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## **Analysis of Tank 43H Samples at the Conclusion of Uranyl Carbonate Addition.**

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## SUMMARY

Three variable depth samples from Tank 43H (TK43-HTF-E-106 [24" below liquid surface], TK43-HTF-E-113 [Middle depth sample, 200"] and TK43-HTF-E-110 [24 " above sludge], were characterized for actinides, cations and other chemical species of interest. Only the last variable depth sample, TK43-HTF-E-110 [24 " above sludge], contained any visible solids.

The U-235 enrichments, as measured by the weight percent of U-235 to U-238, in all three variable depth samples are less than 0.5 wt %; with the 24 " above sludge sample showing the highest average enrichment of  $0.483 \pm 0.037$  wt % (liquid fraction) and  $0.464 \pm 0.042$  wt % (dry and digested solids). The U-235 enrichments in the other variable depth sample averaged  $0.363 \pm 0.008$  wt % for the 24" below liquid surface sample and  $0.361 \pm 0.007$  wt % for the middle depth sample. The U-235 enrichments are well within the acceptance criteria of 0.7 wt % established in the Nuclear Criticality Safety Evaluation (NCSE) for restart of the 2H Evaporator.<sup>1</sup>

Results from the three samples described in this report agreed well with the six samples analyzed at Central Laboratories. The average U-235 enrichment for the three samples analyzed at SRTC measured  $0.402 \pm 0.070$  wt %, compared to an average of  $0.390 \pm 0.007$  wt % for the six samples analyzed at Central Laboratories. This agreement was significant since sample preparation and analysis techniques were different at the two laboratories. The data from both of these sets of samples showed that the reduction in enrichment required for restart of the 2H-Evaporator had been achieved by a wide margin, which was an essential milestone for the successful restart of the evaporator.

We determined the neutron poison (Na, Fe, Cr, Zn and Mn) to equivalent-U-235 weight ratios for each of the supernatant liquids from the "as-received" samples and solids isolated from the sample taken 24" above the sludge layer. The weight ratios indicate that the liquids and solids are well poisoned by two or more neutron poisons.

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## 1.0 INTRODUCTION

Tank 43H serves as the feed Tank to the 2H evaporator. In the months of July and August 2001, about 21,000 gallons of a depleted uranyl carbonate solution were added to Tank 43H and agitated with two Flygt mixers. The depleted uranium addition served to decrease the U-235 enrichment in the Tank 43H supernate so that the supernate could be evaporated with no risk of accumulating enriched uranium.

After all the uranyl carbonate had been added, the Flygt mixers were turned off, and the solids in the tank were allowed to settle. Nine samples were taken and analyzed to demonstrate that the U-235 enrichment in the supernate liquid is below the acceptance criteria of 0.7 wt %. The samples were taken from the H and G risers at three different depths (329.5 inches or 24 inches below liquid surface, 200 inches (middle of Tank), and 86.5 inches above the tank bottom (24 inches above sludge level in the Tank). Six of these samples were sent to Central Laboratories and three to SRTC for analysis.

The three Tank 43H sample containers delivered to SRTC included TK43-HTF-E-106, TK43-HTF-E-113 and TK43-HTF-E-110. These samples were acquired, respectively, from 24" below the top of liquid phase, middle of supernatant liquid and 24" from top of the sludge.

Analytical characterization of these samples complies with the work scope defined in "Task Technical and Quality Assurance Plan for FY2001 Analysis of samples from Tank 43H at the Conclusion of Uranyl Carbonate Addition," WSRC-RP-2001-00576, Rev.1, May 7, 2001, authored by L. N. Oji and D. T. Hobbs.

## 2.0 EXPERIMENTAL

The analyses conducted on these tank samples are summarized in Table 1 below. Of the three variable depth samples received (TK43-HTF-E-106 [24" below liquid surface], TK43-HTF-E-113 [Middle depth sample, 200"] and TK43-HTF-E-110 [24 " above sludge], only the last sample contained any visible solids. The last sample, TK43-HTF-E-110 [24 " above sludge], contained about 50% solids by volume.

All three variable depth samples were centrifuged for several minutes and the liquid fraction from each sample was analyzed. No solids were observed in the bottom of the centrifuge tubes for the 24" below liquid surface and middle depth samples.

The solid fraction from the third variable depth sample (24 " above sludge sample) was dried at 110 °C until constant weight (overnight) and digested in two ways (Aqua Regia and Peroxide Fusion dissolution in triplicate) and analyzed as previously described for the supernate fractions. The solid fraction was not washed to remove interstitial liquids

and soluble solids prior to the drying treatment. We also determined the percent dry solids in the sludge fraction.

The principal analytical techniques employed by ADS in this characterization include the following techniques: Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)<sup>2</sup> for radiochemical analysis for the actinides and plutonium activity by alpha counting for Pu-238/241(PuTTA), elemental analysis by Inductively Coupled Plasma-Emission Spectroscopy (ICP-ES)<sup>3</sup>, wet chemistry (free hydroxide, aluminate and carbonate) and ion chromatography (IC) for anions<sup>4 5</sup>.

The ICP-MS results are given for each atomic mass, and in most cases, with the exception of a few, each mass number represents only one isotope. Overlapping atomic masses include M-238 (U and Pu) and M-241 (Pu and Am). Hence, we used alpha separation and counting techniques to measure Pu-238, Pu-241 and Am-241. The total plutonium results reported in each Table are based on Pu-238 and Pu-241 values from PuTTA and ICP-MS actinide results for Pu-239, Pu-240 and Pu-242. Equivalent U-235 (Wt %) was determined using the following summation:  $U-235 + 4(Pu-239 + Pu-241 + U-233)$ <sup>6</sup>.

For elemental analysis, no results are reported for sodium (Na), zirconium (Zr) and silver (Ag) from peroxide fusion digestions because this technique adds considerable amounts of sodium as a reagent and the digestion crucible is made of zirconium. This technique also introduces significant interference for the determination of silver. Hence, in cases where these elements are not reported for peroxide fusion, the combined average values for these elements are represented by those from aqua regia digestion. In the Tables, values preceded by ‘ < ’ (less than sign) indicates values were below quantification limits and in these cases no standard deviation values are provided. In the ICP-MS summary for actinides a “bdl” symbol is used to indicate below quantification limits. In cases where ADS provided results which were below and above detection limits (samples analyzed in triplicate), the actual values and limits of detection are averaged and preceded by “≤” indicating that the average of the results is “less than or equal to” the given result.

The uncertainties reported for the poison- to equivalent U-235 ratios are based on propagation of errors for individual measurements.

Likewise, no standard deviations are provided for ratios of elements to equivalent uranium or combined apparent averages based on aqua regia dissolution and peroxide fusion digestion because these two methods are entirely different methods of preparing the samples prior to analysis.

For both the “as-received” sample and the digested insoluble solids, concentration units have been reported here as follows:

Elemental analysis (ICP-ES): weight percent (wt %)

Actinides (ICP-MS): weight percent (wt %)

Plutonium (PuTTA): weight percent (wt %) and disintegration per minute per gram solid (dpm/g).

**Table 1. Analyses Conducted on Samples from Tank 43H.**

Analysis/Analytical Method	As-Received Sample* (All depths)	1 <sup>st</sup> Liquid fraction from 24" above sludge**	Dried Solid Fraction from 24" above sludge**
Density	X	-	-
Na <sub>2</sub> O <sub>2</sub> Digestion	-	-	X
Aqua Regia Dissolution	-	-	X
U and Pu-isotopes (ICP-MS)	X	X	X
Pu-243 and 241	X	X	X
Aluminates, Carbonates and Free OH	X	X	-
NO <sub>3</sub> , NO <sub>2</sub> (I.C)	X	X	-
Al, Si, Cr, Na, ,Fe, Mn and Zn (ICP-ES)	X	X	X

\* Characterized for all "as-received" variable depth liquids samples including liquid fraction from the 24" above sludge sample.

\*\* From 24" above sludge sample, the only variable depth sample with solid and liquid fractions.

### 3.0 RESULTS

The densities of the three as-received variable depth samples were determined at shielded cell temperature of 26 °C prior to centrifugation of each sample. The values obtained were, respectively,  $1.236 \pm 0.002$ ,  $1.242 \pm 0.002$  and  $1.330 \pm 0.010$  g/cc for the 24" below liquid surface sample, middle sample and 24" above sludge surface sample. The third variable depth sample containing solids was agitated to ensure a uniform matrix prior to sampling for density measurements.

#### 3.10 As-Received Tank 43H Liquid Fraction Samples

Without further processing, aliquots of Tank 43H as-received liquid fractions resulting from centrifugation were diluted with distilled water and analyzed for actinides, alpha and beta activities, plutonium isotopics, and selected anions and elements. These analytical results are summarized in Tables 2, 3 and 4. The analytical results indicate that the supernatant liquids are comprised chiefly of sodium, aluminum, nitrate, hydroxide and carbonate with smaller quantities of boron, chromium, phosphorus and silicon.

Uranium and plutonium concentrations varied among the three samples (see Table 3). The U-235 enrichment for the supernatant samples taken at the two higher elevations were not statistically different. The U-235 enrichment in the supernatant liquid isolated from the sample taken closest to the sludge layer measured 0.483 wt %, which is about 33% higher than that in the other samples, but is well below the target enrichment of 0.7 wt %. Table 3 also provides the calculated the neutron poison to equivalent U-235 weight ratios for Na, Fe, Cr, Zn and Mn. The weight ratios for all of the poison except Mn are above 50:1. The weight ratios for Na are particularly high ( $\geq 7.29\text{E}+05:1$ ) and reflect the high concentrations of sodium salts and the limited solubility of uranium in alkaline solutions.

**Table 2. ICP-ES Analytical Results for the Tank 43H Variable Depth Samples.**

ICP-ES	24" Below Liquid Surface [TK43-HTF-E-106]			Middle (200") [TK43-HTF-E-113]			24" Above Sludge (Liquid fraction) [TK43-HTF-E-110]		
Element		Average (mg/L)	Std. Dev.		Average (mg/L)	Std. Dev.		Average (mg/L)	Std. Dev.
Ag	<	5.00E+00		<	5.00E+00		<	5.00E+00	
Al		3.77E+03	1.17E+02		3.72E+03	1.90E+02		4.26E+03	1.40E+02
B		1.63E+02	3.20E+00		1.69E+02	7.96E+00		1.82E+02	6.03E+00
Ba	<	3.00E+00		<	3.00E+00		<	3.00E+00	
Ca	<	1.00E+00		<	1.00E+00		<	1.02E+01	
Cd	<	2.00E+00		<	2.00E+00		<	2.00E+00	
Co		3.57E+00	8.14E-01	<	3.00E+00		<	3.00E+00	
Cr		9.82E+01	2.55E+00		9.69E+01	4.05E+00		1.65E+02	4.20E+00
Cu		4.93E+00	1.53E-01		4.27E+00	3.21E-01	<	3.30E+00	
Fe		1.36E+01	1.19E+00		2.20E+01	5.94E+00		2.25E+01	5.84E+00
La	<	1.00E+01		<	1.00E+01		<	1.00E+01	
Li		1.31E+01	4.58E-01		1.30E+01	5.69E-01		9.90E+00	5.20E-01
Mg	<	1.00E+00		<	1.00E+00			2.73E+00	2.58E+00
Mn	<	1.00E+00		<	1.00E+00		<	1.30E+00	
Mo		1.14E+01	1.35E+00		1.23E+01	5.13E-01		1.44E+01	9.45E-01
Na		1.42E+05	4.86E+03		1.44E+05	8.42E+03		1.49E+05	4.44E+03
Ni	<	9.00E+00		<	9.00E+00		<	9.00E+00	
P		2.50E+02	5.89E+00		2.61E+02	1.36E+01		3.11E+02	6.26E+00
Pb	<	4.60E+01		<	4.60E+01		<	4.60E+01	
Si		1.13E+02	1.73E+01		1.23E+02	3.22E+00		1.08E+02	5.27E+00
Sn	<	1.60E+01		<	1.74E+01		<	1.60E+01	
Sr	<	1.00E+00		<	1.00E+00		<	1.00E+00	
Ti		2.13E+00	3.21E-01		1.50E+00	3.00E-01	<	1.00E+00	
U		8.90E+01	8.09E+00	<	8.57E+01		<	7.70E+01	
V	<	3.17E+00		<	3.17E+00		<	3.00E+00	
Zn		9.97E+00	1.19E+00		1.04E+01	7.77E-01		1.29E+01	2.31E+00
Zr	<	3.03E+00		<	3.10E+00		<	3.00E+00	



**Table 3 Radiochemical ICP-MS Analytical Results and Poison-to Equivalent U-235 Ratios for the Tank 43H Variable Depth Samples.**

	24" Below Liquid Surface [TK43-HTF-E-106]			Middle (200") [TK43-HTF-E-113]			24" Above Sludge (Liquid fraction) [TK43-HTF-E-110]	
	Average		Std. Dev.	Average		Dev.dev.	Average	Std. Dev.
Units	dpm/ml			dpm/ml			dpm/ml	
<b>Pu-238/241</b>								
Pu-238		9.63E+03	3.22E+03		1.60E+04	1.77E+03	6.83E+04	3.51E+02
Pu-241	<	4.98E+03			1.51E+04	9.16E+03	3.13E+04	5.90E+03
		mg/L			mg/L		mg/L	
<b>Pu-238/241</b>								
Pu-238		2.54E-04	8.48E-05		4.20E-04	4.66E-05	1.80E-03	9.25E-06
Pu-241		2.16E-05	2.13E-06		6.56E-05	3.98E-05	1.36E-04	2.56E-05
		mg/L			mg/L		mg/L	
M-232 (Th)		1.45E-02	7.07E-04		3.30E-02	1.10E-02	2.03E-02	5.51E-03
M-233 (Pa, U)		bdl			bdl		bdl	
M-234 (Th,U)		bdl			bdl		bdl	
M-235 (U)		1.91E-01	1.12E-02		1.95E-01	1.18E-02	1.01E-01	1.24E-02
M-236 (U)		1.93E-02	1.15E-03		1.83E-02	5.77E-04	1.83E-02	1.53E-03
M-237 (Np)		bdl			bdl		bdl	
M-238 (U, Pu)		5.25E+01	2.38E+00		5.41E+01	2.77E+00	2.08E+01	1.39E+00
		mg/L			mg/L		mg/L	
U-Total		5.28E+01	2.39E+00		5.43E+01	2.79E+00	2.09E+01	1.41E+00
U-235/U-238		3.63E-03	8.00E-05		3.61E-03	5.51E-05	4.83E-03	3.70E-04
U-235/U-Total		3.61E-03	7.51E-05		3.59E-03	5.51E-05	4.80E-03	3.65E-04
Total Pu		2.75E-04	8.48E-05		4.86E-04	6.13E-05	1.94E-03	2.722E-05
Equiv. U-235		1.92E-01	1.120E-02		1.97E-01	1.180E-02	1.08E-01	1.240E-02
Na/Eq.U-235		7.41E+05	5.015E+04		7.29E+05	6.111E+04	1.38E+06	1.628E+05
Fe/Eq.U-235		7.11E+01	7.480E+00		1.12E+02	3.087E+01	2.07E+02	5.885E+01
Cr/Eq.U-235		5.12E+02	3.273E+01		4.92E+02	3.594E+01	1.52E+03	1.786E+02
Zn/Eq.U-235		5.20E+01	6.922E+00		5.30E+01	5.063E+00	1.19E+02	2.524E+01
Mn/Eq.U-235	<	5.21E+00		<	5.08E+00		< 1.20E+01	

**Table 4. Ion Chromatography and Wet Chemistry Analytical Results for the Tank 43H Variable Depth Samples.**

		24" Below Liquid [TK43-HTF-E-106]			Middle [TK43-HTF-E-113]			24" above sludge (Liquid fraction) [TK43-HTF-E-110]	
		Average	Std. Dev.		Average	Std. Dev.		Average	Std. Dev.
Units		mg/L			mg/L			mg/L	
<b>IC-Anions</b>									
Cl <sup>-</sup>	<	2.00E+03			5.53E+03	7.77E+02		5.03E+03	2.89E+02
F <sup>-</sup>	<	2.00E+03		<	2.00E+03		<	2.00E+03	
NO <sub>2</sub> <sup>-</sup>	<	1.00E+04			4.53E+03	9.50E+02		4.10E+03	5.57E+02
NO <sub>3</sub> <sup>-</sup>		1.09E+06	3.25E+04		8.67E+06	8.34E+05		8.80E+06	8.97E+05
SO <sub>4</sub> <sup>2-</sup>		2.67E+03	2.08E+02		3.73E+03	1.53E+02		3.73E+03	1.15E+02
PO <sub>4</sub> <sup>3-</sup>	<	1.00E+04		<	1.00E+04		<	1.00E+04	
CHO <sub>2</sub> <sup>-</sup>	<	1.00E+04		<	1.00E+04		<	1.00E+04	
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	<	1.00E+04		<	1.00E+04		<	1.00E+04	
<b>Wet Chemistry</b>		M			M			M	
AlO <sub>2</sub> <sup>-</sup>	<	6.600E-02		<	6.600E-02		<	6.600E-02	
CO <sub>3</sub> <sup>2-</sup>		1.481E-01	5.978E-02		1.392E-01	4.179E-02		1.317E-01	1.479E-02
Free-OH <sup>-</sup>		3.023E+00	2.791E-01		3.118E+00	2.302E-02		3.263E+00	3.222E-02

**3.20 As-Received Dried Tank 43H Solid fraction**

Three portions of the solid fraction isolated from the third variable depth sample (24" above sludge [TK43-HTF-E-110]) were dried to constant weight at 110 °C. The average weight percent dry solids was  $37.22 \pm 0.85$  %. Samples from the dry solids were digested in triplicate (Aqua regia dissolution and peroxide fusion digestion) and analyzed. Analytical results are summarized in Tables 5 and 6 below.

In general there was good agreement between the two dissolution methods. Slightly higher analytical values were reported for the aqua regia dissolution method compared to those from the peroxide fusion method. For the uranium and plutonium isotopic measurements, there was no statistical difference in the analytical results for the two dissolution methods. The results indicate that the solids are comprised chiefly of Na, Fe, Ca and Al, with smaller amounts of Cr, Mg, Mn, Ni, P and Si. Table 6 also provides the calculated the neutron poison to equivalent U-235 weight ratios for Na, Fe, Cr, Zn and Mn. The weight ratios for Na and Fe are high ( $\geq 274:1$ ). The weight ratios for Cr, Zn and Mn are much lower ( $\leq 7.15:1$ ).

**Table 5. ICP-ES Analytical Results for the Dried Solid Fraction Contained in the Tank 43H Sample [TK43-HTF-E-110].**

	<b>Aqua Regia Dissolution***</b>		<b>Peroxide Fusion Digestion***</b>		<b>Combined Dissolution</b>	
<b>Species</b>	<b>Average</b>	<b>Std. Dev.</b>	<b>Average</b>	<b>Std. Dev.</b>		
<b>ICP-ES</b>	<b>Wt %</b>		<b>Wt %</b>		<b>Wt. %</b>	
Ag	2.087E-02	1.172E-03	NR		2.087E-02	
Al	2.143E+00	3.983E-02	1.434E+00	1.971E-01	1.789E+00	
B	4.887E-02	5.686E-04	1.963E-02	3.761E-03	3.425E-02	
Ba	1.283E-02	5.033E-04	2.967E-03	1.514E-03	7.900E-03	
Be	< 1.033E-03		< 1.000E-04		< 5.667E-04	
Ca	1.496E+00	6.075E-02	1.160E+00	2.208E-01	1.328E+00	
Cd	1.397E-02	1.193E-03	< 3.567E-03	7.638E-04	< 8.767E-03	
Co	1.303E-02	5.859E-04	< 1.917E-02		< 1.610E-02	
Cr	2.130E-01	8.343E-03	1.520E-01		1.825E-01	
Cu	3.833E-02	1.756E-03	1.400E-02	4.933E-03	2.617E-02	
Fe	9.119E+00	3.689E-01	7.046E+00	9.341E-01	8.082E+00	
La	1.263E-02	2.743E-03	< 1.160E-01		< 6.433E-02	
Li	4.430E-02	1.868E-03	2.673E-02	5.179E-03	3.552E-02	
Mg	2.123E-01	9.001E-03	1.567E-01	2.899E-02	1.845E-01	
Mn	8.926E-01	3.918E-02	7.093E-01	9.989E-02	8.009E-01	
Mo	8.900E-03	1.311E-03	< 8.067E-03		< 8.483E-03	
Na	2.699E+01	6.421E-01	NR	NR	2.699E+01	
Ni	2.219E-01	6.461E-03	1.447E-01	2.435E-02	1.833E-01	
P	1.557E-01	1.709E-02	8.923E-02	6.336E-03	1.225E-01	
Pb	< 6.900E-02		< 2.553E-01		< 1.622E-01	
Sb	< 6.767E-02		< 6.660E-02		< 6.713E-02	
Si	6.735E-01	7.790E-02	3.997E-01	6.709E-02	5.366E-01	
Sn	< 2.463E-02		< 4.712E-01		< 2.479E-01	
Sr	6.867E-03	2.082E-04	1.993E-02	2.950E-02	1.340E-02	
Ti	< 1.677E-02		< 4.500E-03		< 1.063E-02	
U	6.352E+00	1.738E-01	NR		6.352E+00	
V	< 1.620E-02		< 1.110E-02		< 1.365E-02	
Zn	5.227E-02	2.967E-03	3.963E-02	7.295E-03	4.595E-02	
Zr	2.110E-02	5.434E-03	NR	NR	2.110E-02	

NR= Not reported.

\*\*\* Average and standard deviation of triplicate samples.

**Table 6. Radiochemical ICP-MS Analytical Results and Poison-to Equivalent U-235 Ratios for the Dried Solid Fractions of the Tank 43H Sample [TK43-HTF-E-110].**

	Aqua Regia Dissolution		Peroxide Fusion Digestion		Combined Digestion Average	
	Wt%	Std. Dev.	Wt%	Std. Dev.		
<b>Pu-238/241</b>						
Pu-238	1.29E-03	6.42E-05	1.08E-03	1.24E-04	1.18E-03	
Pu-241	5.09E-05	2.38E-06	4.61E-05	4.99E-06	4.85E-05	
<b>Pu-238/241</b>	**** dpm/g		dpm/g		dpm/g	
Pu-238	4.91E+08	2.44E+07	4.07E+08	4.53E+07	4.49E+08	
Pu-241	1.17E+08	5.47E+06	1.06E+08	1.10E+07	1.12E+08	
<b>ICP-MS Act.</b>						
M-232 (Th)	6.16E-03	8.32E-04	5.24E-03	1.01E-03	5.70E-03	
M-233 (Pa,U)	bdl		bdl			
M-234 (Th,U)	1.18E-03	1.46E-04	9.87E-04	1.79E-04	1.09E-03	
M-235 (U)	2.44E-02	2.65E-03	2.12E-02	2.98E-03	2.28E-02	
M-236 (U)	3.40E-03	4.57E-04	2.95E-03	4.55E-04	3.18E-03	
M-237 (Np)	3.21E-03	4.11E-04	2.77E-03	3.95E-04	2.99E-03	
M-238 (U, Pu)	5.40E+00	8.43E-01	4.52E+00	8.57E-01	4.96E+00	
M-239 (Pu)	1.69E-03	1.99E-04	1.54E-03	2.54E-04	1.62E-03	
M-240 (Pu)	4.12E-04	4.08E-05	3.95E-04	6.08E-05	4.04E-04	
M-241 (Pu,Am)	2.46E-04	5.10E-05	2.37E-04	3.38E-05	2.41E-04	
M-242 (PU)	7.48E-04	9.85E-05	4.85E-04	1.97E-04	6.16E-04	
M-243 (U, Pu)						
Total U	5.43E+00	8.47E-01	4.55E+00	8.57E-01	4.99E+00	
Total Pu	4.57E-03	5.51E-04	3.23E-03	1.30E-04	3.90E-03	
U-235/U-238	4.55E-03	3.41E-04	4.73E-03	2.50E-04	4.64E-03	
U-235/U-Total	4.52E-03	3.39E-04	4.69E-03	2.42E-04	4.60E-03	
Equivalent U-235	2.98E-02	2.851E-03	2.57E-02	3.477E-03	2.77E-02	
Na/Eq.U-235	9.059E+02	8.934E+01	1.050E+03	1.442E+02	9.779E+02	
Fe/Eq.U-235	3.061E+02	3.180E+01	2.741E+02	5.191E+01	2.901E+02	
Cr/Eq.U-235	7.150E+00	7.394E-01	5.912E+00	1.242E+00	6.531E+00	
Zn/Eq.U-235	1.754E+00	1.952E-01	1.542E+00	3.522E-01	1.648E+00	
Mn/Eq.U-235	1.754E+00	4.820E-01	1.542E+00	3.522E-01	1.648E+00	

Standard deviations are given only for species with measurable quantities.

\*\*\*\* Based on weight of dried and digested (aqua regia and peroxide fusion) sludge samples for Pu-238 and Pu-241 only.

## 4.0 CONCLUSIONS

The average U-235 enrichments (U-235/U-238) in all three variable depth samples are less than 0.5 wt %, with the 24" above sludge sample showing the highest average enrichment of  $0.483 \pm 0.037$  wt % (liquid fraction) and  $0.464 \pm 0.042$  wt % (dry and digested solids). The U-235 enrichments in the other variable depth samples averaged  $0.363 \pm 0.008$  wt % for the 24" above liquid surface sample and  $0.361 \pm 0.007$  wt % for the middle depth sample.

These results indicate that the U-235 enrichment in the supernatant liquid in Tank 43H was successfully diluted below the target of 0.7 wt % by addition of depleted uranium in the form of a uranyl carbonate solution.

Results from the three samples described in this report agreed well with the six samples that were analyzed at Central Laboratories<sup>7</sup>. U-235 enrichment for the three samples measured at SRTC averaged  $0.402 \pm 0.070$  wt %, compared to  $0.390 \pm 0.0007$  wt % for the six samples analyzed at Central Laboratories. This agreement was significant since sample preparation and analysis techniques were different at the two laboratories. The data from both of these sets of samples showed that the reduction in enrichment required for restart of the 2H Evaporator had been achieved by a wide margin, which was an essential milestone for the successful restart of the evaporator.

We determined the neutron poison (Na, Fe, Cr, Zn and Mn) to equivalent-U-235 weight ratios for each of the supernatant liquids from the "as-received" samples and solids isolated from the sample taken 24" above the sludge layer. The weight ratios indicate that the liquids and solids are well poisoned by two or more neutron poisons.


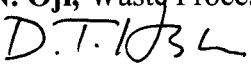
## 5.0 QUALITY ASSURANCE

The SRTC Analytical Development Section (ADS) performed all the analytical requirements for this characterization of Tank 37H according to their operating quality assurance procedures. SRTC Shielded Cell operations personnel conducted all the sample preparation protocols per written instructions provided by Waste Processing Technology personnel. Data obtained from this study reside as records in WSRC-NB-2001-00179 and in WSRC-NB-2001-00142. ADS also maintain analytical results.

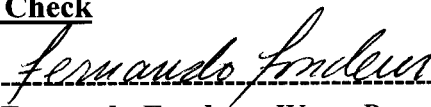

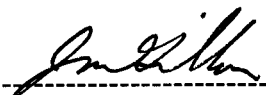


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