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ANALYSIS OF TANK 48H SAMPLE HTF-E-05-021

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LIST OF ACRONYMS

1PB	phenylboronic acid
2PB	diphenylborinic acid
3PB	triphenylborane
4PB	tetraphenylborate anion – $[B(C_6H_5)_4]^-$
AA	Atomic Absorption
ADS	Analytical Development Section
CsTPB	Cesium tetraphenylborate ($Cs[B(C_6H_5)_4]$)
FW	formula weight
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma – Emission Spectrometry
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
ITP	In-Tank Precipitation
KTPB	Potassium tetraphenylborate – $K[B(C_6H_5)_4]$
LOD	Limit of Detection
MST	monosodium titanate
MW	molecular weight
NA	not applicable
NM	not measured
NaTPB	sodium tetraphenylborate – $Na[B(C_6H_5)_4]$
SRNL	Savannah River National Laboratory
TPB	tetraphenylborate - $[B(C_6H_5)_4]^-$
WPTS	Waste Processing Technology Section
95% confidence level	95 out 100 chances the mean value will lie in this interval
Std Dev	The length of the measuring interval or precision

1.0 EXECUTIVE SUMMARY

Personnel analyzed Tank 48H sample taken on March 6, 2005, for chemical and radiological constituents. This report documents the analytical results and analysis of this data. A comparison of the results with the data from other reports (CBU-PIT-2005-00046 and CBU-PIT-2005-00066) revealed the data is consistent.

- The measured potassium tetrphenylborate (KTPB) solid concentration is 2.01 ± 0.21 wt %.
- The calculated monosodium titanate (MST) concentration is 0.15 ± 0.01 wt % MST.
- The measured insoluble solids content was 3.05 ± 0.16 wt %. Both KTPB and MST contributed to 70% of the insoluble solids concentration. This insoluble solids content result is higher than previous Tank 48H analyses (2.18 ± 0.6).
- The free hydroxide concentration in the Tank 48H filtrate sample (1.34 ± 0.01 M) is greater than the Tank 48H limit (1.0 M). This is an increase of 0.55 M since the September 2003 sample due to the addition of 6,424 gallons of 50 wt % sodium hydroxide to Tank 48H.¹
- The soluble potassium content in the filtrate continues to follow a linear trend, which began in 1995 showing a slow radiolytic decomposition of the tetraphenylborate solids.
- The measured ^{137}Cs concentration is $7.53\text{E}+08 \pm 1.07\text{E}+07$ dpm/mL (1.28 Ci/gallon or 313,866 curies of ^{137}Cs) in the slurry which is lower than the expected 1.72 Ci/gal (or 409,000 curies of ^{137}Cs). The filtrate ^{137}Cs concentration is $3.0\text{E}+07 \pm 6.0\text{E}+04$ dpm/mL. This result is consistent with previous results. The filtrate ^{137}Cs does not follow a linear trend with time. Significant analytical data is summarized in Table 1.

Table 1 – Summary of Significant Tank 48H Sample Results

Analyte	Slurry	Supernate	Source
Volume, gal	242,190	N/A	Tank 48H
Density, g/mL	1.165	1.14	Density
Total Solids, wt %	20.19	17.68	Solids
Total Insolubles, wt %	3.05	NM	Calculation
KTPB, wt %	2.01	<0.001	HPLC
MST solids, wt %	0.15	(<0.15 mg/L)	ICP-ES
<u>Metals</u>			
Sodium, M	NM	3.82	ICP-ES
Potassium, M	0.068	0.006	AA
<u>Anions</u>			
Free Hydroxide, M	NM	1.34	Titration
Carbonate, M	NM	0.385*	Titration
Nitrite, M	NM	0.516	Anion
Nitrate, M	NM	0.23	Anion
<u>RadChem</u>			
^{137}Cs , dpm/mL	7.53E+08	3.0E+07	RadChem

NM=Not Measured

LOD = Limit of Detection

* Low number resulted from precipitation during titration

2.0 INTRODUCTION

Due to the need for additional HLW storage, successful disposition of the material in Tank 48H and return of the tank to routine service are two critically needed activities.² As an initial step in the process, SRNL compositionally characterized the components of the Tank 48H slurry. Previously, a Tank 48H slurry sample was collected on August 23, 2004 (HTF-E-04-049 and HTF-E-04-050).³ The August 23, 2004 sample contained approximately 2 Liters of Tank 48H slurry. In December 7, 2004, Tank farm personnel added 3,019 gallons of 50 wt% caustic solution to Tank 48H. On March 6, 2005, about of 4L of slurry was pulled from Tank 48H sample using a new 4.2 L sampler and sent to SRNL. Part of this sample was used for saltstone aggregation studies. The remaining Tank 48H sample was placed in a carboy for future analysis. Small portions of this sample were analyzed and the results are reported in this document.

This document provides the chemical and radiological characterization of a Tank 48H slurry sample as defined in the Technical Task Request Plan⁴ and task technical and quality assurance⁵ plan. At the time of the sampling, the Tank 48H volume was 242,190 gallons (69 inches from the bottom of the tank) and the pumps were ran for 27 hours prior to sampling. The sample was collected within approximately 10 minutes of pump shutdown. A description of the sampler and method is given in Appendix A.⁶ This report compares results with those reported elsewhere (WSRC-TR-2004-00514 [HTF-E-04-049 and HTF-E-04-050] analysis)³, CBU-PIT-2005-00066 [Tank 48 Best Estimate Chemical Characterization as of March 17, 2005]⁷ and CBU-PIT-2005-00046 [Tank 48 Radionuclide Characterization to Support Material Disposition]⁸. Since there was addition of 3,019 gallons of 50 wt% caustic solution to Tank 48H after issuing the WSRC-TR-2004-00514 characterization report, the data from WSRC-TR-2004-00514 needs to be adjusted by dividing the results by a factor of 1.013. This adjustment is necessary to ensure meaningful comparison between data reported here and that reported in WSRC-TR-2004-00514 (or vice versa the results in this report must be increased by 1% for comparison with the WSRC-TR-2004-00514 report). This adjustment factor (1.013) reflects the dilution effect resulting from the addition of 3,109 gallons of caustic solution (where the adjustment factor $1.013 = 1 + \text{volume of caustic added/T48H volume before addition}$).

3.0 EXPERIMENTAL DETAILS

Operations collected and shipped the Tank 48H sample to SRNL. Personnel placed the sample holder (HTF-E-05-021) into the shielded cells on March 6, 2005. Technicians emptied the sample vessels on March 9, 2005 by pumping the contents of the sampler holder into a calibrated 15-L polypropylene carboy using a peristaltic pump with ¼ inch Tygon[®] tubing. The sampler holders were sealed after the transfer.

3.1 PREPARATION OF SLURRY SAMPLES

Personnel diluted the slurry samples with deionized water as required for analyses. The slurry samples were analyzed in duplicate. A portion of the dried slurry was fused with sodium peroxide at 675 °C. The resulting fused mass was digested with stock nitric acid and then diluted with deionized water. This method oxidizes all of the organics in the sample. This procedure utilizes more reagents and therefore results to higher dilution than other digestion methods previously used. Some filtrate samples were diluted with 0.1M nitric acid.

Table 2. Analysis of Tank 48H Slurry Samples

Analysis	Samples	Sample Volume, mL	Dilution Volume, mL	Diluent	Tk 48 Volume, mL
GAMMA SPEC (Cs-137)	3	0.1	3 samples and a blank digested in Cells, then split into sub-samples for analysis		0.3
CVAA HG	3	20			3
RAD K, Hg by AA	3	20			3
RAD ICP-ES (Na, K, Ti, B, Si, Cu, Fe, RCRA metals)	3	1			3
RADICPMS (fission products, actinides, noble metals)	3	1			3
VOA (Benzene)	3	0.1	9.9	DI Water	0.3
SVOA (TPB decomposition products)	3	0.1	9.9	DI Water	0.3
Total Solids	3	3	0	None	9
Density	3	2	0	None	6
HPLC	3	2	8	DI Water	6
Gamma Spec (Cs ¹³⁷)	3	0.1	0	None	0.3

3.2 PREPARATION OF FILTERED SAMPLES

Personnel filtered approximately 60 mL of slurry to produce approximately 50 mL of filtrate for analysis. Technicians moved some of the slurry from the 4-L carboy into a 100 mL polybottle. The contents of the polybottle was well mixed by vigorously shaking and through the use of a magnetic stirrer. A portion of the mixed slurry was removed and filtered using a 0.45 µm supported acrylic copolymer disc filter. They prepared sub-samples from this filtrate without dilution and submitted them for analysis as summarized in Table 3.

Table 3. Listing of Requested Filtrate Analyses

Analysis					
	Samples	Sample Volume, mL	Dilution Volume, mL	Diluent	Tk 48 Volume, mL
GAMMA SPEC	2	0.1	9.9	0.1 M HNO ₃	0.2
HPLC (TPB, 3PB, 2PB, 1PB, & phenol)	2	2	0	None	4
CARBONATE	2	1	0	None	2
TITRATION BASE/OH/OTHER BASE (EXCLUDING CO ₃)	2	1	0	None	2
IC ANIONS [F ⁻ , Cl ⁻ , HCO ₃ ⁻ , (C ₂ O ₄) ²⁻ , (SO ₄) ²⁻ , (NO ₃) ⁻ , (NO ₂) ⁻]	2	1	0	None	2
RAD ICP-ES (Na, K, Ti, B, Si)	2	2	8	0.1 M HNO ₃	4
RAD ICP-MS (actinides)	2	2	8	0.1 M HNO ₃	4
DISSOLVED SOLIDS	2	1	0	None	2
Density	2	5	0	None	10

3.3 IN-CELL ANALYSES

Personnel performed two analyses, each in duplicate, in the shielded cells to minimize the dose to analytical personnel. Personnel determined both the density of the slurry and the total and insoluble solids concentration of the slurry gravimetrically. Note that insoluble solids are defined as those solids that can be removed by filtration, and soluble solids as those solids that can not be filtered, and total solids as sum of the two. These results are reported in wt % solids on a slurry basis.

Personnel gravimetrically determined the density of the slurry at ambient cell temperature (24 °C) using 2 mL Class A micro-volumetric flasks.

Technicians gravimetrically determined the total solids by drying portions of the sample to constant weight at 100 ± 5 °C. They used duplicate analysis of a nominal 15 wt % NaCl standard, slurry sample and the filtered filtrate to measure the total solids and dissolved solids in the Tank 48H material. Technicians pre-weighed clean dry Pyrex™ beakers for each analysis. Personnel mixed the samples thoroughly, removed ~3 mL aliquots, and delivered each aliquot into each beaker (i.e., 3 mL of the 15 wt % NaCl standard into beakers 1, 2, 3 mL; 3 mL of Tank 48H slurry sample into beakers 4-6 and, and 3 mL of Tank 48H filtrate into beakers 7-9). Personnel weighed each beaker with sample immediately after addition then proceeded to dry the samples in a 100-115 °C oven for 8 hours. Samples were allowed to cool in a dessicator for 15 min before additional-weighing. Technicians repeated the drying cycle 3 additional times to ensure complete sample drying.

4.0 EXPERIMENTAL RESULTS

ADS personnel completed the analyses between April 12 and July 25, 2005. The slurry results are generally reported in units of mg/L of slurry and the filtrate results in units of mg/L filtrate. To convert the filtrate results to a slurry bases, the filtrate data is multiplied by a factor of 0.957*. This correction is necessary to compare the filtrate and slurry results to determine the insoluble solids concentration. The results are reported in this section and discussed in detail in Section 5.0, Analysis of Data.

4.1 ADS SLURRY SAMPLE RESULTS

ADS personnel analyzed the filtrate data in duplicate and the digested slurry data in triplicate. When outliers were detected, we reported the average or the value (in the case of duplicate samples) of the remaining sample. We report the analytical data together with the one standard deviation (1σ) uncertainty.

4.1.1 Radionuclide Composition

Many of the radioisotopes are measured using ICP-MS methods. However, personnel determined ^{137}Cs by radio-counting analyses for the Tank 48H slurry – see Table 4. The major radiation hazard in Tank 48H comes from the ^{137}Cs , with a concentration of 1.28 Ci/gallon (2004 measurement reported a value of 1.54 Ci/gallon at total volume of 239,031 gallons and 2003 measurement reported a value of 1.72 Ci/gal). This is a 17% decrease in value relative to the previous measurement and 25% decrease from the 2003 measurement of 1.72 Ci/gal. The current

* This factor is the ratio of filtrate mass (mg/L) to slurry mass (mg/L). It is calculated by the following formula: filtrate correction factor = density slurry/density filtrate*(1-insoluble solids) = $1.165/1.18*(1-0.0305) = 0.957$

value listed on CBU report (CBU-PIT-2005-00046) is 1.72 Ci/gal. As described in section 4.2.4., the ^{137}Cs concentration in the filtrate in this and previous study are similar. The current measurement estimates a total of 310,003 curies of ^{137}Cs in Tank 48H (current volume is 242,190 gallons).

Table 4 – Tank 48H Slurry Radiation Chemistry Analytical Results. Reported value for HTF-E-05-021 are averages of three measurements.

Radionuclide	Sample HTF-E-05-021 (dpm/mL slurry)	Sample HTF-E-04-049 and HTF-E-0050 (dpm/mL slurry)	CBU-PIT-2005-00046 (dpm/mL slurry)
Cs^{137}	$7.53 \pm 0.25 \text{ E}+08$	$9.05 \pm 0.11 \text{ E}+08$	$1.01 \text{ E}+09$

4.1.2 Inductively Coupled Spectroscopy – Emission Spectroscopy for Slurry Samples

Personnel determined the elemental composition (in g/L of slurry) of the digested slurry by ICP-ES. The digestion method used (sodium peroxide fusion) masked the true concentration of sodium in the slurry (previous measurement of sodium measured 3.26 M). The ICP-ES results from another report (HTF-E-05-021) are shown in Table 5. The major constituents in the slurry include Na, K, Al, Fe and B. Aluminum, iron and boron are present in appreciable levels. Approximately equal concentrations of aluminum, manganese, chromium, and titanium were found between this study and previous results. Note that the iron concentration is higher than in previous measurement. The sludge is an aggregate of metal hydroxide/oxide compounds (or particles) and it is possible, through extensive mixing, to separate or disrupt aggregates of particles. Hematite is sub-micron size iron particles which will remain suspended for long times in slurries. The potassium concentration, determined by ICPE-S, measured 5.15 g/L_{slurry}. We suspected this value was too high. This reported value for potassium is not in agreement with the independent measurements obtained by Atomic Absorption ($2.3 \pm 0.04 \text{ g/L}$) as described in a subsequent sections. Previous measurements for two earlier samples collected from the tank report a value similar to the AA result (2.38 g/L). Discussions with both the AA and ICP-ES chemists revealed that the preferred (more accurate and precise) way to measure K is with AA. Elements measured below method detection limits include Ag, Cd, Gd, La, Ni, Pb, and V. Their detection limits are listed in Table 5.

Table 5. The ICP-ES results for the digested slurry from the HTF-E-05-21 sample. All units are given in mg/L slurry

Component	Average	Std. Dev.	Sample HTF-E-04-049 and HTF-E-0050	CBU-PIT-2005- 00066
Ag	<0.01	-		
Al	2014	32	2240	2240
B	867	24	1030	987
Ba	< 0.008	-	2.52	3.47
Be	NM	-		
Ca	43	47	21.4	19.7
Cd	< 0.017	-	NR	0.022
Ce	5	8	7	NR
Cr	70	5	51	46.6
Cu	4	0	3	2.97
Fe	169	29	43	53.9
Gd	< 0.01	-	NR	NR
K	5155 [#]	366	2380	2570
La	< 0.032	-	NR	NR
Li	< 0.026	-	NR	NR
Mg	19	4	18.5	20.2
Mn	6	0	6.38	7.82
Mo	< 0.053	-	13.3	NR
Ni	< 0.015	-	NR	NR
P	129	13	207	NR
Pb	< 0.283	-	NR	NR
S	378	90	245	NR
Sb	< 0.042	-	11.5	NR
Si	125	8	106	NR
Sn	< 0.11	-	22.1	NR
Sr	9	9	5.29	NR
Ti	826	14	840	NR
U	7	12	17.6	NR
V	< 0.007	-	NR	NR
Zn	5	1	12	NR
Zr	1.47	0.13	NR	NR
NM = Not Measured NR = Not Reported *Duplicate measured identical values [#] This value is twice the AA value. This report will use AA value				

4.1.3 Inductively Coupled Spectroscopy – Mass Spectroscopy for Slurry Samples

SRNL performed three analyses of the Tank 48H slurry for actinides. The replicate samples showed excellent precision (Table 6). ^{240}Pu , ^{241}Am , ^{243}Am , and ^{244}Cm (LOD for these elements was less than 0.09 mg/L_{slurry}) are all less than the detection limits. ^{238}Pu can not be detected by this analytical method. No ^{239}Pu was detected in HTF-E-05-021 (LOD = 0.045 mg/L). The limit of detection for ^{239}Pu is above the results previously published.

Table 6. The actinide results for the digested slurry from the HTF-E-05-21 sample. The fourth column shows the actinide data from previous measurements.

Component	Average (mg/L slurry)	Std Dev. (mg/L slurry)	CBU-PIT- 2005-00046 (mg/L slurry)
99 (Tc)	1.23 E0	0.08	NM
237 (Np)	1.73 E-1	0.040	2.83 E-1
239 (Pu)	<0.045 mg/L	-	4.46 E-2
233 (U)	<0.09 mg/L	-	9.44 E-2
234 (U)	2.67 E-1	0.012	4.99 E-1
235 (U)	5.64 E-1	0.050	9.71 E-1
236 (U)	1.25 E-1	0.006	1.48 E0
238 (U)	3.32 E0	0.359	6.16 E0
TOTAL (U)	4.48 E0	0.36	6.32 E0
NM = Not Measured			

We analyzed a slurry sample for transition metals (i.e., Ag, Pd, Rh, and Ru). The elemental results of the Tank 48H slurry sample are presented in Table 7. Contrary to the results for HTF-E-04-049 sample, our analysis found no measurable amounts of these transition metals. The concentrations of these metals (Ru, Rh or Ag) were all below the instrument detection limit. The lower bias shown in this work may be due to the sample preparation involved. Zirconium crucibles were used for peroxide fusion leaching, which normally leads to Zr leaching into the sample matrix. Sodium peroxide fusion method was used to completely oxidize all of the organics. Analytical Division personnel used large amounts of water for dilution and thus raising the limit of detection above the limits previously published. Zirconium interference in the ICP-MS data masked Pd metal detection. Mercury concentration was less than 0.4 mg/L_{slurry} (LOD). The digestion process reached at least 670°C. At this temperature, we expect mercury to volatilize.

Table 7. List the noble metal composition of the slurry HTF-E-05-021 as determined by ICP-MS.

Component	HTF-E-05-021	CBU-PIT-2005-00066
Total Cd, mg/L	1.4 E-02	2.16 E-02
Total Ag, mg/L	< Detection Limit (0.73)	1.88 E-02
Total Pd, mg/L	Interference from Zr	9.28 E-02
Total Rh, mg/L	< Detection Limit (0.7)	2.30 E-01
Total Ru, mg/L	< Detection Limit (1.16)	3.80 E-01

4.1.4 Atomic Absorption Spectroscopy of the Slurry

Personnel determined the potassium content of the digested slurry by AA. The measured potassium concentration in the digested slurry is reported as $2,310 \pm 42$ mg/L (0.06 ± 0.001 M). This concentration is comparable to the value of $2,650 \pm 184$ mg/L (0.068 ± 0.0047 M) reported for HTF-E-04-049 sample. No measurable amounts of As, Hg and Se were detected. The LOD (limit of detection) for As, Hg and Se are 4.6 mg/L, 10.23 mg/L and 4.8 mg/L, respectively. It is worth noting that this method utilizes temperatures that may lead to Hg volatilization. Therefore, the Hg concentration reported here is well below the true value.

4.1.5 Volatile and Semi-Volatile Organic Analyses (VOA and SVOA) for Slurry Samples

SVOA analysis is used to detect aromatic compounds. The SVOA and VOA results from the slurry sample for the HTF-E-05-021 sample are shown in Table 8. Also shown in Table 8 are the results of similar compounds in the HTF-E-04-049 and E-0050 samples. The analysis result indicates the presence of nitro-aromatic compounds and other condensed ring compounds. Although nitro-aromatic compounds are energetic, their low concentrations observed here will not result in a significant temperature rise or gas generation upon decomposition. Table 8 also shows that 1-ethylisopropyl benzene is present. The presence of this compound is not expected nor is it predicted. This compound assignment may be an artifact of the SVOA compound identification method or additives from the sample's bottle. A more precise analytical HPLC was conducted on the HTF-E-05-021 sample and it showed no evidence of this compound or any nitrated aromatics. Volatile analysis indicates the presence of benzene in the slurry. Benzene is a byproduct from the decomposition of TPB and its degradation products.

Table 8. Results from the VOA and SVOA analysis of the slurry HTF-E-05-021.

Component	Average (mg/L slurry)	St Dev (mg/L slurry)	Sample HTF- E-04-049 and HTF-E-0050 (mg/L)
Biphenyl	414.6	105.6	384
Nitrobenzene	180.6	104.4	< 10
Phenol*	98.1	35.7	735
p-terphenyl	182.0	-	< 10
1-ethylisopropyl benzene	64.3	32.7	< 10
Nitrosobenzene	25.3	5.4	< 10
Benzene	17.9	1.0	< 10
*This value is not as precise as the value obtained in Table 9.			

4.1.6 High Performance Liquid Chromatography-Slurry Samples

Personnel measured the concentration of tetraphenylborate and its degradation products in the HTF-E-05-021 slurry sample by HPLC. Table 9 lists the HPLC results for the slurry. The analytical method is considered more sensitive to organic anion measurement than the SVOA method. Both TPB and KTPB concentration, as shown in Table 9, are consistent with previous measurements.

Table 9. The HPLC results of HTF-E-05-021 slurry sample.

Analyte	Concentration, (mg/L slurry)	St. Dev, (mg/L)	CBU-PIT-2005-00066 (mg/L)
TPB Anion	21,000	970	18500
Calculated KTPB	23796	-	20761
3PB	74	16.5	< 50
2PB	142	19	< 50
1PB	151	7.6	< 50
Phenol	771	22	973
Biphenyl	420	54.3	632
NM = Not Measured			

4.2 FILTRATE SAMPLE RESULTS

ADS personnel analyzed duplicate filtrate Tank 48H samples. Personnel removed samples of the Tank 48H filtrate from the cells without dilution. ADS personnel diluted the filtrate as appropriate for the analyses. We report the analytical data together with the one standard deviation (1σ) uncertainty. To compare the slurry results with the filtrate results, the filtrate result is multiplied by a factor of 0.957 to convert to a slurry basis. Note that a filtrate sample has been filtered to remove insoluble solids and a supernate sample has been pulled from an un-slurried tank but has not been filtered. It is expected that supernate results from previous samples would have slightly higher insoluble solids concentrations since additional caustic solution was added to Tank 48H before the collection of the earlier samples.

4.2.1 Anion Analysis by Ion Chromatography for Filtrate Sample

Table 10 contains the measured values for the Tank 48H filtered sample. Table 10 includes the measured anion concentrations for the tank sample. Nitrate and nitrite concentrations are 0.52M and 0.23M, respectively.

Table 10. The anion composition of HTF-E-05-021 filtrate sample. The two set values are from duplicate analysis.

Anion Component	Average Concentration (mg/L)	Standard Deviation (mg/L)	CBU-PIT-2005-00066 (mg/L)
F ⁻	14	0 (exact duplicates)	< 18
Cl ⁻	172	23	125
HCOO ⁻	502	10	432
NO ₂ ⁻	23750	350	21400
NO ₃ ⁻	14250	150	13400
PO ₄ ²⁻	428	9	515
SO ₄ ²⁻	323	1	273
C ₂ O ₄ ²⁻	1440	10	1080

4.2.2 High Performance Liquid Chromatography-Filtrate Sample

ADS completed analysis of the Tank 48H filtrate for tetraphenylborate and 14 of its common decomposition products. Only phenol at 535 ± 5 mg/L occur above the detection limit. TPB, 3PB, 2PB, and 1PB all fell below detection limits of 10 mg/L (see Table 11). Other decomposition products were below method's detections limit.

Table 11. The phenylborate composition of the filtrate as determined by HPLC (mg/L).

Component	1 st sample	2 nd sample	Average or Limit of Detection	CBU-PIT-2005-00066
TPB	< 10	< 10	< 10	< 10
3PB	< 10	< 10	< 10	< 10
2PB	< 10	< 10	< 10	< 10
1PB	< 10	< 10	< 10	< 10
phenol	531	538	534.5 ± 5	706

4.2.3 Wet Chemistry-Filtrate Sample

ADS personnel analyzed the filtrate samples using titration methods and density measurements. The total base concentration is 2.48 ± 0.05 M and the free hydroxide is 1.34 ± 0.01 M. The concentration of other bases is 0.172 ± 0.02 M and carbonate concentration is 0.385 ± 0.01 M. Table 12 lists the titration results for the HTF-E-05-021 sample.

The free hydroxide concentration at 1.34 M has increased since the September 2003 sampling (0.790 M) due to the addition of sodium hydroxide in October 2003 and December 2004.

ADS measured the Tank 48H filtrate density as 1.14 ± 0.01 g/mL.

The dissolved solids concentration of the Tank 48H filtrate was $17.68\% \pm 0.14$ wt %. The dissolved solids concentration of the August 24, 2004 filtrate was 16.28 ± 0.27 wt %. The dissolved solids concentration of the June 3, 2003 and September 23, 2003 filtrate was 16.6 wt %.

Table 12. The base composition of HTF-E-05-021 filtrate sample. The two set values are from duplicate sample analysis. Value in parenthesis represents 1 sigma uncertainty.

Component	(Molar)	(Molar)	Average (Molar)	Sample HTF-E-04-049 and HTF-E-0050 (Molar)
Total Base	2.52 ($\pm 10\%$)	2.45 ($\pm 10\%$)	2.485 ± 0.05	2.04
Free OH ⁻	1.35 ($\pm 10\%$)	1.33 ($\pm 10\%$)	1.34 ± 0.014	1.155
Other Base (excluding CO ₃ ²⁻)	0.156 ($\pm 25\%$)	0.188 ($\pm 25\%$)	0.172 ± 0.023	0.185
CO ₃ ²⁻	0.389 ($\pm 25\%$)*	0.382 ($\pm 25\%$)*	0.3855 ± 0.005	0.492
* precipitates observed during titration				

4.2.4 Radionuclide Composition for Filtrate Sample

ADS personnel analyzed the filtered sample using radio-analytical methods. Cesium (¹³⁷Cs) is the major radioactive analyte in the filtrate at a concentration of $3.0 \pm 0.4 \text{ E}7 \text{ dpm/mL}$. Table 13 summarizes the radionuclide concentrations. Other isotopes were not analyzed. Table 14 provides a comparison with previous sample results.

Personnel analyzed the filtrate radionuclide content by ICP-MS. Table 14 lists the lanthanide and actinide concentration for the HTF-E-05-021 sample.

Table 13. Tank 48H Filtrate radiation chemistry data.

Analyte	Sample HTF-E-005-021	CBU-PIT- 2005- 00046
¹³⁷ Cs (dpm/mL)	$3.0 \pm 0.4 \text{ E}+07$	$2.76 \text{ E}+07$

Table 14. The ICP-MS results of HTF-E-05-021 filtrate sample. The two set values are from duplicate sample analysis.

Actinide	1 st sample (mg/L)	2 nd Sample (mg/L)	Average (mg/L)	CBU-PIT-2005-00046 (mg/L)
²³² Th	1.952 E-2	-	1.952 E-2 ± 0	NM
²³³ U	1.035 E-2	1.009 E-2	1.022 E-2 ± 0.18 E-3	4.94 E-2
²³⁴ U	7.295 E-2	6.960 E-2	7.128 E-2 ± 2.4 E-3	3.58 E-1
²³⁵ U	1.411 E-1	1.450 E-1	1.431 E-1 ± 2.8 E-3	5.74 E-1
²³⁶ U	3.050 E-2	3.150 E-2	3.100 E-2 ± 0.7 E-3	1.41 E0
²³⁷ Np	1.862 E-2	1.851 E-2	1.856 E-2 ± 0.1 E-3	5.39 E-2
²³⁸ U	8.453 E-1	8.054 E-1	8.253 E-1 ± 28 E-3	3.62 E0
Total U	-	-	1.10 E0	6.01 E0

4.2.5 Inductively Coupled Spectroscopy – Emission Spectroscopy-Filtrate Sample

ADS determined the elemental composition of the filtrate by the ICP-ES method. The major constituents found in the filtrate included Na and B, as shown in Table 15. The element sodium is present in the highest concentration at 3.82 M. Boron is present in appreciable levels, too. Elements measured below instrument detection limits include Ag, Ba, Cd, Ce, Fe, Gd, La, Li, Mg, Mn, Ni, Pb, Sn, Ti, U, and Zr (LOD values for these elements are listed in Table 15). Table 15 provides the elemental results of the Tank 48H filtrate samples. One of the samples analyzed showed low K concentration. We attribute this low [K] value to possible over dilution with water. Table 16 provides a comparison with historical data. Inspection of Table 16 reveals higher Na and B concentrations. This we attribute to additions to Tank 48H and to TPB decomposition.

Table 15. The cation composition of HTF-E-05-021 filtrate sample as determined by ICP-ES. The two set values are from duplicate sample analysis.

Cation Component	1 st Sample (mg/L)	2 nd Sample (mg/L)	Average (mg/L) or LOD	Standard Deviation (mg/L)
B ⁺ *	466.7	453.3	460	9.47
K ⁺	< 39	266.7	266.7	-
Na ⁺	85848	89950	87899	2948
Si ⁴⁺	< 125	< 125	< 125	-
Ti ⁴⁺	< 1	< 1	< 1	-
Al	2344	2272	2309	50.7
Si	<125	<125	<125	-
Ag	<3	<3	<3	-
Ba	<6	<6	<6	-
Cd	<1	<1	<1	-
Ce	<35	<35	<35	-
Fe	<3	<3	<3	-
Cu	<3	<3	<3	-
Gd	<4	<4	<4	-
La	<13	<13	<13	-
Li	<12	<12	<12	-
Mg	<1	<1	<1	-
Mn	<1	<1	<1	-
Ni	<10	<10	<10	-
Pb	<196	<196	<196	-
Sn	<77	<77	<77	-
Ti	<1	<1	<1	
U	<50	<50	<50	
Zr	<2	<2	<2	
*The presence of B may be due to Borates such as B(OH) ₂ O ⁻ for example. LOD = Limit of Detection				

Table 16. Comparison between HFT-E-05-021 filtrate and previous sample's analysis

Sample ID Analysis	HFT-E-05-021	CBU-PIT-2005-00066
ICP-ES		
Al, mg/L	2.31 E3	2.18 E3
B, mg/L	4.60 E2	4.43 E2
Na, mg/L	8.79 E4*	6.93 E4
S, mg/L	3.2 E2	NM
K, mg/L	2.67 E2	2.48 E2
AA		
K, mg/L	2.54 E2	-
NM = Not Measured		
* Increase due to addition of NaOH.		

4.3 IN-CELL SLURRY SAMPLE RESULTS

Personnel performed analyses in the shielded cell, because of the high radioactivity of the samples. Solids analyses, slurry titration and density analyses were analyzed in duplicate.

4.3.1 Dissolved Solids and Density Analysis

Based on triplicate analysis, the slurry total solids is $20.19 \pm 0.08\%$ and the filtrate dissolved solids content is $17.68 \pm 0.14\%$. Table 17 shows the gravimetric results from duplicate samples analysis. We calculate the value for the insoluble solids (3.05 ± 0.15 wt %) using the following formula.⁹

$$\text{Insoluble Solids} = \frac{(\text{total solids} - \text{dissolved solids})}{(1 - \text{dissolved solids}/100)}$$

$$\text{Insoluble Solids} = (20.19 - 17.68) / (1 - 17.68/100) = 3.05 \pm 0.15 \text{ wt}\%$$

$$\text{Soluble Solids} = 20.19 - 3.05 = 17.14 \pm 0.15 \text{ wt}\%$$

Based on this calculation, the insoluble solids measurement is consistent with previous analyses (1.69 ± 0.22 % for Ref.3, 2.18 ± 0.61 for Ref. 10, and 2.3 % for Ref. 9).^{9,10} The insoluble solids measurement has a range of 2.9 wt % to 3.2 wt % based on the calculated uncertainties. This overlaps with the sum of the measured KTPB, MST, iron, aluminum, silicon and insoluble organics as shown in Table 18.

Table 17. The gravimetric properties of HTF-E-05-021 filtrate sample.

	Sample 1	Sample 2	Average	Sample HTF-E-04-049 and HTF-E-0050 (Molar)
Total Dissolved Solids (wt%)	17.42	17.94	17.68 ± 0.26	17.68
Density (g/mL)	1.14	1.14	1.14 ± 0.06	1.164

Table 18. Composition of the total solid mass for 1 gram of dried solids.

Component	g/g dry solid	Analytical Method
Amount of MST ($\text{NaTi}_2\text{O}_5\text{H}$)	0.0073	ICP-ES (Table 5)
Amount of Hematite (Fe_2O_3)	0.001	ICP-ES (Table 5)
Amount of Gibbsite ($\text{Al}(\text{OH})_3$)	0.025	ICP-ES (Table 5)
Chromium (Cr_2O_3)	0.001	ICP-ES (Table 5)
KTPB	0.101	AA
Organics	0.0041	SVOA
Silicon (SiO_2)	0.0013	ICP-ES
Sum of all of the above (IS)	0.1384	-
Measured Dissolved solids	0.8494	Weight
Sum Dissolved Solid + Insoluble Solids	0.988 g	-
Expected Dried Total Solids	1g	-
$\text{g/gdry} = \text{g/L} \times (1/\text{slurry density}) \times (100/\text{Total solids}) \times (\text{mol. Wt compound}/\text{mol. Wt element})$		

4.3.2 In-cell Slurry Density

Technicians determined density from the average of triplicate measurements of the Tank 48H slurry. Based on these measurements, the density of the Tank 48H slurry equals $1.165 \pm 0.002\text{g/mL}$ (at 24°C).

5.0 ANALYSIS OF DATA

Completion of an anion and cation balance provides a consistency check on the data. By comparing filtrate potassium and cesium concentrations, information on the decomposition rate for tetraphenylborate in Tank 48H can be obtained.

5.1 TANK 48H ION BALANCE

The sum of the major cations exceeds the sum of the major anions by ~19%. We attribute this significant difference to low carbonate values obtained from titration tests of the filtrate. Personnel reported observing precipitates during the process. We believe the carbonate level should range around 0.4 Molar. Table 19 sums the anions and cations using the filtrate analyses for the major components.

Table 19. Tank 48H Filtrate Anion/Cation Balance

Analyte	Moles ions	1 σ Uncertainty, moles	Method
Al(OH) ₄ ⁻	0.085	0.000	ICP-ES
BO ₃ ³⁻	0.043	0.001	ICP-ES
C ₂ O ₄ ²⁻	0.016	0.001	IC
Cl ⁻	0.014	0.003	IC
COOH ⁻	0.004	0.005	IC
CO ₃ ²⁻	0.385*	0.001	Titration
NO ₂ ⁻	0.516	0.032	IC
NO ₃ ⁻	0.230	0.012	IC
OH ⁻	1.34	0.007	Titration
PO ₄ ³⁻	0.005	0.001	IC
SO ₄ ²⁻	0.003	0.001	IC
Total Anions	3.14	0.06	Calculation
B+	0.042	0.001	ICP-ES
K+	0.006	0.005	AA
Na+	3.82 [#]	0.029	ICP-ES
Total Cations	3.87	0.03	Calculation
*Precipitates were seen during titration of filtrate. We expect the carbonate level to be near 0.4 M.			
[#] This value is higher than the expected value of 3.01 M (CUB-PIT-2005-00066).			

6.0 SUMMARY

Personnel analyzed samples taken from Tank 48H on August 23, 2004 for chemical and radiological constituents. This report documents the analytical results and analysis of this data.

The results demonstrate that samples pulled in September 2003 and August 2004 are very similar in chemical composition. The free hydroxide concentration, sodium concentration, soluble solids and density have all increased as expected due to the addition of 6,424 gallons and 3,019 gallons of 50 wt % sodium hydroxide, respectively, in October 30, 2003 and December 7, 2004.

Data collected during sampling is summarized in Appendix B.

7.0 ACKNOWLEDGEMENTS

This task required the support of a large team of people. We thank the following individuals but note that many others assisted in completing this task.

We thank the dedicated Shielded Cells Technicians, especially Monica Jenkins for receiving the Tank 48H sample, completing the analyses in the cells as requested and preparing the hundreds of samples for transfer to ADS.

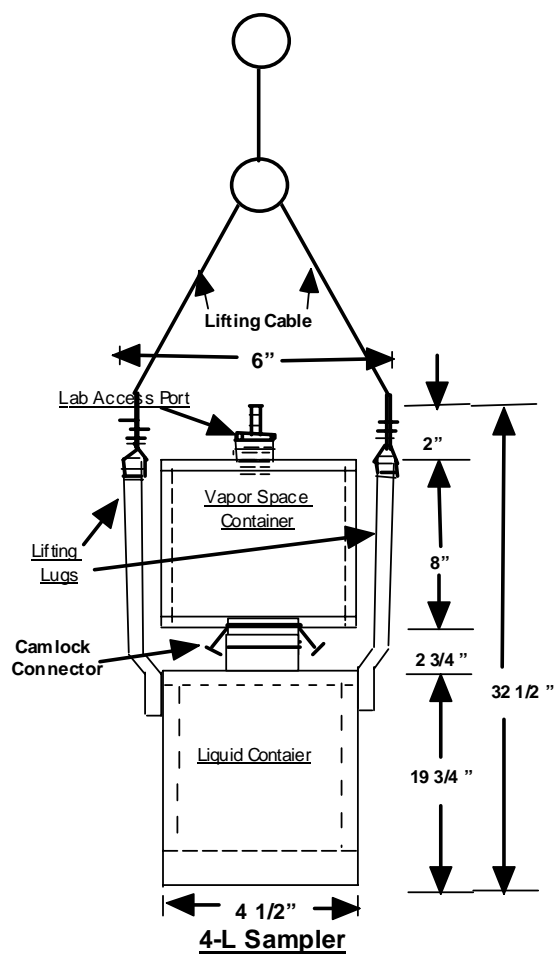
We thank the following personnel in the Analytical Development Section for analyzing samples in a busy time period. We list the researchers involved but omit the many technicians supporting them to complete this task on schedule. Thanks to Leigh Brown, John Young, Tom White, Robert Ray, Curtis Johnson, David DiPrete, Ceci DiPrete, June Hart, Bill Boyce, Chuck Coleman, and Damon Click.

We appreciate the continued support of our WPT technician, Sharon Smith, who prepared the sample bottles for the shielded cells technicians and verified that the written instructions accomplished the intended tasks.

We appreciate the support of the Tank 48H team, for their technical support in helping us accomplish this task.

Appendix A – A picture of the Slurry Sampler

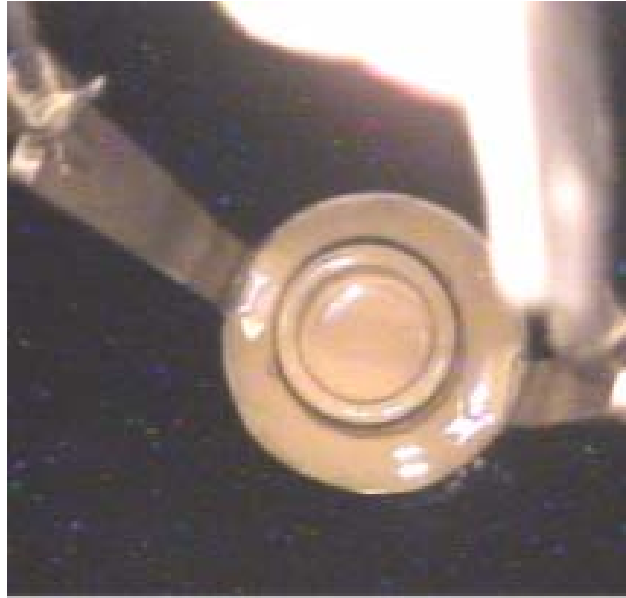
T48 4L Sampler
(March, 05 Sampling)



4 Liter Sampler
Conceptual Sketch



Photo of final fabricated sampler



**Full Sampler with
T48 Material**

Appendix B – Tank 48H Sample Results Summary

Analyte	Slurry	Filtrate	Source
Volume, gal	242,190	NM	Tank 48H
Density, mg/L	1.165	1.14	Density
Total Solids, wt %	20.19%	17.68%	Solids
Total Insolubles	3.05%	NM	Calculation
KTPB, wt %	2.01%	<0.001%	HPLC
MST solids, wt %	0.15%	(<0.15 mg/L)	ICP-ES
Anions			
Free Hydroxide, M	NM	1.34	Titration
Carbonate, M	NM	0.385*	Titration
Nitrite, mg/L	NM	23750	IC
Nitrate, mg/L	NM	14250	IC
Oxalate, mg/L	NM	1440	IC
Formate, mg/L	NM	502	IC
Chloride, mg/L	NM	172	IC
Fluoride, mg/L	NM	14	IC
Phosphate, mg/L	NM	428	IC
Sulfate, mg/L	NM	323	IC
Organic Species			
TPB Anion, mg/L	21,000.	<10	HPLC
3PB, mg/L	74	<10	HPLC
2PB, mg/L	142	<10	HPLC
1PB, mg/L	151	<10	HPLC
Phenol, mg/L	771	535	HPLC
Nitrobenzene, mg/L	180.6	<10	SVOA/HPLC
Nitrososbezene, mg/L	25.3	<10	SVOA/HPLC
4phenylphenol, mg/L	<50	<10	HPLC
2phenylphenol, mg/L	<50	<10	HPLC
Diphenylamine, mg/L	<50	<10	HPLC
Biphenyl, mg/L	420	<10	HPLC
o-terphenyl, mg/L	<50	<10	HPLC
m-terphenyl, mg/L	<50	<10	HPLC
p-terphenyl, mg/L	182.0	<10	SVOA/HPLC
benzene, mg/L	17.9	<10	SVOA/HPLC
* Precipitation seen during analysis (titration)			

Metals	Slurry	Filtrate	Source
Potassium, mg/L	2310	267	AA
Hg, mg/L	< 10.23*	NM	AA
Sodium, mg/L	74980	87899	ICP-ES
Aluminum, mg/L	2014	2210	ICP-ES
Boron, mg/L	867	460	ICP-ES
¹³³ Cesium, mg/L	12	NM	ICP-MS
Sulfur, mg/L	378	320	ICP-ES
Phosphorus, mg/L	129	NM	ICP-ES
Silicon, mg/L	125	<125	ICP-ES
Iron, mg/L	169	<3	ICP-ES
Chromium, mg/L	70	NM	ICP-ES
Magnesium, mg/L	19	< 1	ICP-ES
Calcium, mg/L	43	NM	ICP-ES
Molybdenum, mg/L	<0.05	NM	ICP-ES
Antimony, mg/L	<0.46	NM	ICP-ES
Zinc, mg/L	5	NM	ICP-ES
Manganese, mg/L	6	< 1	ICP-ES
Strontium, mg/L	9	NM	ICP-ES
Barium, mg/L	<0.09	< 6	ICP-ES
Copper, mg/L	4	< 3	ICP-ES
Total Ag, mg/L	<0.73	NM	ICP-MS
Total Pd, mg/L	Prep. Interference	NM	ICP-MS
Total Rh, mg/L	<0.7	NM	ICP-MS
Total Ru, mg/L	< 1.16	NM	ICP-MS
Total Cd, mg/L	0.014	NM	ICP-MS
Total Hg, mg/L	< 0.45*	NM	ICP-MS
Tc-99, mg/L	1.23	NM	ICP-MS
Th-232, mg/L	NM	1.95E-02	ICP-MS
Np-237, mg/L	0.173	0.0186	ICP-MS
Pu-239, mg/L	<0.045	<0.05	ICP-MS
U-233, mg/L	<0.09	0.01	ICP-MS
U-234, mg/L	0.267	0.0713	ICP-MS
U-235, mg/L	0.564	0.1431	ICP-MS
U-236, mg/L	0.125	0.031	ICP-MS
U-238, mg/L	3.32	0.825	ICP-MS
U Total, mg/L	4.48	1.1	ICP-MS
Cs-137, dpm/mL	7.53 E+08	3.0 E+07	ICP-MS
* Hg may have volatilized during sample digestion.			

8.0 References

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