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Tank 41H Salt-Well Sample Analysis

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Summary

This report provides results of the analysis of the Tank 41H salt-well criticality sample TK-41-HTF-E-173. Statistics are provided for the analyses of the wet as-received salt and the solids insoluble in multiple strikes of excess inhibited water. The dried insoluble solids comprise 0.22 wt. % of the wet as-received sample and contain approximately 0.2 wt. % uranium with a ^{235}U enrichment of 12.6%. The bulk of the insoluble solid material (84.4 wt. %) is best represented by $Al_2O_3 * 3H_2O$, which is gibbsite or its polymorph bayerite.

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Abbreviations

C.I. Confidence Interval
N/A Not Applicable
n.d. Not determined

Introduction

The High Level Waste (HLW) division plans to dissolve at least 100,000 gallons of salt in Tank 41H for use as a feed to the Low Curie Salt process.[1] The planned dissolution and removal methods are not covered by the existing Nuclear Criticality Safety Evaluation (NCSE) for Tank 41H. This sample analysis provides results to support the NCSE needed to qualify the entire contents of the tank for dissolution and removal.

Prior to sampling, HLW removed the supernate from the surface of Tank 41H and transferred it to the 3H-Evaporator system. On May 11, 2002, HLW completed mining a salt-well from the Tank 41H saltcake surface (~351") to within 24" of the tank bottom. The solids that remained in the newly mined hole were allowed to settle for about three weeks, after which time two core samples were pulled from near the bottom of this hole.

HLW requested that the Savannah River Technology Center (SRTC) perform Sample-Plan Suite 1 (Salt Criticality Characterization) on the Tank 41H saltcake samples in support of the criticality evaluation[2]. The Task Technical and Quality Assurance Plan documents the planned sample analysis.[3] SRTC analyzed the as-received sample and the washed insoluble solids for uranium and plutonium isotopes and neutron poisons. This analysis covers one of the two samples received (TK-41-HTF-E-173).

Sample Description

On June 4, 2002, HLW obtained two samples of salt from Tank 41H. On the first attempt to use Sampler #1, the sampler moved easily into and down the well and stopped at 132". The mast was disconnected from the crane and pounded to a sample depth of 96", where it stopped. When the sampler was brought out of the tank, the indicator revealed that the sampler was empty. During a second attempt to use Sampler #1, the sampler moved easily into and down the salt-well and stopped well past 96", and was subsequently pounded to 20". The indicator revealed that Sampler #1 was full. Sampler #2 moved easily into and down the well on the first attempt, stopped at 36", and was subsequently pounded to 6". The indicator revealed that Sampler #2 was full.[4]

Table 1: Tank 41H salt-well criticality sample details.

Sample Jar #	Sampler # (and fraction)	Tank Farm ID#	Weight (g)	Description
1	1	TK-41-HTF-E-173	153.8	Overall homogeneity throughout sampler. White/off-white with a few darker specks.
2	2 dark	TK-41-HTF-E-174	64.5	From lower portion of sampler, primarily outside of retaining basket. Darker and wetter than Sample Jars #1 and #3. Slushy with an orange/brown color. Consistent with the appearance of a salt slurry containing sludge.
3	2 light	TK-41-HTF-E-174	80.2	From the top portion of the sampler, packed inside of the retaining basket. Similar appearance to Sample Jar #1, slightly wetter.

The two Tank 41H salt-well core samples arrived at SRTC and were placed in the shielded cells on June 5 and were opened on June 6. Table 1 outlines the description of the two samples as they were placed into three glass jars. Both sample vials were essentially full (see Figure 1), containing portions of salt both inside and outside of the retaining basket. Sampler #1 contained 153.8 grams of salt that appeared similar throughout the vial. Sampler #2 contained 144.7 grams of material that exhibited two fractions of different appearance. The interface between the two fractions was not distinct, but was approximately at the sampler retaining fingers. The material in the upper portion of Sampler #2 (inside of the retaining fingers and packed into the cap) resembled the material from Sampler #1. The material in the lower portion of Sampler #2 (outside of the retaining fingers) appeared darker and wetter than the material from Sampler #1 and the upper portion of Sampler #2. These “light” and “dark” fractions of TK-41-HTF-E-174 were placed in separate sample jars (Jar #3 and #2, respectively) so that a decision could be made as to what portions of the sample to analyze. The samples, including the dark portions, looked lighter than expected based on the appearance of the recent Tank 37H salt-well core sample.[5]

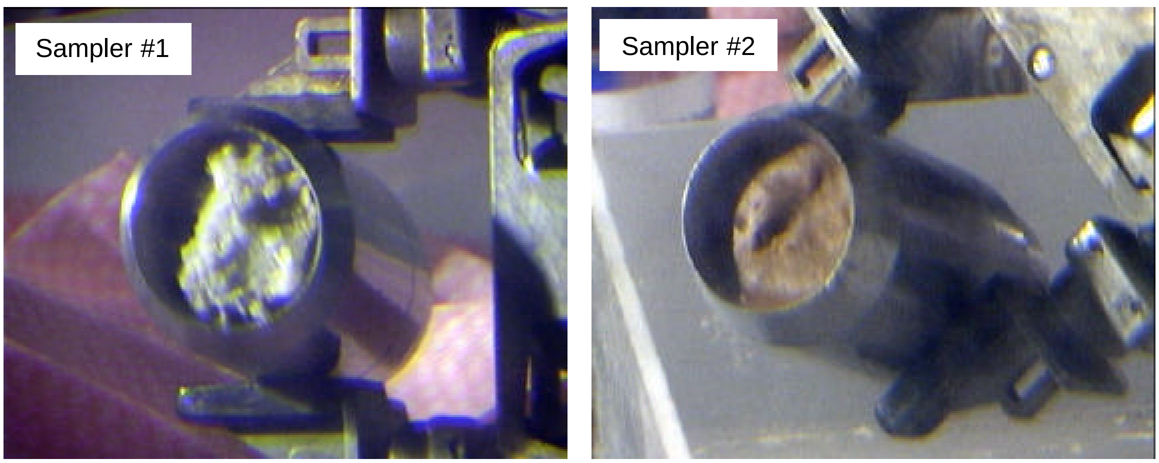

Figure 1: Bottom (outside of retaining fingers) of Sampler #1 (TK-41-HTF-E-173, left) and Sampler #2 (TK-41-HTF-E-174, right).

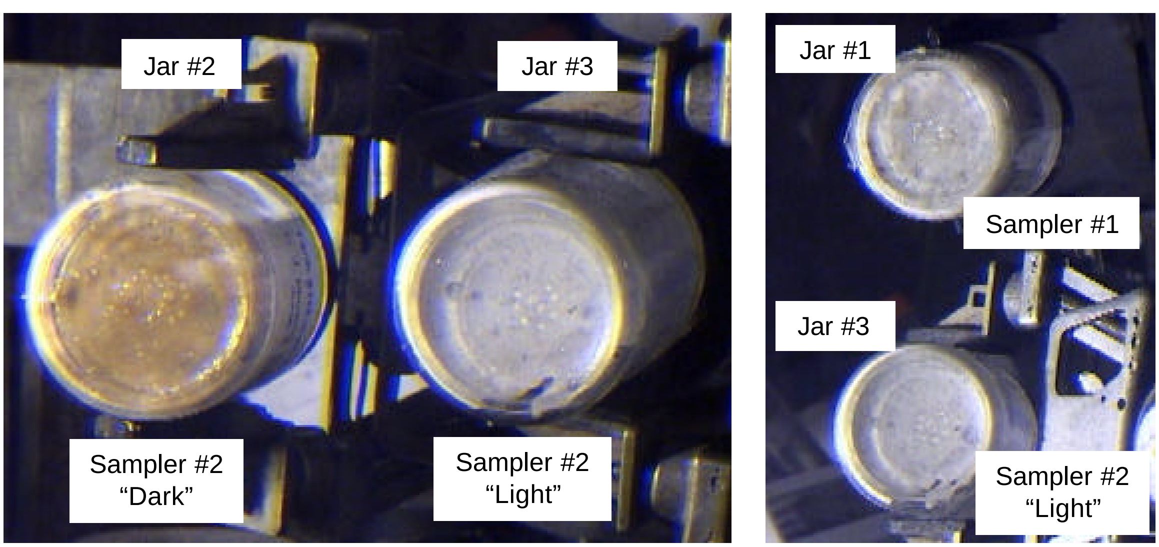

Figure 2: Appearance of the Tank 41H salt sample fractions.

Experimental

The saltcake material from only Sampler #1 (TK-41-HTF-E-173) was homogenized and characterized for uranium and plutonium isotopes and potential neutron poisons. Portions of the as-received sample were digested by two methods and characterized in triplicate. The remainder of the salt from Sampler #1 was washed three times with inhibited water, resulting in insoluble solids that were dried, digested by two methods, and characterized in duplicate. The following are the details of this general procedure.

Three aliquots of the as-received sample were pulled for aqua regia dissolution and three were pulled for sodium peroxide fusion dissolution. Approximately 0.5 g of original sample material was dissolved into 250 mL of liquid. The dissolved samples were removed from the shielded cells and transferred to the Analytical Development Section (ADS) for analysis for actinides and neutron poisons. Analyses conducted include inductively coupled plasma-emission spectrometry (ICP-ES) for various elemental species including neutron poisons, inductively coupled plasma-mass spectrometry (ICP-MS) for various actinide isotopes, and PuTTA (Pu-238/241) analysis for ^{238}Pu and ^{241}Pu. Additional analyses were requested to satisfy requirements for the drain disposal of waste.

Prior to digesting the aliquots with sodium peroxide, those samples were dried to a constant weight at 115 ± 10 °C. (The sodium peroxide reacts with water before the digestion is completed. Drying is not necessary for samples dissolved using aqua regia.) This drying step allowed a measurement of the weight fraction total solids in the sample.

The insoluble solids preparation process uses a conservatively large amount of wash-water (roughly 3 washes with a 3:1 ratio of inhibited water to original salt for each wash) when compared with in-tank dissolution processes. The insoluble solids were separated from the salt cake sample by washing portions of the salt in four polypropylene centrifuge tubes. Each tube was filled with a 10-g aliquot of the as-received sample and washed three times with 30 g of inhibited water (0.0158M NaOH) to dissolve the soluble material. After each wash, the samples were centrifuged to minimize the solids volume and the clear supernate was decanted. The density of the as-received salt was estimated by noting the displacement of the 10 g of salt during the first addition of 30 g of inhibited water. After the last of the three washes, not enough solids remained in the four tubes to allow for adequate analysis. An additional 13 g of salt was added to each tube and washed once with 40 g of inhibited water. The remainder of the sample in Jar #1 was processed by the addition of 10 g of salt to each tube and washing three times with 40 g of inhibited water. The resulting insoluble solids, when dried, was an amount of material adequate for duplicate analysis of the two dissolution methods.

After the final wash, the insoluble solids in each of the tubes were dried at 60 °C. The solids were transferred into polytetrafluoroethylene beakers and dried to a constant weight at 115 ± 10 °C. Two aliquots each of the dried solids were then dissolved using aqua regia and sodium peroxide fusion digestion. Approximately 0.07 g of dried insoluble solids was dissolved into 100 mL of liquid in each of the dissolutions. The dried insoluble solids were submitted for the same analyses as the as-received sample aliquots.

Results

As-received and Insoluble Solids Analysis

The wet as-received salt sample TK-41-HTF-E-173 had a specific gravity of 1.9 (95% C.I. of ±0.2) and a total solids content of 83.5 wt. % (95% C.I. of ±9.3 wt. %). After the washing process was complete, 1.14 g of wet insoluble solids was obtained. In all, 131.3 g of the wet as-received sample was consumed in the washing process, yielding 0.291 g of dry insoluble solids. Thus, the dry insoluble solids comprised 0.22 wt. % of the wet as-received sample.

Table 2 and Table 3 contain the summaries of the analytical results for the wet as-received sample and the dried insoluble solids, respectively. Data from the individual dissolutions used to calculate the statistics reported in Table 2 and Table 3 are contained in the appendix (see Table 5 and Table 6 for the as-received results and Table 7 and Table 8 for the insoluble solids results).

Results from ICP-ES, ICP-MS, and Pu-238/241 are given in weight percentage. The as-received sample results are reported on a wet basis and the insoluble solids results are reported on a dry basis. Sodium values are not reported for peroxide fusion digestions because of the sodium content of the sample is small in relation to that in the added sodium peroxide. It is evident that significant zinc is introduced during peroxide fusion digestion. Aqua regia dissolution is a poor method for the dissolution of silicon, and thus no silicon values are reported for aqua regia of the insoluble solids. A combined average of the three (for as-received sample) or two (for insoluble solids) determinations for each of the two digestion methods is reported. Values that are below detection limits are signified by "<", and averages for replicates that contain a mixture of actual values and below detection limit values are signified by " ". In addition to the analytical results reported by ADS, several calculated values are given: total uranium, total plutonium, and ^{235}U/total uranium. The Pu-238/241 method did not routinely lead to the mass quantification of ^{239}Pu and ^{240}Pu, so the Total Pu value may be low in instances where ICP-MS did not measure ^{239}Pu and ^{240}Pu above the minimum detection limits.

Table 2 and Table 3 contain several statistics: the average of the determinations ($\bar{x}$), the standard deviation of the determinations (s), and the one- and two-sided 95% confidence intervals (C.I.) for the sample average. The reported standard deviations capture a combination of the variance in the composition of the salt sample and the variance from analytical uncertainties. These standard deviations are used to calculate the 95% C.I. about the average by the following equation:

$$\text{C.I.} = t_{\alpha,n-1}\frac{s}{\sqrt{n}}$$

where t is Student's t-statistic, n is the number of determinations, and α is the tail probability (0.025 for the two-sided 95% C.I. and 0.05 for the one-sided 95% C.I.). This can be used to bound the true average value, m, with 95% confidence. As more replicate analyses are performed, this confidence interval on the true average value decreases even if the standard deviation remains the same, due to the influence of the increase in n and the reduction in the t-statistic.

The two-sided confidence interval should be used in cases where you want to bound the average between two numbers with 95% confidence (i.e., $m = \bar{x} \pm$ 95% C.I.). For example, it may be important to know that the ^{235}U enrichment in the insoluble solids was found to be 0.126 ± 0.005 with 95% confidence. The one-sided confidence interval should be used to assure that the average is above or below a specific value with 95% confidence (i.e., $m > \bar{x}$ - 95% C.I. or $m < \bar{x}$ + 95% C.I.). For instance, it also may be important to know that aluminum comprises at least 29.6 - 1.59 wt. % (or 28.0 wt. %) of the insoluble solids with 95% confidence. This approach for calculating the confidence interval assumes that the determined quantities of each analyte are normally distributed about the true average quantity. These confidence intervals are valid for the tank sample analyzed.

Table 2: Summary of the analytical results of the wet as-received Tank 41H salt sample (TK-41-HTF-E-173).

Species/Analysis	Sample Average	Sample Std Dev.	2-sided 95% C.I.	1-sided 95% C.I.
ICP-ES (wt. %)				
Al	2.76E+00	2.88E-01	3.02E-01	2.37E-01
Cr	8.88E-03	6.49E-03	8.06E-03	6.19E-03
Cu	7.76E-03	2.99E-03	3.14E-03	2.46E-03
Fe	1.35E-02	1.38E-02	1.72E-02	1.32E-02
Na	2.49E+01	1.15E-01	2.87E-01	1.95E-01
P	3.64E-02	8.46E-03	1.05E-03	8.06E-03
ICP-MS (wt. %)				
M-235 (U)	1.12E-04	2.04E-05	2.14E-05	1.68E-05
M-238 (U,Pu)	1.02E-03	3.88E-04	4.07E-04	3.19E-04
Pu-238/241 (wt. %)				
^{238}Pu	9.33E-07	1.62E-07	1.70E-07	1.34E-07
^{241}Pu	7.08E-08	2.67E-08	2.81E-08	2.20E-08
Actinides Summary (wt. %)				
Total U	1.16E-03	3.68E-04	3.86E-04	3.03E-04
Total Pu	1.00E-06	1.49E-07	1.57E-07	1.23E-07
Enrichment (mass fraction)				
^{235}U/Tot.U	0.104	0.032	0.033	0.026

Table 3: Summary of the analytical results of the dry insoluble solids from the Tank 41H salt sample (TK-41-HTF-E-173).

Species/Analysis	Sample Average	Sample Std Dev.	2-sided 95% C.I.	1-sided 95% C.I.
ICP-ES (wt. %)				
Al	2.96E+01	1.35E+00	2.15E+00	1.59E+00
Ba	3.76E-02	9.37E-03	1.49E-02	1.10E-02
Ca	7.34E-01	1.67E-01	2.66E-01	1.96E-01
Cr	1.14E+00	4.24E-01	6.75E-01	4.99E-01
Cu	7.76E-02	5.47E-02	8.71E-02	6.44E-02
Fe	2.78E+00	1.71E+00	2.72E+00	2.01E+00
Mn	1.08E-01	4.61E-02	7.33E-02	5.42E-02
Na	7.20E-01	1.98E-02	1.78E-01	8.84E-02
Ni	7.50E-02	2.67E-02	4.25E-02	3.14E-02
Si	4.13E-01	7.71E-02	6.92E-01	3.44E-01
Sr	1.36E-01	4.06E-02	6.46E-02	4.78E-02
Zn	8.73E-02	1.94E-02	1.75E-01	8.68E-02
ICP-MS (wt. %)				
M-232 (Th, U)	1.79E-03	2.58E-04	2.32E-03	1.15E-03
M-233 (U)	1.35E-03	4.60E-04	7.32E-04	5.41E-04
M-234 (U)	7.30E-03	2.70E-03	4.30E-03	3.18E-03
M-235 (U)	2.17E-02	8.02E-03	1.28E-02	9.44E-03
M-236 (U)	7.95E-03	2.77E-03	4.41E-03	3.26E-03
M-237 (Np)	6.44E-03	2.31E-03	3.67E-03	2.71E-03
M-238 (U, Pu)	1.34E-01	4.91E-02	7.81E-02	5.78E-02
M-239 (Pu)	4.42E-04	—	—	—
Pu-238/241 (wt. %)				
^{238}Pu	2.05E-04	8.61E-05	1.37E-04	1.01E-04
^{241}Pu	1.14E-06	6.54E-07	1.04E-06	7.69E-07
Actinides Summary (wt. %)				
Total U	1.72E-01	6.30E-02	1.00E-01	7.41E-02
Total Pu	6.54E-04	4.91E-04	7.81E-04	5.78E-04
Enrichment (mass fraction)				
^{235}U/Tot.U	0.126	0.003	0.005	0.004

Insoluble Solids Assignment

As evident from Table 3, the insoluble solids in the Tank 41H sample consist primarily of aluminum compounds, with the additional presence of calcium, iron, chromium, and other trace compounds. What follows is an assignment discreet compounds to the elemental analysis in order to perform a mass balance for the insoluble solids. The assignment of the compounds is based on previously reported characterization of SRS and Hanford sludge solids as well as solids isolated after multiple contacts with inhibited water.[6] The insoluble solids were prepared by drying at 115 °C, which would remove the free water but retain the waters of hydration. For simplicity, all elements present in quantities < 0.1 wt. % were not included in the mass balance analysis.

The silicon is assumed to be present as sodium aluminosilicate, $Na_8Al_6Si_6O_{24}(NO_3)_2 * 4H_2O$. This aluminosilicate compound requires a small portion of the total aluminum present, and the bulk of the aluminum is assigned as $Al_2O_3 * 3H_2O$ (also written $Al(OH)_3$). This aluminum hydroxide phase is either gibbsite or its polymorph, bayerite. The remainder of the solids assignments does not have a major impact on the overall mass balance. The iron is assigned as goethite, $Fe_2O_3 * H_2O$, the calcium as $CaCO_3$, the manganese as Mn_2O_3, the chromium as $Cr_2O_3 * H_2O$, the uranium as $Na_2U_2O_7$, and the strontium as $SrCO_3$. The excess sodium is assigned as sodium sulfate decahydrate. As seen in Table 4, the solids from this assignment sum to 97.6 wt. % (nearly 100 wt. %). This lends validation to the assignments of the major insoluble components, namely $Al_2O_3 * 3H_2O$. Based on this composition, the waters of hydration represent 30.0 wt % of the solids (excluding the contribution from the presumed $Na_2SO_4 * 10H_2O$).

Aluminum is the major metallic component of the insoluble solids. If a less hydrated form of alumina is substituted in this analysis for gibbsite or bayerite (e.g., boehmite – $Al_2O_3 * H_2O$ or AlOOH), the mass balance does not close. The assignment of aluminum as boehmite yields to only 64.9 wt. % boehmite and a sum of the solid masses of only 78.1 wt. %. In addition, the selection of gibbsite or bayerite is consistent with the production and storage conditions for the saltcake in Tank 41H. Generally, higher temperatures (>100 °C for long periods of time) are required to convert gibbsite and bayerite into boehmite.

Table 4: Mass balance of components assigned to Tank 41H insoluble solids, based on sample TK-41-HTF-E-173.

Assigned Solid	mol/100g	wt. %
$Al_2O_3 * 3H_2O$	0.541	84.4
$Na_8Al_6Si_6O_{24}(NO_3)_2 * 4H_2O$	0.00245	2.68
$Cr_2O_3 * H_2O$	0.0109	1.86
$Fe_2O_3 * H_2O$	0.0249	4.43
Mn_2O_3	0.000981	0.155
$Na_2U_2O_7$	0.000362	0.230
$CaCO_3$	0.0183	1.83
$SrCO_3$	0.00155	0.229
$Na_2SO_4 * 10H_2O$	0.00551	1.77
Total:		97.6

The 84.4 wt. % value for $Al_2O_3 * 3H_2O$ is the best estimate based on the sample average, but conservative lower-end value for gibbsite or bayerite has been requested. If the one-sided 95% confidence interval is subtracted from the average, the insoluble solids were shown to contain >28.0 wt. % aluminum. This corresponds an amount of gibbsite and/or bayerite in the insoluble solids of >79.8 wt%.

Quality Assurance

This work satisfies the requirements of the original task technical and quality assurance plan. Laboratory Notebooks WSRC-NB-2002-00088 and WSRC-NB-2002-00128 contain the experimental data. Personnel in ADS and SCO keep additional notebooks containing this data.

References

[1] L. B. Romanowski, “Low Curie Salt Processing Technical Plan,” HLW-SDT-2002-00031, Rev. 0, March 2002.

[2] J. R. Sessions, “NCSA Sample Analysis for Tank 41,” HLE-TTR-2002-099, Rev. 0, April 30, 2002.

[3] C. J. Martino, “Task Technical and Quality Assurance Plan for the Characterization of the Tank 41H Salt-Well Core Sample,” WSRC-RP-2002-00306, Rev.0, May 29, 2002.

[4] J. R. Sessions, personal communication, June 4, 2002.

[5] R. F. Swingle, “Results of Analyses of Tank 37H Criticality Salt Samples (HTK-493 and 494),” WSRC-TR-2002-00244, Rev. 0, May 29, 2002.

[6] D. T. Hobbs, “Composition of Tank 41H Insoluble Solids,” SRT-LWP-2002-00068, July 18, 2002.

Appendix

Table 5: TK-41-HTF-E-173 wet as-received sample ICP-ES results.

	Aqua Regia Dissolution				Peroxide Fusion Digestion			
Species/Analysis	1	2	3	**Average**	1	2	3	**Average**
ICP-ES (wt. %)								
Ag	< 2.67E-03	< 4.51E-03	< 3.29E-03	**< 3.49E-03**	N/A	N/A	N/A	**N/A**
Al	2.79E+00	2.29E+00	3.02E+00	**2.70E+00**	3.07E+00	2.79E+00	2.59E+00	**2.82E+00**
B	< 9.35E-03	< 1.58E-02	< 1.15E-02	**< 1.22E-02**	< 6.25E-03	< 7.50E-03	< 6.07E-03	**< 6.61E-03**
Ba	< 6.34E-03	< 1.07E-02	< 7.83E-03	**< 8.29E-03**	< 4.24E-03	< 5.09E-03	< 4.12E-03	**< 4.48E-03**
Ca	< 7.68E-03	< 1.30E-02	< 9.47E-03	**< 1.01E-02**	3.80E-02	4.26E-02	3.48E-02	**3.85E-02**
Cd	< 1.40E-03	< 2.37E-03	< 1.73E-03	**< 1.83E-03**	< 9.38E-04	< 1.13E-03	< 9.10E-04	**< 9.93E-04**
Cr	7.42E-03	< 3.39E-03	3.26E-03	**4.69E-03**	2.10E-02	8.94E-03	9.29E-03	**1.31E-02**
Cu	8.05E-03	1.30E-02	8.75E-03	**9.93E-03**	6.38E-03	5.98E-03	4.41E-03	**5.59E-03**
Fe	7.46E-03	< 2.03E-03	4.97E-03	**4.82E-03**	3.95E-02	1.78E-02	9.15E-03	**2.22E-02**
Gd	< 7.81E-03	< 1.32E-02	< 9.64E-03	**< 1.02E-02**	< 5.22E-03	< 6.27E-03	< 5.07E-03	**< 5.52E-03**
K	< 3.13E-01	< 5.29E-01	< 3.86E-01	**< 4.09E-01**	< 2.09E-01	< 2.51E-01	< 2.03E-01	**< 2.21E-01**
La	< 2.40E-03	< 4.06E-03	< 2.97E-03	**< 3.14E-03**	< 1.61E-03	< 1.93E-03	< 1.56E-03	**< 1.70E-03**
Li	< 1.44E-02	< 2.43E-02	< 1.77E-02	**< 1.88E-02**	< 9.60E-03	< 1.15E-02	< 9.32E-03	**< 1.01E-02**
Mg	< 1.77E-03	< 2.99E-03	< 2.18E-03	**< 2.31E-03**	< 1.18E-03	< 1.42E-03	< 1.15E-03	**< 1.25E-03**
Mn	< 2.67E-04	< 4.51E-04	< 3.29E-04	**< 3.49E-04**	< 1.79E-04	< 2.14E-04	< 1.73E-04	**< 1.89E-04**
Mo	< 1.77E-02	< 2.99E-02	< 2.18E-02	**< 2.31E-02**	< 1.18E-02	< 1.42E-02	< 1.15E-02	**< 1.25E-02**
Na	2.50E+01	2.48E+01	2.48E+01	**2.49E+01**	N/A	N/A	N/A	**N/A**
Ni	< 4.51E-03	< 7.62E-03	< 5.56E-03	**< 5.90E-03**	5.04E-03	< 3.62E-03	< 2.92E-03	**3.86E-03**
P	2.86E-02	< 3.89E-02	3.38E-02	**3.38E-02**	4.98E-02	4.04E-02	2.70E-02	**3.91E-02**
Pb	< 1.06E-02	< 1.80E-02	< 1.31E-02	**< 1.39E-02**	< 7.12E-03	< 8.55E-03	< 6.91E-03	**< 7.53E-03**
Sb	< 1.26E-01	< 2.13E-01	< 1.56E-01	**< 1.65E-01**	n.d.	n.d.	n.d.	**n.d.**
Si	< 5.57E-03	< 9.42E-03	< 6.88E-03	**< 7.29E-03**	< 3.73E-03	< 4.47E-03	< 3.62E-03	**< 3.94E-03**
Sn	< 1.50E-02	< 2.54E-02	< 1.85E-02	**< 1.96E-02**	< 1.00E-02	< 1.21E-02	< 9.75E-03	**< 1.06E-02**
Sr	< 2.54E-03	< 4.29E-03	< 3.13E-03	**< 3.32E-03**	6.44E-03	7.27E-03	6.22E-03	**6.64E-03**
Ti	< 2.57E-03	< 4.35E-03	< 3.17E-03	**< 3.36E-03**	< 1.72E-03	< 2.06E-03	< 1.67E-03	**< 1.82E-03**
Zn	< 1.07E-03	< 1.81E-03	< 1.32E-03	**< 1.40E-03**	N/A	N/A	N/A	**N/A**
Zr	< 7.61E-03	< 1.29E-02	< 9.39E-03	**< 9.97E-03**	N/A	N/A	N/A	**N/A**

Table 6: TK-41-HTF-E-173 wet as-received sample actinides results.

	Aqua Regia Dissolution				Peroxide Fusion Digestion			
Species/Analysis	1	2	3	Average	1	2	3	Average
ICP-MS (wt. %)								
M-230 (Th)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-231 (Pa)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-232 (Th,U)	6.20E-05	< 5.36E-05	< 3.91E-05	**5.16E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-233 (U)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-234 (U)	5.30E-05	< 5.36E-05	4.60E-05	**5.09E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-235 (U)	1.39E-04	1.02E-04	9.40E-05	**1.12E-04**	1.20E-04	8.80E-05	1.29E-04	**1.12E-04**
M-236 (U)	4.20E-05	< 5.36E-05	4.40E-05	**4.65E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-237 (Np)	4.60E-05	< 5.36E-05	< 3.91E-05	**4.62E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-238 (U,Pu)	9.63E-04	8.64E-04	7.74E-04	**8.67E-04**	9.19E-04	1.79E-03	7.79E-04	**1.16E-03**
M-239 (Pu)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-240 (Pu)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-241 (Pu,Am)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-242 (Pu)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-243 (Am,Cm)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-244 (Pu,Cm)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-245 (Cm)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-246 (Cm)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
M-247 (Cm,Bk)	< 3.17E-05	< 5.36E-05	< 3.91E-05	**< 4.15E-05**	< 5.36E-05	< 6.43E-05	< 5.20E-05	**< 5.66E-05**
Pu-238/241								
^{239}Pu & ^{240}Pu (dpm/mL)	2.57E+03	5.89E+04	< 2.16E+03	**2.12E+04**	1.08E+04	2.38E+04	2.59E+04	**2.02E+04**
^{238}Pu (wt. %)	1.12E-06	7.71E-07	8.52E-07	**9.14E-07**	1.14E-06	9.18E-07	7.92E-07	**9.52E-07**
^{241}Pu (wt. %)	6.30E-08	1.18E-07	8.62E-08	**8.89E-08**	5.34E-08	4.55E-08	5.90E-08	**5.26E-08**
Actinides Summary (wt. %)								
Total U	1.20E-03	9.66E-04	9.58E-04	**1.04E-03**	1.04E-03	1.88E-03	9.08E-04	**1.28E-03**
Total Pu	1.18E-06	8.89E-07	9.39E-07	**1.00E-06**	1.20E-06	9.64E-07	8.51E-07	**1.00E-06**
Enrichment (mass fraction)								
^{235}U/Tot.U	0.116	0.106	0.098	**0.107**	0.115	0.047	0.142	**0.101**

Table 7: TK-41-HTF-E-173 dry insoluble solids ICP-ES results.

	Aqua Regia Dissolution			Peroxide Fusion Digestion		
Species/Analysis	1	2	**Average**	1	2	**Average**
ICP-ES (wt. %)						
Ag	< 9.20E-03	1.43E-02	**1.18E-02**	N/A	N/A	**N/A**
Al	2.79E+01	3.01E+01	**2.90E+01**	2.93E+01	3.11E+01	**3.02E+01**
B	< 3.22E-02	< 4.91E-02	**< 4.07E-02**	< 4.06E-02	< 3.59E-02	**< 3.83E-02**
Ba	3.87E-02	4.06E-02	**3.97E-02**	4.65E-02	< 2.44E-02	**3.55E-02**
Ca	6.33E-01	6.49E-01	**6.41E-01**	9.83E-01	6.70E-01	**8.27E-01**
Cd	< 4.83E-03	< 7.37E-03	**< 6.10E-03**	< 6.09E-03	< 5.38E-03	**< 5.74E-03**
Ce	< 3.10E-02	< 4.74E-02	**< 3.92E-02**	n.d.	n.d.	**n.d.**
Cr	1.47E+00	1.44E+00	**1.46E+00**	1.08E+00	5.59E-01	**8.20E-01**
Cu	1.58E-01	6.25E-02	**1.10E-01**	5.35E-02	3.63E-02	**4.49E-02**
Fe	5.08E+00	2.91E+00	**4.00E+00**	2.07E+00	1.07E+00	**1.57E+00**
Gd	< 2.69E-02	< 4.11E-02	**< 3.40E-02**	4.50E-02	< 3.00E-02	**3.75E-02**
K	< 1.08E+00	< 1.65E+00	**< 1.37E+00**	< 1.36E+00	< 1.20E+00	**< 1.28E+00**
La	< 8.28E-03	< 1.26E-02	**< 1.04E-02**	1.85E-02	< 9.23E-03	**1.39E-02**
Li	< 4.94E-02	< 7.54E-02	**< 6.24E-02**	< 6.23E-02	< 5.51E-02	**< 5.87E-02**
Mg	1.38E-02	< 9.30E-03	**1.16E-02**	< 7.68E-03	< 6.79E-03	**< 7.24E-03**
Mn	1.61E-01	1.22E-01	**1.42E-01**	9.73E-02	5.10E-02	**7.42E-02**
Mo	< 6.09E-02	< 9.30E-02	**< 7.70E-02**	7.96E-02	< 6.79E-02	**7.38E-02**
Na	7.06E-01	7.34E-01	**7.20E-01**	N/A	N/A	**N/A**
Ni	9.38E-02	9.75E-02	**9.57E-02**	6.88E-02	3.98E-02	**5.43E-02**
P	< 7.93E-02	< 1.21E-01	**< 1.00E-01**	< 1.00E-01	< 8.85E-02	**< 9.43E-02**
Pb	< 3.67E-02	< 5.60E-02	**< 4.64E-02**	< 4.62E-02	< 4.09E-02	**< 4.36E-02**
Si	N/A	N/A	**N/A**	4.67E-01	3.58E-01	**4.13E-01**
Sn	< 5.17E-02	< 7.89E-02	**< 6.53E-02**	1.21E-01	< 5.77E-02	**8.94E-02**
Sr	1.08E-01	1.08E-01	**1.08E-01**	1.94E-01	1.33E-01	**1.64E-01**
Ti	< 8.85E-03	< 1.35E-02	**< 1.12E-02**	1.58E-02	< 9.87E-03	**1.28E-02**
Zn	7.35E-02	1.01E-01	**8.73E-02**	N/A	N/A	**N/A**
Zr	< 2.62E-02	< 4.00E-02	**< 3.31E-02**	N/A	N/A	**N/A**

Table 8: TK-41-HTF-E-173 dry insoluble solids actinides results.

	Aqua Regia Dissolution			Peroxide Fusion Digestion		
Species/Analysis	1	2	Average	1	2	Average
ICP-MS (wt. %)						
M-230 (Th)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-231 (Pa)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-232 (Th,U)	1.97E-03	1.61E-03	**1.79E-03**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-233 (U)	1.52E-03	1.61E-03	**1.57E-03**	1.59E-03	6.58E-04	**1.13E-03**
M-234 (U)	9.14E-03	9.37E-03	**9.25E-03**	7.16E-03	3.53E-03	**5.34E-03**
M-235 (U)	2.69E-02	2.77E-02	**2.73E-02**	2.18E-02	1.03E-02	**1.61E-02**
M-236 (U)	9.68E-03	9.71E-03	**9.70E-03**	8.54E-03	3.87E-03	**6.21E-03**
M-237 (Np)	8.09E-03	7.98E-03	**8.03E-03**	6.57E-03	3.14E-03	**4.86E-03**
M-238 (U,Pu)	1.63E-01	1.70E-01	**1.66E-01**	1.41E-01	6.27E-02	**1.02E-01**
M-239 (Pu)	3.35E-04	< 1.67E-04	**2.51E-04**	1.15E-03	< 3.08E-04	**7.30E-04**
M-240 (Pu)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-241 (Pu,Am)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-242 (Pu)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-243 (Am,Cm)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-244 (Pu,Cm)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-245 (Cm)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-246 (Cm)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
M-247 (Cm,Bk)	< 1.09E-04	< 1.67E-04	**< 1.38E-04**	< 3.48E-04	< 3.08E-04	**< 3.28E-04**
Pu-238/241						
^{239}Pu & ^{240}Pu (dpm/mL)	1.94E+05	1.02E+06	**6.07E+05**	1.42E+06	1.07E+05	**7.64E+05**
^{238}Pu (wt. %)	2.63E-04	2.66E-04	**2.64E-04**	2.07E-04	8.18E-05	**1.45E-04**
^{241}Pu (wt. %)	1.21E-06	1.03E-06	**1.12E-06**	1.96E-06	3.74E-07	**1.17E-06**
Actinides Summary (wt. %)						
Total U	2.10E-01	2.18E-01	**2.14E-01**	1.80E-01	8.11E-02	**1.31E-01**
Total Pu	5.99E-04	2.67E-04	**4.33E-04**	1.36E-03	3.90E-04	**8.76E-04**
Enrichment (mass fraction)						
^{235}U/Tot.U	0.128	0.127	**0.128**	0.121	0.127	**0.124**