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Analysis of Defense Waste Processing Facility Sample: Recycle Collection Tank Sample Batch 4945

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0	Initial Issue	August 2021
1	"As-received" RCT slurry and RCT filtrate viscosity values added in the report on pages 7, 8, and 30. The analytical results for mercury species in both the "as-received" RCT and RCT filtrate samples were updated in section 4.3.	April 2022

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) was requested by Savannah River Remediation (now Savannah River Mission Completion (SRMC)), through a Technical Task Request, to characterization the "as-received" Recycle Collection Tank (RCT) Sample identified as sample batch 4945 [Sludge Receipt and Adjustment Tank (SRAT) batch 796)], which was delivered to SRNL Shielded Cells on January 28, 2021. The RCT characterization data will be used as input to the Defense Waste Processing Facility (DWPF) Recycle Diversion Project.

This RCT report is the first of three sample characterization reports that will be used for this DWPF Project. The other DWPF reports will involve the characterization of the Off-Gas Condensate Tank (OGCT) and the Slurry Mix Evaporator Condensate Tank (SMECT) samples.

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

- The density of the unfiltered, "as-received" RCT sample averaged 1.02 g/mL (1.2 % RSD) and that of the RCT filtrate sample averaged 1.00 g/mL (0.30 %RSD).
- The pH of the "as-received" RCT sample was 12.9 and that of the filtrate was 13.
- The pH of the RCT sample filtrate, based on free-OH concentration calculations in the filtrate, was 13.2 (0.1 %RSD).
- The turbidity of the "as-received" RCT slurry averaged 64.9 NTU (0.4 %RSD) and that of the filtrate averaged 1.3 NTU (1.3 %RSD).
- The weight percent total solids, dissolved solids, calculated weight percent insoluble solids and soluble solids are 2.1 wt.%, (5.7 %RSD), 2.1 wt.% (6.5 %RSD), 0.04 wt.% and 2.1 wt.%, respectively.
- The viscosity of the "as-received" RCT slurry was 0.98 cP (1.2 %RSD) and that of the RCT filtrate was 1.02 cP (2.4 %RSD). These viscosity values for the "as-received" RCT sample and the RCT filtrate are not different from that of ordinary water at 25^{o}C.
- The XRD pattern for the "as-received" RCT solid fraction matches those of several common minerals (sodium nitrite ($NaNO_2$), sodium aluminosilicate ($Na_5(AlSiO_4)_6{*}H_2O$), trisodium carbonate ($Na_3(CO_3)(HCO_3)(H_2O)_2$), gibbsite ($\alpha$-$Al_2O_3.3H_2O$) and hematite ($Fe_2O_3$)). The RCT solid fraction consists of partially crystalline phase minerals and non-crystalline amorphous phases.
- Scanning electron microscope information shows that the principal quantitative elemental constituents present in the RCT sample solid fraction include iron, nickel, aluminum, sodium, chromium, manganese, silicon, calcium, magnesium, mercury, and thorium.
- The particle size distribution result for the "as-received" RCT sample is left skewed (most data falls to the left) and the average particle size is 126.2 ± 125.0 microns (1 sigma standard deviation).
- Total beta activity in the "as-received" RCT sample averaged 1.15E+07 dpm/mL (0.6 %RSD) and an average activity concentration of 5.20E-03 Ci/L. The average total alpha activity was an upper bound at < 1.02E+05 dpm/mL (<4.61E-05 Ci/L).
- The primary beta emitting radionuclides in the "as-received" RCT sample include Sr-90, Y-90 and Cs-137 at average activity concentrations of 3.92E-04, 3.92E-04 and 3.33E-03 Ci/ L, respectively.
- As expected, the primary gamma emitting radionuclide in the "as-received" RCT sample is Ba-137m at average activity concentration of 3.15E-03 Ci/ L (note that the activity concentration for Ba-137m was calculated as 94.7% of the Cs-137 activity concentration).
- Technetium-99 activity in the "as-received" RCT sample averaged 1.37E+03 dpm/mL (6.2 %RSD) and an average activity concentration of 6.16E-07 Ci/L.

- Iodine-129 activity in the “as-received” RCT sample averaged 6.62E+01 dpm/mL (14.5 %RSD) and an average activity concentration of 2.98E-08 Ci/L.
- Plutonium-238 activity in the “as-received” RCT sample averaged 3.12E+04 dpm/mL (5.6 %RSD) and an average activity concentration of 1.41E-05 Ci/L.
- Plutonium-241 activity in the “as-received” RCT sample averaged 5.87E+03 dpm/mL (9.6 %RSD) and an average activity concentration of 2.64E-06 Ci/L.

The activities for total beta, I-129, Pu-238 and Pu-241 in the filtrate RCT sample all showed a significant drop in comparison to their initial activities in the “as-received” RCT sample. Total beta activity in the “as-received” RCT (1.15E+07 dpm/mL (0.6 %RSD)) sample dropped by 18.4% in the filtrate (9.38E+06 dpm/mL, 0.5 %RSD). Iodine-129 activity in the “as-received” RCT sample (6.62E+01 dpm/mL, 14.5 %RSD) dropped to less than minimum detection limit in the filtrate, while Plutonium-238 and Plutonium-241 activities in the “as-received” RCT sample, respectively, at 3.12E+04 dpm/mL (5.6 %RSD) and 5.87E+03 dpm/mL (9.6 %RSD), dropped to less than minimum detection limits in the filtrate sample.

The average activities for Ni-63 (1.38E+04 dpm/mL, 25 %RSD), Th-232 (3.66E-01 dpm/mL, 0.4 %RSD), Np-237 (3.74E+00 dpm/mL, 1.1 %RSD) and Pu-239/240 (1.87E+03 dpm/mL, 13.4 %RSD) were all above instrument detection limit in the “as-received” RCT sample. In the filtrate, the activities for these radionuclides were all less than minimum detection limits, which means they had been retained as part of the solid fraction during the solid liquid separation.

The activities for Cs-137, Tc-99 and U-238 in the “as-received” RCT sample were comparable to their activities in the filtrate sample; activities were about the same order of magnitude in the two types of sample media. The average Sr-90 activity in filtrate was slightly lower than its activity in the “as-received” RCT sample.

Only the analytical results for iron, sodium, manganese, and silicon were above instrument detection limits in the elemental characterization of the “as-received” RCT and filtrate samples. The concentrations for all other elements were below instrument detection limits. Sodium concentration in the “as-received” RCT sample averaged 8301 mg/L (0.6 %RSD) and that for silicon averaged 34.7 mg/L (0.4 %RSD). Sodium concentration in the filtrate RCT sample averaged 8073.5 mg/L (3.4 %RSD) and that for silicon averaged 38.2 mg/L (2.9 %RSD). The sodium and silicon concentrations in the “as-received” RCT sample and RCT filtrate samples do not show any significant differences in concentrations; the small differences are within the analytical uncertainties for the two elements. Both iron and manganese concentrations in the “as-received” RCT sample were above instrument detection limits and averaged 8.3 mg/L (0.9 %RSD) and 3.3 mg/L (0.3%RSD), respectively. Iron and manganese concentrations (<2.4 mg/L and <0.2 mg/L, respectively) in the RCT filtrate were below instrument detection limits possibly due to their retention as part of the solid fractions during filtration.

Total mercury in the “as-received” RCT sample averaged 252.7 mg/L (13.6 %RSD). Methyl mercury, elemental mercury, and ionic mercury (inorganic mercury) concentration in the “as-received” RCT sample measured 47.5 mg/L (0.9 %RSD), 22.2 mg/L, and 42.7 mg/L, respectively.

The average methyl mercury concentration in the RCT filtrate at 50.1 mg/L (0.9 %RSD) was statistically comparable to the methyl mercury concentration in the “as-received” RCT sample (47.5 mg/L (0.9 %RSD). However, the elemental mercury dropped from a high of 22.2 mg/L in the “as-received” RCT sample to

1.81 mg/L in the filtrate, which is a drop of about 92% in the filtrate sample. Ionic mercury concentration dropped from 42.7 mg/L in the "as-received" RCT sample to 27.2 mg/L in the RCT filtrate, which is a drop of about 36% in the RCT filtrate.

Ethyl mercury and dimethyl mercury determinations in the "as-received" RCT and filtrate samples were below instrument detection limits. These mercury species may not be present in the "as-received" RCT sample at detectable levels.

The re-analyses of the RCT 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.

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" RCT and filtrate samples. Because of the extreme saturation level for elemental mercury and other mercury forms in the RCT samples, the analytical uncertainty measurements for elemental 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 have high bias. As a result, there are mercury mass balance issues with the characterization of the "as-received" RCT samples for total mercury and other mercury species.

Filtering of the "as-received" RCT sample to generate the filtrates seem to have resulted in the retention, as part of the solid fraction, of analytes (mercury species, 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" RCT sample. This retention of the analytes results in the decrease in concentration 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
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
SRMC	Savannah River Mission Completion
SRR	Savannah River Remediation

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) recycle stream away from the SRS Tank Farm, SRR (now SRMC) has requested that the Savannah River National Laboratory (SRNL) help in the evaluation and characterization of the process chemistry involved in the DWPF waste stream diversion. Of the 5 Systems Engineering Evaluation[2] options for diverting the DWPF 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) in order 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 ($NaNO_2$). Sodium nitrite is added to meet the tank farm corrosion control requirements. The samples were taken while DWPF was using Antifoam 747 and they have since transitioned to using Momentive™ Y-17112 antifoam[a].

The RCT will serve as the feed 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 in order 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 in regard to chemistry and throughput of the facilities.

The RCT samples (sample 27537) sent to SRNL for characterization were identified as RCT batch 4945 from SRAT batch 796. This RCT material corresponded to Strip Effluent Feed Tank (SEFT) material that was collected during the final SEFT addition of approximately 7160 gallons (27,100 Liters) to the SRAT batch with three Antifoam 747 additions to the Sludge Receipt and Adjustment Tank (SRAT) as this condensate was being collected.

This report presents the analytical results for the characterization of the "as-received" DWPF RCT sample[b]. These RCT sample characterization efforts are governed by a Technical Task Request (TTR)[2] and a Task

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

[b] "As-received" means no chemical or physical adjustment other than dilutions, if necessary, of sample prior to analysis.

Technical and Quality Assurance Plan (TTQAP)[3]. It is the first of three sample characterization reports dealing with this DWPF recycle program. The other future recycle stream reports will be the characterization of the OCGT and the SMECT samples.

Objectives
The customer requested the characterization[1, 3] of the "as-received" DWPF RCT sample (batch 4945 from SRAT batch 796) and the RCT filtrate samples to support the eventual diversion of the DWPF recycle stream away from the SRS Tank Farm.

The initial task performed with the RCT sample involved physical characterizations [bulk densities, particle size distribution (PSD), X-ray diffraction (XRD) and scanning electron microscopy (SEM)], turbidity and pH measurements. Viscosity measurement, which is part of the characterization request, will be performed and reported to the customer later, possibly in a revised version of this report. The RCT sample was also characterized for weight percent total and dissolved solids, density, elemental composition, total mercury and methyl mercury, free-hydroxide and anions and select radionuclides.

2.0 Experimental Setups/Sample description and Preparations/Methodology

The "as-received" RCT 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 after transfer into PMP container are shown in Figure 1, inserts A-C. These samples were solutions with brown coloration due to the presence of brownish particulates. 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 (21 hours) for settling. Figure 1, insert C, shows images of the settled solids, with the liquid portion a lot clearer than after the 2-hour settling period. 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 mass was 630 grams (~ 645 mL).

Analyses for most of the analytes in the RCT sample were performed in triplicate, with the analyses for most the mercury species being the exception. The analyses for total mercury, dimethyl mercury, and ethyl mercury were performed in triplicate and the analysis for methyl mercury in duplicate for both the "as-received" RCT sample and the RCT filtrate samples. Single analysis was performed for all other mercury species in both the "as-received" and filtrate RCT samples. It was necessary to transport small portions (5-10 mL) of the "as-received" RCT sample in green shielded cell bottles, with effective shielding, to the Sensing and Metrology (SaM) group for special storage and characterizations without any dilutions to achieve lower detection limits for I-129, C-14, Cs-135, anions and free-hydroxide (Free-OH^{-1}). The characterization of the RCT sample for other radionuclides, as specified in the TTR[1] and presented in Table 1, involved bulk dilutions of the "as-received" RCT sample and filtrate in 2.0 M HNO_3 and 1.0 M HCl (mercury species) or super-Q water (anion, cation and total organic / inorganic carbons), followed by the removal of aliquot samples in green shielded cell bottles that were sent to 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 may also affect the detection limit for some of the analytes in the final analytical results.

All RCT 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 SaM special storage facility in preparation for analyses in accordance with best practices for mercury handling and analysis [4]. Where sample analyses called for the use of RCT filtrates, these filtrate samples were obtained by liquid /solid separation using a 0.45 µm Nalgene® filter nylon membrane; about 100 mL of the "as-received" RCT slurry were normally filtered through the Nalgene® membrane.

A total of 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 RCT 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) involves 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 RCT sample slurry particle size determination as described in Appendix B.

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. The approach was used in the analytical results for sulfur, arsenic, selenium, and formate anion as well as for I-129 and Cs-135. Since the weight percent insoluble solids for the RCT sample was small (0.04 wt. %), 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" RCT slurry if there was no retention of the analytes in the 0.45 separation membrane, or the particle size of the analytes were larger than 0.45 microns.

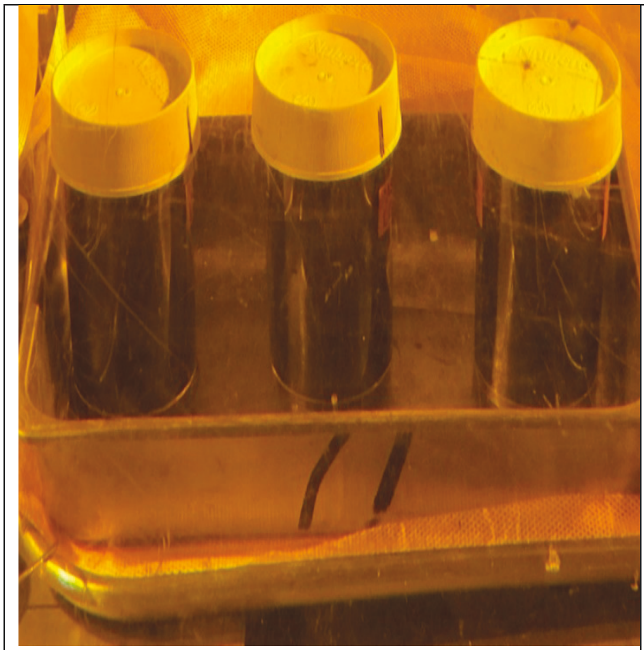	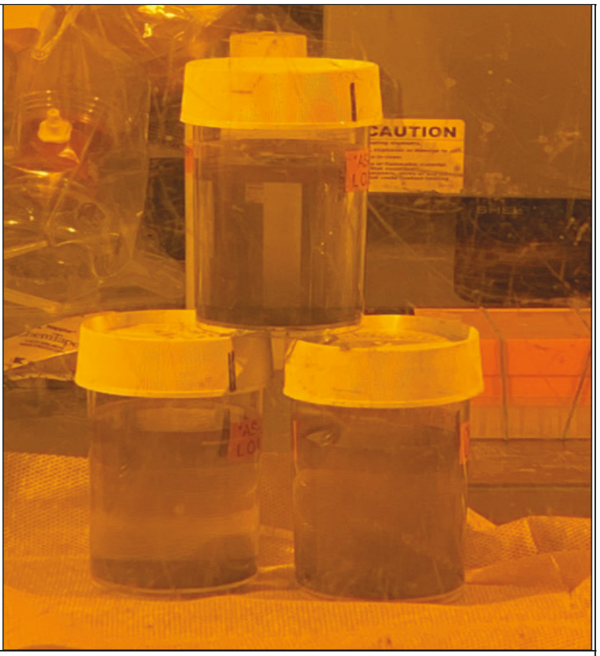
Insert A: "as-received" RCT sample: Dark brown solution with fine suspended particles.	**Insert B:** Clear solution with less than 1% by volume of settled fines after 2 hours settling.
	Intentionally leftt blank
Insert C: Clearer solution with less than 1% by volume of settled fines after 21 hours settling.	

Figure 1. Photo images of "as-received" RCT samples

Table 1 Analytical method Summary for RCT Sample Characterization

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/EDX	Solid fractions	SaM
Sulfur	ICP-AES-axial Sulfur	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	Extraction & LSC	Slurry & filtrate acid dilutions	SaM
Masses 59, 82, 84-114, 116-126, 128, 130, 133-187, 191, 193-196, 198, 203-208, 229-230, 232-251.	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
C-14	C-14	Undiluted Slurry & Filtrates	SaM
Cs-135	Cs-135 extraction/ICP-MS	Undiluted Slurry & Filtrates	SaM
Total alpha/beta	LSC & Rad screen	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^- $C_2O_4^{2-}$, HCO_2^-, PO_4^{3-}, Br^{-1}	IC-anions	Water dilutions & undiluted	SaM
IC-Cations (NH_4^+)	IC-Cations	Water dilutions & undiluted	SaM
Y-90 and Ba-137m	Calculated	n/a	πPI

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; π PI = Principal investigator; ¥SCO = Shielded Cell Operations; SaM = Sensing and Metrology; n/a = not applicable; XRD = X-ray diffraction.

3.0 Data Quality and Blank Evaluations

Appendix A contains the SRNL Analytical Research and 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 (for example, 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 RCT “as-received” slurry analysis results are presented first followed immediately by tables containing the corresponding analysis results for the RCT 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 and b) ~5% for Cs-137 determined by gamma spectroscopy and c) 20% for Cs-135 and mercury species. As such, only one to three of the leading digits reported for the SaM analysis results should be considered significant.

In the RCT 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 proceeded 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) analyses are 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, SQRT((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, upper limit values or the DL values as indicated by the analytical method.

4.0 Results and Discussion

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

4.1 Physical Measurements (Density, Turbidity, Weight percent solids, pH, and Viscosity)

As presented in Table 2, the density of the unfiltered, "as-received" RCT sample averaged 1.02 g/mL (1.2% RSD) and that of the RCT filtrate sample averaged 1.00 g/mL (0.3%RSD). Direct pH measurement with a pH probe gave a pH value of 12.9 for the "as-received" RCT sample and a value of 13.0 for the RCT filtrate (Table 2). To verify the measured pH value for the filtrate, the pH of the filtrate was also calculated from the analytical results for free-OH concentration using the equation pH = 14 + log [Free-OH], where the free-OH concentration averaged 0.14 molar as shown in Table 5. Thus, the pH of the filtrate from free-OH concentration calculations average 13.2, which is about the same order magnitude as the directly measured pH value of 13 for the filtrate.

Table 2. Physical Characterization of RCT Sample (batch 4945; SRAT batch 796) RCT "as-received" Slurry

Parameter	Analysis-1	Analysis-2	Analysis-3	Average	%RSD, N =3
Density, g/mL	1.01	1.04	1.02	1.02	1.2
$^{\alpha}$pH	12.9	12.9	12.9	na	na
Turbidity, NTU	64.6	65.1	65.0	64.9	0.4
Viscosity, cP	0.98	0.99	Not determined	0.98	1.2 N =2
RCT Sample Filtrate					
Parameter	**Analysis-1**	**Analysis-2**	**Analysis-3**	**Average**	**%RSD, N =3**
Density, g/mL	1.00	1.01	1.00	1.00	0.3
$^{\#}$pH	13.2	13.2	13.1	13.2	0.1
Turbidity, NTU	1.4	1.3	1.3	1.3	1.3
Viscosity, cP	1.0	1.03	Not determined	1.02	2.4, N =2

N = number of replicates; $^{\alpha}$No average values for pH; pH is a log function, na = not applicable,
# pH was calculated from free-OH analytical result, but direct pH measurement reading was 12.96.

The turbidity of the "as-received" RCT slurry averaged 64.9 Nephelometric Turbidity Units (NTU) [0.4 %RSD] and that of the filtrate averaged 1.3 NTU (1.30 %RSD). The weight percent total solids [2.1 wt.%, (5.7 %RSD)], dissolved solids [2.1 wt.% (6.5 %RSD)] and calculated weight percent insoluble solids and soluble solids are 0.04 wt.% and 2.1 wt.%, respectively (Table 3). The viscosity of the "as-received" RCT slurry averaged 0.98 cP (1.2 %RSD) and that of the RCT filtrate averaged 1.02 cP (2.4 %RSD).

Table 3. Weight Percent Solids: RCT Sample (batch 4945; SRAT batch 796)

Parameter	Result	Units	Standard deviation, 1 Sigma
¥Wt% Total Solids	2.1	Wt %	0.12
Wt% Dissolved Solids	2.1	Wt %	0.13
Wt% Insoluble Solids	0.04	Wt %	calculated
Wt% Soluble Solids	2.1	Wt %	calculated

¥Measurements based on gram slurry for wt% total solids and gram filtrate for wt% dissolved solids.

4.2 Anion Analysis

The anion analytical results for the "as-received" RCT and filtrate samples are presented in Tables 4 and 5. Analysis of the "as-received" RCT sample required a dilution factor of about 16. At this dilution factor, the analytical results for analytes of particular interest such as formate, free-hydroxide and sulfate were below instrument detection limits (Table 4) because of their initial low concentrations in the undiluted "as-received" RCT sample.

To attain better detection limits for these anions, using the filtrate RCT sample, it was decided to take small aliquots of undiluted RCT filtrate out of the Shielded Cell for analyses. The results for the undiluted filtrate, as expected, produced the desired analytical results for formate, free-hydroxide, sulfate and other anions, as shown in Table 5 for the RCT filtrate.

The diluted "as-received" RCT sample result reported for the formate anion (< 171.3 mg/L) is comparable to the analytical result reported for the undiluted RCT filtrate sample, which averaged 138.7 mg/L (1.1 %RSD) because the less than value for formate anion concentration in the "as-received" RCT slurry points to the same concentration magnitude as the true measured concentration for the formate anion in the RCT filtrate.

The other principal anion analytical results (nitrates, nitrites) for the diluted "as-received" RCT sample and the undiluted RCT filtrate, as presented in Table 4, are comparable in magnitude. Nitrates and nitrites in the "as-received" diluted RCT samples averaged 1582.3 mg/L (0.5 %RSD) and 9076.1 mg/L (3.4 %RSD), respectively, while their concentrations in the undiluted RCT filtrate averaged 1591 mg/L (2.8 %RSD) and 9185 mg/L (0.8 %RSD), respectively. The slight differences in concentration of these analytes in both the diluted "as-received" RCT and the undiluted RCT filtrates are mainly due to dilution effects and analytical uncertainties.

Free-OH analysis result for the "as-received" RCT samples was left out because of titration interferences due to the presence of small fine particles in the as-received sample. Free-OH results reported are for the undiluted RCT filtrate sample, as shown in Table 5, and the average free-OH concentration is 0.14 M (2.5 %RSD). It is worth noting that the pH for the RCT sample is based on this free-OH concentration value. The carbonate concentration in the diluted "as-received" RCT sample averaged 2.2E-02 M (3.8 %RSD) and 1.8E-02 M (9.9 %RSD) for the undiluted filtrate sample. Carbonate results are calculated from the inorganic carbon concentrations in the "as-received" RCT and RCT filtrate samples. The difference between these two carbonate results is about 19 %, which will seem to indicate that some of the inorganic carbon components were retained on the surface of the membrane during filtration to obtain the filtrate from the "as-received" RCT sample.

Both phosphate and sulfate analysis results for the diluted "as-received" RCT sample were below instrument detection limit because of dilution effects (low concentration of phosphate and sulfate in the original "as-received" RCT sample). However, the analyses result for sulfate anions averaged above instrument detection limits (15.2 mg/L (1.7 %RSD)) when undiluted filtrates were characterized for sulfate anions. The concentration for phosphate anion in the undiluted filtrates gave mixed results; one of the triplicate analysis results gave a real value of 11 mg/L and the other two analyses were below instrument detection limits (<10 mg/L) as shown in Table 5. The analysis results for the following anions, ammonium ion, aluminate anion (calculated from elemental Al), fluoride, chloride, bromide, and oxalate were below instrument detection limits.

The %RSD for all anion analyses, those above minimum detection limits, as presented in Tables 4 and 5, are less than 10% and meet the analytical requirements.

The concentrations for total carbon and total organic carbon in the "as-received" RCT samples were small to begin with, and with dilutions by a factor of 16, the analysis results for these diluted RCT samples for these analytes were now below instrument detection limits, averaged <3.4E-01 g/C/L and 8.6E-02 g/C/L, respectively, as shown in Table 4. However, the analytical results for both total carbon and total organic carbon in the undiluted RCT filtrate were above instrument detection limits because there were no dilutions of the filtrate as was done with the "as-received" RCT samples. Total carbon and total organic carbon in the undiluted RCT filtrates now averaged 2.9E-01 gC/L (6.5 %RSD) and 8.3E-02 gC/L (2.4 %RSD), respectively (Table 5).

To check the results, a cation-anion normality balance was performed. The normal concentrations of cations (mainly Na^+, Fe^{+2} and Mn^{+2}) were summed, as were the anions (NO_3^-, NO_2^-, SO_4^{2-}, Cl^-, CO_3^{2-}, PO_4^{3}, AlO_2^-, $C_2O_4^{2-}$ and free OH^-). The two sums were compared. For these comparisons, the primary contributing cations included Na^+ and K^+, while the primary contributing anions included hydroxide, nitrite, nitrate, carbonate, formate, sulfate, phosphate, oxalate, chloride, and aluminate.

For the RCT "as-received" sample, the cations summed to 3.6E-01 M, while the anions summed to 3.9E-01 M. Thus, the cations summed to about 92.3 % of the anions. The differences between the cation and anion molarity values are within ± 10% of each other, which is good when one takes into consideration that the nominal uncertainties (1 sigma) for ICP-AES, IC and OH are about 10%. The small difference can be attributed to analytical uncertainties. This anion/cation comparison may not be valid for the filtrates because some of the analytes are retained on the membrane during the solid/liquid separation to generate the filtrate.

Table 4. IC-Anion/Cation, Total carbon Analyses Results: “as-received” RCT Slurry (batch 4945)

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F$^-$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Formate, HCO$_2^-$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Chloride, Cl$^-$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Nitrite, NO$_2^-$, mg/L	8734	9162	9333	9076	309	3.4
Nitrate, NO$_3^-$, mg/L	1576	1581	1591	1582	8	0.5
Phosphate, PO$_4^{3-}$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Sulfate, SO$_4^{2-}$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Oxalate, C$_2$O$_4^{2-}$, mg/L	<171.3	<171.3	<171.3	< 171.3	na	na
Bromide, Br$^-$, mg/L	<856.2	<856.2	<856.2	< 856.2	na	na
Free Hydroxide, M	-	-	-	-	na	na
Carbonate, CO$_3^{2-}$, M	2.28E-02	2.14E-02	2.14E-02	2.19E-02	8.24E-04	3.8
Al(OH)$_4^-$, M	<1.89E-04	<1.89E-04	<1.89E-04	<1.89E-04	na	na
Ammonium ion, NH$_4^-$ mg/L	<85.62	<85.62	<85.62	<85.62	na	na
Total carbon, gC/L	<3.4E-01	<3.4E-01	<3.4E-01	<3.4E-01	na	na
Inorganic carbon gC/L	2.7E-01	2.6E-01	2.6E-01	2.6E-01	9.9E-03	3.8
Organic carbon, gC/L	<8.6E-02	<8.6E-02	<8.6E-02	<8.6E-02	na	na

N = number of replicates; na = not applicable.

Table 5. IC-Anion/Cation, Total carbon Analyses Results: RCT -Filtrate (batch 4945; SRAT batch 796)@

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F$^-$, mg/L	< 10	< 10	< 10	< 10	na	na
Formate, HCO$_2^-$, mg/L	139	137	140	138.7	1.53	1.1
Chloride, Cl$^-$, mg/L	< 10	< 10	< 10	< 10	na	na
Nitrite, NO$_2^-$, mg/L	9268	9143	9144	9185	71.9	0.8
Nitrate, NO$_3^-$, mg/L	1541	1608	1624	1591	44.0	2.8
Phosphate, PO$_4^{3-}$, mg/L	11	< 10	< 10	≤ 11	na	na
Sulfate, SO$_4^{2-}$, mg/L	15	15.1	15.5	15.2	0.3	1.7
Oxalate, C$_2$O$_4^{2-}$, mg/L	< 10	< 10	< 10	< 10	na	na
Bromide, Br$^-$, mg/L	< 10	< 10	< 10	< 10	na	na
Free Hydroxide, OH$^-$, M	0.14	0.14	0.14	0.14	4.0E-03	3.0
Carbonate, CO$_3^{2-}$, M	1.6E-02	1.8E-02	2.0E-02	1.8E-02	1.8E-03	9.9
Al(OH)$_4^-$, M	<1.9E-04	<1.9E-04	<1.9E-04	<1.9E-04	na	na
Ammonium ion, NH$_4^-$ mg/L	<85.6	<85.6	<85.6	<85.6	na	na
Total carbon, gC/L	2.8E-01	2.9E-01	3.2E-01	2.9E-01	1.9E-02	6.5
Inorganic carbon gC/L	1.9E-01	2.1E-01	2.3E-01	2.1E-01	2.1E-02	9.9
Organic carbon, gC/L	8.5E-02	8.2E-02	8.2E-02	8.3E-02	2.0E-03	2.4

@ Analysis for free-OH based on undiluted filtrates to enhance free-OH detection; na = not applicable; N = number of replicates.

4.3 Elemental analysis, Total mercury and Mercury Species Composition

Elementals, total mercury, and mercury species analytical results for the "as-received" RCT sample and the RCT filtrate samples are presented in Tables 6 and 7, respectively. Only the analytical results for iron, sodium, manganese, and silicon were above instrument detection limits in the elemental characterization of the "as-received" RCT and filtrate samples. The concentrations for all other elements were below instrument detection limits. Sodium concentration in the "as-received" RCT sample averaged 8301 mg/L (0.6 %RSD) and that for silicon averaged 34.7 mg/L (1.1 %RSD). Sodium concentration in the filtrate RCT sample averaged 8074 mg/L (3.4 %RSD) and that for silicon averaged 38.2 mg/L (2.9 %RSD). The sodium and silicon concentrations in the "as-received" RCT sample and RCT filtrate samples do not show any significant differences; the small differences are within the analytical uncertainties for the two elements. Both iron and manganese concentrations in the "as-received" RCT sample were above instrument detection limits and averaged 8.3 mg/L (0.9 %RSD) and 3.3 mg/L (0.3%RSD), respectively. Iron and manganese concentrations (<2.4 mg/L and <0.2 mg/L, respectively) in the RCT filtrate were below instrument detection limits due to their retention as part of the solid fractions during filtration.

The analytical result for arsenic, sulfur and selenium in the 16-fold diluted "as-received" RCT sample were all below instrument detection limits for these analytes (Table 6); the results averaged <0.02 mg/L, <15.6 mg/L and <0.02 mg/L, respectively. To enhance the detection limits for these analytes in the RCT filtrate, it was decided to send out small aliquots of the undiluted filtrate samples from the Shielded Cell to SaM for analysis. The results for arsenic, sulfur, and selenium in the undiluted filtrate sample aliquots, as presented in Table 7, averaged <1.0E-03 mg/L, 4.5 mg/L (1.5 %RSD) and <1.0E-03 mg/L. These results confirm that there are no measurable quantities of both arsenic and selenium in the filtrate and the "as-received" RCT samples. Sulfur was present in the undiluted RCT sample filtrate at 4.5 mg/L (1.5 %RSD), and points to the concentration direction (<15.6 mg/L) for sulfur measured in the diluted "as-received" RCT sample.

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" RCT sample and the RCT filtrate are presented in Tables 6 and 7, respectively.

The analytical results for total mercury, methyl, elemental and ionic mercury in the "as-received" RCT sample were all above instrument detection limit, with dimethyl mercury and ethyl mercury being the exceptions. Total mercury in this sample averaged 252.7 mg/L (13.6 %RSD). Methyl mercury, elemental mercury, and ionic mercury (inorganic mercury) concentration in the "as-received" RCT sample averaged 47.5 mg/L (0.9 %RSD), 22.2 mg/L, and 42.7 mg/L, respectively. The results for dimethyl mercury and ethyl mercury in the "as-received" RCT sample were below the instrument detection limits.

Total, methyl, elemental and ionic mercury analytical results for the RCT filtrate samples are presented in Table 7. Except for total mercury, methyl mercury, elemental and ionic mercury analytical results for this sample, the analytical results for all the other mercury species were below instrument detection limits. Taking into consideration the analytical uncertainties for these mercury species in the RCT samples, the average methyl mercury concentration in the RCT filtrate at 50.1 mg/L (0.9 %RSD) was comparable to the methyl mercury concentration in the "as-received" RCT sample (47.5 mg/L (0.9 %RSD)). However, the elemental mercury dropped from a high of 22.2 mg/L in the "as-received" RCT sample to 1.81 mg/L in the filtrate, which is a drop of about 92% in the filtrate sample. Ionic mercury concentration dropped from 42.7

mg/L in the "as-received" RCT sample to 27.2 mg/L in the RCT filtrate, which is a drop of about 36% in the RCT filtrate.

The concentration of total mercury in the RCT filtrate average 166.5 mg/L (3.6 %RSD), which is about 86 mg lower than the average total mercury concentration in the original "as-received" RCT sample (252.7 mg/L). Neglecting measurement uncertainty, this means that the filtration process resulted in the loss of about 34 % of the total mercury.

Ethyl mercury and dimethyl mercury determinations in the "as-received" RCT and filtrate samples were below instrument detection limits even when undiluted sample aliquots were analyzed for dimethyl mercury and ethyl mercury. This would seem to indicate that these mercury species are not present at a detectable level in the "as-received" RCT sample.

As presented in Table 6, the sum of the average concentrations of all the mercury species, excluding total mercury, in the "as-received" RCT sample is ~ 112.4 mg/L. This sum should equal the average total mercury analyses results (252.7 mg/L) or at least be within the analytical error margin of 10-20% for these methods of analyses. The one sigma analytical uncertainty for elemental (purgeable mercury) and ionic mercury analysis was reported as 40% and 20% for methyl mercury. Because elemental mercury, ionic mercury and methyl mercury comprise a large fraction of the mercury species, these significantly large analytical uncertainties for these mercury species renders it difficult to obtain a reliable mercury mass balance for the RCT samples. In addition, the re-analyses of the RCT samples for mercury species occurred several months after sample receipt at SRNL and therefore the impact of storage time, prior to these re-analyses[c], 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.

The high analytical uncertainty results for elemental mercury and inorganic mercury in the caustic "as-received" RCT sample can be attributed to several factors, which are all related to the solubility of elemental/inorganic mercury and representative aliquot sampling for purgeable mercury (elemental mercury) analysis. The solubility for elemental/ inorganic mercury in aqueous solutions such as water or caustic media is extremely low[d] and ranges from 0.024 to 0.059 mg/L. The measured concentration for elemental mercury in the "as-received" RCT sample (22.2 mg/L) is more than 370-900 times higher than its solubility limit in water or caustic solution. At this extreme saturation level for elemental mercury, several other forms of mercury are also present in such media including elemental mercury, mercury beads, mercury amalgams, mercuric oxide $(HgO)_s$, mercury nanoparticles, and mercuric hydroxides $(Hg(OH)_x)$ to name a few[e]. The presence of these various forms of mercury and the fact that elemental mercury is above its solubility limit in the caustic RCT sample renders the analytical results for elemental mercury biased low and is responsible for the poor mass balance results for total mercury in the "as-received" RCT sample

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[c] A. J. Boggess and T. L. White, "Recalibration and Validation of Mercury Speciation Methods," SRNL-RP-2021-05291, December 22, 2021.

[d] Isao Sanemasa, "The solubility of elemental mercury vapor in water," Bulletin of the Chemical Society of Japan, vol. 48(6), 1795-1798 (1975).

[e] C. 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.

when compared to mercury mass sum for the mercury species. It is also worth mentioning that both elemental mercury and ionic mercury form the bulk of the mercury species in this “as-received” RCT sample. Therefore, if the one sigma analytical uncertainty for each of this two mercury species is 40%, the mercury mass balance in the sample may not be reliable.

Table 6. Elemental Analyses Results: RCT “as-received” Slurry Sample (batch 4945; SRAT batch 796)

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	<0.08	<0.08	<0.08	<0.08	na
Al	<5.09	<5.09	<5.09	<5.09	na
B	<4.15	<4.15	<4.15	<4.2	na
Ba	<0.08	<0.08	<0.08	<0.08	na
Be	<0.02	<0.02	<0.02	<0.02	na
Ca	<1.75	<1.75	<1.75	<1.8	na
Cd	<0.09	<0.09	<0.09	<0.09	na
Ce	<0.51	<0.51	<0.51	<0.5	na
Co	<0.11	<0.11	<0.11	<0.1	na
Cr	<0.72	<0.72	<0.72	<0.7	na
Cu	<0.38	<0.38	<0.38	<0.9	na
Fe	8.39	8.39	8.26	8.3	0.9
Gd	<0.08	<0.08	<0.08	<0.08	na
K	<11.16	<11.16	<11.16	<11.2	na
La	<0.06	<0.06	<0.06	<0.06	na
Li	<6.07	<6.07	<6.07	<6.1	na
Mg	<0.75	<0.75	<0.75	<0.8	na
Mn	3.28	3.30	3.30	3.3	0.3
Mo	<0.24	<0.24	<0.24	<0.2	na
Na	8322	8243	8338	8301	0.6
Ni	<1.56	<1.56	<1.56	<1.6	na
P	<8.31	<8.31	<8.31	<8.3	na
Pb	<2.71	<2.71	<2.71	<2.7	na
S	<15.58	<15.58	<15.58	<15.6	na
Sb	<1.17	<1.17	<1.17	<1.2	na
Si	34.44	35.07	34.44	34.7	1.1
Sn	<9.14	<9.14	<9.14	<9.1	na
Sr	<0.11	<0.11	<0.11	<0.1	na
Th	<1.66	<1.66	<1.66	<1.7	na
Ti	<0.59	<0.59	<0.59	<0.6	na
U	<2.39	<2.39	<2.39	<2.4	na
V	<0.29	<0.29	<0.29	<0.3	na
Zn	<0.43	<0.43	<0.43	<0.4	na
Zr	<0.22	<0.22	<0.22	<0.2	na
As	<0.016	<0.016	<0.016	<0.02	na
Sulfur	<15.60	<15.60	<15.60	<15.6	na
Se	<0.016	<0.016	<0.016	<0.02	na
Total mercury	280.2	263.7	214.2	252.7	13.6
Methyl mercury	not analyzed	47.8	47.2	47.5	0.9
Dimethyl mercury	<0.2	<0.2	<0.2	<0.2	na
[π]Elemental mercury	22.2	not analyzed	not analyzed	22.2	N=1
Ionic mercury[%]	42.7	not analyzed	not analyzed	42.7	N=1
Ethyl mercury	<1	<1	<1	<1	na

N = number of replicates. na = not applicable. [π] One sigma analytical uncertainty reported as 40%, [%] Ionic mercury is also called Inorganic mercury.

Table 7. Elemental Analyses Results: RCT Filtrate Sample **(batch 4945; SRAT batch 796)**

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	<0.08	<0.08	<0.08	<0.08	na
Al	<5.08	<5.08	<5.08	<5.08	na
B	<4.14	<4.14	<4.14	<4.1	na
Ba	<0.08	<0.08	<0.08	<0.08	na
Be	<0.02	<0.02	<0.02	<0.02	na
Ca	<1.75	<1.75	<1.75	<1.8	na
Cd	<0.09	<0.09	<0.09	<0.09	na
Ce	<0.51	<0.51	<0.51	<0.5	na
Co	<0.11	<0.11	<0.11	<0.1	na
Cr	<0.72	<0.72	<0.72	<0.7	na
Cu	<0.38	<0.38	<0.38	<0.4	na
Fe	<2.39	<2.39	<2.39	<2.4	na
Gd	<0.08	<0.08	<0.08	<0.08	na
K	<11.15	<11.15	<11.15	<11.2	na
La	<0.06	<0.06	<0.06	<0.06	na
Li	<6.07	<6.07	<6.07	<6.1	na
Mg	<0.75	<0.75	<0.75	<0.8	na
Mn	<0.24	<0.24	<0.24	<0.2	na
Mo	<0.24	<0.24	<0.24	<0.2	na
Na	7807.94	8349.71	8062.89	8073.5	3.4
Ni	<1.56	<1.56	<1.56	<1.6	na
P	<8.30	<8.30	<8.30	<8.3	na
Pb	<2.71	<2.71	<2.71	<2.7	na
S	<15.57	<15.57	<15.57	<15.6	na
Sb	<1.17	<1.17	<1.17	<1.2	na
Si	36.97	38.88	38.88	38.2	2.9
Sn	<9.13	<9.13	<9.13	<9.1	na
Sr	<0.11	<0.11	<0.11	<0.1	na
Th	<1.66	<1.66	<1.66	<1.7	na
Ti	<0.59	<0.59	<0.59	<0.6	na
U	<2.39	<2.39	<2.39	<2.4	na
V	<0.29	<0.29	<0.29	<0.3	na
Zn	<0.43	<0.43	<0.43	<0.4	na
Zr	<0.22	<0.22	<0.22	<0.2	na
As	<1.0E-03	<1.0E-03	<1.0E-03	<1.0E-03	na
Sulfur	4.5E+00	4.6E+00	4.4E+00	4.5E+00	na
Se	<1.0E-03	<1.0E-03	<1.0E-03	<1.0E-03	na
Total mercury	171.9	167.4	160.2	166.5	3.6
Methyl mercury	50.4	49.8	not analyzed	50.1	0.9, N=2
Dimethyl mercury	<0.2	<0.2	<0.2	<0.2	na
[π] Elemental mercury	1.81	not analyzed	not analyzed	1.81	na
Ionic mercury [%]	27.2	not analyzed	not analyzed	27.2	na
Ethyl mercury	<0.1	<0.1	<0.1	<0.1	na

N = number of replicates; [π] One sigma analytical uncertainty reported as 40%, [%] Ionic mercury is also called inorganic mercury; na = not applicable.

4.4 Select Radionuclides

Total beta activity in the “as-received” RCT sample averaged 1.15E+07 dpm/mL (0.6 %RSD) and an average activity concentration of 5.20E-03 Ci/L. The average total alpha activity was an upper bound at <1.02E+05 dpm/mL (<4.61E-05 Ci/L). As shown in Table 8, average detected radionuclide activities in the unfiltered “as-received” RCT sample included Ni-63 (1.38E+04 dpm/mL, 25.0 %RSD (6.20E-06 Ci/L)); Tc-99 (1.37E+03 dpm/mL, 6.2 %RSD (6.16E-07 Ci/L)); I-129 (6.62E+01 dpm/mL, 14.5 %RSD (2.98E-08 Ci/L)); Th-232 (3.66E-01 dpm/mL, 0.4 %RSD (1.65E-10 Ci/L)); U-235 (9.85E-02 dpm/mL, 1.0 %RSD (4.44E-11 Ci/L)); Np-237 (3.74E+00 dpm/mL, 1.1 %RSD (1.68E-09 Ci/L)); U-238 (1.75E+00 dpm/mL, 0.4 %RSD (7.87E-10 Ci/L)); Pu-238 (3.12E+04 dpm/mL, 5.6 %RSD (1.41E-05 Ci/L)); Pu-239/240 (1.87E+03 dpm/mL, 13.4 %RSD (8.43E-07 Ci/L)); Pu-241 (5.87E+03 dpm/mL, 9.6 %RSD (2.64E-06 Ci/L)).

Both Ba-137m and Y-90 activities were calculated as 94.7%[6] of the Cs-137 and 100 % of the Sr-90 activities, respectively. As presented in Table 8, the primary beta emitting radionuclides in the RCT sample include Sr-90, Y-90 and Cs-137 at average activity of 8.70E+05 dpm/mL, 11.2 %RSD (3.92E-04 Ci/L), 8.70E+05 dpm/mL, 11.2 %RSD (3.92E-04 Ci/L) and 7.39E+06 dpm/mL, 0.5% RSD, (3.33E-03 Ci/L), respectively, in the “as-received” RCT sample. The primary gamma emitting radionuclide is Ba-137m at average activity of 7.00E+06 dpm/mL, 0.5 %RSD (3.15E-03 Ci/L) for the “as-received” RCT sample. The average Cs-135 activity in the “as-received” RCT sample (2.75E+00 dpm/mL,1.6 %RSD) was statistically not different from the average Cs-135 activity (2.66E+00 dpm/mL, 0.9 %RSD) in the filtrate.

Total beta activity in the RCT sample-filtrate averaged 9.38E+06 dpm/mL, 0.5 %RSD (4.23E-03 Ci/L), while the total alpha activity was an upper bound and averaged <1.02E+05 dpm/mL (<4.57E-05 Ci/L). As shown in Table 9, Sr-90, Y-90 and Cs-137 activities in the RCT filtrate sample average 1.29E+05 dpm/mL, 1.4 %RSD (5.83E-05 Ci/L), 1.29E+05 dpm/mL, 1.4 %RSD (5.83E-05 Ci/L), and 7.17E+06 dpm/mL, 0.8 %RSD (3.2E-03 Ci/L), respectively. The primary gamma emitting radionuclide, Ba-137m, activity averaged 6.79E+06 dpm/mL, 0.8 %RSD (3.06E-03 Ci/L) for the RCT filtrate.

The other detected radionuclides (above instrument detection limits) activities in the RCT filtrate sample, as presented in Table 9, included only U-235 (8.68E-02 dpm/mL, 1.0 %RSD (3.91E-11Ci/L)) and U-238 (1.55E+00 dpm/mL, 0.6 %RSD (6.98E-10 Ci/L)), Cs-135 (2.66E+00 dpm/mL, 0.9 %RSD (1.20E-09 Ci/L)) and Tc-99 (1.24+03 dpm/mL, 4.1 %RSD (5.57E-7Ci/L)).

As presented in the analytical results in Tables 8 and 9, there seemed to be a significant drop in the activities of certain radionuclides (Ni-63, I-129, Th-232, Np-237, Pu-238, Pu-238/240, and Pu-241) in the filtrate RCT sample. The activities of these radionuclides in the filtrate dropped below the instrument detection limit compared to their activities in the “as-received” RCT sample. The total beta activity in the RCT filtrate also showed a significant drop. Total beta activity in the “as-received” RCT (1.15E+07 dpm/mL, 0.6 %RSD) sample dropped by about 18.4% in the filtrate (9.38E+06 dpm/mL, 0.5 %RSD). Other radionuclides (Sr-90, Tc-99, U-235, and U-238) showed marginal decrease in activity in the filtrate compared to their activities in the “as-received” RCT sample, as presented in Tables 8 and 9. Cesium-137 and cesium-135 activities did not show any measurable difference between its activities in the RCT filtrate and the “as-received” RCT sample. The slight difference in Cs-137 activity between the RCT filtrate and the "as-received" RCT sample was within the 5% analytical uncertainty by counting method (gamma scan) for cesium-137.

These large differences in the activities of some radionuclides in the filtrate RCT sample compared to their activities in the "as-received" RCT sample would seem to indicate that these radionuclides exist in the "as-received" RCT 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.

The %RSD for all analytes with measurable minimum detectable activity, as summarized in Table 8 and 9 are less than 15%, with analytical results for Ni-63 being the exception. The %RSD for Ni-63 at 25.0 %, with a corresponding one sigma analytical uncertainty of 12.68% (Table 8), is 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). The source of this large variation in Ni-63 result is unknown.

The analytical results for cesium removed gamma characterizations (Na-22, Al-26, K-40, Co-60, Nb-94, Ru-103, Ru-106, Sb-125, Sb-126, Sn-126, Ce-144, Eu-152, Eu-154, Eu-155, Np-239, Am-241 and Am-243) for the RCT samples, both "as-received" RCT and filtrate RCT samples are presented in Tables 10 and 11, respectively. In the analysis of the "as-received" RCT sample, only Eu-154 and Am-241 showed activities above instrument detection limits, and these activities averaged 5.60E+03 dpm/mL (4.0 %RSD) and 3.01E+04 dpm/mL (12.6 %RSD), respectively. The activities of all the other analytes, as shown in Table 10, were below instrument detection limits.

No activities were above instrument detection limits for all the analytes for cesium removed gamma characterizations of the RCT filtrate sample, as shown in Table 11. Hot particles bearing Eu-154 and Am-241, identified in the "as-received" RCT sample, must have been retained in the filter membrane as part of the solid fractions during the solid/liquid separation to produce the RCT filtrate sample. For comparison, it is worth noting that Tables 10 and 11 also show the cesium removed gamma analytical results for the 2 molar nitric acid blanks used for diluting the RCT samples prior to analysis.

Table 8. Select Radionuclide Analytical Results for the "as-received" RCT Slurry (batch 4945; SRAT batch 796)

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L RCT slurry	%RSD N = 3	One sigma % uncertainty[#]
Total alpha	<1.02E+05	<1.02E+05	<1.03E+05	<1.02E+05	<4.61E-05	na	MDA
Total beta	1.15E+07	1.15E+07	1.16E+07	1.15E+07	5.20E-03	0.6	10.0
C-14	<2.17E+01	<4.90E+01	<2.23E+01	<3.10E+01	<1.40E-08	na	MDA
Ni-59	<2.97E+02	<3.01E+02	<2.22E+02	<2.73E+02	<1.23E-07	na	MDA
Ni-63	1.77E+04	1.22E+04	1.14E+04	1.38E+04	6.20E-06	25	12.7
Sr-90	9.39E+05	7.59E+05	9.12E+05	8.70E+05	3.92E-04	11.2	17.8
Y-90	9.39E+05	7.59E+05	9.12E+05	8.70E+05	3.92E-04	11.2	17.8
Tc-99	1.36E+03	1.46E+03	1.29E+03	1.37E+03	6.16E-07	6.2	14.7
I-129	5.98E+01	7.97E+01	5.90E+01	6.62E+01	2.98E-08	14.5	3.8
Cs-134	<3.09E+03	<4.02E+03	<3.82E+03	<3.64E+03	<1.64E-06	na	MDA
Cs-135	2.8E+00	2.72E+00	2.73E+00	2.75E+00	1.24E-09	1.6	20
Cs-137	7.43E+06	7.39E+06	7.35E+06	7.39E+06	3.33E-03	0.5	5
Ba-137m	7.03E+06	7.00E+06	6.96E+06	7.00E+06	3.15E-03	0.5	5
^Th-232	3.65E-01	3.65E-01	3.68E-01	3.66E-01	1.65E-10	0.4	20
U-233	<3.53E+03	<3.53E+03	<3.53E+03	<3.53E+03	<1.59E-06	na	MDA
U-234	<3.53E+03	<3.53E+03	<3.53E+03	<3.53E+03	<1.59E-06	na	MDA
U-235	9.93E-02	9.88E-02	9.74E-02	9.85E-02	4.44E-11	1.0	20
U-236	<3.53E+03	<3.53E+03	<3.53E+03	<3.53E+03	<1.59E-06	na	MDA
Np-237	3.69E+00	3.76E+00	3.77E+00	3.74E+00	1.68E-09	1.1	20
U-238	1.75E+00	1.75E+00	1.74E+00	1.75E+00	7.87E-10	0.4	20
Pu-238	2.92E+04	3.24E+04	3.20E+04	3.12E+04	1.41E-05	5.6	10.2
Pu-239/240	1.58E+03	2.01E+03	2.02E+03	1.87E+03	8.43E-07	13.4	12.8
Pu-241	5.25E+03	6.35E+03	6.01E+03	5.87E+03	2.64E-06	9.6	17.4

Pooled estimate based on Excel function SQRT((SUMSQ(xi)/n)) where applicable. ^Th-232, U-235, Np-237, U-238 and other actinide activities were calculated from ICP-MS data in Table 12, na = not applicable; N = number of replicates; MDA = minimum detectable activity.

Table 9. Select Radionuclide Analytical Results for the RCT Filtrate Sample (batch 4945; SRAT batch 796)

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L RCT filtrate	%RSD N = 3	One sigma % uncertainty[#]
Total alpha	<1.00E+05	<1.01E+05	<1.03E+05	**<1.02E+05**	<4.57E-05	na	MDA
Total beta	9.43E+06	9.37E+06	9.34E+06	9.38E+06	4.23E-03	0.5	11
C-14	<2.26E+01	<2.14E+01	<2.14E+01	<2.18E+01	<9.82E-09	na	MDA
Ni-59	<3.92E+02	<3.38E+02	<4.00E+02	<3.77E+02	<1.70E-07	na	MDA
Ni-63	<7.44E+02	<6.04E+02	<6.18E+02	<6.55E+02	<2.95E-07	na	MDA
Sr-90	1.31E+05	1.28E+05	1.30E+05	1.29E+05	5.83E-05	1.4	14.8
Y-90	1.31E+05	1.28E+05	1.30E+05	1.29E+05	5.83E-05	1.4	14.8
Tc-99	1.18E+03	1.25E+03	1.28E+03	1.24E+03	5.57E-07	4.1	15.4
I-129	<9.76E-01	<1.25E+00	<6.73E-01	<9.66E-01	<4.35E-10	na	MDA
Cs-134	<4.10E+03	<3.82E+03	<4.27E+03	<4.06E+03	<1.83E-06	na	MDA
Cs-135	2.64E+00	2.69E+00	2.66E+00	2.66E+00	1.20E-09	0.9	20
Cs-137	7.14E+06	7.23E+06	7.12E+06	7.17E+06	3.23E-03	0.8	5
Ba-137m	6.76E+06	6.85E+06	6.75E+06	6.79E+06	3.06E-03	0.8	5
Th-232	<3.87E-03	<3.87E-03	<3.87E-03	<3.87E-03	<1.74E-12	na	MDA
U-233	<2.21E+02	<2.21E+02	<2.21E+02	<2.21E+02	<9.93E-08	na	MDA
U-234	<7.63E-02	<7.63E-02	<7.63E-02	<7.63E-02	<3.44E-11	na	MDA
^U-235	8.78E-02	8.64E-02	8.64E-02	8.68E-02	3.91E-11	1.0	20
U-236	<2.28E+00	<2.28E+00	<2.28E+00	<2.28E+00	<1.03E-09	na	MDA
Np-237	<2.49E+01	<2.49E+01	<2.49E+01	<2.49E+01	<1.12E-08	na	MDA
U-238	1.54E+00	1.56E+00	1.54E+00	1.55E+00	6.98E-10	0.6	20
Pu-238	<1.08E+02	<9.72E+01	<1.05E+02	<1.04E+02	<4.67E-08	na	MDA
Pu-239/240	<9.86E+01	<5.18E+01	<7.17E+01	<7.40E+01	<3.34E-08	na	MDA
Pu-241	<1.85E+02	<1.85E+02	<1.85E+02	<1.85E+02	<8.33E-08	na	MDA

Pooled estimate based on Excel function SQRT((SUMSQ(xi)/n)) where applicable. ^U-235, U-238 and other actinide activities were calculated from ICP-MS data in Table 13, na = not applicable; N = number of replicates; MDA = minimum detectable activity.

Table 10. Cesium Removed Gamma Scan: RCT Slurry (batch 4945; SRAT batch 796)

Analyte	Blank dpm/mL	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L RCT slurry	%RSD
Na-22	<2.21E+01	<4.85E+02	<4.62E+02	<4.80E+02	<4.76E+02	<2.14E-07	**na**
Al-26	<2.07E+01	<4.56E+02	<4.35E+02	<4.51E+02	<4.47E+02	<2.02E-07	**na**
K-40	<5.56E+02	<9.73E+03	<9.42E+03	<9.15E+03	<9.43E+03	<4.25E-06	**na**
Co-60	<3.78E+01	<4.77E+02	<6.86E+02	<3.22E+02	<4.95E+02	<2.23E-07	**na**
Nb-94	<3.30E+01	<6.60E+02	<6.39E+02	<6.30E+02	<6.43E+02	<2.90E-07	**na**
Ru-103	<3.30E+01	<7.32E+02	<7.46E+02	<7.29E+02	<7.36E+02	<3.31E-07	**na**
Ru-106	<1.92E+02	<4.19E+03	<4.30E+03	<4.19E+03	<4.23E+03	<1.91E-06	**na**
Sb-125	<9.90E+01	<2.23E+03	<2.25E+03	<2.23E+03	<2.24E+03	<1.01E-06	**na**
Sb-126	<3.31E+01	<7.72E+02	<7.89E+02	<7.49E+02	<7.70E+02	<3.47E-07	**na**
Sn-126	<8.77E+01	<4.07E+03	<4.00E+03	<3.94E+03	<4.00E+03	<1.80E-06	**na**
Ce-144	<2.14E+02	<8.35E+03	<8.48E+03	<8.29E+03	<8.38E+03	<3.77E-06	**na**
Eu-152	<8.40E+01	<2.66E+03	<2.68E+03	<2.57E+03	<2.64E+03	<1.19E-06	na
Eu-154	<5.94E+01	5.48E+03	5.45E+03	5.85E+03	5.60E+03	2.52E-06	4.0
Eu-155	<9.96E+01	<4.66E+03	<4.59E+03	<4.53E+03	<4.59E+03	<2.07E-06	**na**
Np-239	<1.09E+02	<4.93E+03	<4.80E+03	<4.74E+03	<4.82E+03	<2.17E-06	**na**
Am-241	<3.08E+02	2.69E+04	2.90E+04	3.43E+04	3.01E+04	1.35E-05	12.6
Am-243	<8.03E+01	<2.87E+03	<3.49E+03	<3.44E+03	<3.27E+03	<1.47E-06	**na**

na = not applicable

Table 11. Cesium Removed Gamma Scan: RCT Filtrate (batch 4945; SRAT batch 796)

Analyte	Blank dpm/mL	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L RCT filtrate	%RSD
Na-22	<2.21E+01	<3.28E+02	<3.35E+02	<3.51E+02	<3.38E+02	<1.52E-07	**na**
Al-26	<2.07E+01	<3.09E+02	<3.14E+02	<3.30E+02	<3.18E+02	<1.43E-07	**na**
K-40	<5.56E+02	<7.66E+03	<7.49E+03	<7.74E+03	<7.63E+03	<3.44E-06	**na**
Co-60	<3.78E+01	<4.75E+02	<4.89E+02	<4.81E+02	<4.82E+02	<2.17E-07	**na**
Nb-94	<3.30E+01	<4.81E+02	<4.54E+02	<4.61E+02	<4.65E+02	<2.10E-07	**na**
Ru-103	<3.30E+01	<4.56E+02	<4.56E+02	<5.00E+02	<4.71E+02	<2.12E-07	**na**
Ru-106	<1.92E+02	<2.76E+03	<2.88E+03	<2.93E+03	<2.86E+03	<1.29E-06	**na**
Sb-125	<9.90E+01	<1.40E+03	<1.43E+03	<1.34E+03	<1.39E+03	<6.26E-07	**na**
Sb-126	<3.31E+01	<4.67E+02	<4.96E+02	<4.75E+02	<4.79E+02	<2.16E-07	**na**
Sn-126	<8.77E+01	<1.63E+03	<1.63E+03	<1.66E+03	<1.64E+03	<7.37E-07	**na**
Ce-144	<2.14E+02	<3.76E+03	<3.65E+03	<3.63E+03	<3.68E+03	<1.66E-06	**na**
Eu-152	<8.40E+01	<1.48E+03	<1.50E+03	<1.49E+03	<1.49E+03	<6.72E-07	**na**
Eu-154	<5.94E+01	<1.04E+03	<1.06E+03	<1.06E+03	<1.05E+03	<4.73E-07	**na**
Eu-155	<9.96E+01	<1.86E+03	<1.85E+03	<1.91E+03	<1.87E+03	<8.45E-07	**na**
Np-239	<1.09E+02	<2.01E+03	<1.99E+03	<2.04E+03	<2.01E+03	<2.24E-06	**na**
Am-241	<3.08E+02	<5.16E+03	<5.15E+03	<5.10E+03	<5.14E+03	<2.31E-06	**na**
Am-243	<8.03E+01	<1.45E+03	<1.45E+03	<1.46E+03	<1.45E+03	<6.53E-07	**na**

na = not applicable

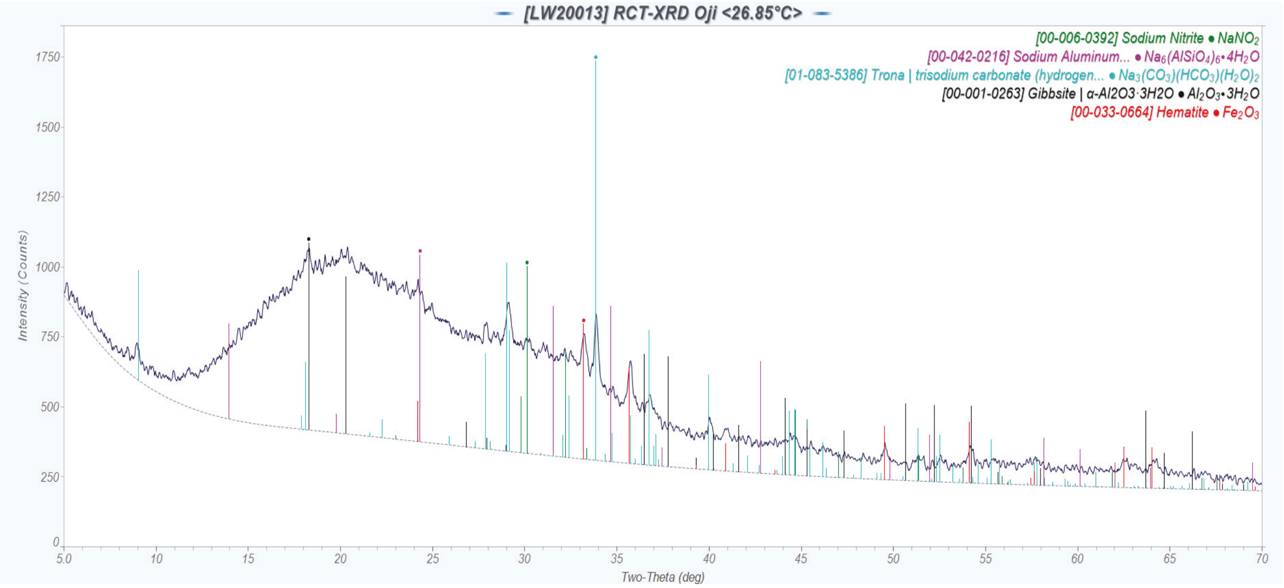


Figure 2. XRD for RCT solid fraction

4.5 <u>XRD Spectra, SEM/EDX and Particle Size Distribution</u>

The XRD patterns for the "as-received" RCT solid fraction, as shown in Figure 2, matches those of a several common minerals (sodium nitrite ($NaNO_2$), sodium aluminosilicate ($Na_5(AlSiO_4).6H_2O$), trisodium carbonate ($Na_3(CO_3)(HCO_3)(H_2O)_2$), gibbsite ($\alpha$-$Al_2O_3.3H_2O$) and hematite ($Fe_2O_3$)). The raised spectra baseline indicates the presence of amorphous materials. Thus, the RCT solid fraction is made up of partially crystalline phase minerals and non-crystalline amorphous phases.

The scanning electron microscope (SEM/EDX) information presented in Figure 3, shows that the principal quantitative elemental constituents present in the RCT sample solid fraction include mercury, iron, aluminum, sodium, chromium, manganese, nickel, thorium, silicon, calcium, and magnesium.

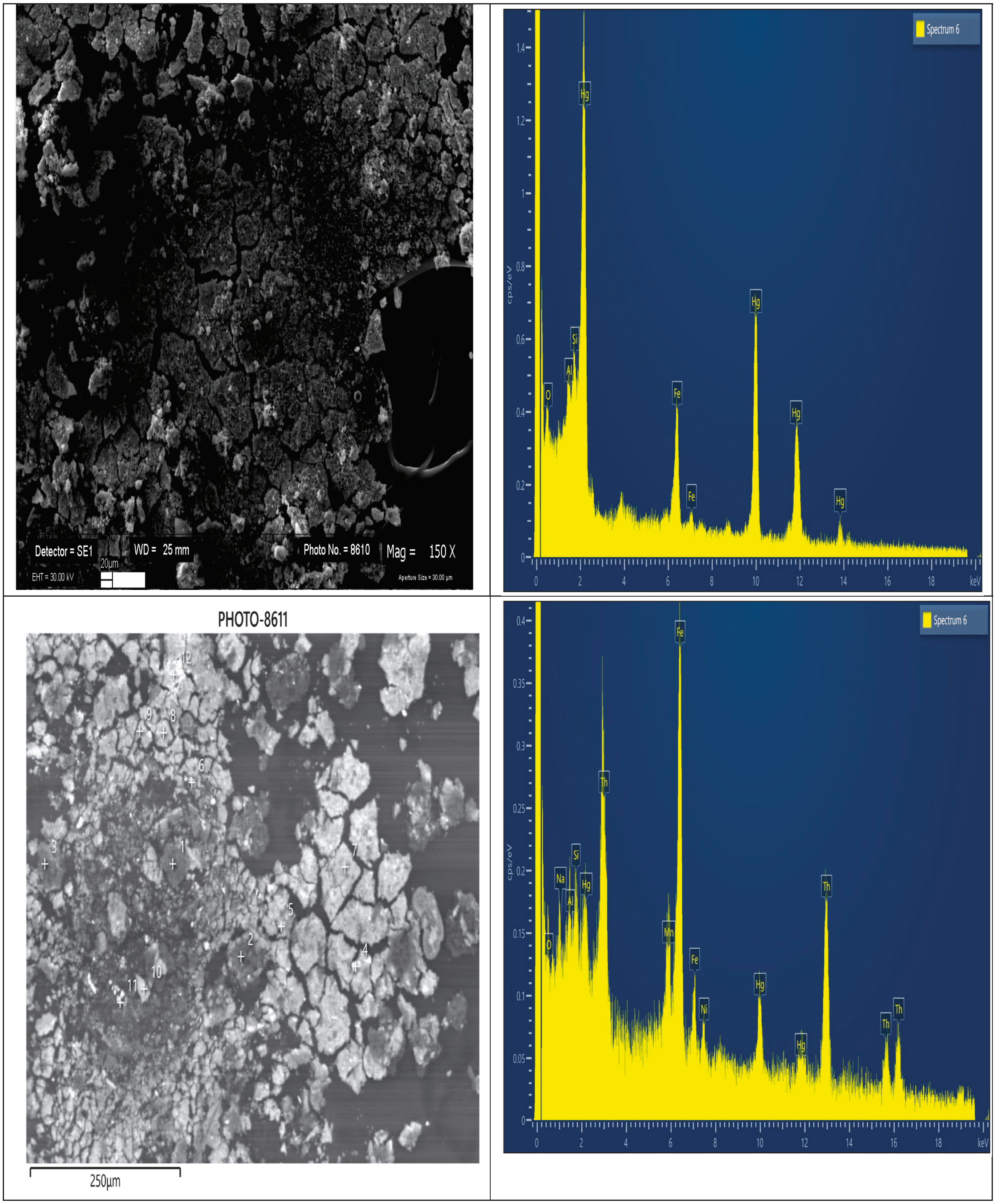
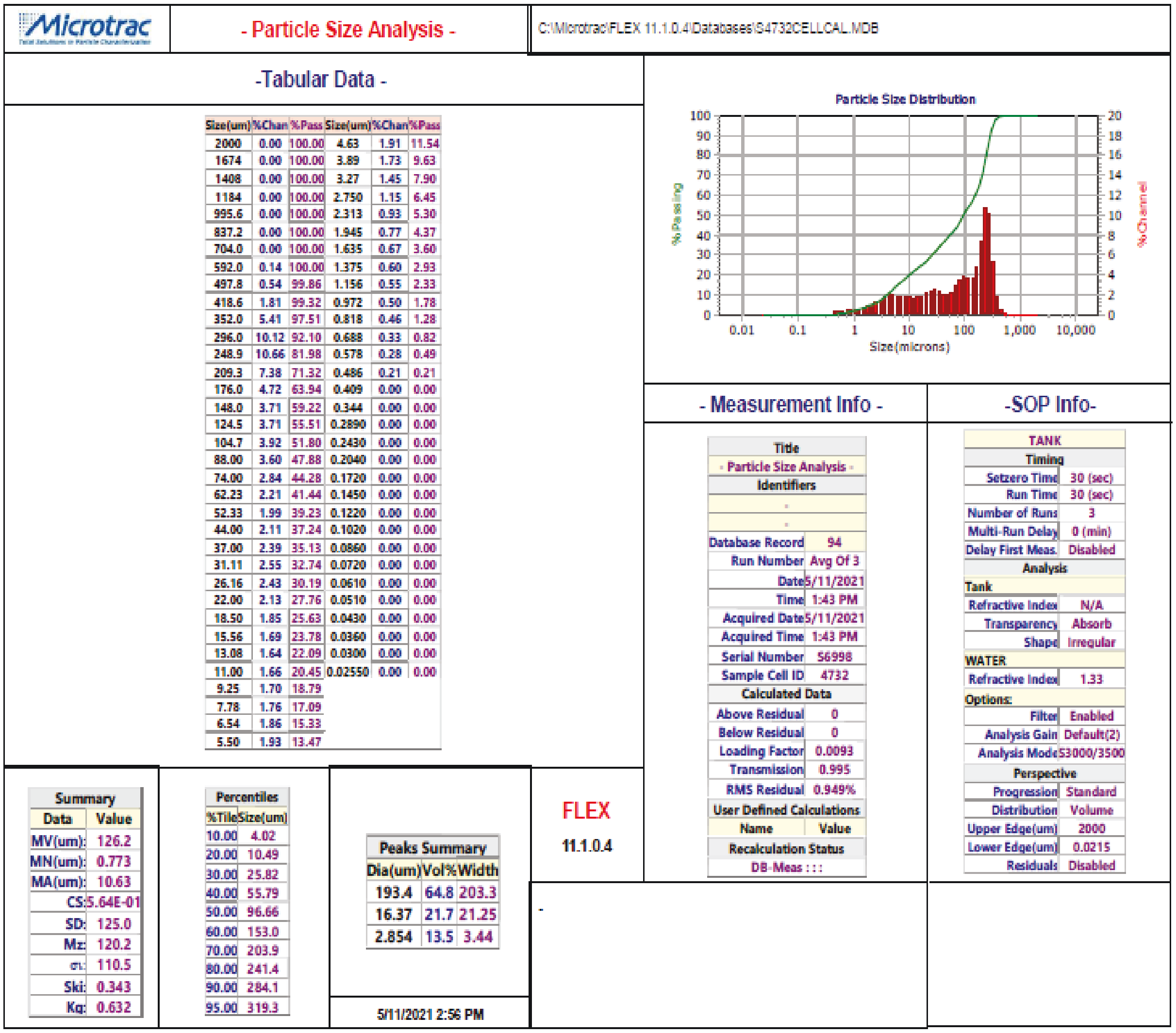


Figure 3. Scanning electron microscope images for the "as-received" RCT solid fractions



- Particle Size Analysis -

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-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
2000	0.00	100.00	4.63	1.91	11.54
1674	0.00	100.00	3.89	1.73	9.63
1408	0.00	100.00	3.27	1.45	7.90
1184	0.00	100.00	2.750	1.15	6.45
995.6	0.00	100.00	2.313	0.93	5.30
837.2	0.00	100.00	1.945	0.77	4.37
704.0	0.00	100.00	1.635	0.67	3.60
592.0	0.14	100.00	1.375	0.60	2.93
497.8	0.54	99.86	1.156	0.55	2.33
418.6	1.81	99.32	0.972	0.50	1.78
352.0	5.41	97.51	0.818	0.46	1.28
296.0	10.12	92.10	0.688	0.33	0.82
248.9	10.66	81.98	0.578	0.28	0.49
209.3	7.38	71.32	0.486	0.21	0.21
176.0	4.72	63.94	0.409	0.00	0.00
148.0	3.71	59.22	0.344	0.00	0.00
124.5	3.71	55.51	0.2890	0.00	0.00
104.7	3.92	51.80	0.2430	0.00	0.00
88.00	3.60	47.88	0.2040	0.00	0.00
74.00	2.84	44.28	0.1720	0.00	0.00
62.23	2.21	41.44	0.1450	0.00	0.00
52.33	1.99	39.23	0.1220	0.00	0.00
44.00	2.11	37.24	0.1020	0.00	0.00
37.00	2.39	35.13	0.0860	0.00	0.00
31.11	2.55	32.74	0.0720	0.00	0.00
26.16	2.43	30.19	0.0610	0.00	0.00
22.00	2.13	27.76	0.0510	0.00	0.00
18.50	1.85	25.63	0.0430	0.00	0.00
15.56	1.69	23.78	0.0360	0.00	0.00
13.08	1.64	22.09	0.0300	0.00	0.00
11.00	1.66	20.45	0.02550	0.00	0.00
9.25	1.70	18.79			
7.78	1.76	17.09			
6.54	1.86	15.33			
5.50	1.93	13.47			

- Measurement Info -

Title	
- Particle Size Analysis -	
Identifiers	
-	
-	
Database Record	94
Run Number	Avg Of 3
Date	5/11/2021
Time	1:43 PM
Acquired Date	5/11/2021
Acquired Time	1:43 PM
Serial Number	S6998
Sample Cell ID	4732
Calculated Data	
Above Residual	0
Below Residual	0
Loading Factor	0.0093
Transmission	0.995
RMS Residual	0.949%
User Defined Calculations	
Name	Value
Recalculation Status	
DB-Meas : : :	

-SOP Info-

TANK	
Timing	
Setzero Time	30 (sec)
Run Time	30 (sec)
Number of Runs	3
Multi-Run Delay	0 (min)
Delay First Meas.	Disabled
Analysis	
Tank	
Refractive Index	N/A
Transparency	Absorb
Shape	Irregular
WATER	
Refractive Index	1.33
Options:	
Filter	Enabled
Analysis Gain	Default(2)
Analysis Mode	S3000/3500
Perspective	
Progression	Standard
Distribution	Volume
Upper Edge(um)	2000
Lower Edge(um)	0.0215
Residuals	Disabled

Summary	
Data	Value
MV(um):	126.2
MN(um):	0.773
MA(um):	10.63
CS:	5.64E-01
SD:	125.0
Mz:	120.2
σι:	110.5
Ski:	0.343
Kg:	0.632

Percentiles	
%Tile	Size(um)
10.00	4.02
20.00	10.49
30.00	25.82
40.00	55.79
50.00	96.66
60.00	153.0
70.00	203.9
80.00	241.4
90.00	284.1
95.00	319.3

Peaks Summary		
Dia(um)	Vol%	Width
193.4	64.8	203.3
16.37	21.7	21.25
2.854	13.5	3.44

5/11/2021 2:56 PM

FLEX
11.1.0.4

-

Figure 4 Particle size distribution for the "as-received" RCT sample. The "as-received" RCT sample mean diameter (MV) is 126.2 microns.

The particle size distribution (PSD) result for the "as-received" RCT sample is provided in Figure 4. The PSD is left skewed (most data fall to the left). The integrated peak summary volume percent is 64.8 % for particles with diameters greater than 193.4 microns and less than 21.7 % for particle diameters less than 16.37 microns. The average particle size for this RCT sample is 126.2 ± 125.0 microns (1 sigma standard deviation).

4.6 Mass Spectral Analysis; Mass 59-252

The “as-received” RCT sample, its filtrate and the diluting acid blanks were also analyzed by mass spectrophotometer (masses 59-252), as presented in Tables 12 and 13. The last column in each table contains information on the most “likely element(s)” 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” RCT sample analysis, Table 12, and the continuations, shows that analytical results for the following masses 59, 85-112, 114-120, 122, 124, 128, 130, 133,135-148, 150, 152,154-158, 160, 182-184, 186, 196, 198, 204, 206-208, 232, 235, 237-239 are above instrument and blank detection limits, while the other masses not cited above were below instrument detection limits for those masses but above reagent blank concentrations for the corresponding masses.

However, for the RCT filtrate samples (Table 13 and continuations), the ICP-MS characterization of the sample shows fewer masses above instrument detection limits. This is expected for the RCT sample because some of the elements are insoluble at high pH. Masses above instrument and blank detection limits include the following masses 92, 94-102,104, 116, 118-120, 133, 137, 196, 198, 204, 235 and 238. The other masses for the RCT filtrate are below instrument detection limits but above reagent blank concentrations for the corresponding masses.

The concentration of Tc-99, averaging 3.20E-02 mg/L (3.1 %RSD) for the “as received” RCT sample, is approximately equal to the average Tc-99 concentration of 2.97E-02 mg/L (1.3 %RSD) in the filtrate. The concentrations of select actinides in the “as received” RCT sample, those above instrument detection limits [Th-232, U-235, Np-237 U-238 and mass 239], with the exception of U-235 and U-238, were all below instrument detection limit in the filtrate sample analytical result. Therefore, the filtration process results in the drastic reduction of the concentration of Th-232, Np-237 and mass 239 in the filtrate sample up to the point where their concentrations are below detection limits. The concentrations of those actinides that pass through the filter membrane such as U-235 and U-238 show small but measurable drops in concentrations. Uranium-235 concentration dropped from an average of 2.06E-02 mg/L (1.0 %RSD) in the “as-received” RCT sample to a low of 1.81E-02 mg/L (0.9 %RSD) in the filtrate, which is a 12 percent drop in concentration. Uranium-238 concentration in the “as-received” RCT sample dropped from an average of 2.3 to 2.1 mg/L; an 11 percent drop in concentration in the filtrate. In contrast to the fate of Pu isotopes, uranium is more soluble in the RCT sample. Therefore, the filtration process to generate the RCT filtrate sample resulted in the decrease of some actinide concentrations in the filtrate.

Table 12. Mass Spectral Analyses of RCT "as-received" Sample Slurry (batch 4945; SRAT batch 796)

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	3.45E-02	3.51E-02	3.47E-02	3.47E-02	8.59E-01	Co
82	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Se
84	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Sr
85	< 1.00E-04	1.14E-02	1.20E-02	1.21E-02	1.18E-02	3.3	Rb
86	< 1.00E-04	1.59E-03	1.59E-03	1.59E-03	1.59E-03	0.0	Sr
87	< 1.00E-04	1.64E-02	1.63E-02	1.57E-02	1.61E-02	2.3	Rb, Sr
88	< 1.00E-04	1.38E-02	1.46E-02	1.52E-02	1.45E-02	4.6	Sr
89	< 1.00E-04	1.42E-02	1.41E-02	1.49E-02	1.44E-02	3.0	Y
90	< 2.50E-04	3.73E-02	3.66E-02	3.76E-02	3.72E-02	1.3	Zr, Sr
91	< 1.00E-04	3.51E-02	3.56E-02	3.58E-02	3.55E-02	1.0	Zr
92	< 1.00E-04	6.22E-02	6.13E-02	6.23E-02	6.20E-02	0.9	Zr, Mo
93	6.40E-04	4.93E-02	4.79E-02	4.94E-02	4.89E-02	1.7	Nb
94	< 1.00E-04	5.66E-02	5.67E-02	5.67E-02	5.67E-02	0.50	Nb, Mo
95	< 1.00E-04	3.20E-02	3.12E-02	3.24E-02	3.19E-02	1.9	Mo
96	< 1.00E-04	6.21E-02	6.11E-02	6.21E-02	6.18E-02	1.0	Ru, Zr, Mo
97	< 1.00E-04	2.06E-02	2.01E-02	2.07E-02	2.05E-02	1.6	Mo, Tc
98	< 1.00E-04	4.70E-02	4.70E-02	4.71E-02	4.71E-02	0.1	Ru, Mo, Tc
99	< 1.00E-04	3.19E-02	3.11E-02	3.31E-02	3.20E-02	3.1	Tc, Ru
100	< 1.00E-04	2.45E-02	2.48E-02	2.49E-02	2.47E-02	0.7	Ru, Mo
101	< 1.00E-04	1.93E-01	1.90E-01	1.93E-01	1.92E-01	0.8	Ru
102	< 1.00E-04	1.72E-01	1.71E-01	1.69E-01	1.71E-01	0.8	Ru, Pd
103	< 1.00E-04	3.84E-02	3.84E-02	3.79E-02	3.82E-02	0.7	Rh
104	< 1.00E-04	1.01E-01	1.01E-01	1.00E-01	1.01E-01	0.4	Ru, Pd
105	< 1.00E-04	3.35E-03	3.47E-03	3.20E-03	3.34E-03	4.0	Pd
106	< 1.00E-04	3.95E-03	3.88E-03	3.84E-03	3.89E-03	1.4	Pd, Cd
107	< 1.00E-04	3.35E-02	3.19E-02	3.26E-02	3.27E-02	2.5	Ag
108	< 1.00E-04	1.61E-03	1.59E-03	1.66E-03	1.62E-03	2.2	Pd, Cd
109	< 1.00E-04	3.35E-02	3.29E-02	3.23E-02	3.29E-02	1.9	Ag
110	< 1.00E-04	4.70E-03	5.05E-03	5.28E-03	5.01E-03	5.8	Pd, Cd
111	< 1.00E-04	6.71E-03	6.52E-03	6.52E-03	6.59E-03	1.7	Cd
112	< 1.00E-04	1.26E-02	1.23E-02	1.32E-02	1.27E-02	3.8	Sn, Cd
113	< 5.00E-04	<7.97E-03	<7.97E-03	<7.97E-03	<7.97E-03	na	In, Cd
114	< 1.00E-04	1.45E-02	1.47E-02	1.39E-02	1.44E-02	2.9	Sn, Cd
116	< 1.50E-04	5.78E-02	5.80E-02	5.73E-02	5.77E-02	0.6	Sn, Cd
117	< 1.00E-04	1.16E-02	1.11E-02	1.20E-02	1.16E-02	4.1	Sn
118	< 3.00E-04	3.48E-02	3.53E-02	3.41E-02	3.48E-02	1.7	Sn
119	< 1.00E-04	4.44E-02	4.39E-02	4.36E-02	4.40E-02	1.0	Sn
120	< 4.00E-04	4.84E-02	4.71E-02	4.83E-02	4.79E-02	1.6	Sn
121	< 9.00E-04	<1.43E-02	<1.43E-02	<1.43E-02	<1.43E-02	na	Sb
122	< 1.00E-04	1.03E-02	1.02E-02	1.03E-02	1.03E-02	0.6	Te, Sn
123	< 7.00E-04	<1.12E-02	<1.12E-02	<1.12E-02	<1.12E-02	na	Sb, Te
124	< 1.50E-04	1.58E-02	1.51E-02	1.49E-02	1.52E-02	3.0	Te, Sn
125	< 2.00E-04	<3.19E-03	<3.19E-03	<3.19E-03	<3.19E-03	na	Sb, Te
126	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Te
128	< 1.00E-04	1.66E-02	1.68E-02	1.54E-02	1.63E-02	4.6	Te
130	< 1.00E-04	8.04E-02	7.98E-02	7.81E-02	7.94E-02	1.5	Te
133	5.84E-04	1.37E-01	1.37E-01	1.35E-01	1.36E-01	0.9	Cs
134	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Ba, Cs

na = not applicable; N = number of replicates.

Table 12 Continued. Mass Spectral Analyses of RCT Sample "As-Received" Slurry (batch 4945; SRAT batch 796)

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	1.51E-02	1.48E-02	1.56E-02	1.51E-02	2.5	Ba, Cs
136	< 1.00E-04	2.88E-03	3.03E-03	2.98E-03	2.96E-03	2.6	Ce, Ba
137	< 1.00E-04	4.39E-02	4.28E-02	4.45E-02	4.38E-02	2.0	Cs, Ba, La
138	2.37E-04	4.19E-02	4.13E-02	4.09E-02	4.13E-02	1.1	Ba, La, Ce
139	< 1.00E-04	3.14E-02	3.04E-02	3.20E-02	3.13E-02	2.6	La
140	< 1.00E-04	1.57E-01	1.57E-01	1.57E-01	1.57E-01	0.2	Ce
141	< 1.00E-04	2.91E-02	2.86E-02	2.91E-02	2.89E-02	1.0	Pr
142	< 1.00E-04	5.64E-02	5.53E-02	5.53E-02	5.57E-02	1.1	Nd, Ce
143	< 1.00E-04	2.78E-02	2.69E-02	2.71E-02	2.73E-02	1.8	Nd., Pm
144	< 1.00E-04	3.10E-02	3.04E-02	3.13E-02	3.09E-02	1.6	Nd, Sm, Pm
145	< 1.00E-04	1.94E-02	1.96E-02	1.93E-02	1.95E-02	0.8	Nd, Pm
146	< 1.00E-04	1.54E-02	1.60E-02	1.53E-02	1.56E-02	2.3	Nd, Sm
147	< 1.00E-04	1.14E-02	1.16E-02	1.16E-02	1.15E-02	1.0	Sm, Ti
148	< 1.00E-04	1.05E-02	1.05E-02	9.90E-03	1.03E-02	3.2	Nd, Gd, Sm
149	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Sm
150	< 1.00E-04	9.25E-03	9.59E-03	9.35E-03	9.39E-03	1.8	Nd, Gd, Sm, Eu
151	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Eu
152	< 1.00E-04	4.63E-03	4.53E-03	4.63E-03	4.60E-03	1.3	Gd, Sm, Eu
153	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Eu
154	< 1.00E-04	3.09E-03	3.06E-03	3.22E-03	3.12E-03	2.7	Gd, Sm, Eu, Dy
155	< 1.00E-04	7.26E-03	7.64E-03	7.50E-03	7.47E-03	2.6	Gd
156	< 1.00E-04	1.09E-02	1.08E-02	1.09E-02	1.08E-02	0.8	Gd, Dy
157	< 1.00E-04	7.93E-03	7.39E-03	7.90E-03	7.74E-03	4.0	Gd, Tb
158	< 1.00E-04	1.23E-02	1.21E-02	1.25E-02	1.23E-02	1.4	Gd, Dy, Tb
159	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Tb
160	< 1.00E-04	1.08E-02	1.03E-02	1.06E-02	1.06E-02	2.4	Gd, Dy
161	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Dy
162	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Dy, Er
163	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Dy, Ho
164	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Dy, Er
165	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Ho
166	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Er, Ho
167	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Er
168	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Er, Yb
169	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Tm
170	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Er, Yb
171	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Yb
172	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Yb
173	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Yb
174	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Yb, Hf
175	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Lu
176	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Lu, Hf, Yb
177	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Hf
178	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Hf
179	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Hf
180	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Hf, W, Ta
181	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Ta
182	< 1.00E-04	6.15E-03	5.94E-03	5.90E-03	6.00E-03	2.2	Hf, W

na = not applicable; N = number of replicates.

Table 12 Continued. Mass Spectral Analyses of RCT Sample "as-received" Slurry (batch 4945; SRAT batch 796)

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	3.24E-03	3.18E-03	3.24E-03	3.22E-03	1.0	W
184	< 1.00E-04	7.09E-03	6.98E-03	7.12E-03	7.06E-03	1.1	W
185	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Re
186	< 1.00E-04	6.97E-03	6.77E-03	6.83E-03	6.86E-03	1.5	Os, W
187	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Re, Os
188	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Os
189	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Os
191	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Ir
193	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Ir, Pt
194	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pt
195	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pt
196	< 1.00E-04	1.74E-01	1.78E-01	1.78E-01	1.77E-01	1.5	Hg, Pt
198	< 4.00E-04	1.10E+01	1.16E+01	1.12E+01	1.13E+01	2.6	Hg, Pt
203	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Tl
204	< 3.00E-04	5.83E+00	6.00E+00	5.78E+00	5.87E+00	2.0	Pb, Hg
205	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Tl
206	1.33E-03	3.78E-02	3.80E-02	3.83E-02	3.80E-02	0.6	Pb
207	1.14E-03	3.21E-02	3.23E-02	3.23E-02	3.22E-02	0.3	Pb
208	2.77E-03	7.89E-02	7.91E-02	8.01E-02	7.94E-02	0.8	Pb
230	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Th
232	< 1.00E-04	1.50E+00	1.50E+00	1.51E+00	1.50E+00	0.4	Th, U
233	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	U
234	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	U
235	< 1.00E-04	2.07E-02	2.06E-02	2.03E-02	2.06E-02	1.0	U
236	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	U
237	< 1.00E-04	2.36E-03	2.40E-03	2.41E-03	2.39E-03	1.2	Np
238	< 1.00E-04	2.34E+00	2.35E+00	2.33E+00	2.34E+00	0.3	U, Pu
239	< 1.00E-04	1.08E-02	1.06E-02	1.02E-02	1.05E-02	2.7	Pu
240	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pu
241	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pu, Am
242	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pu, Am
243	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pu, Cm
244	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Pu, Cm
245	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cm
246	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cm
247	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cm, Bk
248	< 1.00E-04	<3.19E-02	<3.19E-02	<3.19E-02	<3.19E-02	na	Cm
249	< 1.00E-04	<3.19E-03	<3.19E-03	<3.19E-03	<3.19E-03	na	Cf
250	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cf
251	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cf
252	< 1.00E-04	<1.59E-03	<1.59E-03	<1.59E-03	<1.59E-03	na	Cf, Cm

na = not applicable; N = number of replicates.

Table 13. Mass Spectral Analyses of RCT Sample Filtrate (batch 4945; SRAT batch 796)

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	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Co
82	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Se
84	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sr
85	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Rb
86	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sr
87	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Rb, Sr
88	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sr
89	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Y
90	< 2.50E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Zr, Sr
91	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Zr
92	< 1.00E-04	3.55E-02	3.63E-02	3.73E-02	3.64E-02	2.43	Zr, Mo
93	6.40E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Nb
94	< 1.00E-04	2.40E-02	2.46E-02	2.41E-02	2.42E-02	1.3	Nb, Mo
95	< 1.00E-04	3.68E-02	3.80E-02	3.85E-02	3.78E-02	2.3	Mo
96	< 1.00E-04	3.82E-02	3.78E-02	3.84E-02	3.81E-02	0.8	Ru, Zr, Mo
97	< 1.00E-04	2.37E-02	2.34E-02	2.38E-02	2.36E-02	0.9	Mo, Tc
98	< 1.00E-04	5.63E-02	5.73E-02	5.64E-02	5.67E-02	1.0	Ru, Mo, Tc
99	< 1.00E-04	2.94E-02	2.96E-02	3.01E-02	2.97E-02	1.3	Tc, Ru
100	< 1.00E-04	2.58E-02	2.65E-02	2.61E-02	2.62E-02	1.30	Ru, Mo
101	< 1.00E-04	1.36E-01	1.36E-01	1.38E-01	1.37E-01	0.7	Ru
102	< 1.00E-04	1.21E-01	1.21E-01	1.24E-01	1.22E-01	1.5	Ru, Pd
103	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Rh
104	< 1.00E-04	6.82E-02	6.67E-02	6.82E-02	6.77E-02	1.2	Ru, Pd
105	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pd
106	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pd, Cd
107	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Ag
108	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pd, Cd
109	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Ag
110	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pd, Cd
111	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cd
112	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sn, Cd
113	< 5.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	In, Cd
114	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sn, Cd
116	< 1.50E-04	1.62E-02	1.67E-02	1.70E-02	1.66E-02	2.3	Sn, Cd
117	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sn
118	< 3.00E-04	3.24E+01	3.19E+01	3.29E+01	3.24E+01	1.5	Sn
119	< 1.00E-04	3.70E+01	3.72E+01	3.80E+01	3.74E+01	1.4	Sn
120	< 4.00E-04	4.26E+01	4.37E+01	4.40E+01	4.35E+01	1.7	Sn
121	< 9.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sb
122	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Te, Sn
123	< 7.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sb, Te
124	< 1.50E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Te, Sn
125	< 2.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Sb, Te
126	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Te
128	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Te
130	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Te
133	5.84E-04	1.26E-01	1.26E-01	1.29E-01	1.27E-01	1.3	Cs
134	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Ba, Cs

na = not applicable; N = number of replicates.

Table 13 Continued. Mass Spectral Analyses of RCT Sample Filtrate (batch 4945; SRAT batch 796)

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	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ba, Cs
136	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ce, Ba
137	< 1.00E-04	2.86E-02	2.84E-02	2.97E-02	2.89E-02	2.4	Cs, Ba, La
138	2.37E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ba, La, Ce
139	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	La
140	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ce
141	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Pr
142	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Ce
143	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd., Pm
144	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Sm, Pm
145	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Pm
146	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Sm
147	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Sm, Ti
148	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Gd, Sm
149	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Sm
150	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Nd, Gd, Sm, Eu
151	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Eu
152	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Sm, Eu
153	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Eu
154	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Sm, Eu, Dy
155	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd
156	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Dy
157	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Tb
158	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Dy, Tb
159	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Tb
160	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Gd, Dy
161	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Dy
162	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Dy, Er
163	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Dy, Ho
164	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Dy, Er
165	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ho
166	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Er, Ho
167	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Er
168	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Er, Yb
169	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Tm
170	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Er, Yb
171	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Yb
172	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Yb
173	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Yb
174	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Yb, Hf
175	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Lu
176	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Lu, Hf, Yb
177	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Hf
178	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Hf
179	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Hf
180	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Hf, W, Ta
181	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Ta
182	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	**< 1.59E-02**	na	Hf, W

na = not applicable; N = number of replicates.

Table 13 Continued. Mass Spectral Analyses of RCT Sample Filtrate (batch 4945; SRAT batch 796)

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	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	W
184	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	W
185	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Re
186	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Os, W
187	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Re, Os
188	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Os
189	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Os
191	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Ir
193	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Ir, Pt
194	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pt
195	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pt
196	< 1.00E-04	7.94E-02	8.04E-02	8.22E-02	8.06E-02	1.7	Hg, Pt
198	< 4.00E-04	4.96E+00	5.05E+00	5.07E+00	5.03E+00	1.2	Hg, Pt
203	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Tl
204	< 3.00E-04	2.62E+00	2.59E+00	2.57E+00	2.59E+00	0.9	Pb, Hg
205	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Tl
206	1.33E-03	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pb
207	1.14E-03	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pb
208	2.77E-03	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pb
230	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Th
232	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Th, U
233	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	U
234	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	U
235	< 1.00E-04	1.83E-02	1.80E-02	1.80E-02	1.81E-02	0.9	U
236	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	U
237	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Np
238	< 1.00E-04	2.07E+00	2.09E+00	2.07E+00	2.08E+00	0.5	U, Pu
239	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu
240	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu
241	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu, Am
242	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu, Am
243	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu, Cm
244	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Pu, Cm
245	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cm
246	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cm
247	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cm, Bk
248	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cm
249	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cf
250	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cf
251	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cf
252	< 1.00E-04	< 1.59E-02	< 1.59E-02	< 1.59E-02	< 1.59E-02	na	Cf, Cm

na = not applicable; N = number of replicates.

5.0 Conclusions

SRNL was requested by SRR (now SRMC), through a TTR, to characterization the "as-received" RCT Sample identified as sample batch 4945 SRAT batch 796, which was delivered to SRNL Shielded Cells on January 28, 2021. The RCT characterization data will be used as input to the DWPF Recycle Diversion Project.

This RCT report is the first of three sample characterization reports that will be used for this DWPF Project. The other DWPF reports will involve the characterization of the OGCT and the SMECT samples.

A summary of the characterization results for the RCT sample follows.

- The density of the unfiltered, "as-received" RCT sample averaged 1.02 g/mL (0.2% RSD) and that of the RCT filtrate sample averaged 1.00 g/mL (0.3 %RSD).
- The pH of the "as-received" RCT sample was 12.9 and that of the filtrate was 13.
- The pH of the RCT sample filtrate, based on free-OH concentration calculations in the filtrate, was 13.2 (0.1 %RSD).
- The turbidity of the "as-received" RCT slurry averaged 64.9 NTU (0.4 %RSD) and that of the filtrate averaged 1.3 NTU (1.3 %RSD).
- The weight percent total solids, dissolved solids, calculated weight percent insoluble solids and soluble solids are 2.1 wt.%, (5.7 %RSD), 2.1 wt.% (6.5 %RSD), 0.04 wt.% and 2.1 wt.%, respectively.
- The viscosity of the "as-received" RCT slurry was 0.98 cP (1.2 %RSD) and that of the RCT filtrate was 1.02 cP (2.4 %RSD). These viscosity values for the "as-received" RCT sample and the RCT filtrate are not different from that of ordinary water at 25^{o}C.
- The XRD pattern for the "as-received" RCT solid fraction matches those of several common minerals (sodium nitrite ($NaNO_2$), sodium aluminosilicate ($Na_5(AlSiO_4)_6H_2O$), trisodium carbonate ($(Na_3(CO_3)(HCO_3)(H_2O)_2)$), gibbsite ($\alpha$-$Al_2O_3.3H_2O$) and hematite ($Fe_2O_3$)). The RCT solid fraction consists of partially crystalline phase minerals and non-crystalline amorphous phases.
- Scanning electron microscope information shows that the principal quantitative elemental constituents present in the RCT sample solid fraction include iron, nickel, aluminum, sodium, chromium, manganese, silicon, calcium, magnesium mercury, and thorium.
- The particle size distribution result for the "as-received" RCT sample is left skewed (most data falls to the left) and the average particle size is 126.2 ± 125.0 microns (1 sigma standard deviation).
- Total beta activity in the "as-received" RCT sample averaged 1.15E+07 dpm/mL (0.6 %RSD) and an average activity concentration of 5.20E-03 Ci/L. The average total alpha activity was an upper bound at < 1.02E+05 dpm/mL (<4.61E-05 Ci/L).
- The primary beta emitting radionuclides in the "as-received" RCT sample include Sr-90, Y-90 and Cs-137 at average activity concentrations of 3.92E-04, 3.92E-04 and 3.33E-03 Ci/ L, respectively.
- As expected, the primary gamma emitting radionuclide in the "as-received" RCT sample is Ba-137m at average activity concentration of 3.15E-03 Ci/ L (note that the activity concentration for Ba-137m was calculated as 94.7% the Cs-137 activity concentration).
- Technetium-99 activity in the "as-received" RCT sample averaged 1.37E+03 dpm/mL (6.2 %RSD) and an average activity concentration of 6.16E-07 Ci/L.
- Iodine-129 activity in the "as-received" RCT sample averaged 6.62E+01 dpm/mL (14.5 %RSD) and an average activity concentration of 2.98E-08 Ci/L.
- Plutonium-238 activity in the "as-received" RCT sample averaged 3.12E+04 dpm/mL (5.6 %RSD) and an average activity concentration of 1.41E-05 Ci/L.
- Plutonium-241 activity in the "as-received" RCT sample averaged 5.87E+03 dpm/mL (9.6 %RSD) and an average activity concentration of 2.64E-06 Ci/L.

The activities for total beta, I-129, Pu-238 and Pu-241 in the filtrate RCT sample all showed a significant drop in comparison to their initial activities in the "as-received" RCT sample. Total beta activity in the "as-received" RCT (1.15E+07 dpm/mL (0.6 %RSD)) sample dropped by about 18.4% in the filtrate (9.38E+06 dpm/mL, 0.5%RSD). Iodine-129 activity in the "as-received" RCT sample (6.62E+01 dpm/mL, 14.5 %RSD) dropped to less than minimum detection limit in the filtrate, while Plutonium-238 and Plutonium-241 activities in the "as-received" RCT sample, respectively, at 3.12E+04 dpm/mL (5.6%RSD) and 5.87E+03 dpm/mL (9.6 %RSD), dropped to less than minimum detection limits in the filtrate sample.

The average activities for Ni-63 (1.38E+04 dpm/mL, 25 %RSD), Th-232 (3.66E-01 dpm/mL, 0.4 %RSD), Np-237 (3.74E+00 dpm/mL, 1.1 %RSD) and Pu-239/240 (1.87E+03 dpm/mL, 13.4 %RSD) were all above instrument detection limit in the "as-received" RCT sample. In the filtrate, the activities for these radionuclides were all less than minimum detection limits, which means they had been retained as part of the solid fraction during the solid liquid separation.

The activities for Cs-137, Tc-99 and U-238 in the "as-received" RCT sample were comparable to their activities in the filtrate sample; activities were about the same order of magnitude in the two types of sample media. The average Sr-90 activity in filtrate was slightly lower than its activity in the "as-received" RCT sample.

Only the analytical results for iron, sodium, manganese, and silicon were above instrument detection limits in the elemental characterization of the "as-received" RCT and filtrate samples. The concentrations for all other elements were below instrument detection limits. Sodium concentration in the "as-received" RCT sample averaged 8301 mg/L (0.6 %RSD) and that for silicon averaged 34.7 mg/L (0.4 %RSD). Sodium concentration in the filtrate RCT sample averaged 8073.5 mg/L (3.4 %RSD) and that for silicon averaged 38.2 mg/L (2.9 %RSD). The sodium and silicon concentrations in the "as-received" RCT sample and RCT filtrate samples do not show any significant differences in concentrations; the small differences are within the analytical uncertainties for the two elements. Both iron and manganese concentrations in the "as-received" RCT sample were above instrument detection limits and averaged 8.3 mg/L (0.9 %RSD) and 3.3 mg/L (0.3%RSD), respectively. Iron and manganese concentrations (<2.4 mg/L and <0.2 mg/L, respectively) in the RCT filtrate were below instrument detection limits possibly due to their retention as part of the solid fractions during filtration.

Total mercury in the "as-received" RCT sample averaged 252.7 mg/L (13.6 %RSD). Methyl mercury, elemental mercury, and ionic mercury (inorganic mercury) concentration in the "as-received" RCT sample measured 47.5 mg/L (0.9 %RSD), 22.2 mg/L, and 42.7 mg/L, respectively.

The average methyl mercury concentration in the RCT filtrate at 50.1 mg/L (0.9 %RSD) was statistically comparable to the methyl mercury concentration in the "as-received" RCT sample (47.5 mg/L (0.9 %RSD)). However, the elemental mercury dropped from a high of 22.2 mg/L in the "as-received" RCT sample to 1.81 mg/L in the filtrate, which is a drop of about 92% in the filtrate sample. Ionic mercury concentration dropped from 42.7 mg/L in the "as-received" RCT sample to 27.2 mg/L in the RCT filtrate, which is a drop of about 36% in the RCT filtrate.

Ethyl mercury and dimethyl mercury determinations in the “as-received” RCT and filtrate samples were below instrument detection limits. These mercury species may not be present in the “as-received” RCT sample at detectable levels.

The re-analyses of the RCT samples for mercury species occurred several 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 may tend to degrade over time.

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” RCT and filtrate samples. Because of the extreme saturation level for elemental mercury and other mercury forms in the RCT samples, the analytical uncertainty measurements for elemental 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 are biased. As a result, there are mercury mass balance issues with the characterization of the “as-received” RCT samples for total mercury and other mercury species.

Filtering of the “as-received” RCT sample to generate the filtrates seemed to have resulted in the retention or absorption, as part of the solid fraction, of analytes (mercury species, 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” RCT sample. This retention of the analytes resulted in the decrease in concentration 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 Slurry [3]. 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 Request[1].

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 8, 9. 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

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4. 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.
5. C.J. Martino, J.M. Pareizs, and J.D. Newell, "Thermolytic Hydrogen Generation Testing of Tank 22 Material", SRNL-STI-2018-00385, Revision 0, November 2018.
6. 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.
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8. "Technical Reviews", Manual E7, Procedure 2.60, Revision 18, December 2, 2019.
9. "Savannah River National Laboratory Technical Report Design Check Guidelines", WSRC-IM- 2002-00011, Revision 2, August 2004.

Appendix A: RCT Sample Characterization: SaM Tracking Numbers*

Analytes	SRNL SaM Tracking Number (LIMS):
Total alpha	LW20003, LW20004, LW20005, LW20041, LW20042, LW20043
Total /beta	LW20003, LW20004, LW20005, LW20041, LW20042, LW20043
C-14	LW19928-LW19930, LW20666-LW20668
Cs-135	LW20107-LW20109, LW20178-LW20180
Co-60	LW19945- LW19947, LW19996-LW19998
Ni-63	LW19949-LW19952, LW19999-LW20001
Sr-90	LW20051-LW20053, LW19999-LW20001
Tc-99	LW19949-LW19952, LW19999-LW20001
Ru-106	LW19945- LW19947, LW19996- LW19998
Sb-125	LW19945- LW19947, LW19996- LW19998
Sb-126	LW19945- LW19947, LW19996- LW19998
Sn-126	LW19945- LW19947, LW19996- LW19998
I-129	LW20667-LW20669, LW20663-LW20665
Cs-134	LW20044-LW20046, LW20006-LW20008
Cs-135	LW20107- LW20109, LW20178-LW20180
Cs-137	LW20044-LW20046, LW20006-LW20008
Ce-144	LW19945- LW19947, LW19996- LW19998
Eu-152	LW19945- LW19947, LW19996- LW19998
Eu-154	LW19945- LW19947, LW19996- LW19998
Eu-155	LW19945- LW19947, LW19996- LW19998
Th-232	LW20026- LW20029, LW19993- LW19995
U-233	LW20026- LW20029, LW19993- LW19995
U-234	LW20026- LW20029, LW19993- LW19995
U-235	LW20026- LW20029, LW19993- LW19995
U-236	LW20026- LW20029, LW19993- LW19995
U-238	LW20026- LW20029, LW19993- LW19995
NP-237	LW20026- LW20029, LW19993- LW19995
Np-239	LW20026- LW20029, LW19993- LW19995
Pu-238	LW20047-LW20049, LW20009-LW20011
Pu-239/ Pu-240	LW20047-LW20049, LW20009-LW20011
Pu-241	LW20047-LW20049, LW20009-LW20011
Am-241	LW19945- LW19947, LW19996- LW19998
ICP-ES	LW19993- LW19995, LW20030- LW20032
ICP-MS	LW20026- LW20029, LW19993- LW19995
Particle size analysis	LW20012
XRD	LW20013
SEM	LW20014
Free-OH	LW20773- LW20775
TIC/TOC	LW20415- LW20417, LW20773- LW20775
Wt. % total solids	LW19970, LW19971, LW19972
Wt. % dissolved solids	LW19963, LW19964, LW19965

*Project: IDs: LW-AD-PROJ-210105-3, LW-AD-PROJ-210106-2, LW-AD-PROJ-210106-3, and LW-AD-PROJ-210106-4.

Appendix A-Continued: RCT Sample Characterization: SaM Tracking Numbers*

Analyte	SRNL SaM Tracking Number (LIMS):
ICP-AES (Elementals)	LW19993, LW19994, LW19995, LW20030, LW20031, LW20032
As	LW20181, LW20182, LW20183, LW20175, LW20176, LW20177
Sulfur	LW20181, LW20182, LW20183, LW20175, LW20176, LW20177
Se	LW20181, LW20182, LW20183, LW20175, LW20176, LW20177
ICP-MS	LW20026, LW20027, LW20028, LW20028, LW19993, LW19994, LW19995
Total Hg	LW20055, LW20056, LW20057, LW20019, LW20020, LW20021
Methyl Hg	LW19934- LW1996
Dimethyl Hg	LW19934- LW1996
Elemental Hg	LW 19938- LW19941, LW 20015- LW 20017
Ionic mercury	LW 20015- LW 20017, LW19938- 199340
Ethyl Hg	LW20015- LW20017, LW19934- LW19936
IC-Anions	LW19982- LW19984, LW20412- LW20414
IC-Cations	LW19982- LW19984, LW20412- LW20414

*Project: IDs: LW-AD-PROJ-210105-3, LW-AD-PROJ-210106-2, LW-AD-PROJ-210106-3, and LW-AD-PROJ-210106-4.

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-EAS-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 Spectrometer (ICP-AES), which is configured in a radiological containment unit. The ICP-AES 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 RCT 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 RCT 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 RCT 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 RCT 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 Molybdo-phosphate (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 RCT 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

RCT 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 RCT 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 RCT samples that had undergone peroxide fusion dissolution were further 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 RCT 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 µg/g to µCi/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: Methyl, Dimethyl, and ethyl mercury analysis

Methylmercury 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[6].

Gross Alpha/Gross Beta

Aliquots of the RCT 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 RCT slurry or filtrate was oxidized and spiked with Tc-99^{m} 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-99^{m} 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 solid for the RCT sample slurry was 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 RCT filtrate used instead.

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

Wis = (Wts – Wds)/(1 – Wds) and
Wss = Wts - Wis
Where Wds = weight fraction dissolved solids in the supernate,
Wts = weight fraction total solids in the slurry,

Wis = weight fraction insoluble solids in the slurry and
Wss = weight fraction soluble solids in the slurry.

Density Measurement and Volume Measurements

The density of the RCT 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 ºF (18.3 ºC)

Particle Size Analysis

For this RCT 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" RCT slurry sample. A small volume of the RCT slurry (3-5 mL) was suspended in Tank 22 salt simulant described above and the particle size determined.

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