4.17E+02*	*<4.09E+02*	<4.47E+01	-

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-7. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-W-1 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (mg/kg)	Aqua Regia Replicate 2 (mg/kg)	Per Fusion Replicate 1 (mg/kg)	Per Fusion Replicate 2 (mg/kg)	Average (mg/kg)	%RSD
Sr (IE)	5.13E+01	4.45E+01	*5.84E+01*	*6.68E+01*	4.79E+01	10%
Th (IE)	<4.28E+01	<4.46E+01	*<4.07E+02*	*<4.00E+02*	<4.37E+01	-
Ti (IE)	4.24E+01	3.92E+01	1.44E+02	1.37E+02	9.07E+01	64%
U (IE)	<2.55E+03	<2.65E+03	<2.43E+03	<2.38E+03	<2.50E+03	-
V (IE)	<3.67E+00	<3.82E+00	*<3.49E+01*	*<3.43E+01*	<3.75E+00	-
Zn (IE)	4.28E+02	3.93E+02	3.22E+02	2.89E+02	3.58E+02	18%
Zr (IE)	2.40E+02	2.20E+02	-	-	2.30E+02	6.1%
Hg (CV)	2.72E+03	2.49E+03	-	-	2.61E+03	6.2%
^{232}Th (IM)	2.40E+00	1.79E+00	1.68E+00	1.67E+00	1.89E+00	18%
^{233}U (IM)	<7.81E-01	<8.13E-01	<7.43E-01	<7.30E-01	<7.67E-01	-
^{234}U (IM)	<7.81E-01	<8.13E-01	<7.43E-01	<7.30E-01	<7.67E-01	-
^{235}U (IM)	9.90E+00	8.97E+00	8.15E+00	1.10E+01	9.49E+00	13%
^{236}U (IM)	9.07E-01	1.19E+00	1.28E+00	1.09E+00	1.12E+00	14%
^{238}U (IM)	2.55E+02	2.53E+02	2.12E+02	2.68E+02	2.47E+02	9.9%
U-total (IM)	2.66E+02	2.63E+02	2.21E+02	2.80E+02	2.58E+02	9.9%
^{90}Sr (BS)	1.19E+01	6.57E+00	6.41E+00	9.00E+00	8.48E+00	31%
^{137}Cs (GS)	1.67E+01	1.68E+01	1.66E+01	1.52E+01	1.63E+01	4.6%
^{238}Pu (SA)	1.60E-01	1.73E-01	1.17E-01	1.62E-01	1.53E-01	16%
^{99}Tc (IM)	2.54E+01	2.36E+01	3.02E+01	2.74E+01	2.66E+01	11%
^{237}Np (IM)	2.87E+00	2.56E+00	2.49E+00	2.68E+00	2.65E+00	6.3%
^{239}Pu (IM)	5.86E+00	5.33E+00	4.29E+00	5.61E+00	5.27E+00	13%
^{240}Pu (IM)	1.02E+00	1.01E+00	<1.12E+00	1.15E+00	1.07E+00	6.4%

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-7. Composition of the As-Received Solids from Tank 16H Annulus Sample HTF-16-W-1 (continued)

Analyte (Method)	Aqua Regia Replicate 1 (dpm/g)	Aqua Regia Replicate 2 (dpm/g)	Per Fusion Replicate 1 (dpm/g)	Per Fusion Replicate 2 (dpm/g)	Average (dpm/g)	%RSD
^{90}Sr (BS)	3.63E+09	2.00E+09	1.95E+09	2.74E+09	2.58E+09	31%
^{137}Cs (GS)	3.21E+09	3.24E+09	3.20E+09	2.93E+09	3.15E+09	5%
^{238}Pu (SA)	6.07E+06	6.58E+06	4.44E+06	6.15E+06	5.81E+06	16%
$^{239/240}$Pu (SA)	1.11E+06	1.23E+06	8.38E+05	1.17E+06	1.09E+06	16%
^{99}Tc (IM)	9.55E+05	8.89E+05	1.14E+06	1.03E+06	1.00E+06	11%
^{233}U (IM)	<1.67E+04	<1.74E+04	<1.59E+04	<1.56E+04	<1.64E+04	-
^{234}U (IM)	<1.09E+04	<1.13E+04	<1.03E+04	<1.01E+04	<1.07E+04	-
^{235}U (IM)	4.74E+01	4.30E+01	3.91E+01	5.26E+01	4.55E+01	13%
^{236}U (IM)	1.30E+02	1.71E+02	1.84E+02	1.56E+02	1.60E+02	14%
^{237}Np (IM)	4.49E+03	4.01E+03	3.90E+03	4.20E+03	4.15E+03	6.3%
^{238}U (IM)	1.90E+02	1.89E+02	1.58E+02	2.00E+02	1.84E+02	9.9%
^{239}Pu (IM)	7.98E+05	7.25E+05	5.84E+05	7.63E+05	7.17E+05	13%
^{240}Pu (IM)	5.10E+05	5.06E+05	<5.59E+05	5.74E+05	5.30E+05	7.2%

Values in italics were not used in calculating the average. Divide mg/kg values by 1E+04 to convert to wt % basis.

Table 3-8. XRD Result for the As-Received Tank 16H Annulus Samples-Estimated Mineral Phase Composition

Crystalline Phase	HTF-16E2 Replicate 1	HTF-16E2 Replicate 2	HTF-16N1 Replicate 1	HTF-16N1 Replicate 2	HTF-16S2 Replicate 1	HTF-16S2 Replicate 2	HTF-16W1 Replicate 1	HTF-16W1 Replicate 2
Amorphous	25%	28%	22%	21%	21%	22%	19%	21%
SiO_2	20%	25%	25%	20%	15%	10%	2%	-
$Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$	20%	20%	15%	25%	25%	25%	40%	50%
$NaNO_3$	10%	10%	10%	10%	12%	18%	20%	10%
$NaNO_2$	10%	10%	10%	10%	10%	11%	18%	15%
$KAl_2(AlSi_3O_{10})(OH)_2$	10%	-	5%	5%	5%	5%	-	5%
$Al(OH)_3$	5%	-	5%	5%	-	-	-	-
$Na_3H(CO_3)_2.2H_2O$	-	-	5%	5%	13%	10%	-	-

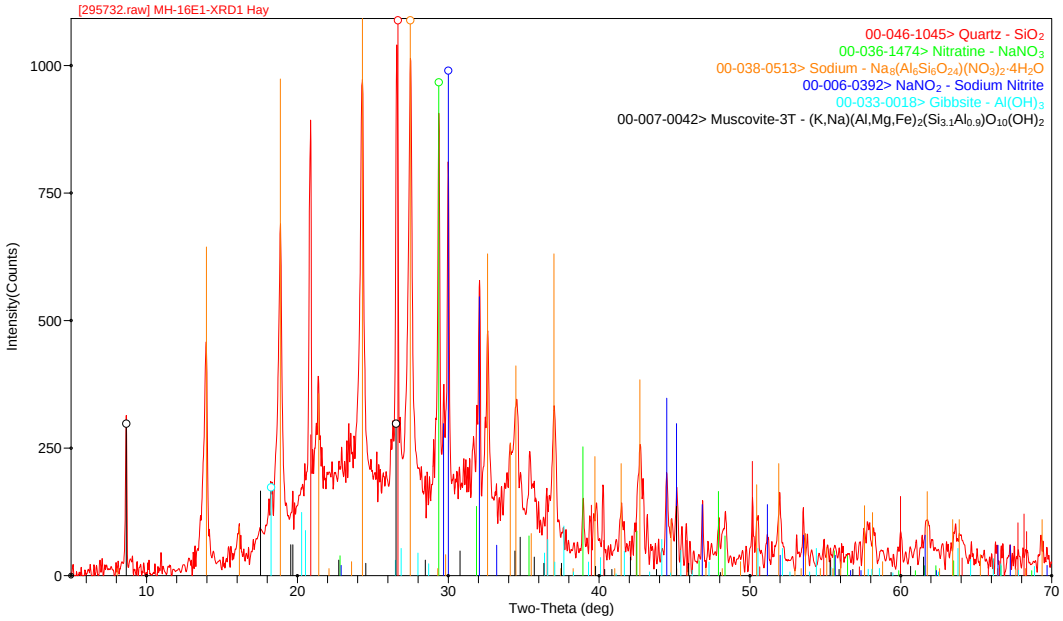

Figure 3-1 XRD Results for Tank 16H Annulus Sample HTF-16E2-1

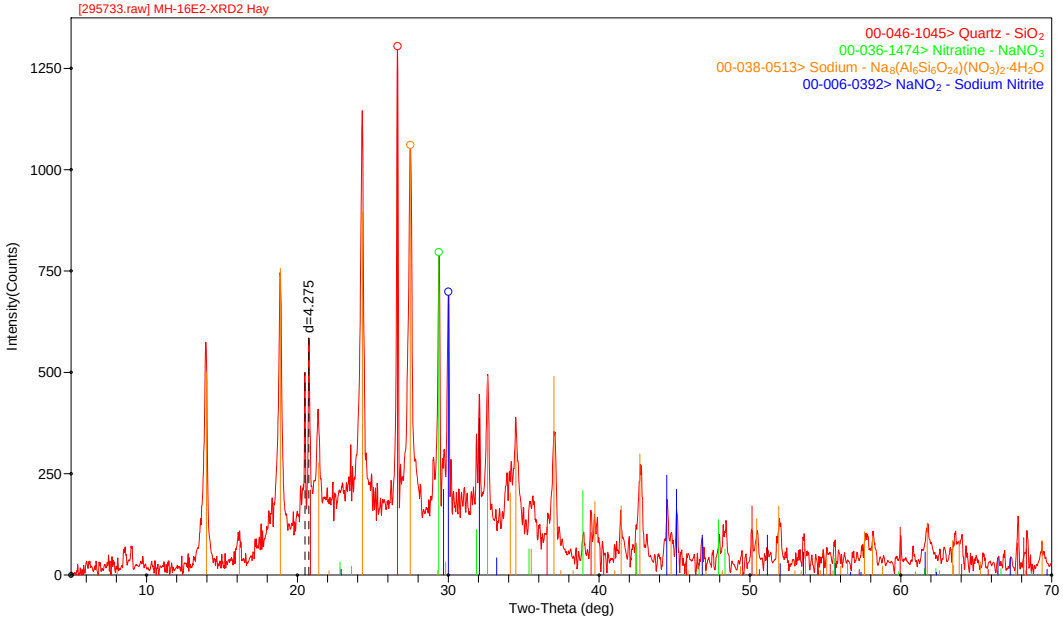

Figure 3-2 XRD Results for Tank 16H Annulus Sample HTF-16E2-2

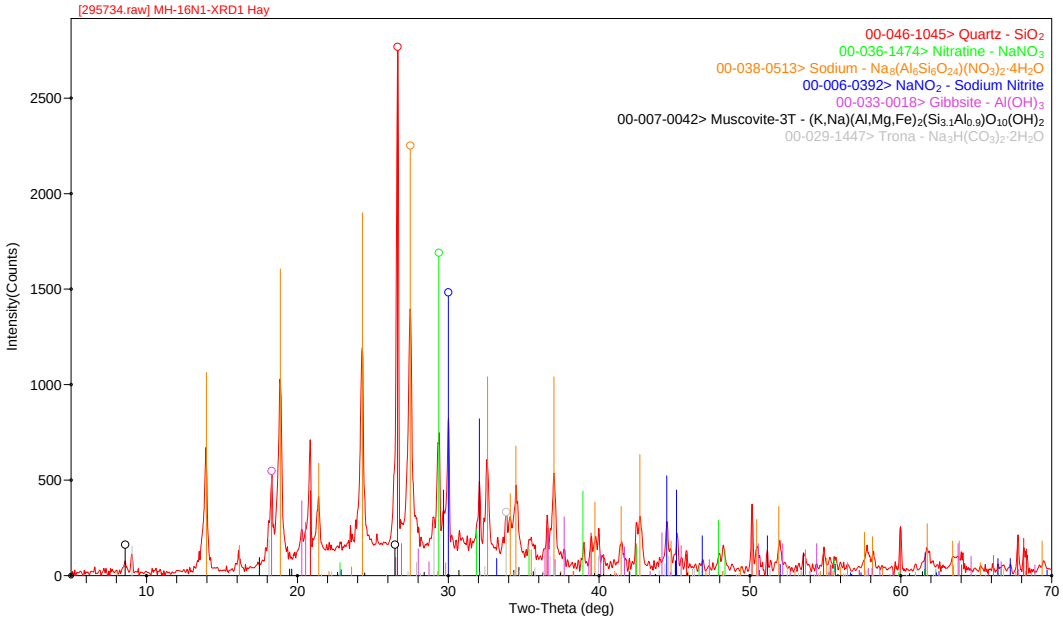

Figure 3-3 XRD Results for Tank 16H Annulus Sample HTF-16N1-1

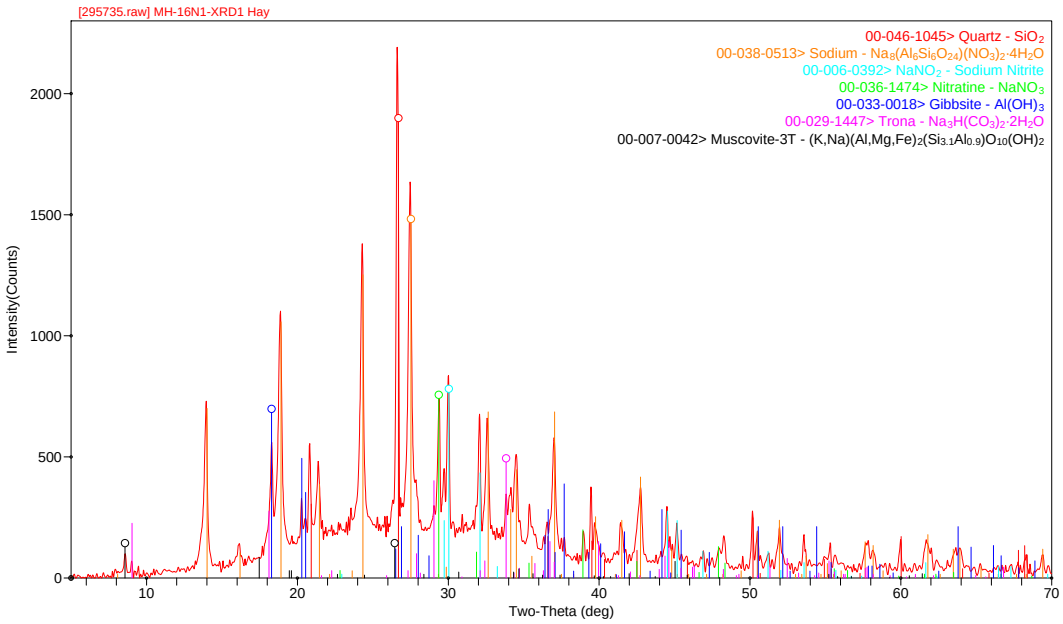

Figure 3-4 XRD Results for Tank 16H Annulus Sample HTF-16N1-2

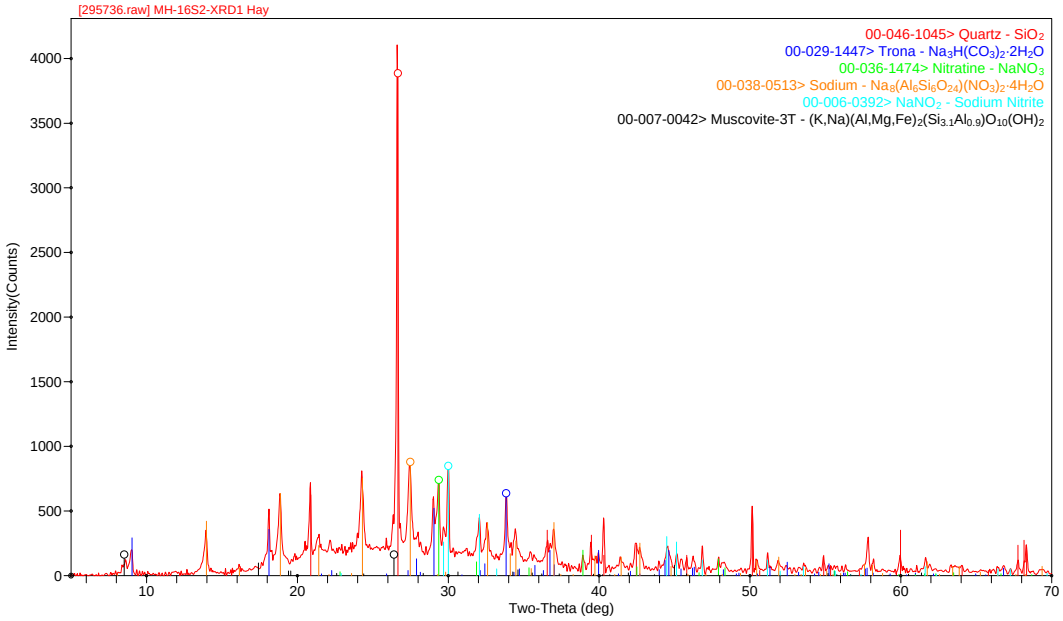

Figure 3-5 XRD Results for Tank 16H Annulus Sample HTF-16S2-1

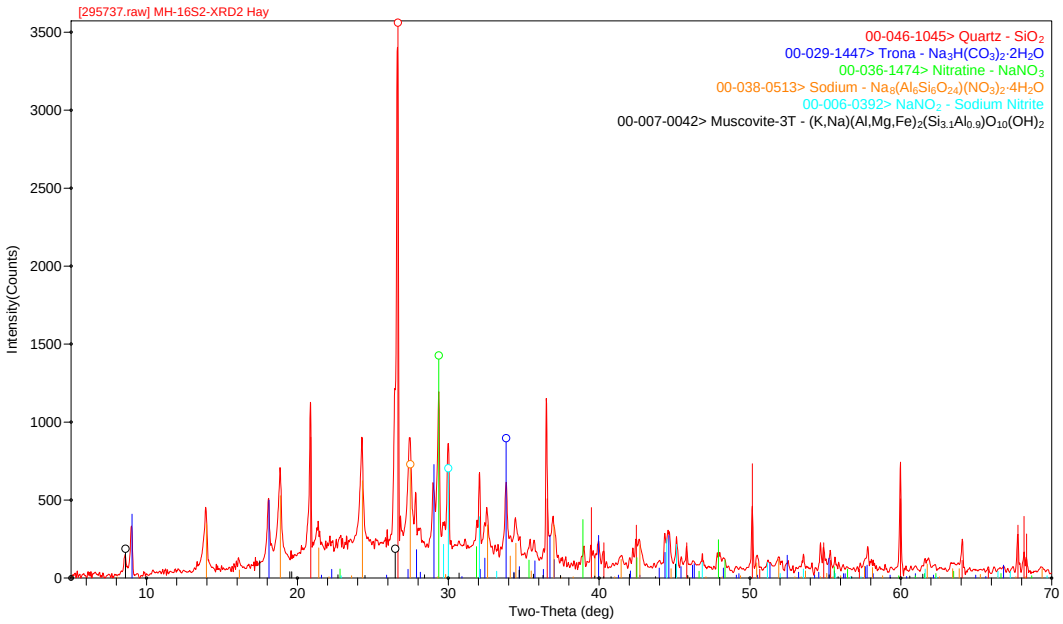

Figure 3-6 XRD Results for Tank 16H Annulus Sample HTF-16S2-2

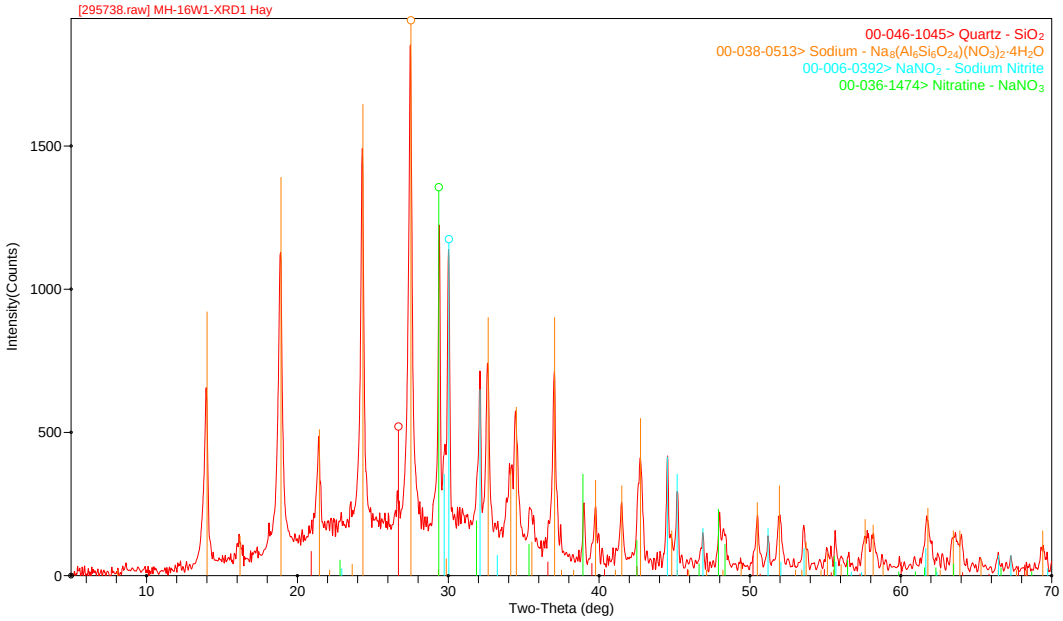

Figure 3-7 XRD Results for Tank 16H Annulus Sample HTF-16W1-1

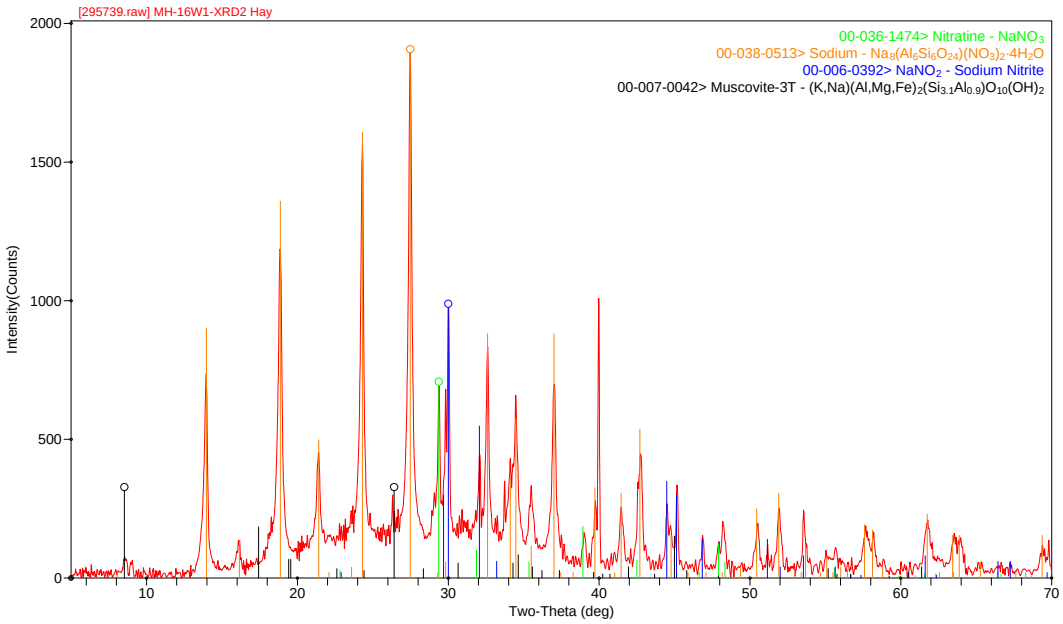

Figure 3-8 XRD Results for Tank 16H Annulus Sample HTF-16W1-2

Table 3-9. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-E-2 from Three Wash Cycles

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
F^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
CH_2O^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
Cl^- (IC)	6.00E+02	1.25E+02	2.40E+01	0.21	0.19
NO_2^- (IC)	1.53E+04	3.46E+03	8.43E+02	0.23	0.24
Br^- (IC)	<1.00E+02	<1.00E+02	<1.00E+01	-	-
NO_3^- (IC)	8.20E+03	1.93E+03	5.15E+02	0.24	0.27
PO_4^{3-} (IC)	1.30E+01	<1.00E+01	<1.00E+01	-	-
SO_4^{2-} (IC)	1.01E+03	2.02E+02	<5.00E+01	0.20	-
$C_2O_4^{2-}$ (IC)	1.72E+02	3.60E+01	1.10E+01	0.21	0.31
Free OH^- (T)	<8.50E+01	<8.50E+01	<8.50E+01	-	-
Ag (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Al (IE)	<6.64E-01	<6.64E-01	7.44E-01	-	-
B (IE)	<4.04E-01	<4.04E-01	<4.04E-01	-	-
Ba (IE)	<1.26E-01	<1.26E-01	<1.26E-01	-	-
Be (IE)	<1.60E-02	<1.60E-02	<1.60E-02	-	-
Ca (IE)	2.24E+00	8.90E-01	1.30E+00	0.40	1.5
Cd (IE)	<1.28E-01	<1.28E-01	<1.28E-01	-	-
Ce (IE)	<1.21E+00	<1.21E+00	<1.21E+00	-	-
Co (IE)	<1.94E-01	<1.94E-01	<1.94E-01	-	-
Cr (IE)	1.31E+00	6.96E-01	5.09E-01	0.53	0.73
Cu (IE)	<1.26E-01	<1.26E-01	<1.26E-01	-	-
Fe (IE)	<6.60E-02	1.18E-01	5.14E-01	-	-
Gd (IE)	<4.30E-01	<4.30E-01	<4.30E-01	-	-
K (IE)	1.53E+02	3.80E+01	1.05E+01	0.25	0.28
La (IE)	<1.34E-01	<1.34E-01	<1.34E-01	-	-
Li (IE)	4.90E-01	<2.92E-01	<2.92E-01	-	-
Mg (IE)	9.50E-02	5.70E-02	1.36E-01	0.60	2.4
Mn (IE)	<4.20E-02	<4.20E-02	<4.20E-02	-	-
Mo (IE)	2.94E+00	1.09E+00	<5.36E-01	0.37	-

Table 3-9. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-E-2 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
Na (IE)	1.52E+04	3.50E+03	1.13E+03	0.23	0.32
Ni (IE)	<9.10E-01	<9.10E-01	<9.10E-01	-	-
P (IE)	4.63E+00	<3.28E+00	<3.28E+00	-	-
Pb (IE)	<1.43E+00	<1.43E+00	<1.43E+00	-	-
S (IE)	3.27E+02	5.99E+01	2.14E+01	0.18	0.36
Sb (IE)	<2.13E+00	<2.13E+00	<2.13E+00	-	-
Si (IE)	1.73E+02	2.79E+02	2.93E+02	1.6	1.1
Sn (IE)	<1.12E+00	<1.12E+00	<1.12E+00	-	-
Sr (IE)	2.10E-02	<1.00E-02	<1.00E-02	-	-
Th (IE)	<5.36E-01	<5.36E-01	<5.36E-01	-	-
Ti (IE)	<7.60E-02	<7.60E-02	<7.60E-02	-	-
U (IE)	4.32E+01	<6.53E+00	<6.53E+00	-	-
V (IE)	<9.40E-02	<9.40E-02	<9.40E-02	-	-
Zn (IE)	<1.32E-01	<1.32E-01	<1.32E-01	-	-
Zr (IE)	<1.10E-01	<1.10E-01	<1.10E-01	-	-
Hg (CV)	9.40E+00	4.63E+00	4.71E+00	0.49	1.0
^{232}Th (IM)	<3.00E+01	<3.00E+01	<3.00E+00	-	-
^{233}U (IM)	1.84E-02	<1.50E-02	<1.50E-03	-	-
^{234}U (IM)	4.80E-02	<1.00E-02	<1.00E-03	-	-
^{235}U (IM)	1.49E+00	9.38E-02	4.97E-03	0.06	0.05
^{236}U (IM)	1.22E-01	1.37E-02	1.68E-03	0.11	0.12
^{238}U (IM)	4.06E+01	2.18E+00	1.20E-01	0.05	0.06
U-total (IM)	4.23E+01	2.29E+00	1.27E-01	0.05	0.06
^{137}Cs (GS)	1.02E-01	2.12E-02	7.37E-03	0.21	0.35
^{99}Tc (IM)	1.11E+00	3.11E-01	1.03E-01	0.28	0.33
^{237}Np (IM)	<2.50E-02	<2.50E-02	3.29E-03	-	-
^{239}Pu (IM)	<1.00E-02	<1.00E-02	2.17E-03	-	-
^{240}Pu (IM)	<2.50E-02	<2.50E-02	<2.50E-03	-	-
pH (pH units)	10.0	10.3	10.5	-	-

Table 3-9. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-E-2 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (dpm/ml)	Decant 2 (dpm/ml)	Decant 3 (dpm/ml)	Decant 2 / Decant 1	Decant 3 / Decant 2
^{137}Cs (GS)	1.96E+07	4.09E+06	1.42E+06	0.21	0.35
^{99}Tc (IM)	4.17E+04	1.17E+04	3.87E+03	0.28	0.33
^{233}U (IM)	3.95E+02	<3.21E+02	<3.21E+01	-	-
^{234}U (IM)	6.67E+02	<1.39E+02	<1.39E+01	-	-
^{235}U (IM)	7.16E+00	4.50E-01	2.38E-02	0.06	0.05
^{236}U (IM)	1.75E+01	1.97E+00	2.41E-01	0.11	0.12
^{237}Np (IM)	<3.91E+01	<3.91E+01	5.15E+00	-	-
^{238}U (IM)	3.03E+01	1.63E+00	8.97E-02	0.05	0.06
^{239}Pu (IM)	<1.36E+03	<1.36E+03	2.95E+02	-	-
^{240}Pu (IM)	<1.25E+04	<1.25E+04	<1.25E+03	-	-

Table 3-10. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-N-1 from Three Wash Cycles

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
F^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
CH_2O^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
Cl^- (IC)	2.51E+02	4.60E+01	1.00E+01	0.18	0.22
NO_2^- (IC)	1.88E+04	3.41E+03	6.99E+02	0.18	0.20
Br^- (IC)	<1.00E+02	<1.00E+02	<1.00E+01	-	-
NO_3^- (IC)	1.16E+04	2.24E+03	5.97E+02	0.19	0.27
PO_4^{3-} (IC)	1.60E+01	1.10E+01	<1.00E+01	-	-
SO_4^{2-} (IC)	1.13E+03	1.90E+02	<5.00E+01	0.17	-
$C_2O_4^{2-}$ (IC)	1.40E+02	3.50E+01	1.00E+01	0.25	0.29
Free OH^- (T)	<8.50E+01	<8.50E+01	<8.50E+01	-	-
Ag (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Al (IE)	<6.64E-01	<6.64E-01	2.74E+00	-	-
B (IE)	<4.04E-01	<4.04E-01	<4.04E-01	-	-
Ba (IE)	<1.26E-01	<1.26E-01	<1.26E-01	-	-
Be (IE)	<1.60E-02	<1.60E-02	<1.60E-02	-	-
Ca (IE)	6.30E+00	1.50E+00	1.23E+00	0.24	0.82
Cd (IE)	<1.28E-01	<1.28E-01	<1.28E-01	-	-
Ce (IE)	<1.21E+00	<1.21E+00	<1.21E+00	-	-
Co (IE)	<1.94E-01	<1.94E-01	<1.94E-01	-	-
Cr (IE)	1.81E+00	6.85E-01	4.72E-01	0.38	0.69
Cu (IE)	8.51E+00	1.84E+00	9.44E-01	0.22	0.51
Fe (IE)	<6.60E-02	1.32E-01	1.35E+00	-	-
Gd (IE)	<4.30E-01	<4.30E-01	<4.30E-01	-	-
K (IE)	1.30E+02	2.56E+01	<6.02E+00	0.20	-
La (IE)	<1.34E-01	<1.34E-01	<1.34E-01	-	-
Li (IE)	4.36E-01	<2.92E-01	<2.92E-01	-	-
Mg (IE)	1.04E+00	1.34E-01	1.12E-01	0.13	0.84
Mn (IE)	1.50E-01	<4.20E-02	<4.20E-02	-	-
Mo (IE)	2.05E+00	5.52E-01	< 0.536	0.27	-

Table 3-10. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-N-1 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
Na (IE)	2.21E+04	4.12E+03	1.03E+03	0.19	0.25
Ni (IE)	<9.10E-01	<9.10E-01	<9.10E-01	-	-
P (IE)	7.22E+00	<3.28E+00	<3.28E+00	-	-
Pb (IE)	<1.43E+00	<1.43E+00	<1.43E+00	-	-
S (IE)	3.73E+02	7.04E+01	<1.50E+01	0.19	-
Sb (IE)	<2.13E+00	<2.13E+00	<2.13E+00	-	-
Si (IE)	9.45E+01	1.56E+02	1.83E+02	1.7	1.2
Sn (IE)	<1.12E+00	<1.12E+00	<1.12E+00	-	-
Sr (IE)	2.40E-02	<1.00E-02	<1.00E-02	-	-
Th (IE)	<5.36E-01	<5.36E-01	<5.36E-01	-	-
Ti (IE)	<7.60E-02	<7.60E-02	<7.60E-02	-	-
U (IE)	1.21E+01	<6.53E+00	<6.53E+00	-	-
V (IE)	<9.40E-02	<9.40E-02	<9.40E-02	-	-
Zn (IE)	1.79E-01	1.73E-01	2.40E-01	0.97	1.4
Zr (IE)	<1.10E-01	<1.10E-01	<1.10E-01	-	-
Hg (CV)	1.33E+01	7.62E+00	6.03E+00	0.57	0.79
^{232}Th (IM)	<1.50E+01	<3.00E+00	<3.00E+00	-	-
^{233}U (IM)	<7.50E-03	<1.50E-03	<1.50E-03	-	-
^{234}U (IM)	2.37E-02	2.94E-03	<1.00E-03	0.12	-
^{235}U (IM)	3.98E-01	6.86E-02	4.87E-03	0.17	0.07
^{236}U (IM)	3.97E-02	5.47E-03	<1.00E-03	0.14	-
^{238}U (IM)	1.06E+01	1.72E+00	7.06E-02	0.16	0.04
U-total (IM)	1.11E+01	1.80E+00	7.54E-02	0.16	0.04
^{137}Cs (GS)	1.22E-01	2.48E-02	4.94E-03	0.20	0.20
^{99}Tc (IM)	7.52E-01	1.76E-01	5.96E-02	0.23	0.34
^{237}Np (IM)	4.77E-02	6.89E-03	7.67E-03	0.14	1.11
^{239}Pu (IM)	1.71E-02	1.21E-03	1.32E-03	0.07	1.09
^{240}Pu (IM)	<1.25E-02	<2.50E-03	<2.50E-03	-	-
pH (pH units)	9.7	10.0	10.2	-	-

Table 3-10. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-N-1 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (dpm/ml)	Decant 2 (dpm/ml)	Decant 3 (dpm/ml)	Decant 2 / Decant 1	Decant 3 / Decant 2
^{137}Cs (GS)	2.35E+07	4.78E+06	9.51E+05	0.20	0.20
^{99}Tc (IM)	2.83E+04	6.63E+03	2.24E+03	0.23	0.34
^{233}U (IM)	<1.61E+02	<3.21E+01	<3.21E+01	-	-
^{234}U (IM)	3.29E+02	4.08E+01	<1.39E+01	0.12	-
^{235}U (IM)	1.91E+00	3.29E-01	2.33E-02	0.17	0.07
^{236}U (IM)	5.70E+00	7.86E-01	<1.44E-01	0.14	-
^{237}Np (IM)	7.46E+01	1.08E+01	1.20E+01	0.14	1.1
^{238}U (IM)	7.90E+00	1.28E+00	5.26E-02	0.16	0.04
^{239}Pu (IM)	2.33E+03	1.64E+02	1.79E+02	0.07	1.1
^{240}Pu (IM)	<6.26E+03	<1.25E+03	<1.25E+03	-	-

Table 3-11. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-S-2 from Three Wash Cycles

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
F^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
CH_2O^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
Cl^- (IC)	7.10E+01	1.40E+01	<1.00E+01	0.20	-
NO_2^- (IC)	2.14E+04	3.68E+03	6.64E+02	0.17	0.18
Br^- (IC)	<5.00E+02	<5.00E+01	<1.00E+01	-	-
NO_3^- (IC)	1.72E+04	3.02E+03	5.77E+02	0.18	0.19
PO_4^{3-} (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
SO_4^{2-} (IC)	1.32E+03	2.11E+02	3.30E+01	0.16	0.16
$C_2O_4^{2-}$ (IC)	1.40E+02	2.60E+01	<1.00E+01	0.19	-
Free OH^- (T)	<8.50E+01	<8.50E+01	<8.50E+01	-	-
Ag (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Al (IE)	<6.64E-01	<6.64E-01	7.22E-01	-	-
B (IE)	<4.04E-01	<4.04E-01	<4.04E-01	-	-
Ba (IE)	<1.26E-01	<1.26E-01	<1.26E-01	-	-
Be (IE)	<1.60E-02	<1.60E-02	<1.60E-02	-	-
Ca (IE)	2.97E+00	9.70E-01	7.98E-01	0.33	0.82
Cd (IE)	<1.28E-01	<1.28E-01	<1.28E-01	-	-
Ce (IE)	<1.21E+00	<1.21E+00	<1.21E+00	-	-
Co (IE)	3.54E-01	<1.94E-01	<1.94E-01	-	-
Cr (IE)	1.26E+00	5.10E-01	3.73E-01	0.40	0.73
Cu (IE)	4.13E+00	2.14E-01	2.58E-01	0.05	1.2
Fe (IE)	1.06E-01	1.48E-01	3.46E-01	1.4	2.3
Gd (IE)	<4.30E-01	<4.30E-01	<4.30E-01	-	-
K (IE)	2.58E+02	4.41E+01	9.58E+00	0.17	0.22
La (IE)	<1.34E-01	<1.34E-01	<1.34E-01	-	-
Li (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Mg (IE)	3.84E-01	4.40E-02	1.49E-01	0.11	3.4
Mn (IE)	<4.20E-02	<4.20E-02	<4.20E-02	-	-
Mo (IE)	4.76E+00	8.97E-01	<5.36E-01	0.19	-

Table 3-11. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-S-2 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
Na (IE)	3.05E+04	5.42E+03	1.16E+03	0.18	0.21
Ni (IE)	<9.10E-01	<9.10E-01	<9.10E-01	-	-
P (IE)	6.33E+00	<3.28E+00	<3.28E+00	-	-
Pb (IE)	1.87E+00	<1.43E+00	<1.43E+00	-	-
S (IE)	4.86E+02	8.62E+01	<1.50E+01	0.18	-
Sb (IE)	<2.13E+00	<2.13E+00	<2.13E+00	-	-
Si (IE)	1.35E+02	1.96E+02	2.53E+02	1.5	1.3
Sn (IE)	<1.12E+00	<1.12E+00	<1.12E+00	-	-
Sr (IE)	3.50E-02	1.00E-02	<1.00E-02	0.29	-
Th (IE)	<5.36E-01	<5.36E-01	<5.36E-01	-	-
Ti (IE)	<7.60E-02	<7.60E-02	<7.60E-02	-	-
U (IE)	8.30E+00	<6.53E+00	<6.53E+00	-	-
V (IE)	1.42E-01	<9.40E-02	<9.40E-02	-	-
Zn (IE)	<1.32E-01	<1.32E-01	<1.32E-01	-	-
Zr (IE)	<1.10E-01	<1.10E-01	<1.10E-01	-	-
Hg (CV)	1.72E+01	1.06E+01	9.81E+00	0.62	0.93
^{232}Th (IM)	4.10E+00	<2.00E+00	<2.00E+00	-	-
^{233}U (IM)	4.11E-03	<2.00E-03	<2.00E-03	-	-
^{234}U (IM)	1.21E-02	2.86E-03	<2.00E-03	0.24	-
^{235}U (IM)	2.84E-01	5.36E-02	4.49E-03	0.19	0.08
^{236}U (IM)	2.23E-02	5.21E-03	<2.00E-03	0.23	-
^{238}U (IM)	6.80E+00	1.28E+00	6.29E-02	0.19	0.05
U-total (IM)	7.12E+00	1.34E+00	6.74E-02	0.19	0.05
^{137}Cs (GS)	1.63E-01	3.23E-02	6.75E-03	0.20	0.21
^{99}Tc (IM)	1.67E+00	3.20E-01	7.87E-02	0.19	0.25
^{237}Np (IM)	5.55E-02	5.33E-03	<2.00E-03	0.10	-
^{239}Pu (IM)	1.53E-02	<2.00E-03	<2.00E-03	-	-
^{240}Pu (IM)	3.00E-03	<2.00E-03	<2.00E-03	-	-
pH (pH units)	9.6	10.0	10.3	-	-

Table 3-11. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-S-2 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (dpm/ml)	Decant 2 (dpm/ml)	Decant 3 (dpm/ml)	Decant 2 / Decant 1	Decant 3 / Decant 2
^{137}Cs (GS)	3.13E+07	6.22E+06	1.30E+06	0.20	0.21
^{99}Tc (IM)	6.30E+04	1.21E+04	2.96E+03	0.19	0.25
^{233}U (IM)	8.79E+01	<4.28E+01	<4.28E+01	-	-
^{234}U (IM)	1.68E+02	3.98E+01	<2.78E+01	0.24	-
^{235}U (IM)	1.36E+00	2.57E-01	2.15E-02	0.19	0.08
^{236}U (IM)	3.20E+00	7.48E-01	<2.87E-01	0.23	-
^{237}Np (IM)	8.68E+01	8.34E+00	<3.13E+00	0.10	-
^{238}U (IM)	5.07E+00	9.53E-01	4.69E-02	0.19	0.05
^{239}Pu (IM)	2.08E+03	<2.72E+02	<2.72E+02	-	-
^{240}Pu (IM)	1.50E+03	<1.00E+03	<1.00E+03	-	-

Table 3-12. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-W-1 from Three Wash Cycles

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
F^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
CH_2O^- (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
Cl^- (IC)	2.28E+02	5.20E+01	1.10E+01	0.23	0.21
NO_2^- (IC)	2.42E+04	5.07E+03	1.03E+03	0.21	0.20
Br^- (IC)	<5.00E+02	<5.00E+01	<1.00E+01	-	-
NO_3^- (IC)	1.47E+04	3.33E+03	8.79E+02	0.23	0.26
PO_4^{3-} (IC)	<1.00E+01	<1.00E+01	<1.00E+01	-	-
SO_4^{2-} (IC)	1.01E+03	1.94E+02	3.60E+01	0.19	0.19
$C_2O_4^{2-}$ (IC)	3.72E+02	7.80E+01	1.40E+01	0.21	0.18
Free OH^- (T)	<8.50E+01	<8.50E+01	<8.50E+01	-	-
Ag (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Al (IE)	<6.64E-01	<6.64E-01	9.47E-01	-	-
B (IE)	<4.04E-01	<4.04E-01	<4.04E-01	-	-
Ba (IE)	<1.26E-01	<1.26E-01	<1.26E-01	-	-
Be (IE)	<1.60E-02	<1.60E-02	<1.60E-02	-	-
Ca (IE)	9.91E+00	1.64E+00	5.02E-01	0.17	0.31
Cd (IE)	<1.28E-01	<1.28E-01	<1.28E-01	-	-
Ce (IE)	<1.21E+00	<1.21E+00	<1.21E+00	-	-
Co (IE)	<1.94E-01	<1.94E-01	<1.94E-01	-	-
Cr (IE)	1.57E+00	4.87E-01	2.94E-01	0.31	0.60
Cu (IE)	2.78E-01	<1.26E-01	<1.26E-01	-	-
Fe (IE)	<6.60E-02	1.24E-01	5.98E-01	-	-
Gd (IE)	<4.30E-01	<4.30E-01	<4.30E-01	-	-
K (IE)	3.36E+02	7.31E+01	1.86E+01	0.22	0.25
La (IE)	<1.34E-01	<1.34E-01	<1.34E-01	-	-
Li (IE)	<2.92E-01	<2.92E-01	<2.92E-01	-	-
Mg (IE)	9.15E-01	5.60E-02	6.00E-02	0.06	1.1
Mn (IE)	<4.20E-02	<4.20E-02	<4.20E-02	-	-
Mo (IE)	7.40E+00	1.55E+00	<5.36E-01	0.21	-

Table 3-12. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-W-1 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (mg/L)	Decant 2 (mg/L)	Decant 3 (mg/L)	Decant 2 / Decant 1	Decant 3 / Decant 2
Na (IE)	2.39E+04	5.27E+03	1.36E+03	0.22	0.26
Ni (IE)	<9.10E-01	<9.10E-01	<9.10E-01	-	-
P (IE)	7.43E+00	<3.28E+00	<3.28E+00	-	-
Pb (IE)	<1.43E+00	<1.43E+00	<1.43E+00	-	-
S (IE)	3.33E+02	6.42E+01	<1.50E+01	0.19	-
Sb (IE)	<2.13E+00	<2.13E+00	<2.13E+00	-	-
Si (IE)	1.37E+02	2.01E+02	2.46E+02	1.5	1.2
Sn (IE)	<1.12E+00	<1.12E+00	<1.12E+00	-	-
Sr (IE)	4.50E-02	4.50E-02	2.50E-02	1.0	0.56
Th (IE)	<5.36E-01	<5.36E-01	<5.36E-01	-	-
Ti (IE)	<7.60E-02	<7.60E-02	<7.60E-02	-	-
U (IE)	7.70E+00	<6.53E+00	<6.53E+00	-	-
V (IE)	<9.40E-02	<9.40E-02	<9.40E-02	-	-
Zn (IE)	<1.32E-01	<1.32E-01	<1.32E-01	-	-
Zr (IE)	<1.10E-01	<1.10E-01	<1.10E-01	-	-
Hg (CV)	1.52E+01	6.78E+00	4.44E+00	0.45	0.65
^{232}Th (IM)	<2.00E+00	<2.00E+00	<2.00E+00	-	-
^{233}U (IM)	3.60E-03	<2.00E-03	<2.00E-03	-	-
^{234}U (IM)	9.62E-03	2.16E-03	<2.00E-03	0.22	-
^{235}U (IM)	2.60E-01	3.94E-02	3.68E-03	0.15	0.09
^{236}U (IM)	2.09E-02	6.59E-03	<2.00E-03	0.32	-
^{238}U (IM)	6.67E+00	9.83E-01	5.85E-02	0.15	0.06
U-total (IM)	6.96E+00	1.03E+00	6.21E-02	0.15	0.06
^{137}Cs (GS)	1.76E-01	3.85E-02	8.57E-03	0.22	0.22
^{99}Tc (IM)	2.33E+00	5.19E-01	1.23E-01	0.22	0.24
^{237}Np (IM)	6.76E-02	9.08E-03	2.27E-03	0.13	0.25
^{239}Pu (IM)	<2.00E-03	<2.00E-03	<2.00E-03	-	-
^{240}Pu (IM)	<2.00E-03	<2.00E-03	<2.00E-03	-	-
pH (pH units)	9.7	10.1	10.3	-	-

Table 3-12. Composition of the Water Leachates from Tank 16H Annulus Sample HTF-16-W-1 from Three Wash Cycles (continued)

Analyte (Method)	Decant 1 (dpm/ml)	Decant 2 (dpm/ml)	Decant 3 (dpm/ml)	Decant 2 / Decant 1	Decant 3 / Decant 2
^{137}Cs (GS)	3.39E+07	7.42E+06	1.65E+06	0.22	0.22
^{99}Tc (IM)	8.76E+04	1.96E+04	4.63E+03	0.22	0.24
^{233}U (IM)	7.70E+01	<4.28E+01	<4.28E+01	-	-
^{234}U (IM)	1.34E+02	3.00E+01	<2.78E+01	0.22	0.93
^{235}U (IM)	1.25E+00	1.89E-01	1.76E-02	0.15	0.09
^{236}U (IM)	3.00E+00	9.46E-01	<2.87E-01	0.32	-
^{237}Np (IM)	1.06E+02	1.42E+01	3.56E+00	0.13	0.25
^{238}U (IM)	4.97E+00	7.33E-01	4.36E-02	0.15	0.06
^{239}Pu (IM)	<2.72E+02	<2.72E+02	<2.72E+02	-	-
^{240}Pu (IM)	<1.00E+03	<1.00E+03	<1.00E+03	-	-

Table 3-13. Estimated Percent of Solids Dissolved in the Water Leaching Tests and Oxalic Acid Leaching Tests on Tank 16H Annulus Samples

Sample ID	Estimated Percent Dissolved in Water Leach	Estimated Percent Dissolved in Oxalic Acid Leach
HTF-16-E-2	20%	34%
HTF-16-N-1	29%	38%
HTF-16-S-2	35%	46%
HTF-16-W-1	33%	47%

Figure 3-9 Water Leaching Test on the Tank 16H Annulus Samples

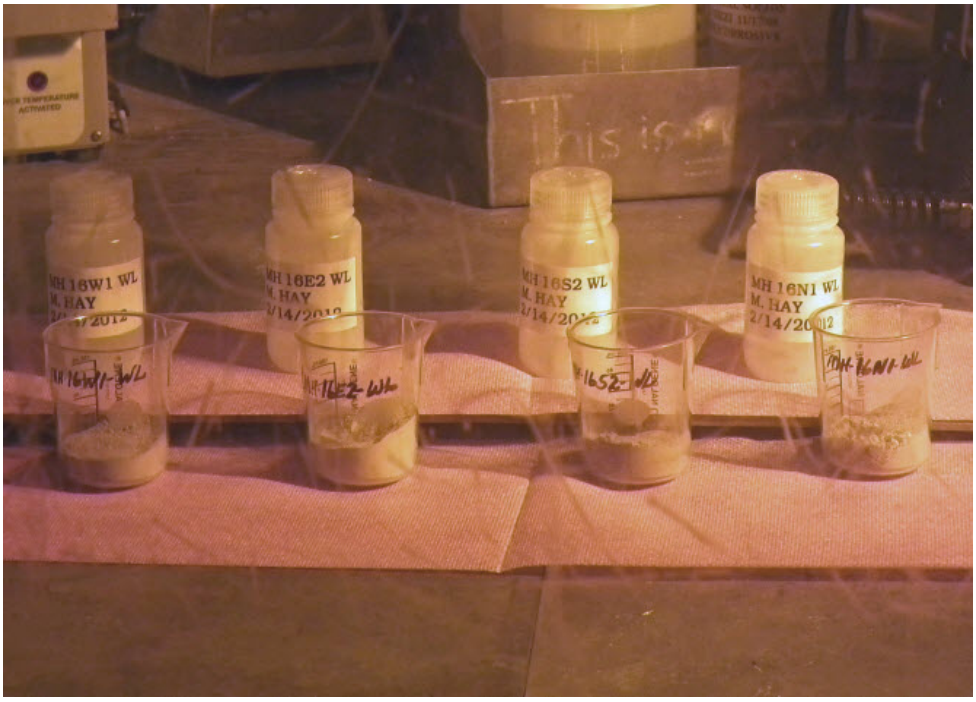

Figure 3-10 Water Leached Solids from the Tank 16H Annulus Samples

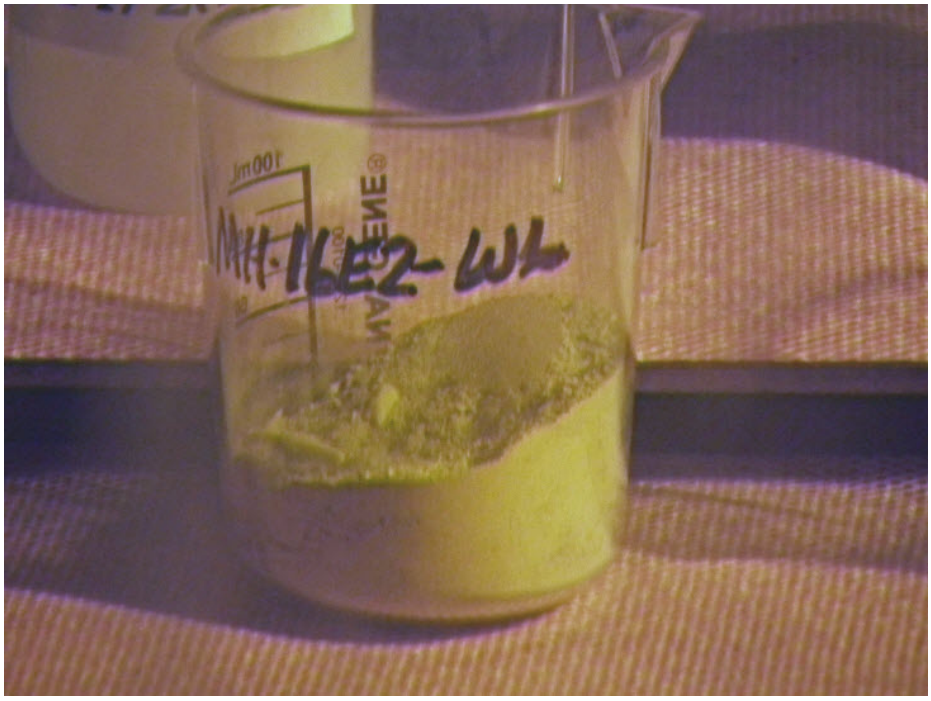

Figure 3-11 Water Leached Solids from Tank 16H Annulus Sample HTF 16-E-2

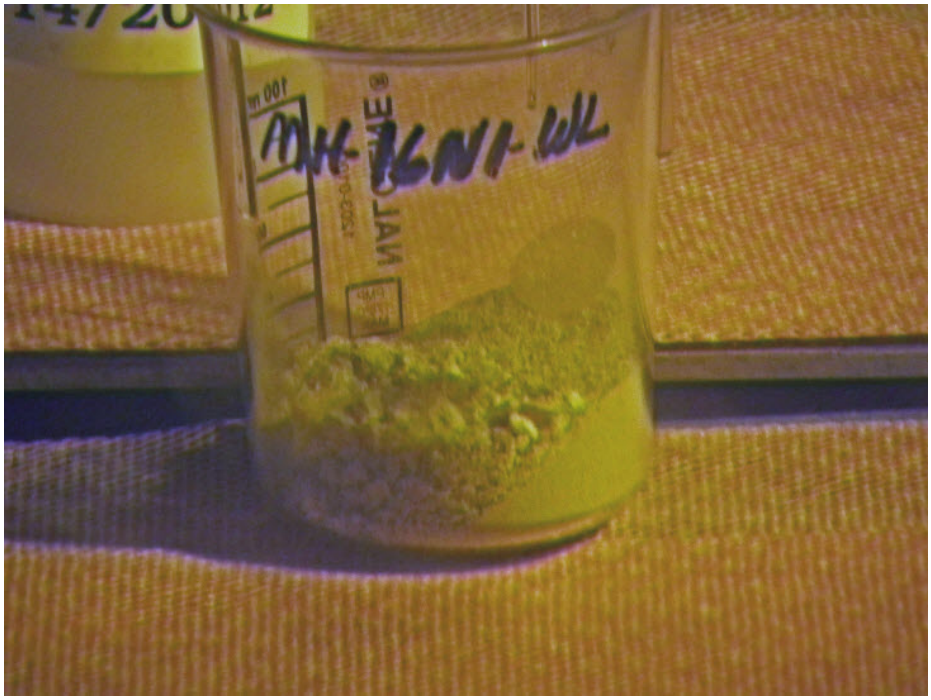

Figure 3-12 Water Leached Solids from Tank 16H Annulus Sample HTF 16-N-1

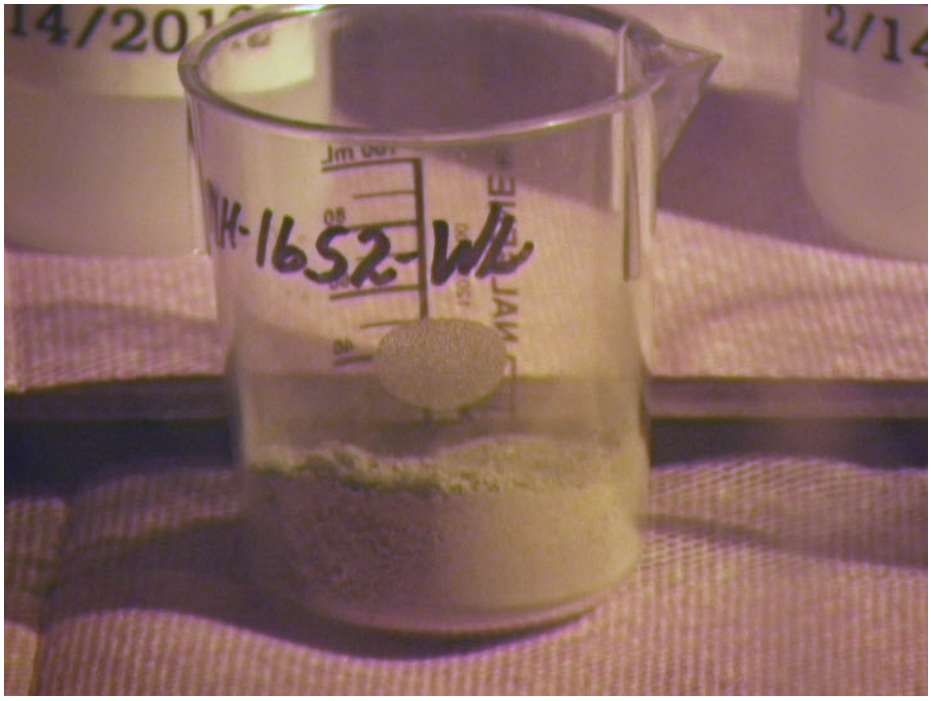

Figure 3-13 Water Leached Solids from Tank 16H Annulus Sample HTF 16-S-2

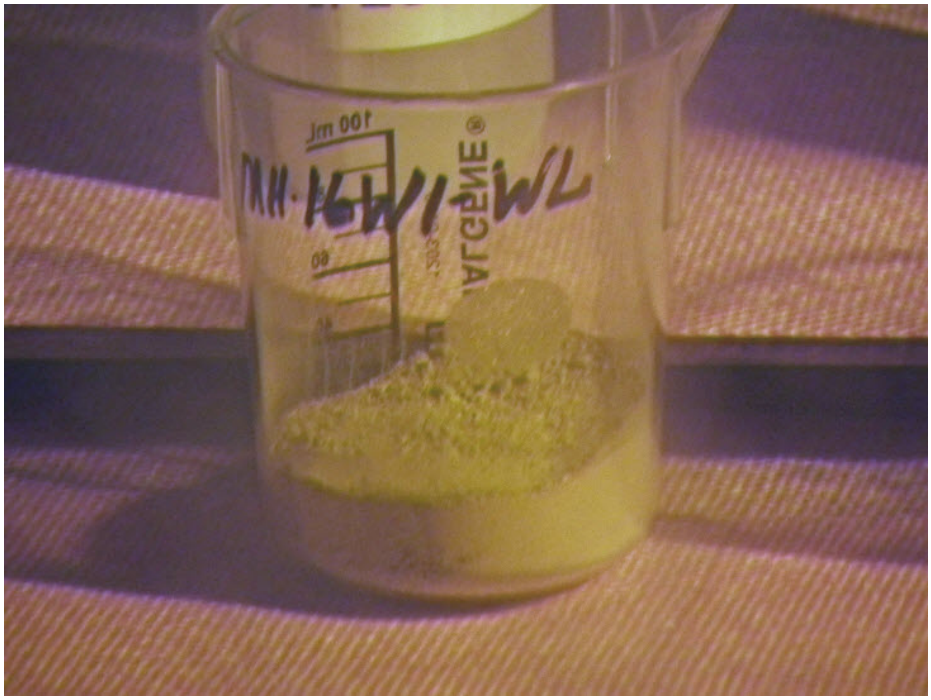

Figure 3-14 Water Leached Solids from Tank 16H Annulus Sample HTF 16-W-1

Table 3-14. XRD Result for the Water Leached Solids from the Tank 16H Annulus Samples-Estimated Mineral Phase Composition

Crystalline Phase	HTF-16E2 Replicate 1	HTF-16E2 Replicate 2	HTF-16N1 Replicate 1	HTF-16N1 Replicate 2	HTF-16S2 Replicate 1	HTF-16S2 Replicate 2	HTF-16W1 Replicate 1	HTF-16W1 Replicate 2
Amorphous	31%	25%	17%	19%	27%	27%	27%	26%
SiO_2	25%	40%	50%	40%	40%	35%	30%	-
$Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$	35%	25%	20%	30%	30%	30%	45%	75%
$NaNO_3$	5%	5%	-	-	-	-	-	-
$KAl_2(AlSi_3O_{10})(OH)_2$	-	-	4%	-	-	5%	-	-
$Al(OH)_3$	5%	5%	10%	10%	5%	5%	-	-

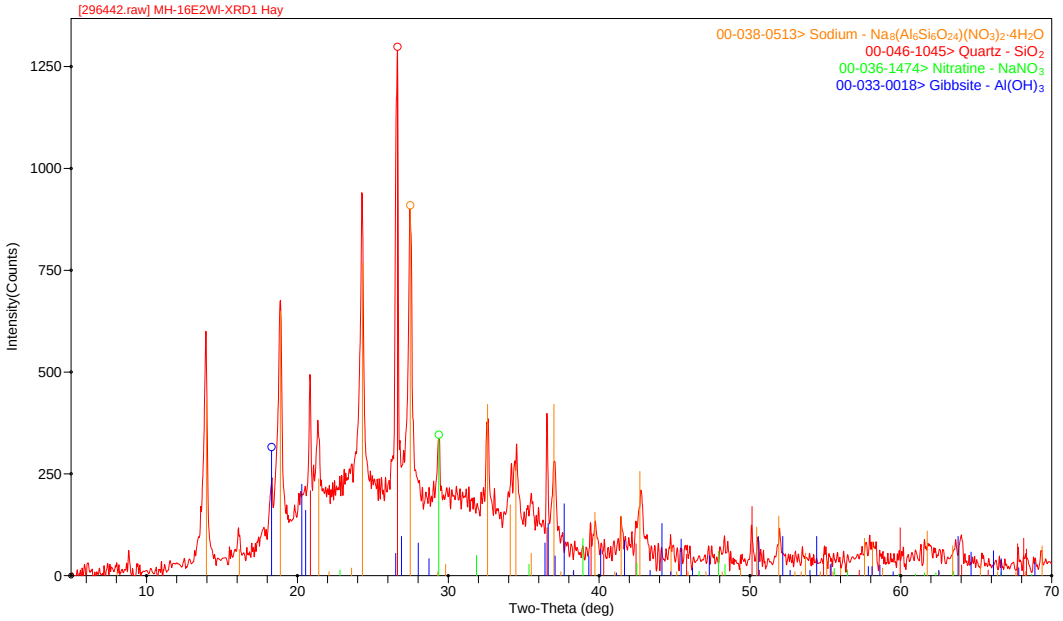

Figure 3-15 XRD Results of the Water Leached Solids for HTF-16E2-1

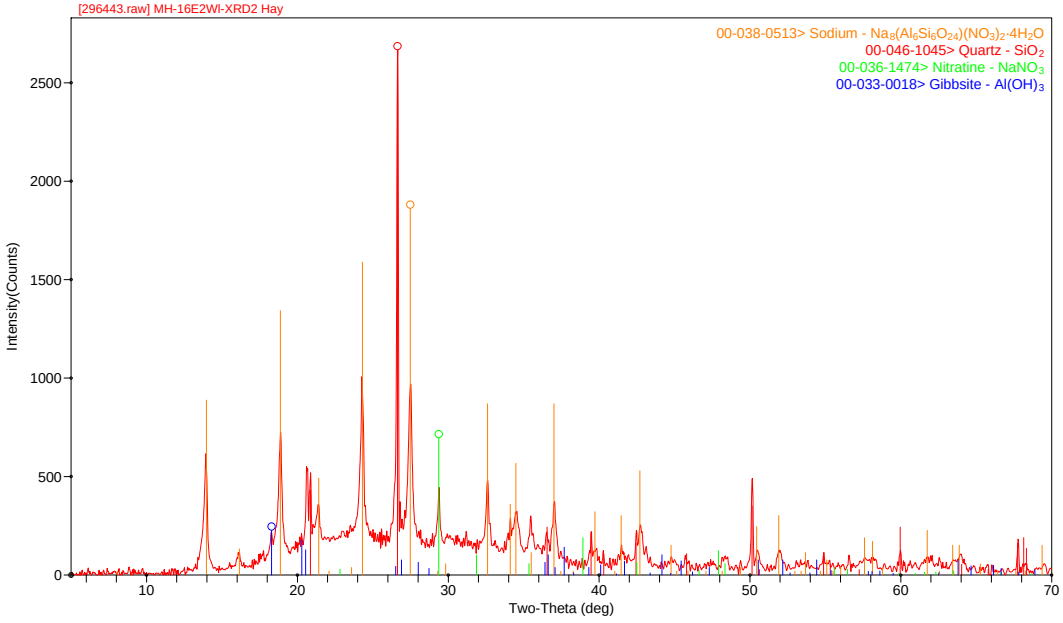

Figure 3-16 XRD Results of the Water Leached Solids for HTF-16E2-2

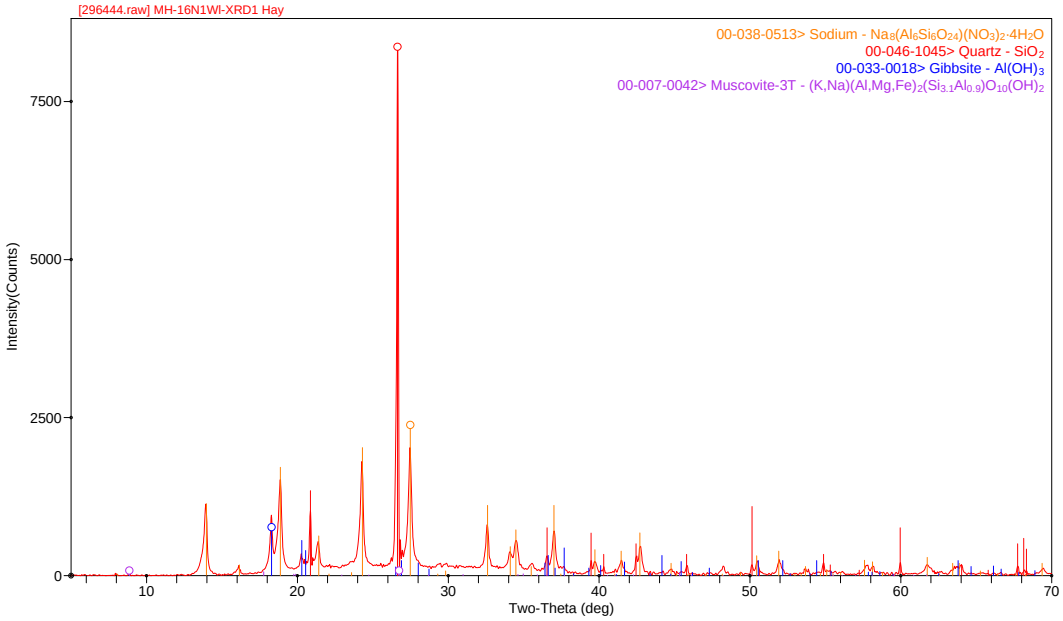

Figure 3-17 XRD Results of the Water Leached Solids for HTF-16N1-1

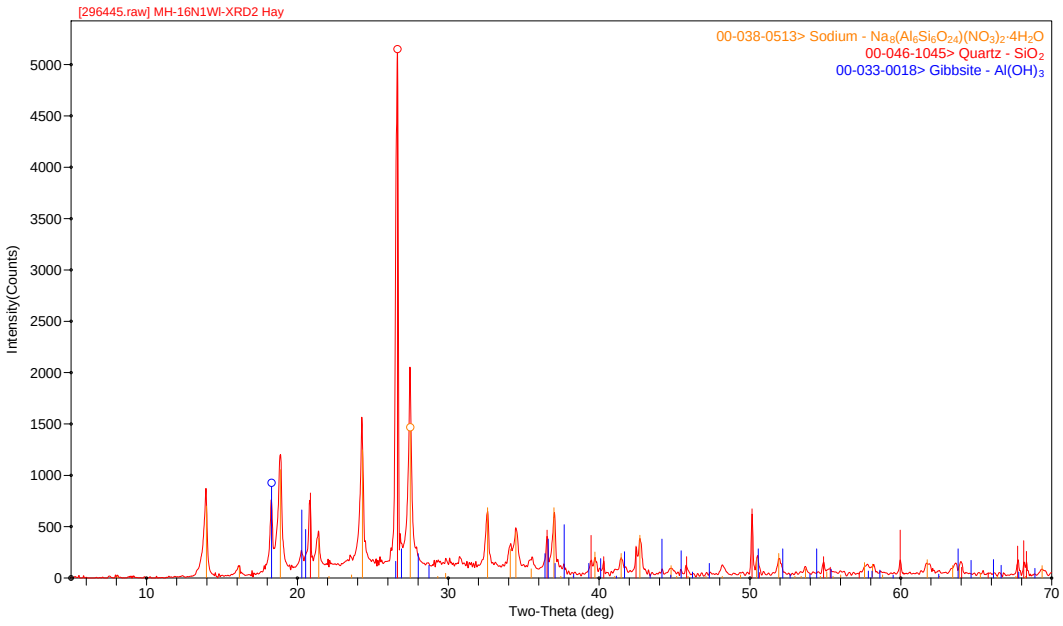

Figure 3-18 XRD Results of the Water Leached Solids for HTF-16N1-2

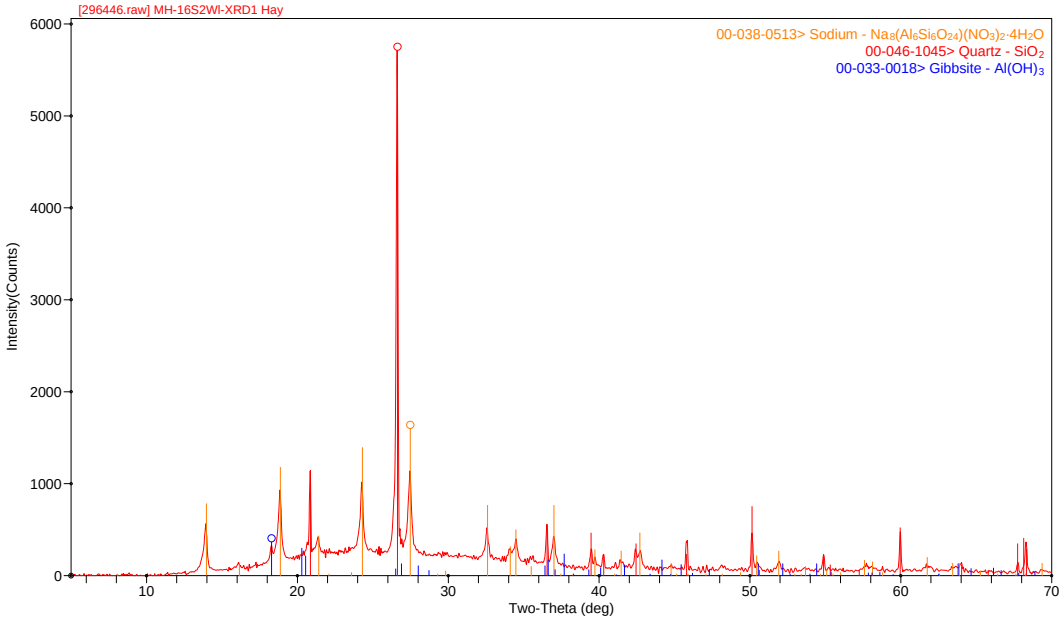

Figure 3-19 XRD Results of the Water Leached Solids for HTF-16S2-1

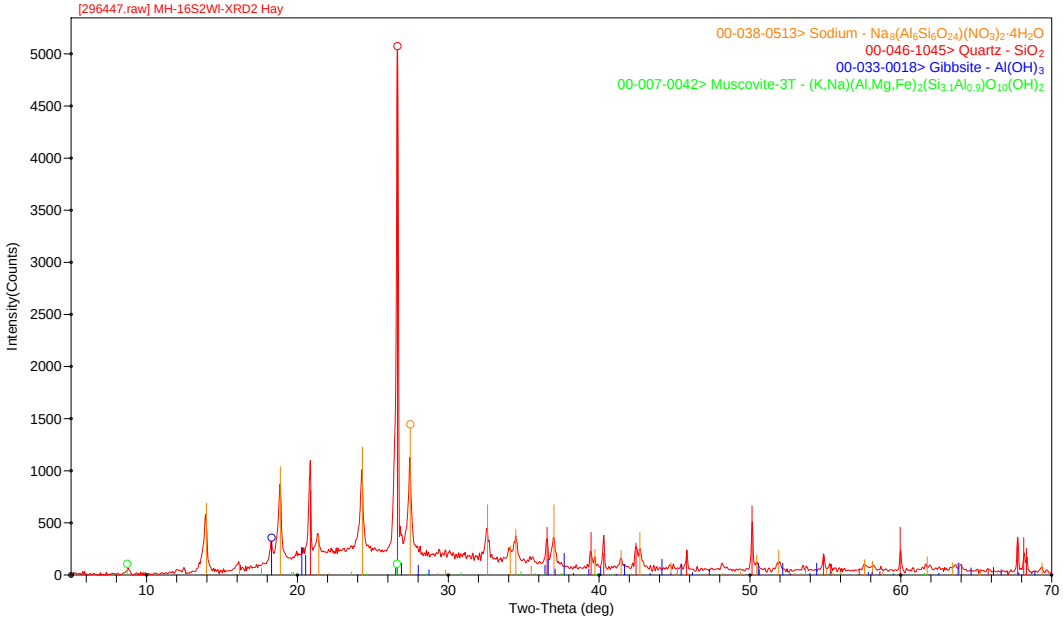

Figure 3-20 XRD Results of the Water Leached Solids for HTF-16S2-2

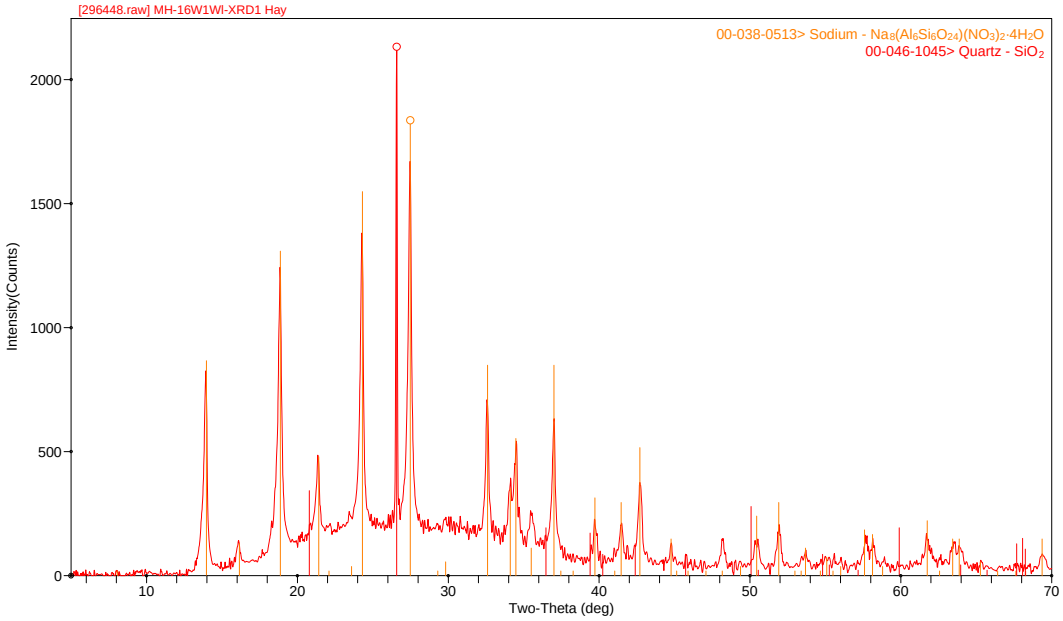

Figure 3-21 XRD Results of the Water Leached Solids for HTF-16W1-1

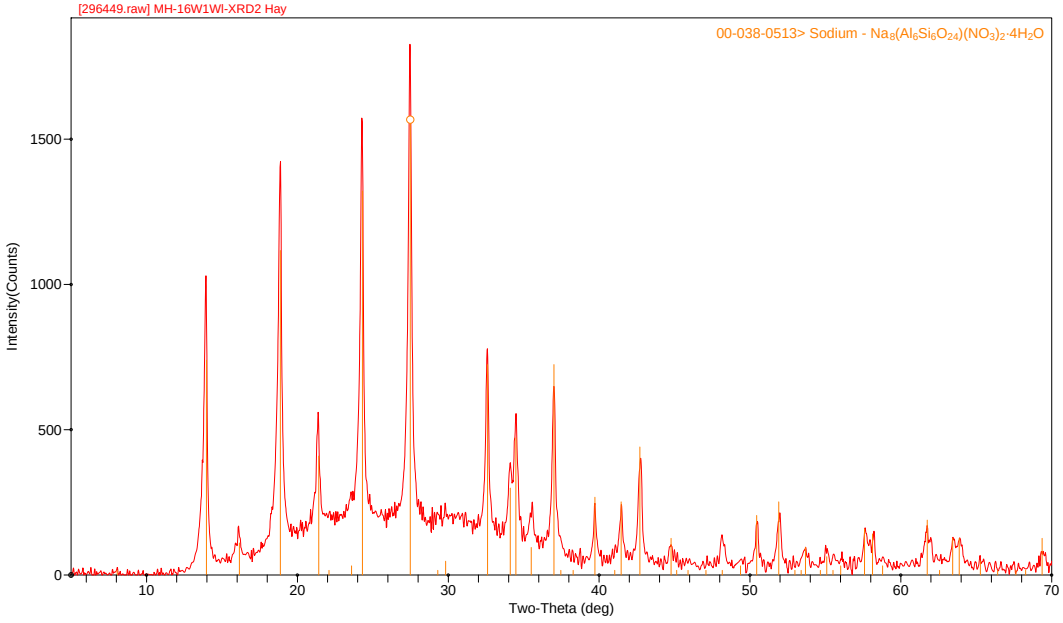

Figure 3-22 XRD Results of the Water Leached Solids for HTF-16W1-2

4.0 Conclusions and Recommendations

Table 4-1 summarizes the composition of the major components of the as-received annulus samples. The analysis found all four annulus samples to be composed mainly of Si, Na, and Al and lesser amounts of other elements. Sample HTF-16-E-2 contains a higher concentration of Fe than the other three samples. The estimated percent of each crystalline mineral phase assigned from the XRD data generally agree with the composition from the chemical analysis. The data indicate quartz (SiO_2) and sodium aluminum nitrate silicate hydrate ($Na_8(Al_6Si_6O_{24})(NO_3)_2.4H_2O$) as the predominant crystalline mineral phases in the samples The XRD data also indicate the presence of crystalline sodium nitrate, sodium nitrite, gibbsite, hydrated sodium bicarbonate, and muscovite.

Table 4-1. Summary of the Composition of the As-Received Solids from Tank 16H Annulus Samples (Wt% Total Dried Solids)

Element	HTF-16-E-2 (wt%)	HTF-16-N-1 (wt%)	HTF-16-S-2 (wt%)	HTF-16-W-1 (wt%)
Si	17.0	21.1	17.1	11.7
Na	12.1	12.1	15.9	17.9
Al	6.62	7.27	4.20	8.35
Fe	6.54	1.12	2.01	3.58
Ca	1.58	0.61	0.47	0.14
S	0.14	0.16	0.55	0.14
Hg	0.21	0.10	0.35	0.26

Based on the weight of solids remaining at the end of the test, the water leaching test results from Table 3-13 indicate approximately 20-35% of the solids dissolved after three contacts with an approximately 3:1 volume of water at 45 °C. The HTF-16-S-2 sample showed the highest dissolution with ~35%, followed by HTF-16-W-1 at ~33%, HTF-16-N-1 at ~29%, and HTF-16-E-2 at ~20%.

Table 4-2 provides a summary of the leaching behavior of the radionuclides analyzed in the samples from the water leaching test. The concentrations of the radionuclides in the first water contact of the leaching test and the initial as-received solids were used to calculate the percent dissolved for each of the radionuclides analyzed. The total strontium results from the ICP-ES method provide insight into the behavior for ^{90}Sr. The water leaching removed large amounts of the uranium in samples HTF-16-E-2 and HTF-16-N-1

and ~1/3 of the ^{99}Tc from all four samples. Most of the other radionuclides analyzed showed low solubility in the water leaching test.

Table 4-2. Summary of the Estimated Percentage Dissolved for each Radionuclide in the Water Leaching Test on Tank 16H Annulus Samples

Radionuclide	HTF-16-E-2	HTF-16-N-1	HTF-16-S-2	HTF-16-W-1
Cs-137	3.0%	3.4%	6.3%	3.8%
Sr	0.1%	0.6%	0.4%	0.3%
Tc-99	28%	33%	38%	31%
U-235	52%	61%	18%	9.7%
U-238	52%	70%	19%	9.6%
Np-237	<3.1%	17%	11%	9.0%
Pu-239	<0.8%	5.0%	2.1%	<0.1%

The chemical analysis of the leachates and the XRD results of the remaining solids indicate sodium salts of nitrate, nitrite, sulfate, and possibly carbonate/bicarbonate make up the majority of the dissolved material. Based on the results of the leachate analysis, the majority of these salts were dissolved in the first water contact and simply diluted with each subsequent water contact. The soluble portion of the ^{137}Cs and ^{99}Tc that dissolved in the three water contacts also dissolved in the first water contact. The data on uranium, plutonium, and neptunium appears more complicated. Solution concentrations from water leaching in the annulus may be lower than found in these small scale tests due to the monolithic structure of the waste in the annulus and resulting lower available surface area.

The preliminary data on the oxalic acid leaching test, shown in Table 3-13, indicate the three acid contacts at 45 °C dissolved from ~34-47% of the solids. The somewhat higher dissolution found in the oxalic acid leaching test versus the water leaching test might be offset by the tendency of the oxalic acid solutions to take on a gel-like consistency. The filtered solids left behind after three oxalic acid contacts were sticky and formed large clumps after drying. These two observations could indicate potential processing difficulties with solutions and solids from oxalic acid leaching. The gel formation might be avoided by using larger volumes of the acid. Further testing would be recommended before using oxalic acid to dissolve the Tank 16H annulus waste to ensure no processing difficulties are encountered in the full scale process.

5.0 References

1. M. S. Hay, S. H. Reboul, “Task Technical and Quality Assurance Plan for the Analysis of Tank 16 Annulus Process Samples”, SRNL-RP-2011-01169, Rev. 1, November 14, 2011.

2. Technical Task Request, HLE-TTR-2011-010, C. B. Sherburne. November 14, 2011.

3. M. S. Hay, “Characterization of Samples from Tank 16H Annulus”, WSRC-STI-2008-00203, Rev. 0, May 2008.

4. C. J. Coleman, R. A. Dewberry, M. F. Bryant, J. J. Gemmill, “SRL's Performance in Round Robin #6 - Analysis of Simulated Defense Waste Glass”, WSRC-TR-91-187, Rev. 0, May 31, 1991.

5. M. S. Hay, T. B. Edwards, “Statistical Analysis of ESP Verification Test Samples”, WSRC-RP-94-1224, Rev. 0, November 4, 1994.

6. M. S. Hay, “Results of the Characterization and Dissolution Tests of Samples from Tank 16H”, WSRC-RP-99-00124, January 19, 1999.

7. L. Reynolds, “Summary of Current Data on Tank 16H Annulus Samples”, DPST-79-360, January 9, 1979.

Appendix A

Table A-1. Composition of Blanks and Standards for the As-Received Solids from Tank 16H Annulus Sample

Analyte (Method)	Aqua Regia Glass Std (mg/kg)	Per Fusion Glass Std (mg/kg)	Glass Std Average (mg/kg)	Glass Std Composition (mg/kg)	Aqua Regia Blank (mg/kg)	Per Fusion Blank (mg/kg)
Ag (IE)	<1.08E+02	<1.17E+03	<6.39E+02	-	<1.15E+02	<1.15E+03
Al (IE)	2.27E+04	2.43E+04	2.35E+04	2.50E+04	<1.41E+01	2.23E+03
B (IE)	2.54E+04	2.43E+04	2.49E+04	2.69E+04	<1.62E+01	<1.14E+02
Ba (IE)	7.99E+02	7.54E+02	7.77E+02	7.90E+02	1.28E+00	<4.16E+01
Be (IE)	2.77E+01	2.83E+01	2.80E+01	-	<4.80E-01	<6.40E+00
Ca (IE)	1.06E+04	1.29E+04	1.18E+04	1.02E+04	5.61E+01	3.10E+03
Cd (IE)	6.82E+00	*<2.51E+01*	6.82E+00	-	<2.48E+00	<2.48E+01
Ce (IE)	<5.30E+01	<4.88E+02	<2.71E+02	-	<5.66E+01	<4.82E+02
Co (IE)	6.33E+01	8.66E+01	7.50E+01	-	<7.76E+00	<6.80E+01
Cr (IE)	6.49E+02	6.58E+02	6.54E+02	6.40E+02	1.15E+01	<8.16E+01
Cu (IE)	2.28E+01	<5.10E+01	2.28E+01	3.00E+01	<5.04E+00	<5.04E+01
Fe (IE)	9.55E+04	1.27E+05	1.11E+05	9.79E+04	7.86E+01	2.39E+02
Gd (IE)	<1.61E+01	<6.32E+01	<3.97E+01	-	<1.72E+01	<6.24E+01
K (IE)	2.15E+04	2.34E+04	2.25E+04	2.26E+04	<2.05E+02	<2.05E+03
La (IE)	1.23E+01	*<5.43E+01*	1.23E+01	-	<4.32E+00	<5.36E+01
Li (IE)	1.43E+04	1.43E+04	1.43E+04	1.49E+04	<1.17E+01	<1.17E+02
Mg (IE)	5.00E+03	5.06E+03	5.03E+03	5.20E+03	9.80E+00	1.24E+01
Mn (IE)	1.41E+04	1.40E+04	1.41E+04	1.46E+04	<4.24E+00	<1.68E+01
Mo (IE)	<2.01E+01	<2.17E+02	<1.19E+02	-	<2.14E+01	<2.14E+02
Na (IE)	8.19E+04	-	8.19E+04	8.52E+04	<5.90E+02	-
Ni (IE)	7.92E+03	7.81E+03	7.87E+03	8.27E+03	<3.64E+01	<3.64E+02
P (IE)	1.19E+03	*<7.01E+02*	1.19E+03	1.10E+03	<1.31E+02	<6.93E+02
Pb (IE)	<5.36E+01	<5.80E+02	<3.17E+02	-	<5.73E+01	<5.73E+02
S (IE)	<5.62E+02	<6.07E+03	<3.32E+03	-	<6.00E+02	<6.00E+03
Sb (IE)	<9.57E+01	<8.63E+02	<4.79E+02	-	<1.02E+02	<8.53E+02
Si (IE)	-	2.03E+05	2.03E+05	2.24E+05	-	<3.51E+02
Sn (IE)	6.90E+01	*<4.54E+02*	6.90E+01	-	<4.49E+01	<4.49E+02

Divide mg/kg values by 1E+04 to convert to wt % basis

Table A-1. Composition of Blanks and Standards for the As-Received Solids from Tank 16H Annulus Sample (continued)

Analyte (Method)	Aqua Regia Glass Std (mg/kg)	Per Fusion Glass Std (mg/kg)	Glass Std Average (mg/kg)	Glass Std Composition (mg/kg)	Aqua Regia Blank (mg/kg)	Per Fusion Blank (mg/kg)
Sr (IE)	3.15E+01	6.07E+01	4.61E+01	3.00E+01	<4.00E-01	2.80E+01
Th (IE)	1.54E+02	*<4.44E+02*	1.54E+02	-	<4.38E+01	<4.38E+02
Ti (IE)	5.25E+03	6.57E+03	5.91E+03	6.90E+03	<3.04E+00	1.84E+01
U (IE)	<2.45E+03	<2.64E+03	<2.55E+03	-	<2.61E+02	<2.61E+03
V (IE)	1.10E+02	9.55E+01	1.03E+02	-	<3.76E+00	<3.76E+01
Zn (IE)	1.64E+02	1.20E+03	6.82E+02	1.60E+02	<7.36E+00	<7.36E+01
Zr (IE)	5.08E+02	-	5.08E+02	9.60E+02	<4.40E+00	-
Hg (CV)	<4.12E+01	-	<4.12E+01	-	<4.40E+01	-
^{233}U (IM)	<7.49E-01	<8.10E-01	<7.79E-01	-	<8.00E-01	<8.00E-01
^{234}U (IM)	<7.49E-01	<8.10E-01	<7.79E-01	-	<8.00E-01	<8.00E-01
^{235}U (IM)	<7.49E-01	<8.10E-01	<7.79E-01	-	<8.00E-01	<8.00E-01
^{236}U (IM)	<7.49E-01	<8.10E-01	<7.79E-01	-	<8.00E-01	<8.00E-01
^{238}U (IM)	6.15E+00	5.55E+00	5.85E+00	-	<8.00E-01	<1.60E+00
U-total (IM)	6.15E+00	5.55E+00	5.85E+00	-	-	-
Analyte (Method)	**Aqua Regia Glass Std (dpm/g)**	**Per Fusion Glass Std (dpm/g)**	**Glass Std Average (dpm/g)**	**Glass Std Composition (dpm/g)**	**Aqua Regia Blank (dpm/g)**	**Per Fusion Blank (dpm/g)**
^{90}Sr (BS)	2.91E+04	*<1.66E+06*	2.91E+04	-	3.79E+04	<1.64E+06
^{137}Cs (GS)	<4.42E+06	<5.63E+06	<5.03E+06	-	<4.20E+06	<6.36E+06
^{238}Pu (SA)	<2.12E+03	<6.43E+03	<4.28E+03	-	3.39E+03	<4.45E+02
$^{239/240}$Pu (SA)	<3.02E+03	<8.71E+03	<5.87E+03	-	<3.19E+03	<1.06E+03
^{99}Tc (IM)	<2.82E+04	<3.05E+04	<2.94E+04	-	<3.01E+04	<3.01E+04
^{237}Np (IM)	<1.17E+03	<1.27E+03	<1.22E+03	-	<1.25E+03	<1.25E+03
^{239}Pu (IM)	<1.02E+05	<1.10E+05	<1.06E+05	-	<1.09E+05	<1.09E+05
^{240}Pu (IM)	<3.75E+05	<6.08E+05	<4.92E+05	-	<4.01E+05	<6.01E+05

Divide mg/kg values by 1E+04 to convert to wt % basis