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**Permanent**

# Analysis of the Tank 5F Feed and Bleed Residual Solids

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July 8, 2011

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

**Prepared for the U.S. Department of Energy Under Contract Number DE-AC09-08SR22470**

SRNL
SAVANNAH RIVER NATIONAL LABORATORY

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## REVIEWS AND APPROVALS

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# LIST OF ACRONYMS

| | |
|---|---|
| LWO | Liquid Waste Organization |
| SMP | Submersible Mixer Pumps |
| SRNL | Savannah River National Laboratory |
| SRR | Savannah River Remediation |
| SRS | Savannah River Site |

## 1.0 SUMMARY

Savannah River Remediation (SRR) is preparing Tank 5F for closure. As part of Tank 5F Closure Mechanical Cleaning, SRR conducted a "Feed and Bleed" process in Tank 5F. Following this "Feed and Bleed" Mechanical Cleaning in Tank 5F, SRR collected two tank heel samples (referred to as sample 1 and sample 2) under Riser 5 to determine the composition of the material remaining in the tanks. This document describes sample analysis results.

The conclusions from this analysis follow.

- The anions measured all had a concentration less than 250 mg/kg, except for oxalate, which had a concentration of 2100 – 2400 mg/kg.
- The measured cations with the highest concentration were iron (432,000 – 519,000 mg/kg), nickel (54,600 – 69,300 mg/kg), and manganese (35,200 – 42,100 mg/kg). All other cations measured less than 13,000 mg/kg.
- The radionuclides present in the highest concentration are $^{90}$Sr (3.0 x $10^{10}$dpm/g), $^{137}$Cs (6.8 x $10^{8}$ dpm/g), and $^{241}$Am (1.4 x $10^{8}$ - 1.8 x $10^{8}$ dpm/g).
- The particle size analysis shows a large fraction of particles greater than 100 μ.

## 2.0 INTRODUCTION

Savannah River Remediation (SRR) is preparing Tank 5F for closure. Part of tank closure is mechanical sludge removal.

Following Phase II Mechanical Cleaning (i.e., "Feed and Bleed") in Tank 5F, SRR collected two tank heel samples (referred to as sample 1 and sample 2) on April 8, 2010 from the solids accumulation under Riser 5 to determine the composition of the material remaining in the tanks. These samples were collected using a scraper sampling tool that collected approximately 20 grams of material per scrape. This document describes sample analysis results. The results include anions, cations, $^{60}$Co, $^{90}$Sr, $^{99}$Tc, $^{137}$Cs, $^{154}$Eu, $^{233}$U, $^{234}$U, $^{235}$U, $^{236}$U, $^{238}$U, $^{237}$Np, $^{238}$Pu, $^{239}$Pu, $^{239/240}$Pu, $^{240}$Pu $^{241}$Pu, $^{244}$Pu, $^{241}$Am, $^{243}$Am, $^{242m}$Am, $^{243}$Cm, $^{245}$Cm, $^{247}$Cm, $^{249}$Cf, $^{251}$Cf, $^{242}$Cm, $^{244}$Cm, and particle size. The purpose of the document is to provide early characterization data for use in the evaluation of the heel removal process in Tank 5F and to support planning for future heel removal efforts.[1,2]

## 3.0 ANALYSES

SRNL received two solid replicate scrape samples from under riser 5 in Tank 5F (see Figure 1). The sample contained no free liquid. SRNL prepared four subsamples from each of the samples. In addition, we prepared one blank sample (using the digestion reagents only). From each subsample, personnel took a sample for digestion by aqua regia, a sample for digestion by sodium peroxide fusion, and a sample for contact wit deionized water. The aqua regia digestion consists of heating the sample at 115 °C for two hours with a 3:1 mixture of concentrated hydrochloric acid and concentrated nitric acid in a sealed Teflon pressure. The sodium peroxide fusion consists of heating the sample at 675 °C for 10 minutes with sodium peroxide in a zirconium crucible followed by dissolution of the fusion residue with nitric acid. The water

contact sample was prepared by contacting approximately 0.5 grams of sample with approximately 25 grams of deionized water.

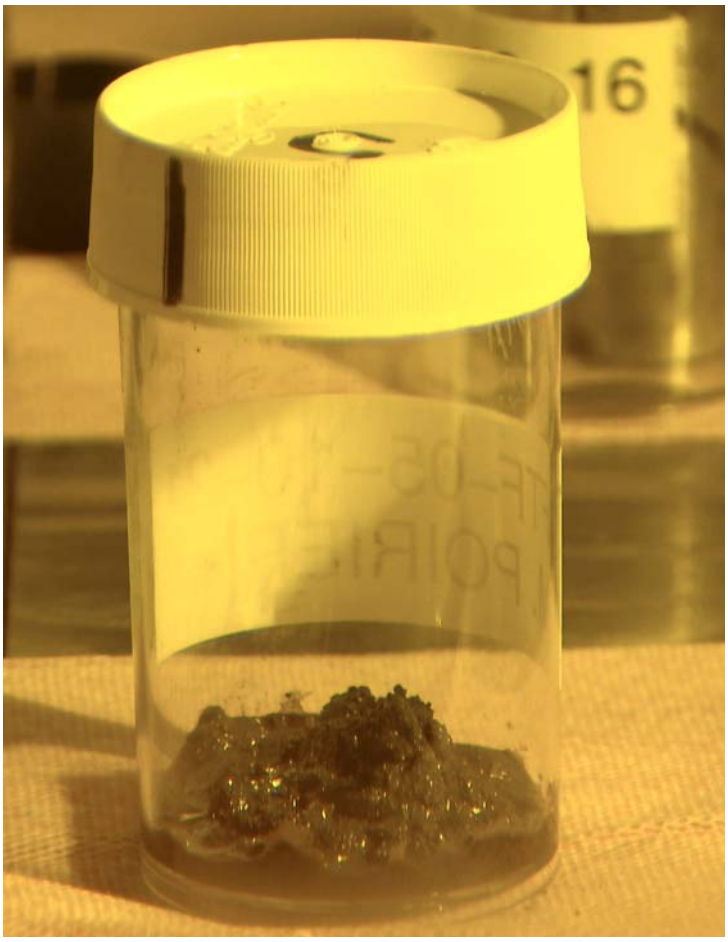


**Figure 1. Tank 5F Feed and Bleed sample**

The samples were analyzed for cations (ICPES), anions (IC), mercury (by Cold Vapor Atomic Absorption, CVAA, spectroscopy), and radionuclides (ICPMS, liquid scintillation counting, gamma scan, PuTTA, $^{90}$Sr, $^{99}$Tc, and Am/Cm methods).

## 4.0 RESULTS

Table 1 shows the anion composition of sample 1, and Table 2 shows the anion composition of sample 2. The samples were prepared by performing a water leach of the solids. The concentrations of all species are less than 250 μg/g, except for oxalate that had a concentration of 2100 - 2400 μg/g. Because the solid particles were contacted with water rather than nitric acid, the actual oxalate concentration in the sludge may be higher.

**Table 1. Sample 1 Anion Analysis**

| Analyte | 300271114 Blank (µg/g) | 300271106 Prelim 1 (µg/g) | 300271107 Prelim 2 (µg/g) | 300271108 Prelim 3 (µg/g) | 300271109 Prelim 4 (µg/g) | Average (µg/g) |
|---|---|---|---|---|---|---|
| Fluoride ($F^-$) | <50 | <50 | <45 | <48 | <50 | <48 |
| Formate ($HCOO^-$) | <50 | 50 | 45 | 48 | 50 | 48 |
| Chloride ($Cl^-$) | 100 | 100 | 90 | 97 | 100 | 97 |
| Nitrite ($NO_2^-$) | <50 | <50 | <45 | <48 | <50 | <48 |
| Nitrate ($NO_3^-$) | 100 | 150 | 90 | 97 | 100 | 110 |
| Phosphate ($PO_4^{3-}$) | <250 | <250 | <220 | <240 | <250 | <240 |
| Sulfate ($SO_4^{2-}$) | 100 | 150 | 180 | 150 | 150 | 160 |
| Oxalate ($C_2O_4^{2-}$) | <50 | 2300 | 2100 | 2100 | 2100 | 2150 |
| Bromide ($Br^-$) | <50 | <50 | <45 | <48 | <50 | <48 |

**Table 2. Sample 2 Anion Analysis**

| Analyte | 300271114 Blank (µg/g) | 300271110 Prelim 5 (µg/g) | 300271111 Prelim 6 (µg/g) | 30027112 2 Prelim 7 (µg/g) | 300271113 Prelim 8 (µg/g) | Average (µg/g) |
|---|---|---|---|---|---|---|
| Fluoride ($F^-$) | <50 | <42 | <41 | <41 | <42 | <42 |
| Formate ($HCOO^-$) | <50 | 42 | 41 | 41 | 42 | 42 |
| Chloride ($Cl^-$) | 100 | 83 | 81 | 83 | 84 | 83 |
| Nitrite ($NO_2^-$) | <50 | <42 | <41 | <41 | <42 | <42 |
| Nitrate ($NO_3^-$) | 100 | 83 | 81 | 83 | 84 | 83 |
| Phosphate ($PO_4^{3-}$) | <250 | <210 | <200 | <210 | <210 | <210 |
| Sulfate ($SO_4^{2-}$) | 100 | 250 | 240 | 250 | 250 | 250 |
| Oxalate ($C_2O_4^{2-}$) | <50 | 2300 | 2400 | 2400 | 2400 | 2400 |
| Bromide ($Br^-$) | <50 | <42 | <41 | <41 | <42 | <41 |

Table 3 and Table 4 show the cation composition for sample 1 and sample 2, respectively. The samples were prepared by performing either an aqua regia digestion or a peroxide fusion digestion. The species with the largest concentration are iron (432,000 – 519,000 mg/kg), nickel (54,600 – 69,300 mg/kg), manganese (35,200 – 42,100 mg/kg), uranium (9,160 – 12,100 mg/kg), silicon (7,790 – 9,580 mg/kg), aluminum (4,520 – 5,510 mg/kg), cerium (3,290 – 4,450 mg/kg), calcium (2,310 – 3,140 mg/kg), mercury (2,010 – 2,090 mg/kg), barium (1,970 – 2,370 mg/kg), and lanthanum (1,250 – 1,820 mg/kg). Since the calcium concentration in the blanks was approximately the same as the calcium concentration in the samples, the calcium may be from the dissolution agents.

**Table 3. Sample 1 Cation Analysis**

| Analyte | 300271087[a] Blank (µg/mL) | 300271079[b] Prelim 1 (µg/g) | 300271080[c] Prelim 2 (µg/g) | 300271081[d] Prelim 3 (µg/g) | 300271082[e] Prelim 4 (µg/g) | Average (µg/g) | Uncertainty (µg/g) |
|---|---|---|---|---|---|---|---|
| Ag | <2.57E+02 | <2.25E+02 | <2.15E+02 | <2.43E+02 | <2.31E+02 | <2.29E+02 | - |
| Al | 8.20E+02 | 4.68E+03 | 4.88E+03 | 5.37E+03 | 5.51E+03 | 5.11E+03 | 3.94E+02 |
| As | <5.50E+00 | <3.63E+00 | <5.08E+00 | <4.30E+00 | <5.27E+00 | <4.57E+00 | - |
| B | <3.00E+02 | <2.63E+02 | <2.51E+02 | <2.84E+02 | <2.70E+02 | <2.67E+02 | - |
| Ba | <9.44E+01 | 1.97E+03 | 2.05E+03 | 2.34E+03 | 2.37E+03 | 2.18E+03 | 2.02E+02 |
| Be | <5.60E+01 | <4.90E+01 | <4.68E+01 | <5.29E+01 | <5.04E+01 | <4.98E+01 | - |
| Ca | 1.90E+03 | 2.31E+03 | 2.45E+03 | 2.41E+03 | 2.94E+03 | 2.53E+03 | 2.81E+02 |
| Cd | <1.22E+02 | <1.08E+02 | <1.03E+02 | <1.16E+02 | <1.11E+02 | <1.10E+02 | |
| Ce | <5.28E+02 | 3.29E+03 | 3.46E+03 | 3.97E+03 | 3.93E+03 | 3.66E+03 | 3.40E+02 |
| Co | <6.80E+01 | 2.06E+02 | 2.02E+02 | 2.43E+02 | 2.59E+02 | 2.28E+02 | 2.80E+01 |
| Cr | <8.40E+01 | 3.00E+02 | 3.25E+02 | 3.97E+02 | 3.93E+02 | 3.54E+02 | 4.87E+01 |
| Cu | <1.14E+02 | 7.11E+02 | 6.82E+02 | 8.10E+02 | 7.96E+02 | 7.50E+02 | 6.29E+01 |
| Fe | 1.71E+02 | 4.36E+05 | 4.32E+05 | 5.19E+05 | 5.13E+05 | 4.75E+05 | 4.74E+04 |
| Gd | <3.40E+02 | <2.96E+02 | <2.84E+02 | <3.20E+02 | <3.06E+02 | <3.02E+02 | - |
| Hg | <2.20E+01 | 2.05E+03 | 2.05E+03 | 2.06E+03 | 2.13E+03 | 2.07E+03 | 3.86E+01 |
| K | <2.79E+03 | <2.44E+03 | <2.35E+03 | <2.64E+03 | <2.51E+03 | <2.49E+03 | - |
| La | <8.64E+01 | 1.25E+03 | 1.29E+03 | 1.49E+03 | 1.47E+03 | 1.38E+03 | 1.23E+02 |
| Li | <1.38E+02 | 4.98E+02 | 5.11E+02 | 5.65E+02 | 5.78E+02 | 5.38E+02 | 3.94E+01 |
| Mg | <1.76E+01 | 4.11E+02 | 4.47E+02 | 5.19E+02 | 5.44E+02 | 4.80E+02 | 6.18E+01 |
| Mn | <1.60E+01 | 3.53E+04 | 3.65E+04 | 4.13E+04 | 4.21E+04 | 3.88E+04 | 3.40E+03 |
| Mo | <5.01E+02 | <4.38E+02 | <4.18E+02 | <4.73E+02 | <4.51E+02 | <4.45E+02 | - |
| Ni | <1.88E+02 | 5.46E+04 | 5.67E+04 | 6.40E+04 | 6.46E+04 | 6.00E+04 | 5.07E+03 |
| P | <1.35E+03 | <1.18E+03 | <1.13E+03 | <1.28E+03 | <1.22E+03 | <1.20E+03 | - |
| Pb | <5.85E+02 | 8.20E+02 | 8.26E+02 | 7.81E+02 | 9.60E+02 | 8.47E+02 | 7.81E+01 |
| S | <6.00E+03 | <5.25E+03 | <5.01E+03 | <5.67E+03 | <5.40E+03 | <5.33E+03 | - |
| Sb | <5.50E+02 | <4.82E+02 | <4.60E+02 | <5.20E+02 | <4.95E+02 | <4.89E+02 | - |
| Se | <1.10E+01 | <7.27E+00 | <1.02E+01 | <8.59E+00 | <1.05E+01 | <9.14E+00 | - |
| Si | <1.85E+02 | 7.79E+03 | 8.24E+03 | 9.16E+03 | 9.46E+03 | 8.66E+03 | 7.80E+02 |
| Sn | <5.15E+02 | <4.50E+02 | <4.31E+02 | <4.86E+02 | <4.64E+02 | <4.58E+02 | - |
| Sr | <2.00E+01 | 3.30E+02 | 3.36E+02 | 3.97E+02 | 3.96E+02 | 3.65E+02 | 3.67E+01 |
| Ti | <1.36E+01 | 2.42E+02 | 2.51E+02 | 2.86E+02 | 2.84E+02 | 2.66E+02 | 2.25E+01 |
| U | <6.95E+03 | 9.16E+03 | 9.21E+03 | 1.04E+04 | 1.20E+04 | 1.02E+04 | 1.33E+03 |
| V | <8.32E+01 | <7.28E+01 | <6.94E+01 | <7.86E+01 | <7.48E+01 | <7.39E+01 | - |
| Zn | <1.21E+02 | 3.66E+02 | 3.94E+02 | 4.50E+02 | 4.43E+02 | 4.13E+02 | 4.02E+01 |

[a] Arsenic, mercury, and selenium obtained from LIMS number 300271096.
[b] Arsenic, mercury, and selenium obtained from LIMS number 300271088.
[c] Arsenic, mercury, and selenium obtained from LIMS number 300271089.
[d] Arsenic, mercury, and selenium obtained from LIMS number 300271090.
[e] Arsenic, mercury, and selenium obtained from LIMS number 300271091.

**Table 4. Sample 2 Cation Analysis**

| Analyte | 300271087[f] Blank (µg/g) | 300271083[g] Prelim 5 (µg/g) | 300271084[h] Prelim 6 (µg/g) | 300271085[i] Prelim 7 (µg/g) | 300271086[j] Prelim 8 (µg/g) | Average (µg/g) | Uncertainty (µg/g) |
|---|---|---|---|---|---|---|---|
| Ag | <2.57E+02 | <2.51E+02 | <2.18E+02 | <2.57E+02 | <2.34E+02 | <2.40E+02 | - |
| Al | 8.20E+02 | 4.52E+03 | 4.89E+03 | 4.93E+03 | 4.75E+03 | 4.77E+03 | 1.85E+02 |
| As | <5.50E+00 | <3.84E+00 | <4.25E+00 | <4.94E+00 | <5.00E+00 | <4.51E+00 | - |
| B | <3.00E+02 | <2.93E+02 | <2.54E+02 | <3.00E+02 | <2.73E+02 | <2.80E+02 | - |
| Ba | <9.44E+01 | 2.18E+03 | 2.27E+03 | 2.19E+03 | 2.21E+03 | 2.21E+03 | 4.03E+01 |
| Be | <5.60E+01 | <5.46E+01 | <4.73E+01 | <5.60E+01 | <5.10E+01 | <5.22E+01 | - |
| Ca | 1.90E+03 | 2.70E+03 | 2.65E+03 | 2.58E+03 | 3.14E+03 | 2.77E+03 | 2.53E+02 |
| Cd | <1.22E+02 | <1.20E+02 | <1.04E+02 | <1.22E+02 | <1.12E+02 | <1.15E+02 | - |
| Ce | <5.28E+02 | 4.44E+03 | 4.33E+03 | 4.45E+03 | 4.02E+03 | 4.31E+03 | 2.01E+02 |
| Co | <6.80E+01 | 2.48E+02 | 2.39E+02 | 2.45E+02 | 2.64E+02 | 2.49E+02 | 1.07E+01 |
| Cr | <8.40E+01 | 3.63E+02 | 4.14E+02 | 3.62E+02 | 3.76E+02 | 3.79E+02 | 2.43E+01 |
| Cu | <1.14E+02 | 7.71E+02 | 7.78E+02 | 8.00E+02 | 8.17E+02 | 7.92E+02 | 2.10E+01 |
| Fe | 1.71E+02 | 4.70E+05 | 4.51E+05 | 4.74E+05 | 4.53E+05 | 4.62E+05 | 1.17E+04 |
| Gd | <3.40E+02 | <3.30E+02 | <2.86E+02 | <3.40E+02 | <3.08E+02 | <3.16E+02 | - |
| Hg | <2.20E+01 | 2.09E+03 | 2.04E+03 | 2.01E+03 | 2.06E+03 | 2.05E+03 | 3.37E+01 |
| K | <2.79E+03 | <2.72E+03 | <2.36E+03 | <2.79E+03 | <2.54E+03 | <2.60E+03 | - |
| La | <8.64E+01 | 1.82E+03 | 1.79E+03 | 1.78E+03 | 1.75E+03 | 1.79E+03 | 2.89E+01 |
| Li | <1.38E+02 | 5.80E+02 | 5.66E+02 | 5.65E+02 | 5.88E+02 | 5.75E+02 | 1.12E+01 |
| Mg | <1.76E+01 | 5.17E+02 | 4.68E+02 | 4.48E+02 | 4.54E+02 | 4.72E+02 | 3.13E+01 |
| Mn | <1.60E+01 | 3.52E+04 | 4.07E+04 | 3.60E+04 | 3.60E+04 | 3.70E+04 | 2.51E+03 |
| Mo | <5.01E+02 | <4.88E+02 | <4.23E+02 | <5.01E+02 | <4.56E+02 | <4.67E+02 | - |
| Ni | <1.88E+02 | 6.91E+04 | 6.82E+04 | 6.93E+04 | 6.90E+04 | 6.89E+04 | 4.83E+02 |
| P | <1.35E+03 | <1.32E+03 | <1.14E+03 | <1.35E+03 | <1.23E+03 | <1.26E+03 | - |
| Pb | <5.85E+02 | 1.08E+03 | 9.77E+02 | 8.90E+02 | 9.47E+02 | 9.74E+02 | 7.96E+01 |
| S | <6.00E+03 | <5.85E+03 | <5.07E+03 | <6.00E+03 | <5.46E+03 | <5.60E+03 | - |
| Sb | <5.50E+02 | <5.37E+02 | <4.65E+02 | <5.50E+02 | <5.01E+02 | <5.13E+02 | - |
| Se | <1.10E+01 | <7.68E+00 | <8.50E+00 | <9.87E+00 | <1.00E+01 | <9.01E+00 | - |
| Si | <1.85E+02 | 8.45E+03 | 8.93E+03 | 8.52E+03 | 9.58E+03 | 8.87E+03 | 5.19E+02 |
| Sn | <5.15E+02 | <5.03E+02 | <4.35E+02 | <5.15E+02 | <4.68E+02 | <4.80E+02 | - |
| Sr | <2.00E+01 | 3.12E+02 | 3.46E+02 | 3.13E+02 | 3.08E+02 | 3.20E+02 | 1.76E+01 |
| Ti | <1.36E+01 | 2.74E+02 | 2.75E+02 | 2.81E+02 | 2.84E+02 | 2.79E+02 | 4.80E+00 |
| U | <6.95E+03 | 1.15E+04 | 1.21E+04 | 1.02E+04 | 1.09E+04 | 1.12E+04 | 8.14E+02 |
| V | <8.32E+01 | <8.12E+01 | <7.04E+01 | <8.32E+01 | <7.58E+01 | <7.77E+01 | - |
| Zn | <1.21E+02 | 1.37E+03 | 1.30E+03 | 1.32E+03 | 1.40E+03 | 1.35E+03 | 4.57E+01 |

Table 5 and Tank 6 show ICP-MS results ($^{235}U$, $^{237}Np$, $^{238}U$, and $^{239}Pu$) for sample 1 and sample 2, respectively. The samples were prepared by performing a peroxide fusion digestion.

[f] Arsenic, mercury, and selenium obtained from LIMS number 300271096.
[g] Arsenic, mercury, and selenium obtained from LIMS number 300271092.
[h] Arsenic, mercury, and selenium obtained from LIMS number 300271093.
[i] Arsenic, mercury, and selenium obtained from LIMS number 300271094.
[j] Arsenic, mercury, and selenium obtained from LIMS number 300271095.

**Table 5. Sample 1 ICP-MS Analysis**

| Analyte | 300271087 Blank (µg/g) | 300271079 Prelim 1 (µg/g) | 300271080 Prelim 2 (µg/g) | 300271081 Prelim 3 (µg/g) | 300271082 Prelim 4 (µg/g) | Average (µg/g) | Uncertainty (µg/g) |
|---|---|---|---|---|---|---|---|
| $^{235}U$ | <8.00E+00 | 5.43E+01 | 5.37E+01 | 6.66E+01 | 7.50E+01 | 6.24E+01 | 1.03E+01 |
| $^{237}Np$ | <4.00E+00 | 1.65E+01 | 2.76E+01 | 2.18E+01 | 1.70E+01 | 2.07E+01 | 5.17E+00 |
| $^{238}U$ | <1.00E+01 | 8.29E+03 | 8.62E+03 | 1.01E+04 | 1.04E+04 | 9.35E+03 | 1.05E+03 |
| $^{239}Pu$ | <4.00E+00 | 9.66E+01 | 1.06E+02 | 1.25E+02 | 1.23E+02 | 1.13E+02 | 1.37E+01 |
| | dpm/g | dpm/g | dpm/g | dpm/g | dpm/g | dpm/g | |
| $^{235}U$ | <3.84E+01 | 2.60E+02 | 2.58E+02 | 3.20E+02 | 3.60E+02 | 2.99E+02 | 4.94E+01 |
| $^{237}Np$ | <6.26E+03 | 2.58E+04 | 4.32E+04 | 3.41E+04 | 2.66E+04 | 3.24E+04 | 8.09E+03 |
| $^{238}U$ | <7.46E+00 | 6.19E+03 | 6.43E+03 | 7.54E+03 | 7.76E+03 | 6.98E+03 | 7.85E+02 |
| $^{239}Pu$ | <5.52E+05 | 1.33E+07 | 1.46E+07 | 1.72E+07 | 1.70E+07 | 1.55E+07 | 1.89E+06 |

**Table 6. Sample 2 ICP-MS Analysis**

| Analyte | 300271087 Blank (µg/g) | 300271083 Prelim 5 (µg/g) | 300271084 Prelim 6 (µg/g) | 300271085 Prelim 7 (µg/g) | 300271086 Prelim 8 (µg/g) | Average (µg/g) | Uncertainty (µg/g) |
|---|---|---|---|---|---|---|---|
| $^{235}U$ | <8.00E+00 | 6.48E+01 | 6.75E+01 | 6.14E+01 | 6.17E+01 | 6.39E+01 | 2.88E+00 |
| $^{237}Np$ | <4.00E+00 | 2.69E+01 | 2.78E+01 | 2.55E+01 | 2.83E+01 | 2.71E+01 | 1.23E+00 |
| $^{238}U$ | <1.00E+01 | 9.95E+03 | 1.04E+04 | 9.00E+03 | 9.78E+03 | 9.78E+03 | 5.84E+02 |
| $^{239}Pu$ | <4.00E+00 | 1.26E+02 | 1.26E+02 | 1.20E+02 | 1.29E+02 | 1.25E+02 | 3.77E+00 |
| | dpm/g | dpm/g | dpm/g | dpm/g | dpm/g | dpm/g | |
| $^{235}U$ | <3.84E+01 | 3.11E+02 | 3.24E+02 | 2.95E+02 | 2.96E+02 | 3.06E+02 | 1.38E+01 |
| $^{237}Np$ | <6.26E+03 | 4.21E+04 | 4.35E+04 | 3.99E+04 | 4.43E+04 | 4.24E+04 | 1.92E+03 |
| $^{238}U$ | <7.46E+00 | 7.43E+03 | 7.76E+03 | 6.72E+03 | 7.30E+03 | 7.30E+03 | 4.36E+02 |
| $^{239}Pu$ | <5.52E+05 | 1.74E+07 | 1.74E+07 | 1.66E+07 | 1.78E+07 | 1.73E+07 | 5.21E+05 |

Table 7 and Table 8 show the radionuclide analysis of sample 1 and sample 2, respectively.

Table 9 compares the concentrations of select cations and radionuclides in the Feed and Bleed Residual solids, the Process Sample[3], and a sample collected prior to Mechanical Sludge Removal in Tank 5F[4]. Most of the species show good agreement between their concentration in the Feed and Bleed Sample and their concentration in the Process Sample.

The following species showed lower concentrations in the Feed and Bleed Samples than in the Process Sample. The chromium concentration was 75% lower in the Feed and Bleed samples than in the process sample. The Nickel concentration was 22% lower in the Feed and Bleed Sample than in the Process Sample. The silicon concentration was about 50% lower in the Feed and Bleed Sample than in the Process Sample.

The following samples showed higher concentrations in the Feed and Bleed Samples than in the Process Sample. The aluminum concentration was approximately four times larger in the Feed and Bleed Sample. The barium concentration was approximately two times larger in the Feed and Bleed Sample. The calcium concentration was approximately nine times larger in the Feed and Bleed Sample. The calcium concentration in the blank is the same order of magnitude as the samples. Therefore, the calcium in the samples might be from the dissolution reagents. The strontium (total strontium and $^{90}Sr$) concentration was approximately three times larger in the Feed and Bleed Sample. The uranium concentration was more than six times larger in the Feed

and Bleed Sample. The $^{137}Cs$ was approximately nine times larger in the Feed and Bleed Sample. We are not sure of the reason for these differences.

Figures 1 and 2 show the particle size distribution of four samples analyzed by Microtrac. The samples had a median particle size of 60 – 109 micron, a 90$^{th}$ percentile particle size of 350 – 640 micron, and a maximum particle size of 704 micron or larger. Figure 2 shows peaks between 296 and 704 micron. Some of the samples show two multiple peaks. Figure 2 shows a flat distribution between 0.5 micron and 100 micron.

Figures 3 and 4 show the particle size distribution of four samples by SEM. The samples had a median particle size of 600 – 700 micron, and an 85$^{th}$ percentile of 900 micron. Figure 4 shows a peak at 900 micron. More than 90% of the particles are greater than 200 micron.

Figures 5 and 6 compare the particle size measures by the Microtrac and SEM methods. The particle size distribution of the particles measured by Microtrac is smaller than the particle size distribution of the particles measured by SEM. One likely cause of this difference is the shearing of the particles that occurs when their size is measured by Microtrac.

**Table 7. Sample 1 Radionuclide Analysis**

| Analyte | 300271087 Blank (dpm/g) | 300271079 Prelim 1 (dpm/g) | 300271080 Prelim 2 (dpm/g) | 300271081 Prelim 3 (dpm/g) | 300271082 Prelim 4 (dpm/g) | Average (dpm/g) | Uncertainty (dpm/g) |
|---|---|---|---|---|---|---|---|
| $^{60}Co$ | <3.42E+05 | 1.69E+07 | 1.67E+07 | 1.91E+07 | 1.87E+07 | 1.79E+07 | 1.23E+06 |
| $^{90}Sr$ | <8.78E+07 | 1.96E+10 | 2.33E+10 | 3.62E+10 | 4.19E+10 | 3.03E+10 | 1.05E+10 |
| $^{99}Tc$[k] | <5.99E+01 | 3.82E+03 | 2.25E+03 | 3.19E+03 | 3.86E+03 | 3.28E+03 | 7.52E+02 |
| $^{137}Cs$ | <3.46E+05 | 6.08E+08 | 6.56E+08 | 7.19E+08 | 7.29E+08 | 6.78E+08 | 5.68E+07 |
| $^{154}Eu$ | <3.80E+05 | 6.39E+07 | 6.60E+07 | 7.67E+07 | 7.26E+07 | 6.98E+07 | 5.91E+06 |
| $^{155}Eu$ | # | # | # | 1.18E+07 | # | n/a | - |
| $^{233}U$ | <3.09E+03 | <2.29E+02 | <2.24E+02 | <2.55E+02 | <3.20E+02 | <2.72E+02 | - |
| $^{234}U$ | <1.98E+03 | 6.53E+03 | 6.66E+03 | 7.70E+03 | 9.24E+03 | 7.53E+03 | 1.25E+03 |
| $^{235}U$ | <6.90E-01 | 2.60E+02 | 2.73E+02 | 3.17E+02 | 3.263E+02 | 2.94E+02 | 3.27E+01 |
| $^{236}U$ | <2.07E+01 | 3.13E+02 | 3.20E+02 | 3.73E+02 | 4.44E+02 | 3.62E+02 | 6.06E+01 |
| $^{238}U$ | <7.46E+00 | 6.19E+03 | 6.44E+03 | 7.55E+03 | 7.77E+03 | 6.99E+03 | 7.87E+02 |
| $^{241}Pu$ | <4.08E+06 | <3.57E+07 | <1.79E+07 | <2.02E+07 | <2.86E+07 | <2.56E+07 | - |
| $^{238}Pu$ | <1.73E+06 | 5.21E+06 | 4.37E+06 | 3.33E+06 | 5.20E+06 | 4.53E+06 | 8.90E+05 |
| $^{239/240}Pu$ | 3.40E+06 | 1.29E+07 | 1.73E+07 | 1.58E+07 | 2.37E+07 | 1.74E+07 | 4.56E+06 |
| $^{239}Pu$ | <7.30E+05 | 1.03E+07 | 1.37E+07 | 1.26E+07 | 1.87E+07 | 1.38E+07 | 3.57E+06 |
| $^{240}Pu$ | <2.66E+06 | 2.60E+06 | 3.53E+06 | 3.24E+06 | 4.938E+06 | 3.57E+06 | 9.83E+05 |
| $^{242}Pu$ | <4.64E+04 | 4.95E+02 | 6.35E+02 | 5.42E+02 | 7.53E+02 | 6.06E+02 | 1.14E+02 |
| $^{244}Pu$ | <3.24E+02 | <2.64E+00 | <4.02E+00 | <2.80E+00 | <2.33E+00 | <2.95E+00 | - |
| $^{241}Am$ | <1.60E+06 | 1.22E+08 | 1.34E+08 | 1.49E+08 | 1.35E+08 | 1.35E+08 | 1.10E+07 |
| $^{243}Am$ | <4.47E+05 | 8.60E+05 | 9.84E+05 | 1.12E+06 | 1.02E+06 | 9.96E+05 | 1.07E+05 |
| $^{242m}Am$ | <1.61E+05 | 3.59E+05 | 3.68E+05 | 3.64E+05 | 3.43E+05 | 3.59E+05 | 1.10E+04 |
| $^{243}Cm$ | <1.66E+06 | <6.10E+05 | <2.23E+06 | <5.75E+05 | <7.85E+05 | <1.05E+06 | - |
| $^{245}Cm$ | <1.36E+06 | <7.40E+05 | <5.64E+05 | <4.82E+05 | <4.01E+05 | <5.47E+05 | - |
| $^{247}Cm$ | <2.09E+06 | <3.52E+05 | <2.25E+05 | <2.64E+05 | <1.60E+05 | <2.50E+05 | - |
| $^{249}Cf$ | <2.09E+06 | <3.62E+05 | <2.26E+05 | <2.60E+05 | <1.61E+05 | <2.52E+05 | - |
| $^{251}Cf$ | <1.54E+06 | <2.23E+05 | <1.99E+05 | <2.16E+05 | <1.38E+05 | <1.94E+05 | - |
| $^{242}Cm$ | <1.33E+05 | 2.97E+05 | 3.04E+05 | 3.01E+05 | 2.83E+05 | 2.96E+05 | 9.29E+03 |
| $^{244}Cm$ | <5.28E+05 | 7.20E+06 | 7.02E+06 | 8.45E+06 | 7.54E+06 | 7.55E+06 | 6.36E+05 |

# not reported

[k] $^{99}Tc$ samples had LIMS numbers of 300271105, 300271097, 300271098, 300271099, 300271100

**Table 8. Sample 2 Radionuclide Analysis**

| Analyte | 300271087 Blank (dpm/g) | 300271083 Prelim 5 (dpm/g) | 300271084 Prelim 6 (dpm/g) | 300271085 Prelim 7 (dpm/g) | 300271086 Prelim 8 (dpm/g) | Average (dpm/g) | Uncertainty (dpm/g) |
|---|---|---|---|---|---|---|---|
| $^{60}$Co | <3.42E+05 | 2.00E+07 | 1.96E+07 | 2.01E+07 | 1.87E+07 | 1.96E+07 | 6.38E+05 |
| $^{90}$Sr | <8.78E+07 | 2.52E+10 | 3.72E+10 | 3.26E+10 | 2.64E+10 | 3.04E+10 | 5.60E+09 |
| $^{99}$Tc[1] | <5.99E+01 | 4.40E+03 | 3.52E+03 | 2.92E+03 | 5.65E+03 | 4.12E+03 | 1.19E+03 |
| $^{137}$Cs | <3.46E+05 | 6.66E+08 | 7.11E+08 | 6.47E+08 | 6.93E+08 | 6.79E+08 | 2.84E+07 |
| $^{154}$Eu | <3.80E+05 | 8.95E+07 | 8.56E+07 | 9.03E+07 | 8.62E+07 | 8.79E+07 | 2.35E+06 |
| $^{155}$Eu | # | # | # | # | # | n/a | - |
| $^{233}$U | <3.09E+03 | <2.49E+02 | <2.01E+02 | <3.17E+02 | <2.58E+02 | <2.56E+02 | - |
| $^{234}$U | <1.987E+03 | 7.02E+03 | 8.24E+03 | 7.04E+03 | 7.57E+03 | 7.46E+03 | 5.75E+02 |
| $^{235}$U | <6.90E-01 | 3.15E+02 | 3.24E+02 | 2.82E+02 | 3.04E+02 | 3.06E+02 | 1.82E+01 |
| $^{236}$U | <2.07E+01 | 3.37E+02 | 4.00E+02 | 3.40E+02 | 3.71E+02 | 3.62E+02 | 2.941E+01 |
| $^{238}$U | <7.46E+00 | 7.41E+03 | 7.77E+03 | 6.70E+03 | 7.30E+03 | 7.30E+03 | 4.43E+02 |
| $^{241}$Pu | <4.08E+06 | <2.92E+07 | <1.90E+07 | <2.81E+07 | <1.54E+07 | <2.29E+07 | - |
| $^{238}$Pu | <1.73E+06 | 7.51E+06 | 4.61E+06 | 5.73E+06 | 2.58E+06 | 5.11E+06 | 2.07E+06 |
| $^{239/240}$Pu | 3.40E+06 | 2.11E+07 | 3.14E+07 | 2.09E+07 | 2.55E+07 | 2.47E+07 | 4.93E+06 |
| $^{239}$Pu | <7.30E+05 | 1.68E+07 | 2.51E+07 | 1.67E+07 | 2.04E+07 | 1.97E+07 | 3.95E+06 |
| $^{240}$Pu | <2.66E+06 | 4.28E+06 | 6.35E+06 | 4.17E+06 | 5.15E+06 | 4.99E+06 | 1.01E+06 |
| $^{242}$Pu | <4.64E+04 | 7.02E+02 | 9.99E+02 | 6.77E+02 | <1.97E+03 | 7.93E+02 | 1.79E+02 |
| $^{244}$Pu | <3.24E+02 | <3.62E+00 | <5.79E+00 | <4.11E+00 | <1.37E+01 | <6.82E+00 | - |
| $^{241}$Am | <1.60E+06 | 1.87E+08 | 1.76E+08 | 1.67E+08 | 1.76E+08 | 1.77E+08 | 8.19E+06 |
| $^{243}$Am | <4.47E+05 | 1.32E+06 | 1.21E+06 | 1.21E+06 | 1.35E+06 | 1.27E+06 | 7.32E+04 |
| $^{242m}$Am | <1.61E+05 | 4.48E+05 | 4.97E+05 | 4.83E+05 | 5.01E+05 | 4.82E+05 | 2.41E+04 |
| $^{243}$Cm | <1.66E+06 | <1.02E+06 | <1.18E+06 | <6.81E+05 | <1.98E+06 | <1.22E+06 | - |
| $^{245}$Cm | <1.36E+06 | <9.65E+05 | <4.52E+05 | <5.71E+05 | <4.39E+05 | <6.07E+05 | - |
| $^{247}$Cm | <2.09E+06 | <3.51E+05 | <1.68E+05 | <3.12E+05 | <1.78E+05 | <2.52E+05 | - |
| $^{249}$Cf | <2.09E+06 | <3.51E+05 | <1.69E+05 | <3.09E+05 | <1.77E+05 | <2.52E+05 | - |
| $^{251}$Cf | <1.54E+06 | <2.96E+05 | <1.40E+05 | <2.55E+05 | <1.56E+05 | <2.12E+05 | - |
| $^{242}$Cm | <1.33E+05 | 3.71E+05 | 4.11E+05 | 3.99E+05 | 4.14E+05 | 3.99E+05 | 1.96E+04 |
| $^{244}$Cm | <5.28E+05 | 1.05E+07 | 1.15E+07 | 1.05E+07 | 1.15E+07 | 1.10E+07 | 5.77E+05 |

# not reported

[1] $^{99}$Tc samples had LIMS numbers of 300271105, 300271101, 300271102, 300271103, 300271104

**Table 9. Comparison of Feed and Bleed Sample with 2007 Tank 5F Sample and Tank 5F Process Sample**

| Species | Sample 1 (μg/g) | Sample 2 (μg/g) | Process Sample (μg/g) | 2007 Sample (μg/g) |
|---|---|---|---|---|
| Al | 5,110 | 4,770 | 1290 | 14,400 |
| Ba | 2,180 | 2,210 | 1090 | 1,820 |
| Ca | 2,530 | 2,770 | 303 | 3,470 |
| Cr | 354 | 379 | 1470 | <1,100 |
| Fe | 475,000 | 462,000 | 177,000 | 373,000 |
| Hg | 2,070 | 2,050 | 1480 | 1,290 |
| Mg | 480 | 472 | 409 | <850 |
| Mn | 38,800 | 37,000 | 34,600 | 68,400 |
| Ni | 60,000 | 68,900 | 83,000 | 44,500 |
| Si | 8,660 | 8,870 | 19,100 | 11,800 |
| Sr | 365 | 320 | 108 | 1,500 |
| U | 10,200 | 11,200 | <1560 | 100,000 |
| | (dpm/g) | (dpm/g) | (dpm/g) | (dpm/g) |
| $^{60}Co$ | 1.79E07 | 1.96E07 | 2.4E07 | 3.1E07 |
| $^{90}Sr$ | 3.03E10 | 3.04E10 | 1.2E10 | 8.1E10 |
| $^{99}Tc$ | 3.28E03 | 4.12E03 | <5.2E03 | 2.9E04 |
| $^{137}Cs$ | 6.78E08 | 6.79E08 | 7.7E07 | 2.3E09 |
| $^{154}Eu$ | 6.98E07 | 8.79E07 | 6.5E07 | * |
| $^{241}Pu$ | <2.56E07 | <2.29E07 | 2.9E07 | 1.8E07 |
| $^{238}Pu$ | 4.53E06 | 5.11E06 | 6.3E06 | 4.3E06 |
| $^{239/240}Pu$ | 1.74E07 | 2.47E07 | 2.4E07 | 1.9E07 |
| $^{241}Am$ | 1.35E08 | 1.77E08 | 1.4E08 | 1.1E08 |
| $^{243}Am$ | 9.96E05 | 1.27E06 | 3.5E06 | 7.4E05 |
| $^{242m}Am$ | 3.59E05 | 4.82E05 | 4.0E05 | 3.2E05 |
| $^{243}Cm$ | <1.05E06 | <1.22E06 | <1.1E06 | * |
| $^{245}Cm$ | <5.47E05 | <6.07E05 | <1.0E06 | * |
| $^{247}Cm$ | <2.50E05 | <2.52E05 | <2.6E05 | * |
| $^{249}Cf$ | <2.52E05 | <2.52E05 | <2.7E05 | * |
| $^{251}Cf$ | <1.94E05 | <2.12E05 | <4.2E05 | * |
| $^{242}Cm$ | 2.96E05 | 3.99E05 | 3.5E05 | 2.7E05 |
| $^{244}Cm$ | 7.55E06 | 1.10E07 | 1.1E07 | 8.5E06 |

* Not reported

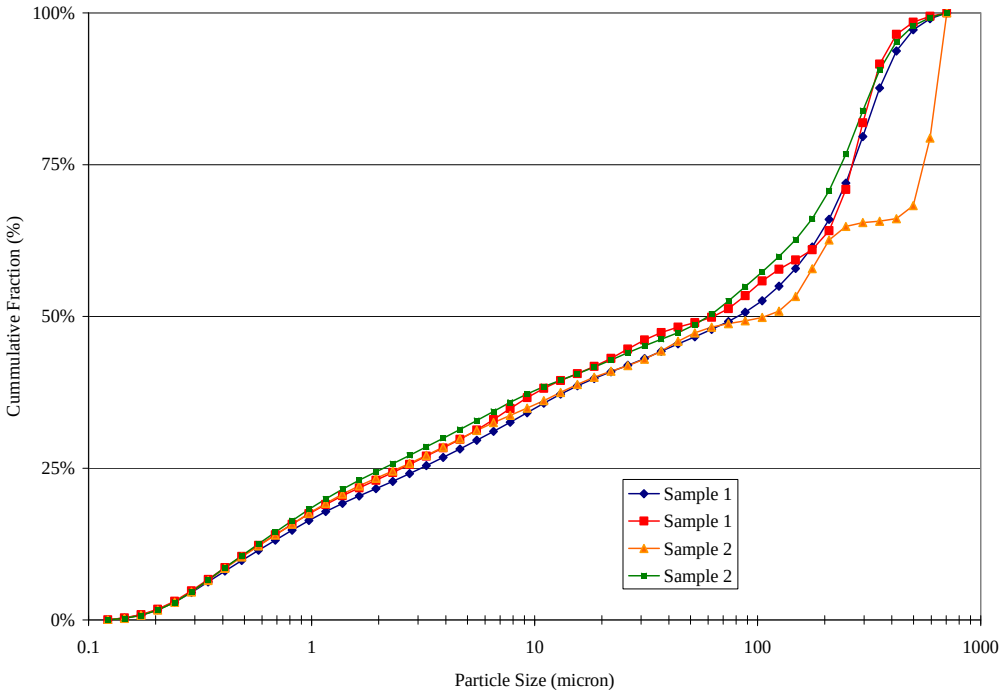


**Figure 1. Particle Size Distribution of Tank 5F Feed and Bleed Residual Solids Samples by Microtrac**

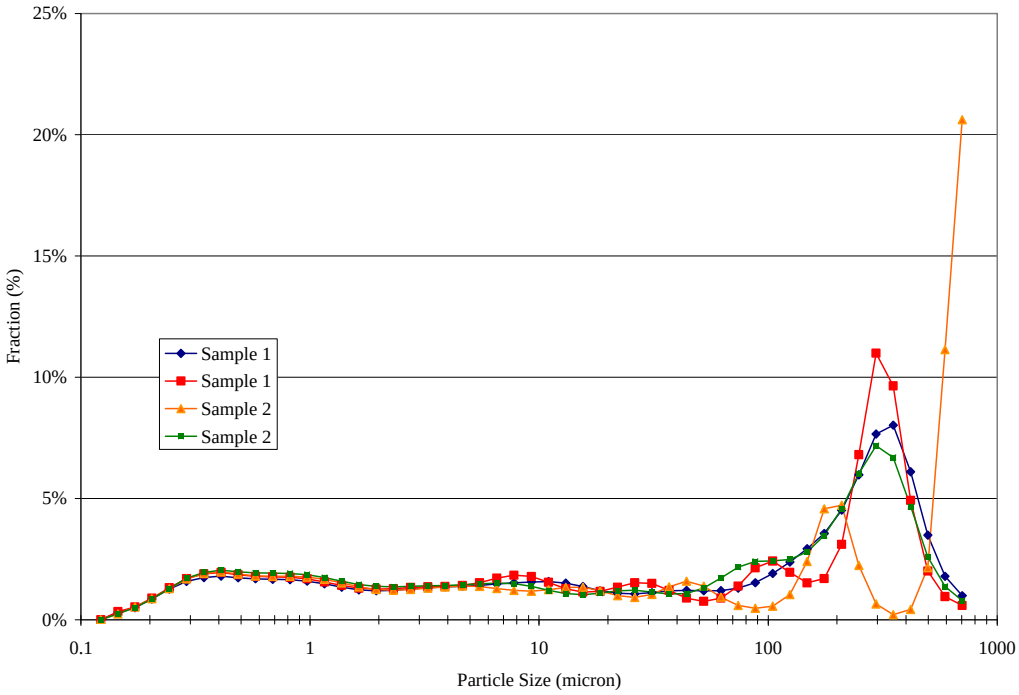


**Figure 2. Particle Size Distribution of Tank 5F Feed and Bleed Residual Solids Samples by Microtrac**

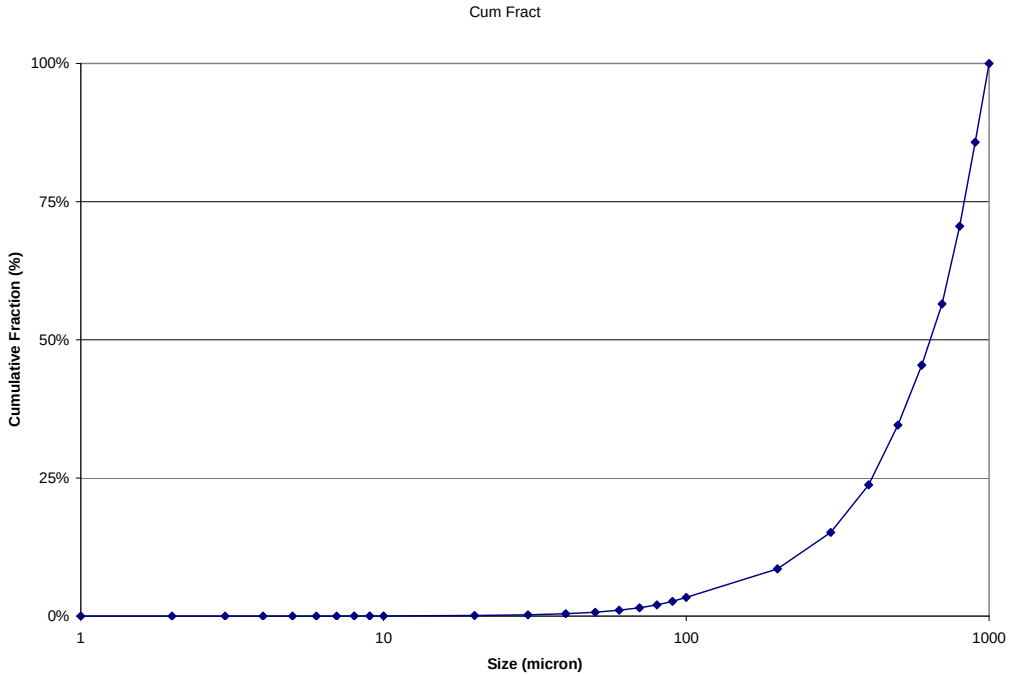


**Figure 3. Particle Size Distribution of Tank 5F Feed and Bleed Residual Solids Samples by SEM**

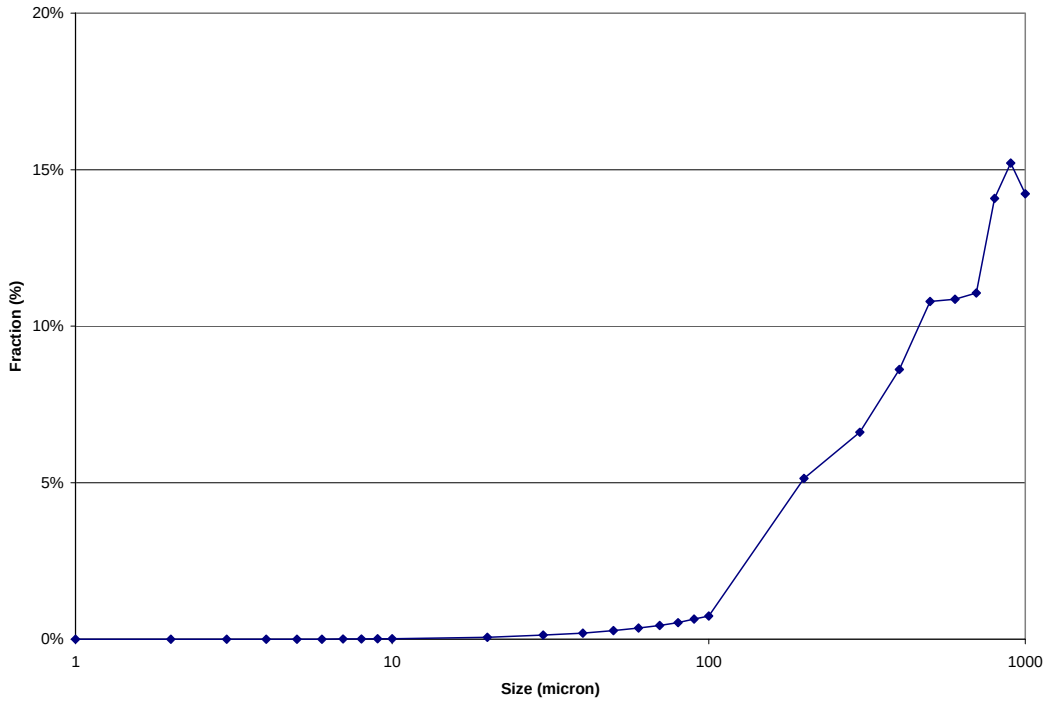


**Figure 4. Particle Size Distribution of Tank 5F Feed and Bleed Residual Solids Samples by SEM**

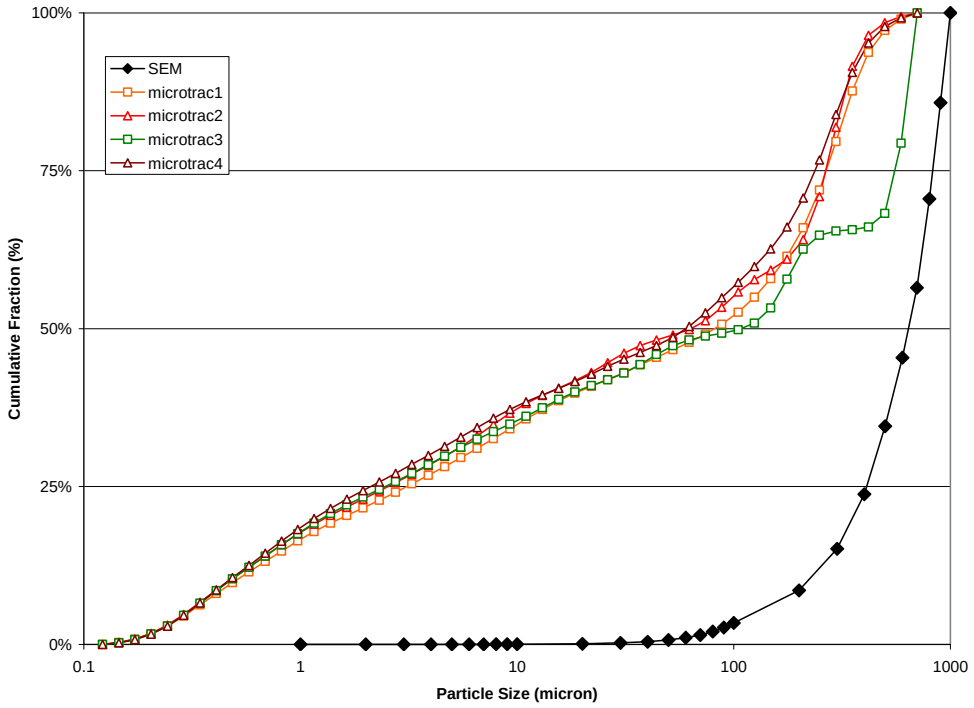


**Figure 5. Comparison of Particle Size Measurements by Microtrac and SEM**

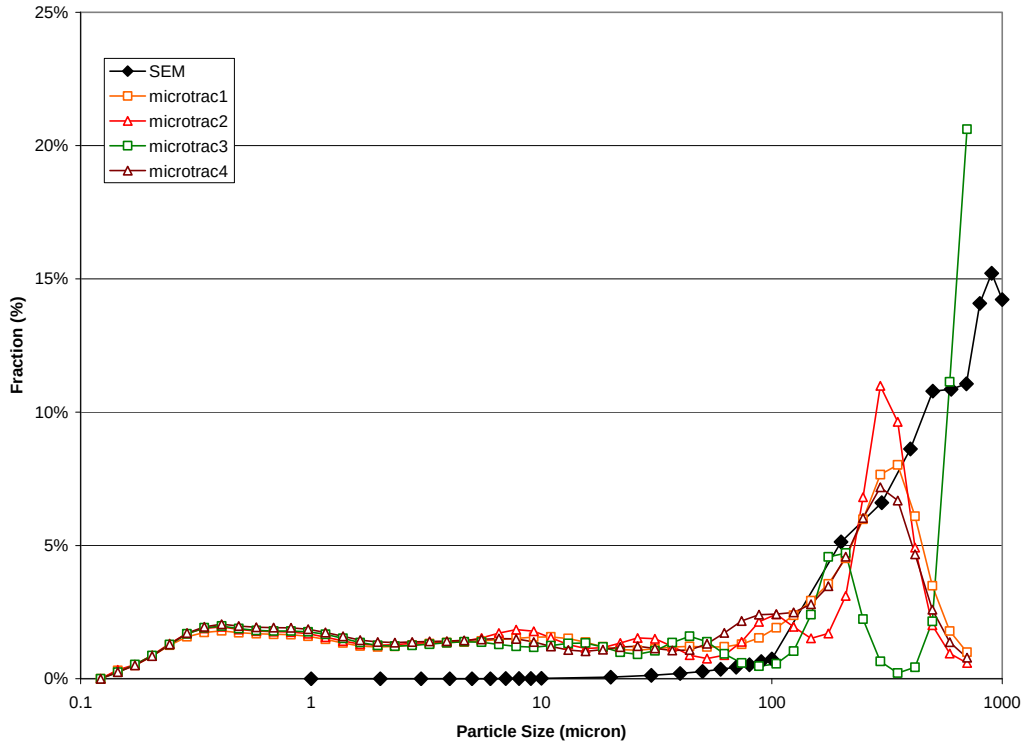


**Figure 6. Comparison of Particle Size Measurements by Microtrac and SEM**

## 5.0 CONCLUSIONS

The conclusions from this analysis follow.

- All anions measured had a concentration less than 250 mg/kg, except for oxalate, which had a concentration of 2100 – 2400 mg/kg.
- The measured cations with the highest concentration were iron (432,000 – 519,000 mg/kg), nickel (54,600 – 69,300 mg/kg), and manganese (35,200 – 42,100 mg/kg). All other cations measured less than 13,000 mg/kg.
- The radionuclides present in the highest concentration are $^{90}Sr$ ($3.0 \times 10^{10}$dpm/g), $^{137}Cs$ ($6.8 \times 10^{8}$ dpm/g), and $^{241}Am$ ($1.4 \times 10^{8}$ - $1.8 \times 10^{8}$ dpm/g).
- The particle size analysis shows a large fraction of particles greater than 100 μ.

## 6.0 REFERENCES

[1] G. D. Thaxton, “Tank 5 Feed and Bleed Residual Solids Sample Analysis”, HLE-TTR-2010-014, Rev. 1, April 27, 2010.
[2] M. R. Poirier, D. DiPrete, and C. Coleman, “Task Technical and Quality Assurance Plan for Analysis of the Tank 5F Feed and Bleed Residual Solids”, SRNL-RP-2010-00729, Rev. 0, April 27, 2010.
[3] M. R. Poirier and S. D. Fink, “Analysis of Samples from Tank 5F Chemical Cleaning”, SRNL-STI-2009-00492, December 9, 2009.
[4] M. S. Hay, K. P. Crapse, S. D. Fink, and J. M. Pareizs, "Characterization and Actual Waste Tests with Tank 5F Samples," WSRC-STI-2007-00192, Washington Savannah River Company (2007).