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Analysis of Defense Waste Processing Facility Sample: Slurry Mix Evaporator Condensate Tank Sample Batch 4638

L. N. Oji

May 2022

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) was requested by Savannah River Remediation (SRR, now Savannah River Mission Completion (SRMC)), through a Technical Task Request, to characterize the “as-received” Slurry Mix Evaporator Condensate Tank (SMECT) sample identified as sample batch 4638, which was delivered to SRNL Shielded Cells on March 19, 2021. The SMECT characterization data will be used as input to the Defense Waste Processing Facility (DWPF) Recycle Diversion Project.

This SMECT report is the second of three sample characterization reports that will be used for this DWPF Project. The other two DWPF characterization reports involve the characterization of the Recycle Collection Tank (RCT)⁴ sample and the future Off-Gas Condensate Tank (OGCT) characterization report.

A summary of the average analytical results for this DWPF SMECT sample characterization follows.

- The densities of the “as-received” SMECT sample and the SMECT filtrate averaged 1.01 g/mL, [0.4 %RSD] and 1.00 g/mL, [0.1 percent relative standard deviation (%RSD)], respectively.
- The pH of the “as-received” SMECT sample was 1.63 and that of the SMECT filtrate was 1.63.
- The turbidity of the “as-received” SMECT slurry averaged 139 nephelometric turbidity units (NTU) (1.1 %RSD) and that of the SMECT filtrate averaged 8.5 NTU (0.3 %RSD).
- The viscosity of the “as-received” SMECT slurry was 0.96 cP (1.9 %RSD) and that of the SMECT filtrate was 0.98 cP (4.2 %RSD). These viscosity values for the “as-received” SMECT sample and the SMECT filtrate are not different from that of ordinary water at 25°C.
- The SMECT sample weight percent total solids, dissolved solids, calculated weight percent insoluble solids, and soluble solids are 0.11 wt.%, (25.7 %RSD), <0.02 wt.%, (MDL), <0.09 wt.%, and <0.02 wt.%, respectively.
- The SMECT sample particle size distribution (PSD) shows a unimodal and symmetric distribution of particles lying between 0.09 microns and 0.25 microns with an average particle size of 0.17 ± 0.03 microns (1 sigma standard deviation).
- The X-ray diffraction (XRD) mineral compositions for the small and fine SMECT solids include mainly hematite (Fe_2O_3), gibbsite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) and generally sodium aluminosilicate complexes, for example $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 3\text{H}_2\text{O}$.
- Scanning electron microscope (SEM) information shows that the principal elemental constituents present in the “as-received” SMECT solid fraction include aluminum, silicon, mercury, iron, copper, thorium, nickel, titanium, barium, sodium, cerium, manganese, zirconium, uranium, rubidium, and neodymium.
- Total beta activity in the “as-received” SMECT sample averaged $4.65\text{E}+06$ dpm/mL (1.6 %RSD) with an average activity concentration of $2.09\text{E}-03$ Ci/L. The average total alpha activity was below instrument detection limit at $< 3.64\text{E}+05$ dpm/mL ($< 1.64\text{E}-04$ Ci/L).
- The primary beta emitting radionuclides in the “as-received” SMECT sample include Sr-90, Y-90 and Cs-137 at average activity of $1.70\text{E}+06$ dpm/mL, 8.0 %RSD ($7.64\text{E}-04$ Ci/L), $1.70\text{E}+06$ dpm/mL, 8.0 %RSD ($7.64\text{E}-04$ Ci/L), and $1.32\text{E}+06$ dpm/mL, 2.5 %RSD ($5.95\text{E}-04$ Ci/L), respectively.
- The primary gamma emitting radionuclide in the “as-received” SMECT sample is Ba-137m at average activity of $1.25\text{E}+06$ dpm/mL, 2.5 %RSD ($5.63\text{E}-04$ Ci/L) (note that the activity concentration for Ba-137m was calculated as 94.7% the Cs-137 activity concentration).
- The average activities for Ni-63, Eu-154 and Th-232 in the “as-received” SMECT sample were all above instrument detection limits and measured for Ni-63 ($1.20\text{E}+04$ dpm/mL, $5.42\text{E}-06$ Ci/L, 15.7 %RSD), Eu-154 ($1.05\text{E}+03$ dpm/mL, $4.74\text{E}-07$ Ci/L, 6.2 %RSD), and Th-232 ($2.58\text{E}-01$ dpm/mL, $1.16\text{E}-10$ Ci/L, 5.8 %RSD).

- Technetium-99 and Cs-135 activities in the “as-received” SMECT sample averaged 8.68E+01 dpm/mL, 4.1 %RSD (3.91E-08 Ci/L) and 7.15E-01 dpm/mL, 6.7 %RSD (3.22E-10 Ci/L), respectively.
- Average actinide activities above instrument detection limits in the “as-received” SMECT sample include U-235 (1.90E-01 dpm/mL, 8.56E-11 Ci/L, 1.8 %RSD), U-236 (3.27E-01 dpm/mL, 1.47E-10 Ci/L, 1.1 %RSD), Np-237 (5.09E+00 dpm/mL, 2.29E-09 Ci/L, 1.7 %RSD) and U-238 (2.13E+00 dpm/mL, 9.60E-10 Ci/L, 0.5 %RSD).
- Iodine-129 activity in the “as-received” SMECT sample averaged 5.39E+01 dpm/mL, 2.43E-08 Ci/L, (1.4 %RSD), while the activity for Am-241 averaged 3.95E+03 dpm/mL, 1.78E-06 Ci/L (12.6 %RSD).
- The activities for Pu-238, Pu-239/240 and Pu-241 in the “as-received” SMECT sample averaged 3.27E+04 dpm/mL, 1.47E-05 Ci/L (12.6 %RSD), 1.95E+03 dpm/mL, 8.77E-07 Ci/L (14.9 %RSD), and 5.84E+03 dpm/mL, 2.63E-06 Ci/L (5.0 %RSD).

The average activities for total beta, Ni-63, Sr-90, Eu-154, and Th-232 in the filtrate SMECT sample all showed a significant decrease in activity in comparison to their initial activities in the “as-received” SMECT sample. Total beta average activity dropped by 17% in the filtrate; going from an average activity of 4.65E+06 dpm/mL in the “as-received” SMECT sample to 3.86E+06 dpm/mL in the SMECT filtrate. Nickel-63, Sr-90, Th-232, and Eu-154 average activities in the SMECT filtrate dropped by 33%, 28%, 75%, and 43%, respectively. Their average activities in the “as-received” SMECT sample dropped from 1.20E+04 dpm/mL to 8.10E+03 dpm/mL, 1.70E+06 dpm/mL to 1.22E+06 dpm/mL, 2.58E-01 dpm/mL to 6.34E-02 dpm/mL, and 1.05E+03 dpm/mL to 5.94E+02 dpm/mL in the filtrate, respectively.

The average activities for some uranium isotopes (U-235, U-236 and U-238) did not show significant changes in the SMECT filtrate sample when compared to their average activities in the “as-received” SMECT sample. Uranium-235 average activity in the “as-received” SMECT was nearly equal to its activity in the SMECT filtrate. Uranium-236 and U-238 average activity percent changes in the “as-received” SMECT were within the analytical percent relative standard deviation for these measurements and averaged less than 10%; respectively, 5.2% and 0.9%.

Neptunium-237 average activity in the SMECT filtrate sample dropped by 26% when compared to its average activity in the “as-received” SMECT sample; its average activity dropped from 5.09E+00 dpm/mL to 3.77E+00 dpm/mL in the SMECT filtrate.

The average activities for I-129 and Cs-137 in the “as-received” SMECT sample was comparable to their average activities in the SMECT filtrate sample; their average activity changes in the “as-received” SMECT sample were 5.39E+01 dpm/mL to 5.46E+01 dpm/mL and 1.32E+06 dpm/mL to 1.27E+06 dpm/mL in the SMECT filtrate, respectively.

The average activities for the plutonium isotopes (Pu-238, Pu-239/240 and Pu-241) in the “as-received” SMECT sample dropped significantly in the SMECT filtrate sample. Plutonium-238, Pu-239/240, and Pu-241 average activities in the SMECT filtrate dropped by 76%, 45% [absolute value for the activity average used for Pu-239/240 in the filtrate], and 42%, respectively. Their average activities in the “as-received” SMECT sample dropped from 3.27E+04 dpm/mL to 7.97E+03 dpm/mL, 1.95E+03 dpm/mL to $\leq 1.08E+03$ dpm/mL, and 5.84E+03 dpm/mL to 3.39E+03 dpm/mL, respectively.

Americium-241 average activity in the SMECT filtrate sample dropped by at least 38% [absolute value for the activity average for Am-241 in the filtrate used] when compared to its average activity in the “as-received” SMECT sample. The average activity dropped from $3.95\text{E}+03$ dpm/mL to $\leq 2.45\text{E}+03$ dpm/mL in the SMECT filtrate.

Only the analytical results for aluminum, iron, manganese, and silicon were above instrument detection limits in the elemental characterization of the “as-received” SMECT sample at concentrations of 2.6 mg/L (3.7 %RSD), 11.1 mg/L (2.0 %RSD), 5.3 mg/L (0.4 %RSD), and 28.7 mg/L (1.1 %RSD), respectively.

The analytical results show that there were more elements with concentrations above instrument detection levels in the SMECT filtrate and these include aluminum (2.6 mg/L, 10.3 %RSD), calcium (1.5 mg/L, 1.3 %RSD), copper (0.5 mg/L, 77 %RSD), iron (8.3 mg/L, 0.4 %RSD), magnesium (0.3 mg/L, 48 %RSD), manganese (5.9 mg/L, 1.3 %RSD), sodium (18.0 mg/L, 0.9 %RSD), nickel (0.8 mg/L, 1.1 %RSD), silicon (24.7 mg/L, 0.0 %RSD), titanium (0.3 mg/L, 3.2 %RSD), uranium (2.9 mg/L, 0.4 %RSD) and zinc (0.4 mg/L, 2.5 %RSD). All other elements in both the “as-received” SMECT sample and SMECT filtrate were below instrument detection limit. Analytical results for selenium, arsenic, and sulfur were below instrument detection limits in both the “as-received” SMECT and SMECT filtrate samples.

The predominant anion species present in both the “as-received” SMECT and SMECT filtrates samples were formate and nitrate anions. The average concentration for formate and nitrate anions in both the “as-received” SMECT and SMECT filtrate samples were equal in magnitude, at a concentration of 17.0 mg/L (0.3 %RSD) for the formate, and 2,400 mg/L (0.6 %RSD) for the nitrate. The average concentration for the aluminate anion ($\text{Al}(\text{OH})_4^-$) in both the “as-received” SMECT and SMECT filtrate samples were just about equal, averaging $9.46\text{E}-05$ M, 0.6 %RSD and $9.52\text{E}-05$ M, 10.5 %RSD, respectively. On the other hand, the average carbonate concentration in the “as-received” SMECT sample at $1.86\text{E}-03$ M, 1.4 %RSD, is higher than the average carbonate concentration in the SMECT filtrate sample at $5.15\text{E}-04$ M, 4.5 %RSD, which is a decrease of 72 % carbonate in the filtrate. The total carbon average concentration in the “as-received” SMECT sample at $6.73\text{E}-02$ M, 1.7 %RSD, is higher than the average total carbon average concentration in the SMECT filtrate sample at $3.10\text{E}-02$ M, 1.2 %RSD, which is a decrease of 54% total carbon in the filtrate.

The average total acid at pH 7 ($3.4\text{E}-02$ M) for the “as-received” SMECT sample was statistically equivalent to the total acid at pH 7 ($3.8\text{E}-02$ M) for the SMECT filtrate sample. The corresponding calculated total acid results at pH 9 and 11 were also equal for these two SMECT sample types, respectively at $4.3\text{E}-02$ M and $4.3\text{E}-02$ M at pH 9, and $9.4\text{E}-02$ M and $9.2\text{E}-02$ M at pH 11. Therefore, the small fine particulates present in the “as-received” SMECT sample did not play any measurable negative role in the titration results for the “as-received” SMECT sample when compared with the SMECT filtrate sample.

Total mercury concentration in the “as-received” SMECT sample averaged 285 mg/L (26.0 %RSD) and averaged 269 mg/L (14.8 %RSD) in the SMECT filtrate sample.

Elemental mercury concentration dropped from an average high of 83 mg/L in the “as-received” SMECT supernate sample to a low of 28 mg/L in the filtrate, while ionic mercury showed about a 19% drop in concentration in the SMECT filtrate sample; ionic mercury concentration in the “as-received” SMECT sample dropped from an average of 27 mg/L to 22 mg/L in the SMECT filtrate sample.

The average methyl mercury concentration in the “as-received” SMECT sample, at 36 mg/L (3.5 %RSD), was not that different from its average concentration of 36 mg/L (7.3 %RSD) in the SMECT filtrate.

Ethyl mercury and dimethyl mercury determinations in the “as-received” SMECT and filtrate samples were below instrument detection limits. Therefore, these mercury species may not be present in the “as-received” SMECT sample at detectable levels. Representative aliquot sampling for elemental mercury analysis becomes a problem when elemental mercury and other forms of mercury concentration are above their saturation levels in a sample media. This was the case with the “as-received” SMECT and filtrate samples. Because of the extreme saturation level for elemental mercury and other mercury forms in the SMECT samples, the analytical uncertainty measurements for elemental and ionic mercury were high. The one sigma analytical uncertainty for elemental mercury (purgeable mercury) and ionic mercury analyses were reported as 40%, which means the analytical results would be biased. As a result, there are mercury mass balance issues with the characterization of both the “as-received” and filtrate SMECT samples for total mercury and other mercury species. Additionally, the re-analyses of the SMECT samples for mercury species occurred several months after sample receipt at SRNL, and therefore the impact of storage time, prior to these re-analyses, on the magnitude of the analytical results for these species may be negative, in part, because some of these mercury species may tend to degrade over time.

Filtering of the “as-received” SMECT sample to generate the SMECT filtrate seemed to have resulted in the retention or absorption, as part of the solid fraction, of analytes (for example Pu isotopes) with particles sizes greater than 0.45 micron (membrane pore size) or those analytes which are latched onto solid particles in the “as-received” SMECT sample. This retention of the analytes resulted in the decrease in concentrations or activities of the analytes in the filtrate or liquid fraction of the liquid/solid separation process.

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LIST OF ABBREVIATIONS

AMP	Ammonium Molybdophosphate
BT	Base titration
BUOGCT	Back-up Off-Gas Condensate Tank
cP	centi-Poise
CPC	Chemical Processing Cell
CST	crystalline silicotitanate
CSSX	caustic side solvent extraction-based
CVAFS	cold-vapor atomic fluorescence spectroscopy
DL	detection limit
DMA	direct mercury analysis
DMG	dimethylglyoxime
DWPF	Defense Waste Processing Facility
DWTT	Decontamination Waste Treatment Tank
ETF	Effluent Treatment Facility
GC-AFS	gas chromatography-atomic fluorescence spectroscopy
IC	ion chromatography
ICP-AES	inductively coupled plasma atomic emission spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
IDL	instrument detection limit (IDL)
LIMS	laboratory information management system
LSC	liquid scintillation counting
MDA	minimum detectable activity
MDL	minimum detection limit
MST	Monosodium titanate
NTU	Nephelometric Turbidity Units
OGCT	Off-Gas Condensate Tank
PI	principal investigator
PiPs	passivated, implanted, planar silicon
P&T	purge and trap
PMP	polymethyl pentane
PSA	particle size analysis
PSD	particle size distribution
PUTTA	plutonium extraction with thenoyltrifluoroacetone
QA	Quality assurance

RCT	Recycle Collection Tank
REDC	remote equipment decontamination cell
RSD	relative standard deviation
SaM	Sensing and Metrology
SC	Shielded Cells
SCO	Shielded cell operation
SEM/EDX	scanning electron microscopy/ energy dispersive x-ray
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRMC	Savannah River Mission Completion
SWPF	Salt Waste Processing Facility
TTA	thenoyltrifluoroacetone
TIC	total inorganic carbon
TOC	total organic carbon
TTQAP	task technical and quality assurance plan
TTR	technical task request
UL	upper limit
XRD	X-ray diffraction

1.0 Introduction

As part of ongoing efforts to develop a working plan for the eventual diversion of the Defense Waste Processing Facility (DWPF)¹ recycle stream away from the SRS Tank Farm, SRR (now SRMC) has requested that the Savannah River National Laboratory (SRNL) assist with the evaluation and characterization of the process chemistry involved in the DWPF waste stream diversion. Of the 5 Systems Engineering Evaluation¹ options for diverting the DWPF's recycle stream, Option 3, which involves crossflow filtration and evaporation as the main unit operations, was chosen.

Based on each of the unit operations, recycle streams will be generated that require a path back to DWPF, Salt Waste Processing Facility (SWPF), and/or Effluent Treatment Facility (ETF) to achieve the goal of decoupling the DWPF recycle stream from the Tank Farm. To select the correct facility for the returns for Option 3, certain upfront decisions need to be made regarding the process chemistry of the Recycle Collection Tank (RCT). The RCT, which receives condensate from the Slurry Mix Evaporator Condensate Tank (SMECT), is in the Chemical Processing Cell (CPC) of DWPF. The RCT also receives condensate from the Off-Gas Condensate Tank (OGCT), solutions from the laboratory and floor drains, transfers of material from the Decontamination Waste Treatment Tank (DWTT), the Back-up Off-Gas Condensate Tank (BUOGCT) and the Remote Equipment Decontamination Cell (REDC), along with many other less frequent sources. The nominal operating volume of the RCT is ~9,000 gallons and upon completing a transfer out, the RCT maintains a heel of ~1500 gallons. The streams that are transferred to the RCT can be acidic or caustic. Currently, the acidic streams that enter the RCT are neutralized and maintained basic (pH~13). This is accomplished via an addition of caustic (NaOH) and sodium nitrite (NaNO₂). Sodium nitrite is added to meet the tank farm corrosion control requirements. The SMECT samples characterized in this report were taken while DWPF was using Antifoam 747, and they have since transitioned to using MumentiveTM Y-17112 antifoam^a.

The SMECT will serve as one of the feeds for evaluating the process chemistry of the resulting streams that are generated as a function of the unit operations for Option 3. Decisions regarding the process chemistry include determining an optimal pH to enhance the unit operations (filtration and evaporation) planned for Option 3, determining the impact of glycolate for the new evaporator operation, and the resulting impacts to the downstream facilities for receipt of these new streams regarding chemistry and throughput of the facilities.

The SMECT samples sent to SRNL for characterization were identified as SMECT sample coming from batch 4638. This report presents the analytical results for the characterization of the "as-received" DWPF SMECT samples and the filtrates. These SMECT sample characterization efforts are governed by a Technical Task Request (TTR)² and a Task Technical and Quality Assurance Plan (TTQAP)³. It is the second of the three sample characterization reports supporting this DWPF recycle program. The other two DWPF reports involve the characterization of the RCT⁴ samples and the future OGCT characterization report.

^a Antifoam procedure SW4-15.85-8.14, Revision 6. 6/9/2021.

Objectives

The customer requested the characterization^{2,3} of the “as-received” DWPF SMECT sample (batch 4638), SMECT filtrate and pH adjusted SMECT samples to support the eventual diversion of the DWPF recycle stream away from the SRS Tank Farm.

The initial task performed with the SMECT sample involved physical characterizations [bulk densities, particle size distribution (PSD), X-ray diffraction (XRD) and scanning electron microscopy (SEM)], turbidity, pH measurements, and viscosity. The SMECT sample was also characterized for weight percent total and dissolved solids, density, elemental composition, total mercury and other mercury species, total acids, anions and select radionuclides. Although the characterization of the post pH adjusted SMECT samples was also requested, it was not fulfilled due to insufficient sample volume.

2.0 Experimental Setups/Sample description and Preparations/Methodology

The “as-received” SMECT sample delivered to SRNL-Shielded Cell came in three capped 250 mL capacity stainless steel containers. The samples were each transferred into a clear/transparent polymethyl pentane (PMP) container. Visual images of the samples are shown in Figure 1, insert A. These unsettled samples solutions were brown in color due to the presence of very fine dark brown particles. After about two hours of settling, a brown layer of fine solids was visible at the bottom of each PMP container (Figure 1, insert B). The settled layer of solids formed was less than 1% by volume when compared to the total volume of each sample. The samples were then left overnight to settle some more. The settled solid volumes at the bottom of each container were visually confirmed to be less than 1% by volume. After consulting with SRR (now SRMC), these three sample portions were combined, and the total combined sample volume was 430 mL.

In general, the analysis of this SMECT sample were performed in triplicate. To attain lower detection limits for I-129, Cs-135, and some anions, it was necessary to transport small portions (5-10 mL) of the “as-received” SMECT sample in shielded bottles to the Sensing and Metrology (SaM) group for special storage and characterization for these analytes without dilutions. The characterization of the SMECT sample for other radionuclides, as specified in the TTR¹ and presented in Table 1, involved simple bulk dilutions of the “as-received” SMECT sample in 2.0 M HNO₃ and 1.0 M HCL (mercury species), followed by the removal of aliquots from the Shielded Cell using shielded bottles that were sent to SRNL-SaM for analysis. These dilutions and shielding reduce exposure to personnel transporting and working with these samples. However, it is worth noting that these dilutions also affect the detection limit of the final analytical results. The fine brown particles in the “as-received” SMECT slurry samples did not dissolve in water. As a result, samples which required only water dilutions (IC-anion and total carbon, total inorganic carbon) prior to submission to SaM for analysis were sent in small aliquots of not more than 5 mL without any form of dilutions for both the “as-received” SMECT and SMECT filtrate. As earlier mentioned, undiluted samples were also used in the characterization of other analytes (Iodine-129, Cs-135 and total acidity) to enhance their detection.

All SMECT mercury sample dilutions in the Shielded Cells were in a solution of 1.0 M hydrochloric acid and aliquots sent to SaM for mercury analysis were refrigerated at the SaM special storage facility in preparation for analyses in accordance with best practices for mercury handling and analysis⁵. Where sample analyses called for the use of SMECT filtrates, these filtrate samples were obtained by a liquid /solid

separation using a 0.45 μm nylon Nalgene® filter membrane; about 100 mL of the “as-received” SMECT slurry were normally filtered through the Nalgene® membrane.

About 200 mL sample volume is required for particle size analysis (PSA) determinations using a Microtrac equipment. It is normally unsafe to transport and work with this large volume of radioactive SMECT sample slurry in a regular radioactive hood. Therefore, the normal approach to determine the particle size of radioactive solution samples outside the SRNL Shielded Cells (SC) involved the transport of a small volume of the radioactive material, usually less than 10 mL, out of the SC and the mixing of the radioactive sample with a simulant salt solution which is comparable in ionic strength with that of the radioactive solution under consideration for PSA. In this instance, a simulant salt solution was used as the suspending media for the SMECT sample slurry particle size determination as described in Appendix B.

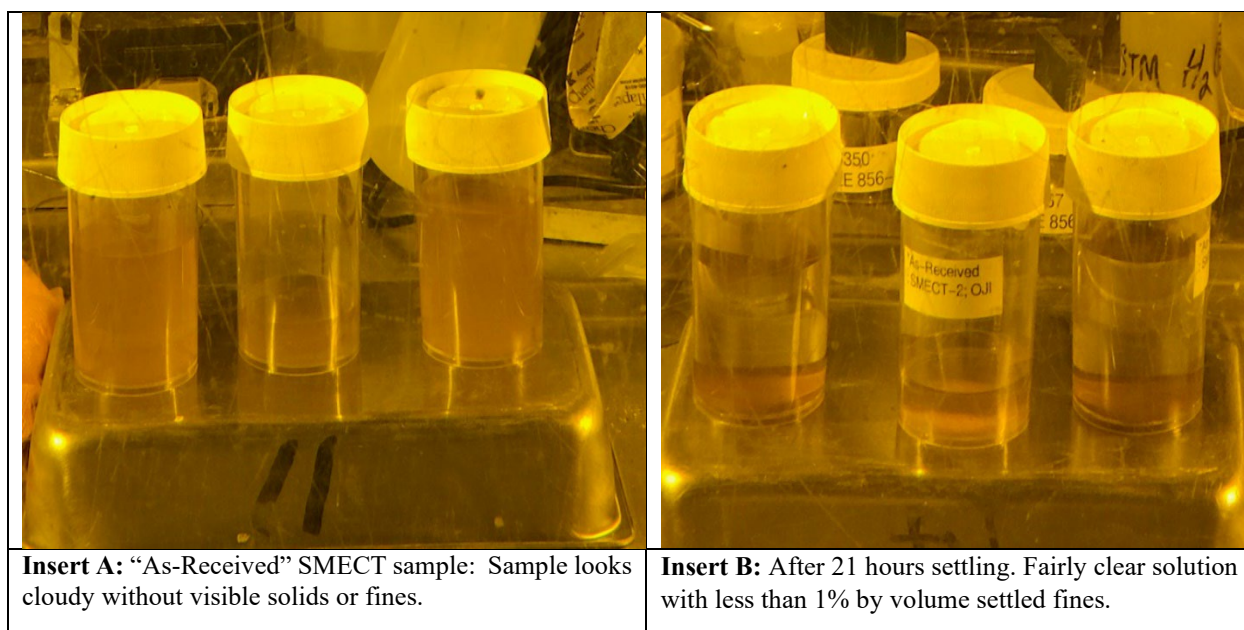


Figure 1. Photo images of the “as-received” SMECT sample

In some cases, where the analytical results for some select analytes were below instrument detection limits due to dilutions, “neat” undiluted filtrate and the “as-received” aliquots were sent to SaM for analysis. In many cases, this approach gave better results which were higher than the instrument detection limits. This approach was used in the analytical results for sulfur, arsenic, selenium, and formate as well as for I-129 and Cs-135. Since the weight percent insoluble solids for the SMECT sample was small, it can be assumed that the concentration of the analytes in the filtrate were about the same order of magnitude as that of the “as-received” SMECT slurry if there is no retention of the analytes in the 0.45 separation membrane, or the particle size of the analytes were larger than 0.45 microns.

Table 1. Analytical method Summary for SMECT Sample

Analysis	Method	Preparations	Laboratory
Density	Gravimetric/volumetric	Slurry & Filtrate	[¥] SCO
[#] Turbidity	Turbidity meter	Slurry & Filtrate	SCO
pH	pH meter, Free-OH calculation	Slurry & Filtrate	SCO
[#] Viscosity	Viscometer/Rheometer	Slurry & Filtrate	SCO
Elementals	ICP-AES	Slurry & filtrate acid dilutions	SaM
Particle size analysis (PSA)	Microtrac	Slurry	SaM
XRD	XRD	Solid fractions	SaM
SEM/EDX	SEM	Solid fractions	SaM
Axial sulfur	ICP-AES-axial S	Acid dilutions & undiluted	SaM
As, Se	ICP-MS-As/Se	Acid dilutions & undiluted	SaM
Total mercury	DMA	Slurry & filtrate acid dilutions	SaM
Methyl Hg, Dimethyl-Hg, Ethyl Hg, Ionic Hg, Elemental Hg	GC-AFS*	Slurry & filtrate acid dilutions	SaM
Wt. % total and dissolved solids	Gravimetric/thermal	Slurry & Filtrates	SaM
Sr-90	Extraction/beta counting	Acid dilutions	SaM
Cs-137	Gamma scan	Slurry & filtrate acid dilutions	SaM
Co-60, Ce-144, Eu-154, Ru-106, Sb-125 and Am-241	Cs-removed gamma scan	Slurry & filtrate acid dilutions	SaM
Pu-238 and Pu-241	Rad screen/LSC	Slurry & filtrate acid dilutions	SaM
Total acid	Titration	Slurry and undiluted filtrate	SaM
Masses 59, 84-114, 116-126, 128, 130, 133-187, 191, 193-196, 198, 203-208, 229-230, 232-252.	ICP-MS	Slurry & filtrate acid dilutions	SaM
Tc-99	Separation and LSC	Slurry & filtrate acid dilutions	SaM
I-129	I-129 with separation	Undiluted Slurry & Filtrates	SaM
Cs-135	Cs-135 extraction/ICP-MS	Undiluted Slurry & Filtrates	SaM
Total alpha/beta	Rad screen, LSC	Slurry & filtrate acid dilutions	SaM
Al(OH) ₄ ⁻	Calculated from ICP-AES	n/a	PI
Free-OH, TIC/TOC	Carbonate removal & BT	Water dilutions & undiluted	SaM
CO ₃ ²⁻	TIC/TOC calculations	Water dilutions & undiluted	SaM
NO ₂ ⁻ , NO ₃ ⁻ , SO ₄ ²⁻ , Cl ⁻ , F ⁻ , C ₂ O ₄ ²⁻ , HCO ₂ ⁻ , PO ₄ ³⁻ , Br ⁻	IC-anions	Water dilutions & undiluted	SaM
IC-Cations (NH ₃ ⁺)	IC-Cations	Water dilutions & undiluted	SaM
Y-90 and Ba-137m	Calculated	n/a	^π PI

ICP-AES = inductively coupled plasma atomic emission spectroscopy; DMA = Direct mercury analysis; ICP-MS = inductively coupled plasma mass spectroscopy; LSC = liquid scintillation counting; BT = base titration; IC = ion chromatography, TIC/TOC = total inorganic carbon/total organic carbon, GC-AFS = Gas chromatography atomic fluorescence spectroscopy, SEM/EDX =

scanning electron microscopy/energy dispersive x-ray, #qualitative data only, * Low level dilutions (ppt) required for these methods, *PI = Principal investigator and *SCO = Shielded Cell Operations.

3.0 Data Quality and Blank Evaluations

Appendix C contains the SRNL Analytical Development Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report. The sample analysis completion dates are tracked in LIMS. The SRNL Sensing and Metrology (SaM) Group used reagent blanks based on dilute acids, de-ionized water, and other test preparation techniques specific to each analytical method used in the sample preparation and characterizations in preparation for analysis.

Inductively coupled plasma mass spectrometry (ICP-MS) results are given for each atomic mass and in most cases each mass number represents only one isotope. An example of an exception is mass 238, since both uranium and plutonium are included in this mass number. However, since the mass contribution of U-238 is significantly greater than that of Pu-238, the 238 signal is used to quantify U-238, not Pu-238. For this reason, Pu-238 was determined by chemical separation coupled with alpha spectroscopy (plutonium extraction with thenoyltrifluoroacetone (PUTTA)). In cases where ICP-MS and radiochemistry data give similar results for a species (e.g., Tc-99), radiochemistry was typically selected and reported due to better sensitivity and precision.

3.1 Format of the Reported Results

In general, tables containing SMECT “as-received” slurry analytical results are presented first followed immediately by tables containing the corresponding analysis results for the SMECT filtrate samples.

The mean results, based on the average of all applicable analytical determinations, are reported in this document, along with the percent relative standard deviation (%RSD). The %RSD provides an indication of the measurement variation between triplicate determinations but is typically not an indicator of analytical accuracy. In general, the one sigma analytical uncertainty as reported by SaM was 10%, although it was sometimes lower or higher. Specifically, the one sigma analytical uncertainties reported by SaM were: a) ~20% for ICP-MS, b) ~5% for Cs-137 determined by gamma spectroscopy, and c) 40% for elemental and ionic mercury species. As such, only two to three of the leading digits reported for the SaM analysis results should be considered significant.

In the SMECT sample characterization results presented in the tables in this report, values preceded by “<” (less than sign) indicate values were below minimum detection limits (MDLs), and values preceded by “≤” (less than or equal to sign) indicate that for replicates, at least one of the analysis values was above the MDL and at least one of the analysis values was below the detection limit or was an upper limit. Thus, where replicate analyses were both above and below the detection limit, the average of all replicates above and below the detection limit is given a “≤” sign that precedes the average value. Likewise, where replicate analyses were all less than values the average is reported as the average of the less than values. The standard and percent relative deviations were calculated only for values that were all above the detection limits. The minimum detectable activity (MDA) is defined as the value above which instrument signal can be considered quantitative relative to the signal-to-noise ratio, and the upper limit (UL) is defined as activity observed but biased high due to spectral interference or blank contamination. The detection limit (DL), as used in mass spectrometer or Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-AES) analysis, is equivalent to three times the standard deviation of the blank measurements.

The one sigma percent counting uncertainty for each radionuclide reported in the tables is based on the pooled estimate derived from the individual uncertainties for each replicate measurement for that radionuclide using a Microsoft Excel function, $\text{SQRT}((\text{SUMSQ}(x_i)/n))$, where n is the number of replicates and x_i is the individual uncertainty associated with each radionuclide for each analysis. Here it is assumed that the radio-analytical processes, be it counting or other techniques, are of the same precision for each individual measurement.

Occasionally, situations may be encountered where the samples prepared and analyzed in triplicate gave mixed results with one or two of the triplicate analyses results being less than the MDA. In these cases, the reporting of the one sigma percent uncertainty is presented in a slightly different format. In this situation, the individual percent uncertainty associated with each analysis for that radionuclide is reported along with MDA, UL values, or DL values as indicated by the analytical method.

4.0 Results and Discussion

Analyses were performed on the “as-received” SMECT sample aliquots and the SMECT filtrate aliquots. A combination of routine measurement techniques and “tailor made” separation/digestion/isolation analysis methods were used to quantify select radionuclides as requested by SRR (now SRMC) as shown in Table 1. Details of the analytical methodologies employed in these characterizations are summarized in Appendix B.

4.1 Physical measurements (Density, Turbidity, pH, Weight Percent Solids, and Viscosity)

As presented in Table 2, the density of the “as-received” SMECT sample averaged 1.01 g/mL (0.4 %RSD) and that of the SMECT filtrate sample averaged 1.00 g/mL (0.1 %RSD). The pH for the “as-received” SMECT and the filtrate samples were all the same at a pH of 1.63 (Table 2). The turbidity of the “as-received” SMECT slurry averaged 139 NTU (1.1 %RSD) and that of the filtrate averaged 8.6 NTU (0.3 %RSD). The average weight percent total solids, dissolved solids and the calculated weight percent insoluble solids and soluble solids were 0.11 wt.% (25.7 %RSD), <0.02 wt.%, (MDL), <0.09 wt.% and <0.02 wt.%, respectively (Table 3). The viscosity of the “as-received” SMECT slurry averaged 0.96 cP (centi-Poise) [1.9 %RSD] and that of the SMECT filtrate averaged 0.98 cP (4.2 %RSD). These viscosity values for the “as-received” SMECT sample and the SMECT filtrate are not different from that of ordinary water at 25°C.

Table 2. Physical Characterization of SMECT Sample (batch 4638)
SMECT “as-received” Slurry

Parameter	Analysis-1	Analysis-2	Analysis-3	Average	%RSD, N =3
Density, g/mL	1.01	1.01	1.00	1.01	0.4
pH	1.63	1.63	1.63	na	na
Turbidity, NTU	141	139	138	139	1.1
Viscosity, cP	0.94	0.97	Not determined	0.96	1.9, N =2
SMECT Sample Filtrate					
Parameter	Analysis-1	Analysis-2	Analysis-3	Average	%RSD, N =3
Density, g/mL	1.00	1.00	1.00	1.00	0.1
pH	1.63	1.63	1.63	na	na
Turbidity, NTU	8.57	8.55	8.52	8.5	0.3
Viscosity, cP	0.95	1.01	Not determined	0.98	4.2, N =2

N = number of replicates, “No average values for pH; pH is a log function.

Table 3. Weight Percent Solids: SMECT Sample (batch 4638)

Parameter	Result	Units	Standard deviation	%RSD,
Total Solids (slurry basis)	0.11	Wt.%	0.03	25.7; N=2
Dissolved Solids (supernate basis)	<0.02	Wt.%	MDL	MDL
Insoluble Solids (slurry basis)	<0.09	Wt.%	Calculated	na
Soluble Solids (slurry basis)	<0.02	Wt.%	Calculated	na

4.2 Anions and Total Acid Analysis

The anion analytical results for the “as-received” SMECT and filtrate samples are presented in Tables 4 and 5. Since the concentrations of some of the anions like formate were expected to be less than 20 mg/L, it was decided to use undiluted samples for both the “as-received” and filtrate SMECT samples in the anion analyses to minimize dilution effects on the analytical results. Therefore, small aliquots of undiluted SMECT filtrate and “as-received” samples were taken out of the Shielded Cell for analyses by SaM. The results for the undiluted samples, as expected, produced the desired analytical results for formate, and nitrate as shown in Tables 4 and 5. The concentrations of all other anions were less than instrument detection limits in both the SMECT filtrate and the “as-received” SMECT samples. The concentration of formate in both the “as-received” SMECT and filtrate samples averaged 17.0 mg/L (0.3 %RSD), and the concentration of nitrate anions also averaged 2400 mg/L (0.6 %RSD) in both sample types. Hence, the liquid solid separation process (filtration) had no effect on the concentration of both formate and nitrate anions in this acidic “as-received” SMECT and filtrate samples.

The analytical results for both total carbon and total organic carbon in the undiluted “as-received” SMECT and its filtrate were above instrument detection limits because analysis were performed with undiluted samples. Total carbon and inorganic carbon in the undiluted “as-received” SMECT sample averaged 6.73E-02 gC/L (1.7 %RSD), 2.23E-02 gC/L (1.4 %RSD), respectively and 3.10E-02 gC/L (1.2 %RSD) and 6.18E-03 gC/L (4.5 %RSD) in the undiluted SMECT filtrate samples, respectively (Tables 4 and 5). The organic carbon in the “as-received” SMECT and SMECT filtrate samples averaged 4.50E-02 gC/L (2.0 %RSD) and 2.48E-02 gC/L (0.8 %RSD), respectively. Thus, the decrease in total carbon, inorganic carbon, and organic carbon between the “as-received” SMECT sample and the SMECT filtrate were 54%, 72%, and 45%, respectively.

The carbonate concentration in the undiluted “as-received” SMECT sample averaged 1.86E-03 M (1.4 %RSD) and 5.15E-04 M (4.5 %RSD) for the undiluted SMECT filtrate sample. Carbonate results are calculated from the inorganic carbon concentrations in the “as-received” SMECT and SMECT filtrate samples. The difference between these two carbonate results is about 72%, which indicates a significant amount of the inorganic carbon components were retained on the surface of the membrane during filtration to obtain the filtrate from the “as-received” SMECT sample.

Table 4. IC-Anions, Cations, and Total carbon Analytical Results: SMECT “as-received” Slurry

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Formate, HCO ₂ ⁻ , mg/L	17.0	17.0	16.9	17.0	0.1	0.3
Chloride, Cl ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Nitrite, NO ₂ ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Nitrate, NO ₃ ⁻ , mg/L	2410	2400	2380	2400	15	0.6
Phosphate, PO ₄ ³⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Sulfate, SO ₄ ²⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Oxalate, C ₂ O ₄ ²⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Bromide, Br ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Carbonate, CO ₃ ²⁻ , M	1.85E-03	1.89E-03	1.84E-03	1.86E-03	2.68E-05	1.4
Al(OH) ₄ ⁻ , M	9.85E-05	9.30E-05	9.22E-05	9.46E-05	5.24E-07	0.6
Ammonium ion, NH ₄ ⁺ mg/L	<5.0	<5.0	<5.0	<5.0	na	na
Total carbon, gC/L	6.79E-02	6.80E-02	6.60E-02	6.73E-02	1.13E-03	1.7
Inorganic carbon gC/L	2.22E-02	2.27E-02	2.21E-02	2.23E-02	3.22E-04	1.4
Organic carbon, gC/L	4.57E-02	4.54E-02	4.40E-02	4.50E-02	9.07E-04	2.0

Table 5. IC-Anions, Cations, and Total carbon Analytical Results: SMECT -Filtrate (batch 4638)

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Formate, HCO ₂ ⁻ , mg/L	17.0	17.0	16.9	17.0	0.1	0.3
Chloride, Cl ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Nitrite, NO ₂ ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Nitrate, NO ₃ ⁻ , mg/L	2410	2400	2380	2400	15	0.6
Phosphate, PO ₄ ³⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Sulfate, SO ₄ ²⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Oxalate, C ₂ O ₄ ²⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Bromide, Br ⁻ , mg/L	<10.0	<10.0	<10.0	<10.0	na	na
Carbonate, CO ₃ ²⁻ , M	4.89E-04	5.34E-04	5.21E-04	5.15E-04	2.31E-05	4.5
Al(OH) ₄ ⁻ , M	not valid	8.81E-05	1.02E-04	9.52E-05	9.95E-06	10.5; N=2
Ammonium ion, NH ₄ ⁺ mg/L	<5.0	<5.0	<5.0	<5.0	na	na
Total carbon, gC/L	3.07E-02	3.14E-02	3.09E-02	3.10E-02	3.61E-04	1.2
Inorganic carbon gC/L	5.87E-03	6.41E-03	6.25E-03	6.18E-03	2.77E-04	4.5
Organic carbon, gC/L	2.49E-02	2.50E-02	2.46E-02	2.48E-02	2.08E-04	0.8

4.3 SMECT Sample Total Acidity and Titration Curves

SMECT solutions (“as-received” SMECT slurry and SMECT filtrate) are acidic with a pH value of 1.63. Titration curves resulting from the determination of the total acidity for both the undiluted “as-received” SMECT slurry and the undiluted SMECT filtrate are shown in Appendix A. These titrations were performed in duplicate. Titrations were performed with 0.01 N sodium hydroxide (NaOH) solutions using 0.5 mL sample volumes at pH end point of 7. A summary of the total acid molarities from the titrations at a target pH value of 7 is presented in Table 6. The summary data shows that there are statistically no differences in

total acid molarities at pH 7 for the “as-received” SMECT slurry and the SMECT filtrate; the average pH 7 titration molarities are 0.034 (1.7 %RSD) and 0.038 (4.1 %RSD), respectively.

Based the titration curves provided by SaM and shown in Appendix A, the average total acid required for the titration of the “as-received” SMECT slurry (4.3E-02 M) is equal in magnitude to the total acid required to titrate the SMECT filtrate (4.3E-02 M) to a pH 9. The corresponding average total acid required for the attainment of pH of 11 for both the SMECT slurry and SMECT filtrate are 9.4E-02 M, and 9.2E-02 M, respectively. Therefore, the small fine particulates present in the “as-received” SMECT sample did not play any measurable negative role in the titration results for the “as-received” SMECT sample when compared with the SMECT filtrate sample.

These base titration curve data, volume, and molarity, with appropriate adjustments, could be used to estimate the total sodium hydroxide requirements to titrate the “as-received” SMECT slurry to an acceptable pH value of between 10 and 13, which is the target pH process requirement.

Table 6. “As-received” SMECT Slurry and SMECT Filtrate Total Acids at pH 7

“as-received” SMECT Slurry					
Parameter	Analysis-1	Analysis-2	Analysis-3	Average	%RSD, N =3
Total acid, M	3.4E-02	3.3E-02	3.4E-02	3.4E-02	1.7
SMECT Filtrate					
Parameter	Analysis-1	Analysis-2	Analysis-3	Average	%RSD, N =3
Total acid, M	3.9E-02	3.6E-02	3.8E-02	3.8E-02	4.1

4.4 Elemental Analysis, Total Mercury and Mercury Species Compositions

Total mercury, and mercury species analytical results for the “as-received” SMECT sample and the SMECT filtrate sample are presented in Tables 7 and 8, respectively. Only the analytical results for aluminum (average concentration 2.6 mg/L, 3.7 %RSD), iron (average concentration 11.1 mg/L, 2.0 %RSD), manganese (average concentration 5.3 mg/L, 0.4 %RSD), total mercury (average concentration 285.3 mg/L, 26.0 %RSD), and silicon (average concentration 28.7 mg/L, 1.1 %RSD) were above instrument detection limits in the elemental characterization of the “as-received” SMECT samples. The elements above instrument detection level in the SMECT filtrate sample include aluminum (average concentration 2.6 mg/L, 10.3 %RSD), calcium (average concentration 1.5 mg/L, 1.3 %RSD), Copper (average concentration 0.5 mg/L, 77 %RSD), iron (average concentration 8.3 mg/L, 0.4 %RSD), magnesium (average concentration 0.3 mg/L, 48 %RSD), manganese (average concentration 5.9 mg/L, 1.3 %RSD), sodium (average concentration 18.0 mg/L, 0.9 %RSD), nickel (average concentration 0.8 mg/L, 1.1 %RSD), silicon (average concentration 24.7 mg/L, 0.0 %RSD), titanium (average concentration 0.3 mg/L, 3.2 %RSD), uranium (average concentration 2.9 mg/L, 0.4 %RSD), total mercury (average concentration 269.2 mg/L, 14.8 %RSD), and zinc (average concentration 0.4 mg/L, 2.5 %RSD).

The analytical results for arsenic, sulfur, and selenium in the “as-received” SMECT sample were all below instrument detection limits for these analytes (Table 7); the results averaged <8.5E-03 mg/L, <1.5E+00 mg/L, and <4.3E-02 mg/L, respectively. The results for arsenic, sulfur, and selenium in the SMECT filtrate sample aliquots, as presented in Table 8, also averaged <8.5E-03 mg/L, <1.5E+00 mg/L, and <4.3E-02

mg/L, respectively. These results confirm that there are no measurable quantities of arsenic, sulfur, and selenium in the filtrate and in the “as-received” SMECT samples.

It is also worth mentioning that the room temperature dissolution of the “as-received” SMECT sample, with its fine solid particles, in dilute nitric acids prior to analysis for the chemical elements does not guarantee 100% dissolution of all the particles. In some cases, such incomplete digestions of the solid particles and dilution effects lead to the existence of colloids, which result in high instrument detection limit, especially if the analytes of interest are still bound in the small fraction of undigested solids and colloids. This incomplete acid digestion sometimes leads to cases where the filtrate sample characterization results in the detection of more analytes compared to the “as-received” sample, especially when the concentration of the analytes of interest are near the instrument detection limit. Yet, in other instances excessive dilution of the sample performed to protect the inductively coupled plasma atomic emission spectroscopy (ICP-AES) plasma torch from salt accumulation damage, especially sodium salts, can also lead to large analytical uncertainties and differences. These two conditions may be responsible for the analytical result issues with Ni, U, Ti, Ca, Cu, Mg, Na, and Zn in the “as-received” SMECT samples.

For example, the analytical result for Ni in the “as-received” SMECT sample is <0.8 mg/L, while its average concentration in the SMECT filtrate is 0.8 mg/L; for U, the average concentration in the “as-received” SMECT sample is < 2.9 mg/L, while the average concentration in the SMECT filtrate is 2.9 mg/L; for Ti, the average concentration in the “as-received” SMECT sample is <0.5 mg/L, while the average concentration in the SMECT filtrate is 0.3 mg/L; for Ca, the average concentration in the “as-received” SMECT sample is < 1.1 mg/L, while the average concentration in the SMECT filtrate is 1.5 mg/L; for Cu, the average concentration in the “as-received” SMECT sample is <0.3 mg/L, while the average concentration in the SMECT filtrate is 0.5 mg/L; for Na, the average concentration in the “as-received” SMECT sample is <17 mg/L, while the average concentration in the SMECT filtrate is 18 mg/L; for Zn, the average concentration in the “as-received” SMECT sample is < 0.4 mg/L, while the average concentration in the SMECT filtrate is 0.4 mg/L, and for Mg, the average concentration in the “as-received” SMECT sample is < 0.03 mg/L, while the average concentration in the SMECT filtrate is 0.3 mg/L.

All the ICP-AES elemental analytical results for the “as-received” sample are comparable in magnitude to those of the filtrate results.

The analysis for total mercury, dimethyl mercury, and ethyl mercury in both the “as-received” SMECT and SMECT filtrate samples were performed in triplicate and the analysis for the methyl mercury in duplicate. Single analysis was performed for all other mercury species (elemental and ionic mercury) in both the “as-received” and filtrate SMECT samples. Due to an instrument malfunction, the sample characterization for methyl, ethyl, elemental and ionic mercury had to be re-analyzed. These re-analyses may have impacted the analytical results for these mercury species, which tend to be unstable with long storage.

The analytical results for total mercury and all other mercury species (methyl mercury, dimethyl mercury, elemental mercury, ionic mercury, and ethyl mercury) for both the “as-received” SMECT sample and the SMECT filtrate sample are presented in Tables 7 and 8, respectively. The analytical results in the “as-received” SMECT sample were all above instrument detection limit, with dimethyl mercury and ethyl mercury being the exceptions. Total mercury concentration in this “as-received” SMECT sample averaged 285 mg/L (26 %RSD). Methyl mercury, elemental mercury, and ionic mercury (inorganic mercury)

concentrations in the “as-received” SMECT sample were 36 mg/L (3.5 %RSD), 83 mg/L, and 27 mg/L, respectively.

Total mercury and mercury species analytical results for the SMECT filtrate samples are presented in Table 8. Except for total mercury, elemental, ionic mercury, and methyl mercury analytical results for this sample, the analytical result for all the other mercury species were below instrument detection limits. Methyl mercury concentration in the “as-received” SMECT sample averaged 36 mg/L (3.5 %RSD) compared to an average of 36 mg/L (7.3 %RSD) in the SMECT filtrate, meaning there were no measurable difference in concentration for methyl mercury content in the “as-received” SMECT sample and the SMECT filtrate.

The elemental mercury concentration in the SMECT filtrate sample, at 28 mg/L, was about 66% lower than the elemental mercury concentration in the “as-received” SMECT sample (83 mg/L). The ionic mercury concentration in the SMECT filtrate sample, at 22 mg/L, was about 19% lower than the measured ionic mercury concentration in the “as-received” SMECT sample (27 mg/L).

The total mercury concentration in the SMECT filtrate averaged 269 mg/L (14.8 %RSD), which is about 16 mg/L lower than the average total mercury concentration in the original “as-received” SMECT sample [285 mg/L (26 %RSD)]. This analytical result difference is within the analytical uncertainty for total mercury measurements and thus the difference in concentrations is not significant.

As presented in Table 7, the sum of the average concentrations of all the mercury species, excluding total mercury, in the “as-received” SMECT sample is ~ 146 mg/L. This sum should equal the average total mercury analytical results (285 mg/L) or at least be within the analytical error margin of 10-20% for these methods of analyses. The mass balance difference between these two mercury sum numbers is about 49%. The one sigma analytical uncertainty for elemental mercury (purgeable mercury) and ionic mercury analyses was reported as 40%. Because elemental mercury, ionic mercury, and methyl mercury comprise a large fraction of the mass balance this difference in mass balance between total mercury and the other mercury species may also be attributed to the large analytical uncertainties for both elemental mercury and ionic mercury. In addition, the re-analyses^b of the SMECT samples for mercury species occurred five months after sample receipt at SRNL, and therefor the impact of storage time, prior to these re-analyses, on the magnitude of the analytical results for these species may be negative, in part, because some of these mercury species tend to degrade over time.

Overall, this high analytical uncertainty result for elemental mercury in the acidic “as-received” SMECT sample can be attributed to several factors, which are all related to the solubility of elemental mercury and representative aliquot sampling for purgeable mercury (elemental mercury) analysis. The solubility for elemental mercury in aqueous solutions such as water or acidic/caustic media is extremely low⁶ and ranges from 0.024 to 0.059 mg/L. For example, the measured concentration for elemental mercury in the “as-received” SMECT sample (83 mg/L) is more than 1,400 times higher than its solubility limit in water or acidic solution. At this extreme saturation level for elemental mercury, several other forms of mercury are also present in such a media and this includes, elemental mercury, mercury beads, mercury amalgams,

^b A. J. Boguess and T. L. White, “Recalibration and Validation of Mercury Speciation Methods,” SRNL-RP-2021-05291, December 22, 2021.

mercuric oxide (HgO)_s, mercury nanoparticles, mercuric hydroxides (Hg(OH)_x) to name a few⁷. The presence of these various forms of mercury and the fact that elemental mercury is above its solubility limit in the acidic SMECT sample renders the analytical results for elemental mercury biased low, likely due to the expected scatter from sampling and analyzing of suspensions.

Table 7. Elemental Analytical Results: “as-received” SMECT Slurry Sample (batch 4638)

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	<0.05	<0.05	<0.05	<0.05	na
Al	2.7	2.5	2.5	2.6	3.7
B	<2.0	<2.0	<2.0	<2.0	na
Ba	<0.01	<0.01	<0.01	<0.01	na
Be	<0.01	<0.01	<0.01	<0.01	na
Ca	<1.1	<1.1	<1.1	<1.1	na
Cd	<0.03	<0.03	<0.03	<0.03	na
Ce	<0.3	<0.3	<0.3	<0.3	na
Co	<0.1	<0.1	<0.1	<0.1	na
Cr	<0.1	<0.1	<0.1	<0.1	na
Cu	<0.3	<0.3	<0.3	<0.3	na
Fe	11.4	10.9	11.0	11.1	2.0
Gd	<0.1	<0.1	<0.1	<0.1	na
K	<1.6	<1.6	<1.6	<1.6	na
La	<0.04	<0.04	<0.04	<0.04	na
Li	<0.2	<0.2	<0.2	<0.2	na
Mg	<0.03	<0.03	<0.03	<0.03	na
Mn	5.3	5.2	5.3	5.3	0.4
Mo	<0.1	<0.1	<0.1	<0.1	na
Na	<17	<17	<17	<17	na
Ni	<0.8	<0.8	<0.8	<0.8	na
P	<3.4	<3.4	<3.4	<3.4	na
Pb	<1.5	<1.5	<1.5	<1.5	na
Sb	<0.7	<0.7	<0.7	<0.7	na
Si	28.9	28.4	29.0	28.7	1.1
Sn	<1.0	<1.0	<1.0	<1.0	na
Sr	<0.1	<0.1	<0.1	<0.1	na
Th	<1.1	<1.1	<1.1	<1.1	na
Ti	<0.5	<0.5	<0.5	<0.5	na
U	<2.9	<2.9	<2.9	<2.9	na
V	<0.2	<0.2	<0.2	<0.2	na
Zn	<0.4	<0.4	<0.4	<0.4	na
Zr	<0.2	<0.2	<0.2	<0.2	na
As	<0.008	<0.008	<0.008	<0.008	na
Sulfur	<1.50	<1.50	<1.50	<1.50	na
Se	<0.043	<0.043	<0.043	<0.043	na
Total mercury	242.5	242.5	370.9	285	26.0
Methyl mercury	37.0	35.2	not analyzed	36	3.5, N=2
Dimethyl mercury	< 0.1	< 0.1	< 0.1	< 0.1	na
Elemental mercury ^a	82.8	not analyzed	not analyzed	83	N= 1
Ionic mercury ^a	not analyzed	not analyzed	27.2	27	N= 1
Ethyl mercury	< 0.001	< 0.001	< 0.001	< 0.001	na

N = number of replicates. na = not applicable, ^a 40 % one sigma analytical uncertainty.

Table 8. Elemental Analyses Results: SMECT Filtrate Sample (batch 4638)

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	<0.06	<0.06	<0.06	<0.06	na
Al	data not valid	2.4	2.8	2.6	10.3, N=2
B	<1.8	<1.8	<1.8	<1.8	na
Ba	<0.03	<0.03	<0.03	<0.03	na
Be	<0.01	<0.01	<0.01	<0.01	na
Ca	1.5	1.5	1.5	1.5	1.3
Cd	<0.05	<0.05	<0.05	<0.05	na
Ce	<0.5	<0.5	<0.5	<0.5	na
Co	<0.1	<0.1	<0.1	<0.1	na
Cr	<0.3	<0.3	<0.3	<0.3	na
Cu	1.0	0.3	0.3	0.5	77
Fe	8.3	8.3	8.3	8.3	0.4
Gd	<0.1	<0.1	<0.1	<0.1	na
K	<1.6	<1.6	<1.6	<1.6	na
La	<0.03	<0.03	<0.03	<0.03	na
Li	<0.2	<0.2	<0.2	<0.2	na
Mg	0.5	0.2	0.2	0.3	48
Mn	6.0	5.9	5.9	5.9	1.3
Mo	<0.04	<0.04	<0.04	<0.04	na
Na	18.2	17.9	18.0	18.0	0.9
Ni	0.8	0.8	0.8	0.8	1.1
P	<0.5	<0.5	<0.5	<0.5	na
Pb	<1.5	<1.5	<1.5	<1.5	na
Sb	<0.6	<0.6	<0.6	<0.6	na
Si	24.7	24.7	24.7	24.7	0.0
Sn	<1.0	<1.0	<1.0	<1.0	na
Sr	<0.01	<0.01	<0.01	<0.01	na
Th	<0.5	<0.5	<0.5	<0.5	na
Ti	0.3	0.3	0.3	0.3	3.2
U	2.9	2.9	2.9	2.9	0.4
V	<0.1	<0.1	<0.1	<0.1	na
Zn	0.4	0.4	0.4	0.4	2.5
Zr	<0.03	<0.03	<0.03	<0.03	na
As	<0.008	<0.008	<0.008	<0.008	na
Sulfur	<1.50	<1.50	<1.50	<1.50	na
Se	<0.043	<0.043	<0.043	<0.043	na
Total mercury	314.4	254.1	239.1	269	14.8
Methyl mercury	37.6	33.9	not analyzed	36	7.3, N=2
Dimethyl mercury	<0.2	<0.2	<0.2	<0.2	na
Elemental mercury	27.8	not analyzed	not analyzed	28	na, N=1
Ionic mercury ^μ	22.1	not analyzed	not analyzed	22	na, N=1
Ethyl mercury	< 0.001	< 0.001	< 0.001	< 0.001	na

N = number of replicates, na = not applicable, ^μ Ionic mercury is also called inorganic mercury, ^α 40 % one sigma analytical uncertainty.

4.5 Analyses for Select Radionuclides

Total beta activity in the “as-received” SMECT sample averaged 4.65E+06 dpm/mL (1.6 %RSD) and an average activity concentration of 2.09E-03 Ci/L. The average total alpha activity was upper bound at <3.64E+05 dpm/mL (<1.64E-04 Ci/L). As shown in Table 9, the detected radionuclide average activities

in the “as-received” SMECT sample included Ni-63 (1.20E+04 dpm/mL, 15.7 %RSD (5.42E-06 Ci/L)), Cs-135 (7.15E-01 dpm/mL, 6.7 %RSD (3.22E-10 Ci/L)), I-129 (5.39E+01 dpm/mL, 1.4 %RSD (2.43E-08 Ci/L)), Th-232 (2.58E-01 dpm/mL, 5.8 %RSD (1.16E-10 Ci/L)), U-234 (1.27E+01 dpm/mL, 6.4 %RSD (5.72E-09 Ci/L)), U-235 (1.90E-01 dpm/mL, 1.8 %RSD (8.56E-11 Ci/L)), U-236 (3.27E-01 dpm/mL, 1.1 %RSD (1.47E-10 Ci/L)), Np-237 (5.09E+00 dpm/mL, 1.7 %RSD (2.29E-09 Ci/L)), U-238 (2.13E+00 dpm/mL, 0.5 %RSD (9.60E-10 Ci/L)), Pu-238 (3.27E+04 dpm/mL, 12.6 %RSD (1.47E-05 Ci/L)), Pu-239/240 (1.95E+03 dpm/mL, 14.9 %RSD (8.77E-07 Ci/L)), Pu-241 (5.84E+03 dpm/mL, 5.0 %RSD (2.63E-06 Ci/L)), Am-241 (3.95E+03 dpm/mL, 12.6 %RSD (1.78E-06 Ci/L)), and Eu-154 (1.05E+03 dpm/mL, 6.2 %RSD (4.74E-07 Ci/L)). As presented in Table 9, the activities for the other radionuclides (Na-22, Al-26, K-40, Nb-94, Ni-59, Co-60, Ru-103, Ru-106, Sb-125, Sb-126, Sn-126, Cs-134, Ce-144, Eu-152, Eu-155, U-233, Np-239, and Am-243) were below instrument detection limits. The activity for Tc-99 was calculated from the ICP-MS data. Assuming the contribution of mass 99 from Ru-99 was negligible, the average activity for Tc-99 in the “as-received” SMECT sample is calculated as 8.68E+01 dpm/mL (3.91E-08 Ci/L, 4.1 %RSD).

As presented in Table 9, the primary beta emitting radionuclides in the SMECT sample include Sr-90, Y-90 and Cs-137 at average activity of 1.70E+06 dpm/mL, 8.0 %RSD (7.64E-04 Ci/L), 1.70E+06 dpm/mL, 8.0 %RSD (7.64E-04 Ci/L) and 1.32E+06 dpm/mL, 2.5 %RSD (5.95E-04 Ci/L), respectively, in the “as-received” SMECT sample. Both Ba-137m and Y-90 activities were calculated as 94.7% of the Cs-137 and 100% of the Sr-90 activities, respectively. The primary gamma emitting radionuclide is Ba-137m at average activity of 1.25E+06 dpm/mL, 2.5 %RSD (5.63E-04 Ci/L) for the “as-received” SMECT sample.

As shown in Table 10, total beta activity in the SMECT sample-filtrate averaged 3.86E+06 dpm/mL, 3.2 %RSD (1.74E-03 Ci/L). In the total alpha activity measurements for the SMECT filtrate, two of the three analytical values were above instrument minimum detection limit, and the third analysis was below instrument detection limit. Thus, the average of the replicates (above and below the detection limit) was assigned a “≤” sign that precedes the average activity in units of dpm/mL and average activity concentration in units of Ci/L for total alpha; ≤1.00E+05 dpm/mL (≤4.52E-05 Ci/L).

The activities for Sr-90, Y-90 and Cs-137, as presented in Table 10, for the SMECT filtrate sample averaged 1.22E+06 dpm/mL, 4.9 %RSD (5.50E-04 Ci/L), 1.22E+06 dpm/mL, 4.9 %RSD (5.50E-04 Ci/L), and 1.27E+06 dpm/mL, 2.0 %RSD (5.73E-04 Ci/L), respectively. The primary gamma emitting radionuclide, Ba-137m, activity averaged 1.21E+06 dpm/mL, 2.0 %RSD (5.43E-04 Ci/L) for the SMECT filtrate. The Eu-154 activity in the SMECT sample filtrate averaged 5.94E+02 dpm/mL, 19.7 %RSD (2.68E-07 Ci/L), which corresponds to about 43% drop in Eu-154 activity in the SMECT filtrate when compared to Eu-154 activity in the “as-received” SMECT sample.

The other detected radionuclides with activities above instrument detection limits in the SMECT filtrate sample, as presented in Table 10, include Ni-63 (8.10E+03 dpm/mL, 11.3 %RSD (3.65E-06 Ci/L)), I-129 (5.46E+01 dpm/mL, 3.1 %RSD (2.46E-08 Ci/L)), Th-232 (6.34E-02 dpm/mL, 1.6 %RSD (2.86E-11 Ci/L)), U-235 (1.87E-01 dpm/mL, 2.9 %RSD (8.42E-11 Ci/L)), U-236 (3.10E-01 dpm/mL, 2.4 %RSD (1.40E-10 Ci/L)), Np-237 (3.77E+00 dpm/mL, 1.4 %RSD (1.70E-09 Ci/L)), U-238 (2.11E+00 dpm/mL, 0.2 %RSD (9.50E-10 Ci/L)), Pu-238 (7.97E+03 dpm/mL, 17.8 %RSD (3.59E-06 Ci/L)) and Pu-241 (3.39E+03 dpm/mL, 5.1 %RSD (1.53E-06 Ci/L)).

In the activity measurements for Pu-239/240 and Am-241 in the SMECT filtrate, two of the three analytical results for both Pu-239/240 and Am-241 were above instrument minimum detection limit, and a third analysis was below instrument detection limit. Hence, the average analytical results are reported with a “≤” sign; Pu-239/240 ($\leq 1.08\text{E}+03$ dpm/mL, ($\leq 4.89\text{E}-07$ Ci/L)), Am-241 ($\leq 2.45\text{E}+03$ dpm/mL ($\leq 1.10\text{E}-06$ Ci/L)). Using the absolute values for the filtrate activity averages for these radionuclides, the average activity for Pu-239/240 and Am-241 in the SMECT filtrate had dropped by about 45% and 38%, respectively, when compared to their activities in the “as-received” SMECT sample.

Because the liquid scintillation counting for Tc-99 was below instrument detection limit, the activity for Tc-99 was calculated from the mass spectral data in Table 12. The average activity for Tc-99 in the SMECT filtrate sample is reported as $7.68\text{E}+01$ dpm/mL ($3.46\text{E}-08$ Ci/L, 6.7 %RSD). Technetium-99 concentration in the “as-received” SMECT sample, averaging $8.68\text{E}+01$ dpm/mL ($3.91\text{E}-08$ Ci/L, 4.1 %RSD), is statistically not different from the Tc-99 concentration in the SMECT filtrate sample.

As presented in the analytical results in Tables 9 and 10, there seem to be a significant drop in the activities of certain radionuclides (Ni-63, Th-232, Np-237, and Pu-238) in the filtrate SMECT sample when compared to their activities in the “as-received” SMECT sample. The average activity for Ni-63, Th-232, Np-237 and Pu-238 dropped from $1.20\text{E}+04$ dpm/mL to $8.10\text{E}+03$ dpm/mL, $2.58\text{E}-01$ dpm/mL to $6.34\text{E}-02$ dpm/mL, $5.09\text{E}+00$ dpm/mL to $3.77\text{E}+00$ dpm/mL, and $3.27\text{E}+04$ dpm/mL to $7.97\text{E}+03$ dpm/mL, respectively, from the “as-received” SMECT sample to the SMECT filtrate. This indicates that portions of the Ni, Th, Np, and Pu in the “as-received” SMECT sample were contained in the solid fraction made of dark brown insoluble solids. There were no analytical results for Cs-135 in the SMECT filtrate in part because of limited sample volume.

In summary, the changes in the average activity/concentration of the radioactive analytes in the “as-received” SMECT sample and SMECT filtrate can be classified into five categories: (1) analytes where there are no changes in activities between the “as-received” SMECT and SMECT filtrate or where the changes in average activity is less than 5% and this includes Cs-137, Ba-137m, U-235, U-236, U-238 and I-129, (2) analytes where the changes in average activity between the “as-received” SMECT samples and SMECT filtrate lie between 5 and 20%; total beta with an average drop of 17% from its initial average activity in the “as-received” sample and Tc-99 with a drop of 12% in the SMECT filtrate are the only analytes in this category, (3) this category includes those radionuclides where the difference in average activity between the “as-received” SMECT sample and the SMECT filtrate is greater than 20% and include Ni-63, Sr-90, Y-90, Eu-154, Th-232, Np-237, Pu-238, Pu-241, Pu-239/240 and Am-241, (4) analytes where the average activity drops to less than instrument detection limits in the filtrate and this includes U-234, and (5) analytes with average activities less than or equal to instrument detection limits in both the “as-received” SMECT sample and the SMECT filtrate and include Ni-59, Co-60, Ru-106, Sb-125, Sb-126, Sn-126, Cs-134, Eu-152, U-233, Am-243, and total alpha. For isotopes in categories 4 and 5, it may be assumed that the actual difference between the “as-received” SMECT and SMECT filtrate would follow the trend of other isotopes of that element. Cesium-135 is not included in any of these categories because it was not analyzed for in the SMECT filtrate.

These differences in the activities of some radionuclides in the filtrate SMECT sample compared to their activities in the “as-received” SMECT sample would seem to indicate that these radionuclides exist in the “as-received” SMECT sample as part of the solid fraction (insoluble components) with particles sizes

greater than 0.45 microns (pore size of the filter membrane used in the liquid/solid separations to obtain the filtrate). This would lead to the solid particles being retained on the filter membrane as part of the solid fraction while those like Cs-137, which are quite soluble, would easily become part of the liquid fraction and show no activity or concentration difference in both the “as-received” and filtrate samples.

The %RSD for all analytes with measurable minimum detectable activity, as summarized in Table 9 and 10 are less than or equal to 20%, and thus meets the analytical expectations except for Eu-154 with one sigma analytical uncertainty of 22.2%. However, the one sigma analytical uncertainties of 29.6, 35.5, and 46.7% (Table 10), for Am-241, total alpha, and Pu-239/240 analytical results, respectively, are on the high end of the analytical uncertainty for radioanalytical methods, which normally range from 5-20% for radionuclide quantified by counting methods (gamma spectroscopy, alpha spectroscopy and liquid scintillation counting). However, it is worth noting that for Am-241, total alpha and Pu-238/240, two of the triplicate analytical results were above instrument minimum detection limit and the third was below instrument detection limits, which may indicate the existence of sampling inconsistencies / analyte distribution issues during sample preparations and thus lead to large analytical uncertainties.

Table 9. Select Radionuclide Analytical Results: “as-received” SMECT Slurry Sample

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L slurry	%RSD N = 3	1 sigma % uncertainty
Total alpha	<1.43E+05	<1.78E+05	<7.71E+05	<3.64E+05	<1.64E-04	na	MDA
Total beta	4.63E+06	4.73E+06	4.59E+06	4.65E+06	2.09E-03	1.6	11 [#]
Na-22	<1.55E+02	<1.36E+02	<1.08E+02	<1.33E+02	<5.98E-08	na	MDA
Al-26	<1.45E+02	<1.27E+02	<1.01E+02	<1.24E+02	<5.60E-08	na	MDA
K-40	<3.22E+03	<3.02E+03	<2.30E+03	<2.85E+03	<1.28E-06	na	MDA
Ni-59	<1.12E+02	<6.20E+01	<1.34E+02	<1.03E+02	<4.62E-08	na	MDA
Co-60	<2.14E+02	<2.17E+02	<1.05E+02	<1.79E+02	<8.05E-08	na	MDA
Ni-63	1.22E+04	1.38E+04	1.01E+04	1.20E+04	5.42E-06	15.7	20
Sr-90	1.62E+06	1.61E+06	1.85E+06	1.70E+06	7.64E-04	8.0	12.3
Y-90	1.62E+06	1.61E+06	1.85E+06	1.70E+06	7.64E-04	8.0	12.3
Nb-94	<1.98E+02	<1.90E+02	<1.40E+02	<1.76E+02	<7.92E-08	na	MDA
^Tc-99	9.08E+01	8.49E+01	8.45E+01	8.68E+01	3.91E-08	4.1	10
Ru-103	<2.20E+02	<2.23E+02	<1.49E+02	<1.98E+02	<8.90E-08	na	MDA
Ru-106	<1.33E+03	<1.34E+03	<9.31E+02	<1.20E+03	<5.41E-07	na	MDA
Sb-125	<7.05E+02	<6.34E+02	<4.77E+02	<6.05E+02	<2.72E-07	na	MDA
Sb-126	<2.37E+02	<2.26E+02	<1.63E+02	<2.09E+02	<9.40E-08	na	MDA
Sn-126	<1.11E+03	<1.10E+03	<7.86E+02	<9.99E+02	<4.50E-07	na	MDA
I-129	5.48E+01	5.34E+01	5.36E+01	5.39E+01	2.43E-08	1.4	4.1
Cs-134	<4.20E+02	<4.40E+02	<5.31E+02	<4.64E+02	<2.09E-07	na	MDA
Cs-135	7.33E-01	7.51E-01	6.60E-01	7.15E-01	3.22E-10	6.7	20
Cs-137	1.30E+06	1.31E+06	1.36E+06	1.32E+06	5.95E-04	2.5	5.0
Ba-137m	1.23E+06	1.24E+06	1.29E+06	1.25E+06	5.63E-04	2.5	5.0
Ce-144	<2.36E+03	<2.35E+03	<1.67E+03	<2.12E+03	<9.57E-07	na	MDA
Eu-152	<7.96E+02	<7.84E+02	<5.49E+02	<7.10E+02	<3.20E-07	na	MDA
Eu-154	9.82E+02	1.11E+03	1.07E+03	1.05E+03	4.74E-07	6.2	14.8
Eu-155	<1.28E+03	<1.26E+03	<9.05E+02	<1.15E+03	<5.18E-07	na	MDA
Th-232	2.65E-01	2.41E-01	2.67E-01	2.58E-01	1.16E-10	5.8	20
U-233	<1.84E+01	<1.84E+01	<1.84E+01	<1.84E+01	<8.27E-09	na	MDA
U-234	1.36E+01	1.24E+01	1.21E+01	1.27E+01	5.72E-09	6.4	20
U-235	1.93E-01	1.90E-01	1.87E-01	1.90E-01	8.56E-11	1.8	20
U-236	3.29E-01	3.29E-01	3.23E-01	3.27E-01	1.47E-10	1.1	20
Np-237	5.13E+00	4.99E+00	5.15E+00	5.09E+00	2.29E-09	1.7	20
U-238	2.14E+00	2.14E+00	2.12E+00	2.13E+00	9.60E-10	0.5	20
Pu-238	3.19E+04	2.90E+04	3.71E+04	3.27E+04	1.47E-05	12.6	6.7
Np-239	<1.35E+03	<1.35E+03	<9.65E+02	<1.22E+03	<5.50E-07	na	MDA
Pu-239/240	1.72E+03	1.85E+03	2.27E+03	1.95E+03	8.77E-07	14.9	14.9
Pu-241	5.66E+03	5.67E+03	6.17E+03	5.84E+03	2.63E-06	5.0	15.7
Am-241	3.50E+03	3.86E+03	4.48E+03	3.95E+03	1.78E-06	12.6	20.0
Am-243	<9.56E+02	<9.48E+02	<5.41E+02	<8.15E+02	<3.67E-07	na	MDA

N = number of replicates. # Pooled estimate based on Excel function SQRT((SUMSQ(xi)/n)) where applicable. ^Tc-99, Th-232, U-235, Np-237, U-238 and other actinide activities were calculated from ICP-MS data in Table 11. na = not applicable.

Table 10. Select Radionuclide Analytical Results: SMECT Filtrate Sample (batch 4638)

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L of slurry	%RSD N = 3	1 sigma % uncertainty
Total alpha	1.10E+05	1.05E+05	<8.67E+04	≤1.00E+05	≤4.52E-05	na	35.5/MDA
Total beta	3.84E+06	3.75E+06	4.00E+06	3.86E+06	1.74E-03	3.2	12 [#]
Na-22	<1.43E+02	<1.43E+02	<1.44E+02	<1.43E+02	<6.45E-08	na	MDA
Al-26	<1.38E+02	<1.34E+02	<1.35E+02	<1.36E+02	<6.12E-08	na	MDA
K-40	<3.03E+03	<3.10E+03	<3.09E+03	<3.07E+03	<1.38E-06	na	MDA
Ni-59	<2.76E+01	<2.25E+01	<3.19E+01	<2.74E+01	<1.23E-08	na	MDA
Co-60	<2.10E+02	<2.23E+02	<2.25E+02	<2.19E+02	<9.88E-08	na	MDA
Ni-63	9.10E+03	7.90E+03	7.30E+03	8.10E+03	3.65E-06	11.3	20
Sr-90	1.28E+06	1.22E+06	1.17E+06	1.22E+06	5.50E-04	4.9	10.9
Y-90	1.28E+06	1.22E+06	1.17E+06	1.22E+06	5.50E-04	4.9	10.9
Nb-94	<2.26E+02	<2.04E+02	<2.02E+02	<2.11E+02	<9.49E-08	na	MDA
^Tc-99	7.98E+01	7.98E+01	7.09E+01	7.68E+01	3.46E-08	6.7	10
Ru-103	<2.21E+02	<2.24E+02	<2.09E+02	<2.18E+02	<9.82E-08	na	MDA
Ru-106	<1.34E+03	<1.32E+03	<1.26E+03	<1.30E+03	<5.87E-07	na	MDA
Sb-125	<6.70E+02	<6.40E+02	<6.53E+02	<6.54E+02	<2.95E-07	na	MDA
Sb-126	<2.23E+02	<2.34E+02	<2.38E+02	<2.32E+02	<1.04E-07	na	MDA
Sn-126	<1.05E+03	<1.07E+03	<1.07E+03	<1.07E+03	<4.80E-07	na	MDA
I-129	5.64E+01	5.44E+01	5.30E+01	5.46E+01	2.46E-08	3.1	5.0
Cs-134	<4.90E+02	<5.29E+02	<4.81E+02	<5.00E+02	<2.25E-07	na	MDA
Cs-135	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	na	Not analyzed.
Cs-137	1.25E+06	1.30E+06	1.27E+06	1.27E+06	5.73E-04	2.0	5.0
Ba-137m	1.18E+06	1.23E+06	1.20E+06	1.21E+06	5.43E-04	2.0	5.0
Ce-144	<2.25E+03	<2.23E+03	<2.26E+03	<2.25E+03	<1.01E-06	na	MDA
Eu-152	<7.43E+02	<7.65E+02	<7.70E+02	<7.59E+02	<3.42E-07	na	MDA
Eu-154	5.34E+02	5.20E+02	7.29E+02	5.94E+02	2.68E-07	19.7	22.2
Eu-155	<1.22E+03	<1.23E+03	<1.22E+03	<1.22E+03	<5.52E-07	na	MDA
Th-232	6.36E-02	6.43E-02	6.23E-02	6.34E-02	2.86E-11	1.6	20
U-233	<3.65E+01	<3.65E+01	<3.65E+01	<3.65E+01	<1.65E-08	na	MDA
U-234	<2.36E+01	<2.36E+01	<2.36E+01	<2.36E+01	<1.06E-08	na	MDA
U-235	1.85E-01	1.93E-01	1.83E-01	1.87E-01	8.42E-11	2.9	20
U-236	3.05E-01	3.19E-01	3.07E-01	3.10E-01	1.40E-10	2.4	20
Np-237	3.75E+00	3.73E+00	3.83E+00	3.77E+00	1.70E-09	1.4	20
U-238	2.11E+00	2.11E+00	2.11E+00	2.11E+00	9.50E-10	0.2	20
Pu-238	9.35E+03	8.05E+03	6.51E+03	7.97E+03	3.59E-06	17.8	13.2
Np-239	<1.28E+03	<1.28E+03	<1.30E+03	<1.29E+03	<5.80E-07	na	MDA
Pu-239/240	1.09E+03	<7.53E+02	1.41E+03	≤1.08E+03	≤4.89E-07	na	MDA/46.7
Pu-241	3.59E+03	3.27E+03	3.31E+03	3.39E+03	1.53E-06	5.1	17.7
Am-241	2.20E+03	2.00E+03	<3.15E+03	≤2.45E+03	≤1.10E-06	na	MDA/29.6
Am-243	<9.18E+02	<9.10E+02	<9.01E+02	<9.10E+02	<4.10E-07	na	MDA

N = number of replicates. # Pooled estimate based on Excel function SQRT((SUMSQ(xi)/n)) where applicable. ^Tc-99, Th-232, U-235, Np-237 U-238 and other actinide activities were calculated from ICP-MS data in Table 12.

4.6 XRD Spectra, SEM/EDX and Particle Size Distribution

The “as-received” SMECT slurry sample contained very little amount of solids fraction. As a result, the XRD signal intensities for the solid fraction were very weak, as shown in Figure 2. The large shift in base line is due to the presence of the sample holder (filter membrane) bearing the traces of SMECT solid fractions. The XRD mineral compositions for the small SMECT solids peaks include mainly hematite (Fe₂O₃), and possibly sodium aluminosilicate complexes, for example NaO₂•Al₂O₃SiO₂•3H₂O. However, because the average aluminum concentration in the “as-received” and filtrate samples are equal at 2.6 mg/L

(Tables 7 and 8), the existence of insoluble gibbsite mineral in the sample, as shown in Figure 2, may be a misidentification due in part to high XRD spectral background noise and baseline shifts.

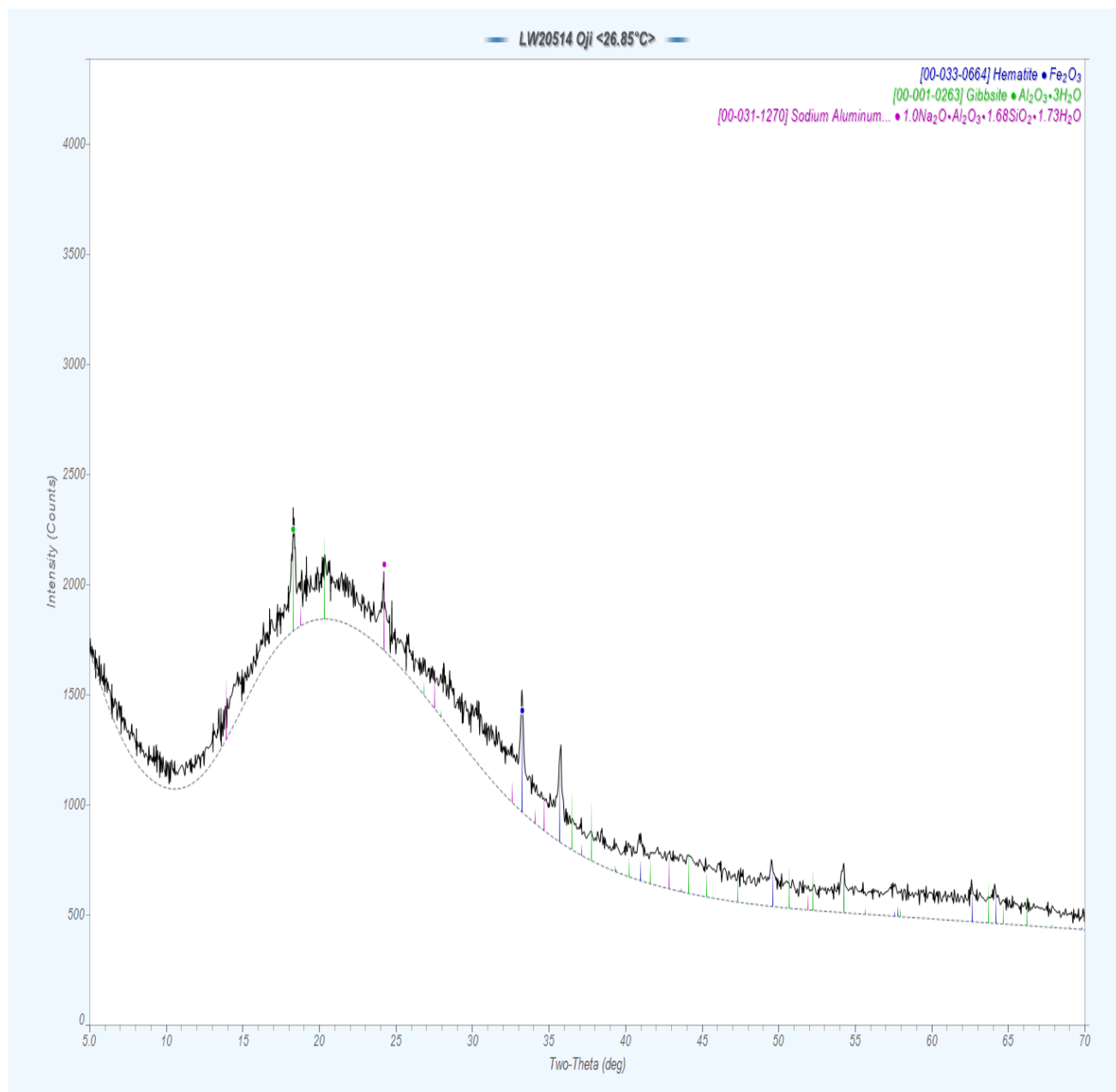
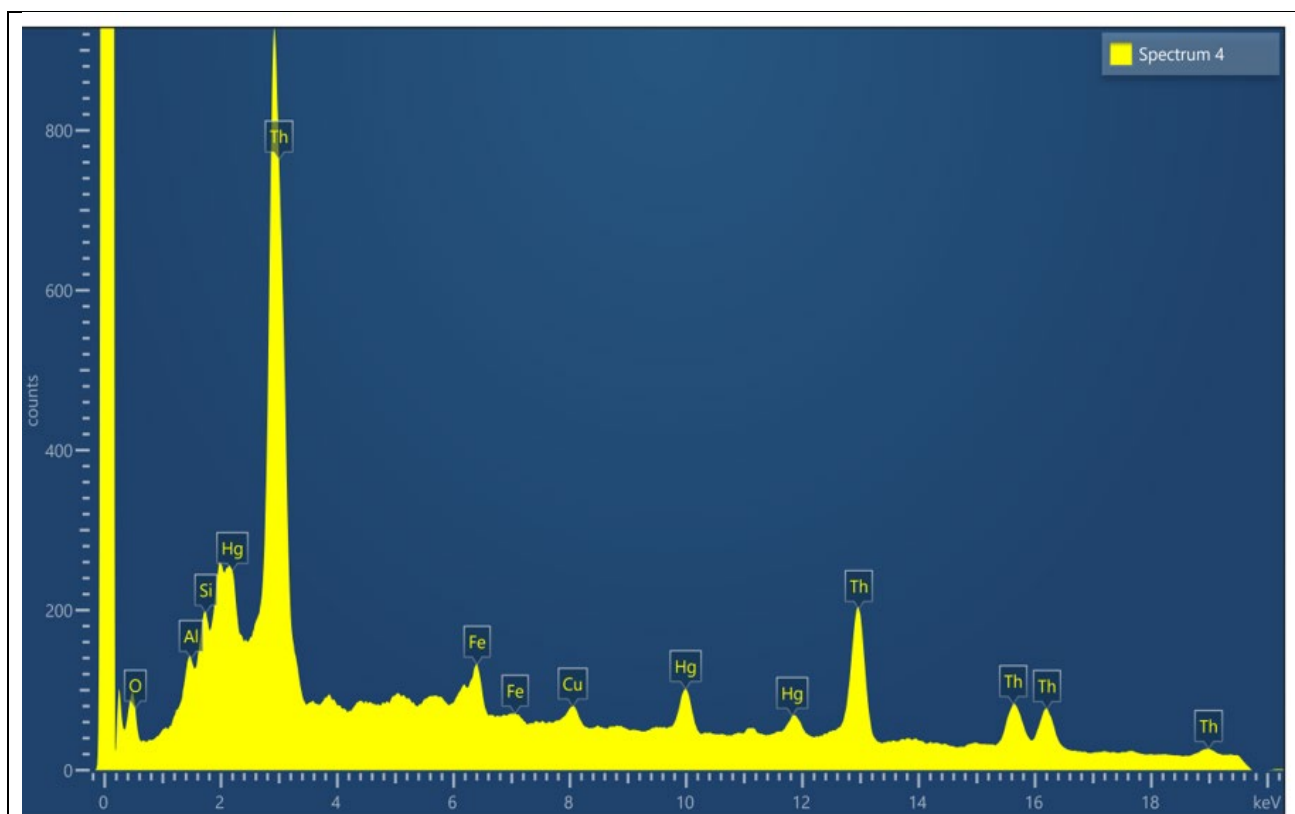
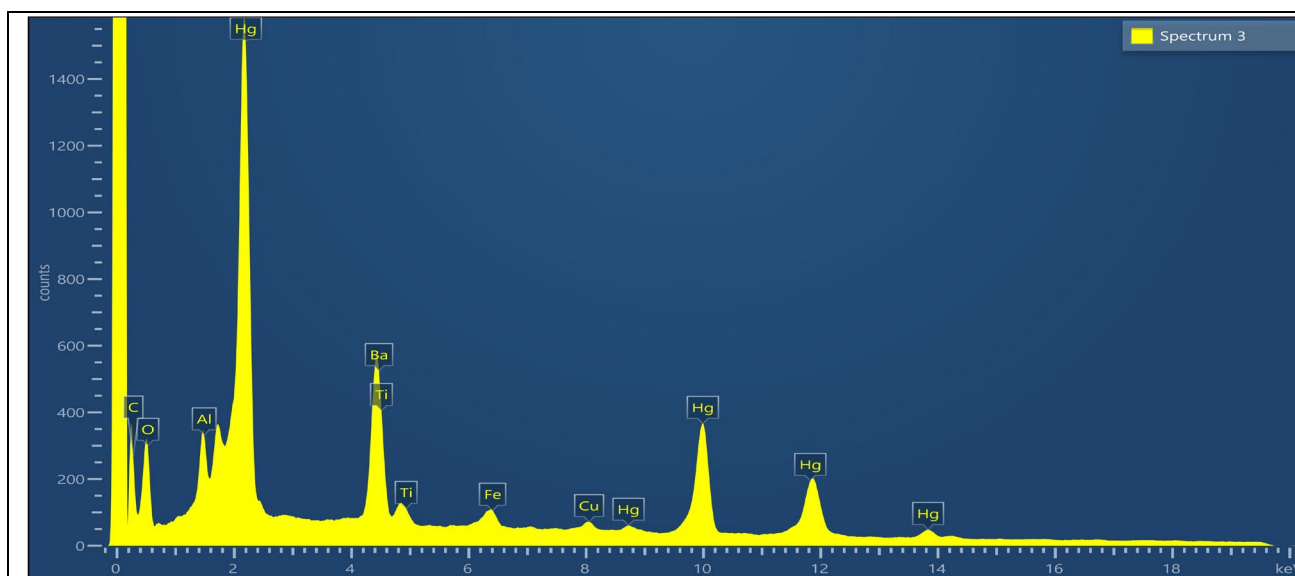


Figure 2. XRD Spectra for the SMECT Solid Fraction



Elemental composition includes aluminum (Al), silicon (Si), mercury (Hg), iron (Fe), copper (Cu), and thorium (Th).

Figure 3a. SEM/EDX Elemental Composition for the SMECT Solid Fraction.



Elemental composition also includes barium (Ba), and titanium (Ti).

Figure 3b. SEM/EDX Elemental Composition for the SMECT Solid Fraction-Continued.

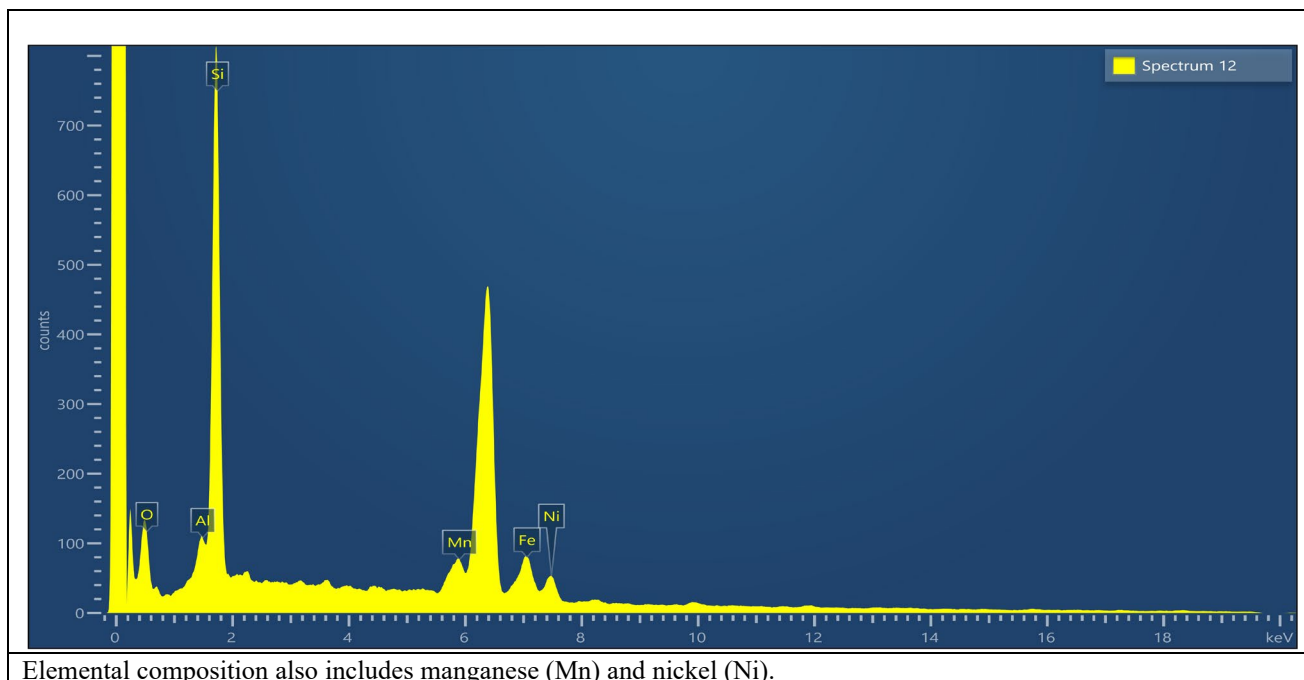


Figure 3c. SEM/EDX Elemental Composition for the SMECT Solid Fraction-Continued.

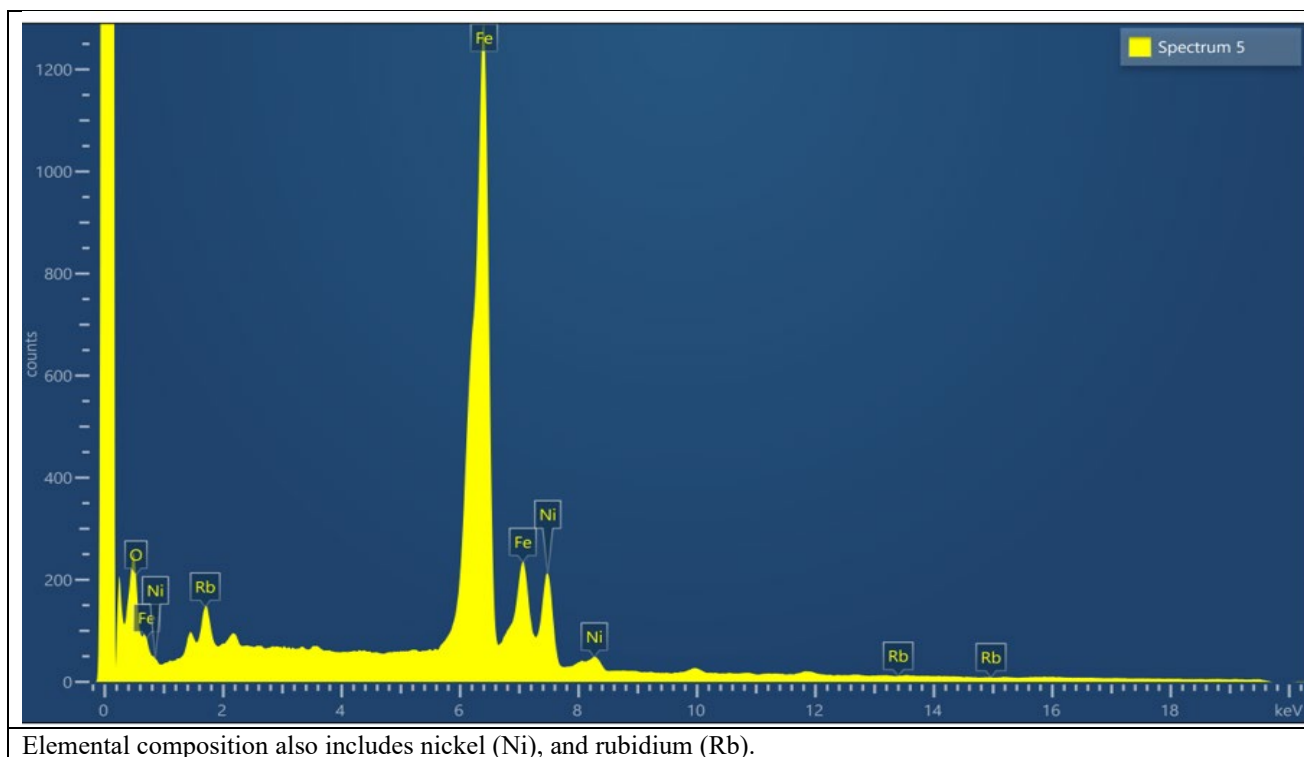


Figure 4a. SEM/EDX Elemental Composition for the SMECT Solid Fraction.

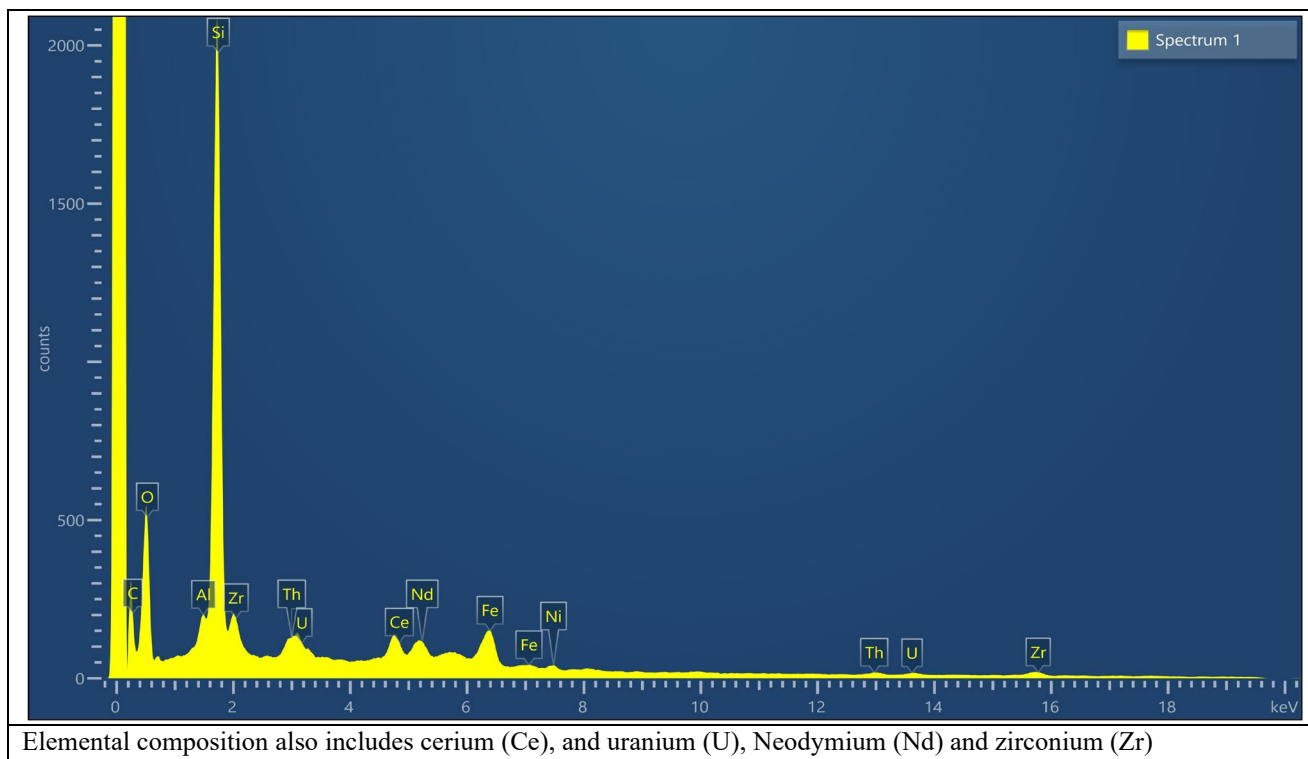


Figure 4b. SEM/EDX Elemental Composition for the SMECT Solid Fraction-Continued.

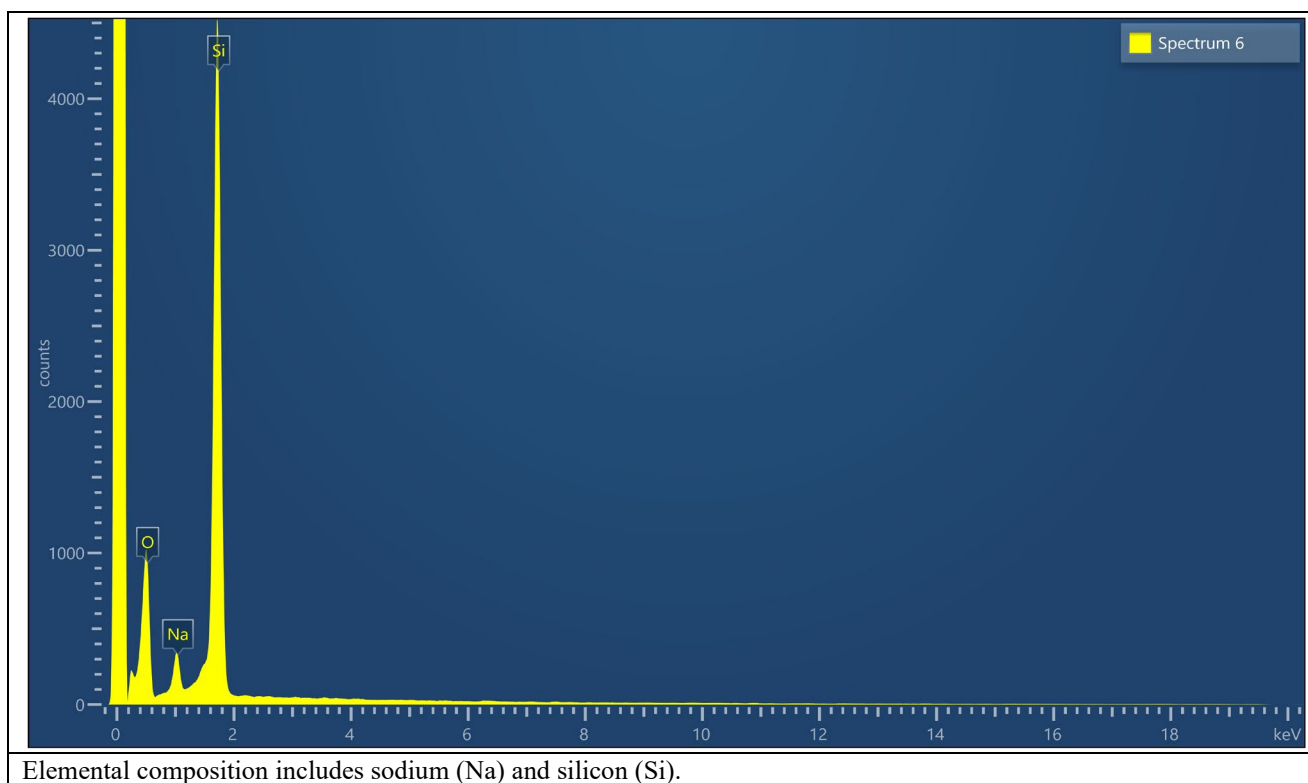


Figure 4c. SEM/EDX Elemental Composition for the SMECT Solid Fraction-Continued.

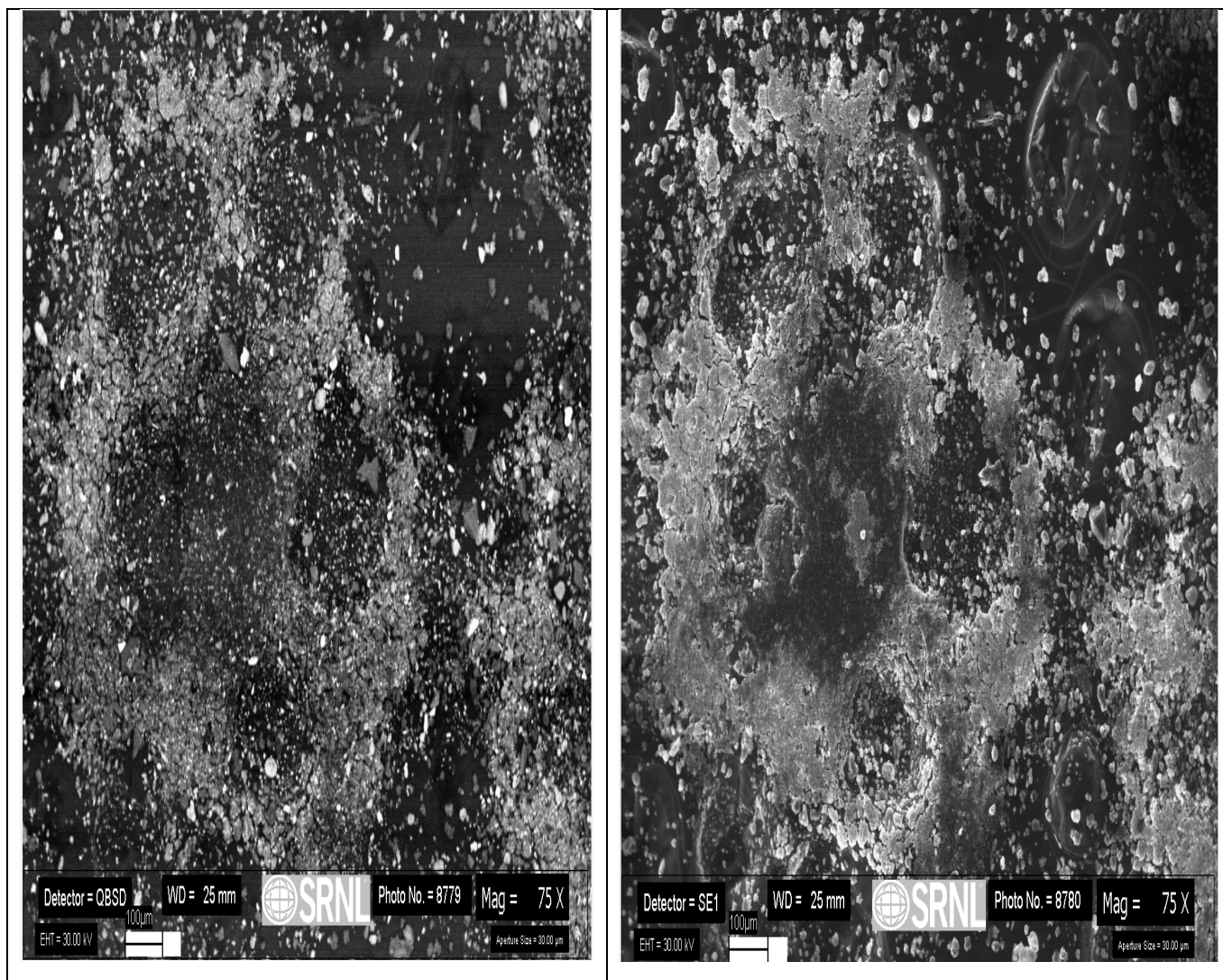


Figure 5. Scanning Electron Microscope Images for the “as-received” SMECT Solid Fractions

The scanning electron microscopy/ energy dispersive x-ray (SEM/EDX) characterization images for the SMECT solid fraction are presented in Figures 3-5. The identified elemental components of the SMECT solids include aluminum, silicon, mercury, iron, copper, thorium, nickel, barium, neodymium, titanium, sodium, cerium, manganese, zirconium, uranium, and rubidium.

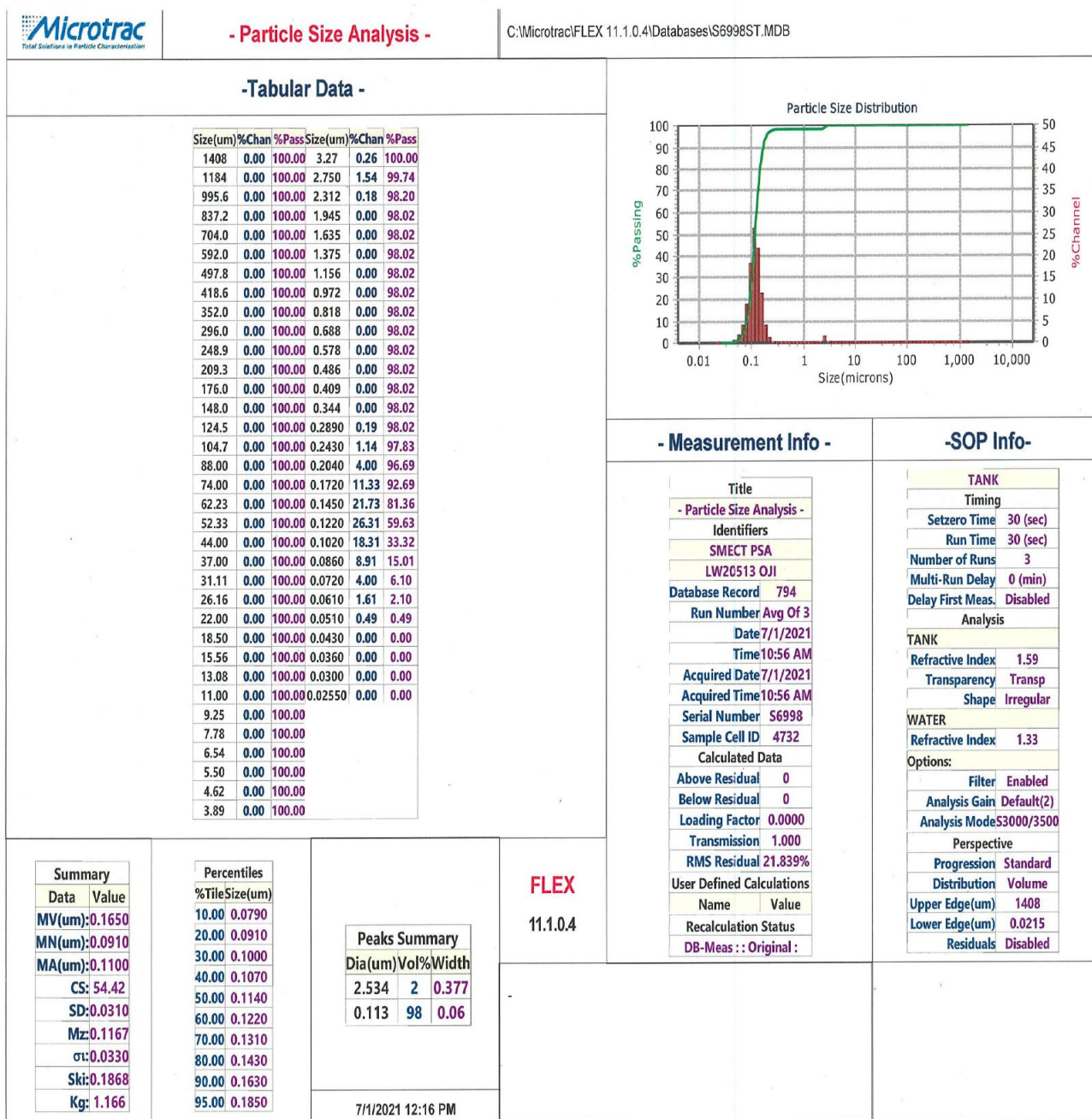


Figure 6. Particle size distribution: “as-received” SPECT Sample.

The PSD result for the “as-received” SPECT sample is provided in Figure 6. The SPECT sample PSD shows a unimodal and symmetric distribution of particles lying between 0.09 microns and 0.25 microns with an average particle size of 0.17 ± 0.03 microns (1 sigma standard deviation). The integrated peak summary volume percent is 2.0 % for particles with diameter greater than 2.53 microns and less than 98 % for particle diameters less than 0.11 microns.

4.7 Mass Spectra Analysis: Mass 59-252

The “as-received” SMECT sample, its filtrate and the diluting acid blanks were analyzed by mass spectrophotometer (masses 59-252), as presented in Tables 11 and 12. The last column in each table contains information on the most likely element or elements with that atomic mass. The assigning of “likely element(s)” for any atomic mass in the ICP-MS data in the tables are based on the nuclide isotopic abundance, atomic weight, and half-lives. Some isotopic masses, for example masses 127 (iodine), 190 and 192 (osmium), 197 (gold) and 199-202 (Hg), require special method development efforts for their isolation and quantification. Therefore, these methods are not within the analytical capabilities of the ICP-MS method employed here. The ICP-MS data for the “as-received” SMECT sample analysis, Table 11, and the continuations, shows that analytical results for the following masses 59, 85, 87-99, 101-104, 107, 109, 111-114, 116-120, 123, 128, 130, 133-148, 150-158, 160, 182, 184, 186, 196, 198, 203-204, 206-208, 232, and 234-240 are above instrument detection limits and blank concentrations, while the other masses not cited above were below instrument detection limits for those masses but above reagent blank concentrations for the corresponding masses.

The ICP-MS characterization for the SMECT filtrate samples is shown in Table 12 and continuations. Masses above instrument and blank detection limits include the following masses 59, 85, 87-90, 92-94, 99, 101-104, 107, 109, 111-112, 114, 116, 118-121, 130, 133, 135-148, 150, 152, 154-158, 160, 196, 198, 203-208, and 232-239. The other masses for the SMECT filtrate are below instrument detection limits but above reagent blank concentrations for the corresponding masses. The blank data for masses 93, 133, 138, 206-208 are above instrument detection level but were still lower than the corresponding mass concentrations for the SMECT filtrate.

The ICP-MS analysis of the “as-received” SMECT sample slurry indicated the presence at levels above the detection limit of Co, Rb, Sr, Y, Nb, Zr, Mo, Tc, Ru, Rh, Pd, Ag, Cd, Sn, In, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Pm, Sm, Ti, Eu, Gd, Dy, Tb, Hf, Os, W, Hg, Pt, Tl, Pb, Th, U, Np, and Pu. Analysis of the filtrate indicated the presence of soluble Co, Rb, Sr, Y, Mo, Nb, Zr, Tc, Ru, Pd, Rh, Ag, Cd, Sn, Sb, Te, Ba, Cs, La, Ce, Pr, Nd, Pm, Sm, Ti, Gd, Eu, Dy, Tb, Hg, Pt, Tl, Pb, Th, U, Np, and Pu.

The concentration of Tc-99, averaging 2.31E-03 mg/L (4.1 %RSD) in the “as-received” SMECT sample, dropped to a concentration of 2.04E-03 mg/L (6.7 %RSD) in the SMECT filtrate, which is about 12 % lower than its initial concentration in the “as-received” SMECT sample. The concentrations for the actinides [Th-232, U-234, U-235, U-236, Np-237, U-238, and mass 239-40] in the “as-received” SMECT sample were all above instrument detection limits. Only the concentration for U-233 was below instrument detection limit in the “as-received” SMECT sample.

The average concentration of the actinides was again above instrument detection limit in the SMECT filtrate except for mass 240; the average concentrations for mass 240 was now below instrument detection limit (<1.70E-03 mg/L). However, the average concentration of some other actinides in the SMECT filtrate, Th-232, Np-237, and Pu-239, had dropped by 75%, 26%, and 63% in concentration, respectively, when compared to their average concentrations in the “as-received” SMECT sample (Table 11). Therefore, the filtration process used to generate the SMECT filtrate sample may have resulted in the drastic reduction of the concentrations for Th-232, Np-237, Pu-239 and mass 240 in the SMECT filtrate sample. The analytes may have been trapped on the filter membrane as part of the solid fractions.

Statistically, there are no measurable differences in the average concentrations for U-235, U-236, and U-238 in the SMECT filtrate in comparison with their corresponding concentrations in the “as-received” SMECT sample; the concentration differences were less than or equal to 5%, which is within the analytical uncertainty for these measurements. However, the average concentrations for U-233 ($<1.70\text{E-}03$ mg/L) and U-234 ($<1.70\text{E-}03$ mg/L) in the SMECT filtrate and U-233 ($<8.54\text{E-}04$ mg/L) in the “as-received” SMECT sample were below instrument detection limit, while U-234 concentration ($9.16\text{E-}04$ mg/L, 6.4 %RSD) in the “as-received” SMECT samples was above instrument detection limit. This unusual analytical results for U-233 in the SMECT filtrate and the “as-received” SMECT sample may be attributed to dilution effects and possibly to incomplete dissolution in dilute nitric acid of solids in the “as-received” SMECT sample.

Table 11. Mass Spectral Analytical Results: “as-received” SMECT Sample Slurry (batch 4638)

ICP-MS, m/z	BLANK, mg/L	Analysis-1, mg/L	Analysis 2, mg/L	Analysis 3, mg/L	Average, mg/L	% RSD N = 3	Likely element (s)
59	< 1.00E-04	1.25E-02	1.24E-02	1.22E-02	1.23E-02	1.42	Co
84	< 1.00E-04	< 8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Sr
85	< 1.00E-04	1.66E-02	1.65E-02	1.62E-02	1.65E-02	1.3	Rb
86	< 1.00E-04	< 1.71E-03	<1.71E-03	<1.71E-03	<1.71E-03	na	Sr
87	< 1.00E-04	1.09E-02	1.04E-02	9.50E-03	1.03E-02	6.8	Rb, Sr
88	< 1.00E-04	2.04E-02	1.98E-02	1.96E-02	1.99E-02	2.0	Sr
89	< 1.00E-04	1.86E-02	1.73E-02	1.98E-02	1.86E-02	6.7	Y
90	< 2.50E-04	2.64E-02	2.48E-02	2.60E-02	2.57E-02	3.4	Zr, Sr
91	< 1.00E-04	2.57E-02	2.16E-02	2.81E-02	2.51E-02	13.0	Zr
92	< 1.00E-04	2.63E-02	2.33E-02	2.79E-02	2.58E-02	8.9	Zr, Mo
93	6.40E-04	3.35E-02	2.93E-02	3.55E-02	3.28E-02	9.7	Nb
94	< 1.00E-04	2.84E-02	2.49E-02	2.90E-02	2.74E-02	8.1	Nb, Mo
95	< 1.00E-04	4.57E-03	3.83E-03	4.09E-03	4.16E-03	9.0	Mo
96	< 1.00E-04	2.60E-02	2.21E-02	2.48E-02	2.43E-02	8.2	Ru, Zr, Mo
97	< 1.00E-04	3.29E-03	2.78E-03	3.23E-03	3.10E-03	9.0	Mo, Tc
98	< 1.00E-04	5.04E-03	5.75E-03	5.60E-03	5.46E-03	6.8	Ru, Mo, Tc
99	< 1.00E-04	2.41E-03	2.26E-03	2.25E-03	2.31E-03	4.1	Tc, Ru
100	< 1.00E-04	< 8.54E-03	< 8.54E-03	< 8.54E-03	< 8.54E-03	na	Ru, Mo
101	< 1.00E-04	2.73E-02	2.46E-02	2.57E-02	2.59E-02	5.21	Ru
102	< 1.00E-04	2.64E-02	2.17E-02	2.34E-02	2.38E-02	9.91	Ru, Pd
103	< 1.00E-04	1.32E-02	1.16E-02	1.15E-02	1.21E-02	8.13	Rh
104	< 1.00E-04	1.47E-02	1.47E-02	1.55E-02	1.50E-02	3.11	Ru, Pd
105	< 1.00E-04	< 8.54E-03	<8.54E-03	<8.54E-03	<8.54E-03	na	Pd
106	< 1.00E-04	< 8.54E-03	<8.54E-03	<8.54E-03	<8.54E-03	na	Pd, Cd
107	< 1.00E-04	2.65E-02	2.95E-02	2.70E-02	2.76E-02	5.80	Ag
108	< 1.00E-04	< 8.54E-03	<8.54E-03	<8.54E-03	<8.54E-03	na	Pd, Cd
109	< 1.00E-04	3.00E-02	3.25E-02	3.05E-02	3.10E-02	4.24	Ag
110	< 1.00E-04	< 8.54E-03	<8.54E-03	<8.54E-03	<8.54E-03	na	Pd, Cd
111	< 1.00E-04	2.24E-03	2.18E-03	2.32E-03	2.25E-03	3.3	Cd
112	< 1.00E-04	4.16E-03	4.21E-03	3.77E-03	4.04E-03	5.92	Sn, Cd
113	< 5.00E-04	1.42E-03	1.87E-03	1.48E-03	1.59E-03	15.6	In, Cd
114	< 1.00E-04	5.01E-03	4.90E-03	5.03E-03	4.98E-03	1.4	Sn, Cd
116	< 1.50E-04	3.39E-02	3.07E-02	3.26E-02	3.24E-02	5.0	Sn, Cd
117	< 1.00E-04	8.75E-04	9.00E-04	8.54E-04	8.76E-04	2.7	Sn
118	< 3.00E-04	2.51E-03	2.73E-03	2.18E-03	2.48E-03	11.2	Sn
119	< 1.00E-04	4.79E-02	4.64E-02	4.61E-02	4.68E-02	2.1	Sn
120	< 4.00E-04	3.17E-03	3.34E-03	3.27E-03	3.26E-03	2.7	Sn
121	< 9.00E-04	<4.27E-03	<4.27E-03	<4.27E-03	<4.27E-03	na	Sb
122	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Te, Sn
123	< 7.00E-04	2.38E-03	2.35E-03	2.54E-03	2.43E-03	4.3	Sb, Te
124	< 1.50E-04	<4.27E-03	<4.27E-03	<4.27E-03	<4.27E-03	na	Te, Sn
125	< 2.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Sb, Te
126	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Te
128	< 1.00E-04	4.50E-03	3.06E-03	3.95E-03	3.83E-03	19.0	Te
130	< 1.00E-04	1.96E-02	1.77E-02	1.88E-02	1.87E-02	5.2	Te
133	5.84E-04	1.02E-01	9.82E-02	9.65E-02	9.88E-02	2.71	Cs
134	< 1.00E-04	1.46E-03	1.50E-03	1.62E-03	1.53E-03	5.4	Ba, Cs

Table 11 Continued. Mass Spectral Analytical Results: “as-received” SMECT Sample Slurry (batch 4638)

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3	Likely element (s)
135	< 1.00E-04	3.58E-03	4.38E-03	3.66E-03	3.87E-03	11.5	Ba, Cs
136	< 1.00E-04	2.34E-03	2.65E-03	2.54E-03	2.51E-03	6.3	Ce, Ba
137	< 1.00E-04	2.88E-02	2.87E-02	2.78E-02	2.84E-02	2.0	Cs, Ba, La
138	2.37E-04	5.35E-02	5.49E-02	5.30E-02	5.38E-02	1.8	Ba, La, Ce
139	< 1.00E-04	4.64E-02	4.35E-02	4.58E-02	4.52E-02	3.3	La
140	< 1.00E-04	1.98E-01	1.79E-01	2.03E-01	1.93E-01	6.8	Ce
141	< 1.00E-04	4.07E-02	3.71E-02	3.92E-02	3.90E-02	4.7	Pr
142	< 1.00E-04	6.49E-02	5.87E-02	6.75E-02	6.37E-02	7.1	Nd, Ce
143	< 1.00E-04	3.87E-02	3.65E-02	3.83E-02	3.78E-02	3.1	Nd., Pm
144	< 1.00E-04	4.08E-02	3.74E-02	4.00E-02	3.94E-02	4.6	Nd, Sm, Pm
145	< 1.00E-04	2.72E-02	2.60E-02	2.73E-02	2.68E-02	2.61	Nd, Pm
146	< 1.00E-04	2.32E-02	2.16E-02	2.35E-02	2.28E-02	4.6	Nd, Sm
147	< 1.00E-04	1.40E-02	1.35E-02	1.44E-02	1.40E-02	3.3	Sm, Ti
148	< 1.00E-04	1.37E-02	1.34E-02	1.38E-02	1.37E-02	1.7	Nd, Gd, Sm
149	< 1.00E-04	<8.54E-04	<8.54E-04	<8.73E-04	<8.60E-04	na	Sm
150	< 1.00E-04	1.30E-02	1.23E-02	1.29E-02	1.27E-02	2.6	Nd, Gd, Sm, Eu
151	< 1.00E-04	1.16E-03	1.06E-03	1.13E-03	1.12E-03	4.6	Eu
152	< 1.00E-04	4.66E-03	4.14E-03	4.43E-03	4.41E-03	5.9	Gd, Sm, Eu
153	< 1.00E-04	1.77E-03	1.59E-03	1.89E-03	1.75E-03	8.7	Eu
154	< 1.00E-04	4.02E-03	3.71E-03	3.75E-03	3.83E-03	4.4	Gd, Sm, Eu, Dy
155	< 1.00E-04	1.61E-02	1.57E-02	1.61E-02	1.60E-02	1.7	Gd
156	< 1.00E-04	2.42E-02	2.33E-02	2.32E-02	2.36E-02	2.3	Gd, Dy
157	< 1.00E-04	1.76E-02	1.71E-02	1.70E-02	1.72E-02	1.9	Gd, Tb
158	< 1.00E-04	2.87E-02	2.72E-02	2.81E-02	2.80E-02	2.8	Gd, Dy, Tb
159	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Tb
160	< 1.00E-04	2.57E-02	2.48E-02	2.49E-02	2.51E-02	2.1	Gd, Dy
161	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Dy
162	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Dy, Er
163	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Dy, Ho
164	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Dy, Er
165	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Ho
166	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Er, Ho
167	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Er
168	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Er, Yb
169	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Tm
170	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Er, Yb
171	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Yb
172	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Yb
173	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Yb
174	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Yb, Hf
175	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Lu
176	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Lu, Hf, Yb
177	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Hf
178	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Hf
179	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Hf
180	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Hf, W, Ta
181	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Ta
182	< 1.00E-04	1.41E-03	1.48E-03	1.43E-03	1.44E-03	2.3	Hf, W

Table 11 Continued. Mass Spectral Analytical Results: “as-received” SMECT Sample Slurry (batch 4638)

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3	Likely element (s)
183	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	W
184	< 1.00E-04	1.62E-03	1.44E-03	1.59E-03	1.55E-03	6.3	W
185	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Re
186	< 1.00E-04	1.36E-03	1.29E-03	1.45E-03	1.37E-03	6.0	Os, W
187	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Re, Os
191	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Ir
193	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Ir, Pt
194	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pt
195	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pt
196	< 1.00E-04	7.21E-01	7.37E-01	7.11E-01	7.23E-01	1.9	Hg, Pt
198	< 4.00E-04	4.74E+01	4.85E+01	4.66E+01	4.75E+01	2.0	Hg, Pt
203	< 1.00E-04	2.67E-03	2.61E-03	2.41E-03	2.56E-03	5.3	Tl
204	< 3.00E-04	2.56E+01	2.63E+01	2.52E+01	2.57E+01	2.0	Pb, Hg
205	< 1.00E-04	<1.71E-03	<1.71E-03	<1.71E-03	<1.71E-03	na	Tl
206	1.33E-03	4.58E-02	4.47E-02	4.44E-02	4.50E-02	1.7	Pb
207	1.14E-03	4.11E-02	4.00E-02	3.96E-02	4.02E-02	1.9	Pb
208	2.77E-03	9.82E-02	9.69E-02	9.66E-02	9.72E-02	0.9	Pb
229	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Th
230	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Th
232	< 1.00E-04	1.09E+00	9.88E-01	1.10E+00	1.06E+00	5.8	Th, U
233	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	U
234	< 1.00E-04	9.82E-04	8.96E-04	8.69E-04	9.16E-04	6.4	U
235	< 1.00E-04	4.03E-02	3.96E-02	3.89E-02	3.96E-02	1.8	U
236	< 1.00E-04	2.29E-03	2.29E-03	2.25E-03	2.28E-03	1.1	U
237	< 1.00E-04	3.28E-03	3.19E-03	3.29E-03	3.25E-03	1.71	Np
238	< 1.00E-04	2.86E+00	2.87E+00	2.84E+00	2.86E+00	0.5	U, Pu
239	< 1.00E-04	1.08E-02	1.02E-02	1.10E-02	1.07E-02	3.7	Pu
240	< 1.00E-04	1.15E-03	1.07E-03	1.06E-03	1.09E-03	4.4	Pu
241	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pu, Am
242	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pu, Am
243	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pu, Cm
244	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Pu, Cm
245	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cm
246	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cm
247	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cm, Bk
248	< 1.00E-04	<2.56E-02	<2.56E-02	<2.56E-02	<2.56E-02	na	Cm
249	< 1.00E-04	<2.56E-03	<2.56E-03	<2.56E-03	<2.56E-03	na	Cf
250	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cf
251	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cf
252	< 1.00E-04	<8.54E-04	<8.54E-04	<8.54E-04	<8.54E-04	na	Cf, Cm

Table 12. Mass Spectral Analytical Results: SMECT Filtrate Sample (batch 4638)

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis 2, mg/L	Analysis 3, mg/L	Average, mg/L	% RSD, N = 3	Likely element (s)
59	< 1.00E-04	1.08E-02	1.04E-02	1.09E-02	1.07E-02	2.7	Co
84	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sr
85	< 1.00E-04	1.72E-02	1.78E-02	1.56E-02	1.69E-02	6.8	Rb
86	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sr
87	< 1.00E-04	9.26E-03	1.02E-02	1.04E-02	9.96E-03	6.2	Rb, Sr
88	< 1.00E-04	1.80E-02	1.97E-02	1.71E-02	1.83E-02	7.2	Sr
89	< 1.00E-04	1.18E-02	1.25E-02	1.15E-02	1.19E-02	4.3	Y
90	< 2.50E-04	7.41E-03	7.05E-03	6.82E-03	7.09E-03	4.2	Zr, Sr
91	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Zr
92	< 1.00E-04	2.05E-03	2.01E-03	1.75E-03	1.93E-03	8.4	Zr, Mo
93	6.40E-04	5.77E-03	5.54E-03	3.88E-03	5.06E-03	20.3	Nb
94	< 1.00E-04	1.87E-03	1.86E-03	1.85E-03	1.86E-03	0.6	Nb, Mo
95	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Mo
96	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ru, Zr, Mo
97	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Mo, Tc
98	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ru, Mo, Tc
99	< 1.00E-04	2.12E-03	2.12E-03	1.89E-03	2.04E-03	6.7	Tc, Ru
100	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ru, Mo
101	< 1.00E-04	1.71E-02	1.78E-02	1.75E-02	1.75E-02	2.1	Ru
102	< 1.00E-04	1.56E-02	1.50E-02	1.56E-02	1.54E-02	2.1	Ru, Pd
103	< 1.00E-04	1.01E-02	1.08E-02	1.04E-02	1.04E-02	3.3	Rh
104	< 1.00E-04	8.06E-03	7.99E-03	8.66E-03	8.24E-03	4.5	Ru, Pd
105	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pd
106	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pd, Cd
107	< 1.00E-04	3.07E-02	3.53E-02	3.14E-02	3.24E-02	7.6	Ag
108	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pd, Cd
109	< 1.00E-04	3.24E-02	3.67E-02	3.32E-02	3.41E-02	6.8	Ag
110	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pd, Cd
111	< 1.00E-04	1.91E-03	2.50E-03	2.02E-03	2.14E-03	14.7	Cd
112	< 1.00E-04	3.68E-03	4.42E-03	3.55E-03	3.88E-03	12.1	Sn, Cd
113	< 5.00E-04	<3.40E-03	<3.40E-03	<3.40E-03	<3.40E-03	na	In, Cd
114	< 1.00E-04	3.92E-03	5.46E-03	4.21E-03	4.53E-03	18.1	Sn, Cd
116	< 1.50E-04	1.00E-02	1.01E-02	9.63E-03	9.91E-03	2.4	Sn, Cd
117	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sn
118	< 3.00E-04	2.23E-03	3.10E-03	1.71E-03	2.35E-03	30	Sn
119	< 1.00E-04	4.49E-02	4.40E-02	4.51E-02	4.47E-02	1.3	Sn
120	< 4.00E-04	2.90E-03	3.66E-03	2.03E-03	2.86E-03	28.5	Sn
121	< 9.00E-04	2.12E-03	2.00E-03	1.98E-03	2.03E-03	3.7	Sb
122	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Te, Sn
123	< 7.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sb, Te
124	< 1.50E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Te, Sn
125	< 2.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sb, Te
126	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Te
128	< 1.00E-04	<2.55E-03	<2.55E-03	<2.55E-03	<2.55E-03	na	Te
130	< 1.00E-04	1.14E-02	1.05E-02	1.12E-02	1.10E-02	4.2	Te
133	5.84E-04	9.47E-02	9.59E-02	9.71E-02	9.59E-02	1.3	Cs
134	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ba, Cs

Table 12 Continued. Mass Spectral Analytical Results: SMECT Filtrate Sample (batch 4638)

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3	Likely element (s)
135	< 1.00E-04	3.42E-03	3.27E-03	3.54E-03	3.41E-03	3.9	Ba, Cs
136	< 1.00E-04	<1.70E-03	1.71E-03	<1.70E-03	<1.71E-03	na	Ce, Ba
137	< 1.00E-04	2.38E-02	2.47E-02	2.38E-02	2.41E-02	2.2	Cs, Ba, La
138	2.37E-04	3.81E-02	3.80E-02	3.72E-02	3.78E-02	1.3	Ba, La, Ce
139	< 1.00E-04	3.14E-02	3.16E-02	3.10E-02	3.14E-02	0.9	La
140	< 1.00E-04	3.63E-02	3.72E-02	3.66E-02	3.67E-02	1.1	Ce
141	< 1.00E-04	2.39E-02	2.37E-02	2.39E-02	2.39E-02	0.4	Pr
142	< 1.00E-04	1.32E-02	1.32E-02	1.25E-02	1.29E-02	2.9	Nd, Ce
143	< 1.00E-04	2.29E-02	2.31E-02	2.37E-02	2.32E-02	1.7	Nd., Pm
144	< 1.00E-04	2.34E-02	2.32E-02	2.27E-02	2.31E-02	1.6	Nd, Sm, Pm
145	< 1.00E-04	1.70E-02	1.72E-02	1.75E-02	1.72E-02	1.3	Nd, Pm
146	< 1.00E-04	1.40E-02	1.48E-02	1.43E-02	1.44E-02	3.1	Nd, Sm
147	< 1.00E-04	8.60E-03	8.97E-03	8.84E-03	8.80E-03	2.1	Sm, Ti
148	< 1.00E-04	8.44E-03	8.08E-03	8.44E-03	8.32E-03	2.5	Nd, Gd, Sm
149	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Sm
150	< 1.00E-04	7.84E-03	8.15E-03	7.64E-03	7.88E-03	3.3	Nd, Gd, Sm, Eu
151	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Eu
152	< 1.00E-04	2.74E-03	2.85E-03	2.71E-03	2.77E-03	2.7	Gd, Sm, Eu
153	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Eu
154	< 1.00E-04	3.13E-03	3.26E-03	2.92E-03	3.10E-03	5.4	Gd, Sm, Eu, Dy
155	< 1.00E-04	1.34E-02	1.39E-02	1.40E-02	1.38E-02	2.2	Gd
156	< 1.00E-04	1.92E-02	1.96E-02	1.92E-02	1.93E-02	1.3	Gd, Dy
157	< 1.00E-04	1.43E-02	1.44E-02	1.43E-02	1.43E-02	0.3	Gd, Tb
158	< 1.00E-04	2.36E-02	2.41E-02	2.32E-02	2.36E-02	1.8	Gd, Dy, Tb
159	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Tb
160	< 1.00E-04	2.08E-02	2.13E-02	2.06E-02	2.09E-02	1.8	Gd, Dy
161	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Dy
162	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Dy, Er
163	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Dy, Ho
164	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Dy, Er
165	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ho
166	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Er, Ho
167	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Er
168	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Er, Yb
169	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Tm
170	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Er, Yb
171	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Yb
172	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Yb
173	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Yb
174	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Yb, Hf
175	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Lu
176	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Lu, Hf, Yb
177	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Hf
178	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Hf
179	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Hf
180	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Hf, W, Ta
181	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ta
182	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Hf, W

Table 12 Continued. Mass Spectral Analytical Results: SMECT Filtrate Sample (batch 4638)

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3	Likely element (s)
183	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	W
184	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	W
185	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Re
186	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Os, W
187	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Re, Os
191	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ir
193	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Ir, Pt
194	< 1.00E-04	<3.40E-03	<3.40E-03	<3.40E-03	<3.40E-03	na	Pt
195	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pt
196	< 1.00E-04	6.14E-01	6.80E-01	6.96E-01	6.63E-01	6.6	Hg, Pt
198	< 4.00E-04	7.60E+00	1.02E+01	1.45E+01	1.08E+01	32.5	Hg, Pt
203	< 1.00E-04	2.32E-03	2.72E-03	2.53E-03	2.52E-03	7.8	Tl
204	< 3.00E-04	4.24E+00	5.63E+00	8.08E+00	5.99E+00	32.5	Pb, Hg
205	< 1.00E-04	<2.55E-03	<2.55E-03	<2.55E-03	<2.55E-03	na	Tl
206	1.33E-03	4.19E-02	4.31E-02	4.12E-02	4.21E-02	2.3	Pb
207	1.14E-03	3.63E-02	3.88E-02	3.74E-02	3.75E-02	3.7	Pb
208	2.77E-03	8.77E-02	9.01E-02	8.82E-02	8.87E-02	1.4	Pb
229	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Th
230	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Th
232	< 1.00E-04	2.61E-01	2.64E-01	2.56E-01	2.60E-01	1.6	Th, U
233	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	U
234	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	U
235	< 1.00E-04	3.87E-02	4.02E-02	3.80E-02	3.90E-02	2.9	U
236	< 1.00E-04	2.12E-03	2.22E-03	2.14E-03	2.16E-03	2.4	U
237	< 1.00E-04	2.39E-03	2.38E-03	2.44E-03	2.41E-03	1.4	Np
238	< 1.00E-04	2.82E+00	2.83E+00	2.82E+00	2.82E+00	0.2	U, Pu
239	< 1.00E-04	3.98E-03	3.89E-03	3.87E-03	3.91E-03	1.4	Pu
240	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pu
241	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pu, Am
242	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pu, Am
243	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pu, Cm
244	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Pu, Cm
245	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cm
246	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cm
247	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cm, Bk
248	< 1.00E-04	<5.95E-03	<5.95E-03	<5.95E-03	<5.95E-03	na	Cm
249	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cf
250	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cf
251	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cf
252	< 1.00E-04	<1.70E-03	<1.70E-03	<1.70E-03	<1.70E-03	na	Cf, Cm

5.0 Conclusions

The “as-received” DWPF SMECT sample, batch 4638, has been characterized for select analytes and radionuclides as requested by SRR (now SRMC) and described in the TTR². This sample was delivered to SRNL Shielded Cells in March 2021. The SMECT characterization data will be used as input to the DWPF Recycle Diversion Project.

A summary of the analytical results for the SMECT sample follows.

- The densities of the “as-received” SMECT sample and the SMECT filtrate averaged 1.01 g/mL, [0.4 %RSD] and 1.00 g/mL, [0.1 %RSD], respectively.
- The pH of the “as-received” SMECT sample was 1.63 and that of the SMECT filtrate was 1.63.
- The turbidity of the “as-received” SMECT slurry averaged 139 NTU (1.1 %RSD) and that of the SMECT filtrate averaged 8.5 NTU (0.3 %RSD). These viscosity values for the “as-received” SMECT sample and the SMECT filtrate are not different from that of ordinary water at 25°C.
- The SMECT sample weight percent total solids, dissolved solids, calculated weight percent insoluble solids and soluble solids are 0.11 wt.%, (25.7 %RSD), <0.02 wt.%, (MDL), <0.09 wt.% and <0.02 wt.%, respectively.
- The SMECT PSD shows a unimodal and symmetric distribution of particles lying between 0.09 microns and 0.25 microns with an average particle size of 0.17 ± 0.03 microns (1 sigma standard deviation).
- The X-ray diffraction (XRD) mineral compositions for the small and fine SMECT solids include mainly hematite (Fe_2O_3), gibbsite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) and generally sodium aluminosilicate complexes, for example $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 3\text{H}_2\text{O}$.
- SEM/EDX information shows that the principal elemental constituents present in the “as-received” SMECT solid fraction include aluminum, silicon, mercury, iron, copper, thorium, nickel, titanium, barium, sodium, cerium, manganese, zirconium, uranium, rubidium, and neodymium.
- Total beta activity in the “as-received” SMECT sample averaged $4.65\text{E}+06$ dpm/mL (1.6 %RSD) with an average activity concentration of $2.09\text{E}-03$ Ci/L. The average total alpha activity was below instrument detection limit at $< 3.64\text{E}+05$ dpm/mL ($<1.64\text{E}-04$ Ci/L).
- The primary beta emitting radionuclides in the “as-received” SMECT sample include Sr-90, Y-90 and Cs-137 at average activity of $1.70\text{E}+06$ dpm/mL, 8.0 %RSD ($7.64\text{E}-04$ Ci/L), $1.70\text{E}+06$ dpm/mL, 8.0 %RSD ($7.64\text{E}-04$ Ci/L), and $1.32\text{E}+06$ dpm/mL, 2.5 %RSD ($5.95\text{E}-04$ Ci/L), respectively.
- The primary gamma emitting radionuclide in the “as-received” SMECT sample is Ba-137m at average activity of $1.25\text{E}+06$ dpm/mL, 2.5 %RSD ($5.63\text{E}-04$ Ci/L) (note that the activity concentration for Ba-137m was calculated as 94.7% the Cs-137 activity concentration).
- The average activities for Ni-63, Eu-154 and Th-232 in the “as-received” SMECT sample were all above instrument detection limits and measured for Ni-63 ($1.20\text{E}+04$ dpm/mL, $5.42\text{E}-06$ Ci/L, 15.7 %RSD), Eu-154 ($1.05\text{E}+03$ dpm/mL, $4.74\text{E}-07$ Ci/L, 6.2 %RSD), and Th-232 ($2.58\text{E}-01$ dpm/mL, $1.16\text{E}-10$ Ci/L, 5.8 %RSD).
- Technetium-99 and Cs-135 activities in the “as-received” SMECT sample averaged $8.68\text{E}+01$ dpm/mL, 4.1 %RSD ($3.91\text{E}-08$ Ci/L) and $7.15\text{E}-01$ dpm/mL, 6.7 %RSD ($3.22\text{E}-10$ Ci/L), respectively.
- Average actinide activities above instrument detection limits in the “as-received” SMECT sample include U-235 ($1.90\text{E}-01$ dpm/mL, $8.56\text{E}-11$ Ci/L, 1.8% RSD), U-236 ($3.27\text{E}-01$ dpm/mL, $1.47\text{E}-10$ Ci/L, 1.1 %RSD), Np-237 ($5.09\text{E}+00$ dpm/mL, $2.29\text{E}-09$ Ci/L, 1.7 %RSD) and U-238 ($2.13\text{E}+00$ dpm/mL, $9.60\text{E}-10$ Ci/L, 0.5 %RSD).

- Iodine-129 activity in the “as-received” SMECT sample averaged $5.39\text{E}+01$ dpm/mL, $2.43\text{E}-08$ Ci/L, (1.4 %RSD), while the activity for Am-241 averaged $3.95\text{E}+03$ dpm/mL, $1.78\text{E}-06$ Ci/L (12.6 %RSD).
- The activities for Pu-238, Pu-239/240 and Pu-241 in the “as-received” SMECT sample averaged $3.27\text{E}+04$ dpm/mL, $1.47\text{E}-05$ Ci/L (12.6 %RSD), $1.95\text{E}+03$ dpm/mL, $8.77\text{E}-07$ Ci/L (14.9 %RSD), and $5.84\text{E}+03$ dpm/mL, $2.63\text{E}-06$ Ci/L (5.0 %RSD).

The average activities for total beta, Ni-63, Sr-90, Eu-154, and Th-232 in the filtrate SMECT sample all showed a significant decrease in activity in comparison to their initial activities in the “as-received” SMECT sample. Total beta average activity dropped by 17% in the filtrate; going from an average activity of $4.65\text{E}+06$ dpm/mL in the “as-received” SMECT sample to $3.86\text{E}+06$ dpm/mL in the SMECT filtrate. Nickel-63, Sr-90, Th-232, and Eu-154 average activities in the SMECT filtrate dropped by 33 %, 28%, 75%, and 43%, respectively. Their average activities in the “as-received” SMECT sample dropped from $1.20\text{E}+04$ dpm/mL to $8.10\text{E}+03$ dpm/mL, $1.70\text{E}+06$ dpm/mL to $1.22\text{E}+06$ dpm/mL, $2.58\text{E}-01$ dpm/mL to $6.34\text{E}-02$ dpm/mL, and $1.05\text{E}+03$ dpm/mL to $5.94\text{E}+02$ dpm/mL in the filtrate, respectively.

The average activities for some uranium isotopes (U-235, U-236 and U-238) did not show significant changes in the SMECT filtrate sample when compared to their average activities in the “as-received” SMECT sample. Uranium-235 average activity in the “as-received” SMECT was nearly equal to its activity in the SMECT filtrate. Uranium-236 and U-238 average activity percent changes in the “as-received” SMECT were within the analytical percent relative standard deviation for these measurements and averaged less than 10%; respectively, 5.2% and 0.9%.

Neptunium-237 average activity in the SMECT filtrate sample dropped by 26% when compared to its average activity in the “as-received” SMECT sample; its average activity dropped from $5.09\text{E}+00$ dpm/mL to $3.77\text{E}+00$ dpm/mL in the SMECT filtrate.

The average activities for I-129 and Cs-137 in the “as-received” SMECT sample was comparable to their average activities in the SMECT filtrate sample; their average activity changes in the “as-received” SMECT sample were $5.39\text{E}+01$ dpm/mL to $5.46\text{E}+01$ dpm/mL and $1.32\text{E}+06$ dpm/mL to $1.27\text{E}+06$ dpm/mL in the SMECT filtrate, respectively.

The average activities for the plutonium isotopes (Pu-238, Pu-239/240 and Pu-241) in the “as-received” SMECT sample dropped significantly in the SMECT filtrate sample. Plutonium-238, Pu-239/240, and Pu-241 average activities in the SMECT filtrate dropped by 76%, 45% [absolute value for the activity average used for Pu-239/240 in the filtrate], and 42%, respectively. Their average activities in the “as-received” SMECT sample dropped from $3.27\text{E}+04$ dpm/mL to $7.97\text{E}+03$ dpm/mL, $1.95\text{E}+03$ dpm/mL to $\leq 1.08\text{E}+03$ dpm/mL [two real values used], and $5.84\text{E}+03$ dpm/mL to $3.39\text{E}+03$ dpm/mL in the SMECT filtrate, respectively.

Americium-241 average activity in the SMECT filtrate sample dropped by 38% [absolute value for the activity average for Am-241 in the filtrate used] when compared to its average activity in the “as-received” SMECT sample. The average activity dropped from $3.95\text{E}+03$ dpm/mL to $\leq 2.45\text{E}+03$ dpm/mL in the SMECT filtrate.

Only the analytical results for aluminum, iron, manganese, and silicon were above instrument detection limits in the elemental characterization of the “as-received” SMECT sample at concentrations of 2.6 mg/L (3.7 %RSD), 11.1 mg/L (2.0 %RSD), 5.3 mg/L (0.4 %RSD), and 28.7 mg/L (1.1 %RSD), respectively.

The analytical results show that there were more elements with concentrations above instrument detection levels in the SMECT filtrate and these include aluminum (2.6 mg/L, 10.3 %RSD), calcium (1.5 mg/L, 1.3 %RSD), copper (0.5 mg/L, 77 %RSD), iron (8.3 mg/L, 0.4 %RSD), magnesium (0.3 mg/L, 48 %RSD), manganese (5.9 mg/L, 1.3 %RSD), sodium (18.0 mg/L, 0.9 %RSD), nickel (0.8 mg/L, 1.1 %RSD), silicon (24.7 mg/L, 0.0 %RSD), titanium (0.3 mg/L, 3.2 %RSD), uranium (2.9 mg/L, 0.4 %RSD) and zinc (0.4 mg/L, 2.5 %RSD). All other elements in both the “as-received” SMECT sample and SMECT filtrate were below instrument detection limit. Analytical results for selenium, arsenic, and sulfur were below instrument detection limits in both the “as-received” SMECT and SMECT filtrate samples.

The predominant anion species present in both the “as-received” SMECT and SMECT filtrates samples were formate and nitrate anions. The concentration for formate and nitrate anions in both the “as-received” SMECT and SMECT filtrate samples were equal in magnitude, at a concentration of 17.0 mg/L (0.3 %RSD) for the formate, and 2,400 mg/L (0.6 %RSD) for the nitrate. The average concentration for the aluminate anion ($\text{Al}(\text{OH})_4^-$) in both the “as-received” SMECT and SMECT filtrate samples were just about equal, averaging 9.46E-05 M, 0.6 %RSD and 9.52E-05 M, 10.5 %RSD, respectively. On the other hand, the average carbonate concentration in the “as-received” SMECT sample at 1.86E-03 M, 1.4 %RSD, is higher than the average carbonate concentration in the SMECT filtrate sample at 5.15E-04 M, 4.5 %RSD, which is a decrease of 72% carbonate in the filtrate. The total carbon average concentration in the “as-received” SMECT sample at 6.73E-02 M, 1.7 %RSD, is higher than the average total carbon average concentration in the SMECT filtrate sample at 3.10E-02 M, 1.2 %RSD, which is a decrease of 54% total carbon in the filtrate.

The average total acid at pH 7 (3.4E-02 M) for the “as-received” SMECT sample was statistically equivalent to the total acid at pH 7 (3.8E-02 M) for the SMECT filtrate sample. The corresponding calculated total acid results at pH 9 and 11 were also equal for these two SMECT sample types, respectively at 4.3E-02 M and 4.3E-02 M at pH 9, and 9.4E-02 M and 9.2E-02 M at pH 11. Therefore, the small fine particulates present in the “as-received” SMECT sample did not play any measurable negative role in the titration results for the “as-received” SMECT sample when compared with the SMECT filtrate sample.

Total mercury concentration in the “as-received” SMECT sample averaged 285 mg/L (26.0 %RSD) and averaged 269 mg/L (14.8 %RSD) in the SMECT filtrate sample.

Elemental mercury concentration dropped from an average high of 83 mg/L in the “as-received” SMECT supernate sample to a low of 28 mg/L in the filtrate, while ionic mercury showed about a 19% drop in concentration in the SMECT filtrate sample; ionic mercury concentration in the “as-received” SMECT sample dropped from an average of 27 mg/L to 22 mg/L in the SMECT filtrate sample.

The average methyl mercury concentration in the “as-received” SMECT sample, at 36 mg/L (3.5 %RSD), was not that different from its average concentration of 36 mg/L (7.3 %RSD) in the SMECT filtrate.

Ethyl mercury and dimethyl mercury determinations in the “as-received” SMECT and filtrate samples were below instrument detection limits. Therefore, these mercury species may not be present in the “as-received”

SMECT sample at detectable levels. Representative aliquot sampling for elemental mercury analysis becomes a problem when elemental mercury and other forms of mercury concentration are above their saturation levels in a sample media. This was the case with the “as-received” SMECT and filtrate samples. Because of the extreme saturation level for elemental mercury and other mercury forms in the SMECT samples, the analytical uncertainty measurements for elemental and ionic mercury were high. The one sigma analytical uncertainty for elemental mercury (purgeable mercury) and ionic mercury analyses were reported as 40%, which means the analytical results would be biased. As a result, there are mercury mass balance issues with the characterization of both the “as-received” and filtrate SMECT samples for total mercury and other mercury species. Additionally, the re-analyses of the SMCET samples for mercury species occurred several months after sample receipt at SRNL, and therefore the impact of storage time, prior to these re-analyses, on the magnitude of the analytical results for these species may be negative, in part, because some of these mercury species may tend to degrade over time.

Filtering of the “as-received” SMECT sample to generate the SMECT filtrate seemed to have resulted in the retention or absorption, as part of the solid fraction, of analytes (for example Pu isotopes) with particles sizes greater than 0.45 micron (membrane pore size) or those analytes which are latched onto solid particles in the “as-received” SMECT sample. This retention of the analytes resulted in the decrease in concentrations or activities of the analytes in the filtrate or liquid fraction of the liquid/solid separation process.

6.0 Quality Assurance

The Task Technical and Quality Assurance Plan details the planned activities and associated quality assurance implementing procedures for the characterization of the DWPF Recycle Diversion - Recycle Collection Tank Slurry³. The documents referenced in the TTQAP include the following: L. N. Oji: ELN: L5575-00080-16 (Electronic Notebook (Production)); SRNL, Aiken, SC 29808 (2014) and various SaM notebooks contain the analytical data. Other relevant QA documents include the Technical Task Request².

The TTR requested that a functional classification of Safety Significant Class applies to this work. Equipment with a General Service functional classification comprises the analytical measurement systems used to collect data for these characterizations. Standards used to calibrate these systems were purchased at level 2 with a certificate of analysis. Chemicals and reagents used in testing and sample preparation are purchased at levels 2 or 3 and standards are uniquely identified and traceable to NIST or equivalent per 1Q, 2-7 section 5.2.3.

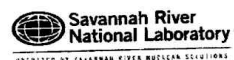
To match the requested functional classification, the reports, calculations, and technical memoranda issued from this testing received technical review by design verification (E7 Manual Procedure 2.60, Section 5.3). This document, including all calculations, was reviewed by Design Verification by Document Review.^{10, 11} SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. The experimental work, the analyses, and peer checks all comply with the customer quality assurance (QA) requirements.

7.0 References

1. “Diversion of DWPF Recycle from HLW Tank Farms System Engineering Alternatives Analysis,” G-AES-S-00005, Revision 0, May 26, 2021.
2. “DWPF Recycle Diversion Characterization”, X-TTR-H-00107, Rev. 1, 5/26/2021.
3. L. N. Oji, “Task Technical and Quality Assurance Plan for the Analysis of the Recycle Collection Tank, Slurry Mix Evaporator Condensate Tank and the Off-Gas Condensate Tank Vessel Samples” SRNL-RP-2020-00915, Revision 0, 01/21/2021.
4. L. N. Oji, “Analysis of Defense Waste Processing Facility Sample: Recycle Collection Tank Sample Batch 4945” SRNL-STI-2021-00228, Revision 1, April 2022.
5. D. J. McCabe, C. L. Crawford, C. J. Bannochie, A. J. Boggess and S. G. Bishop, “Best handling Practices for Elemental Mercury, Organo-Mercury compounds, and Inorganic Mercury Compounds” SRNL-TR-2019-00243, Revision 1, June 2020.
6. Isao Sanemasa, “The solubility of elemental mercury vapor in water,” Bulletin of the Chemical Society of Japan, vol. 48(6), 1795-1798 (1975).
7. Staun, N. Bansal and J. Vaughan, “Electro-crystallization and solubility of mercury in alkaline solution,” Can. J. Chem. 96: 385-393 (2018) dx.doi.org10.1139/cjc-2017-0592.
8. A. J. Boggess, T. L. White, M.A. Jones, T. B. Edwards and S. P. Harris, “Development and Comparison of Purgeable Mercury Values in SRR Samples Measured by SRNL and Eurofins FGS”, SRNL-STI-2019-00300, revision 0, September 2019.
9. A. J. Boggess, T. L. White, M.A. Jones, T. B. Edwards, “Analysis of Ionic Mercury Species in SRR Samples Measured by SRNL and Eurofins FGS”, SRNL-STI-2020-00081, Revision 0, May 2020.
10. “Technical Reviews”, Manual E7, Procedure 2.60, Revision 19, August 25, 2016.
11. “Savannah River National Laboratory Technical Report Design Check Guidelines”, WSRC-IM- 2002-00011, Revision 2, August 2000.

Appendix A: Total Acid Titration Curves: undiluted SMECT and undiluted SMECT filtrates

APD Laboratory Titration Report



O₂ Total Acid

Method

Method ID
Name TA using 0.01 N NaOH
Total Acid to pH 7, 9, 11

21274-81

Summary

Samples

Sample ID	Name	Result	State
1 2179-1	Sample size	0.5 mL	
	Consumption to pH 7 R1	1.9221 mL	OK
	Total acid to pH 7 R2	0.0388 M	OK
	Consumption to pH 9 R4	2.1456 mL	OK
	Total acid to pH 9 R5	0.0433 M	OK
	Consumption to pH 11 R6	4.8100 mL	OK
	Total acid to pH 11 R7	0.0972 M	OK
2 2179-2	Sample size	0.5 mL	
	Consumption to pH 7 R1	1.8801 mL	OK
	Total acid to pH 7 R2	0.0380 M	OK
	Consumption to pH 9 R4	2.1388 mL	OK
	Total acid to pH 9 R5	0.0432 M	OK
	Consumption to pH 11 R6	4.6090 mL	OK
	Total acid to pH 11 R7	0.0931 M	OK
3 2180-1	Sample size	0.5 mL	
	Consumption to pH 7 R1	1.6526 mL	OK
	Total acid to pH 7 R2	0.0334 M	OK
	Consumption to pH 9 R4	1.9545 mL	OK
	Total acid to pH 9 R5	0.0395 M	OK
	Consumption to pH 11 R6	4.7910 mL	OK
	Total acid to pH 11 R7	0.0968 M	OK
4 2180-2	Sample size	0.5 mL	
	Consumption to pH 7 R1	1.8634 mL	OK
	Total acid to pH 7 R2	0.0376 M	OK
	Consumption to pH 9 R4	2.0842 mL	OK
	Total acid to pH 9 R5	0.0421 M	OK

$$\begin{array}{r} 0.0388 \\ + 0.0380 \\ \hline 0.0768 \\ \hline 2 \\ \hline = 0.0384 \end{array}$$

$$\begin{array}{r} 0.0334 \\ + 0.0376 \\ \hline 0.071 \\ \hline 2 \\ \hline = 0.035 \end{array}$$

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APD Laboratory Titration Report



5	2181-1	Consumption to pH 11 R6	4.6970 mL	OK
		Total acid to pH 11 R7	0.0949 M	OK
		Sample size	0.5 mL	
		Consumption to pH 7 R1	1.9243 mL	OK
		Total acid to pH 7 R2	0.0389 M	OK
6	2181-2	Consumption to pH 9 R4	2.1566 mL	OK
		Total acid to pH 9 R5	0.0436 M	OK
		Consumption to pH 11 R6	4.6820 mL	OK
		Total acid to pH 11 R7	0.0946 M	OK
		Sample size	0.5 mL	
7	end std-1	Consumption to pH 7 R1	1.8971 mL	OK
		Total acid to pH 7 R2	0.0383 M	OK
		Consumption to pH 9 R4	2.1275 mL	OK
		Total acid to pH 9 R5	0.0430 M	OK
		Consumption to pH 11 R6	4.6180 mL	OK
8	end std-2	Total acid to pH 11 R7	0.0933 M	OK
		Sample size	0.1 mL	
		Consumption to pH 7 R1	10.5942 mL	OK
		Total acid to pH 7 R2	1.0700 M	OK
		Consumption to pH 9 R4	10.9013 mL	OK
		Total acid to pH 9 R5	1.1010 M	OK
		Consumption to pH 11 R6	14.1480 mL	OK
		Total acid to pH 11 R7	1.4289 M	OK
		Sample size	0.1 mL	
		Consumption to pH 7 R1	10.6253 mL	OK
		Total acid to pH 7 R2	1.0732 M	OK
		Consumption to pH 9 R4	10.9609 mL	OK
		Total acid to pH 9 R5	1.1071 M	OK
		Consumption to pH 11 R6	14.4350 mL	OK
		Total acid to pH 11 R7	1.4579 M	OK

0.0389
 $+ 0.0383$
 $\hline 0.0772 = 0.0386$
 2

Statistics

R1

5/12/2021 4:08:48 PM

SystemInternal

2/28

Titration report

LabX

Overall state OK
Result 4.8100 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.810 mL

Total acid to pH 11 (R7)

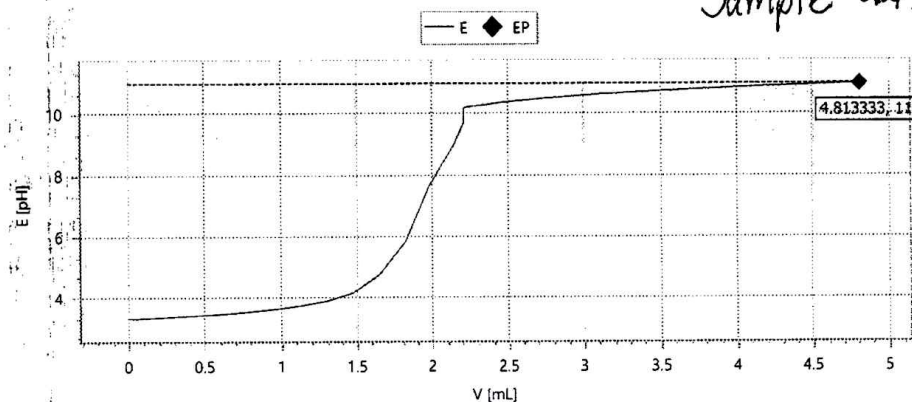
Creation time 5/12/2021 3:35:15 PM
Overall state OK
Result 0.0972 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.810 mL

Conditioning (Conditioning1)

Creation time 5/12/2021 3:35:15 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 1/8

E - V Curve Scope 1/1, Sample 1/8



Used Instruments

Name B-122 Titrator Serial number B909284082

Responsible User

Titration report

LabX

Overall state OK
Result 4.6090 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.6090 mL

Total acid to pH 11 (R7)

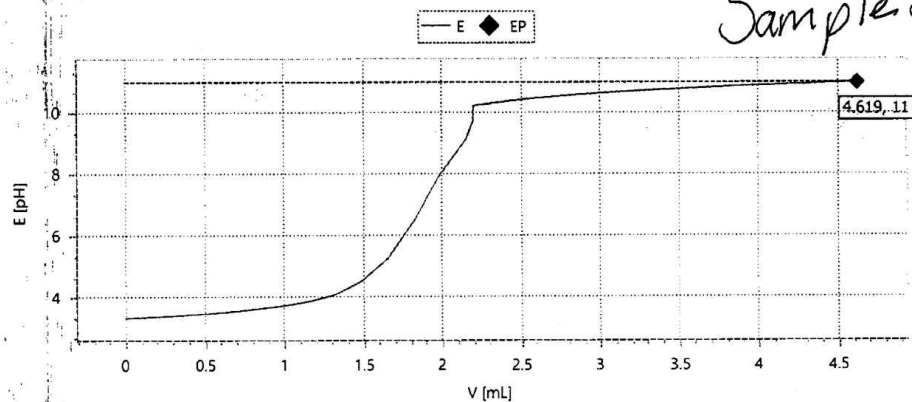
Creation time 5/12/2021 3:38:48 PM
Overall state OK
Result 0.0931 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.6090 mL

Conditioning (Conditioning1)

Creation time 5/12/2021 3:38:48 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 2/8

E - V Curve Scope 1/1, Sample 2/8



Used Instruments

Name B-122 Titrator Serial number B909284082

Responsible User

Titration report

LabX

Overall state OK
Result 4.7910 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.791 mL

Total acid to pH 11 (R7)

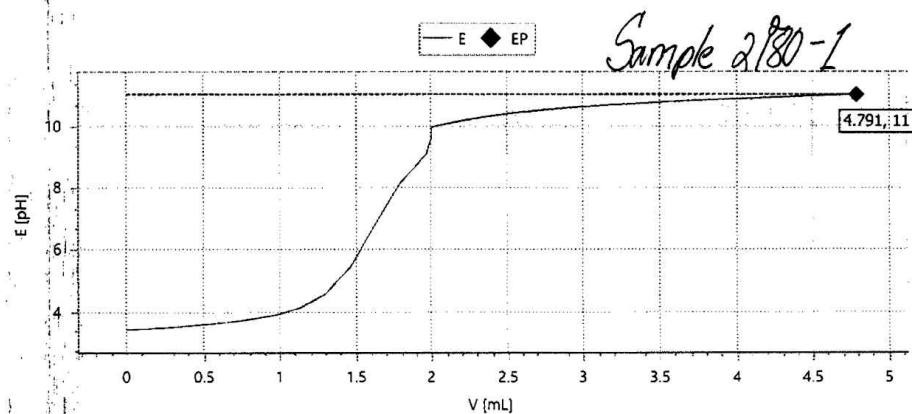
Creation time 5/12/2021 3:42:15 PM
Overall state OK
Result 0.0968 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.791 mL

Conditioning (Conditioning1)

Creation time 5/12/2021 3:42:15 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 3/8

E - V Curve Scope 1/1, Sample 3/8



Used Instruments

Name B-122 Titrator Serial number B909284082

Responsible User

Titration report

LabX

Overall state OK
Result 4.6970 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.697 mL

Total acid to pH 11 (R7)

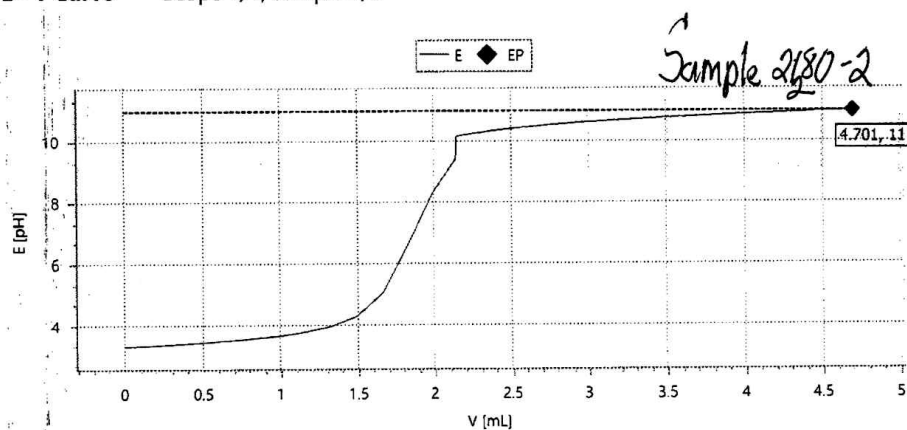
Creation time 5/12/2021 3:45:55 PM
Overall state OK
Result 0.0949 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.697 mL

Conditioning (Conditioning1)

Creation time 5/12/2021 3:45:56 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 4/8

E - V Curve Scope 1/1, Sample 4/8



Used Instruments

Name B-122 Titrator

Serial number B909284082

Responsible User

Titration report

LabX

Overall state OK
Result 4.6820 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.682 mL

Total acid to pH 11 (R7)

Creation time 5/12/2021 3:50:02 PM
Overall state OK
Result 0.0946 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.682 mL

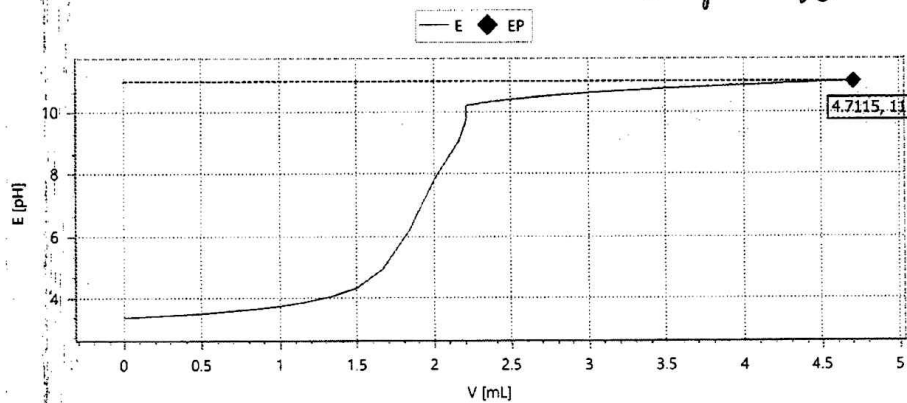
Conditioning (Conditioning1)

Creation time 5/12/2021 3:50:03 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 5/8

E - V Curve Scope 1/1, Sample 5/8

Sample 2181-1



Used Instruments

Name B-122 Titrator Serial number B909284082

Responsible User

Titration report

LabX

Overall state OK
Result 4.6180 mL
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.618 mL

Total acid to pH 11 (R7)

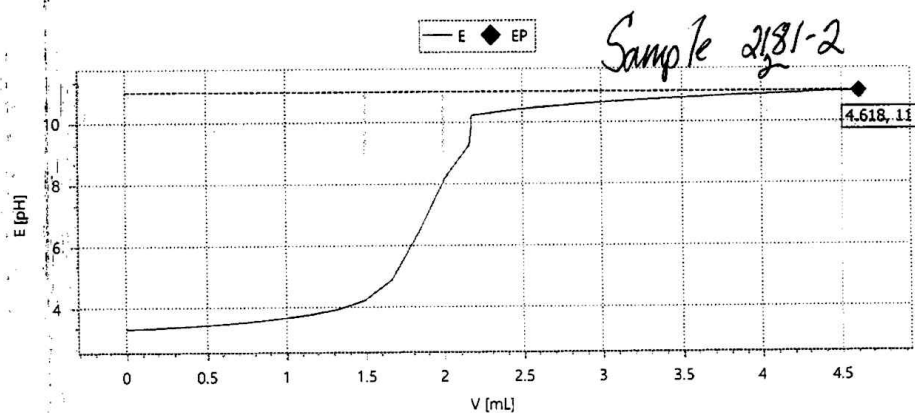
Creation time 5/12/2021 3:53:51 PM
Overall state OK
Result 0.0933 M
Decimal places 4
Substance None
M 1 g/mol
z 1
VE(11.0) 4.618 mL

Conditioning (Conditioning1)

Creation time 5/12/2021 3:53:51 PM
Overall state OK

Titration (EP) [1] Scope 1/1, Sample 6/8

E - V Curve Scope 1/1, Sample 6/8



Used Instruments

Name B-122 Titrator Serial number B909284082

Responsible User

Appendix A Continued: Total Acids[†] at pH 9, and 11.

“As-received” SMECT Slurry		
	Total acid at pH 9 (Units in M)	Total acid at pH 11 (Units in M)
Analysis-1	4.3E-02 (2.15 mL)	9.6E-02 (4.81 mL)
Analysis-2	4.3E-02 (2.14 mL)	9.2E-02 (4.61 mL)
Average total acid, M	4.30E-02	9.40E-02
SMECT Filtrate		
	Total acid at pH 9, M	Total acid at pH 11, M
Analysis-1	4.3E-02 (2.16 mL)	9.2E-02 (4.62 mL)
Analysis-2	4.3E-02 (2.13 mL)	9.2E-02 (4.62 mL)
Average total acid, M	4.28E-02	9.2E-02

[†] Base concentration = 0.01 N, Sample volume = 0.5 mL; where mL base consumed*base concentration, M/sample volume, mL = M total acid. Numbers in bracket correspond to the respective volumes of base (mL) consumed during titration.

Appendix B: Summary of Analytical Methods

Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP-AES)

Samples are diluted as necessary to bring analytes within the instrument range. A scandium internal standard is added to all samples after dilution at a concentration of 2 mg/L. The instrument is calibrated daily with a blank and two standards: 5 and 10 mg/L NIST traceable multi-element standards in dilute acid. Background and internal standard correction were applied to the results.

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

Samples are diluted as necessary to bring analytes within the instrument range. An internal standard with bismuth and indium is added to all samples after dilution. The instrument is calibrated daily with a blank and a minimum of four calibration standards that are NIST traceable multi-element standards in dilute acid. Background and internal standard correction were applied to the results.

Sulfur, Arsenic, Se (ICP-AES-Axial S, ICP-MS-Se and ICP-MS-As)

Quantitative analyses of As and Se were performed on an Agilent 7700x Inductively Coupled Plasma Mass Spectrometer (ICP-MS), which is configured in a radiological containment unit. ICP-MS provides multi-element analyses of aqueous solutions based on the measurement of atomic species from their ions created in the plasma. The high temperature plasma ionizes metallic species, and the ions are separated through a quadrupole mass filter. The detector (electron multiplier) measures the signal for calibration and analysis at ppb levels with a 20% method uncertainty. For As and Se, samples were diluted at 10x in 2% nitric acid and measured at 75 m/z for As and 82 m/z for Se using single element standards for calibration and the application of a He collision cell in the spectrometer.

Quantitative analysis for S is performed on the Agilent 5110 Inductively Coupled Plasma Atomic Emission Atomic Spectrometer (ICP-AES), which is configured in a radiological containment unit. The ICP-ES provides multi-elemental analyses of solutions. Measurements are based on atomic emission from excited atoms and ions. Liquid samples are nebulized, and the aerosol produced is transported to an argon plasma. The plasma is created and sustained by coupling a radio frequency signal to the argon gas. In the high temperature plasma (10,000 K), atomic species are excited to higher energy states resulting in characteristic atomic and ion line emission. The Agilent 5110 instrument has a Dichroic Spectral Combiner to enable

synchronous radial and axial measurements. Wavelengths of light are reflected and transmitted into an echelle based polychromator combined with a vertical torch and VistaChip II CCD detector. The VistaChip II CCD detector provides highspeed and continuous wavelength coverage. For S, samples were diluted at 2X in 2% nitric acid and measured at wavelengths 180.669 and 181.972 nm.

Sr-90

Aliquots of the SMECT samples were spiked with an elemental strontium carrier. The strontium species were extracted from the matrix using a crown-ether-based solid phase extractant. Sr-90 concentrations were measured by liquid scintillation analysis. Elemental strontium carrier yields were measured by neutron activation analysis and were used to correct the Sr-90 analyses for any strontium losses from the radiochemical separations.

Co-60, Am-241 (Cs-removed gamma analysis)

Aliquots of the SMECT samples were subjected to a Cs-removal process utilizing Bio Rad AMP-1 resin. The Cs-removed solutions were analyzed by coaxial high purity germanium spectrophotometers to measure the gamma-emitting radionuclides listed above. Aliquots of SMECT samples were analyzed for Co-60 and Am-241. Sb-125, Sb-126 and Sn-126 were also measured from these analyses.

Pu-238, 239/240, 241

Aliquots of the SMECT samples were spiked with Pu-236 tracer. The plutonium was extracted from the matrix using thenoyltrifluoroacetone (TTA) following a series of oxidation-state adjustments. The TTA extracts were mounted on stainless steel counting plates and counted for Pu-238 and Pu-239/240 using passivated, implanted, planar silicon (PIPs) detectors. Each separation was traced based on the Pu-236 recovery. Aliquots of sample were also subjected to Cs-removal with Bio-Rad Ammonium Molybdophosphate (AMP) resin and extracted using TEVA columns (TEVA is the brand name for one of Eichrom's resins). The Pu-containing extracts were measured by liquid scintillation analysis to determine Pu-241 concentrations. Laboratory reagent blanks and a Pu-238 standard were run as controls.

Ni-63

Aliquots of the SMECT samples were spiked with an elemental nickel carrier. The nickel species were extracted from the matrix using dimethylglyoxime (DMG) based extractant. Ni-59 concentrations were measured using low energy photon/x-ray, thin-windowed, semi-planar high purity germanium spectrometers. Ni-63 concentrations were measured by liquid scintillation analysis. Elemental nickel carrier yields were measured by ICP-AES and were used to correct the radioactive nickel species' analyses for any nickel losses from the radiochemical separations. Reagent blanks, a Ni-63 standard and a Ni-59 standard were run as controls.

I-129

SMECT samples were dissolved in concentrated acid with an added KI carrier. A matrix blank and matrix blank containing an I-129 spike were also prepared using sodalite. The samples were rendered caustic and decontaminated with strikes with crystalline silicotitanate (CST) and monosodium titanate (MST) followed by a filtration step. The samples were then acidified and treated with Actinide and AMP resins to facilitate removal of interfering isotopes. Sodium sulfite was added to the material to reduce the iodine. Silver nitrate was added to the solution to precipitate the iodine as AgI, which was separated via filtration. The filtrate is analyzed for I-129 content using low energy photon/x-ray, thin-windowed, semi-planar, high

purity germanium spectrometers. Elemental iodine yields were measured by neutron activation analysis and were used to correct the I-129 analyses for any iodine losses from the radiochemical separation.

C-14

The SMECT sample was added to a mixture of sodium hydroxide and sodium carbonate/sodium hydroxide. A series of oxidation and reduction steps designed to liberate C-14 containing carbon dioxide were carried out, which selectively trapped the C-14 in a basic solution. The basic solutions were acidified, and the C-14 containing carbon dioxide was captured in Carbosorb E and measured by liquid scintillation analysis. A laboratory blank, a C-14 calibration standard and a C-14 control standard were also run through the process.

Cs-135

Aliquots of SMECT samples that had undergone peroxide fusion dissolutions were purified using a solvent-solvent caustic side solvent extraction-based (CSSX) extraction system. The purified Cs-containing aliquots were analyzed using ICP-MS to measure Cs-135 masses. Cs-137 was measured in the purified Cs-containing aliquots by gamma spectrometry. Cs yields were determined by using the ratio of the Cs-137 concentrations measured in the purified aliquots to the Cs-137 concentrations previously measured on dissolutions of the SMECT samples. The Cs yield was applied to the Cs-135 masses measured to determine the Cs-135 mass concentrations. The Cs-135 result was then converted from $\mu\text{g/g}$ to $\mu\text{Ci/g}$ using the specific activity of Cs-135.

Mercury Analysis (Total Hg, MeHg, DMeHg, Ethyl Hg, Ionic Mercury, and Elemental Mercury)

Total mercury was analyzed by DMA.

With direct mercury analysis (DMA) method for total mercury analysis, controlled heating in an oxygenated decomposition furnace is used to liberate mercury from solid and aqueous samples in the instrument. The sample is dried and then thermally and chemically decomposed within the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. With the completion of oxidation, halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

The typical working range for this method is 0.05 - 600 ng. The mercury vapor is first carried through a long pathlength absorbance cell and then a short pathlength absorbance cell. (The lengths of the first cell and the second cell are in a ratio of 10:1 or another appropriate ratio.). The same quantity of mercury is measured twice, using two different sensitivities, resulting in a dynamic range that spans at least four orders of magnitude. The instrument detection limit (IDL) for this method is 0.01 ng of total mercury.

Gas chromatography/atomic fluorescence spectroscopy: Dimethyl, Methyl, and ethyl mercury analysis

Methylmercury, dimethylmercury, and ethylmercury are analytically separated and quantified from aqueous samples by purge and trap (P&T) gas chromatography (GC) cold-vapor atomic fluorescence spectroscopy (CVAFS). The methyl- or ethylmercury species are first derivatized using sodium tetraethylborate or sodium tetrapropylborate, respectively, to induce volatility prior to sample purge using nitrogen. The purged vapor enters a GC module where the various mercury species are separated

isothermally prior to ballistic pyrolysis to convert all mercury species to fully reduced elemental mercury. The ground-state mercury travels to the CVAFS for detection.

Ionic and Elemental Hg

Analysis for inorganic mercury (ionic mercury) is a two-step process involving, first, the removal of traces of volatile elemental mercury species (purgeable mercury- suspended colloidal, mercury particulates, dense-phase mercury, and any volatile organomercury species) from the sample. In this initial processing to remove elemental mercury, the sample is purged with nitrogen and other gases and the purged mercury (purgeable mercury) trapped onto an adsorbent media for elemental mercury analysis. Following this initial purge, stannous chloride is used to reduce ionic mercury (Hg(I) and Hg(II)) to Hg(0) in the post gas purged sample. Finally, a second purge is performed to remove the reduced ionic mercury, where it can then be trapped on gold-coated beads. Details of the experimental procedure for analyzing SRS waste samples for mercury species are detailed in another report.^{8,9}

Gross Alpha/Gross Beta

Aliquots of the SMECT slurry or filtrates was added to liquid scintillation cocktail and analyzed for gross alpha and gross beta activity using liquid scintillation analysis. Alpha/beta spillover was determined for each aliquot analyzed, and subsequently used for accurately determining alpha and beta activity, via the addition of a known amount of plutonium to an identical aliquot of each sample.

Tc-99

Aliquots of the SMECT slurry or filtrate was oxidized and spiked with Tc-99m that had been extracted from molybdenum which had been activated in SRNL's Cf-252 neutron activation analysis facility. The technetium species were extracted from the matrix using an Aliquat-336 based solid phase extractant.

Tc-99 concentrations were measured by liquid scintillation analysis. Tc-99m yields were measured with a NaI-well gamma spectrometer and were used to correct the Tc-99 analyses for any technetium losses from the radiochemical separations. Alternatively, Tc-99 was also measured by ICP-MS and the value from both methods compared favorably.

Anions in the Supernatant

In preparation for the anion analyses (prior to AD submittal), two supernatant aliquots were each diluted by a factor of ~11 (on a volume basis), using de-ionized water. IC was performed on the diluted supernatant aliquots, to quantify bromide, chloride, fluoride, formate, nitrate, nitrite, oxalate, phosphate, and sulfate. Total inorganic carbon (TIC) analyses were performed to quantify carbonate, and base titration analyses were performed to quantify free hydroxide. Aluminate was quantified based on the ICP-AES supernatant aluminum concentration, assuming 100% of the aluminum was present as aluminate. Dilution-correction of the results was performed prior to reporting.

Weight Percent Solids Measurement (Solids Distribution)

The weight percent total solids for the SMECT sample slurry were measured in the Shielded Cells using a conventional drying oven at 110 °C. An aliquot of the sample was placed in a 150-mL capacity beaker container. Three individual slurry aliquots and three individual supernatant aliquots were utilized in the measurements. The mass of each aliquot was ~5.0 g. The container was placed in the oven. The weights of the dried sample were checked periodically over 72 hours until two consecutive weights yielded comparable results. The weight fraction solid to the total slurry weight was calculated by dividing the dry

weight of the sample by the initial weight of the sample slurry. The weight percent dissolved solids were determined as described above with the SMECT filtrate used instead.

The insoluble solids and soluble solids were calculated from the total solids and dissolved solids (filtrate) using the following equations:

$$W_{is} = (W_{ts} - W_{ds}) / (1 - W_{ds}) \text{ and}$$

$$W_{ss} = W_{ts} - W_{is}$$

Where W_{ds} = weight fraction dissolved solids in the supernate,

W_{ts} = weight fraction total solids in the slurry,

W_{is} = weight fraction insoluble solids in the slurry and

W_{ss} = weight fraction soluble solids in the slurry.

Density Measurement and Volume Measurements

The density of the SMECT sample slurry and filtrates were determined using a 2.0 mL capacity reference glass container. Using a 3-digit balance, the mass of slurry or filtrate required to fill the reference glass up to the 2.0 mL reference mark was measured by difference and recorded. Water was used as the reference media and the Shielded Cell temperatures was 65 °F (18.3 °C)

Particle Size Analysis

For this SMECT sample PSA, about a 200 mL of Tank 22 simulant salt solution, based mainly on Tank 22 precursor salts [sodium nitrite (17.1 g/L), sodium nitrate (5.48 g/L), sodium hydroxide (7.56 g/L), sodium sulfate (0.75 g/L), sodium oxalate (0.343 g/L), sodium carbonate (3.18 g/L), and sodium aluminate (0.0425 g/L)], was prepared, filtered through a 0.45 micron filter membrane and the filtrate sent to SaM for use in PSA for the “as-received” SMECT slurry sample. A small volume of the SMECT slurry (3-5 mL) was suspended in Tank 22 salt simulant described above and the particle size determined.

Appendix C: SMECT Sample Characterization: SaM Tracking Numbers

Analytes	SRNL SaM Tracking Number (LIMS):
Total alpha	LW20465, LW20466, LW20467, LW21289, LW21290, LW21291
Total beta	LW20465, LW20466, LW20467, LW21289, LW21290, LW21291
Cs-135	LW2047, LW2048, LW2049
Ni59/63	LW20461, LW20462, LW20463, LW21285, LW21286, LW21287
Co-60	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Sr-90	LW20461, LW20462, LW20463, LW21285, LW21286, LW21287
Tc-99	LW20461, LW20462, LW20463, LW21285, LW21286, LW21287
Ru-106	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Sb-125	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Sb-126	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Sn-126	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
I-129	LW20474, LW20475, LW20476, LW21298, LW21299, LW21300
Cs-134	LW20468, LW20469, LW20470, LW21292, LW21293, LW21294
Cs-137	LW20468, LW20469, LW20470, LW21292, LW21293, LW21294
Ce-144	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Eu-152	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Eu-154	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Eu-155	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Th-232	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
U-233	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
U-234	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
U-235	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
U-236	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
U-238	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
NP-237	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
Pu-238	LW20471, LW20472, LW20473, LW21295, LW21296, LW21297
Pu-239/ Pu-240	LW20471, LW20472, LW20473, LW21295, LW21296, LW21297
Pu-241	LW20471, LW20472, LW20473, LW21295, LW21296, LW21297
Am-241	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
Am-243	LW20458, LW20459, LW20460, LW21282, LW21283, LW21284
ICP-AES (Elementals)	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
ICP-MS	LW20486, LW20487, LW20488, LW21342, LW21343, LW21344
Particle size	LW20513
XRD	LW20514
SEM	LW20515
TIC/TOC	LW20495, LW20496, LW20497, LW21354, LW21355, LW21356
Total acid	LW20455, LW2046, LW20457, LW21279, LW21280, LW21281
Wt. % total solids	LW20480, LW20481
Wt. % dissolved solids	LW20483
As, Axial Sulfur, Se	LW20489, LW20490, LW20491, LW21345, LW21346, LW21347
Total mercury	LW20498, LW20499, LW20450, LW21357, LW21358, LW21359
Methyl mercury	LW21368, LW21369, LW21370, LW20819, LW20820, LW20821
Dimethyl mercury	LW21368, LW21369, LW21370, LW20819, LW20820, LW20821
Elemental Hg	LW20509, LW20510, LW20511, LW21364, LW21366
Ionic mercury	LW20509, LW20510, LW20511, LW21364, LW21366
Ethyl mercury	LW21364, LW21366, LW20819, LW20820, LW20821
IC-Anions	LW21351, LW21352, LW21353, LW21371, LW21372, LW21373
IC-Cations	LW20492, LW20493, LW20494, LW21348, LW21349, LW21350

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