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Characterization of the Tank 38H Variable Depth and Core Samples

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Summary

The Tank Farm has transferred a number of samples from Tank 38H to SRTC for analysis in the past year. The primary emphasis on the analyses of these samples was criticality safety. Results of analyses of several core salt samples and dip and variable depth samples are presented herein. Results are given for both the as-received and dried composited salt sample, for insoluble solids separated from the core sample, for wash water produced in dissolving the soluble salts in inhibited water, and for both filtered and unfiltered dip and variable depth samples.

The uranium isotopic enrichment appears fairly consistent among the current analyses (0.87 wt % to 1.7 wt % with and average ~1.5 wt % U-235) and somewhat lower than analyses from 1996 samples (1.7 to 3.8 wt % U-235), although consistent with more recent analyses. There continues to be significant quantities of neutron poisons (sodium, iron and manganese) present to mitigate the potential for nuclear criticality.

Introduction

The High Level Waste Tank Farms store and process high-level liquid wastes from a number of sources including F- and H-Canyon, Receiving Basin for Offsite Fuels (RBOF) and the Defense Waste Processing Facility (DWPF)s and a recycle stream from the Defense Waste Processing Facility (DWPF). The waste is relatively dilute as received and is concentrated in one of several evaporators to minimize the space required to store the waste. The concentrated waste is transferred to one of several concentrate receipt tanks for storage, currently Tank 38H. Recently, the 2H Evaporator was shut down due to crystallization of aluminum silicates in the evaporator and the co-precipitation of enriched

uranium with the aluminum silicate. This raised a concern about the criticality safety of the evaporator. The shutdown of the evaporator has caused problems with storage space in the H Tank Farm. To alleviate the space problem, it has been suggested that material from certain tanks, including Tank 38H, be dissolved, possibly filtered, and then transferred to the Saltstone Facility for disposal.

A variable depth sample and a core sample were pulled during Fall 1999 to check for organics in the tank and perform initial criticality analyses. Both sets of analyses were performed. Concentration, Storage and Transfer Engineering (CSTE) issued a request for more information. Two additional variable depth samples (VDS, HTF-230 and 231) and three core samples (no HTF numbers given) were pulled from Tank 38H during July 2000 to perform routine criticality analyses. A task technical and quality assurance plan was written to cover this work. The core samples were composited and analyzed as received. The sample was lost during washing to separate soluble material from the insoluble material. Two additional core samples (HTF-395 and HTF-396) were pulled in January 2001 and composited. Separation of soluble and insoluble solids was completed on the composite sample and the insoluble solids and wash water were analyzed, and the results are reported below.

The original VDSs arrived without reference to the depth at which they were taken. The customer requested that these VDSs not be analyzed, but that another set of samples with a better pedigree be pulled and analyzed. Surface dip sample HTF-391 was pulled in December 2000 and VDS HTF-400 was pulled in January 2001 from a level of 286 inches above the bottom of Tank 38H. These samples were analyzed with the results presented herein.

In addition to the analyses requested in the task technical request, CSTE requested the contents of Tank 38H be characterized for the feasibility of dissolving the contents and transferring them directly to the Saltstone Facility for disposal as saltstone. CSTE also desired an estimate of the amount of Tank 38H saltcake material dissolved by the supernate transferred to Tank 38H from Tank 22H in December of 1999. The current report contains the characterization results but makes no comparison against saltstone limits. Additionally, no estimate of the amount of Tank 38H material dissolved by Tank 22H supernate is provided. The additional information will be provided at a later date or may be derived from the information provided herein by CSTE personnel.

Discussion

Experimental

Core Samples

Initially, three core samples and two variable depth were pulled from Tank 38H in the July 2000. These samples were transferred to SRTC for analysis. The core samples were pulled from three separate holes in close proximity to each other. These samples were composited for analysis. Aliquots of the as-received sample were digested in triplicate by two different methods (aqua regia dissolution and sodium peroxide fusion digestion) and analyzed for a number of different species. Separate aliquots of the composite sample were dried to a constant weight, and the dried samples were digested by the two methods noted above in triplicate and analyzed for same species as the as-received samples. Table 1 gives the analyses requested.

While compositing the core sample, it was noted that there seemed to be two types of solid material in the sample: small white pebbles and a black sludgy material. This was pointed out to the customer. It was decided at that time to analyze each of these materials separately. Portions of each of these materials were separated as well as possible. Aliquots of each material were digested using either aqua regia or sodium peroxide (one digestion each for each of the two materials). These samples were submitted for the same analyses as the as-received samples and the dried solids.

Portions of the initial composite core sample were then washed to separate the undissolved soluble salts. A portion of the core sample (~10 g) was placed into each of three centrifuge tubes, and 30 mL of inhibited water (0.015 M NaOH) was added to each tube. The centrifuge tubes were shaken for several minutes and allowed to settle at least 12 hours. The tubes were centrifuged for at least two runs of at least 15 minutes each, with additional centrifuge runs performed until consecutive centrifuge runs produced no apparent reduction in volume of undissolved solids. After centrifuging the salt solution was decanted from each tube. This process (addition of inhibited water, mixing, settling, centrifuging

and decanting) was repeated two more times for each of the three tubes. The insoluble solids remaining after the three washes were then dried to a constant weight. Portions of dried insoluble solids from each of the three tubes were digested using both aqua regia dissolution and sodium peroxide fusion digestion, and the resulting digested solids were submitted for the analyses given in Table 1. There was an insufficient amount of the insoluble solids to allow submission of any the material for scanning electron microscopy (SEM). Aliquots of each wash water (three tubes, three washes each) were diluted and submitted for the analyses given in Table 1.

During the first attempt at washing the core solids, the tubes were broken during centrifuging. Since all of the sample had been previously used, the Tank Farm was requested to send more sample material. The Facility pulled two additional samples in January 2001. It was decided that the as-received analyses of the new sample were not necessary, so only the washing was done on the new sample. The centrifuge instructions were adjusted and the washing of the second sample solids proceeded without incident. The as-received (including the white pebble and black sludge material) and dried sample analyses documented in this report were from the first core sample. The washed sample analyses (both wash water and insoluble solids) reported herein were conducted on the second composite core sample.

Dip and Variable Depth Samples

The dip and/or variable depth samples were also analyzed in several different ways. Aliquots of each sample were filtered through 0.45-micron filters, diluted and then submitted for the analyses given in Table 1. (The filters used were the same as those used in the first filtration of the current method of silicon analysis.) This gives information of the dissolved material contained in the samples. Other aliquots of each sample were then digested using both aqua regia and sodium peroxide fusion in triplicate. The digested samples were then submitted for the analyses given in Table 1. The results of the digested sample analyses give when compared with the filtrate results give some indication about any undissolved solids in the supernate.

Initially, variable depth samples were pulled from two different depths. However, there was no information defining which sample was taken from which depth, and it was decided not to analyze these samples. New samples were pulled in December 2000 (dip surface sample) and January 2001 (variable depth sample from a level of 286 inches above the bottom of the tank). The results of the analyses of these samples are documented herein

Table 1. Analyses Conducted on the Tank 38H Samples

Analytical Method	Digested VDS	VDS Filtrate and Core Wash Water Samples	As-Received Core Solids	Washed Insoluble Core Solids
Density	X	X (VDS Filtrate only)		X
Na_2O_2 Digestion	X	-	X	X
Aqua Regia Dissolution	X	-	X	X
Tank 50 Rad Screen	X	X	X	X
Pu 238/241	X	X	X	X
Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)	X	X	X	X
Inductively Coupled Plasma – Emission Spectrometry (ICP-ES)	X	X	X	X
Cold Vapor – Atomic Absorption (Hg)	-	-	-	X

Ion Chromatography (IC) – Anions	-	X	-	-
Wet Chemistry (Free OH^-, AlO_2^-, CO_3^{-2})	-	X	-	-
Gamma Scan	X	X	X	X
Sr-90	X	X	X	X
Scanning Electron Microscopy (SEM)	-	-	-	X

Results

ICP-MS results are given for each atomic mass. In most cases, each mass number can realistically represent only one isotope. In some cases however, two isotopes are a realistic possibility. The most notable of these cases are mass # 238, which can be either uranium or plutonium, and mass # 241, which can be either plutonium or americium. For mass 238, the vast majority is assumed to be uranium, but Pu-238 is measured separately by a chemical separation and counting technique. The same is true for Pu-241. In the ICP-MS results reported in the tables below, the value for mass 238 is for uranium. The Pu-238 value is reported separately below. The value for Pu-241 is determined by a chemical separation and counting technique and is reported below the ICP-MS results in each table. No results are given for Na, Ag and Zr for the peroxide fusion digestions, because the peroxide fusion digestion adds significant Na from the reagents and Zr from the vessels used. The peroxide fusion also introduces significant interference for the determination of Ag. Where no analysis is performed, the result is given as "na". Many species were not detected using one or both of the detection methods. In those cases "<" followed by the limit of detection is given when available. Where no detection limit is available, "bdl" is given for "below detection limit." In some cases, ADS provided results both above and below the detection limit. In those cases both actual values and limits of detection are averaged and result is preceded by "≤" indicating that the that the average of the results is "less than or equal to" the result given. These values are probably somewhat higher than the actual concentration. In a very few cases a single value of limit of detection is greater than one order of magnitude different from the other values. In those cases, the one value different from the other two is considered to be an outlier and is not included in the result. No standard deviation is provided for those values preceded by "<" or "≤". For the ratios of elements to equivalent uranium, any value preceded by "<" is considered to be "0" and is accounted for in averages as such.

As-Received and Dried Core Sample Analyses

The results for as-received core sample are given in Table 2. The dried core sample results are given in Table 3. In general, the averages and standard deviations are given for three determinations. The one exception in these two sets of sample results is the actinide results for the dried sample. One of the samples submitted for ICP-MS analysis was apparently misplaced and was not analyzed. Unless otherwise noted the results for solid samples are given as an average and standard deviation of three determinations each for aqua regia dissolution and sodium peroxide fusion digestion, followed by a combined average and standard deviation for all six determinations.

High Level Waste saltcake is composed primarily of sodium salts (sodium hydroxide, sodium nitrate and sodium nitrate). If the saltcake were evenly divided between these three salts, the sodium concentration in a dried sample would be expected to be about 35 wt %. The higher sodium content found in the dried sample (Table 3) indicates that the sodium content may be somewhat skewed toward sodium hydroxide. Since the saltcake was found to be about 20 wt % water, a lower sodium concentration such as the 29 wt % (Table 2) measured in the as-received sample is not unreasonable.

Table 2. Results of As-Received Core Sample

	Aqua Regia Dissolution		Peroxide Fusion Digestion		Combined Dissolutions	

Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-ES		(wt %)			(wt %)			(wt %)	
Ag	<	2.80E-03	--		na	na	<	2.80E-03	--
Al		2.17E-01	6.19E-02		1.84E-01	1.01E-02		2.01E-01	4.36E-02
B	<	2.34E-03	--	<	2.37E-03	--	<	2.35E-03	--
Ba	<	9.34E-04	--	<	9.47E-04	--	<	9.41E-04	--
Ca		6.14E-02	2.46E-02		6.02E-02	8.81E-03		6.08E-02	1.65E-02
Cd	£	1.42E-03	--	<	1.42E-03	--	£	1.42E-03	--
Co	<	2.34E-03	--	<	9.00E-03	--	<	5.67E-03	--
Cr	£	5.58E-03	--		8.53E-03	3.22E-04	£	7.06E-03	--
Cu	<	9.34E-04	--	<	2.84E-03	--	<	1.89E-03	--
Fe		3.60E-01	2.83E-01		2.73E-01	6.53E-02		3.17E-01	1.90E-01
La	<	5.14E-03	--	<	5.45E-02	--	<	2.98E-02	--
Li	<	9.34E-04	--	<	3.79E-03	--	<	2.36E-03	--
Mg		1.92E-02	1.21E-02		1.28E-02	1.54E-03		1.60E-02	8.49E-03
Mn		3.22E-02	2.37E-02		2.43E-02	2.89E-03		2.82E-02	1.57E-02
Mo	<	2.80E-03	--	<	3.79E-03	--	<	3.30E-03	--
Na		2.98E+01	7.92E-01		na	na		2.98E+01	7.92E-01
Ni	£	4.81E-03	--		1.37E-02	5.19E-04	£	9.27E-03	--
P	£	1.55E-02	--		2.37E-02	8.96E-04	£	1.96E-02	--
Pb	<	1.31E-02	--	<	1.20E-01	--	<	6.65E-02	--
Si		4.18E-02	1.37E-02		1.35E-01	8.58E-03		8.85E-02	5.21E-02
Sn	<	7.01E-03	--	<	2.21E-01	--	<	1.14E-01	--
Sr	<	4.67E-04	--	<	9.47E-04	--	<	7.07E-04	--
Ti	<	9.34E-04	--	<	1.42E-03	--	<	1.18E-03	--
V	<	1.40E-03	--	<	5.21E-03	--	<	3.31E-03	--
Zn	£	2.91E-03	--	<	2.84E-03	--	£	2.88E-03	--
Zr	£	2.05E-03	--		na	na	£	2.05E-03	--

Table 2. Results of As-Received Core Sample (cont'd)

	Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions		
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
Gamma Scan		(dpm/g)			(dpm/g)			(dpm/g)	
Co-60		2.15E+04	1.36E+04		1.38E+04	3.44E+03		1.76E+04	9.86E+03
Sb-125		1.07E+06	4.48E+04		9.69E+05	7.82E+04		1.02E+06	7.99E+04
Cs-134		5.28E+04	7.00E+03		5.01E+04	1.15E+03		5.17E+04	5.20E+03

Cs-137		7.91E+07	1.27E+07		7.37E+07	8.06E+05		7.64E+07	8.58E+06
Eu-154		2.09E+05	1.06E+05		1.33E+05	8.99E+03		1.71E+05	7.94E+04
Eu-155		1.46E+05	--		bdl	bdl		1.46E+05	--
Am-241		4.75E+05	1.79E+05		4.37E+05	7.68E+04		4.56E+05	1.25E+05
Tk 50 Rad Scr		(dpm/g)			(dpm/g)			(dpm/g)	
Alpha		4.55E+07	1.35E+07		3.27E+07	7.73E+06		3.91E+07	1.21E+07
Beta		2.29E+08	9.00E+07		1.92E+08	5.91E+06		2.10E+08	6.05E+07
Pu-238/241		(dpm/g)			(dpm/g)			(dpm/g)	
Pu-239/240		6.16E+05	6.88E+04		5.53E+05	9.99E+03		5.85E+05	5.58E+04
Pu-238		3.52E+07	8.38E+06		2.78E+07	1.71E+06		3.15E+07	6.75E+06
Pu-241		2.18E+07	1.36E+06		1.92E+07	7.76E+05		2.05E+07	1.71E+06
Sr-90		(dpm/g)			(dpm/g)			(dpm/g)	
Sr-90		6.56E+07	3.07E+07		4.33E+07	4.61E+06		5.45E+07	2.31E+07
ICP-MS Act.		(wt %)			(wt %)			(wt %)	
M-230(Th)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-231(Pa)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-232(Th,U)		1.29E-03	8.43E-04		1.08E-03	2.17E-04		1.18E-03	5.63E-04
M-233(U)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-234(U)		8.54E-04	3.13E-04		7.57E-04	1.41E-04		8.05E-04	2.23E-04
M-235(U)		1.57E-03	8.18E-05		1.45E-03	2.46E-05		1.51E-03	8.75E-05
M-236(U)		5.04E-04	1.58E-05		4.44E-04	2.78E-05		4.74E-04	3.87E-05
M-237(Np)		7.75E-04	3.96E-05		6.57E-04	7.02E-05		7.16E-04	8.25E-05
M-238(U,Pu)		1.09E-01	8.04E-03		9.56E-02	1.55E-04		1.02E-01	8.91E-03
M-239(Pu)		3.18E-04	1.06E-04		2.84E-04	2.07E-05		3.01E-04	7.11E-05
M-240(Pu)		9.12E-05	2.14E-05	<	1.28E-06	--	£	4.62E-05	--
M-241(Pu,Am)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-242(Pu)		2.52E-04	3.05E-05		2.26E-04	2.24E-05		2.39E-04	2.79E-05
M-243(Am,Cm)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-244(Pu,Cm)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-245(Cm)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-246(Cm)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--
M-247(Cm,Bk)	<	5.61E-07	--	<	1.28E-06	--	<	9.20E-07	--

Table 2. Results of As-Received Core Sample (cont'd)

	Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions		
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
Pu from Pu-238/241		wt %			wt %			wt %	
Pu-238		9.25E-05	2.21E-05		7.32E-05	4.50E-06		8.28E-05	1.78E-05
Pu-241		9.51E-06	5.94E-07		8.40E-06	3.39E-07		8.96E-06	7.46E-07
Total U		1.12E-01	8.29E-03		9.82E-02	2.24E-04		1.05E-01	9.12E-03
Total Pu		7.63E-04	1.58E-04		5.91E-04	1.96E-05		6.77E-04	1.38E-04
Equivalent U-235		2.88E-03	3.46E-04		2.61E-03	1.07E-04		2.75E-03	2.72E-04
U-235/Tot. U		1.41E-02	1.66E-03		1.47E-02	2.18E-04		1.44E-02	1.11E-03
Na/Eq. U-235		1.04E+04	1.43E+03		na	na		1.04E+04	1.43E+03
Fe/Eq. U-235		1.19E+02	8.10E+01		1.04E+02	2.20E+01		1.12E+02	5.37E+01
Cr/Eq. U-235		1.50E+00	1.38E+00		bdl	bdl		7.52E-01	1.20E+00
Zn/Eq. U-235		7.25E-01	1.26E+00		bdl	bdl		3.63E-01	8.89E-01
Mn/Eq. U-235		1.07E+01	6.68E+00		9.27E+00	7.52E-01		9.97E+00	4.32E+00

In most cases, the results from the both dissolution methods are similar. Additionally, the results of the dried core samples given in Table 3 are generally a little higher than the results given for the as-received sample in Table 2. This reflects the weight of the water lost in drying. A determination of the solids content indicated that the sample was 77.9 $\pm$ 8.2 wt % solids.

Table 3. Results of Analyses of Dried Core Sample

	Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions		
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-ES		(wt %)			wt %			wt %	
Ag	<	5.98E-03	--		na	na	<	5.98E-03	--
Al		2.17E-01	4.06E-03		2.12E-01	2.18E-02		2.14E-01	1.43E-02
B	<	4.98E-03	--	<	4.87E-03	--	<	4.92E-03	--
Ba	<	1.99E-03	--	<	1.95E-03	--	<	1.97E-03	--
Ca		3.21E-02	5.79E-03		9.15E-02	8.61E-03		6.18E-02	3.32E-02
Cd	<	2.99E-03	--	<	2.92E-03	--	<	2.95E-03	--
Co	<	4.98E-03	--	<	1.85E-02	--	<	1.17E-02	--
Cr	<	6.97E-03	--	<	1.75E-02	--	<	1.22E-02	--
Cu	<	1.99E-03	--	<	5.84E-03	--	<	3.92E-03	--
Fe		4.06E-01	1.65E-01		4.55E-01	2.74E-01		4.31E-01	2.04E-01

La	<	1.10E-02	--	<	1.12E-01	--	<	6.14E-02	--
Li	<	1.99E-03	--	<	7.78E-03	--	<	4.89E-03	--
Mg		1.71E-02	3.18E-04		1.51E-02	6.96E-04		1.61E-02	1.19E-03
Mn		3.09E-02	2.28E-03		3.17E-02	6.84E-03		3.13E-02	4.59E-03
Mo	<	--	6.27E-05	<	7.78E-03	--	<	6.88E-03	--
Na		3.91E+01	2.55E-01		na	na		3.91E+01	2.55E-01
Ni	<	6.97E-03	--	<	2.82E-02	--	<	1.76E-02	--
P	<	2.59E-02	--	<	4.87E-02	--	<	3.73E-02	--
Pb	<	2.79E-02	--	<	2.46E-01	--	<	1.37E-01	--
Si		5.18E-02	6.10E-03		1.96E-01	1.03E-02		1.24E-01	7.96E-02
Sn	<	1.49E-02	--	<	4.54E-01	--	<	2.35E-01	--
Sr	<	9.96E-04	--	<	1.95E-03	--	<	1.47E-03	--
Ti	<	1.99E-03	--	<	2.92E-03	--	<	2.46E-03	--
V	<	2.99E-03	--	<	1.07E-02	--	<	6.85E-03	--
Zn	<	2.99E-03	--	<	5.84E-03	--	<	4.41E-03	--
Zr	<	3.98E-03	--		na	na	<	3.98E-03	--
Gamma Scan		(dpm/g)			(dpm/g)			(dpm/g)	
Co-60		1.87E+04	8.13E+02		1.45E+04	--		1.77E+04	2.22E+03
Sb-125		1.32E+06	6.23E+04		1.13E+06	4.79E+04		1.25E+06	1.16E+05
Cs-134		6.03E+04	--		7.07E+04	--		6.55E+04	7.36E+03
Cs-137		8.94E+07	1.42E+06		9.77E+07	5.60E+06		9.27E+07	5.43E+06
Eu-154		1.82E+05	2.28E+04		1.82E+05	2.16E+04		1.82E+05	1.94E+04
Eu-155		bdl	bdl		3.60E+03	--		3.60E+03	--
Am-241		3.49E+05	1.21E+05		4.56E+05	2.34E+05		3.92E+05	1.56E+05

Table 3. Results of Analyses of Dried Core Sample (cont'd)

		Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
Tk 50 Rad Scr		(dpm/g)			(dpm/g)			(dpm/g)	
Alpha		5.79E+07	1.53E+07		4.50E+07	1.53E+07		5.15E+07	1.54E+07
Beta		2.27E+08	2.04E+07		2.21E+08	8.92E+06		2.24E+08	1.45E+07
Pu-238/241		(dpm/g)			(dpm/g)			(dpm/g)	

Pu-239/240		6.26E+05	6.93E+04		4.37E+05	3.15E+05		5.32E+05	2.29E+05
Pu-238		3.71E+07	1.64E+06		3.26E+07	1.03E+06		3.48E+07	2.74E+06
Pu-241		2.52E+07	9.98E+05		2.17E+07	8.36E+05		2.34E+07	2.12E+06
Sr-90		(dpm/g)			(dpm/g)			(dpm/g)	
Sr-90		6.06E+07	3.33E+06		4.75E+07	1.41E+06		5.41E+07	7.51E+06
ICP-MS Act.		(wt %)			(wt %)			(wt %)	
M-230(Th)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-231(Pa)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-232(Th,U)		1.50E-03	2.69E-05		1.88E-03	2.11E-04		1.73E-03	2.53E-04
M-233(U)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-234(U)		1.41E-03	1.36E-04		1.47E-03	1.06E-04		1.44E-03	1.06E-04
M-235(U)		1.86E-03	1.25E-04		1.85E-03	1.45E-04		1.85E-03	1.20E-04
M-236(U)		6.00E-04	8.79E-06		5.15E-04	8.66E-05		5.49E-04	7.70E-05
M-237(Np)		8.78E-04	8.25E-05		8.61E-04	1.76E-04		8.68E-04	1.32E-04
M-238(U,Pu)		1.14E-01	7.90E-03		1.12E-01	1.39E-02		1.13E-01	1.07E-02
M-239(Pu)		3.03E-04	3.39E-06		3.62E-04	6.33E-05		3.38E-04	5.55E-05
M-240(Pu)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-241(Pu,Am)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-242(Pu)		2.49E-04	4.96E-06		2.90E-04	1.79E-05		2.73E-04	2.60E-05
M-243(Am,Cm)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-244(Pu,Cm)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-245(Cm)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-246(Cm)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
M-247(Cm,Bk)	<	1.20E-06	--	<	2.63E-06	--	<	2.06E-06	--
Pu from Pu-238/241		(wt %)			(wt %)			(wt %)	
Pu-238		9.76E-05	4.31E-06		8.58E-05	2.71E-06		9.17E-05	7.21E-06
Pu-240		4.12E-05			8.55E-05			6.34E-05	
Pu-241		1.10E-05	4.36E-07		9.47E-06	3.65E-07		1.03E-05	9.26E-07

Table 3. Results of Analyses of Dried Core Sample (cont'd)

		Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
Total U		1.18E-01	8.15E-03		1.16E-01	1.43E-02		1.17E-01	1.09E-02

Total Pu		6.58E-04	3.29E-06		7.47E-04	8.39E-05		7.11E-04	7.70E-05
Equivalent U-235		3.11E-03	1.41E-04		3.34E-03	3.01E-04		3.25E-03	2.56E-04
U-235/Tot. U		1.58E-02	2.79E-05		1.60E-02	7.09E-04		1.59E-02	5.18E-04
Na/Eq. U-235		1.26E+04	5.64E+02		na	na		1.26E+04	5.64E+02
Fe/Eq. U-235		1.00E+02	6.61E+00		1.42E+02	9.90E+01		1.25E+02	7.38E+01
Cr/Eq. U-235		2.26E+00	1.08E-01		bdl	bdl		9.03E-01	1.24E+00
Zn/Eq. U-235		9.67E-01	4.64E-02		bdl	bdl		3.87E-01	5.30E-01
Mn/Eq. U-235		9.55E+00	1.99E-02		9.69E+00	3.03E+00		9.63E+00	2.14E+00

Two distinct solid phases were apparent in the core sample, a sludgy black form and a white pebble-like form. The customer requested an attempt to isolate and separately characterize the two phases. Difficulties encountered in the separation lead to incomplete separation of the two phases. A single aqua regia and a single peroxide fusion dissolution were made of each phase. The average results from the two dissolutions are given in Table 4. The ratio of the concentration of the species in the black sludge material to its concentration in the white pebbles is also given where appropriate. In Table 4, minimum detectable limits are given as below detection limit or "bdl", since the values in Table 4 are for comparison only.

For most species, there is no significant difference in the concentration of the species in the two phases. However, a few species indicative of sludge material (Al, Fe, and Mn) are somewhat more prevalent in the black sludge material. This indicates that the white pebbles were almost entirely saltcake. The black sludgy material was also almost entirely saltcake but may have contained a small amount of HLW sludge.

Table 4. Results of Analyses of Black Sludge and White Pebble Material

		Black Sludge			White Pebble			Black Sludge/
Species		Average	St. Dev.		Average	St. Dev.		White Pebble
ICP-ES		(wt %)			(wt %)			
Ag		bdl	bdl		bdl	bdl		bdl
Al		2.70E-01	--		3.06E-01	--		8.81E-01
B		bdl	bdl		bdl	bdl		bdl
Ba		bdl	bdl		bdl	bdl		bdl
Ca		4.38E-02	--		1.07E-01	1.32E-01		4.08E-01
Cd		bdl	bdl		bdl	bdl		bdl
Co		bdl	bdl		bdl	bdl		bdl
Cr		bdl	bdl		bdl	bdl		bdl
Cu		bdl	bdl		bdl	bdl		bdl
Fe		4.66E-01	--		2.50E-01	1.14E-01		1.86E+00
La		bdl	bdl		bdl	bdl		bdl
Li		bdl	bdl		bdl	bdl		bdl
Mg		2.09E-02	--		1.87E-02	9.32E-03		1.12E+00

Mn		3.83E-02	--		1.77E-02	5.29E-03		2.17E+00
Mo		bdl	bdl		bdl	bdl		bdl
Na		3.92E+01	--		4.34E+01	--		9.04E-01
Ni		bdl	bdl		bdl	bdl		bdl
P		bdl	bdl		bdl	bdl		bdl
Pb		bdl	bdl		bdl	bdl		bdl
Si		7.49E-02	4.94E-03		2.27E-01	2.67E-01		3.29E-01
Sn		bdl	bdl		bdl	bdl		bdl
Sr		bdl	bdl		bdl	bdl		bdl
Ti		bdl	bdl		9.08E-02	--		bdl
V		bdl	bdl		bdl	bdl		bdl
Zn		bdl	bdl		bdl	bdl		bdl
Zr		bdl	bdl		7.42E-02	--		bdl
Gamma Scan		(dpm/g)			(dpm/g)			
Sb-125		1.63E+06	3.89E+04		1.50E+06	4.42E+05		1.08E+00
Cs-137		9.81E+07	5.48E+04		9.95E+07	6.57E+06		9.86E-01
Eu-154		2.11E+05	4.13E+03		bdl	bdl		bdl
Am-241		6.55E+05	--		bdl	bdl		bdl
AA		(wt %)			(wt %)			
As		bdl	bdl		bdl	bdl		bdl
Se		1.12E-04	1.30E-05		2.02E-04	2.88E-05		5.55E-01

Table 4. Results of Analyses of Black Sludge and White Pebble Material (cont'd)

		Black Sludge			White Pebble			Black Sludge/
Species		Average	St. Dev.		Average	St. Dev.		White Pebble
Pu-238/241		(dpm/g)			(dpm/g)			
Pu-239/240		7.34E+05	2.64E+05		1.16E+06	7.85E+05		6.32E-01
Pu-238		4.59E+07	4.06E+06		4.52E+07	1.63E+07		1.02E+00
Pu-241		3.15E+07	2.14E+06		3.02E+07	1.27E+07		1.04E+00
Sr-90		(dpm/g)			(dpm/g)			
Sr-90		7.29E+07	6.56E+06		6.44E+07	3.97E+07		1.13E+00
ICP-MS Act.		(wt %)			(wt %)			
M-230		bdl	bdl		bdl	bdl		bdl

M-231		bdl	bdl		bdl	bdl		bdl
M-232		2.02E-03	4.89E-04		1.74E-03	9.12E-04		1.16E+00
M-233		bdl	bdl		bdl	bdl		bdl
M-234		1.32E-03	3.84E-05		1.89E-03	3.97E-04		7.01E-01
M-235		2.33E-03	3.02E-04		3.31E-03	6.33E-04		7.03E-01
M-236		7.88E-04	3.04E-05		8.31E-04	2.08E-04		9.49E-01
M-237		1.28E-03	2.27E-05		1.38E-03	2.13E-04		9.28E-01
M-238		1.57E-01	1.00E-02		1.67E-01	4.68E-02		9.40E-01
M-239		4.00E-04	5.49E-05		bdl	bdl		bdl
M-240		1.26E-04	--		bdl	bdl		bdl
M-241		bdl	bdl		bdl	bdl		bdl
M-242		4.97E-04	1.73E-04		bdl	bdl		bdl
M-243		bdl	bdl		bdl	bdl		bdl
M-244		bdl	bdl		bdl	bdl		bdl
M-245		bdl	bdl		bdl	bdl		bdl
M-246		bdl	bdl		bdl	bdl		bdl
M-247		bdl	bdl		bdl	bdl		bdl

Core Sample Washing Activities

As described above, three aliquots of the core sample were each washed three times. The insoluble solids remaining after the third wash were dried to a constant weight. The insoluble solids were found to comprise 0.795 $\pm$ 0.099 wt % of the core sample material. They were also found to comprise 17.7 $\pm$ 3.6 wt % of the wet insoluble solids. After drying, each of the aliquots of the dried insoluble solids was divided into two portions, one for aqua regia dissolution and analysis and one for peroxide fusion digestion and analysis. In the process of preparing the samples, one of the peroxide fusion samples was lost, so the peroxide fusion results reflect the analyses of only two samples. There were insufficient solids remaining to submit a portion for SEM analysis. The results of the insoluble solids analyses are given in Table 5. These results have been corrected for the dissolved solids left behind from the third wash when the solids were dried. In most cases the correction was very small.

Table 5. Results of Analyses of Insoluble Solids

	Aqua Regia Dissolution		Peroxide Fusion Digestion		Combined Dissolutions	
Species	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
ICP-ES	(wt %)		(wt %)		(wt %)	
Ag	4.98E-02	1.49E-02	na	na	4.98E-02	1.49E-02
Al	8.27E+00	2.29E+00	4.23E+00	1.73E-01	6.65E+00	2.75E+00
B	1.34E-02	3.50E-03	4.48E-02	7.34E-03	2.60E-02	1.78E-02
Ba	6.48E-01	1.06E+00	5.60E-02	5.29E-02	4.11E-01	8.15E-01
Ca	1.95E+00	4.92E-01	1.37E+00	3.94E-02	1.71E+00	4.71E-01
Cd	3.47E-02	1.07E-02	1.46E-02	6.83E-04	2.67E-02	1.33E-02
Co	1.10E-02	2.64E-03	7.39E-02	1.38E-02	3.62E-02	3.52E-02
Cr	3.18E-01	1.17E-01	2.98E-01	7.04E-02	3.10E-01	9.08E-02
Cu	5.08E-02	8.51E-03	4.42E-02	6.52E-03	4.81E-02	7.72E-03
Fe	1.13E+01	1.78E+00	4.44E+00	1.89E-01	8.54E+00	3.95E+00
La	2.04E-02	4.42E-03	3.95E-02	6.56E-03	2.81E-02	1.14E-02
Li	4.43E-02	1.39E-02	2.11E-02	3.10E-03	3.50E-02	1.62E-02
Mg	5.66E-01	1.35E-01	2.91E-01	6.05E-03	4.56E-01	1.78E-01
Mn	1.52E+00	5.44E-01	6.94E-01	1.30E-01	1.19E+00	5.96E-01
Mo	7.69E-03	1.85E-03	2.47E-02	2.90E-03	1.45E-02	9.52E-03
Na	1.04E+01	2.69E+00	na	na	1.04E+01	2.69E+00
Ni	2.62E-01	7.66E-02	1.69E-01	1.39E-02	2.25E-01	7.46E-02
P	1.48E-01	4.27E-02	2.22E-01	2.46E-02	1.78E-01	5.21E-02
Pb	7.88E-02	2.38E-02	7.75E-01	1.47E-01	3.57E-01	3.89E-01
Si	4.04E+00	1.12E+00	4.12E+00	3.20E-01	4.07E+00	8.07E-01
Sn	2.87E-02	7.98E-03	1.39E+00	2.86E-01	5.74E-01	7.60E-01
Sr	5.85E-02	7.42E-02	3.69E-02	5.43E-03	4.98E-02	5.38E-02
Ti	3.64E-02	9.30E-03	2.78E-01	4.91E-02	1.33E-01	1.35E-01
V	1.76E-02	5.74E-03	1.03E-01	2.03E-02	5.18E-02	4.81E-02
Zn	4.14E-02	7.65E-03	3.04E-02	3.01E-03	3.70E-02	8.24E-03
Zr	4.37E-01	4.63E-01	na	na	4.37E-01	4.63E-01

Table 5. Results of Analyses of Insoluble Solids (cont'd)

	Aqua Regia Dissolution		Peroxide Fusion Digestion		Combined Dissolutions	
Species	Average	St. Dev.	Average	St. Dev.	Average	St. Dev.
Gamma Scan	(dpm/g)		(dpm/g)		(dpm/g)	
Co-60	bdl	bdl	bdl	bdl	bdl	bdl
Sb-125	1.41E+08	5.12E+07	5.95E+07	7.23E+06	1.08E+08	5.77E+07
Cs-134	bdl	bdl	bdl	bdl	bdl	bdl
Cs-137	1.20E+09	3.23E+08	3.57E+07	3.08E+05	7.36E+08	6.79E+08
Eu-154	4.71E+07	3.18E+06	2.43E+07	1.10E+07	3.80E+07	1.38E+07
Eu-155	1.08E+07	1.82E+05	bdl	bdl		
Am-241	4.67E+07	8.56E+06	1.96E+07	2.21E+06	3.59E+07	1.61E+07
CVAA	(wt %)		(wt %)		(wt %)	
Hg	4.01E-01	1.41E-01	6.15E-04	5.77E-04	2.41E-01	2.41E-01
Tk 50 Rad Scr	(dpm/g)		(dpm/g)		(dpm/g)	

Alpha		3.71E+09	2.10E+09		2.17E+09	3.30E+07		3.10E+09	1.71E+09
Beta		1.51E+10	3.32E+09		6.29E+09	8.86E+08		1.16E+10	5.37E+09
Pu-238/241		(dpm/g)			(dpm/g)			(dpm/g)	
Pu-239/240		6.99E+07	2.55E+07		2.96E+07	9.08E+06		5.38E+07	2.88E+07
Pu-238		5.53E+09	1.72E+09		2.51E+09	4.14E+08		4.32E+09	2.06E+09
Pu-241		2.57E+09	7.76E+08		1.24E+09	2.71E+08		2.04E+09	9.22E+08
Sr-90		(dpm/g)			(dpm/g)			(dpm/g)	
Sr-90		5.33E+09	1.31E+09		2.06E+09	1.31E+08		4.02E+09	2.02E+09

Table 5. Results of Analyses of Insoluble Solids (cont'd)

		Aqua Regia Dissolution			Peroxide Fusion Digestion			Combined Dissolutions	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-MS Act.		(wt %)			(wt %)			(wt %)	
M-230(Th)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
M-231(Pa)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
M-232(Th,U)		4.21E-02	1.41E-02		2.27E-02	2.48E-03		3.43E-02	1.47E-02
M-233(U)		2.64E-03	8.56E-04		1.38E-03	1.12E-04		2.14E-03	9.18E-04
M-234(U)		1.63E-01	4.98E-02		8.38E-02	8.50E-03		1.31E-01	5.60E-02
M-235(U)		1.93E-01	5.84E-02		1.02E-01	8.65E-03		1.57E-01	6.52E-02
M-236(U)		4.86E-02	1.42E-02		2.59E-02	1.94E-03		3.95E-02	1.60E-02
M-237(Np)		6.65E-02	1.90E-02		3.60E-02	2.68E-03		5.43E-02	2.14E-02
M-238(U,Pu)		1.27E+01	3.97E+00		6.97E+00	8.53E-01		1.04E+01	4.22E+00
M-239(Pu)		2.55E-02	6.82E-03		1.42E-02	4.65E-04		2.10E-02	7.85E-03
M-240(Pu)		8.84E-03	2.35E-03		4.86E-03	2.87E-04		7.25E-03	2.74E-03
M-241(Pu,Am)		1.75E-03	6.54E-04		1.03E-03	2.51E-05		1.46E-03	6.07E-04
M-242(Pu)		2.20E-02	6.23E-03		1.19E-02	7.87E-04		1.80E-02	7.09E-03

M-243(Am,Cm)	£	4.87E-05	--	<	1.26E-06	--	£	2.97E-05	--
M-244(Pu,Cm)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
M-245(Cm)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
M-246(Cm)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
M-247(Cm,Bk)	<	1.76E-06	--	<	1.26E-06	--	<	1.56E-06	--
Pu from Pu-238/241		(wt %)			(wt %)			(wt %)	
Pu-238		1.45E-02	4.52E-03		6.60E-03	1.09E-03		1.06E-02	5.05E-03
Pu-241		1.12E-03	3.39E-04		5.42E-04	1.18E-04		8.32E-04	3.77E-04
Total U		1.31E+01	4.09E+00		7.19E+00	8.72E-01		1.07E+01	4.35E+00
Total Pu		7.20E-02	1.91E-02		3.81E-02	2.75E-03		5.85E-02	2.30E-02
Equivalent U-235		3.03E-01	8.76E-02		1.62E-01	1.11E-02		2.46E-01	9.90E-02
U-235/Tot. U		1.48E-02	1.84E-04		1.42E-02	5.17E-04		1.46E-02	4.61E-04
Na/Eq. U-235		3.47E+01	1.51E+00		na	na		3.47E+01	1.51E+00
Fe/Eq. U-235		3.85E+01	7.02E+00		2.74E+01	7.07E-01		3.40E+01	7.84E+00
Cr/Eq. U-235		1.03E+00	1.74E-01		1.83E+00	3.09E-01		1.35E+00	4.79E-01
Zn/Eq. U-235		1.41E-01	2.29E-02		1.88E-01	5.73E-03		1.60E-01	3.05E-02
Mn/Eq. U-235		4.94E+00	3.71E-01		4.27E+00	5.12E-01		4.67E+00	5.20E-01

The wash water from all three washes of all three sample aliquots were collected and submitted for analysis. Two additional analyses were requested for the wash water samples: ion chromatography for various anions and wet chemistry for free OH^-, AlO_2^- and CO_3^{-2}. All of the first washes were averaged together, as were the second washes and the third washes. The results are given in Table 6. As expected the concentrations in the washes for most species decreases going from the 1st wash to the 2nd wash to the 3rd wash. One notable exception to this is the uranium concentration. The uranium is probably present as sodium diuranate, which is slightly soluble. More uranium dissolves with each subsequent wash, so that the concentration stays about the same from wash to wash. The variations noted may be the result of changes in solubility with the changing pH going from wash to wash.

Table 6. Results of Analyses of Core Sample Wash Water Samples

		First Wash			Second Wash			Third Wash	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-ES		(mg/L)			(mg/L)			(mg/L)	
Ag	<	6.10E-01	--	<	6.09E-01	--	<	6.23E-01	--
Al		2.75E+02	1.09E+01		3.26E+01	8.73E+00		3.70E+00	8.10E-01
B		6.37E+00	2.78E-01	<	1.93E+00	--	<	1.97E+00	--
Ba	<	3.05E-01	--	<	3.05E-01	--	<	3.12E-01	--
Ca	<	2.03E-01	--	<	2.03E-01	--	<	2.08E-01	--
Cd	<	2.03E-01	--	<	2.03E-01	--	<	2.08E-01	--
Co	<	7.11E-01	--	<	7.11E-01	--	<	7.27E-01	--
Cr		6.43E+00	2.68E-01		1.52E+00	2.36E-01	£	6.61E-01	--
Cu	<	1.02E-01	--	<	1.02E-01	--	<	1.04E-01	--
Fe		4.99E+00	2.85E+00		4.22E+00	1.76E+00		3.68E+00	1.12E+00
La	<	1.32E+00	--	<	1.32E+00	--	<	1.35E+00	--
Li		5.08E-01	2.10E-03	<	2.03E-01	--	<	2.08E-01	--
Mg		4.41E-01	2.58E-01	£	3.04E-01	--		2.41E-01	5.35E-02
Mn	<	1.02E-01	--	£	2.35E-01	--	<	1.04E-01	--
Mo		9.48E-01	1.55E-01	<	3.05E-01	--	<	3.12E-01	--
Na		7.63E+04	5.86E+02		1.53E+04	3.05E+03		1.19E+03	4.96E+02
Ni	<	7.11E-01	--	<	7.11E-01	--	<	7.27E-01	--
P		3.10E+01	2.13E+00	£	4.59E+00	--	<	3.84E+00	--
Pb	<	3.05E+00	--		3.05E+00	7.18E-02	<	3.12E+00	--
Si		3.19E+00	1.34E+00	£	1.89E+00	--	<	1.77E+00	--
Sn	£	1.86E+00	--	<	1.73E+00	--	<	1.77E+00	--
Sr	<	1.02E-01	--	<	1.02E-01	--	<	1.04E-01	--
Ti	<	1.02E-01	--	<	1.02E-01	--	<	1.04E-01	--
V	<	3.05E-01	--	<	3.05E-01	--	<	3.12E-01	--
Zn		1.12E+00	8.00E-01	<	3.05E-01	--	<	3.12E-01	--
Zr	<	2.03E-01	--	<	2.03E-01	--	<	2.08E-01	--

Table 6. Results of Analyses of Core Sample Wash Water Samples (cont'd)

		First Wash			Second Wash			Third Wash	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
Gamma		(dpm/mL)			(dpm/mL)			(dpm/mL)	

Scan									
Co-60	<	1.73E+05	--	<	7.54E+03	--	<	4.33E+03	--
Sb-125	<	9.61E+05	--	<	5.67E+04	--	<	2.77E+04	--
Cs-134	<	1.36E+05	--	<	6.76E+03	--	<	6.25E+03	--
Cs-137		1.77E+07	2.37E+06		1.40E+06	3.90E+05		1.76E+05	1.48E+05
Eu-154	<	3.01E+05	--	<	1.54E+04	--	<	7.45E+03	--
Am-241	<	1.33E+06	--	<	7.57E+04	--	<	3.10E+04	--
IC-Anions		(mg/L)			(mg/L)			(mg/L)	
Cl-	<	1.99E+02	--	<	2.00E+02	--	<	1.96E+02	--
F-	<	1.99E+02	--	<	2.00E+02	--	<	1.96E+02	--
NO2-		2.31E+03	1.70E+02		2.43E+02	6.46E+01	<	9.82E+02	--
NO3-		5.90E+03	9.55E+02		5.13E+02	1.58E+02	<	9.82E+02	--
SO4--		2.27E+04	1.85E+03		3.36E+03	1.29E+03	<	4.91E+02	--
PO4---	<	9.95E+02	--	<	1.00E+03	--	<	9.82E+02	--
CHO2-	<	9.95E+02	--	<	1.00E+03	--	<	9.82E+02	--
C2O2--		2.56E+03	2.81E+01		5.66E+02	5.92E+01	<	9.82E+02	--
Wet Chemistry		(M)			(M)			(M)	
Free OH-		3.38E-01	1.51E-02		3.20E-02	1.20E-03	£	2.45E-02	--
CO3--		1.20E+00	8.43E-03		2.65E-01	4.77E-02	<	1.97E-02	--
AlO2-	<	1.99E-01	--	<	2.00E-02	--	<	1.96E-02	--
Tk 50 Rad Scr		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Alpha		3.80E+05	4.01E+04		1.40E+05	1.37E+05		4.63E+04	4.89E+04
Beta		2.06E+07	1.02E+06		2.20E+06	8.79E+05		3.01E+05	1.70E+05
Pu-238/241		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Pu-239/240		6.26E+03	1.82E+03	£	2.12E+03	--	<	1.36E+03	--
Pu-238		3.72E+05	3.52E+04		1.32E+05	1.22E+05		3.61E+04	3.16E+04
Pu-241		2.25E+05	1.38E+04	£	7.88E+04	--	<	2.64E+04	--
Sr-90		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Sr-90		8.40E+05	1.60E+05		2.49E+05	2.33E+05	<	6.39E+04	--

Table 6. Results of Analyses of Core Sample Wash Water Samples (cont'd)

		First Wash			Second Wash			Third Wash	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-MS Act.		(mg/L)			(mg/L)			(mg/L)	
M-230(Th)	<	2.64E-03	--	<	5.10E-03	--	<	2.70E-03	--
M-231(Pa)	<	2.64E-03	--	<	9.43E-03	--	<	2.70E-03	--
M-232(Th,U)		1.69E-02	6.84E-03	£	2.91E-02	--	£	4.84E-03	--
M-233(U)	<	2.64E-03	--	£	1.31E-02	--	<	2.70E-03	--
M-234(U)		6.33E-02	1.19E-02	£	2.61E-01	--		1.40E-01	6.36E-02
M-235(U)		1.07E-01	2.06E-02	£	3.27E-01	--		1.50E-01	6.93E-02
M-236(U)		3.52E-02	5.54E-03	£	9.88E-02	--		4.16E-02	1.73E-02
M-237(Np)		1.30E-01	1.49E-02	£	1.31E-01	--		9.53E-03	4.46E-03
M-238(U,Pu)		6.29E+00	1.18E+00		2.08E+01	1.92E+00		1.02E+01	4.69E+00
M-239(Pu)		3.52E-02	7.03E-03	£	1.71E-02	--	£	4.36E-03	--
M-240(Pu)		1.29E-02	2.51E-03	<	1.08E-02	--	<	2.70E-03	--
M-241(Pu,Am)	<	2.64E-03	--		7.76E-03	8.84E-03	<	2.70E-03	--
M-242(Pu)		2.84E-02	4.39E-03	£	2.09E-02	--	£	3.84E-03	--
M-243(Am,Cm)	<	2.64E-03	--	<	8.43E-03	--	<	2.70E-03	--
M-244(Pu,Cm)	<	2.64E-03	--	<	8.43E-03	--	<	2.70E-03	--
M-245(Cm)	<	2.64E-03	--	<	1.08E-02	--	<	2.70E-03	--
M-246(Cm)	<	2.64E-03	--	<	1.04E-02	--	<	2.70E-03	--
M-247(Cm,Bk)	<	2.64E-03	--	<	1.11E-02	--	<	2.70E-03	--
Pu from Pu-238/241		(mg/L)			(mg/L)			(mg/L)	
Pu-238		9.79E-03	9.25E-04		3.47E-03	3.22E-03		9.49E-04	8.31E-04
Pu-240								1.53E-03	
Pu-241		9.85E-04	6.04E-05		3.45E-04	2.40E-04		bdl	
Total U		6.50E+00	1.22E+00		2.12E+01	1.99E+00		1.06E+01	4.84E+00
Total Pu		7.65E-02	1.33E-02		1.22E-02	4.03E-03		3.95E-03	5.59E-03
Equivalent U-235		2.48E-01	4.77E-02		3.63E-01	3.84E-02		1.60E-01	7.73E-02
U-235/Tot. U		1.65E-02	9.31E-05		1.54E-02	1.08E-03		1.41E-02	1.20E-04
Na/Eq. U-235		3.15E+05	5.61E+04		3.80E+04	2.77E+03		7.92E+03	1.90E+03
Fe/Eq. U-235		2.16E+01	1.55E+01		9.04E+00	2.58E+00		2.74E+01	1.48E+01

Cr/Eq. U-235		2.65E+01	4.00E+00		3.93E+00	2.81E-01		1.27E+00	2.20E+00
Zn/Eq. U-235		4.94E+00	4.20E+00		bdl	bdl		bdl	bdl
Mn/Eq. U-235		bdl	bdl		bdl	bdl		bdl	bdl

HTF-391 Dip and HTF-400 Variable Depth Samples

Two liquid samples were also received from Tank 38H, a surface dip sample, HTF-391, and a subsurface variable depth sample, HTF-400, taken from an elevation of ~286 inches above the tank bottom. Each of these samples received three sets of analyses. An aliquot of each was filtered and diluted. The diluted filtrate from each sample was submitted in triplicate to Analytical Development Section for analysis. As with the core sample wash water samples the filtrate samples were submitted for the additional IC-anions and wet chemistry analyses.

A second triplicate set of aliquots from each sample was mixed with aqua regia to dissolve any residual solids. A third triplicate set of aliquots from each sample was dried to remove the water, digested using a peroxide fusion digestion and submitted to ADS for analysis. (Water reacts with the sodium peroxide used in the peroxide fusion digestions.) Neither the aqua regia dissolutions nor the peroxide fusion digestions were submitted for IC-anions and wet chemistry analyses. In many cases the aqua regia dissolutions exhibited higher concentrations than the filtrate, indicating the possible presence of some undissolved solids in the liquid samples. Nearly all of the peroxide fusion results are lower than the aqua regia results and in most cases the filtrate results. This is believed to have resulted from loss of part of the sample during drying.

Table 7 contains the results for the HTF-391 Dip sample and Table 8 contains the results for the HTF-400 Variable Depth sample.

Table 7. Results of Analyses of HTF-391 Dip Sample

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-ES		(mg/L)			(mg/L)			(mg/L)	
Ag	<	3.25E-01	--	<	6.39E-01	--		na	na
Al		1.00E+03	3.17E+00		1.21E+03	1.36E+01		6.97E+02	2.12E+02
B		3.51E+01	2.48E-01		4.44E+01	2.28E+00		2.52E+01	6.23E+00
Ba	<	1.63E-01	--	<	3.20E-01	--	<	5.38E-01	--
Ca		1.47E+00	7.57E-01	<	3.20E-01	--	£	3.97E+01	--
Cd	£	1.54E-01	--	<	9.59E-01	--	<	8.07E-01	--
Co	<	3.80E-01	--	<	1.60E+00	--	<	1.61E+00	--
Cr		1.89E+01	2.46E-01		2.27E+01	7.18E-01		1.51E+01	5.91E+00
Cu		1.95E-01	1.22E-01	£	3.83E-01	--	<	1.61E+00	--
Fe		7.61E-01	1.71E-01		1.53E+01	7.87E+00		3.00E+01	2.88E+01
La	<	7.05E-01	--	<	3.20E+00	--	<	3.09E+01	--
Li		3.93E+00	1.21E-01		4.69E+00	2.23E-01	£	2.30E+00	--
Mg	<	5.42E-02	--	<	3.20E-01	--	£	6.31E-01	--
Mn	£	6.43E-02	--		1.16E+00	6.36E-01	£	8.65E-01	--
Mo		4.22E+00	1.55E-02		4.52E+00	1.15E-01	<	2.15E+00	--

Na		4.32E+04	1.81E+00		5.29E+04	5.25E+02		na	na
Ni	£	5.90E-01	--	<	3.20E+00	--	<	7.80E+00	--
P		5.91E+01	4.50E+00		6.35E+01	2.77E+00		3.98E+01	1.45E+01
Pb	£	2.08E+00	--	<	1.63E+01	--	<	6.81E+01	--
Si		4.51E+01	3.84E+00		8.36E+01	1.71E+01		4.06E+01	1.35E+01
Sn		2.73E+00	1.02E+00	<	6.39E+00	--	<	1.26E+02	--
Sr	<	5.42E-02	--		8.40E-01	4.33E-01	<	5.38E-01	--
Ti	£	1.44E-01	--	<	3.20E-01	--	£	1.20E+00	--
V	£	2.28E-01	--	<	6.39E-01	--	<	2.96E+00	--
Zn		3.39E+00	1.31E+00		4.64E+00	2.36E-01	£	2.96E+00	--
Zr	£	1.18E-01	--	<	6.39E-01	--		na	na
Gamma Scan		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Co-60		bdl	bdl		bdl	bdl		bdl	bdl
Sb-125		bdl	bdl		bdl	bdl		bdl	bdl
Cs-134		bdl	bdl		bdl	bdl		bdl	bdl
Cs-137		1.39E+08	4.20E+06		2.28E+08	5.42E+07		1.43E+08	3.96E+07
Eu-154		bdl	bdl		bdl	bdl		bdl	bdl
Am-241		bdl	bdl		bdl	bdl		bdl	bdl

Table 7. Results of Analyses of HTF-391 Dip Sample (cont'd)

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
IC-Anions		(mg/L)			(mg/L)			(mg/L)	
Cl-		1.99E+02	2.58E+00		na	na		na	na
F-		1.99E+02	2.58E+00		na	na		na	na
NO2-		1.71E+04	6.84E+02		na	na		na	na
NO3-		1.44E+04	5.72E+02		na	na		na	na
SO4--		4.02E+02	1.22E+01		na	na		na	na
PO4---		9.94E+02	1.29E+01		na	na		na	na
C2O4--	<	9.94E+02	--		na	na		na	na
Wet Chemistry		(M)			(M)			(M)	
Free OH-		9.39E-01	5.25E-02		na	na		na	na
CO3--		na	na		na	na		na	na

AlO2-	<	1.99E-01	--		na	na		na	na
Tk 50 Rad Scr		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Alpha	<	2.01E+05	--		1.22E+06	7.38E+04		1.22E+06	6.89E+05
Beta		1.66E+08	7.46E+05		1.38E+08	1.03E+08		1.09E+08	3.59E+07
Pu-238/241		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Pu-239/240		1.01E+03	2.39E+02		2.87E+03	2.30E+03		1.26E+03	1.28E+03
Pu-238		7.59E+04	7.73E+02		2.54E+05	8.01E+04		5.65E+04	4.72E+04
Pu-241		3.05E+04	1.38E+03		9.00E+04	3.88E+04		4.44E+04	1.27E+03
Sr-90		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Sr-90		2.22E+05	1.05E+04		4.45E+05	1.24E+05		2.17E+05	1.20E+05

Table 7. Results of Analyses of HTF-391 Dip Sample (cont'd)

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-MS Act.		(mg/L)			(mg/L)			(mg/L)	
M-230(Th)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-231(Pa)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-232(Th,U)		7.45E-03	6.06E-04	<	8.63E-04	--		8.58E-02	4.72E-02
M-233(U)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-234(U)		1.82E-02	4.53E-05		8.58E-04	1.11E-05	<	3.61E-04	--
M-235(U)		1.54E-01	5.30E-03		2.51E-01	3.97E-02		9.56E-02	5.09E-02
M-236(U)		2.66E-02	6.96E-04	<	8.63E-04	--	<	3.63E-04	--
M-237(Np)		9.84E-03	6.90E-04	<	8.63E-04	--	<	3.61E-04	--
M-238(U,Pu)		1.76E+01	2.87E-01		2.06E+01	2.75E+00		6.89E+00	4.01E+00
M-239(Pu)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-240(Pu)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-241(Pu,Am)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-242(Pu)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-243(Am,Cm)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-244(Pu,Cm)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-245(Cm)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--

M-246(Cm)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
M-247(Cm,Bk)	<	1.39E-04	--	<	8.63E-04	--	<	3.61E-04	--
Pu from Pu-238/241		(mg/L)			(mg/L)			(mg/L)	
Pu-238		2.00E-03	2.03E-05		6.68E-03	2.11E-03		1.49E-03	1.24E-03
Pu-241		1.33E-04	6.03E-06		3.94E-04	1.70E-04		1.94E-04	5.54E-06
Total U		1.78E+01	2.93E-01		2.09E+01	2.84E+00		7.00E+00	4.08E+00
Total Pu		2.13E-07	2.64E-09		7.08E-07	2.28E-07		1.68E-07	1.24E-07
Equivalent U-235		1.54E-01	5.30E-03		2.51E-01	3.97E-02		9.56E-02	5.09E-02
U-235/Tot. U		8.65E-03	1.55E-04		1.20E-02	5.65E-04		1.40E-02	2.28E-03
Na/Eq. U-235		2.81E+05	9.67E+03		2.15E+05	3.39E+04		na	na
Fe/Eq. U-235		4.93E+00	9.44E-01		5.97E+01	2.34E+01		2.74E+02	1.24E+02
Cr/Eq. U-235		1.23E+02	5.83E+00		9.22E+01	1.67E+01		1.67E+02	3.37E+01
Zn/Eq. U-235		2.19E+01	7.75E+00		1.88E+01	3.09E+00		1.22E+01	2.11E+01
Mn/Eq. U-235		9.54E-02	1.35E-01		4.48E+00	1.84E+00		3.28E+00	5.67E+00

Table 8. Results of Analyses of HTF-400 Variable Depth Sample

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-ES		(mg/L)			(mg/L)			(mg/L)	
Ag	<	5.80E-01	--	<	6.47E-01	--		na	na
Al		1.64E+03	6.77E+00		2.14E+03	7.21E+01		6.95E+02	1.53E+02
B		6.39E+01	1.77E-01		8.22E+01	2.68E+00		2.57E+01	7.95E+00
Ba	<	1.16E+00	--	<	3.24E-01	--	<	4.90E-01	--
Ca	<	1.93E-01	--	<	3.24E-01	--	£	3.08E+01	--
Cd	<	1.93E-01	--	<	9.71E-01	--	<	7.36E-01	--
Co	<	6.77E-01	--	<	1.62E+00	--	<	1.47E+00	--
Cr		3.53E+01	2.34E-01		4.54E+01	2.18E+00		1.51E+01	4.84E+00
Cu		1.52E-01	2.02E-02		1.14E+00	2.15E-01	<	1.47E+00	--
Fe		1.24E+00	1.04E-01		8.96E+00	4.49E-01		1.80E+01	8.99E+00
La	<	1.26E+00	--	<	3.24E+00	--	<	2.82E+01	--
Li		5.77E+00	4.72E-02		7.13E+00	3.00E-01	£	2.21E+00	--
Mg	<	9.67E-02	--	£	3.46E-01	--	£	1.39E+00	--

Mn	<	9.67E-02	--	<	3.22E-01	--	<	4.90E-01	--
Mo		6.59E+00	1.05E-01	£	8.13E+00	--	<	1.96E+00	--
Na		7.40E+04	2.62E+02		9.81E+04	2.90E+03		na	na
Ni	<	6.77E-01	--	<	3.24E+00	--	<	7.11E+00	--
P		1.11E+02	2.24E+00		1.34E+02	2.25E+00		4.29E+01	1.19E+01
Pb	<	2.90E+00	--	<	1.65E+01	--	<	6.20E+01	--
Si		5.43E+01	3.35E+00		6.98E+01	6.90E+00		2.93E+01	9.34E+00
Sn		3.67E+00	2.50E-01	<	6.47E+00	--	<	1.14E+02	--
Sr	<	9.67E-02	--	£	3.35E-01	--	<	4.90E-01	--
Ti	<	9.67E-02	--	<	3.24E-01	--	<	7.36E-01	--
V	<	2.90E-01	--	<	6.47E-01	--	<	2.70E+00	--
Zn		3.77E+00	4.90E-01		6.16E+00	1.71E-01		2.34E+00	6.14E-01
Zr	<	1.93E-01	--	<	6.47E-01	--		na	na
Gamma Scan		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Co-60		bdl	bdl		bdl	bdl		bdl	bdl
Sb-125		bdl	bdl		bdl	bdl		bdl	bdl
Cs-134		bdl	bdl		bdl	bdl		bdl	bdl
Cs-137		1.81E+08	5.27E+06		2.74E+08	3.51E+07		1.22E+08	6.91E+07
Eu-154		bdl	bdl		bdl	bdl		bdl	bdl
Am-241		bdl	bdl		bdl	bdl		bdl	bdl

Table 8. Results of Analyses of HTF-400 Variable Depth Sample (cont'd)

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
IC-Anions		(mg/L)			(mg/L)			(mg/L)	
Cl-	<	1.93E+02	--		na	na		na	na
F-	<	1.93E+02	--		na	na		na	na
NO2-		2.97E+04	6.58E+03		na	na		na	na
NO3-		2.59E+04	7.41E+03		na	na		na	na
SO4--		1.31E+03	7.99E+01		na	na		na	na
PO4---	<	9.67E+02	--		na	na		na	na
C2O4--	<	9.67E+02	--		na	na		na	na
Wet Chemistry		(M)			(M)			(M)	

Free OH-		1.94E+00	2.00E-01		na	na		na	na
CO3--	£	2.24E-01	--		na	na		na	na
AlO2-	<	1.93E-01	--		na	na		na	na
Tk 50 Rad Scr		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Alpha	<	1.99E+05	--		1.67E+06	1.17E+05		1.70E+06	1.16E+06
Beta		2.11E+08	2.32E+06		2.69E+08	8.75E+06		9.42E+07	4.10E+07
Pu-238/241		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Pu-239/240		2.69E+03	5.28E+02		3.05E+03	1.62E+03		5.83E+02	2.06E+02
Pu-238		1.51E+05	3.83E+04		2.40E+05	4.93E+04		4.82E+04	2.22E+04
Pu-241		6.51E+04	1.37E+04		9.12E+04	1.69E+04		6.90E+04	4.78E+04
Sr-90		(dpm/mL)			(dpm/mL)			(dpm/mL)	
Sr-90		1.07E+05	1.07E+03		3.33E+05	5.50E+04		1.46E+05	5.48E+04

Table 8. Results of Analyses of HTF-400 Variable Depth Sample (cont'd)

		Filtrate			Aqua Regia Dissolution			Peroxide Fusion Dissolution	
Species		Average	St. Dev.		Average	St. Dev.		Average	St. Dev.
ICP-MS Act.		(mg/L)			(mg/L)			(mg/L)	
M-230(Th)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-231(Pa)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-232(Th,U)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-233(U)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-234(U)		2.89E-02	3.24E-04	<	8.74E-04	--	<	3.29E-04	--
M-235(U)		1.96E-01	3.60E-03		3.10E-01	4.92E-02		8.64E-02	5.61E-02
M-236(U)		4.37E-02	3.34E-04	<	8.74E-04	--	<	3.22E-04	--
M-237(Np)		2.69E-02	1.43E-03	<	8.74E-04	--	<	3.29E-04	--
M-238(U,Pu)		1.52E+01	1.94E-01		1.76E+01	1.60E+00		5.93E+00	3.83E+00
M-239(Pu)		1.83E-02	8.45E-04	<	8.74E-04	--	<	3.29E-04	--
M-240(Pu)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-241(Pu,Am)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-242(Pu)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-243(Am,Cm)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--

M-244(Pu,Cm)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-245(Cm)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-246(Cm)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
M-247(Cm,Bk)	<	1.35E-04	--	<	8.74E-04	--	<	3.29E-04	--
Pu from Pu-238/241		(mg/L)			(mg/L)			(mg/L)	
Pu-238		3.98E-03	1.01E-03		6.30E-03	1.30E-03		1.27E-03	5.83E-04
Pu-240		3.90E-04							
Pu-241		2.85E-04	6.01E-05		3.99E-04	7.39E-05		3.02E-04	2.09E-04
Total U		1.55E+01	1.98E-01		1.80E+01	1.64E+00		6.03E+00	3.92E+00
Total Pu		1.83E-02	8.45E-04		6.70E-07	1.36E-07		1.57E-07	4.23E-08
Equivalent U-235		2.45E-01	4.21E-02		3.10E-01	4.92E-02		8.64E-02	5.61E-02
U-235/Tot U.		1.27E-02	7.19E-05		1.72E-02	1.43E-03		1.43E-02	1.17E-03
Na/Eq. U-235		3.10E+05	6.03E+04		3.21E+05	3.84E+04		na	na
Fe/Eq. U-235		5.14E+00	7.38E-01		2.94E+01	5.08E+00		2.23E+02	4.85E+01
Cr/Eq. U-235		1.48E+02	2.90E+01		1.48E+02	1.53E+01		2.22E+02	1.51E+02
Zn/Eq. U-235		1.55E+01	1.43E+00		2.01E+01	2.45E+00		3.15E+01	1.00E+01
Mn/Eq. U-235		bdl	bdl		bdl	bdl		bdl	bdl

Comparison with Previous Results

The results reported in this report were compared with previously reported data." In the current set of Tank 38H samples the U-235 enrichment in general ranges from 1.4 to 1.65 % for all samples: total solids in salt cake, insoluble solids, dissolved salt and supernate. There are a few outliers from as low as 0.87 to as high as 1.7 in the dip sample analyses. Previous results reported by Swingle from samples taken in 1999 showed results of total core sample solids ranging from 1.04 to 1.4 % enrichment, variable depth sample solids at 1.1 % enrichment and variable depth sample supernate at 3.6 % enrichment. Previous results reported by Hobbs in 1996 showed insoluble solids from variable depth samples in the range of 1.4 to 1.7 % enrichment. Similar variable depth samples were analyzed as they were received and gave 2.8 to 3.8 % enrichment, while an analysis of total undissolved solids from samples taken at the same time gave 1.7 to 2.7 % enrichment. From these samples it appears that U-235 enrichments in the current samples are pretty much in-line with solids analyzed in most of the recent previous samples. The supernate from the previous samples may have been somewhat higher. The supernate in Tank 38 might be expected to vary somewhat as material from different sources is transferred in and out, so this is probably not unexpected.

In the 1999 sample, Swingle reported a total uranium concentration of 0.15 – 0.17 wt % in the core sample compared with the current value of ~0.11 wt % on an as-received basis. Hobbs reported ~0.0004 wt % total uranium on a dry basis in 1996 compared with the current 0.12 wt % on a dry basis. Though as a drop tank for the evaporator the concentrations in Tank 38H can vary significantly as new material is processed, it is not clear why the two results differ so greatly.

Quality Assurance

All analyses were conducted by ADS according to their routine operating and quality assurance procedures. All sample preparation work was conducted by Shielded Cells Operations personnel, according to written instructions provided by Waste Processing Technology personnel. All results were recorded in laboratory notebooks WSRC-NB-2000-00197, WSRC-ND-2001-00079, and WSRC-NB-2001-00089, along with several notebooks kept by ADS personnel.

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