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- Changed units for prediction of tetraphenylborate decomposition rate in Tank 48H from “0.0898 mg K/L/hr” to “0.0898 mg K/L/day”. This number is documented in the 3rd paragraph in the conclusion section on page 14.
- Changed date of report to “May 2005” and changed revision number from “0” to “1”.
- Deleted extra period in values reported using engineering notation in Tables 7, 8 and Appendix B. For example, changed “2.22.E-01” to “2.22E-01” for Np-237 in Table 7.
- Deleted redundant units in Tables 14 (“M” was listed twice in each row).
- Added units “, mg/L” to title of Table 9.
- All changes were noted using a change bar in the margin of the report.

**ANALYSIS OF TANK 48H SAMPLES
HTF-E-04-049 AND HTF-E-04-050
(AUGUST 23, 2004)**

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| May 2005

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Prepared for the U.S. Department of Energy Under Contract Number
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SAVANNAH RIVER NATIONAL LABORATORY

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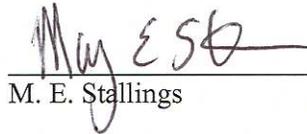
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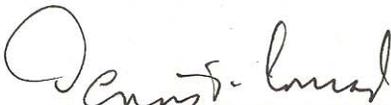
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LIST OF ACRONYMS

| | |
|--------|----------------------------------------------------------------------------|
| 1PB | phenylboronic acid |
| 2PB | diphenylborinic acid |
| 3PB | triphenylborane |
| 4PB | tetraphenylborate anion – $[\text{B}(\text{C}_6\text{H}_5)_4]^-$ |
| AA | Atomic Absorption |
| ADS | Analytical Development Section |
| CsTPB | Cesium tetraphenylborate ($\text{Cs}[\text{B}(\text{C}_6\text{H}_5)_4]$) |
| 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 – $\text{K}[\text{B}(\text{C}_6\text{H}_5)_4]$ |
| MST | monosodium titanate |
| MW | Molecular Weight |
| NA | Not Applicable |
| NM | Not Measured |
| NaTPB | Sodium tetraphenylborate – $\text{Na}[\text{B}(\text{C}_6\text{H}_5)_4]$ |
| SRNL | Savannah River National Laboratory |
| TPB | Tetraphenylborate - $[\text{B}(\text{C}_6\text{H}_5)_4]^-$ |
| WPTS | Waste Processing Technology Section |

1.0 EXECUTIVE 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 measured potassium tetrphenylborate (KTPB) concentration is 1.95 ± 0.22 wt %.
- The calculated monosodium titanate (MST) concentration is 0.15 ± 0.015 wt % MST.
- The measured insoluble solids content was 1.69 ± 0.22 wt %. The sum of KTPB and MST is 24% higher than the insoluble solids concentration. The insoluble solids result is lower than the tank based on previous Tank 48H analyses.
- The free hydroxide concentration in the Tank 48H filtrate sample (1.16 ± 0.007 M) is greater than the Tank 48H limit (1.0 M). This is an increase of 0.37 M since the September 2003 sample due to the addition of 6,424 gallons of 50 wt % sodium hydroxide to Tank 48H on October 30, 2003.
- The soluble potassium content in the filtrate continues to follow the linear trend that began in 1995 showing slow, radiolytic decomposition of the tetraphenylborate solids.
- The measured ^{137}Cs concentration is $9.05\text{E}+08 \pm 1.07+07$ dpm/mL (1.54 Ci/gallon) in the slurry and $2.57\text{E}+07 \pm 6.01\text{E}+04$ dpm/mL in the filtrate. This is equivalent to 367,000 total ^{137}Cs curies in Tank 48H. The ^{137}Cs does not follow the earlier linear trend.
- The measured Total Alpha concentration is $1.01\text{E}+04 \pm 6.51\text{E}+02$ dpm/mL in the slurry and $2.00\text{E}+03 \pm 3.891\text{E}+02$ dpm/mL in the filtrate. This is equivalent to 4.13 total curies from alpha emitters in Tank 48H.
- Three tetraphenylborate decomposition products, triphenylborane (3PB), diphenylborinic acid (2PB), phenylboronic acid (1PB), were all detected in the slurry but not in the filtered sample.

The more significant analytical data is summarized in Table 1.

Deleted: Table

Table 1 – Summary of Significant Tank 48H Sample Results

| Analyte | Slurry | Supernate | Source |
|----------------------------|----------|-----------|-------------|
| Volume, gal | 239,031 | N/A | Tank 48H |
| Density, g/mL | 1.162 | 1.164 | Density |
| Total Solids, wt % | 19.08% | 17.68% | Solids |
| Total Insolubles, wt % | 1.69% | NM | Calculation |
| KTPB, wt % | 1.95% | <0.001% | HPLC |
| MST solids, wt % | 0.15% | NM | ICP-ES |
| Metals | | | |
| Sodium, M | 3.26 | 2.99 | ICP-ES |
| Potassium, M | 0.068 | 0.0065 | AA |
| Anions | | | |
| Free Hydroxide, M | NM | 1.155 | Titration |
| Carbonate, M | NM | 0.492 | Titration |
| Nitrite, M | NM | 0.649 | Anion |
| Nitrate, M | NM | 0.304 | Anion |
| RadChem | | | |
| ^{137}Cs , dpm/mL | 9.05E+08 | 2.57E+07 | RadChem |
| Total Alpha, dpm/mL | 1.01E+04 | 2.00E+03 | RadChem |

NM=Not Measured

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.¹ As an initial step in the process, SRNL compositionally characterized the components of the Tank 48H slurry. A nominal Tank 48H Tank 48H slurry sample was collected on August 23, 2004 (HTF-E-04-049 and HTF-E-04-050). The August 23, 2004 sample contained approximately 2 Liters of Tank 48H slurry.

This document provides the chemical and radiological properties of a Tank 48H slurry sample. A Technical Task Request defines the required analyses². A Task Plan summarized the analyses required and the methods for completing these analyses³. The Tank 48H volume was 239,000 gallons (68.2 inches) at the time of the sampling.

5-L Sample Vessels

The Tank 48H team requested collection of two five-liter samples of Tank 48H slurry material after a minimum of 24 hours of slurry pump operations and immediately after pump shutdown. Sample collection utilized a sampler fabricated by Salt Works.

The total volume of the sampler is 6.54 liters with a liquid fill volume of 4.95 liters. The sample enters the 5-Liter vessel through the inlet port located on the top of the sampler. A crane lowered the sampler approximately 18 inches below the liquid surface. Once filled, the crane raised the sampler to the surface and capped the sampler. The sampler was washed, pulled to the top of the tank, and radiation rates monitored by Radiological Control inspector to confirm the presence of material in the sampler prior to placement in the 8-ton cask.⁴

Collection of the Tank 48H sample occurred on August 23, 2004 by lowering the uncapped 5-L sampler into Tank 48H. Personnel pulled sample HTF-E-04-0049 within 2 minutes of shutting down all four slurry pumps and following 11 hours of continuous operation at full speed (1180 rpm). The four slurry pumps were restarted and operated for an additional 2 continuous hours at full speed (1180 rpm). Personnel pulled sample HTF-E-04-0050 within 8 minutes of shutting down all four slurry pumps. Data concerning the pump operational times, Tank 48H volume, and seal leakage estimates are summarized in Appendix A.

3.0 EXPERIMENTAL DETAILS

Operations shipped two samples to SRNL. Personnel placed the two samplers (HTF-E-03-127) into the shielded cells on August 24, 2004. Technicians emptied the sample vessels, on August 25, 2004, by pumping the contents of the sampler into the calibrated 15-L polypropylene carboy using a peristaltic pump with ¼ inch Tygon[®] tubing. The sample was later transferred to a 4-L carboy. Researchers recovered 1.9 L of slurry from the two samplers. The samplers were sealed after emptying.

3.1 PREPARATION OF SLURRY SAMPLES

Personnel diluted the slurry samples as required for analyses. Analysis of all slurry samples occurred in duplicate. Dilution used deionized water while digestion used an acid/hydrogen

peroxide solution to destroy the organic, dissolve the metals and dilute the samples. Table 2 summarizes the slurry samples submitted for analysis.

Table 2 – Listing of Requested Slurry Analyses and Dilutions

| OPERATION | Samples | Sample Volume, mL | Dilution Volume, mL | Diluent | Tk 48 Volume, mL |
|-------------------------|---------|-------------------|---------------------------|----------|------------------|
| HLC-DENSITY | 2 | 2 | 0 | None | 4 |
| DISSOLVED SOLIDS (B101) | 2 | 3 | 0 | None | 6 |
| TOTAL SOLIDS (B101) | 2 | 3 | 0 | None | 6 |
| HPLC (B123) | 2 | 2 | 8 | DI Water | 4 |
| Gross Alpha (B145) | 2 | 0.5 | Nitric acid Prep in Cells | | 1 |
| GAMMA SPEC (B145) | 2 | 0.1 | | | 0.2 |
| CVAA HG (B143) | 2 | 1 | | | 2 |
| RAD K by AA | 2 | 1 | | | 2 |
| RAD ICP-ES LCS (B151) | 2 | 1 | | | 2 |
| RADICPMS (B067) | 2 | 1 | | | 2 |

3.2 PREPARATION OF FILTERED SAMPLES

Personnel filtered approximately 60 mL of slurry to produce approximately 50 mL of filtrate for analysis. Technicians removed a portion of the slurry from the 4-L carboy and filtered it using a 0.45 μm supported acrylic copolymer disc filter. They prepared sub-samples from this filtrate without dilution and submitted for analysis as summarized in Table 3.

Table 3 – Listing of Requested Filtrate Analyses

| OPERATION | Samples | Sample Volume, mL | Dilution Volume, mL | Diluent | Tk 48 Volume, mL |
|-------------------------------------|---------|-------------------|---------------------|-------------|------------------|
| DISSOLVED SOLIDS (B101) | 2 | 1 | 0 | None | 2 |
| HPLC (B123) | 2 | 2 | 0 | None | 4 |
| IC ANIONS LCS (B134) | 2 | 1 | 0 | None | 2 |
| Gross Alpha (B145) | 2 | 0.1 | 9.9 | .1 M Nitric | 0.2 |
| GAMMA SPEC (B145) | 2 | 0.1 | 9.9 | .1 M Nitric | |
| RAD ICP-ES LCS (B151) | 2 | 2 | 8 | .1 M Nitric | 4 |
| RAD K by AA | 2 | 2 | 8 | .1 M Nitric | 4 |
| CARBONATE (B154) | 2 | 1 | 0 | None | 2 |
| T BASE/OH/OTHER BASE EXC CO3 (B154) | 2 | 1 | 0 | None | 2 |

3.3 IN-CELL ANALYSES

Personnel performed two analyses, each in duplicate, in Shielded Cell Block B to minimize the dose to analytical personnel. Personnel gravimetrically determined the density of the slurry, and gravimetrically determined the total and insoluble solids concentration of the slurry. Note that insoluble solids are defined as those solids that can be removed by filtration, soluble solids are those solids that can not be filtered, and total solids are a 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 Shielded Cells temperature using 2 mL Class A micro-volumetric flasks.

Technicians gravimetrically determined the total solids by drying portions of the sample to constant mass at 100 ± 5 °C. They used duplicate analysis of a nominal 15 wt % NaCl standard, slurry sample and the filtered filtrate to measure 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 to each beaker (i.e., 3 mL of the 15 wt % NaCl standard to beakers 1-2, 3 mL of Tank 48H slurry sample to beakers 3-4 and, and 3 mL of Tank 48H filtrate to beakers 5-6). 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 cooled in a dessicator for 15 min before additional-weighing. Technicians repeated the drying cycle 3 additional times to ensure complete drying.

4.0 EXPERIMENTAL RESULTS

ADS personnel completed the analyses between September 1, 2004 and October 21, 2004. The slurry results are generally reported as mg/L of slurry. The filtrate results are reported as mg/L filtrate. To convert the filtrate results to a slurry basis, multiply filtrate result, in mg/L, by 0.981*. 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 Section 5.0, Analysis of Data.

4.1 ADS SLURRY SAMPLE RESULTS

ADS personnel analyzed the diluted or digested slurry in duplicate. We report the analytical data, together with the one standard deviation (1σ) uncertainty.

4.1.1 High Performance Liquid Chromatography

HPLC analysis is used to detect TPB, phenylborates (3PB, 2PB, and 1PB), phenol, and other TPB decomposition products. HPLC analysis of a Tank 48H slurry sample provided detectable quantities of TPB, triphenylborane (3PB), diphenylborinic acid (2PB), phenylboronic acid (1PB) nitrobenzene, phenol and biphenyl (Table 4). Less than detectable quantities (<50 mg/L) of nitrosobenzene, 4-phenylphenol, 2-phenylphenol, diphenylamine, o-terphenyl, m-terphenyl, and p-terphenyl existed. The KTPB concentration is 22,600 mg/L or 1.95 ± 0.22 wt % assuming all the TPB is present as KTPB.

The prior Tank 48H sample (September 2003) did not have a detectable concentration of 3PB, 2PB or 1PB (each has a detection limit of <50 mg/L after accounting for the dilution necessary to move samples from the shielded cells to ADS for analysis). A reanalysis of the slurry confirmed the presence of the phenylborate intermediates.

* 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) = $1.162/1.164*(1-0.0169) = 0.981$

Table 4 – HPLC Slurry Results for the Tank 48H sample

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 (mg/L) | 1 σ Uncertainty (mg/L) |
|-----------------|-------------------------------------------|-------------------------------|
| TPB | 20,100 | 235 |
| Calculated KTPB | 22,600 | 263 |
| Phenol | 735 | 24.0 |
| Biphenyl | 384 | 61.4 |
| 3PB | 162 | 35.0 |
| 2PB | 123 | 6.1 |
| 1PB | 120 | 5.6 |

Analytical uncertainty is 10% for all analytes except biphenyl (20%)

4.1.2 Radionuclide Composition

Many of the radioisotopes are measured using ICP-MS methods. However, personnel determined ^{90}Sr , ^{137}Cs and gross alpha by radio-counting analyses for the Tank 48H slurry – see Table 5. The major radiation hazard in Tank 48H comes from the ^{137}Cs , with a concentration of 1.54 Ci/gallon. This is equivalent to 367,000 total ^{137}Cs curies in Tank 48H. The gross alpha analysis is used to calculate 4.13 total curies from alpha emitters in Tank 48H.

Table 5 – Tank 48H Slurry Radiation Chemistry Analytical Results

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|----------------------------|------------------------------------|------------------------|
| ^{137}Cs (dpm/mL) | 9.05E+08 | 1.07E+07 |
| Gross Alpha (dpm/mL) | 1.01E+04 | 6.51E+02 |

4.1.3 Inductively Coupled Spectroscopy – Emission Spectroscopy

Personnel determined the elemental composition of the digested slurry by ICP-ES. The major constituents found in the slurry include Na, K, Al, and B. The element sodium is present in the highest concentration at $74,900 \pm 660$ mg/L (3.26 ± 0.029 M). Aluminum and boron are present in appreciable levels. Elements measured below instrument detection limits include Ag, Be, Cd, Gd, La, Ni, Pb, V, and Zr. The elemental results of the Tank 48H slurry sample are presented in Table 6.

Table 6 – Major Elemental Constituents by ICP-ES in the Tank 48H Slurry Sample

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|---------|------------------------------------------|------------------------|
| Al | 2,240 | 0.00 |
| B | 1,030 | 1.64 |
| Ba | 2.52 | 0.03 |
| Ca | 21.4 | 1.23 |
| Ce | 6.89 | 0.11 |
| Cr | 51.1 | 0.16 |
| Cu | 2.97 | 0.16 |
| Fe | 43.4 | 0.16 |
| K | 2,380 | 16 |
| Mg | 18.5 | 0.25 |
| Mn | 6.38 | 0.06 |
| Mo | 13.30 | 0.08 |
| Na | 74,900 | 657 |
| P | 207 | 4.93 |
| S | 245 | 1.64 |
| Sb | 11.5 | 0.16 |
| Si | 106 | 0.99 |
| Sn | 22.1 | 2.05 |
| Sr | 5.29 | 0.17 |
| Ti | 840 | 0.82 |
| U | 17.6 | 0.74 |
| Zn | 12 | 0.08 |

4.1.4 Inductively Coupled Spectroscopy – Mass Spectroscopy

SRNL performed three analyses of the Tank 48H slurry for actinides (with one of the samples analyzed at two different dilutions). The replicate samples showed excellent precision (Table 7). ²⁴⁰Pu, ²⁴¹Am, ²⁴³Am, and ²⁴⁴Cm are all less than the detection limits. ²³⁸Pu can not be detected by this analytical method.

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Table 7 – Actinide Results for the Slurry Sample

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|---------------|------------------------------------------|------------------------|
| Np-237, mg/L | 2.22E-01 | 8.09E-03 |
| Pu-239, mg/L | 3.33E-02 | 5.55E-03 |
| U-233, mg/L | 6.13E-02 | 7.39E-03 |
| U-234, mg/L | 3.25E-01 | 1.94E-02 |
| U-235, mg/L | 6.53E-01 | 4.43E-02 |
| U-236, mg/L | 1.44E-01 | 4.85E-03 |
| U-238, mg/L | 4.12E+00 | 2.24E-01 |
| U Total, mg/L | 5.31E+00 | 2.89E-01 |

We analyzed a slurry sample for metals (Ag, Pd, Rh, and Ru). The elemental results of the Tank 48H slurry sample are presented in Table 8.

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Table 8 – ICP-MS Catalytic Components Present in the Tank 48H Slurry Sample

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|----------------|------------------------------------------|------------------------|
| Total Ag, mg/L | 1.88E-02 | 2.12E-03 |
| Total Pd, mg/L | 9.28E-02 | 6.78E-03 |
| Total Rh, mg/L | 1.53E-01 | 1.61E-02 |
| Total Ru, mg/L | 3.80E-01 | 8.43E-03 |

4.1.5 Atomic Absorption Spectroscopy

Personnel determined the elemental composition of the digested slurry by AA for K only. The element potassium is present in $2,650 \pm 184$ mg/L (0.068 ± 0.0047 M).

4.2 FILTRATE SAMPLE RESULTS

ADS personnel analyzed the filtrate in duplicate for the Tank 48H sample. 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, multiply filtrate results by 0.982 to convert them 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 unslurried tank but has not been filtered. It is expected that supernate results from previous samples would have slightly higher insoluble solids concentrations.

4.2.1 Anion Analysis by Ion Chromatography

Table 9 and Table 10 contain the measured values of the Tank 48H filtered sample. Table 9 includes the measured anion concentrations for the tank sample. Nitrate and nitrite concentrations are 0.216 M and 0.465 M respectively.

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Table 9 – Anion Results for the Filtered Tank 48H sample, mg/L

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|---------------------------------------------|------------------------------------------|------------------------|
| F ⁻ | <20 | NA |
| CO ₂ H ⁻ | 680.0 | 428 |
| Cl ⁻ | 370 | 183 |
| NO ₂ ⁻ | 30,400 | 1,800 |
| Br ⁻ | <100 | NA |
| NO ₃ ⁻ | 18,750 | 926 |
| PO ₄ ³⁻ | 916 | 222 |
| SO ₄ ²⁻ | 528 | 210 |
| C ₂ O ₄ ²⁻ | 1613 | 116 |

For comparison, Table 10 contains the measured values from this Tank 48H sample, but also other recent measurements.

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Table 10 – Comparison of Filtrate Anion Values, Molarity

| Anion | HTF-E-04-049 and HTF-E-0050 Aug 04 | HTF-E-03-127 Sept 03 | HTF-E-03-73 June 03 |
|---------------------------------------------|------------------------------------------|-------------------------|------------------------|
| NO ₃ ⁻ | 0.304 | 0.216 | 0.22 |
| NO ₂ ⁻ | 0.649 | 0.465 | 0.45 |
| SO ₄ ²⁻ | 0.00643 | 0.00285 | 0.005 |
| Cl ⁻ | 0.0127 | 0.00351 | 0.005 |
| HCO ₂ ⁻ | 0.0192 | 0.0096 | 0.009 |
| PO ₄ ³⁻ | 0.0102 | 0.00542 | 0.016 |
| C ₂ O ₄ ²⁻ | 0.0183 | 0.0123 | 0.023 |

4.2.2 High Pressure Liquid Chromatography

ADS completed analysis of the Tank 48H filtrate for tetraphenylborate and 14 of its common decomposition products. Only phenol at 526 ± 2.47 mg/L occurs above the detection limit. Nitrobenzene, Nitrosobenzene, 4-phenylphenol, 2-phenylphenol, diphenylamine, biphenyl, o-terphenyl, m-terphenyl, p-terphenyl, TPB, 3PB, 2PB, and 1PB all fell below detection limits (10 mg/L).

4.2.3 Wet Chemistry

ADS personnel analyzed the filtrate samples using titration methods, carbon analyses and density measurements. The total base is 2.04 ± 0.071 M, the free hydroxide is 1.16 ± 0.007 M, other base is 0.185 ± 0.004 M, and carbonate is 0.492 ± 0.001 M.

The free hydroxide concentration (1.16 M) has increased since the September 2003 sample (0.790 M) due to the addition of sodium hydroxide in October 2003.

The shielded cells technicians measured the Tank 48H filtrate density as 1.164 ± 0.001 g/mL.

The total solids concentration of the Tank 48H filtrate was $17.68\% \pm 0.14$ wt %. The total solids concentration of the Sept 2003 filtrate was 16.28 ± 0.269 wt %.

4.2.4 Radionuclide Composition

ADS personnel analyzed the filtered sample using radio-analytical methods. Cesium (^{137}Cs) is the major radioactive analyte in the filtrate at a concentration of $5.22 \text{ E} +05$ nCi/g. Table 11 summarizes the radionuclide concentrations. Other isotopes were not analyzed. Table 12 provides a comparison with previous sample results.

Table 11 – Tank 48H Filtrate Radiation Chemistry Data

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|----------------------------|------------------------------------------|---------------------------|
| ^{137}Cs (dpm/mL) | 2.57E+07 | 6.01E+04 |
| Gross Alpha (dpm/mL) | 2.00E+03 | 3.89E+02 |

Table 12 – Radionuclide Comparison with Previous Samples

| Sample ID Radiochemical Analysis | Sample HTF-E-04-049 and HTF-E-0050 | HTF-E-03-127 3-L sample Sept. 2003 | HTF-E-03-73 June 2003 | Previous Sample Measurements |
|----------------------------------------|------------------------------------------|------------------------------------------|--------------------------|---------------------------------|
| ^{137}Cs (nCi/g) | 9.95 E+03 | 1.08 E+04 | 1.12 E+04 | 1.50 E+04 [Ref. 6] |
| ^{90}Sr (mg/L) | Not Measured | 7.12 E-06 | 8.59E-06 | <2.44 E-06 [Ref. 8] |
| ^{99}Tc (mg/L) | Not Measured | 2.26 E+00 | 1.15 E+00 | 1.85 E+00 [Ref. 8] |

4.2.5 Atomic Absorption Spectroscopy

ADS analyzed the filtered samples for potassium using atomic absorption spectroscopy. The K concentration of the Tank 48H filtrate was 255 ± 24.7 mg/L.

4.2.6 Inductively Coupled Spectroscopy – Emission Spectroscopy

ADS determined the elemental composition of the filtrate by the ICP-ES method. The major constituents found in the filtrate included Na, Al, and B. The element sodium is present in the highest concentration at 2.81 M. Aluminum and boron are present in appreciable levels. Elements measured below instrument detection limits include Ag, Ba, Cd, Ce, Fe, Gd, La, Li, Mg, Mn, Ni, Pb, Sn, Ti, U, and Zr. Table 13 provides the elemental results of the Tank 48H filtrate samples. Table 14 provides a comparison with historical data.

Table 13 – Tank 48H Filtrate ICP-ES Data

| Analyte | Sample HTF-E-04-049 and HTF-E-0050 | 1 σ Uncertainty |
|----------|------------------------------------------|---------------------------|
| B, mg/L | 412 | 12.7 |
| Cu, mg/L | 1.014 | 0.221 |
| K, mg/L | 372.5 | 53.0 |
| Na, mg/L | 68,800 | 2,263 |
| Si, mg/L | 4.675 | 0.361 |

Table 14 – Major Elemental Constituents Present in the Tank 48H Filtrate Sample

| Sample ID Analysis | Units | Sample HTF-E-04-049 and HTF-E-0050 | HTF-E-03-127 3-L sample | HTF-E-03-73 Tk 48-2L-1 | 1998 Analyses ⁵ |
|-----------------------|-------|------------------------------------------|----------------------------|---------------------------|----------------------------|
| ICP-ES | | | | | |
| Al | M | Not Measured | 0.0808 | 0.074 | 0.082 |
| B | M | 0.038 | 0.0410 | 0.041 | 0.034 |
| Na | M | 2.99 | 3.012 | 2.81 | 2.56 |
| S | M | Not Measured | 0.00872 | 0.01 | NM |
| K | M | 0.010 | 0.00633 | 0.01 | NM |
| AA | | | | | |
| K | M | 0.0065 | 0.00550 | 0.005 | 0.0004 |
| C s | M | Not Measured | 2.88E-06 | 3.61E-06 | NM |

4.3 IN-CELL SLURRY SAMPLE RESULTS

Personnel performed analyses in the cells, because of the high radiation dose of the sample. Solids analyses, slurry titration and density analyses were completed in duplicate. These analytical data are summarized together with the one standard deviation (1 σ) uncertainty.

4.3.1 In-cell Solids Analyses

Based on the average of three analyses, the slurry contained total solids of $19.08 \pm 0.08\%$ and the filtrate contained dissolved solids of $17.68 \pm 0.14\%$. We calculate the value for the insoluble solids (1.69 ± 0.22 wt %) using the following formula.⁶

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

$$\text{Insoluble Solids} = 19.08 - (100 - 19.08) * 17.68/100 / (1 - 17.68/100) = 1.69$$

Based on this calculation, the insoluble solids measurement was significantly lower than expected results based on previous analyses (2.3%).⁷ The insoluble solids measurement has a range of 1.47 wt% to 1.91 wt% based on the calculated uncertainties. This overlaps with the sum of the measured KTPB plus MST concentration which has a range of 1.86 wt % to 2.34 wt %.

4.3.2 In-cell Density

Technicians determined density from the average of duplicate measurements of the Tank 48H slurry. Based on these measurements, the density of the Tank 48H slurry equals 1.16 ± 0.009 g/mL.

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, one can obtain an understanding of the decomposition rate for the tetraphenylborate in Tank 48H.

5.1 TANK 48H ION BALANCE

The sum of the major cations exceeds the sum of the major anions by ~8%, well within the accuracy typically experienced for such analyses. Table 15 sums the anions and cations using the filtrate analyses for the major components.

Table 15 – Tank 48H Filtrate Anion/Cation Balance

| Analyte | Moles ions | 1 σ Uncertainty, moles | Method |
|---------------------------------------------|-------------|-------------------------------|--------------------|
| AlO ₂ ⁻ | 0.083 | 0.0000 | ICP-ES |
| BO ₃ ³⁻ | 0.286 | 0.0005 | ICP-ES |
| C ₂ O ₄ ²⁻ | 0.037 | 0.0013 | IC |
| Cl ⁻ | 0.010 | 0.0026 | IC |
| COOH ⁻ | 0.015 | 0.0048 | IC |
| CO ₃ ²⁻ | 0.932 | 0.0007 | Titration |
| NO ₂ ⁻ | 0.661 | 0.0320 | IC |
| NO ₃ ⁻ | 0.302 | 0.0124 | IC |
| OH ⁻ | 1.155 | 0.0071 | Titration |
| PO ₄ ³⁻ | 0.029 | 0.0012 | IC |
| SO ₄ ²⁻ | 0.011 | 0.0011 | IC |
| Total Anions | 3.52 | 0.064 | Calculation |
| K ⁺ | 0.007 | 0.0047 | AA |
| Na ⁺ | 3.259 | 0.0286 | ICP-ES |
| Total Cations | 3.27 | 0.033 | Calculation |

5.2 TANK 48H SOLUBLE POTASSIUM AND CESIUM

The tetraphenylborate in Tank 48H continues to slowly decompose. As the tetraphenylborate decomposes, cesium and potassium are released from the insoluble solids and return to solution. As a result, the concentrations of cesium and potassium continue to increase over time. Data exist for the soluble cesium and potassium concentrations in the filtrate for the last eight years. Figure 1 and Figure 2 provide the measured values, with 10% error bars. Note that we corrected the data for changes in Tank 48H volume (normalizing to the volume as of 240,000, the volume on 11-3-96) and ¹³⁷Cs radioactive decay.

Based on the potassium data, the tetraphenylborate apparently continues to degrade at the same rate as experienced since 1996. The potassium data, shown on Figure 1, fits a linear curve ($r^2=0.993$) almost as well as a 2nd order polynomial ($r^2 = 0.995$). The ¹³⁷Cs data, shown on Figure 2, is no longer following the straight line. A better fit of the ¹³⁷Cs data fit is a 2nd order polynomial curve ($r^2 = 0.993$) better than a linear curve ($r^2 = 0.90$). The drop in ¹³⁷Cs concentration could be caused by establishment of a new equilibrium due to decomposition or to the increase in ionic strength causing precipitation of cesium (due to the depletion of hydroxide followed by addition of sodium hydroxide to control corrosion).

Figure 1 – K Soluble Concentration Increase Due to KTPB Decomposition
(K Data volume corrected to 240,000 gallons in Tank 48H)

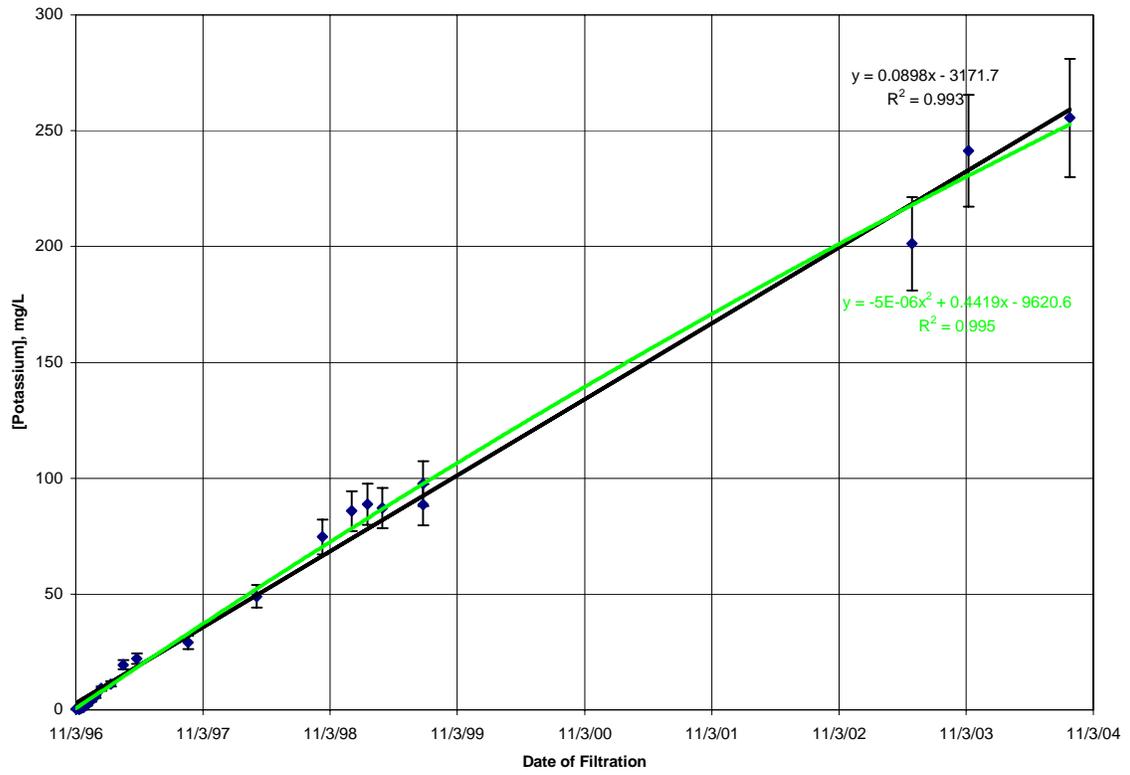
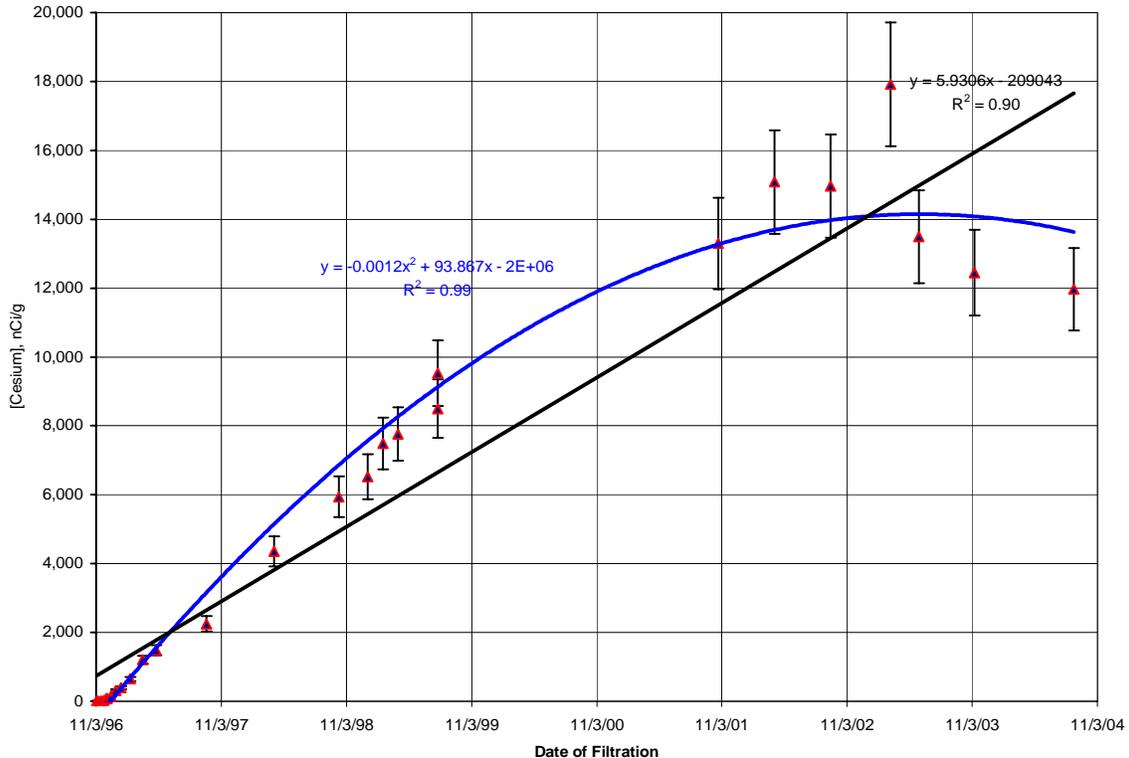


Figure 2 – ¹³⁷Cs Concentration Increase Due to CsTPB Decomposition

(Cs Data was corrected to 240,000 gal and adjusted for radioactive decay since 1996)



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 of 50 wt % sodium hydroxide on October 30, 2003.

The tetraphenylborate in Tank 48H continues to slowly decompose at a rate of approximately 0.0898 mg K/L/day or 1.3 % TPB decomposition per year. This is consistent with the measured decomposition over the last eight years.

The analyses requested were primarily designed to support an in-tank catalyzed hydrolysis process and the actual waste testing of the hydrolysis and aggregation process alternatives. The Tank 48H sample contains 9.28E-02±6.78E-03 mg/L Pd, 2.97±0.156 mg/L Cu and 20.4±0.86 mg/L Hg. Additional analyses may be required for alternative processes such as Aggregation.

Data collected during sampling is summarized in Appendix A. The Tank 48H analytical results are 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 Debbie Burckhalter, Nan Stanley and Dee Wheeler 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, Jon Kuhns, Kim Mitchell, 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 technicians, Kim Wyszynski and Liz Coleman, 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, especially Rick Fowler, Steve Strohmeier, Ben Dean, Delane Maxwell, Dennis Conrad, and Bernice Rogers for their technical support in helping us accomplish this task.

8.0 REFERENCES

- ¹ T. B. Peters, D. P. Lambert, M. E. Smith, and S. D. Fink, "Task Technical and Quality Assurance Plan for Destruction of Tetraphenylborate in Tank 48H", WSRC-RP-2003-00396, Rev. 1, June 10, 2003.
- ² Tank 48H 10 Liter Sample Analyses, SP-TTR-2004-00020, August 4, 2004.
- ³ D. P. Lambert, T. B. Peters, M. S. Hay, S. D. Fink, "Task Technical and Quality Assurance Plan for Analysis and Acceptance of Tank 48H Sample", WSRC-RP-2004-00598, Revision 0, August 2004.
- ⁴ W. B. Dean, "Tank 48H Dissolution Sampling Plan", CBU-SPT-2003-00104, Rev. 0, May 15, 2003.
- ⁵ D. T. Hobbs, et al., "Radioactive Testing Results in Support of the In-Tank Precipitation Facility", WSRC-TR-98-00070, Rev. 0, April 2, 1998.
- ⁶ C. J. Coleman, D. D. Walker, "Statistical Study of Weight % Insoluble Methods for In-Tank Precipitation (ITP) Cold Run Support", WSRC-TR-92-256, December 9, 1992.
- ⁷ D. D. Walker, W. T. Boyce, C. J. Coleman, D. P. DiPrete, T. B. Edwards, A. A. Ekechuckwu, C. W. Hsu, S. F. Peterson, L. L. Tovo, and M. J. Whitaker, "Tank 48H Waste Composition and Results of Investigations of Analytical Methods", WSRC-TR-97-0063, April 2, 1997.

Appendix A
Data Collected during Sampling

Figure A-1 – Temperature and Pump Data during Sampling

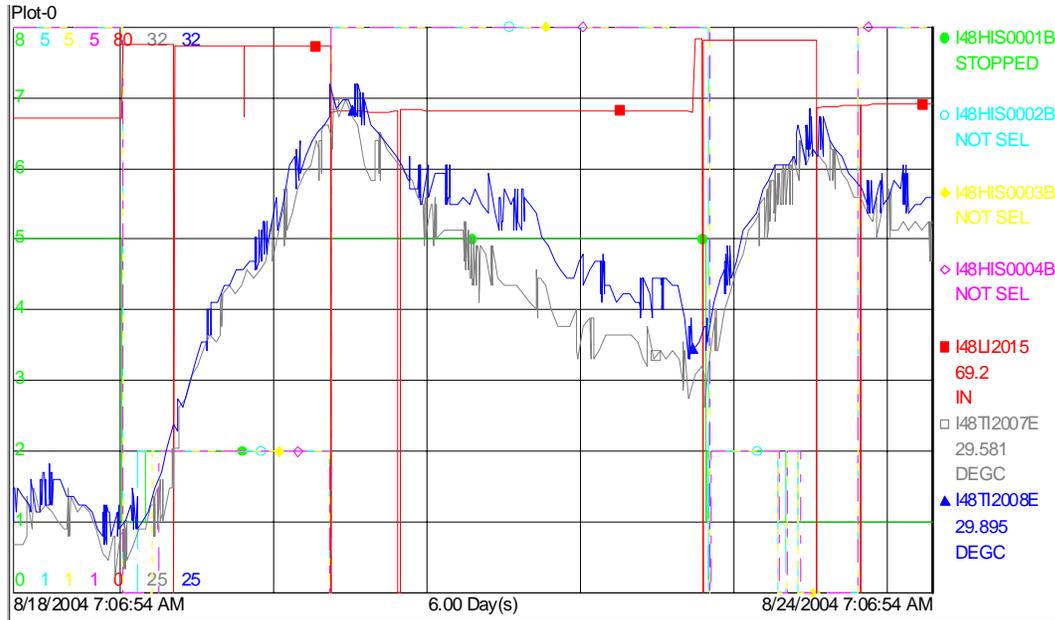


Table A-1 – Pump Start and Stop Time for Samples HTF-E-04-049 and 04-050

| Pump | Start time | Stop Time | Duration #1, hrs | Downtime, hrs |
|------|---------------|---------------|------------------|---------------|
| B1 | 8/19/04 2:51 | 8/20/04 8:42 | 30 | 60 |
| | 8/22/04 19:58 | 8/22/04 7:01 | 11 | 13 |
| | 8-23-04 20:08 | 8-23-04 21:58 | 2 | |
| B4 | 8/19/04 3:53 | 8/20/04 7:48 | 28 | 60 |
| | 8/22/04 20:13 | 8/22/04 7:30 | 11 | 13 |
| | 8-23-04 20:13 | 8-23-04 22:03 | 2 | |
| V1 | 8/19/04 5:08 | 8/20/04 8:15 | 27 | 60 |
| | 8/22/04 20:27 | 8/22/04 7:30 | 11 | 13 |
| | 8-23-04 20:21 | 8-23-04 22:03 | 2 | |
| V2 | 8/19/04 6:00 | 8/20/04 8:20 | 26 | 60 |
| | 8/22/04 20:27 | 8/22/04 7:30 | 11 | 13 |
| | 8-23-04 20:28 | 8-23-04 22:28 | 2 | |

The tank level increased from 67.2 to 69.1 inches as a result of the sampling. This is a volume increase of 6,670 gallons. The pumps operated for a total time of 163 hours for a calculated inleakage of 41 gallons per hour.

Table A-3 -- Data collected During June sampling

| Pump | Bearing Water flow | |
|-------|--------------------|------------|
| | mL/min | gallons/hr |
| V2 | Not recorded | |
| V1 | 150 | 2.4 |
| B4 | 150 | 2.4 |
| B1 | 200 | 3.2 |
| Total | >500 | >8.0 |

Appendix B – Tank 48H Sample Results Summary (Results > 1 mg/L)

| Analyte | Slurry | Filtrate | Source |
|-------------------------|---------|----------|-------------|
| Volume, gal | 239,031 | NM | Tank 48H |
| Density, mg/L | 1.162 | 1.164 | Density |
| Total Solids, wt % | 19.08% | 17.68% | Solids |
| Total Insolubles | 1.69% | NM | Calculation |
| KTPB, wt % | 1.95% | <0.001% | HPLC |
| MST solids, wt % | 0.15% | NM | ICP-ES |
| Anions | | | |
| Free Hydroxide, M | NM | 1.16 | Titration |
| Carbonate, M | NM | 0.492 | Titration |
| Nitrite, M | NM | 0.649 | Anion |
| Nitrate, M | NM | 0.304 | Anion |
| Oxalate, mg/L | NM | 1,613 | Anion |
| Formate, mg/L | NM | 680 | Anion |
| Chloride, mg/L | NM | 370.25 | Anion |
| Phosphate, mg/L | NM | 915.75 | Anion |
| Sulfate, mg/L | NM | 527.5 | Anion |
| Organic Species | | | |
| TPB Anion, mg/L | 20,100 | <10 | HPLC |
| 3PB, mg/L | 162 | <10 | HPLC |
| 2PB, mg/L | 123 | <10 | HPLC |
| PBA, mg/L | 120 | <10 | HPLC |
| PHENOL, mg/L | 735 | 529 | HPLC |
| Nitrobenzene, mg/L | <50 | <10 | HPLC |
| Nitrososbezene, mg/L | <50 | <10 | HPLC |
| 4phenylphenol, mg/L | <50 | <10 | HPLC |
| 2phenylphenol, mg/L | <50 | <10 | HPLC |
| Diphenylamine, mg/L | <50 | <10 | HPLC |
| Biphenyl, mg/L | 384 | <10 | HPLC |
| o-terphenyl, mg/L | <50 | <10 | HPLC |
| m-terphenyl, mg/L | <50 | <10 | HPLC |
| p-terphenyl, mg/L | <50 | <10 | HPLC |

| Metals | Slurry | Filtrate | Source |
|------------------|---------------|-----------------|---------------|
| Potassium, M | 0.068 | 0.0061 | AA |
| Hg, mg/L | 22.0 | NM | AA |
| Sodium, M | 3.26 | 2.99 | ICP-ES |
| Potassium, M | 0.061 | 0.0095 | ICP-ES |
| Aluminum, mg/L | 2,242 | NM | ICP-ES |
| Boron, mg/L | 1,030 | 412 | ICP-ES |
| Sulfur, mg/L | 245.08 | NM | ICP-ES |
| Phosphorus, mg/L | 206.75 | NM | ICP-ES |
| Silicon, mg/L | 106.28 | 4.675 | ICP-ES |
| Iron, mg/L | 43.44 | <0.709 | ICP-ES |
| Chromium, mg/L | 51.11 | NM | ICP-ES |
| Magnesium, mg/L | 18.53 | NM | ICP-ES |
| Calcium, mg/L | 21.43 | NM | ICP-ES |
| Molybdenum, mg/L | 13.30 | NM | ICP-ES |
| Antimony, mg/L | 11.50 | NM | ICP-ES |
| Zinc, mg/L | 11.91 | NM | ICP-ES |
| Manganese, mg/L | 6.38 | NM | ICP-ES |
| Strontium, mg/L | 5.29 | NM | ICP-ES |
| Barium, mg/L | 2.52 | NM | ICP-ES |
| Copper, mg/L | 2.97 | 1.014 | ICP-ES |
| Total Ag, mg/L | 1.88E-02 | 2.12E-03 | ICP-MS |
| Total Pd, mg/L | 9.28E-02 | 6.78E-03 | ICP-MS |
| Total Rh, mg/L | 1.53E-01 | 1.61E-02 | ICP-MS |
| Total Ru, mg/L | 3.80E-01 | 8.43E-03 | ICP-MS |
| Total Cd, mg/L | 2.16E-02 | 1.57E-02 | ICP-MS |
| Total Hg, mg/L | 4.45E+00 | 6.73E-02 | ICP-MS |
| Np-237, mg/L | 2.22E-01 | 8.09E-03 | ICP-MS |
| Pu-239, mg/L | 3.33E-02 | 5.55E-03 | ICP-MS |
| U-233, mg/L | 6.13E-02 | 7.39E-03 | ICP-MS |
| U-234, mg/L | 3.25E-01 | 1.94E-02 | ICP-MS |
| U-235, mg/L | 6.53E-01 | 4.43E-02 | ICP-MS |
| U-236, mg/L | 1.44E-01 | 4.85E-03 | ICP-MS |
| U-238, mg/L | 4.12E+00 | 2.24E-01 | ICP-MS |
| U Total, mg/L | 5.31E+00 | 2.89E-01 | ICP-MS |

Distribution**WSRC-TR-2004-00514, Rev 1**

| | | | | | | | |
|-------|---------------|------------------|--------|----------|----------------|--------------------------|--------|
| R. A. | Adams | 241-162H, Rm. 4 | (E) | D. B. | Little | 703-H, Rm. 3 | (E) |
| J. W. | Barber | 704-2H, Rm. 197 | (E) | S. R. | Loflin | 773-41A, Rm. 223 | (E) |
| J. L. | Barnes | 704-S, Rm. 19 | (E) | N. P. | Malik | 704-26F, Rm. 11 | (E) |
| M. J. | Barnes | 773-A, Rm. B-132 | (E) | J. C. | Marra | 773-42A, Rm. 173 | (E) |
| W. M. | Barnes | 704-56H, Rm. 164 | (E) | D. J. | Martin | 703-H, Rm. 84 | (E) |
| S. M. | Blanco | 766-H, Rm. 2434 | (E) | K. B. | Martin | 773-42A, Rm. 14 | (E) |
| L. R. | Bragg | 766-H, Rm. 2434 | (E) | C. J. | Martino | 735-11A, Rm. 121 | (E) |
| T. E. | Britt | 742-4G, Rm. 3 | (E) | | | | |
| H. L. | Bui | 742-4G, Rm. 3 | (E) | G. J. | Matis | 766-H, Rm. 1066F | (E) |
| S. G. | Campbell | 703-H, Rm. 107 | (E) | D. | Maxwell | 766-H, Rm. 2231 | (E) |
| L. | Carey | 766-H, Rm. 2005A | (E) | D. J. | McCabe | 73-42-A, Rm. 153 | (E) |
| J. T. | Carter | 703-H, Rm. 122 | (E) | J. W. | McCullough | 766-H, Rm. 2411 | (E) |
| W. D. | Clark | 766-H, Rm. 2412 | (E) | L. T. | McGuire | 766-H, Rm. 2441 | (E) |
| S. L. | Clifford | 766-H, Rm. 2443 | (E) | M. S. | Miller | 772-7B, Rm. 6 | (E) |
| J. J. | Connelly | 773-41A, Rm. 231 | (E) | C. A. | Nash | 773-42A, Rm. 182 | (E) |
| D. T. | Conrad | 766-H, Rm. 2007 | (E) | L. M. | Nelson | 773-43A, Rm. 222 | (E) |
| D. R. | Cox | 730-2B, Rm. 118 | (E) | M. A. | Norato | 704-27S, Rm. 6 | (E) |
| A. D. | Cozzi | 773-43A, Rm. 218 | (E) | M. R. | Norton | 66-H, Rm. 2002 | (E) |
| C. L. | Crawford | 773-41A, Rm. 180 | (E) | J. E. | Occhipinti | 704-S, Rm. 18 | (E) |
| D. A. | Crowley | 773-A, Rm. A-262 | (E) | L. D. | Olson | 703-H, Rm. 5 | (E) |
| N. R. | Davis | 766-H, Rm. 1006 | (E) | L. M. | Papouchado | 773-A, Rm. A-263 | (P) |
| W. B. | Dean | 766-H, Rm. 2243 | (E) | T. B. | Peters | 773-42A, Rm. 128 | (E) |
| V. G. | Dickert | 703-H, Rm. 4 | (E) | J. A. | Pike | 703-H, Rm. 99 | (E) |
| C. L. | Donahue | 241-162H, Rm. 6 | (E) | M. R. | Poirier | 773-42A, Rm. 123 | (E) |
| M. D. | Drumm | 766-H, Rm. 2050 | (E) | S. H. | Reboul | 703-H, Rm. 84 | (E) |
| M. C. | Duff | 773-43A, Rm. 217 | (E) | T. R. | Reynolds | 704-S, Rm. 65 | (E) |
| C. R. | Dyer | 766-H, Rm. 2426 | (E) | M.A. | Rios-Armstrong | 766-H, Rm. 2054 | (E) |
| R. E. | Eibling | 999-W, Rm. 335 | (E) | S. J. | Robertson | 766-H, Rm. 2500 | (P) |
| G. N. | Eide | 241-121H, Rm. 6 | (E) | B. C. | Rogers | 766-H, Rm. 2008 | (E) |
| H. H. | Elder | 703-H, Rm. 95 | (E) | R. A. | Runnels | 766-H, Rm. 2011 | (E) |
| S. D. | Fink | 773-A, Rm. B-112 | (E, P) | P. J. | Ryan | 704-61S, Rm. 6 | (E) |
| F. F. | Fondeur | 773-A, Rm. B-124 | (E) | E. | Saldivar | 766-H, Rm. 2004 | (E) |
| R. C. | Fowler | 703-H, Rm. 98 | (E) | S. C. | Shah | 766-H, Rm. 2037 | (E) |
| L. M. | Fox | 703-H, Rm. 3 | (E) | D. C. | Sherburne | 704-S, Rm. 18 | (E) |
| M.W. | Geeting | 766-H, Rm. 2035 | (E) | T. J. | Spears | 766-H, Rm. 2015 | (E) |
| B. A. | Gifford | 766-H, Rm. 1066D | (E) | R. H. | Spires | 766-H, Rm. 2003 | (E) |
| A. P. | Giordano | 703-H, Rm. 79 | (E) | M. E. | Stallings | 773-A, Rm. B-117 | (E) |
| J. C. | Griffin | 773-A, Rm. A-231 | (E) | W. E. | Stevens | 773-A, Rm. A-261 | (E) |
| B. A. | Hamm | 766-H, Rm. 2237 | (E) | S. J. | Strohmeier | 766-H, Rm. 2022 | (E) |
| H. D. | Harmon | 766-H, Rm. 2014 | (P) | S. G. | Subosits | 766-H, Rm. 2052 | (E) |
| K. D. | Harp | 755-H, Rm. 1066B | (E) | P. C. | Suggs | 766-H, Rm. 2436 | (E) |
| E. W. | Harrison | 766-H, Rm. 2034 | (E) | G. A. | Taylor | 703-H, Rm. 96 | (E) |
| K. A. | Hauer | 703-H, Rm. 11 | (E) | S. A. | Thomas | 766-H, Rm. 2016 | (E) |
| D. T. | Herman | 735-11A, Rm. 104 | (E) | P. J. | Valenti | 730-4B, Rm. 2062 | (E) |
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| R. N. | Hinds | 766-H, Rm. 2430 | (E) | D. D. | Walker | 773-A, Rm. B-124 | (E) |
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