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Analysis of Defense Waste Processing Facility Sample: Off-Gas Condensate Tank Sample Batch 27862

L. N. Oji

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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 (X-TTR-H-00107), to characterize the "as-received" Off-Gas Condensate Tank (OGCT) Sample identified as sample batch 27862, which was delivered to SRNL Shielded Cells on April 30, 2021.

This OGCT report is the third of three sample characterization reports dealing with this Defense Waste Processing Facility (DWPF) Recycle Diversion Project. The other two recycling stream characterization reports are the Recycle Collection Tank (RCT)[4] and the Slurry Mix Evaporator Condensate Tank (SMECT)[5] sample report.

A summary of the average analytical results for this DWPF OGCT Sample follows.

- The densities of the "as-received" OGCT sample and the OGCT filtrate averaged 1.02 g/mL, [0.1 %RSD] and 1.00 g/mL, [0.2%RSD], respectively.
- The turbidity for the "as-received" OGCT sample and the OGCT filtrate averaged 28 NTU [0.4 %RSD], and 1.52 NTU [0.8 %RSD], respectively.
- The OGCT sample was acidic, and the sample pH was 1.52 for both the "as-received" OGCT sample the OGCT filtrate.
- The OGCT sample weight percent total solids, dissolved solids, calculated weight percent insoluble solids and soluble solids are 0.29 wt.%, (10.3 %RSD), 0.23 wt.% (4.3 %RSD), 0.06 wt.% and 0.23 wt.%, respectively.
- The "as-received" OGCT sample PSD shows a bimodal distribution of OGCT particles. The particle sizes range from 0.3 to 10 microns with a mean particle size of 2.76 microns. The two modes for the distributions are at 0.84 and 9.30 microns. The integrated peak summary volume percent is 45.3% for particles with diameters greater than 4.64 microns and less than 54.7% for particle diameters less than 0.84 microns.
- The XRD mineral compositions for the OGCT solids fraction include traces of hematite (Fe_2O_3), and sodium nitrite ($NaNO_2$). These mineral peaks are relatively weak in signal intensities and not a lot of other peaks are above the instrument background signal-to-noise ratio.
- SEM/EDX information shows that the principal elemental constituents present in the "as-received" OGCT solid fraction include elemental mercury, iron, manganese, nickel, thorium, silicon, magnesium, aluminum, sodium, and ruthenium.
- Total beta activity in the "as-received" OGCT sample averaged 5.24E+08 dpm/mL (0.9 %RSD) with an average activity concentration of 2.36E-01 Ci/L. The average total alpha activity for the "as-received" OGCT sample was below instrument detection limit at <3.12E+07 dpm/mL (<1.41E-02 Ci/L).
- The primary beta emitting radionuclides in the "as-received" OGCT sample include Sr-90, Y-90 and Cs-137 at average activities of 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L), 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L), and 4.60E+08 dpm/mL, 1.0 %RSD (2.07E-01 Ci/L), respectively.
- The primary gamma emitting radionuclide in the "as-received" OGCT sample is Ba-137m at an average activity of 4.36E+08 dpm/mL, 1.0 %RSD (1.96E-01 Ci/L).

- Technetium-99, Cs-135, I-129, and Am-241 activities in the "as-received" OGCT sample averaged 7.89E+04 dpm/mL, 7.1 %RSD (3.55E-05 Ci/L), and 2.65E+03 dpm/mL, 3.5 %RSD (1.20E-6 Ci/L), 4.47E+02 dpm/mL, 5.3 %RSD (2.02E-07), and 5.65E+04 dpm/mL, 15.3 %RSD (2.55E-05 Ci/L), respectively.
- The average activities for Ni-63, Eu-154 and Th-232 in the "as-received" OGCT sample were 1.01E+05 dpm/mL (4.55E-05 Ci/L, 95 %RSD), 1.22E+04 dpm/mL (5.50E-06 Ci/L, 16.5 %RSD), and 2.90E+00 dpm/mL (1.30E-09 Ci/L, 3.9 %RSD), respectively.
- Average actinide and Neptunium activities in the "as-received" OGCT sample include U-233 (2.10E+02 dpm/mL, 9.50E-08 Ci/L, 4.6 %RSD), U-234 (1.60E+02 dpm/mL, 7.40E-08 Ci/L, 5.0 % RSD), U-235 (2.50E+00 dpm/mL, 1.10E-09 Ci/L, 4.4 % RSD), U-236 (4.40E+00 dpm/mL, 2.00E-09 Ci/L, 5.9 %RSD), Np-237 (5.90E+01 dpm/mL, 2.60E-08 Ci/L, 3.0 %RSD) and U-238 (2.80E+01 dpm/mL, 1.30E-08 Ci/L, 3.7 %RSD).
- The activities for Pu-238, Pu-239, Pu-239/240 and Pu-241 in the "as-received" OGCT sample averaged Pu-238 (4.39E+05 dpm/mL, 1.98E-04 Ci/L (9.7 %RSD)), Pu-239 (2.40E+04 dpm/mL, 1.10E-05 Ci/L (2.0 %RSD)), Pu-239/240 (≤3.90E+04 dpm/mL, ≤1.76E-05 Ci/L), and Pu-241 (8.44E+04 dpm/mL, 3.80E-05 Ci/L (11.2 %RSD)).

The average activities for Ni-59 (absolute values used), Ni-63, I-129, Cs-135, Eu-154, Th-232, Pu-238, Pu-239, Pu-239/240 (absolute values used), Pu-241, and Am-241 in the OGCT filtrate sample all showed a significant decrease in activity in comparison to their initial activities in the "as-received" OGCT sample.

The difference in average activities for total beta, Sr-90, Y-90, Tc-99, Cs-137, Ba-137m, the uranium isotopes (U-233, U-234, U-235, U-236, U-238), and Np-237 in the OGCT filtrates in comparison to their average activities in the "as-received" OGCT sample were within the measurement uncertainties of 20% for these radionuclides.

Overall, there were significant drops in the ICP-AES analyte concentrations in the OGCT filtrates when compared to their ICP-AES concentrations in the "as-received" OGCT sample.

Of the 40 elements, including total mercury and mercury species, analyzed for in the "as-received" OGCT sample only the analytical results for 20 elements were above instrument detection limits.

Twenty six of the 40 elements analyzed in the OGCT filtrate were measured above instrument detection limits.

The elements with the most significant percentage drop in concentration (in the bracket) in the OGCT filtrate compared to their initial concentration in the "as-received" OGCT sample include Al (43%), Ba (23%), Ca (46%), Cr (33%), Fe (68%), Mg (62%), Mn (42%), Ni (72%), Th (63%), Ti (39%), As (82%), and Se (64%).

The analytical results for some elements in the OGCT filtrates were above instrument detection limits but below instrument detection limits in the "as-received" OGCT sample; possibly because of interferences due to incomplete digestions of the solids in the "as-received" OGCT sample prior to analysis or the existence of colloids after digestions. These two conditions will affect the detection limits for certain analytes, especially if the concentrations of these analytes are near their instrument detection limits. Elements that fall into this category include Ag, Cd, Gd, La, Sr, and Zn.

The predominant anion species present in both the "as-received" OGCT and OGCT filtrate samples were fluoride, chloride, nitrate, sulfate, and aluminate anion ($Al(OH)_4^-$). All other anions were less than minimum detection limits in both sample mediums. The concentrations for fluoride, chloride, nitrate, and sulfate anions in both the "as-received" OGCT and OGCT filtrate samples were almost equal in magnitude.

There seem to be a significant data scatter in the average total aid titration results for the undiluted OGCT filtrate in comparison with the titration results for the undiluted OGCT supernatant sample at pH 7, 9, and 11. Therefore, their corresponding average total acid titration results for these two types of samples are not significantly different from each other.

In the OGCT filtrate sample, the concentration for elemental mercury dropped from an average high of 82.2 mg/L in the "as-received" OGCT supernatant sample to an average of 14.7 mg/L in the OGCT filtrate, which is a drop of about 82%. The measured average concentration for ionic mercury in the OGCT supernatant sample [14.3 mg/L (11.0 %RSD)] is significantly lower than the average ionic mercury concentration in the OGCT filtrate sample, which averaged 24.5 mg/L (11.1 %RSD). The difference in ionic mercury concentration in both sample types can be attributed to the large one sigma analytical uncertainty of 40% for ionic mercury determinations.

Ethyl mercury, dimethyl mercury, and methyl mercury are not present in the "as-received" OGCT supernatant sample and OGCT filtrate samples at detectable levels.

Mercury mass balance in both the "as-received" OGCT supernatant, and OGCT filtrate sample are poor due to the concentrations of some mercury species (elemental and ionic mercury species) being above their saturation levels in the OGCT sample medium. As a result, the one sigma analytical uncertainty for elemental (purgeable mercury) and inorganic mercury analyses were reported as 40%, which means the analytical results for these two mercury species are biased low in these cases.

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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
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)[1] recycle stream away from the SRS Tank Farm, Savannah River Remediation (SRR; now Savannah River Mission Completion (SRMC)) has requested that the Savannah River National Laboratory (SRNL) assist with the evaluation and characterization of the process chemistry involved in the DWPF waste stream diversion. Of the 5 Systems Engineering Evaluation[1] options for diverting the DWPF's recycle stream, Option 3, which involves crossflow filtration and evaporation as the main unit operations, was chosen.

Based on each of the unit operations, recycle streams will be generated that require a path back to DWPF, Salt Waste Processing Facility (SWPF), and/or Effluent Treatment Facility (ETF) to achieve the goal of decoupling the DWPF recycle stream from the Tank Farm. To select the correct facility for the returns for Option 3, certain upfront decisions need to be made regarding the process chemistry of the Recycle Collection Tank (RCT). The RCT, which receives condensate from the Slurry Mix Evaporator Condensate Tank (SMECT), is in the Chemical Processing Cell (CPC) of DWPF. The RCT also receives condensate from the Off-Gas Condensate Tank (OGCT), solutions from the laboratory and floor drains, transfers of material from the Decontamination Waste Treatment Tank (DWTT), the Back-up Off-Gas Condensate Tank (BUOGCT) and the Remote Equipment Decontamination Cell (REDC), along with many other less frequent sources. The nominal operating volume of the RCT is ~9,000 gallons and upon completing a transfer out, the RCT maintains a heel of ~1,500 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 OGCT samples characterized in this report were taken while DWPF was using Antifoam 747, and DWPF has since transitioned to using Momentive™ Y-17112 antifoam*.

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

The OGCT sample sent to SRNL for characterization was identified as OGCT sample coming from batch 27862. This report presents the analytical results for the characterization of the "as-received" DWPF OGCT sample and the filtrates of this sample. These OGCT sample characterization efforts are governed by a Technical Task Request (TTR)[2] and a Task Technical and Quality Assurance Plan (TTQAP)[3]. It is the third of the three sample characterization reports supporting this DWPF recycle program. The other two DWPF reports involve the characterization of the RCT[4] and the SMECT[5] samples.

The OGCT sample was pulled on 3/15/21. This sample was taken two days after completion of pouring DWPF Batch 798. DWPF had continuously been processing DWPF Batches 797 and 798 through the melter

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

from March 4 through March 13, cycling about 15,200 gallons of condensate into the OGCT and transferring about 15,700 gallons out to recycle prior to taking the OGCT sample. Batch 798 had a heel of 1,193 gallons, on top of which it received 5,947 gallons of sludge, 1,400 gallons of flush water, 2,153 gallons of PRFT, 380 gallons of formic acid, 95 gallons of nitric acid, 20,944 of gallons SE, 15 gallons of antifoam, and 340,570 lbs of steam for concentration and mercury stripping in the SRAT, with a concentration endpoint of 5,867 gallons. The SME had a heel of 1,381 gallons from previous batches. It received 4,691 gallons from this SRAT batch, where it received an additional 10,709 lbs of frit (2 can decontaminations and the rest process frit) and 4 gallons of antifoam resulting in a final waste loading of 31.9 wt%.

Objectives

The customer requested the characterization[2,3] of the "as-received" DWPF OGCT sample (batch 27862), and the filtered OGCT sample to support the eventual diversion of the DWPF recycle stream away from the SRS Tank Farm.

The initial tasks performed on the OGCT sample involved physical characterizations [bulk densities, particle size distribution (PSD), X-ray diffraction (XRD) and scanning electron microscopy (SEM)], turbidity and pH measurements. Viscosity measurements and pH adjusted OGCT sample characterization, which were supposed to be part of this characterization request, were not performed because of high activity of the OGCT sample and the customer cancellation of the pH adjusted characterization, respectively. The OGCT sample was also characterized for weight percent total and dissolved solids, density, elemental constituents, total mercury and other mercury species, anions, total acidity and for select radionuclides.

2.0 Experimental Setups/Sample description and Preparations/Methodology

The "as-received" OGCT sample delivered to SRNL-Shielded Cell came in three capped 250 mL capacity stainless steel containers. The samples were each transferred into a clear/transparent polymethyl pentane (PMP) container. Visual images of the samples are shown in Figure 1, inserts A and B. These samples were solutions with brown coloration due to the presence of very fine brownish particles. After about 24 hours of settling, a brown layer of fine solids was visible at the bottom of each PMP container (Figure 1, insert C). 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 over the weekend (96 hours) to settle more. Figure 1, insert D, shows images of the settled solids, with the liquid fraction a lot clearer than the post 24-hour settling period. The settled solid volumes at the bottom of each container were 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 weight was 630 grams.

Analyses of this OGCT sample were performed in triplicate in most cases. To attain the detection limits for I-129, Cs-135, anions and total acid titrations, it was necessary to transport small portions (3-4 mL) of the "as-received" and undiluted OGCT sample in green shielded cell bottles, with effective shielding, to the Sensing and Metrology (SaM) group for special storage and characterizations. Normally, the characterization of radioactive tank samples for radionuclides, as specified in the TTR[2] and presented in Table 1, would involve simple bulk dilutions of the "as-received" sample in 2.0 M HNO_3 and 1.0 M HCL (mercury species) or super-Q water (cations and TIC/TOC), 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 theses sample. However, it is worth noting that these dilutions also affect the detection limit of the final analytical results.

Water dilutions or dilutions in dilute acids such as hydrochloric acid (HCL) and even hydrofluoric acid (HF) at room temperature (Shielded Cell temperature) failed to dissolve the fine brown particles, which were present in the "as-received" OGCT sample. It is possible to dissolve these types of samples in hot concentrated acid conditions such as the use of aqua regia (AQR) digestion [thermal treatment of sample aliquots in a mixture of concentration HCl and nitric acid at about 200 °C], or peroxide fusion (PF) [thermal treatment of sample aliquots in sealed mixture of alkali salts and concentrated HF acid at 600 °C] digestion. However, these hash condition cannot be used for sample characterizations for mercury species such as organo-mercury compounds; the organo-mercury compounds degrade under heat treatment. As a result of this insolubility of the OGCT solid fines at room temperature, SRNL sought and obtained permission from DWPF to make changes in some of the OGCT sample processing in preparations for their characterizations. In some analyses, as mentioned above, the "as received" sample could not be used because of the presence on insoluble fines. Therefore, it was necessary to use the decanted supernatant from the OGCT sample and AQR/PF digested slurry sample aliquots for some of these characterizations. The decanted supernatant OGCT sample was obtained by letting about 75 mL of the combined "as-received" OGCT sample to settle in an optically clear 125 mL capacity glass container for about 96 hours. After settling, the volume of fine OGCT solids at the bottom of the container was less than 0.5% of the combined "as-received" OGCT sample volume. A syringe, located about two inches from the bottom of the settling container, was used to pull some of the supernatant OGCT sample volume without disturbing the bottom layer containing the OGCT solid fines.

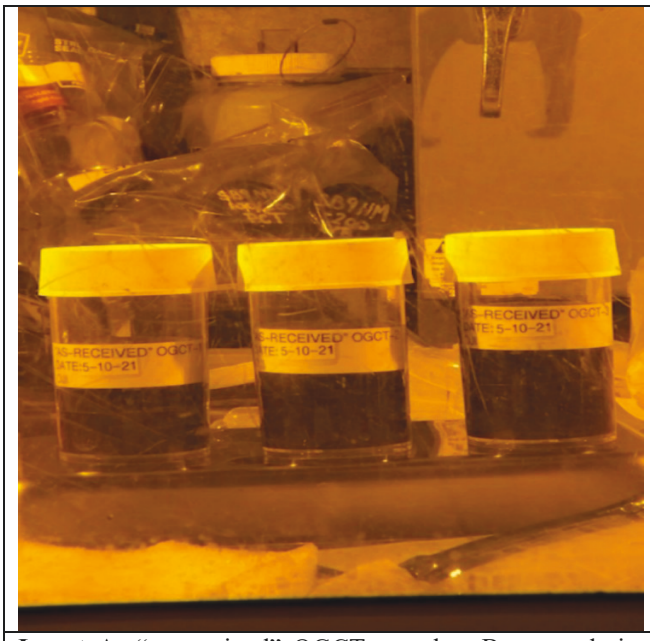
Analysis for plutonium and uranium isotopes require the complete dissolution/digestion of the "as-received" OGCT sample including the solid fines, and thus the hot acid and thermal treatment digestion methods (AQR and PF) of the "as-received" sample were used. Some of the analyses which required AQR digestion of the "as-received" OGCT sample prior to analysis included ICP-MS, ICP-AES (inorganic elements), gamma scan (Cs-134, Cs-137), cesium removed gamma scan, Ni-63/59, Sr-90, Tc-99; As, Se and S; and total mercury. To enhance the detections for Cs-135, As, Se and S, it was necessary to use the undiluted OGCT sample. Analysis for the plutonium isotopes (Pu-238, Pu-239/240 and Pu-241), total alpha and total beta were based on PF digestions, and their corresponding analysis in the filtrate were based on acid diluted OGCT filtrate.

Analyses which were based on the use of undiluted OGCT supernatant liquid (decanted), in place of the use of the "as-received" OGCT sample slurry with hard to dissolve fines, included IC-anion, total acids, I-129, and TIC/TOC. These analytes were also analyzed in the OGCT filtrate using the undiluted OGCT filtrate. The diluted OGCT decanted supernatant sample was used for mercury species quantification and the corresponding diluted OGCT filtrate was also used for mercury species. Only diluted OGCT samples are used for these mercury species analyses because of low calibration requirements for these purge and trap (P&T) gas chromatography (GC) cold-vapor atomic fluorescence spectroscopy (CVAFS) analytical methods.

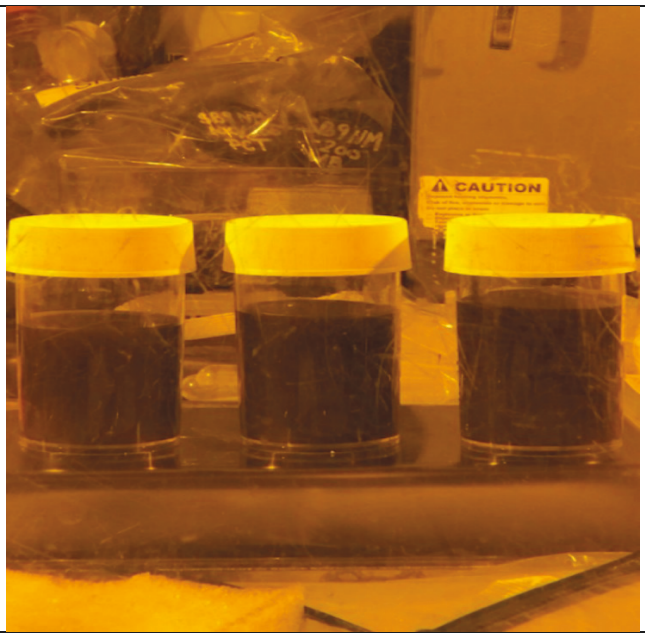
The normal determinations for density, pH, turbidity, and wt.% solids were performed using the "as-received" OGCT sample slurry and the corresponding OGCT filtrate.

The OGCT filtrate sample was diluted using dilute acids, HCL for mercury species and total mercury and nitric acids for all other analytical methods for radionuclides. All filtrate OGCT mercury sample dilutions in the SCO were in a solution of 1.0 M hydrochloric acid. The aliquots were sent to SaM for mercury analysis with refrigeration at SaM special storage facility in preparation for analyses in accordance with best practices for mercury handling and analysis [6, 7, 8, 9]. Where sample analyses called for the use of OGCT filtrate, filtered sample was obtained by a liquid /solid separation using a 0.45 µm Nalgene® nylon filter membrane. About 100 mL of the combined "as-received" OGCT sample was filtered through a Nalgene® nylon membrane to generate the OGCT filtrate sample.

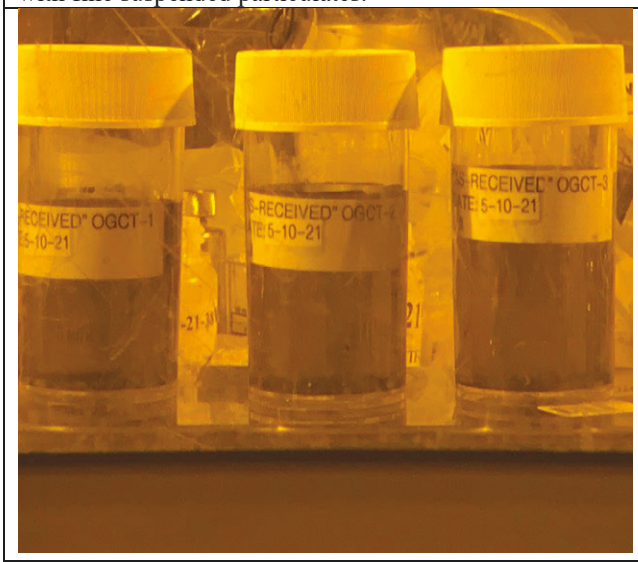
About 200 mL sample volume is required for particle size analysis (PSA) determinations using the Microtrac equipment. It is normally unsafe to transport and work with this large volume of radioactive OGCT sample slurry in a regular radioactive hood. Therefore, the normal approach to determine the particle size of radioactive samples outside the SCO involves the transport of a small volume of the radioactive material, usually less than 5 mL in this case, out of the SCO and mixing 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 OGCT sample slurry particle size determination as described in Appendix C.



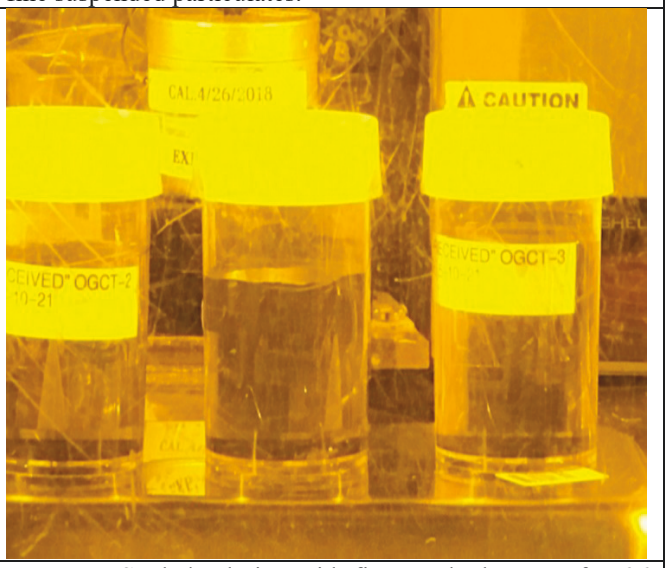
Insert A: "as-received" OGCT sample: Brown solution with fine suspended particulates.



Insert B: "as-received" OGCT sample: Brown solution with fine suspended particulates.



Insert C: Settled solution with fines at the bottom after 24 hours settling.



Insert D: Settled solution with fines at the bottom after 96 hours settling.

Figure 1. Photo images of the "as-received" OCGT sample at different settling times.

Table 1. Analytical method Summary for OGCT Sample

Analysis	Method	Preparations	Laboratory
Density	Gravimetric/volumetric	Slurry & Filtrates	SCO
#Turbidity	Turbidity meter	Slurry & Filtrates	SCO
pH	pH meter	Slurry & Filtrates	SCO
#Viscosity	Viscometer	Not determined	
Particle size analysis (PSA)	Microtrac	Slurry & Filtrates	SaM
XRD	XRD	Solid fractions	SaM
SEM/EDX	SEM/EDX	Solid fractions	SaM
Axial sulfur	ICP-AES-axial S	AQR digestion and undiluted filtrate	SaM
As, Se	ICP-MS-As/Se	AQR digestion and undiluted filtrate	SaM
Total acid	Total acid	Undiluted supernatant and undiluted filtrate	SaM
Total mercury	DMA	AQR digestion and diluted filtrate	SaM
Methyl Hg, Dimethyl-Hg, Ethyl Hg, Ionic Hg, Elemental Hg	GC-AFS*	Undiluted supernatant and undiluted filtrate	SaM
Wt. % total and dissolved solids	Gravimetric/thermal	Slurry & Filtrates	SaM
Sr-90	Extraction/beta counting	AQR digestion and diluted filtrate	SaM
Cs-137	Gamma scan	AQR digestion and diluted filtrate	SaM
Co-60, Ce-144, Eu-154, Ru-106, Sb-125 and Am-241	Cs-removed gamma scan	AQR digestion and diluted filtrate	SaM
Pu-238 and Pu-241	Extraction/alpha PHA & LSC	PF and diluted filtrate	SaM
Masses 59, 82, 84-114, 116-126, 128, 130, 133-187, 191, 193-196, 198, 203-208, 229-230, 232-251.	ICP-MS	AQR digestion and diluted filtrate	SaM
Elemental composition	ICP-AES	AQR digestion and diluted filtrate	SaM
Tc-99	Separation and LSC	AQR digestion and diluted filtrate	SaM
I-129	I-129 with separation	Undiluted supernatant and undiluted filtrate	SaM
Cs-135	Cs-135 extraction/ICP-MS	AQR digestion and undiluted filtrate	SaM
Total alpha/beta	RadScreen/LSC	PF and diluted filtrate	SaM
$Al(OH)_4^-$	Calculated from ICP-AES	n/a	PI
CO_3^{2-}	Calculated, TIC/TOC analyzer	n/a	SaM
NO_2^-, NO_3^-, SO_4^{2-}, Cl^-, F^- $C_2O_4^{2-}$, HCO_2^-, PO_4^{3-}, Br^-	IC-anions	Undiluted supernatant and undiluted filtrate	SaM
IC-Cations (NH_3^+)	IC-Cations	Undiluted supernatant and undiluted filtrate	SaM
TIC/TOC	TIC/TOC	Undiluted supernatant and undiluted filtrate	SaM
Y-90 and Ba-137m	Calculated	n/a	PI

ICP-AES = inductively coupled plasma atomic emission spectroscopy; DMA = Direct mercury analysis; ICP-MS = inductively coupled plasma mass spectroscopy; LSC = liquid scintillation counting; BT = base titration; IC = ion chromatography, TIC/TOC = total inorganic carbon/total organic carbon, and 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, ᵀ PI = Principal investigator and ¥SCO = Shielded Cell Operations, AQR = aqua regia digestion, PF = peroxide fusion, SaM = Sensing and Metrology, n/a = not applicable, XRD = X-ray diffraction.

3.0 Data Quality and Blank Evaluations

Appendix B contains the SRNL SaM Laboratory Information Management System (LIMS) numbers for tracking the analytical data presented in this report. The sample analysis completion dates are also tracked in LIMS. The SaM used reagent blanks based on dilute acids, and other reagents specific to each analytical method used in the sample preparation for analysis.

The inductively coupled plasma mass spectrometry (ICP-MS) results are given for each atomic mass and in most cases each mass number represents more than one isotope. An example 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 (PUTTA). In cases where ICP-MS and radiochemistry data give similar results for a species (e.g., Tc-99), radiochemistry was typically selected and reported due to better sensitivity and precision.

3.1 Format of the Reported Results

In general, tables containing the "as-received" OGCT slurry and supernatant analytical results are presented first followed immediately by tables containing the corresponding analytical results for the OGCT 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 or duplicate determinations but is typically not an indicator of analytical accuracy. In general, the one sigma analytical uncertainty as reported by SaM was 10%, although it was sometimes lower or higher. Specifically, the one sigma analytical uncertainties reported by SaM were: a) ~20% for ICP-MS, b) ~5% for Cs-137 determined by gamma spectroscopy, and c) 40% for elemental and ionic mercury species. As such, only two to three of the leading digits reported for the SaM analysis results should be considered significant.

In the OGCT sample characterization results presented in the tables in this report, values preceded by "<" (less than sign) indicate values for all replicates were below minimum detection limits (MDLs), and values proceeded by "≤" (less than or equal to sign) indicate that at least one of the replicate 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. 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, is equivalent to three times the standard deviation of the blank measurements.

The one sigma percent counting uncertainty for each radionuclide reported in the tables is based on the pooled estimate derived from the individual uncertainties for each replicate measurement for that radionuclide using a Microsoft Excel function, 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, UL values, or the DL values as indicated by the analytical method.

4.0 Results and Discussion

As described above, for some select analyses like those for mercury species and I-129, the OGCT decanted supernatant sample was used in the characterization for these analytes where the sample characterizations originally called for the use of the acid diluted "as-received" OGCT sample slurry. This change in sample processing for these characterizations were due to the inability of the "as-received" OGCT sample fines to dissolve in dilute acids at SRNL Shielded Cell temperature (room temperature). Thus, analyses where the analytical results are based on undiluted OGCT decanted supernatant samples instead of acid diluted "as-received" OCGT slurry sample include anions and cations (Tables 4), total acids [Table 6 (Undiluted OGCT Supernatant)], mercury species excluding total mercury (Tables 7), As, Se and sulfur (Table 7), and I-129 (Table 9). Since the weight percent insoluble solids for the "as-received" OGCT sample was small (0.06 wt. %), it is assumed that the concentration of analytes in the undiluted OGCT decanted supernatant sample were about the same order of magnitude as those of the undiluted "as-received" OGCT slurry.

Aqua regia digestions and characterization of the "as-received" OGCT sample slurry instead of acid diluted "as-received" OCGT slurry sample were used for elemental analysis (Table 7), total mercury (Table 7), select radionuclides (Table 9), and mass spectral data (Table 11 and continuations). All other analyses which called for the use of OGCT filtrate samples were performed with the filtrates using acid dilutions in some cases and in other cases, like total acid titrations and Cs-135, the filtrate samples were used without dilutions (Tables 5, 6 (filtrate only), 8, 10 and, 12 with continuations) to improve on analytical detection limits and minimize dilution effects.

4.1 Physical measurements (Density, Turbidity and pH)

The density of the "as-received" OGCT sample averaged 1.02 g/mL, 0.1 %RSD and that of the OGCT filtrate sample averaged 1.00 g/mL, 0.2 %RSD as summarized in Table 2. The pH for the combined "as-received" OGCT sample and the filtrate OGCT sample were the same, at 1.52, and the turbidity of both samples averaged 28.0 Nephelometric Turbidity unit (NTU), 0.4 %RSD and 1.52 NTU, 0.8 %RSD, respectively. The average weight percent total solids, dissolved solids and calculated weight percent insoluble and soluble solids were 0.29 wt.% (10.3 %RSD), 0.23 wt.% (4.3 %RSD), 0.06 wt.% and 0.23 wt.%, respectively (Table 3).

About 15 mL each of undiluted "as-received" OGCT sample and the OGCT filtrate is required for viscosity measurements. However, the high gamma activity for these volumes of the OGCT samples ("as-received" and filtrate samples) exceeded the whole-body dose requirement for transfer of the samples to the appropriate laboratory where the viscosity equipment (a rheometer) was located. After conferring with DWPF, it was decided to cancel the viscosity measurements for the OGCT samples. Based on experience working with the OGCT, RCT, and SMECT samples, the viscosity of the "as-received" OGCT and OGCT

filtrate samples may not be different from that of ordinary water at room temperature and like the viscosity of the RCT and SMCET samples [4, 5].

4.2 Anion Analysis

Anion analytical results are summarized in Tables 4 and 5 using the undiluted OGCT decanted supernatant sample and the undiluted OGCT filtrate, respectively. Small volumes, 3-5 mL, of these undiluted OGCT samples were taken out of the SRNL Shielded Cell for anion analysis by SaM. In these anion analytical results using both the undiluted OGCT decanted supernatant sample and the undiluted OGCT filtrate, only fluoride, chloride, nitrate, and sulfate and aluminate anion (Al(OH)4-, calculated from elemental aluminum concentration) result were above instrument detection limits. The anion analytical result for fluoride, chloride, and nitrate in both sample types using the undiluted OGCT supernatant sample and the undiluted OGCT filtrate samples were essentially the same magnitude. These concentrations for the undiluted OGCT supernatant sample averaged 15.2 mg/L, 0.4 %RSD for fluoride, 46.6 mg/L, 1.4 %RSD for chloride, 4,910 mg/L, 0.7 %RSD for nitrate, 271.0 mg/L, 1.3 %RSD for sulfate and 5.02E-03 M, 31.0 %RSD for the aluminate anion. These anion concentrations in the undiluted OGCT filtrate averaged 14.8 mg/L, 0.0 %RSD for fluoride, 46.1 mg/L, 0.9 %RSD for chloride, 4,990 mg/L, 0.0 %RSD for nitrate, 250 mg/L, 0.0 %RSD for sulfate, and 2.83E-03 M, 4.1 %RSD for the aluminate anion. The corresponding concentrations for fluoride, chloride and nitrate in both sample types were essentially equal in magnitude, meaning that the solid-liquid separations process used to generate the OGCT filtrate had no effect on the concentration of these analytes in the filtrate sample. Similarly, the concentration for the aluminate anion in the undiluted OGCT decanted supernatant sample (5.02E-03 M, 31.0 %RSD) was about 44 % higher than its concentration in the undiluted OGCT filtrate sample at 2.83E-03 M, 4.1 %RSD. This indicates that the OGCT decanted supernatant may contain some insoluble material with aluminum components which were removed during filtration.

The carbonate species (total carbon, inorganic carbon, and organic carbon) in both the “as-received” OGCT sample and the OGCT filtrate were below instrument detection limits as presented in Tables 4 and 5.

4.2.1 Total Acid and Titration Curves

The determination of total acids for the OGCT sample was based on undiluted OGCT supernatant sample and the undiluted OGCT filtrate samples. The titration curves for total acids are shown in Appendix A, and the titration data are summarized in Table 6. These titrations were performed in triplicate for pH end points of 7, 9 and 11 and 0.01N sodium hydroxide solutions were used for the titrations.

The pH 7 average total acid titration results for the undiluted OGCT filtrate and the undiluted OGCT decanted supernatant samples were 0.10 M (31.6 %RSD), and 0.06 M (9.4 %RSD), respectively. The average total acid for these two titrations at pH 7 seem to be quite different. However, the large percent relative standard deviation (%RSD) for the filtrate titration at pH 7 (31.6 %RSD) would seem to indicate a significant variation in the titration data. Therefore, the average acid titration values may not be that different. The case is the same with average total acid titration at pH 9 for both types of samples. At this pH titration level (pH 9), there is significant data scatter for the undiluted OGCT filtrate (27.0 %RSD), while the data scatter for the undiluted OGCT decanted supernatant sample is significantly smaller at 9.6 %RSD. The average total acid titration results at pH 11 for these two types of samples also follows the same trend as discussed above; there is a significant data scatter in the undiluted OGCT filtrate with 39.8 %RSD compared with a 5.9 %RSD in the data for the undiluted OGCT decanted supernatant sample. Based

on the data scatter seen in these total acid titrations, it may be acceptable to conclude that the corresponding average total acids for these two types of samples are not that different from each other.

4.3 Elemental Analysis

The two OGCT sample types ("as-received" OGCT, and undiluted OGCT filtrate) were analyzed for a total of 37 elemental components. As mentioned previously, the "as-received" OGCT sample was first digested by AQR and the resulting digested solution analyzed for the elements by ICP-AES method, while the undiluted OGCT filtrate samples was analyzed directly for elementals. Specialized methods for selenium, arsenic, and sulfur were used as presented in Appendix C. Of the 40 elements, including total mercury and mercury species presented in Table 7, the analytical results for 21 elements were measured above instrument detection limit in the AQR digested sample solution. The other elements were below instrument detection limits. On the other hand, 26 elements were measured above instrument detection limits in the undiluted OGCT filtrate samples as shown in Table 8, while the other elements were below instrument detection limits.

The elements above instrument detection level in the AQR digested "as-received" OGCT sample include Al (average concentration of 1.35E+02 mg/L, 31 %RSD), B (average concentration of 8.40E+01 mg/L, 1.4 %RSD), Ba (average concentration of 8.00E-01 mg/L, 1.6 %RSD), Ca (average concentration of 2.57E+01 mg/L, 18.9 %RSD), Cr (average concentration of 9.70E+00 mg/L, 9.7 %RSD), Fe (average concentration of 2.60E+02 mg/L, 1.0 %RSD), Mg (average concentration of 6.45E+00 mg/L, 41.4 %RSD), Mn (average concentration of 8.41E+01 mg/L, 1.2 %RSD), Na (average concentration of 4.72E+02 mg/L, 0.0 %RSD), Ni (average concentration of 1.95E+01 mg/L, 1.6 %RSD), Si (average concentration of 1.10E+02 mg/L, 3.3 %RSD), Th (average concentration of 1.34E+01 mg/L, 1.2 %RSD), Ti (average concentration of 1.38E+01 mg/L, 0.4 %RSD), U (average concentration of 3.80E+01 mg/L, 2.5 %RSD), Zr (average concentration of 1.85E+00 mg/L, 0.0 %RSD), As (average concentration of 1.09E-01 mg/L, 0.1 %RSD), S (average concentration of 8.85E+01 mg/L, 3.1 %RSD), Se (average concentration of 9.01E-01 mg/L, 2.1 %RSD). The concentrations for total mercury (average concentration of 161 mg/L, 3.2 %RSD), elemental mercury (average concentration of 82.2 mg/L, 15 %RSD), and ionic mercury (average concentration of 14.3 mg/L, 11 %RSD) were also above instrument detection limits. The concentration of all the other elements and mercury species, as shown in Table 7, were below instrument detection limit.

Twenty six of the 40 elements measured for the filtrate OCGT sample were above instrument detection level in the OGCT filtrate sample (Table 8), and these included Ag (average concentration of 2.54E-01 mg/L, 2.0 %RSD), Al (average concentration of 7.64E+01 mg/L, 4.1 %RSD), B (average concentration of 8.02E+01 mg/L, 0.7 %RSD), Ba (average concentration of 6.16E-01 mg/L, 0.0 %RSD), Ca (average concentration of 1.39E+01 mg/L, 1.6 %RSD), Cd (average concentration of 4.05E-01 mg/L, 3.2 %RSD), Cr (average concentration of 6.49E+00 mg/L, 0.3 %RSD), Fe (average concentration of 8.41E+01 mg/L, 3.3 %RSD), GD (average concentration of 9.26E-01 mg/L, 0.5 %RSD), La (average concentration of 3.42E-01 mg/L, 2.5 %RSD), Mg (average concentration of 2.46E+00 mg/L, 2.2 %RSD), Mn (average concentration of 4.84E+01 mg/L, 0.6 %RSD), Na (average concentration of 4.30E+02 mg/L, 0.6 %RSD), Ni (average concentration of 5.45E+00 mg/L, 0.8 %RSD), Si (average concentration of 1.04E+02 mg/L, 0.5%RSD), Sr (average concentration of 2.94E-01 mg/L, 1.7 %RSD), Th (average concentration of 4.97E+00 mg/L, 0.5 %RSD), Ti (average concentration of 8.39E+00 mg/L, 0.6 %RSD), U (average concentration of 3.51E+01 mg/L, 0.6 %RSD), Zn (average concentration of 7.10E-01 mg/L, 4.3 %RSD), As (average concentration of 3.88E-02 mg/L, 1.3 %RSD), S (average concentration of 8.78E+01 mg/L, 0.2

%RSD), Se (average concentration of 2.73E-01 mg/L, 0.7 %RSD), total Hg (average concentration of 1.34E+02 mg/L, 3.8 %RSD), elemental mercury (14.7 mg/L, 3.6 %RSD), and ionic mercury (24.5 mg/L, 11 %RSD). The concentration of all the other elements and mercury species, as shown in Table 8, were below instrument detection limits.

The major changes in concentrations between the 'as-received" OGCT sample and the OGCT filtrate were seen in the following elements, Al, Ba, Ca, Cr, Fe, Mg, Mn, Ni, Th, Ti, Zr, As, Se, and total Hg; with the average percent drops in concentration in the filtrate as 43%, 23%, 46%, 33%, 68%, 62%, 42%, 72%, 63%, 39%, 82%, 64%, 70%, and 17%, respectively. These measurable changes in elemental concentrations between the "as-received" OGCT sample and the OGCT filtrate may be due to their entrapment in the solid fraction during the liquid to solid separations performed to generate the filtrate solution.

Statistically there were no measurable changes for sodium, silicon, sulfur, boron, and uranium concentrations between the "as-received" OGCT sample and the OGCT filtrate. The percentage differences were within the analytical uncertainties for these elements and were less than 10% and occurred possibly because these elements and their mineral components are quite soluble in that acidic OGCT media.

The concentration of a few elements in the "as-received" OGCT sample analytical results were below instrument detection limits but were, however, detected at measurable levels in the OGCT filtrate. These include Ag, Cd, Gd, La, Sr, and Zn. These measured values in the filtrate are lower concentrations than the detection limit for the "as-received" OGCT sample.

The aqua regia digestion/dissolution of the "as-received" OGCT sample with its fine solid particles in nitric and hydrochloric acids prior to analysis for the elementals may not guarantee complete dissolution of all the particles. In some cases, such incomplete digestions of the solid particles may lead to the existence of colloids, which results in high instrument detection limit, especially if the analytes of interest are still bound in the small fraction of undigested solids or colloids. Yet, in other instances, excessive dilution of the sample, which is normally performed to ensure the calibrations curves meet the analytical conditions and to protect the inductively coupled plasma atomic emission spectroscopy (ICP-AES) plasma torch from salt accumulation damage, especially sodium salts, can also lead to large analytical uncertainties and differences. These two conditions may be responsible for the analytical result issues with the "as-received" OGCT samples.

The incomplete acid digestion and excessive dilution problems can sometimes lead to cases where the filtrate sample analysis results in the detection of more analytes compared to the "as received" sample, especially when the concentration of the analyte is near the instrument detection limit. This may be the case with the analytical results for Ag [average concentration in the "as-receive" OGCT sample is <0.30 mg/L, while the average concentration in the OGCT filtrate is 0.25 mg/L, Cd [average concentration in the "as-receive" OGCT sample is <0.71 mg/L, while the average concentration in the OGCT filtrate is 0.41 mg/L], Gd [average concentration in the "as-receive" OGCT sample is <1.84 mg/L, while the average concentration in the OGCT filtrate is 0.93 mg/L], La [average concentration in the "as-receive" OGCT sample is <0.55 mg/L, while the average concentration in the OGCT filtrate is 0.34 mg/L], Sr [average concentration in the "as-receive" OGCT sample is <0.31 mg/L, while the average concentration in the OGCT filtrate is 0.29 mg/L], and Zn [average concentration in the "as-receive" OGCT sample is <2.7 mg/L, while the average concentration in the OGCT filtrate is 0.71 mg/L]. In all these cases, the absolute

magnitude of the concentrations in the "as-received" OGCT analytical results are always comparable to their concentration in the OGCT filtrate.

4.4 Total Mercury and Mercury Species Compositions

The analytical results for total mercury and other mercury species are presented in Tables 7 and 8. The average total mercury concentration in the "as-received" OGCT decanted supernatant sample was 161 mg/L (3.2 %RSD) and 134 mg/L (3.8 %RSD) in the OGCT filtrate, which is a drop of ~17% in total mercury in the OGCT filtrate. For both sample types, the concentrations for methyl mercury, dimethyl mercury and ethyl mercury were below instrument detection limits. The average concentrations for elemental mercury in the OGCT decanted supernatant and OGCT filtrate solutions were 82.2 mg/L (15.0 %RSD) and 14.7 mg/L (3.6 %RSD), respectively. This is a drop of about 82% for elemental mercury in the OGCT filtrate.

The average concentration for ionic mercury in the OGCT supernatant was 14.3 mg/L (11 %RSD) and 24.5 mg/L (11.1 %RSD) in the OGCT filtrate sample. The measured average ionic mercury concentration in the "as-received" OGCT supernatant sample is significantly lower than the average ionic mercury concentration in the OGCT filtrate sample, which may be due to the large analytical uncertainty (40%) for ionic mercy by this method of analysis or other undefined sample preparation errors.

Both elemental mercury and ionic mercury in the "as-received" OGCT decanted supernatant sample constitute the bulk of the mercury species in the sample and thus are expected to sum up to the concentration of total mercury in the sample. However, the total mercury concentration in the "as-received" OGCT decanted supernatant sample (161 mg/L (3.2 %RSD)) is about 40% higher than the combined concentration of both elemental mercury and ionic mercury (96.5 mg/L) in the "as-received" OGCT supernatant, which is a difference of ~64.5 mg/L. This significant difference in mass balance for mercury can be attributed again to the 40% analytical uncertainty (one sigma) for these ionic mercury and elemental mercury species.

The one sigma analytical uncertainty for elemental mercury (purgeable mercury) and inorganic mercury (ionic mercury) analyses were reported as 40% for the "as-received" OGCT supernatant and the OGCT filtrates sample. The poor mercury mass balance mentioned above for these samples 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 moderately acidic samples is extremely low † and ranges from 0.024 to 0.059 mg/L. For example, the average measured concentration for elemental mercury in the "as-received" OGCT supernatant sample (82.2 mg/L) is more than 1,400-3,400 times higher than its solubility limit in water or acidic solution such as the OGCT samples.

At this extreme saturation level for elemental mercury or inorganic mercury, several other forms of mercury are also present in such a media, including elemental mercury, mercury beads, mercury amalgams, mercuric oxide (HgO)s, mercury nanoparticles, and mercuric hydroxides (Hg(OH)x) to name a few‡.. The presence of these various forms of mercury, and the fact that elemental mercury and inorganic mercury are above their solubility limit in the acidic OGCT samples, renders the analytical results for elemental mercury and inorganic mercury biased low and may be responsible for the poor mercury mass balance results in the "as-

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

received" OGCT sample. Additionally, the re-analyses[§] of the OGCT 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.

Table 2. Physical Characterization of OGCT Sample

OGCT "As-Received" Slurry					
Parameter	**Analysis-1**	**Analysis-2**	**Analysis-3**	**Average**	**%RSD, N =3**
Density, g/mL	1.02	1.02	1.02	1.02	0.1
[α]pH	1.52	-	-	na	na, N=1
Turbidity, NTU	27.9	28.1	28.1	28.0	0.4
OGCT Sample Filtrate					
Parameter	**Analysis-1**	**Analysis-2**	**Analysis-3**	**Average**	**%RSD, N =3**
Density, g/mL	1.00	1.00	1.00	1.00	0.2
[α]pH	1.52	-	-	na	na, N =1
Turbidity, NTU	1.53	1.51	1.51	1.52	0.8

N = number of replicates. [α] No average values for pH; pH is a logarithm function.

Table 3. Weight Percent Solids: OGCT Sample

Parameter	Result	Units	STDV, 1 Sigma	%RSD
Wt% Total Solids	0.29	Wt %	0.03	10.3
Wt% Dissolved Solids	0.23	Wt %	0.01	4.3
Wt% Insoluble Solids	0.06	Wt %	calculated	na
Wt% Soluble Solids	0.23	Wt %	calculated	na

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

Table 4. Anions and Total carbon Analyses Results: OGCT-Undiluted Supernatant

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F^-, mg/L	15.2	15.3	15.2	15.2	0.1	0.4
Formate, HCO_2^-, mg/L	<10	<10	<10	<10	na	na
Chloride, Cl^-, mg/L	47.1	46.9	45.9	46.6	0.6	1.4
Nitrite, NO_2^-, mg/L	<10	<10	<10	<10	na	na
Nitrate, NO_3^-, mg/L	4880	4950	4910	4913	35.1	0.7
Phosphate, PO_4^{3-}, mg/L	<10	<10	<10	<10	na	na
Sulfate, SO_4^{2-}, mg/L	267	273	273	271.0	3.5	1.3
Oxalate, $C_2O_4^{2-}$, mg/L	<10	<10	<10	<10	na	na
Bromide, Br^{1-}, mg/L	<50	<50	<50	<50	na	na
Carbonate, CO_3^{2-}, M	<1.67E-03	<1.67E-03	<1.67E-03	<1.67E-03	na	na
$Al(OH)_4^-$, M	4.74E-03	6.70E-03	3.63E-03	5.02E-03	1.56E-03	31.0
Ammonium ion, NH_4^-, M	<2.8E-04	<2.8E-04	<2.8E-04	<2.8E-04	na	na
Total carbon, gC/L	<2.0E-02	<2.0E-02	<2.0E-02	<2.0E-02	na	na
Inorganic carbon gC/L	<2.0E-02	<2.0E-02	<2.0E-02	<2.0E-02	na	na
Organic carbon, gC/L	<2.0E-02	<2.0E-02	<2.0E-02	<2.0E-02	na	na

N = number of replicates.

Table 5. Anion and Total carbon Analyses Results: Undiluted OGCT-Filtrate

Analyte	Analysis-1	Analysis-2	Analysis-3	Average	Standard deviation	%RSD N = 3
Fluoride, F^-, mg/L	14.8	14.8	14.8	14.8	0.0	0.0
Formate, HCO_2^-, mg/L	<10	<10	<10	<10	na	na
Chloride, Cl^-, mg/L	45.8	46.6	45.9	46.1	0.4	0.9
Nitrite, NO_2^-, mg/L	<10	<10	<10	<10	na	na
Nitrate, NO_3^-, mg/L	4990	4990	4990	4990	0.0	0.0
Phosphate, PO_4^{3-}, mg/L	<10	<10	<10	<10	na	na
Sulfate, SO_4^{2-}, mg/L	250	250	250	250	0.0	0.0
Oxalate, $C_2O_4^{2-}$, mg/L	<10	<10	<10	<10	na	na
Bromide, Br^{1-}, mg/L	<50	<50	<50	<50	na	na
Carbonate, CO_3^{2-}, M	<4.17E-04	<4.17E-04	<4.17E-04	<4.17E-04	na	na
$Al(OH)_4^-$, M	2.79E-03	2.96E-03	2.74E-03	2.83E-03	1.17E-04	4.1
Ammonium ion, NH_4^-, M	<5.6E-04	<5.6E-04	<5.6E-04	<5.6E-04	na	na
Total carbon, gC/L	<5.00E-03	<5.00E-03	<5.00E-03	<5.00E-03	na	na
Inorganic carbon gC/L	<5.00E-03	<5.00E-03	<5.00E-03	<5.00E-03	na	na
Organic carbon, gC/L	<5.00E-03	<5.00E-03	<5.00E-03	<5.00E-03	na	na

Table 6. Undiluted Supernatant OGCT Slurry and OGCT Filtrate Total Acids at pH 7, 9 and 11

Undiluted OGCT Supernatant					
Parameter	**Analysis-1**	**Analysis-2**	**Analysis-3**	**Average**	**%RSD, N =3**
Total acid, pH 7, M	0.06	0.06	0.07	0.06	9.4
Total acid, pH 9, M	0.07	0.08	0.09	0.08	9.6
Total acid, pH 11, M	0.17	0.19	0.19	0.18	5.9
Undiluted OGCT Sample Filtrate					
Parameter	**Analysis-1**	**Analysis-2**	**Analysis-3**	**Average**	**%RSD, N =3**
Total acid, pH 7, M	0.13	0.07	0.09	0.10	31.6
Total acid, pH 9, M	0.14	0.08	0.12	0.11	27.0
Total acid, pH 11, M	0.15	0.08	0.19	0.14	39.8

Table 7. Elemental Analyses Results: "as-received" OGCT Sample -AQR Digestion

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	<3.02E-01	<3.02E-01	<3.02E-01	<3.02E-01	na
Al	1.28E+02	1.81E+02	9.79E+01	1.35E+02	31
B	8.52E+01	8.28E+01	8.41E+01	8.40E+01	1.4
Ba	7.83E-01	8.60E-01	7.59E-01	8.00E-01	1.6
Be	<1.43E-01	<1.43E-01	<1.43E-01	<1.43E-01	na
Ca	2.31E+01	3.12E+01	2.27E+01	2.57E+01	18.9
Cd	<7.10E-01	<7.10E-01	<7.10E-01	<7.10E-01	na
Ce	<4.03E+00	<4.03E+00	<4.03E+00	<4.03E+00	na
Co	<1.06E+00	<1.06E+00	<1.06E+00	<1.06E+00	na
Cr	1.04E+01	8.64E+00	1.00E+01	9.70E+00	9.7
Cu	<5.90E+00	<5.90E+00	<5.90E+00	<5.90E+00	na
Fe	2.63E+02	2.58E+02	2.59E+02	2.60E+02	1.0
Gd	<1.84E+00	<1.84E+00	<1.84E+00	<1.84E+00	na
K	<2.32E+01	<2.32E+01	<2.32E+01	<2.32E+01	na
La	<5.49E-01	<5.49E-01	<5.49E-01	<5.49E-01	na
Li	<3.81E+01	<3.81E+01	<3.81E+01	<3.81E+01	na
Mg	5.41E+00	9.47E+00	4.45E+00	6.45E+00	41.4
Mn	8.50E+01	8.41E+01	8.31E+01	8.41E+01	1.2
Mo	<1.41E+00	<1.41E+00	<1.41E+00	<1.41E+00	na
Na	4.72E+02	4.72E+02	4.72E+02	4.72E+02	0.0
Ni	1.98E+01	1.95E+01	1.92E+01	1.95E+01	1.6
P	<7.79E+00	<7.79E+00	<7.79E+00	<7.79E+00	na
Pb	<1.89E+00	<1.89E+00	<1.89E+00	<1.89E+00	na
Sb	<5.32E+00	<5.32E+00	<5.32E+00	<5.32E+00	na
Si	1.13E+02	1.06E+02	1.10E+02	1.10E+02	3.3
Sn	<7.73E+00	<7.73E+00	<7.73E+00	<7.73E+00	na
Sr	<3.06E-01	<3.06E-01	<3.06E-01	<3.06E-01	na
Th	1.36E+01	1.34E+01	1.33E+01	1.34E+01	1.2
Ti	1.38E+01	1.39E+01	1.38E+01	1.38E+01	0.4
U	3.76E+01	3.91E+01	3.74E+01	3.80E+01	2.5
V	<1.05E+00	<1.05E+00	<1.05E+00	<1.05E+00	na
Zn	<2.73E+00	<2.73E+00	<2.73E+00	<2.73E+00	na
Zr	1.85E+00	1.85E+00	1.85E+00	1.85E+00	0.0
As	1.16E-01	1.06E-01	1.04E-01	1.09E-01	0.1
Sulfur	8.75E+01	9.16E+01	8.65E+01	8.85E+01	3.1
Se	9.00E-01	9.21E-01	8.84E-01	9.01E-01	2.1
Total mercury	1.66E+02	1.56E+02	1.61E+02	1.61E+02	3.2
Methyl mercury	<1	<1	<1	<1	na
Dimethyl mercury	<0.25	<0.25	<0.25	<0.25	na
[þ] Elemental mercury	68.3	86.4	91.9	82.2	15.0
[þ] Ionic mercury	16.1	13.6	13.2	14.3	11.0
Ethyl mercury	<0.1	<0.1	<0.1	<0.1	na

na = not applicable. Undiluted OGCT supernatant used for mercury species excluding total mercury. [þ]1 sigma Analytical uncertainty for Elemental mercury and ionic mercury are reported as 40%.

Table 8. Elemental Analyses Results: OGCT Filtrate

Element	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3
Ag	2.57E-01	2.57E-01	2.48E-01	2.54E-01	2.0
Al	7.53E+01	8.00E+01	7.40E+01	7.64E+01	4.1
B	8.05E+01	8.06E+01	7.96E+01	8.02E+01	0.7
Ba	6.16E-01	6.16E-01	6.16E-01	6.16E-01	0.0
Be	<2.43E-02	<2.43E-02	<2.43E-02	<2.43E-02	na
Ca	1.39E+01	1.42E+01	1.38E+01	1.39E+01	1.6
Cd	4.19E-01	4.02E-01	3.93E-01	4.05E-01	3.2
Ce	<1.30E+00	<1.30E+00	<1.30E+00	<1.30E+00	na
Co	<1.51E-01	<1.51E-01	<1.51E-01	<1.51E-01	na
Cr	6.47E+00	6.51E+00	6.49E+00	6.49E+00	0.3
Cu	<8.47E-01	<8.47E-01	<8.47E-01	<8.47E-01	na
Fe	8.20E+01	8.30E+01	8.72E+01	8.41E+01	3.3
Gd	9.24E-01	9.24E-01	9.32E-01	9.26E-01	0.5
K	<1.63E+01	<1.63E+01	<1.63E+01	<1.63E+01	na
La	3.42E-01	3.51E-01	3.34E-01	3.42E-01	2.5
Li	<1.95E+01	<1.95E+01	<1.95E+01	<1.95E+01	na
Mg	2.45E+00	2.52E+00	2.42E+00	2.46E+00	2.2
Mn	4.82E+01	4.87E+01	4.83E+01	4.84E+01	0.6
Mo	<2.39E-01	<2.39E-01	<2.39E-01	<2.39E-01	na
Na	4.32E+02	4.32E+02	4.28E+02	4.30E+02	0.6
Ni	5.44E+00	5.50E+00	5.41E+00	5.45E+00	0.8
P	<8.48E+00	<8.48E+00	<8.48E+00	<8.48E+00	na
Pb	<2.40E+00	<2.40E+00	<2.40E+00	<2.40E+00	na
Sb	<1.84E+00	<1.84E+00	<1.84E+00	<1.84E+00	na
Si	1.03E+02	1.04E+02	1.03E+02	1.04E+02	0.5
Sn	<1.22E+01	<1.22E+01	<1.22E+01	<1.22E+01	na
Sr	2.91E-01	2.99E-01	2.91E-01	2.94E-01	1.7
Th	4.99E+00	4.99E+00	4.94E+00	4.97E+00	0.5
Ti	8.42E+00	8.42E+00	8.33E+00	8.39E+00	0.6
U	3.50E+01	3.53E+01	3.50E+01	3.51E+01	0.6
V	<3.83E-01	<3.83E-01	<3.83E-01	<3.83E-01	na
Zn	6.84E-01	7.44E-01	7.01E-01	7.10E-01	4.3
Zr	<3.25E-01	<3.25E-01	<3.25E-01	<3.25E-01	na
As	3.94E-02	3.85E-02	3.86E-02	3.88E-02	1.3
Sulfur	8.77E+01	8.77E+01	8.80E+01	8.78E+01	0.2
Se	2.71E-01	2.73E-01	2.75E-01	2.73E-01	0.7
Total mercury	1.33E+02	1.40E+02	1.30E+02	1.34E+02	3.8
Methyl mercury	<1	<1	<1	<1	na
Dimethyl mercury	<0.1	<0.1	<0.1	<0.1	na
[þ] Elemental mercury	14.9	15.1	14.1	14.7	3.6
[þ] Ionic mercury	21.4	26.2	26	24.5	11.1
Ethyl mercury	<1	<1	<1	<1	na

na = not applicable. [þ]1 sigma Analytical uncertainty for Elemental mercury and ionic mercury are reported as 40%.

4.5 Analytical results for Select Radionuclides

Total beta activity in the "as-received" OGCT sample averaged 5.24E+08 dpm/mL (0.9 %RSD) and an average activity concentration of 2.36E-01 Ci/L. The average total alpha activity was below instrument detection limit at <3.12E+07 dpm/mL (<1.41E-02 Ci/L). As shown in Table 9, the average detected radionuclide activities in the "as-received" OGCT sample included Ni-63 (1.01E+05 dpm/mL, 95 %RSD (4.55E-05 Ci/L)); Tc-99 (7.89E+04 dpm/mL, 7.1 %RSD (3.55E-05 Ci/L)); I-129 (4.47E+02 dpm/mL, 5.3 %RSD (2.02E-07 Ci/L)); Cs-135 (2.65E+03 dpm/mL, 3.5 %RSD (1.20E-06 Ci/L)); Eu-154 (1.22E+04 dpm/mL, 16.5 %RSD (5.50E-06 Ci/L)).

Both Ba-137m and Y-90 activities were calculated as 94.6% [6] of the Cs-137 and 100 % of the Sr-90 activities, respectively. As presented in Table 9, the primary beta emitting radionuclides in the "as-received" OGCT sample include Sr-90, Y-90 and Cs-137 at average activities of 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L), 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L) and 4.60E+08 dpm/mL, 1.0 % RSD, (2.07E-01 Ci/L), respectively. The primary gamma emitting radionuclide is Ba-137m at average activity of 4.36E+08 dpm/mL, 1.0 %RSD (1.96E-01 Ci/L).

Other measured radionuclides in the "as-received" OGCT sample include Th-232 (2.90E+00 dpm/mL, 3.9 %RSD (1.30E-09 Ci/L)); U-233 (2.10E+02 dpm/mL, 4.6 %RSD (9.50E-08 Ci/L)); U-234 (1.60E+02 dpm/mL, 5.0 %RSD (7.40E-08 Ci/L)); U-235 (2.50E+00 dpm/mL, 4.4 %RSD (1.10E-09 Ci/L)); U-236 (4.40E+00 dpm/mL, 5.9 %RSD (2.00E-09 Ci/L)); Np-237 (5.90E+01 dpm/mL, 3.0 %RSD (2.60E-08 Ci/L)); U-238 (2.80E+01 dpm/mL, 3.7 %RSD (1.30E-08 Ci/L)); Pu-238 (4.39E+05 dpm/mL, 9.7 %RSD (1.98E-04 Ci/L)); Pu-239 (2.40E+04 dpm/mL, 2.0 %RSD (1.10E-05 Ci/L)); Pu-241 (8.44E+04 dpm/mL, 11.2 %RSD (3.80E-05 Ci/L)); Am-241 (5.65E+04 dpm/mL, 15.3 %RSD (2.55E-05 Ci/L)). All other radionuclide activities, as shown in Table 9, are below instrument detection limits.

Total beta activity in the OGCT sample-filtrate averaged 5.45E+08 dpm/mL, 1.6 %RSD (2.46E-01 Ci/L), while the total alpha activity was below instrument detection limit and averaged <2.26E+07 dpm/mL (<1.02E-02 Ci/L).

As presented in Table 10, the average detected radionuclide activities in the OGCT filtrate sample included Ni-63 (2.24E+04 dpm/mL, 42.9 %RSD (1.01E-05 Ci/L)); Tc-99 (7.51E+04 dpm/mL, 1.8 %RSD (3.38E-05 Ci/L)); I-129 (4.72E+01 dpm/mL, 6.7 %RSD (2.12E-08 Ci/L)); Cs-135 (2.67E+02 dpm/mL, 10.3 %RSD (1.20E-07 Ci/L)); Eu-154 (9.72E+03 dpm/mL, 10.2 %RSD (4.38E-06 Ci/L)). As shown in Table 10, Sr-90, Y-90 and Cs-137 activities in the OGCT filtrate sample averaged 1.62E+07 dpm/mL, 8.5 %RSD (7.28E-03 Ci/L), 1.62E+07 dpm/mL, 8.5 %RSD (7.28E-03 Ci/L), and 4.17E+08 dpm/mL, 10.6 %RSD (1.88E-01 Ci/L), respectively. The primary gamma emitting radionuclide, Ba-137m, activity averaged 3.95E+08 dpm/mL, 10.6 %RSD (1.78E-01 Ci/L) for the OGCT filtrate.

The other detected radionuclides activities (above instrument detection limits) in the OGCT filtrate sample, as presented in Table 10, include Th-232 (1.11E+00 dpm/mL, 0.3 %RSD (5.01E-10 Ci/L)); U-233 (1.87E+02 dpm/mL, 1.3 %RSD (8.43E-08 Ci/L)); U-234 (1.51E+02 dpm/mL, 2.4 %RSD (6.28E-08 Ci/L)); U-235 (2.39E+00 dpm/mL, 0.3 %RSD (1.08E-09 Ci/L)); U-236 (4.17E+00 dpm/mL, 0.9 %RSD (1.88E-09 Ci/L)), Np-237 (5.18E+01 dpm/mL, 0.8 %RSD (2.33E-08 Ci/L)); U-238 (2.65E+01 dpm/mL, 0.2 %RSD (1.20E-08 Ci/L)); Pu-238 (2.82E+04 dpm/mL, 9.2 %RSD (1.27E-05 Ci/L)); Pu-239 (1.49+03 dpm/mL 1.8 %RSD (6.72E-07Ci/L)); Pu-241 (1.13+04 dpm/mL 20.7 %RSD (5.10E-06 Ci/L)); Am-241 (3.32E+04 dpm/mL 10.7 %RSD (1.49E-05 Ci/L)); and Co-60 (4.70E+02 dpm/mL, 10.0 %RSD (2.12E-

07Ci/L)). The activities for all the other radionuclides, as shown in Table 10, were below instrument detection limits in the OGCT filtrate.

As presented in the analytical results in Tables 9 and 10, there seem to be a significant drop in the activities of certain radionuclides (Ni-59 (absolute values used), Ni-63, Sr-90, Y-90, I-129, Cs-135, Eu-154, Th-232, Pu-238, Pu-239, Pu-239/240 (absolute values used), Pu-241, and Am-241) in the filtrate OGCT sample when compared to their initial activities in the "as-received" OGCT sample. The activities for these radionuclides in the filtrate dropped by 78%, 78%, 17%, 17%, 89%, 90%, 20%, 62%, 94%, 94%, 95%, 87% and 41%, respectively.

Other radionuclides showed marginal activity differences between the "as-received" OCGT sample and the OGCT filtrate. Radionuclides in this category include total beta (4.0% difference), Tc-99 (4.8% difference), Cs-137 and Ba-137m (9.3% and 9.4% difference, respectively), U-234 (5.6% difference), U-235 (4.4% difference), U-236 (5.2% difference), U-238 (5.4% difference), U-233 (11.0% difference), and Np-237 (12.2% difference).

These differences in the activities of some radionuclides in the OGCT filtrate sample compared to their activities in the "as-received" OGCT sample would seem to indicate that these radionuclides exist in the "as-received" OGCT 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, Tc-99, Cs-137, and the uranium isotopes, which are soluble, would easily become part of the liquid fraction in this OGCT acidic media.

The activity for Co-60 in the "as-received" OGCT sample was below instrument detection limit at <9.16E+02 dpm/mL (<4.12E-07 Ci/L). However, the OGCT filtrate activity for Co-60 was above instrument detection limit and averaged 4.70E+02 dpm/mL, 10 %RSD (2.12E-07 Ci/L). The reason for this difference for Co-60 determinations is not well known but can be attributed to dilution effects, and/or colloids in the "as-received" OGCT sample, which will affect the detection limit for Co-60. Meanwhile, it is also worth noting that the absolute average activity for Co-60 in the "as-received" OGCT is almost equal to Co-60 average activity in the OGCT filtrate.

The %RSD for all analytes with measurable minimum detectable activity, as summarized in Table 9 and 10, are less than or equal to 20%, with analytical results for Ni-63 and Pu-241 in the OGCT filtrate being the exception. The %RSD for Ni-63 at 43 % in the OGCT filtrate, and 95 % in the "as-received" OGCT sample with a corresponding one sigma analytical uncertainties of 20 % is on the high end of the analytical uncertainty for radioanalytical methods, which normally range from 5-20% for radionuclides quantified by counting methods (gamma spectroscopy, alpha spectroscopy and liquid scintillation counting). The source of this large variation in Ni-63 result is unknown.

Table 9. Radionuclide Analysis Results for the "as-received" OGCT-AQR /PF Digestion

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L	%RSD N = 3	One sigma % uncertainty
Total alpha	<2.55E+07	<2.55E+07	<4.27E+07	<3.12E+07	<1.41E-02	na	MDA
Total beta	5.18E+08	5.28E+08	5.28E+08	5.24E+08	2.36E-01	0.9	10
Na-22	< 8.39E+02	< 8.74E+02	< 8.55E+02	< 8.56E+02	<3.86E-07	na	MDA
Al-26	< 7.88E+02	< 8.24E+02	< 8.04E+02	< 8.05E+02	<3.63E-07	na	MDA
K-40	<1.62E+04	<1.91E+04	<1.90E+04	<1.81E+04	<8.15E-06	na	MDA
Ni-59	3.05E+03	<1.75E+03	<3.69E+03	≤2.83E+03	≤1.27E-06	na	21.2/MDA
Ni-63	2.11E+05	4.35E+04	4.79E+04	1.01E+05	4.55E-05	95	20
Co-60	<1.28E+03	<6.72E+02	<7.98E+02	<9.16E+02	<4.12E-07	na	MDA
Sr-90	2.00E+07	1.89E+07	1.95E+07	1.95E+07	8.77E-03	2.9	15.4
Y-90	2.00E+07	1.89E+07	1.95E+07	1.95E+07	8.77E-03	2.9	15.4
Tc-99	8.53E+04	7.51E+04	7.62E+04	7.89E+04	3.55E-05	7.1	14.1
Ru-103	< 1.35E+03	< 1.33E+03	< 1.38E+03	< 1.35E+03	<6.09E-07	na	MDA
Ru-106	< 7.28E+03	< 7.59E+03	< 7.38E+03	< 7.42E+03	<3.34E-06	na	MDA
Sb-125	< 4.30E+03	< 4.41E+03	< 4.31E+03	< 4.34E+03	<1.95E-06	na	MDA
Sb-126	< 1.46E+03	< 1.43E+03	< 1.50E+03	< 1.46E+03	<6.59E-07	na	MDA
Sn-126	< 8.24E+03	< 8.39E+03	< 8.55E+03	< 8.39E+03	<3.78E-06	na	MDA
I-129	4.23E+02	4.49E+02	4.70E+02	4.47E+02	2.02E-07	5.3	3.9
Cs-134	<7.33E+04	<9.10E+04	<7.93E+04	<8.12E+04	<3.66E-05	na	MDA
Cs-135	2.58E+03	2.72E+03	not analyzed	2.65E+03	1.20E-06	3.5, N =2	20
Cs-137	4.57E+08	4.66E+08	4.58E+08	4.60E+08	2.07E-01	1.0	5
Ba-137m	4.33E+08	4.41E+08	4.34E+08	4.36E+08	1.96E-01	1.0	5
Ce-144	< 1.73E+04	< 1.75E+04	< 1.80E+04	< 1.76E+04	<7.91E-06	na	MDA
Eu-152	< 4.98E+03	< 4.93E+03	< 5.11E+03	< 5.01E+03	<2.26E-06	na	MDA
Eu-154	1.18E+04	1.04E+04	1.44E+04	1.22E+04	5.50E-06	16.5	5.5
Eu-155	< 9.45E+03	< 9.65E+03	< 9.85E+03	< 9.65E+03	<4.35E-06	na	MDA
Th-232	2.8E+00	3.0E+00	3.0E+00	2.90E+00	1.30E-09	3.9	20
U-233	2.0E+02	2.1E+02	2.2E+02	2.10E+02	9.50E-08	4.6	20
U-234	1.6E+02	1.7E+02	1.6E+02	1.60E+02	7.40E-08	5.0	20
U-235	2.4E+00	2.6E+00	2.5E+00	2.50E+00	1.10E-09	4.4	20
U-236	4.1E+00	4.7E+00	4.4E+00	4.40E+00	2.00E-09	5.9	20
Np-237	5.7E+01	6.0E+01	5.9E+01	5.90E+01	2.60E-08	3.0	20
U-238	2.7E+01	2.9E+01	2.9E+01	2.80E+01	1.30E-08	3.7	20
Np-239	<9.40E+03	<9.61E+03	<9.75E+03	<9.59E+03	<4.32E-06	na	MDA
Pu-238	4.87E+05	4.23E+05	4.06E+05	4.39E+05	1.98E-04	9.7	15.7
Pu-239	2.3E+04	2.4E+04	2.4E+04	2.40E+04	1.1E-05	2.0	20
Pu-239/240	<4.27E+04	5.30E+04	2.12E+04	≤3.90E+04	≤1.76E-05	na	MDA/40.7
Pu-241	7.61E+04	9.46E+04	8.25E+04	8.44E+04	3.80E-05	11.2	14.0
Am-241	4.72E+04	5.82E+04	6.42E+04	5.65E+04	2.55E-05	15.3	11.9
Am-243	< 5.61E+03	< 7.13E+03	< 7.38E+03	< 6.70E+03	<3.02E-06	na	MDA

Undiluted supernatant used for I-129 analysis, but other analyses were based on AQR or PF digestion of the "as-received" OGCT slurry.

Table 10. Radionuclide Analysis Results for the OGCT Filtrate Sample

Analyte	Analysis-1 dpm/mL	Analysis-2 dpm/mL	Analysis-3 dpm/mL	Average dpm/mL	Average Ci/L	%RSD N = 3	One sigma % uncertainty
Total alpha	<2.11E+07	<2.82E+07	<1.85E+07	<2.26E+07	<1.02E-02	na	MDA
Total beta	5.50E+08	5.35E+08	5.51E+08	5.45E+08	2.46E-01	1.6	10
Na-22	<2.24E+02	<6.46E+01	< 2.20E+02	<1.69E+02	<7.63E-08	na	MDA
Al-26	<1.79E+02	<3.04E+01	<1.61E+02	<1.23E+02	<5.55E-08	na	MDA
K-40	<3.40E+03	<7.15E+02	<3.34E+03	<2.49E+03	<1.12E-06	na	MDA
Ni-59	1.09E+03	4.74E+02	<2.94E+02	≤6.21E+02	≤2.80E-07	na	15.1/MDA
Ni-63	2.08E+04	1.37E+04	3.27E+04	2.24E+04	1.01E-05	42.9	20
Co-60	4.17E+02	4.85E+02	5.08E+02	4.70E+02	2.12E-07	10	10.1
Sr-90	1.48E+07	1.62E+07	1.75E+07	1.62E+07	7.28E-03	8.5	14.8
Y-90	1.48E+07	1.62E+07	1.75E+07	1.62E+07	7.28E-03	8.5	14.8
Tc-99	7.67E+04	7.43E+04	7.44E+04	7.51E+04	3.38E-05	1.8	10.1
Ru-103	< 4.09E+02	<1.21E+02	< 4.09E+02	<3.13E+02	<1.41E-07	na	MDA
Ru-106	< 1.95E+03	<5.60E+02	< 1.91E+03	<1.47E+03	<6.63E-07	na	MDA
Sb-125	< 1.37E+03	<4.15E+02	< 1.35E+03	<1.04E+03	<4.71E-07	na	MDA
Sb-126	< 4.15E+02	<2.47E+02	< 4.13E+02	< 3.58E+02	<1.61E-07	na	MDA
Sn-126	< 3.08E+03	<1.51E+03	< 3.06E+03	< 2.55E+03	<1.56E-06	na	MDA
I-129	5.08E+01	4.56E+01	4.51E+01	4.72E+01	2.12E-08	6.7	5
Cs-134	<1.04E+04	<1.00E+04	<9.66E+03	<1.00E+04	<4.52E-06	na	MDA
Cs-135	2.85E+02	2.80E+02	2.35E+02	2.67E+02	1.20E-07	10.3	20
Cs-137	4.46E+08	4.39E+08	3.66E+08	4.17E+08	1.88E-01	10.6	5
Ba-137m	4.22E+08	4.15E+08	3.47E+08	3.95E+08	1.78E-01	10.6	5
Ce-144	< 6.38E+03	< 2.60E+03	< 6.33E+03	< 5.10E+03	< 2.30E-06	na	MDA
Eu-152	< 1.03E+03	< 2.71E+02	< 1.06E+03	< 7.89E+02	<3.55E-07	na	MDA
Eu-154	9.24E+03	1.09E+04	9.07E+03	9.72E+03	4.38E-06	10.2	9.3
Eu-155	< 3.54E+03	< 1.57E+03	< 3.52E+03	< 2.88E+03	< 1.30E-06	na	MDA
Th-232	1.11E+00	1.11E+00	1.11E+00	1.11E+00	5.01E-10	0.3	20
U-233	1.89E+02	1.84E+02	1.88E+02	1.87E+02	8.43E-08	1.3	20
U-234	1.48E+02	1.55E+02	1.51E+02	1.51E+02	6.82E-08	2.4	20
U-235	2.38E+00	2.40E+00	2.38E+00	2.39E+00	1.08E-09	0.3	20
U-236	4.22E+00	4.14E+00	4.16E+00	4.17E+00	1.88E-09	0.9	20
Np-237	5.22E+01	5.14E+01	5.18E+01	5.18E+01	2.33E-08	0.8	20
U-238	2.65E+01	2.66E+01	2.65E+01	2.65E+01	1.20E-08	0.2	20
Np-239	< 3.16E+03	< 1.17E+03	< 3.16E+03	< 2.50E+03	< 1.13E-06	na	MDA
Pu-238	2.91E+04	3.03E+04	2.53E+04	2.82E+04	1.27E-05	9.2	7.9
Pu-239	1.51E+03	1.46E+03	1.50E+03	1.49E+03	6.72E-07	1.8	20
Pu-239/240	2.41E+03	<9.92E+02	<2.08E+03	≤1.83E+03	≤8.23E-07	na	30.3/MDA
Pu-241	9.92E+03	1.40E+04	1.00E+04	1.13E+04	5.10E-06	20.7	12.8
Am-241	3.47E+04	3.57E+04	2.91E+04	3.32E+04	1.49E-05	10.7	6.9
Am-243	< 2.62E+03	< 1.35E+03	< 2.06E+03	< 2.01E+03	< 9.05E-07	na	MDA

Undiluted OGCT filtrate used for I-129 and Cs-135 analysis.

4.6 XRD Spectra, SEM/EDX and Particle Size Distribution

As presented in Figure 2, the "as-received" OGCT sample contained only a small solids fraction. Therefore, the XRD spectra signal intensities for crystalline solids were very weak. The large amorphous base line shift is due in part to the presence of the sample holder bearing traces of the OGCT solids. The XRD mineral composition for the OGCT sample solids includes peaks for traces of hematite (Fe_2O_3), and sodium nitrite ($NaNO_2$). These mineral peaks are relatively very low in intensity and not a lot of other peaks are above the signal-to-noise ratio.

The scanning electron microscope (SEM/EDX) information presented in Figures 3, 4 and 5, shows that the main quantitative elemental constituents present in the OGCT sample solid fraction include elemental mercury, iron, manganese, nickel, thorium, silicon, magnesium, aluminum, sodium, and ruthenium.

As shown in Figure 6, the particle size analytical result for the "as-received" OGCT sample slurry shows a bimodal distribution of OGCT particles. The particle sizes range from 0.3 to 10 microns with a mean or median (center of distribution) particle size of 2.76 microns. The two modes or peaks, which are clusters of particles, for the distributions are at 0.84 and 9.30 microns. Particle sizes which constitute the first mode or peak ranges from 0.3 to 2.8 microns while the second peak particles range from 2.8 microns to 10 microns. The integrated peak summary volume percent is 45.3 % for particles with diameters greater than 4.64 microns and 54.70 % for particle diameters less than 0.84 microns.

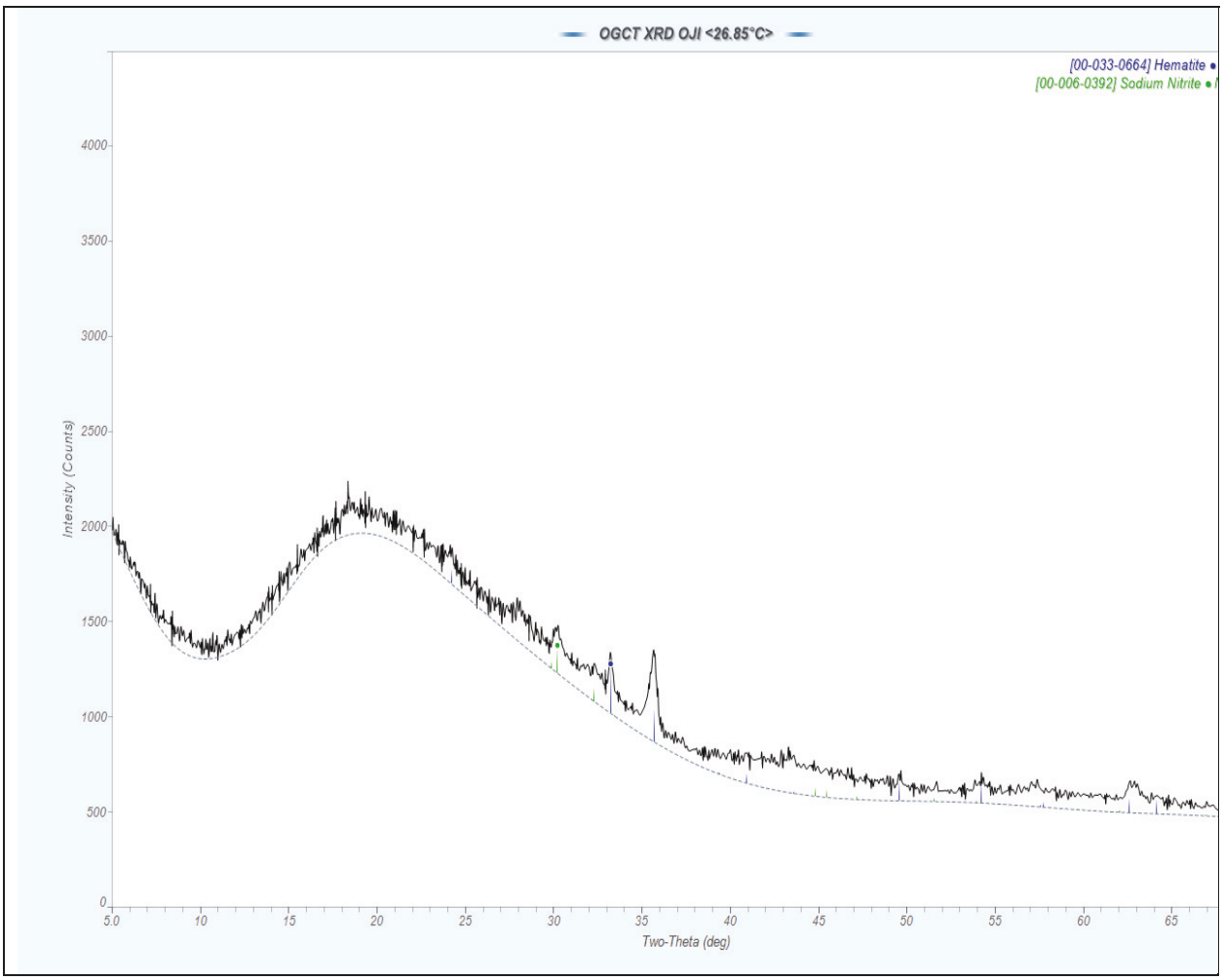
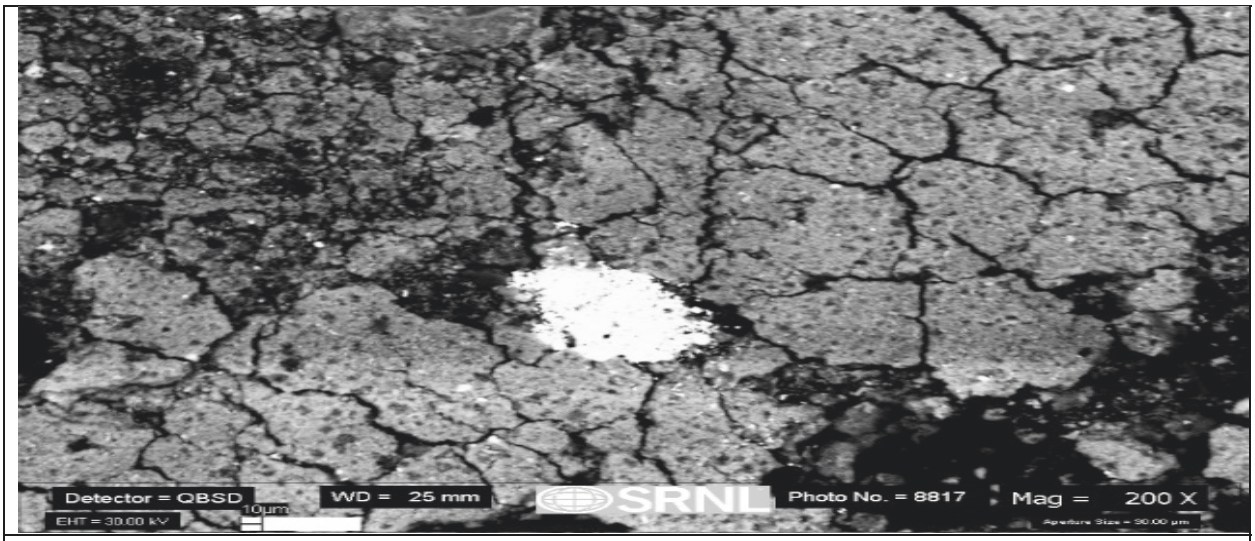
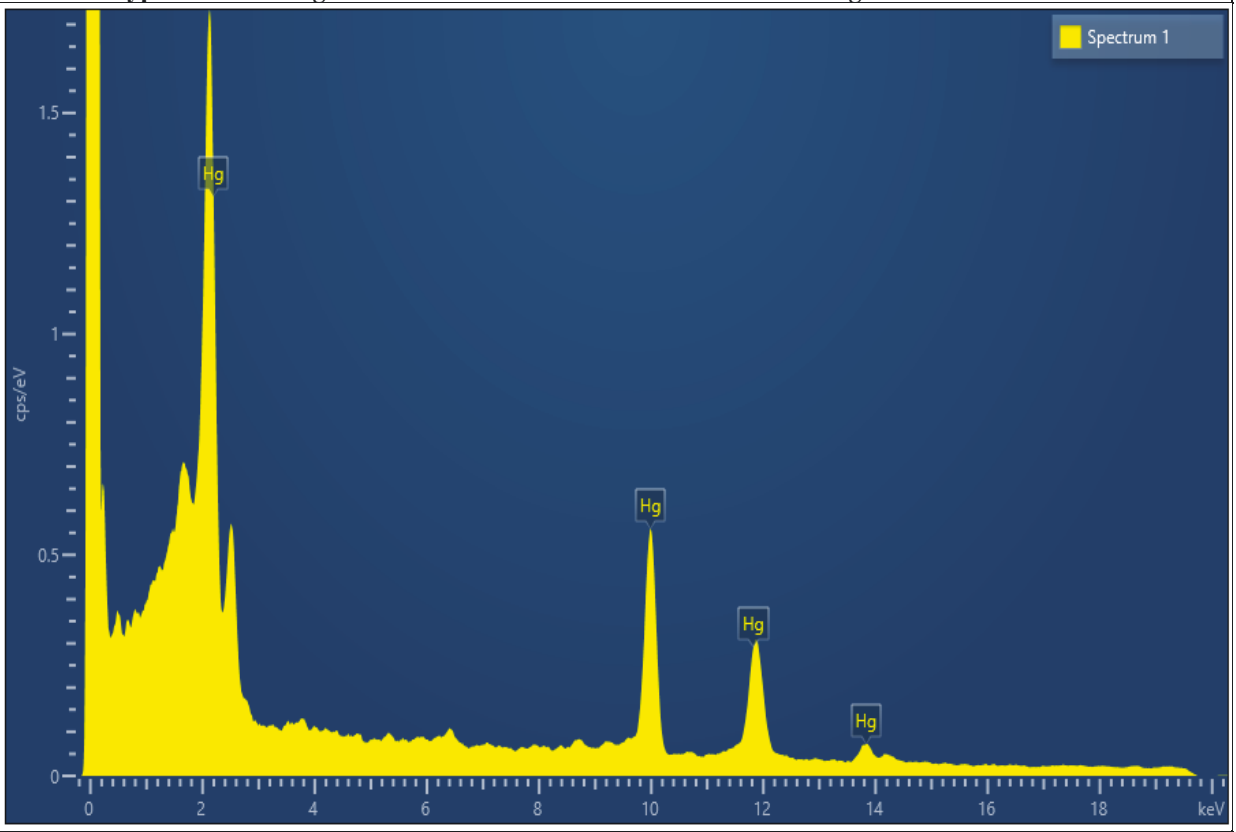


Figure 2. OGCT XRD Spectra for the Solids Fraction

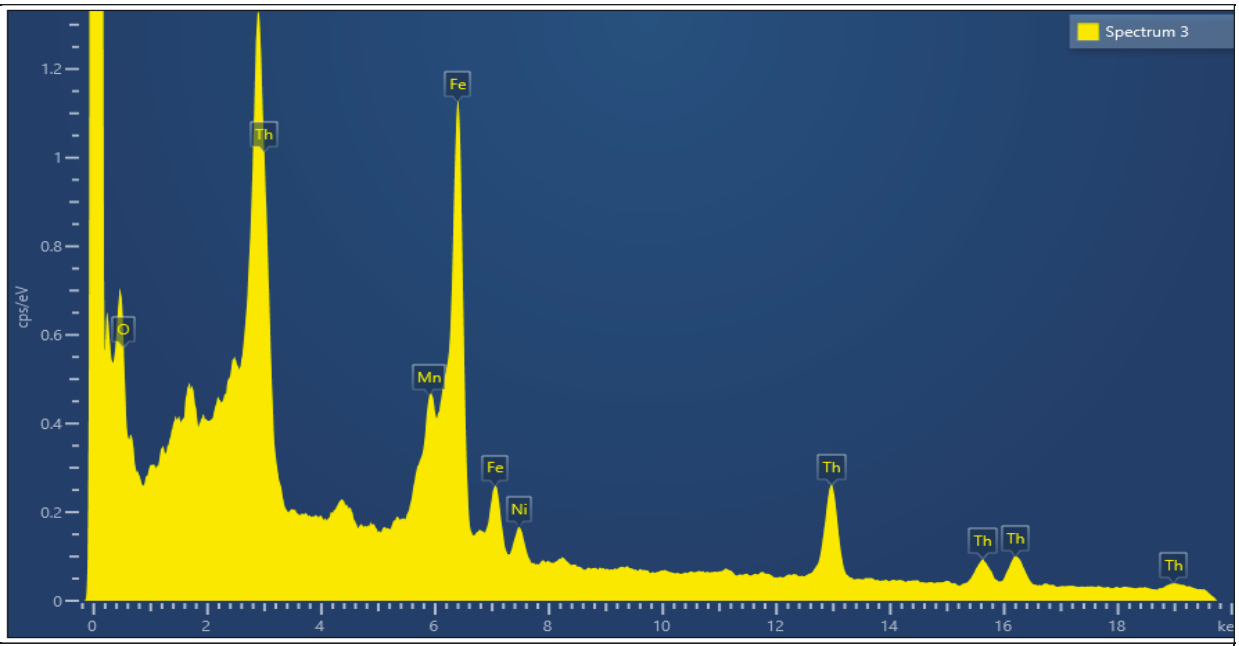


Insert A: Typical SEM Image of the OCGT Solids Fraction with 200 X Magnification

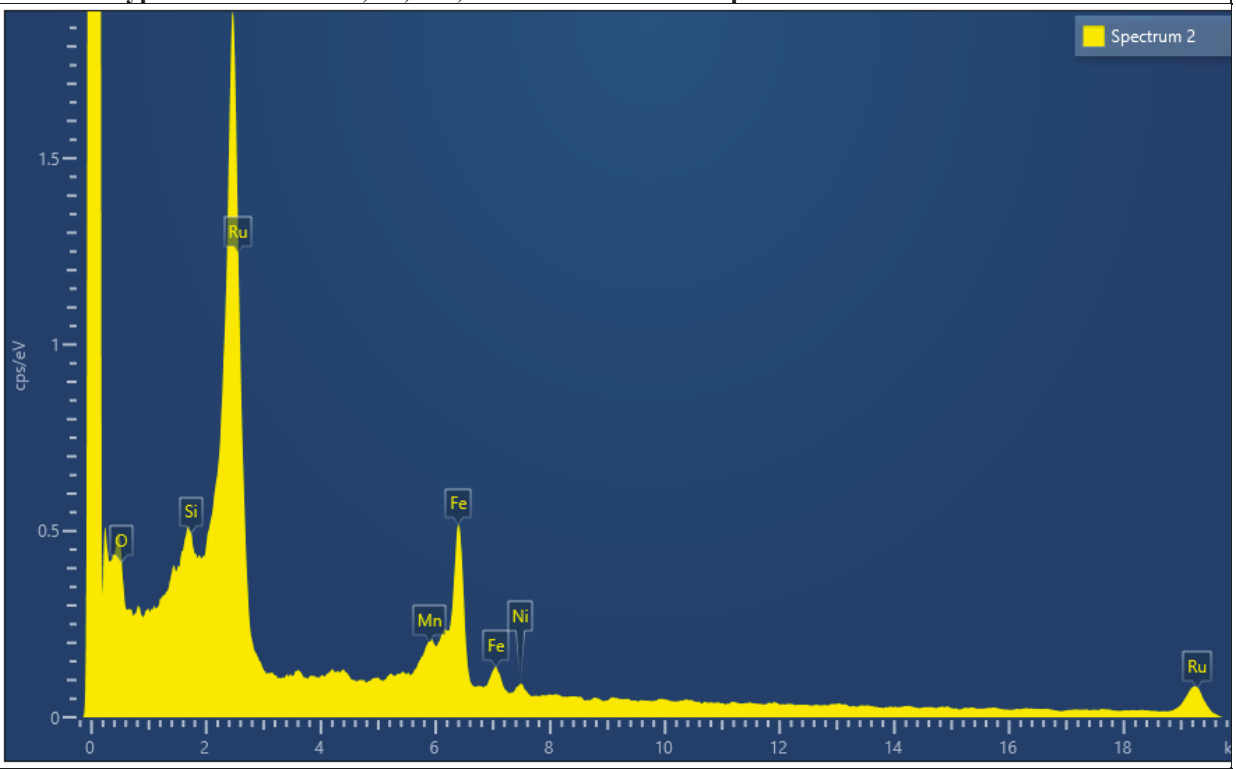


Insert B: Typical SEM/EDX Elemental Hg Composition for the OGCT Solid Fraction

Figure 3. SEM Photo Images for OGCT solids and SEM/EDX Data for Elemental Mercury

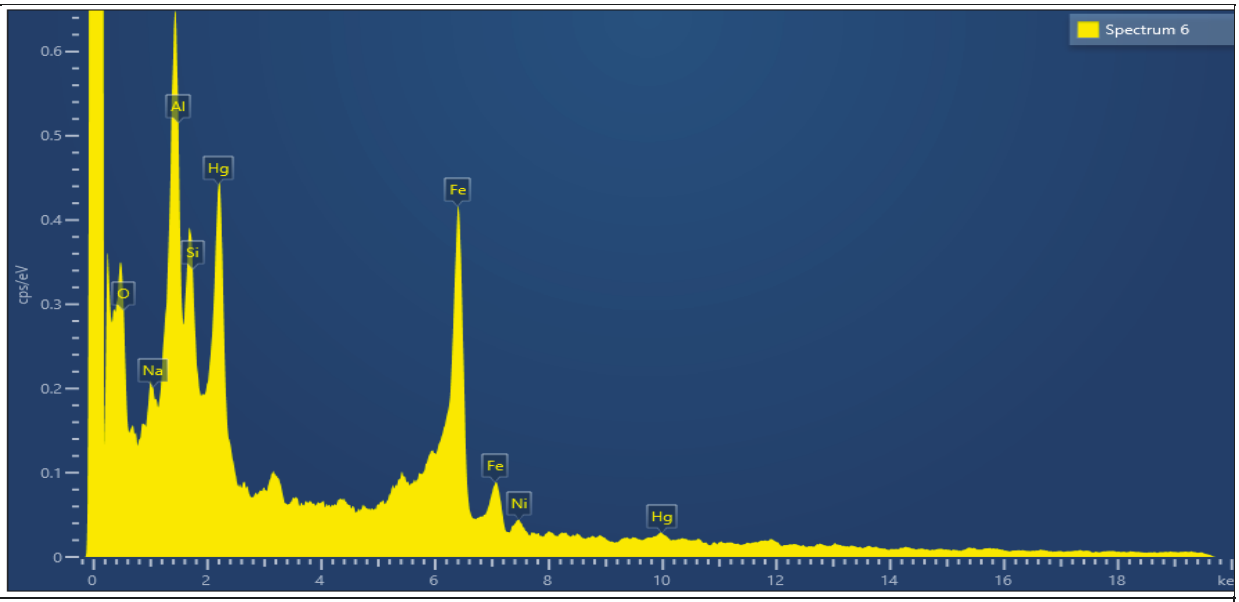


Insert A: Typical SEM/EDX Th, Fe, Mn, and Ni Elemental Composition for the OGCT Solid Fraction

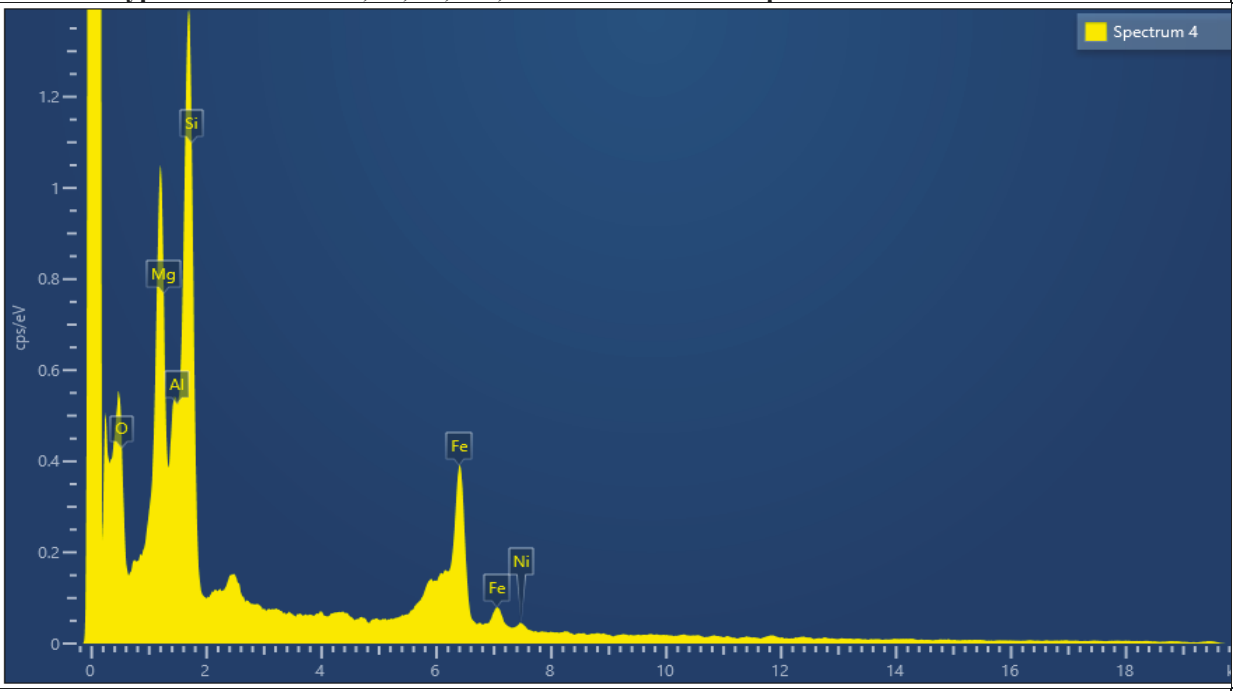


Insert B: Typical SEM/EDX Si, Ru, Mn, Fe, and Ni Elemental Composition for the OGCT Solid Fraction

Figure 4. Representative SEM/EDX Data for Elemental composition for the OGCT Solid Sample



Insert A: Typical SEM/EDX Na, Al, Fe, Mn, and Si Elemental Composition for the OGCT Solid Fraction



Insert B: Typical SEM/EDX Mg, Al, Si, Fe, and Ni Elemental Composition for the OGCT Solid Fraction

Figure 5. Representative SEM/EDX Data for Elemental composition for the OGCT Solid Sample

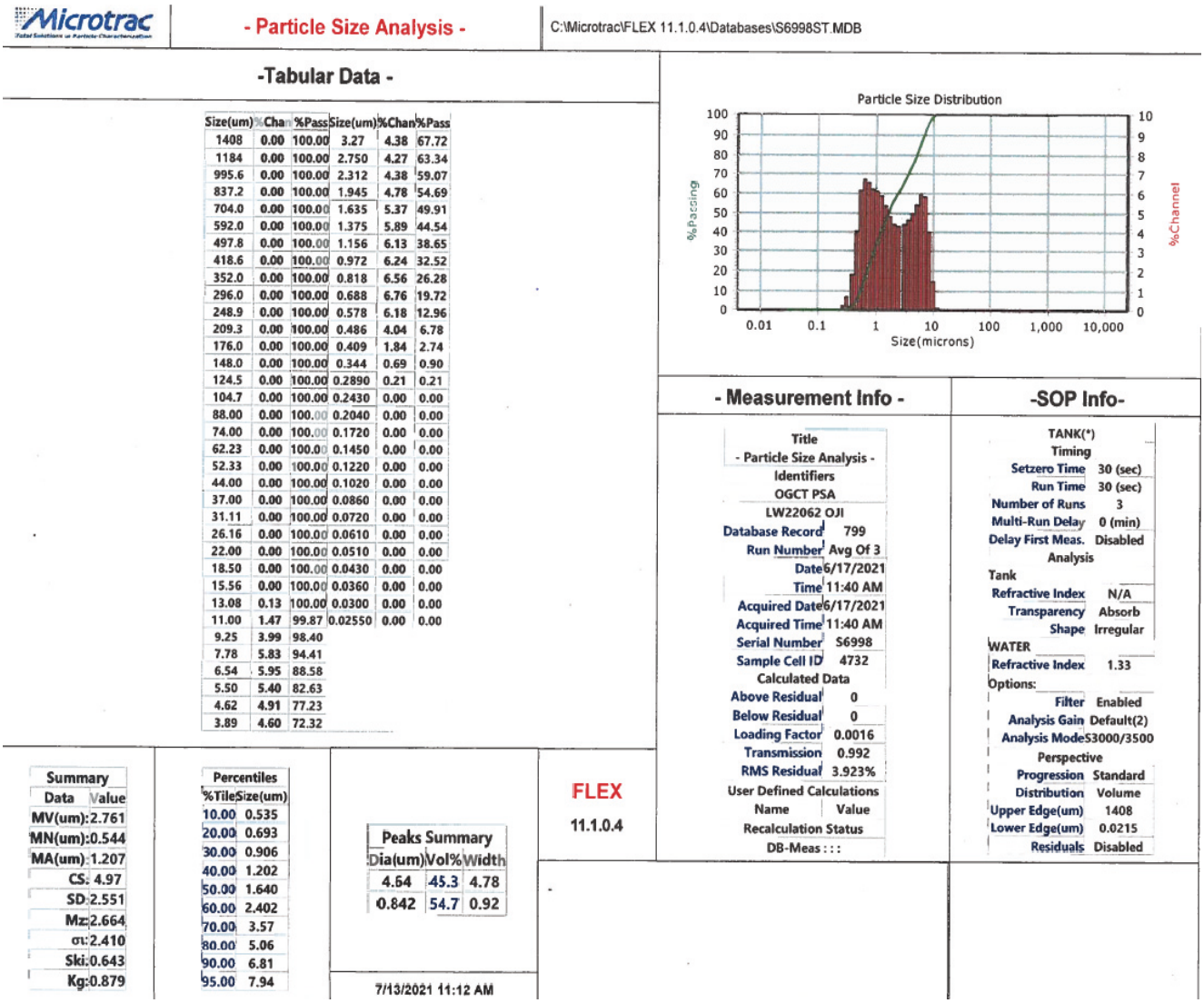


Figure 6 Particle Size Distribution: "as-received" OGCT sample.

4.7 Mass Spectral Analysis; Mass 59-252

The "as-received" OGCT sample, its filtrate and the diluting acid blanks were also analyzed by mass spectrometry (masses 59-252), as presented in Tables 11 and 12. 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" OGCT sample analysis, Table 11 and the continuations, shows that analytical results for the following masses 59, 85-107, 109-114, 116-120, 125-126, 128, 130, 133-158, 160, 182-184, 186, 196, 198, 203-208, and 232-241, are above instrument and blank detection limits, while the other analytical results were below instrument detection limits but above

reagent blank concentrations for the corresponding masses. The ICP-MS characterization of the OGCT filtrate sample, as presented in Table 12 and continuations, show the following masses above instrument and blank detection limits 59, 85-107, 109-114, 116-121, 123-126, 128, 130, 133-162, 182, 184, 186-187, 196, 198, 203-208, 232-241, and 252. Other masses not cited above were below instrument detection limits but above reagent blank concentrations for the corresponding masses.

In the ICP-MS analytical results for the "as-received" OGCT sample, 88 elements were above instrument detection limits, while 94 elements in the OGCT filtrate were above instrument detection limits.

The average percent differences between the mass (m/z) concentrations of the analytes in the "as received" OGCT sample and the OGCT filtrate are summarized in Table 13 and continuations. This percent differences table is only for those analytical results which were above instrument detection limits for one or both of the "as received" OGCT sample and the OGCT filtrate samples.

The concentration of stable Co-59, and Tc-99, averaged 2.10E-01 mg/L (2.6 %RSD) and 1.70E+00 mg/L (2.9 %RSD) in the "as received" OGCT sample, respectively. In the OGCT filtrate sample the concentrations for Co-59 and Tc-99 averaged 1.09E-01 mg/L (0.6 %RSD) and 1.50E+00 mg/L (0.6 %RSD), which are differences of 48.1 and 11.8 %, respectively. Generally, the radchem values reported in Tables 9 and 10 provide more reliable Tc-99 results.

The concentrations for the actinides (mases 232-236, 238), mass 237, and masses 239-241 in the "as received" OGCT sample, and the OGCT filtrate were all above instrument detection limits. The radionuclides in these categories, which showed the highest average concentration changes between the "as-received" OGCT samples and the OGCT filtrate were Th-232 (62 % difference), Pu-239 (93.6 % difference), Pu-240 (96.5 % difference), Pu-241 (45.6 % difference). The mass 241 is likely biased high from Am-241 contributions. Those radionuclides with less than 10% average changes in concentration between the "as received" OGCT sample and the OGCT filtrate were U-234 (9.2 % difference), U-235 (4.2 % difference), U-236 (6.1 % difference), and U-238 (6.3 % difference). Those with greater than 10% difference in average concentration changes, but less than 20% difference, include U-233 (11.4 % difference), and Np-237 (10.5 % difference).

In general, there are no measurable differences in the analytical results for the concentration of some analytes, because the analytical result concentration differences between the "as-received" OGCT sample and the OCGT filtrates are within the analytical uncertainties for this ICP-MS method. These include mass 107, 109, 198, 204, 135, 196, 234, 235, 236, and 238, where the percent differences are less than or equal to 10%.

Most of the analyte concentrations in the OGCT filtrate were less than their concentration in the "as-received" OGCT sample. These decrease in concentration range from 0% for mass 107 to a high of 98.6% for mass 91. The concentration of analytes with mass 242-252 were all below instrument detection limits in both the "as-received" OGCT and the OGCT filtrate samples.

For some analytes, as shown in Table 13 and continuations, the measured concentration of these analytes in the "as-received" OGCT sample were less than instrument detection limit, but above instrument detection limit in the OGCT filtrate. The less than values for these concentrations in the "as-received" OGCT sample are still in the same order of magnitude as in the measured values in the filtrate. Analytes in this category

of analytical results include those with mass 121, 123-124, 159, 161-162, and 187. This analytical difference is normally observed in cases where the solid fractions, which are not present in the filtrate, interfere, with the analytical method detection limits, especially if there are colloids in the sample. The analytical results only show one occasion when the instrument detection limit for one analyte concentration in the OGCT filtrate, mass 183, was less than the instrument detection limit, and this was due in part because most of that analyte was retained in the solid fraction.

Table 11. Mass Spectral Analyses of "as-received" OGCT Sample- AQR Digestion

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	2.1E-01	2.1E-01	2.2E-01	2.1E-01	2.6	Co
84	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	MDA	Sr
85	< 1.00E-04	3.5E-01	3.7E-01	3.8E-01	3.7E-01	5.1	Rb
86	< 1.00E-04	1.6E-02	1.7E-02	1.7E-02	1.7E-02	4.8	Sr
87	< 1.00E-04	6.8E-01	7.3E-01	7.4E-01	7.2E-01	3.9	Rb, Sr
88	< 1.00E-04	2.9E-01	3.0E-01	3.0E-01	3.0E-01	3.1	Sr
89	< 1.00E-04	2.1E-01	2.2E-01	2.3E-01	2.2E-01	3.2	Y
90	< 2.50E-04	2.6E-01	2.9E-01	2.8E-01	2.8E-01	6.0	Zr, Sr
91	< 1.00E-04	3.1E-01	3.3E-01	3.3E-01	3.2E-01	4.8	Zr
92	< 1.00E-04	3.6E-01	4.0E-01	4.0E-01	3.9E-01	6.1	Zr, Mo
93	6.40E-04	3.6E-01	4.2E-01	4.0E-01	4.0E-01	7.9	Nb
94	< 1.00E-04	3.7E-01	4.0E-01	4.1E-01	3.9E-01	6.0	Nb, Mo
95	< 1.00E-04	1.4E-01	1.6E-01	1.6E-01	1.5E-01	5.3	Mo
96	< 1.00E-04	3.7E-01	4.0E-01	4.0E-01	3.9E-01	4.5	Ru, Zr, Mo
97	< 1.00E-04	1.0E-01	1.1E-01	1.1E-01	1.1E-01	5.1	Mo, Tc
98	< 1.00E-04	1.6E-01	1.9E-01	1.8E-01	1.8E-01	6.5	Ru, Mo, Tc
99	< 1.00E-04	1.6E+00	1.7E+00	1.7E+00	1.7E+00	2.9	Tc, Ru
100	< 1.00E-04	4.2E-01	4.5E-01	4.4E-01	4.4E-01	4.7	Ru, Mo
101	< 1.00E-04	1.3E+01	1.3E+01	1.4E+01	1.3E+01	4.6	Ru
102	< 1.00E-04	1.1E+01	1.2E+01	1.2E+01	1.2E+01	4.5	Ru, Pd
103	< 1.00E-04	3.4E-01	3.5E-01	3.6E-01	3.5E-01	3.3	Rh
104	< 1.00E-04	6.5E+00	7.0E+00	7.1E+00	6.9E+00	4.9	Ru, Pd
105	< 1.00E-04	8.3E-03	1.0E-02	1.1E-02	9.7E-03	13.3	Pd
106	< 1.00E-04	1.5E-02	2.1E-02	1.8E-02	1.8E-02	16.1	Pd, Cd
107	< 1.00E-04	1.6E-01	1.9E-01	1.8E-01	1.8E-01	7.5	Ag
108	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pd, Cd
109	< 1.00E-04	1.7E-01	1.9E-01	1.9E-01	1.8E-01	7.9	Ag
110	< 1.00E-04	4.3E-02	5.0E-02	4.7E-02	4.7E-02	6.8	Pd, Cd
111	< 1.00E-04	8.3E-02	9.2E-02	8.8E-02	8.8E-02	4.7	Cd
112	< 1.00E-04	1.4E-01	1.6E-01	1.5E-01	1.5E-01	5.1	Sn, Cd
113	< 5.00E-04	7.3E-02	7.8E-02	7.2E-02	7.4E-02	5.1	In, Cd
114	< 1.00E-04	1.7E-01	1.8E-01	1.8E-01	1.8E-01	4.5	Sn, Cd
116	< 1.50E-04	4.3E-01	4.6E-01	4.6E-01	4.5E-01	4.1	Sn, Cd
117	< 1.00E-04	5.5E-03	7.4E-03	<5.1E-03	≤6.0E-03	na	Sn
118	< 3.00E-04	1.3E-02	1.7E-02	1.1E-02	1.4E-02	22.3	Sn
119	< 1.00E-04	7.1E-01	7.7E-01	7.6E-01	7.5E-01	4.5	Sn
120	< 4.00E-04	1.2E-02	2.0E-02	9.9E-03	1.4E-02	38.2	Sn
121	< 9.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Sb
122	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Te, Sn
123	< 7.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Sb, Te
124	< 1.50E-04	<2.0E-02	<2.0E-02	<2.0E-02	<2.0E-02	na	Te, Sn
125	< 2.00E-04	5.2E-02	7.3E-02	6.7E-02	6.4E-02	16.3	Sb, Te
126	< 1.00E-04	1.3E-02	1.4E-02	1.6E-02	1.4E-02	11.5	Te
128	< 1.00E-04	5.5E-01	5.7E-01	5.9E-01	5.7E-01	3.9	Te
130	< 1.00E-04	2.6E+00	2.9E+00	2.9E+00	2.8E+00	6.4	Te
133	5.84E-04	1.9E+01	2.0E+01	2.1E+01	2.0E+01	3.1	Cs
134	< 1.00E-04	1.8E-02	2.2E-02	1.8E-02	2.0E-02	12.1	Ba, Cs

Table 11 Continued. Mass Spectral Analyses of "as-received" OGCT Sample- AQR Digestion

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	8.1E-01	8.3E-01	8.5E-01	8.3E-01	2.5	Ba, Cs
136	< 1.00E-04	2.1E-02	3.0E-02	2.0E-02	2.4E-02	23.2	Ce, Ba
137	< 1.00E-04	2.1E+00	2.2E+00	2.2E+00	2.2E+00	3.3	Cs, Ba, La
138	2.37E-04	6.0E-01	7.1E-01	6.2E-01	6.5E-01	9.2	Ba, La, Ce
139	< 1.00E-04	5.2E-01	5.6E-01	5.7E-01	5.5E-01	4.2	La
140	< 1.00E-04	2.4E+00	2.5E+00	2.5E+00	2.5E+00	4.2	Ce
141	< 1.00E-04	4.5E-01	4.8E-01	4.9E-01	4.7E-01	3.6	Pr
142	< 1.00E-04	8.0E-01	8.6E-01	8.5E-01	8.4E-01	4.0	Nd, Ce
143	< 1.00E-04	4.4E-01	4.7E-01	4.8E-01	4.6E-01	4.0	Nd., Pm
144	< 1.00E-04	4.7E-01	5.0E-01	5.0E-01	4.9E-01	3.0	Nd, Sm, Pm
145	< 1.00E-04	3.1E-01	3.2E-01	3.3E-01	3.2E-01	3.5	Nd, Pm
146	< 1.00E-04	2.6E-01	2.7E-01	2.7E-01	2.7E-01	3.6	Nd, Sm
147	< 1.00E-04	1.6E-01	1.7E-01	1.8E-01	1.7E-01	4.4	Sm, Ti
148	< 1.00E-04	1.5E-01	1.7E-01	1.7E-01	1.6E-01	4.6	Nd, Gd, Sm
149	< 1.00E-04	9.9E-03	1.0E-02	1.1E-02	1.0E-02	3.6	Sm
150	< 1.00E-04	1.4E-01	1.5E-01	1.6E-01	1.5E-01	4.0	Nd, Gd, Sm, Eu
151	< 1.00E-04	1.3E-02	1.3E-02	1.4E-02	1.3E-02	2.3	Eu
152	< 1.00E-04	4.9E-02	5.1E-02	5.3E-02	5.1E-02	3.7	Gd, Sm, Eu
153	< 1.00E-04	2.0E-02	2.1E-02	2.1E-02	2.1E-02	3.8	Eu
154	< 1.00E-04	4.2E-02	4.4E-02	4.4E-02	4.3E-02	3.2	Gd, Sm, Eu, Dy
155	< 1.00E-04	1.8E-01	1.9E-01	1.9E-01	1.9E-01	3.5	Gd
156	< 1.00E-04	2.6E-01	2.8E-01	2.8E-01	2.7E-01	4.1	Gd, Dy
157	< 1.00E-04	1.9E-01	2.0E-01	2.0E-01	2.0E-01	3.9	Gd, Tb
158	< 1.00E-04	3.0E-01	3.3E-01	3.3E-01	3.2E-01	4.7	Gd, Dy, Tb
159	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Tb
160	< 1.00E-04	2.7E-01	2.9E-01	2.9E-01	2.8E-01	4.6	Gd, Dy
161	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Dy
162	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Dy, Er
163	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Dy, Ho
164	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Dy, Er
165	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Ho
166	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Er, Ho
167	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Er
168	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Er, Yb
169	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Tm
170	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Er, Yb
171	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Yb
172	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Yb
173	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Yb
174	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Yb, Hf
175	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Lu
176	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Lu, Hf, Yb
177	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Hf
178	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Hf
179	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Hf
180	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Hf, W, Ta
181	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Ta
182	< 1.00E-04	5.1E-02	5.1E-02	5.1E-02	5.1E-02	0.0	Hf, W

Table 11 Continued. Mass Spectral Analyses of "as-received" OGCT Sample AQR Digestion

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	1.2E-02	1.2E-02	1.2E-02	1.2E-02	0.3	W
184	< 1.00E-04	2.5E-02	2.5E-02	2.5E-02	2.5E-02	0.5	W
185	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03		Re
186	< 1.00E-04	2.2E-02	2.2E-02	2.1E-02	2.2E-02	2.3	Os, W
187	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Re, Os
191	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Ir
193	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Ir, Pt
194	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pt
195	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pt
196	< 1.00E-04	9.5E-02	1.0E-01	1.0E-01	9.8E-02	3.4	Hg, Pt
198	< 4.00E-04	6.2E+00	6.7E+00	6.7E+00	6.6E+00	4.4	Hg, Pt
203	< 1.00E-04	1.4E-02	1.4E-02	1.3E-02	1.3E-02	4.3	Tl
204	< 3.00E-04	3.5E+00	3.8E+00	3.8E+00	3.7E+00	4.8	Pb, Hg
205	< 1.00E-04	3.1E-02	2.9E-02	3.0E-02	3.0E-02	2.7	Tl
206	1.33E-03	2.2E-01	2.6E-01	2.4E-01	2.4E-01	7.0	Pb
207	1.14E-03	2.0E-01	2.2E-01	2.1E-01	2.1E-01	6.4	Pb
208	2.77E-03	4.6E-01	5.4E-01	5.0E-01	5.0E-01	8.0	Pb
229	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Th
230	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Th
232	< 1.00E-04	1.1E+01	1.2E+01	1.2E+01	1.2E+01	3.9	Th, U
233	< 1.00E-04	9.4E-03	1.0E-02	1.0E-02	9.9E-03	4.6	U
234	< 1.00E-04	1.1E-02	1.3E-02	1.2E-02	1.2E-02	5.0	U
235	< 1.00E-04	5.0E-01	5.4E-01	5.3E-01	5.2E-01	4.4	U
236	< 1.00E-04	2.9E-02	3.2E-02	3.0E-02	3.1E-02	5.9	U
237	< 1.00E-04	3.6E-02	3.8E-02	3.8E-02	3.7E-02	3.0	Np
238	< 1.00E-04	3.6E+01	3.9E+01	3.9E+01	3.8E+01	3.7	U, Pu
239	< 1.00E-04	1.7E-01	1.7E-01	1.7E-01	1.7E-01	2.0	Pu
240	< 1.00E-04	2.8E-02	2.5E-02	2.2E-02	2.5E-02	10.9	Pu
241	< 1.00E-04	7.4E-03	8.2E-03	7.7E-03	7.7E-03	4.9	Pu, Am
242	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pu, Am
243	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pu, Cm
244	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Pu, Cm
245	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Cm
246	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Cm
247	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Cm, Bk
248	< 1.00E-04	<3.1E-01	<3.1E-01	<3.1E-01	<3.1E-01	na	Cm
249	< 1.00E-04	<3.1E-02	<3.1E-02	<3.1E-02	<3.1E-02	na	Cf
250	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Cf
251	< 1.00E-04	<1.5E-02	<1.5E-02	<1.5E-02	<1.5E-02	na	Cf
252	< 1.00E-04	<5.1E-03	<5.1E-03	<5.1E-03	<5.1E-03	na	Cf, Cm

Table 12. Mass Spectral Analyses of OGCT Sample Filtrate

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis 2, mg/L	Analysis 3, mg/L	Average, mg/L	% RSD, N = 3	Likely element (s)
59	< 1.00E-04	1.08E-01	1.09E-01	1.10E-01	1.09E-01	0.6	Co
84	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Sr
85	< 1.00E-04	3.24E-01	3.15E-01	3.20E-01	3.19E-01	1.4	Rb
86	< 1.00E-04	1.25E-02	1.26E-02	1.24E-02	1.25E-02	1.0	Sr
87	< 1.00E-04	6.36E-01	6.29E-01	6.28E-01	6.31E-01	0.7	Rb, Sr
88	< 1.00E-04	2.34E-01	2.30E-01	2.32E-01	2.32E-01	1.0	Sr
89	< 1.00E-04	1.70E-01	1.70E-01	1.67E-01	1.69E-01	1.1	Y
90	< 2.50E-04	2.90E-02	2.87E-02	2.79E-02	2.85E-02	2.0	Zr, Sr
91	< 1.00E-04	4.21E-03	4.29E-03	4.50E-03	4.33E-03	3.4	Zr
92	< 1.00E-04	1.39E-02	1.37E-02	1.36E-02	1.37E-02	1.3	Zr, Mo
93	6.40E-04	9.00E-03	6.55E-03	6.58E-03	7.38E-03	19.1	Nb
94	< 1.00E-04	1.08E-02	1.08E-02	1.10E-02	1.08E-02	1.1	Nb, Mo
95	< 1.00E-04	1.93E-02	1.87E-02	1.84E-02	1.88E-02	2.4	Mo
96	< 1.00E-04	1.50E-02	1.51E-02	1.49E-02	1.50E-02	0.9	Ru, Zr, Mo
97	< 1.00E-04	1.32E-02	1.36E-02	1.34E-02	1.34E-02	1.4	Mo, Tc
98	< 1.00E-04	2.29E-02	2.31E-02	2.26E-02	2.29E-02	1.0	Ru, Mo, Tc
99	< 1.00E-04	1.51E+00	1.50E+00	1.49E+00	1.50E+00	0.6	Tc, Ru
100	< 1.00E-04	2.24E-01	2.24E-01	2.24E-01	2.24E-01	0.2	Ru, Mo
101	< 1.00E-04	8.13E+00	8.13E+00	8.11E+00	8.12E+00	0.1	Ru
102	< 1.00E-04	7.20E+00	7.13E+00	7.17E+00	7.16E+00	0.5	Ru, Pd
103	< 1.00E-04	1.49E-01	1.46E-01	1.46E-01	1.47E-01	1.0	Rh
104	< 1.00E-04	4.03E+00	4.01E+00	3.98E+00	4.01E+00	0.6	Ru, Pd
105	< 1.00E-04	1.54E-03	8.55E-04	8.55E-04	1.08E-03	36.3	Pd
106	< 1.00E-04	5.78E-03	3.29E-03	3.06E-03	4.05E-03	37.3	Pd, Cd
107	< 1.00E-04	1.82E-01	1.78E-01	1.79E-01	1.80E-01	1.3	Ag
108	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pd, Cd
109	< 1.00E-04	1.87E-01	1.85E-01	1.88E-01	1.87E-01	0.9	Ag
110	< 1.00E-04	3.63E-02	3.49E-02	3.52E-02	3.55E-02	2.1	Pd, Cd
111	< 1.00E-04	6.35E-02	6.28E-02	6.30E-02	6.31E-02	0.6	Cd
112	< 1.00E-04	1.12E-01	1.11E-01	1.12E-01	1.12E-01	0.4	Sn, Cd
113	< 5.00E-04	5.41E-01	5.22E-01	5.31E-01	5.31E-01	1.8	In, Cd
114	< 1.00E-04	1.30E-01	1.29E-01	1.30E-01	1.30E-01	0.5	Sn, Cd
116	< 1.50E-04	1.78E-01	1.78E-01	1.84E-01	1.80E-01	1.9	Sn, Cd
117	< 1.00E-04	2.97E-03	3.23E-03	3.05E-03	3.08E-03	4.3	Sn
118	< 3.00E-04	8.59E-03	8.68E-03	8.90E-03	8.73E-03	1.9	Sn
119	< 1.00E-04	6.65E-01	6.70E-01	6.72E-01	6.69E-01	0.6	Sn
120	< 4.00E-04	6.49E-03	6.56E-03	6.83E-03	6.63E-03	2.7	Sn
121	< 9.00E-04	4.34E-03	4.01E-03	3.74E-03	4.03E-03	7.5	Sb
122	< 1.00E-04	<1.71E-03	<1.71E-03	<1.71E-03	<1.71E-03	na	Te, Sn
123	< 7.00E-04	2.84E-03	3.20E-03	3.00E-03	3.01E-03	6.0	Sb, Te
124	< 1.50E-04	3.44E-03	3.58E-03	3.52E-03	3.51E-03	2.0	Te, Sn
125	< 2.00E-04	3.13E-02	3.13E-02	3.25E-02	3.17E-02	2.1	Sb, Te
126	< 1.00E-04	2.53E-03	2.13E-03	3.28E-03	2.65E-03	22.1	Te
128	< 1.00E-04	2.91E-01	2.96E-01	2.97E-01	2.95E-01	1.1	Te
130	< 1.00E-04	1.44E+00	1.43E+00	1.45E+00	1.44E+00	0.6	Te
133	5.84E-04	1.81E+01	1.76E+01	1.73E+01	1.77E+01	2.3	Cs
134	< 1.00E-04	1.20E-02	1.20E-02	1.20E-02	1.20E-02	0.3	Ba, Cs

Table 12 Continued. Mass Spectral Analyses of OGCT Sample Filtrate

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	7.58E-01	7.45E-01	7.53E-01	7.52E-01	0.9	Ba, Cs
136	< 1.00E-04	9.93E-03	9.92E-03	1.06E-02	1.01E-02	3.6	Ce, Ba
137	< 1.00E-04	1.92E+00	1.90E+00	1.90E+00	1.91E+00	0.4	Cs, Ba, La
138	2.37E-04	3.95E-01	3.96E-01	3.97E-01	3.96E-01	0.2	Ba, La, Ce
139	< 1.00E-04	3.98E-01	3.96E-01	4.01E-01	3.98E-01	0.7	La
140	< 1.00E-04	7.15E-01	7.16E-01	7.17E-01	7.16E-01	0.2	Ce
141	< 1.00E-04	3.30E-01	3.31E-01	3.32E-01	3.31E-01	0.4	Pr
142	< 1.00E-04	2.60E-01	2.61E-01	2.62E-01	2.61E-01	0.5	Nd, Ce
143	< 1.00E-04	3.26E-01	3.24E-01	3.26E-01	3.25E-01	0.3	Nd., Pm
144	< 1.00E-04	3.47E-01	3.42E-01	3.45E-01	3.45E-01	0.7	Nd, Sm, Pm
145	< 1.00E-04	2.27E-01	2.23E-01	2.27E-01	2.26E-01	1.0	Nd, Pm
146	< 1.00E-04	1.87E-01	1.85E-01	1.86E-01	1.86E-01	0.6	Nd, Sm
147	< 1.00E-04	1.25E-01	1.26E-01	1.26E-01	1.26E-01	0.4	Sm, Ti
148	< 1.00E-04	1.15E-01	1.15E-01	1.16E-01	1.15E-01	0.4	Nd, Gd, Sm
149	< 1.00E-04	7.75E-03	7.45E-03	7.52E-03	7.57E-03	2.1	Sm
150	< 1.00E-04	1.08E-01	1.09E-01	1.09E-01	1.09E-01	0.6	Nd, Gd, Sm, Eu
151	< 1.00E-04	9.71E-03	9.57E-03	9.68E-03	9.65E-03	0.7	Eu
152	< 1.00E-04	3.71E-02	3.71E-02	3.74E-02	3.72E-02	0.4	Gd, Sm, Eu
153	< 1.00E-04	1.53E-02	1.50E-02	1.51E-02	1.51E-02	1.1	Eu
154	< 1.00E-04	3.35E-02	3.39E-02	3.41E-02	3.38E-02	1.0	Gd, Sm, Eu, Dy
155	< 1.00E-04	1.52E-01	1.53E-01	1.53E-01	1.53E-01	0.21	Gd
156	< 1.00E-04	2.18E-01	2.15E-01	2.19E-01	2.17E-01	0.9	Gd, Dy
157	< 1.00E-04	1.62E-01	1.60E-01	1.61E-01	1.61E-01	0.6	Gd, Tb
158	< 1.00E-04	2.61E-01	2.58E-01	2.61E-01	2.60E-01	0.6	Gd, Dy, Tb
159	< 1.00E-04	1.58E-03	1.56E-03	1.58E-03	1.57E-03	0.6	Tb
160	< 1.00E-04	2.31E-01	2.30E-01	2.32E-01	2.31E-01	0.4	Gd, Dy
161	< 1.00E-04	1.06E-03	1.08E-03	9.37E-04	1.02E-03	7.5	Dy
162	< 1.00E-04	9.48E-04	9.21E-04	8.55E-04	9.08E-04	5.3	Dy, Er
163	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Dy, Ho
164	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Dy, Er
165	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Ho
166	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Er, Ho
167	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Er
168	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Er, Yb
169	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Tm
170	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Er, Yb
171	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Yb
172	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Yb
173	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Yb
174	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Yb, Hf
175	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Lu
176	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Lu, Hf, Yb
177	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Hf
178	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Hf
179	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Hf
180	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Hf, W, Ta
181	< 1.00E-04	<4.28E-03	<4.28E-03	<4.28E-03	<4.28E-03	na	Ta
182	< 1.00E-04	1.55E-03	1.34E-03	1.29E-03	1.39E-03	9.8	Hf, W

Table 12 Continued. Mass Spectral Analyses of OGCT Sample Filtrate

ICP-MS, m/z	Blank, mg/L	Analysis-1, mg/L	Analysis-2, mg/L	Analysis-3, mg/L	Average, mg/L	%RSD, N = 3	Likely element (s)
183	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	W
184	< 1.00E-04	1.78E-03	1.52E-03	1.41E-03	1.57E-03	12.0	W
185	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Re
186	< 1.00E-04	1.67E-03	1.36E-03	1.25E-03	1.43E-03	15.2	Os, W
187	< 1.00E-04	1.34E-03	1.36E-03	1.35E-03	1.35E-03	0.8	Re, Os
191	< 1.00E-04	<4.28E-03	<4.28E-03	<4.28E-03	<4.28E-03	na	Ir
193	< 1.00E-04	<8.55E-03	<8.55E-03	<8.55E-03	<8.55E-03	na	Ir, Pt
194	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pt
195	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pt
196	< 1.00E-04	1.05E-01	1.03E-01	1.03E-01	1.04E-01	1.3	Hg, Pt
198	< 4.00E-04	6.30E+00	6.32E+00	6.33E+00	6.32E+00	0.3	Hg, Pt
203	< 1.00E-04	1.11E-02	1.11E-02	1.09E-02	1.11E-02	1.0	Tl
204	< 3.00E-04	3.56E+00	3.56E+00	3.59E+00	3.57E+00	0.5	Pb, Hg
205	< 1.00E-04	2.66E-02	2.59E-02	2.60E-02	2.62E-02	1.5	Tl
206	1.33E-03	1.62E-01	1.60E-01	1.60E-01	1.61E-01	0.7	Pb
207	1.14E-03	1.38E-01	1.37E-01	1.37E-01	1.37E-01	0.4	Pb
208	2.77E-03	3.36E-01	3.38E-01	3.33E-01	3.36E-01	0.7	Pb
229	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Th
230	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Th
232	< 1.00E-04	4.56E+00	4.58E+00	4.55E+00	4.56E+00	0.3	Th, U
233	< 1.00E-04	8.86E-03	8.64E-03	8.81E-03	8.77E-03	1.3	U
234	< 1.00E-04	1.06E-02	1.12E-02	1.09E-02	1.09E-02	2.4	U
235	< 1.00E-04	4.96E-01	4.99E-01	4.97E-01	4.98E-01	0.3	U
236	< 1.00E-04	2.94E-02	2.88E-02	2.90E-02	2.91E-02	0.9	U
237	< 1.00E-04	3.33E-02	3.28E-02	3.31E-02	3.31E-02	0.8	Np
238	< 1.00E-04	3.56E+01	3.56E+01	3.55E+01	3.56E+01	0.2	U, Pu
239	< 1.00E-04	1.09E-02	1.06E-02	1.09E-02	1.08E-02	1.8	Pu
240	< 1.00E-04	8.96E-04	9.00E-04	8.56E-04	8.84E-04	2.8	Pu
241	< 1.00E-04	4.11E-03	4.27E-03	4.19E-03	4.19E-03	1.9	Pu, Am
242	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pu, Am
243	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pu, Cm
244	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Pu, Cm
245	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Cm
246	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Cm
247	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Cm, Bk
248	< 1.00E-04	<1.03E-01	<1.03E-01	<1.03E-01	<1.03E-01	na	Cm
249	< 1.00E-04	4.53E-06	7.49E-03	5.05E-03	<7.66E-03	86	Cf
250	< 1.00E-04	<8.55E-04	<8.55E-04	<8.55E-04	<8.55E-04	na	Cf
251	< 1.00E-04	<1.20E-02	<1.20E-02	<1.20E-02	<1.20E-02	na	Cf
252	< 1.00E-04	1.09E-03	1.16E-03	1.25E-03	1.16E-03	6.9	Cf, Cm

Table 13. Percent Difference in Concentrations Between the "as-received" OGCT Sample and OGCT Filtrate ICP-MS Analytes.

m/z	Average concentration, "as-received" OGCT, mg/L	Average concentration, OGCT Filtrate, mg/L	% difference
59	2.10E-01	1.09E-01	48.1
85	3.70E-01	3.19E-01	13.8
86	1.70E-02	1.25E-02	26.5
87	7.20E-01	6.31E-01	12.4
88	3.00E-01	2.32E-01	22.7
89	2.20E-01	1.69E-01	23.2
90	2.80E-01	2.85E-02	89.8
91	3.20E-01	4.33E-03	98.6
92	3.90E-01	1.37E-02	96.5
93	4.00E-01	7.38E-03	98.2
94	3.90E-01	1.08E-02	97.2
95	1.50E-01	1.88E-02	87.5
96	3.90E-01	1.50E-02	96.2
97	1.10E-01	1.34E-02	87.8
98	1.80E-01	2.29E-02	87.3
99	1.70E+00	1.50E+00	11.8
100	4.40E-01	2.24E-01	49.1
101	1.30E+01	8.12E+00	37.5
102	1.20E+01	7.16E+00	40.3
103	3.50E-01	1.47E-01	58.0
104	6.90E+00	4.01E+00	41.9
105	9.70E-03	1.08E-03	88.9
106	1.80E-02	4.05E-03	77.5
107	1.80E-01	1.80E-01	0.0
109	1.80E-01	1.87E-01	3.7
110	4.70E-02	3.55E-02	24.5
111	8.80E-02	6.31E-02	28.3
112	1.50E-01	1.12E-01	25.3
113	7.40E-02	5.31E-01	86.1
114	1.80E-01	1.30E-01	27.8
116	4.50E-01	1.80E-01	60.0
117	<6.00E-03	3.08E-03	48.7
118	1.40E-02	8.73E-03	37.6
119	7.50E-01	6.69E-01	10.8
120	1.40E-02	6.63E-03	52.6
121	<5.1E-03	4.03E-03	< value in the "as-received" sample
123	<5.1E-03	3.01E-03	< value in the "as-received" sample
124	<2.0E-02	3.51E-03	< value in the "as-received" sample
125	6.40E-02	3.17E-02	50.5
126	1.40E-02	2.65E-03	81.1
128	5.70E-01	2.95E-01	48.2
130	2.80E+00	1.44E+00	48.6
133	2.00E+01	1.77E+01	11.5
134	2.00E-02	1.20E-02	40.0
135	8.30E-01	7.52E-01	9.4
136	2.40E-02	1.01E-02	57.9
137	2.20E+00	1.91E+00	13.2
138	6.50E-01	3.96E-01	39.1
139	5.50E-01	3.98E-01	27.6

Table 13 Continued. Percent Difference in Concentrations Between the "as-received" OGCT Sample and OGCT Filtrate ICP-MS Analytes

m/z	Average concentration, "as-received" OGCT, mg/L	Average concentration, OGCT Filtrate, mg/L	% difference
140	2.50E+00	7.16E-01	71.4
141	4.70E-01	3.31E-01	29.6
142	8.40E-01	2.61E-01	68.9
143	4.60E-01	3.25E-01	29.3
144	4.90E-01	3.45E-01	29.6
145	3.20E-01	2.26E-01	29.4
146	2.70E-01	1.86E-01	31.1
147	1.70E-01	1.26E-01	25.9
148	1.60E-01	1.15E-01	28.1
149	1.00E-02	7.57E-03	24.3
150	1.50E-01	1.09E-01	27.3
151	1.30E-02	9.65E-03	25.8
152	5.10E-02	3.72E-02	27.1
153	2.10E-02	1.51E-02	28.1
154	4.30E-02	3.38E-02	21.4
155	1.90E-01	1.53E-01	19.5
156	2.70E-01	2.17E-01	19.6
157	2.00E-01	1.61E-01	19.5
158	3.20E-01	2.60E-01	18.8
159	<5.1E-03	1.57E-03	< value in the "as-received" sample
160	2.80E-01	2.31E-01	17.5
161	<5.1E-03	1.02E-03	< value in the "as-received" sample
162	<5.1E-03	9.08E-04	< value in the "as-received" sample
182	5.10E-02	1.39E-03	97.3
183	1.20E-02	<8.55E-04	Below detect in filtrate
184	2.50E-02	1.57E-03	93.7
186	2.20E-02	1.43E-03	93.5
187	<5.1E-03	1.35E-03	< value in the "as-received" sample
196	9.80E-02	1.04E-01	5.8
198	6.60E+00	6.32E+00	4.2
203	1.30E-02	1.11E-02	14.6
204	3.70E+00	3.57E+00	3.5
205	3.00E-02	2.62E-02	12.7
206	2.40E-01	1.61E-01	32.9
207	2.10E-01	1.37E-01	34.8
208	5.00E-01	3.36E-01	32.8
232	1.20E+01	4.56E+00	62.0
233	9.90E-03	8.77E-03	11.4
234	1.20E-02	1.09E-02	9.2
235	5.20E-01	4.98E-01	4.2
236	3.10E-02	2.91E-02	6.1
237	3.70E-02	3.31E-02	10.5
238	3.80E+01	3.56E+01	6.3
239	1.70E-01	1.08E-02	93.6
240	2.50E-02	8.84E-04	96.5
241	7.70E-03	4.19E-03	45.6
252	<5.1E-03	1.16E-03	< value in the "as-received" sample

5.0 Conclusions

The "as-received" OGCT sample, batch 27862, has been characterized for select analytes and radionuclides as requested in the TTR[2].

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

- The densities of the "as-received" OGCT sample and the OGCT filtrate averaged 1.02 g/mL, [0.1 %RSD] and 1.00 g/mL, [0.2%RSD], respectively.
- The turbidity for the "as-received" OGCT sample and the OGCT filtrate averaged 28 NTU [0.4 %RSD], and 1.52 NTU [0.8 %RSD], respectively.
- The OGCT sample was acidic, and the sample pH was 1.52 for both the "as-received" OGCT sample the OGCT filtrate.
- The OGCT sample weight percent total solids, dissolved solids, calculated weight percent insoluble solids and soluble solids are 0.29 wt.%, (10.3 %RSD), 0.23 wt.% (4.3 %RSD), 0.06 wt.% and 0.23 wt.%, respectively.
- The "as-received" OGCT sample PSD shows a bimodal distribution of OGCT particles. The particle sizes range from 0.3 to 10 microns with a mean particle size of 2.76 microns. The two modes for the distributions are at 0.84 and 9.30 microns. The integrated peak summary volume percent is 45.3% for particles with diameters greater than 4.64 microns and less than 54.7% for particle diameters less than 0.84 microns.
- The XRD mineral compositions for the OGCT solids fraction include traces of hematite (Fe_2O_3), and sodium nitrite ($NaNO_2$). These mineral peaks are relatively weak in signal intensities and not a lot of other peaks are above the instrument background signal-to-noise ratio.
- SEM/EDX information shows that the principal elemental constituents present in the "as-received" OGCT solid fraction include elemental mercury, iron, manganese, nickel, thorium, silicon, magnesium, aluminum, sodium, and ruthenium.
- Total beta activity in the "as-received" OGCT sample averaged 5.24E+08 dpm/mL (0.9 %RSD) with an average activity concentration of 2.36E-01 Ci/L. The average total alpha activity for the "as-received" OGCT sample was below instrument detection limit at <3.12E+07 dpm/mL (<1.41E-02 Ci/L).
- The primary beta emitting radionuclides in the "as-received" OGCT sample include Sr-90, Y-90 and Cs-137 at average activities of 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L), 1.95E+07 dpm/mL, 2.9 %RSD (8.77E-03 Ci/L), and 4.60E+08 dpm/mL, 1.0 %RSD (2.07E-01 Ci/L), respectively.
- The primary gamma emitting radionuclide in the "as-received" OGCT sample is Ba-137m at an average activity of 4.36E+08 dpm/mL, 1.0 %RSD (1.96E-01 Ci/L).
- Technetium-99, Cs-135, I-129, and Am-241 activities in the "as-received" OGCT sample averaged 7.89E+04 dpm/mL, 7.1 %RSD (3.55E-05 Ci/L), 2.65E+03 dpm/mL, 3.5 %RSD (1.20E-6 Ci/L), 4.47E+02 dpm/mL, 5.3 %RSD (2.02E-07), and 5.65E+04 dpm/mL, 15.3 %RSD (2.55E-05 Ci/L), respectively.
- The average activities for Ni-63, Eu-154 and Th-232 in the "as-received" OGCT sample were 1.01E+05 dpm/mL (4.55E-05 Ci/L, 95 %RSD), 1.22E+04 dpm/mL (5.50E-06 Ci/L, 16.5 %RSD), and 2.90E+00 dpm/mL (1.30E-09 Ci/L, 3.9 %RSD), respectively.

- Average actinide and Neptunium activities in the "as-received" OGCT sample include U-233 (2.10E+02 dpm/mL, 9.50E-08 Ci/L, 4.6 %RSD), U-234 (1.60E+02 dpm/mL, 7.40E-08 Ci/L, 5.0 % RSD), U-235 (2.50E+00 dpm/mL, 1.10E-09 Ci/L, 4.4 % RSD), U-236 (4.40E+00 dpm/mL, 2.00E-09 Ci/L, 5.9 %RSD), Np-237 (5.90E+01 dpm/mL, 2.60E-08 Ci/L, 3.0 %RSD) and U-238 (2.80E+01 dpm/mL, 1.30E-08 Ci/L, 3.7 %RSD).
- The activities for Pu-238, Pu-239, Pu-239/240 and Pu-241 in the "as-received" OGCT sample averaged Pu-238 (4.39E+05 dpm/mL, 1.98E-04 Ci/L (9.7 %RSD)), Pu-239 (2.40E+04 dpm/mL, 1.10E-05 Ci/L (2.0 %RSD)), Pu-239/240 (≤3.90E+04 dpm/mL, ≤1.76E-05 Ci/L), and Pu-241 (8.44E+04 dpm/mL, 3.80E-05 Ci/L (11.2 %RSD)).

The average activities for Ni-59 (absolute values used), Ni-63, I-129, Cs-135, Eu-154, Th-232, Pu-238, Pu-239, Pu-239/240 (absolute values used), Pu-241, and Am-241 in the OGCT filtrate sample all showed a significant decrease in activity in comparison to their initial activities in the "as-received" OGCT sample.

The difference in average activities for total beta, Sr-90, Y-90, Tc-99, Cs-137, Ba-137m, the uranium isotopes (U-233, U-234, U-235, U-236, U-238), and Np-237 in the OGCT filtrates in comparison to their average activities in the "as-received" OGCT sample were within the measurement uncertainties of 20% for these radionuclides.

Overall, there were significant drops in the ICP-AES analyte concentrations in the OGCT filtrates when compared to their ICP-AES concentrations in the "as-received" OGCT sample.

Of the 40 elements, including total mercury and mercury species, analyzed for in the "as-received" OGCT sample only the analytical results for 20 elements were above instrument detection limits.

Twenty six of the 40 elements analyzed in the OGCT filtrate were measured above instrument detection limits.

The elements with the most significant percentage drop in concentration (in the bracket) in the OGCT filtrate compared to their initial concentration in the "as-received" OGCT sample include Al (43%), Ba (23%), Ca (46%), Cr (33%), Fe (68%), Mg (62%), Mn (42%), Ni (72%), Th (63%), Ti (39%), As (82%), and Se (64%).

The analytical results for some elements in the OGCT filtrates were above instrument detection limits but below instrument detection limits in the "as-received" OGCT sample; possibly because of interferences due to incomplete digestions of the solids in the "as-received" OGCT sample prior to analysis or the existence of colloids after digestions. These two conditions will affect the detection limits for certain analytes, especially if the concentrations of these analytes are near their instrument detection limits. Elements that fall into this category include Ag, Cd, Gd, La, Sr, and Zn.

The predominant anion species present in both the "as-received" OGCT and OGCT filtrate samples were fluoride, chloride, nitrate, sulfate, and aluminate anion ($Al(OH)_4^-$). All other anions were less than minimum detection limits in both sample mediums. The concentrations for fluoride, chloride, nitrate, and sulfate anions in both the "as-received" OGCT and OGCT filtrate samples were almost equal in magnitude.

There seem to be a significant data scatter in the average total aid titration results for the undiluted OGCT filtrate in comparison with the titration results for the undiluted OGCT supernatant sample at pH 7, 9, and

11. Therefore, their corresponding average total acid titration results for these two types of samples are not significantly different from each other.

In the OGCT filtrate sample, the concentration for elemental mercury dropped from an average high of 82.2 mg/L in the "as-received" OGCT supernatant sample to an average of 14.7 mg/L in the OGCT filtrate, which is a drop of about 82%. The measured average concentration for ionic mercury in the OGCT supernatant sample [14.3 mg/L (11.0 %RSD)] is significantly lower than the average ionic mercury concentration in the OGCT filtrate sample, which averaged 24.5 mg/L (11.1 %RSD). The difference in ionic mercury concentration in both sample types can be attributed to the large one sigma analytical uncertainty of 40% for ionic mercury determinations.

Ethyl mercury, dimethyl mercury, and methyl mercury are not present in the "as-received" OGCT supernatant sample and OGCT filtrate samples at detectable levels.

Mercury mass balance in both the "as-received" OGCT supernatant, and OGCT filtrate sample are poor due to the concentrations of some mercury species (elemental and ionic mercury species) being above their saturation levels in the OGCT sample medium. As a result, the one sigma analytical uncertainty for elemental (purgeable mercury) and inorganic mercury analyses were reported as 40%, which means the analytical results for these two mercury species are biased low in these cases.

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 [10, 11]. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. The experimental work, the analyses, and peer checks all comply with the customer quality assurance (QA) requirements.

7.0 References

1. "Diversion of DWPF Recycle from HLW Tank Farms System Engineering Alternatives Analysis," G-AES-S-00005 Revision 0, September 25, 2019.
2. "DWPF Recycle Diversion - OGCT Characterization", X-TTR-H-00107, Rev. 0, 5/26/2021.
3. L. N. Oji, "Task Technical and Quality Assurance Plan for the Analysis of the Recycle Collection Tank, Slurry Mix Evaporator Condensate Tank and the Off-Gas Condensate Tank Vessel Samples" SRNL-RP-2020-00915, Revision 0, 01/21/2021.
4. L. N. Oji, "Analysis of Defense Waste Processing Facility Sample: Recycle Collection Tank Sample Batch 4945," SRNL-STI-2021-00228, Revision 1, April 2022.
5. L. N. Oji, "Analysis of Defense Waste Processing Facility Sample: Slurry Mix Evaporator Condensate Tank Sample Batch 4638," SRNL-STI-2021-00231, Revision 0, May 2022.
6. 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.
7. C.J. Martino, J.M. Pareizs, and J.D. Newell, "Thermolytic Hydrogen Generation Testing of Tank 22 Material", SRNL-STI-2018-00385, Revision 0.
8. A. J. Boggess, T. L. White, M.A. Jones, T. B. Edwards, and S. P. Harris, "Development and Comparison of Purgeable Mercury Values in SRR Samples Measured by SRNL and Eurofins FGS", SRNL-STI-2019-00300, revision 0, September 2019.
9. A. J. Boggess, T. L. White, M.A. Jones, T. B. Edwards, "Analysis of Ionic Mercury Species in SRR Samples Measured by SRNL and Eurofins FGS", SRNL-STI-2020-00081, Revision 0, May 2020.
10. "Technical Reviews", Manual E7, Procedure 2.60, Revision 17, August 25, 2016.
11. "Savannah River National Laboratory Technical Report Design Check Guidelines", WSRC-IM-2002-00011, Revision 2, August 2000.

Appendix A: Total Acid Titration Curves: Undiluted OGCT and Undiluted OGCT Filtrates

APD Laboratory Titration Report

Savannah River National Laboratory

Method

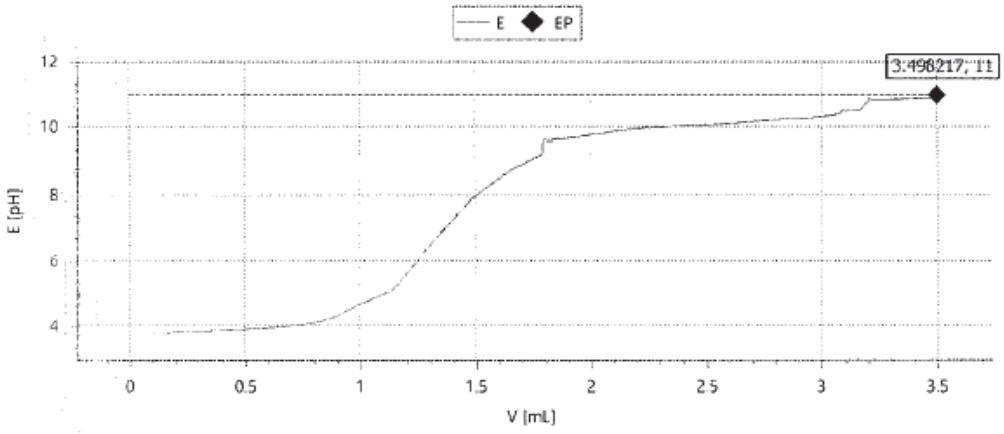
Method ID TA
Name Total Acid to pH 7, 9, 11

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
98-2		Consumption to pH 7	R1	1.3701 mL	OK	6/11/2021 1:27:03 PM
98-2		Total acid to pH 7	R2	0.0686 M	OK	6/11/2021 1:27:03 PM
98-2		Consumption to pH 9	R4	1.7251 mL	OK	6/11/2021 1:27:03 PM
98-2		Total acid to pH 9	R5	0.0864 M	OK	6/11/2021 1:27:03 PM
98-2		Consumption to pH 11	R6	3.4982 mL	OK	6/11/2021 1:27:03 PM
98-2		Total acid to pH 11	R7	0.1753 M	OK	6/11/2021 1:27:03 PM

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

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



6/16/2021 12:49:05 PM SystemInternal 1/2

APD Laboratory Titration Report

Savannah River National Laboratory

Method

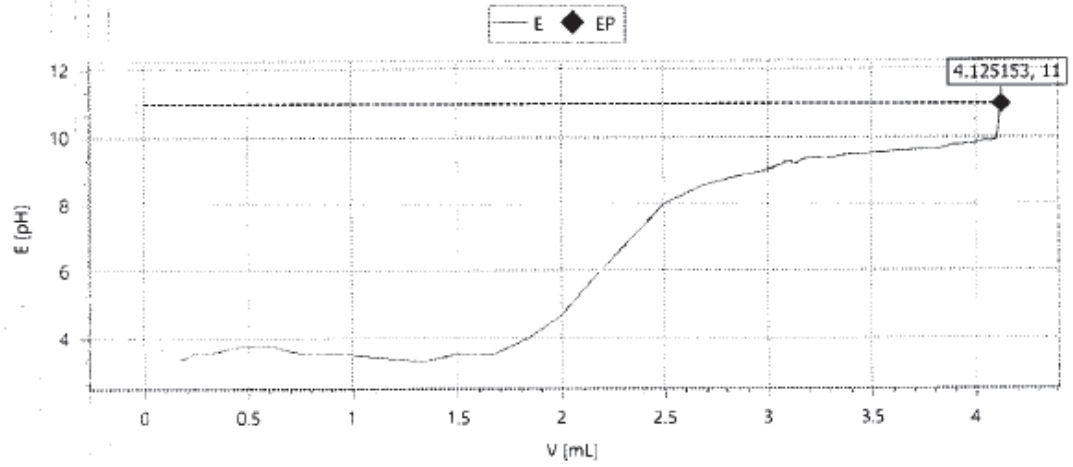
Method ID TA
Name Total Acid to pH 7, 9, 11

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
98-1		Consumption to pH 7	R1	2.3488 mL	OK	6/11/2021 1:24:29 PM
98-1		Total acid to pH 7	R2	0.1177 M	OK	6/11/2021 1:24:29 PM
98-1		Consumption to pH 9	R4	2.9948 mL	OK	6/11/2021 1:24:29 PM
98-1		Total acid to pH 9	R5	0.1500 M	OK	6/11/2021 1:24:29 PM
98-1		Consumption to pH 11	R6	4.1252 mL	OK	6/11/2021 1:24:29 PM
98-1		Total acid to pH 11	R7	0.2067 M	OK	6/11/2021 1:24:29 PM

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

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



APD Laboratory Titration Report

Savannah River National Laboratory

Method

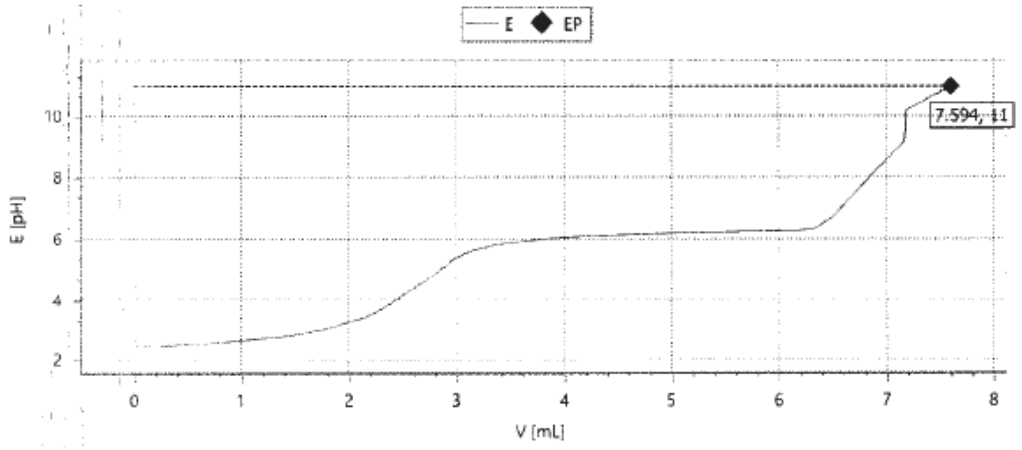
Method ID TA
Name Total Acid to pH 7, 9, 11

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
96		Consumption to pH 7	R1	6.5758 mL	OK	6/11/2021 1:35:13 PM
96		Total acid to pH 7	R2	0.1318 M	OK	6/11/2021 1:35:13 PM
96		Consumption to pH 9	R4	7.1164 mL	OK	6/11/2021 1:35:13 PM
96		Total acid to pH 9	R5	0.1426 M	OK	6/11/2021 1:35:13 PM
96		Consumption to pH 11	R6	7.5940 mL	OK	6/11/2021 1:35:13 PM
96		Total acid to pH 11	R7	0.1522 M	OK	6/11/2021 1:35:13 PM

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

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



APD Laboratory Titration Report

Savannah River National Laboratory

Method

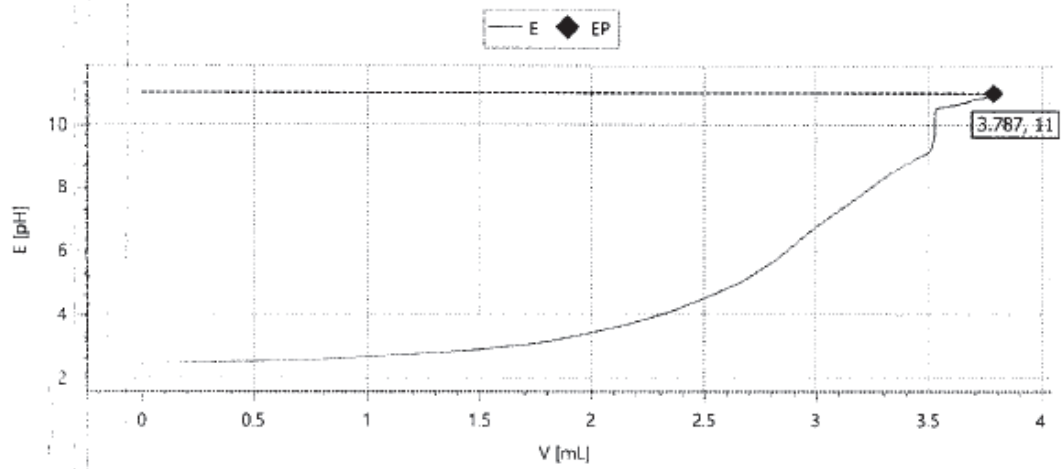
Method ID TA
Name Total Acid to pH 7, 9, 11

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
97		Consumption to pH 7	R1	3.0427 mL	OK	6/11/2021 1:44:01 PM
97		Total acid to pH 7	R2	0.0678 M	OK	6/11/2021 1:44:01 PM
97		Consumption to pH 9	R4	3.4652 mL	OK	6/11/2021 1:44:01 PM
97		Total acid to pH 9	R5	0.0772 M	OK	6/11/2021 1:44:01 PM
97		Consumption to pH 11	R6	3.7870 mL	OK	6/11/2021 1:44:01 PM
97		Total acid to pH 11	R7	0.0843 M	OK	6/11/2021 1:44:01 PM

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

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



Results Editor

LabX

Result Overview

San	Sampl	Sample s	Over	Excluc	Correc	Unit o	Temp	Unit o	Volum	Unit o	Consu	Unit o	Data r	Total :	Unit o	Data r	Consu	Unit o	Data r	Densit	Unit o	Comn	Total :	Unit o	Data r	Consu	Unit o	Data r	Total :	Unit o	Data r
1	48-1	8/3/202	OK		1.0		25.0	°C	0.5	mL	2.967	mL		0.059	M		3.815	mL		1.0	g/mL		0.077	M		9.233	mL		0.186	M	
2	48-2	8/3/202	OK		1.0		25.0	°C	0.5	mL	3.373	mL		0.068	M		4.149	mL		1.0	g/mL		0.083	M		9.219	mL		0.186	M	
3	49-1	8/3/202	OK		1.0		25.0	°C	0.5	mL	3.426	mL		0.069	M		4.258	mL		1.0	g/mL		0.086	M		9.194	mL		0.185	M	
4	49-2	8/3/202	OK		1.0		25.0	°C	0.5	mL	3.465	mL		0.070	M		4.290	mL		1.0	g/mL		0.086	M		9.368	mL		0.189	M	

E248A

APD Laboratory Titration Report

Savannah River National Laboratory

Method

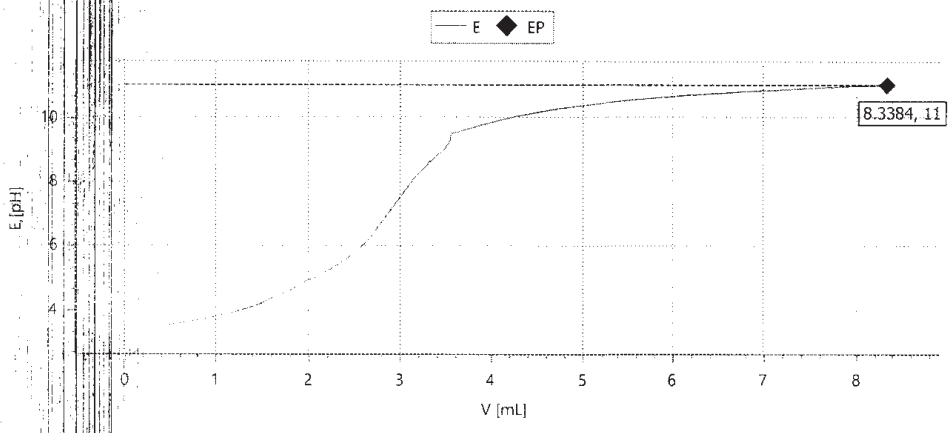
Method ID TA using 0.01 N NaOH
Name Total Acid to pH 7, 9, 11

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
22047		Consumption to pH 7	R1	2.8760 mL	OK	8/3/2021 10:29:42 AM
22047		Total acid to pH 7	R2	0.0581 M	OK	8/3/2021 10:29:42 AM
22047		Consumption to pH 9	R4	3.4940 mL	OK	8/3/2021 10:29:42 AM
22047		Total acid to pH 9	R5	0.0706 M	OK	8/3/2021 10:29:42 AM
22047		Consumption to pH 11	R6	8.2980 mL	OK	8/3/2021 10:29:42 AM
22047		Total acid to pH 11	R7	0.1676 M	OK	8/3/2021 10:29:42 AM

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

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



8/3/2021 10:34:29 AM SystemInternal 1/2

APD Laboratory Titration Report

Savannah River National Laboratory

Method

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

Main Results

Sample ID1	Sample ID2	Result Title	Name	Result	State	Sample start time
48-1		Consumption to pH 7	R1	2.9678 mL	OK	8/3/2021 10:42:46 AM
48-1		Total acid to pH 7	R2	0.0599 M	OK	8/3/2021 10:42:46 AM
48-1		Consumption to pH 9	R4	3.8154 mL	OK	8/3/2021 10:42:46 AM
48-1		Total acid to pH 9	R5	0.0771 M	OK	8/3/2021 10:42:46 AM
48-1		Consumption to pH 11	R6	9.2330 mL	OK	8/3/2021 10:42:46 AM
48-1		Total acid to pH 11	R7	0.1865 M	OK	8/3/2021 10:42:46 AM
48-2		Consumption to pH 7	R1	3.3730 mL	OK	8/3/2021 10:47:00 AM
48-2		Total acid to pH 7	R2	0.0681 M	OK	8/3/2021 10:47:00 AM
48-2		Consumption to pH 9	R4	4.1491 mL	OK	8/3/2021 10:47:00 AM
48-2		Total acid to pH 9	R5	0.0838 M	OK	8/3/2021 10:47:00 AM
48-2		Consumption to pH 11	R6	9.2190 mL	OK	8/3/2021 10:47:00 AM
48-2		Total acid to pH 11	R7	0.1862 M	OK	8/3/2021 10:47:00 AM
49-1		Consumption to pH 7	R1	3.4260 mL	OK	8/3/2021 10:51:52 AM
49-1		Total acid to pH 7	R2	0.0692 M	OK	8/3/2021 10:51:52 AM
49-1		Consumption to pH 9	R4	4.2581 mL	OK	8/3/2021 10:51:52 AM
49-1		Total acid to pH 9	R5	0.0860 M	OK	8/3/2021 10:51:52 AM
49-1		Consumption to pH 11	R6	9.1940 mL	OK	8/3/2021 10:51:52 AM

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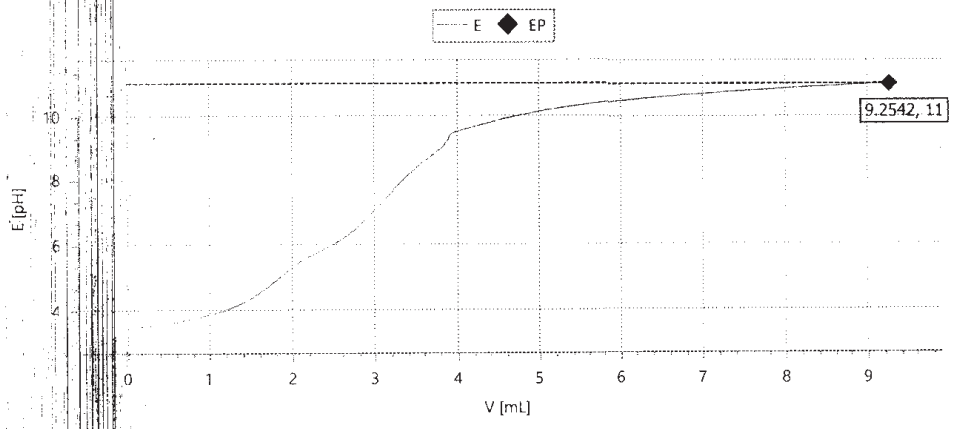
49-1	Total acid to pH 11	R7	0.1857 M	OK	8/3/2021 10:51:52 AM
49-2	Consumption to pH 7	R1	3.4652 mL	OK	8/3/2021 10:58:13 AM
49-2	Total acid to pH 7	R2	0.0700 M	OK	8/3/2021 10:58:13 AM
49-2	Consumption to pH 9	R4	4.2904 mL	OK	8/3/2021 10:58:13 AM
49-2	Total acid to pH 9	R5	0.0867 M	OK	8/3/2021 10:58:13 AM
49-2	Consumption to pH 11	R6	9.3687 mL	OK	8/3/2021 10:58:13 AM
49-2	Total acid to pH 11	R7	0.1892 M	OK	8/3/2021 10:58:13 AM

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

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



Titration (EP) [1] Scope 1/1, Sample 2/4

E - V Curve Scope 1/1, Sample 2/4

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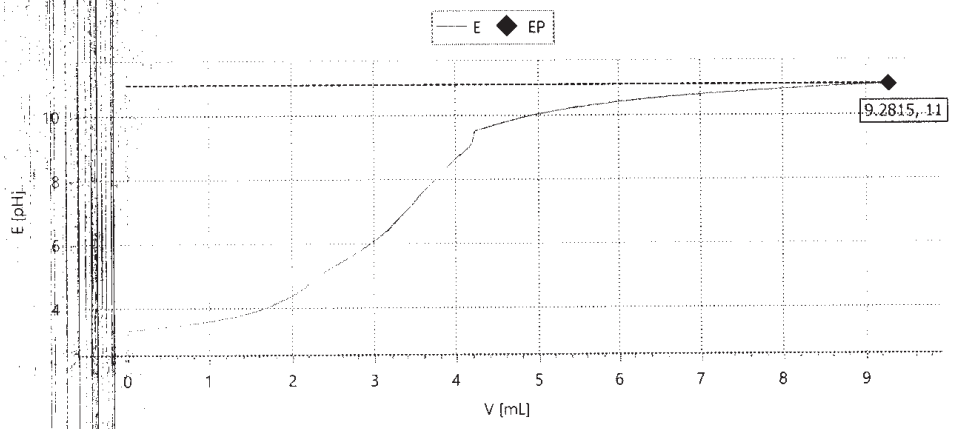
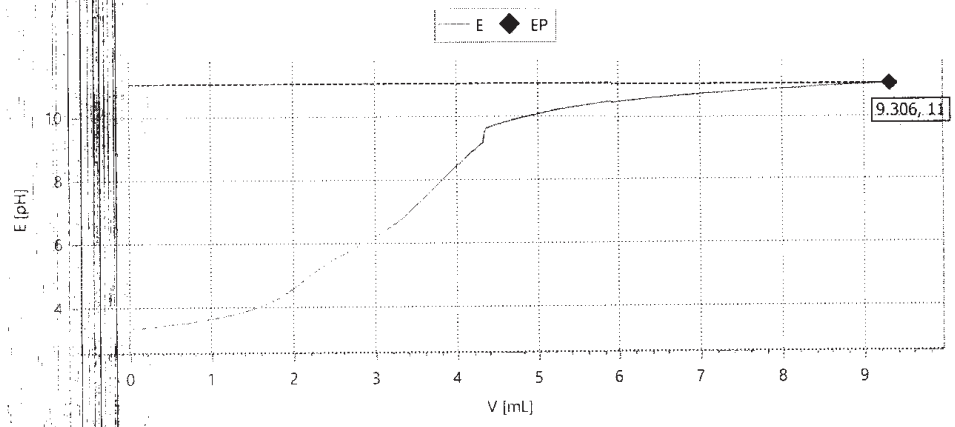


Titration (EP) [1] Scope 1/1, Sample 3/4

E - V Curve Scope 1/1, Sample 3/4



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

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

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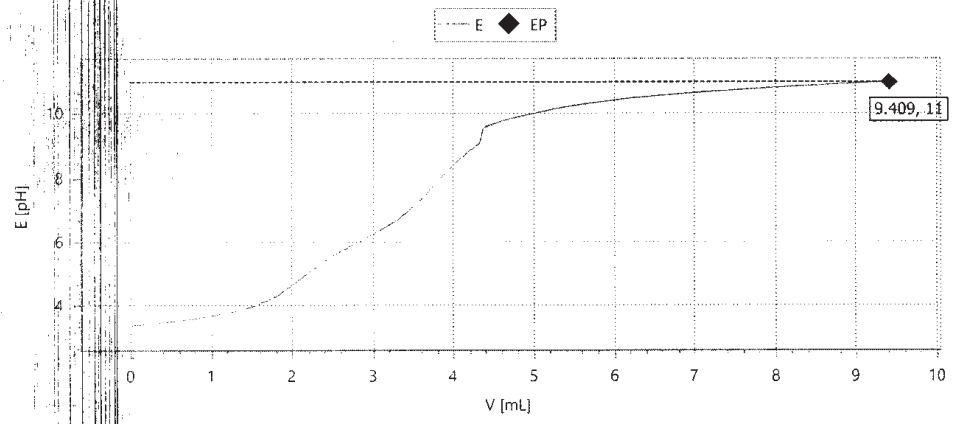

Used Instruments

Name	B-122 Titrator	**Serial number**	B909284082

Appendix B: OGCT Sample Characterization: SaM Tracking Numbers*

Analytes	SRNL SaM Tracking Number (LIMS)
Total alpha	LW22087-LW22089, LW22041-LW22042
Total beta	LW22087-LW22089, LW22041-LW22042
Cs-135	LW22020-LW22021, LW22102-LW22104
Ni59/63	LW22084- LW22086, LW22014- LW22016
Co-60	LW22081- LW22083, LW22011- LW22013
Sr-90	LW22084- LW22086, LW22014- LW22016
Tc-99	LW22084- LW22086, LW22014- LW22016
Ru-106	LW22081- LW22083, LW22011- LW22013
Sb-125	LW22081- LW22083, LW22011- LW22013
Sb-126	LW22081- LW22083, LW22011- LW22013
Sn-126	LW22081- LW22083, LW22011- LW22013
I-129	LW22099-LW22101, LW22050-LW22052
Cs-134	LW22017-LW22019, LW22090-LW22092
Cs-137	LW22017-LW22019, LW22090-LW22092
Ce-144	LW22081- LW22083, LW22011- LW22013
Eu-152	LW22081- LW22083, LW22011- LW22013
Eu-154	LW22081- LW22083, LW22011- LW22013
Eu-155	LW22081- LW22083, LW22011- LW22013
Th-232	LW22111- LW22113, LW22026- LW22029
U-233	LW22111- LW22113, LW22026- LW22029
U-234	LW22111- LW22113, LW22026- LW22029
U-235	LW22111- LW22113, LW22026- LW22029
U-236	LW22111- LW22113, LW22026- LW22029
U-238	LW22111- LW22113, LW22026- LW22029
NP-237	LW22111- LW22113, LW22026- LW22029
Np-239	LW22081- LW22083, LW22011- LW22013
Pu-238	LW22093- LW22095, LW22037- LW22039
Pu-239/ Pu-240	LW22093- LW22095, LW22037- LW22039
Pu-241	LW22093- LW22095, LW22037- LW22039
Am-241	LW22081- LW22083, LW22011- LW22013
Am-243	LW22081- LW22083, LW22011- LW22013
Wt. % total solids	See Electronic Notebook L. N. Oji: ELN: L5575-00080-16
Wt. % dissolved solids	See Electronic Notebook L. N. Oji: ELN: L5575-00080-16

*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-Continued: OGCT Sample Characterization: SaM Tracking Numbers*

Analyte	SRNL SaM Tracking Number (LIMS):
ICP-AES (Elementals)	LW22111-LW22113, LW22027-LW22029
As, S, Se	LW22030-LW22032, LW22114-LW22116
ICP-MS	LW22026-LW22028, LW22111-LW22113
Total acids	LW22096-LW22098, LW22047-LW22049
Total Hg	LW22117-LW22119, LW22033-LW22035
Methyl Hg	LW22120- LW22122
Dimethyl Hg	LW22126- LW22128, LW22078-LW 22080
Elemental Hg	LW22123- LW22125, LW22075- LW22077
Ionic mercury	LW22123- LW22125, LW22075- LW22077
Ethyl Hg	LW22126- LW22128, LW22078-LW 22080
TIC/TOC	LW22108-LW22110, LW22069-LW22071
IC-Anions	LW22105-LW22107, LW22065-LW22068
IC-Cations	LW22105-LW22107, LW22065-LW22068
Particle size analysis	LW22062
XRD	LW22063
SEM	LW22064

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

Appendix C: Summary of Analytical Methods

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

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

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

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

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

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

Quantitative analysis for S is performed on the Agilent 5110 Inductively Coupled Plasma Atomic Emission 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 OGCT 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 OGCT samples were subjected to a Cs-removal process utilizing Bio Rad AMP-1 resin. The Cs-removed solutions were analyzed by coaxial high purity germanium spectrophotometers to measure the gamma-emitting radionuclides listed above. Aliquots of SMECT samples were analyzed for Co-60 and Am-241. Sb-125, Sb-126 and Sn-126 were also measured from these analyses.

Pu-238, 239/240, 241

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

Ni-63

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

I-129

OGCT 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 OGCT 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 OGCT samples that had undergone peroxide fusion dissolutions were purified using a solvent-solvent caustic side solvent extraction-based (CSSX) extraction system. The purified Cs-containing aliquots were analyzed using ICP-MS to measure Cs-135 masses. Cs-137 was measured in the purified Cs-containing aliquots by gamma spectrometry. Cs yields were determined by using the ratio of the Cs-137 concentrations measured in the purified aliquots to the Cs-137 concentrations previously measured on dissolutions of the OGCT 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: Dimethyl, Methyl, and ethyl mercury analysis

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

Ionic and Elemental Hg

Analysis for inorganic mercury (ionic mercury) is a two-step process involving, first, the removal of traces of volatile elemental mercury species (purgeable mercury- suspended colloidal, mercury particulates, dense-phase mercury, and any volatile organomercury species) from the sample. In this initial processing to remove elemental mercury, the sample is purged with nitrogen and other gases and the purged mercury (purgeable mercury) trapped onto an adsorbent media for elemental mercury analysis. Following this initial

purge, stannous chloride is used to reduce ionic mercury (Hg(I) and Hg(II)) to Hg(0) in the post gas purged sample. Finally, a second purge is performed to remove the reduced ionic mercury, where it can then be trapped on gold-coated beads. Details of the experimental procedure for analyzing SRS waste samples for mercury species are detailed in another report.[8,9]

Gross Alpha/Gross Beta

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

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

Anions in the Supernatant

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

Weight Percent Solids Measurement (Solids Distribution)

The weight percent total solids for the OGCT 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 OGCT 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 OGCT 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 OGCT 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" OGCT slurry sample. A small volume of the OGCT slurry (3-5 mL) was suspended in Tank 22 salt simulant described above and the particle size determined.

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