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Cesium Ion Exchange Testing Using Crystalline Silicotitanate with Hanford Tank Waste 241-AP-107

August 2018

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U.S. DEPARTMENT OF
ENERGY

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Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

At the time of this testing, the Low-Activity Waste Pretreatment System (LAWPS) was to provide for the initial production of immobilized low-activity waste by feeding Hanford tank supernate from tank farms to the Hanford Tank Waste Treatment and Immobilization Plant (WTP) Low-Activity Waste (LAW) Facility for immobilization. Washington River Protection Solutions requested that Hanford tank waste collected from tank 241-AP-107 (hereafter called AP-107) be processed using conceived pretreatment steps (suspended solids removal by filtration, Cs removal by ion exchange) then vitrified. A small-scale test platform to demonstrate the solids filtration, Cs removal, and LAW vitrification was constructed and installed at Pacific Northwest National Laboratory. Bench-scale ion exchange testing with approximately 9 L of AP-107 supernate was conducted using crystalline silicotitanate (CST) ion exchange media. The IONSIV R9140-B CST was provided by Honeywell UOP, LLC in 2018 (Batch 2081000057). The ion exchange media was first tested with simulant and was previously described.[1] This report describes the Cs ion exchange batch contact and column test results with the AP-107 tank waste.

Batch contact testing helps to evaluate CST performance on tank waste supernate prior to processing it in the ion exchange columns. Batch contacts were performed with the waste at four Cs concentrations at a phase ratio of 200 (liquid volume to exchanger mass) with AP-107. The distribution coefficient (K_d) at the equilibrium condition of 8.57 µg Cs/mL (AP-107 feed condition) was determined to be 669 mL AP-107/g CST. With a CST bed density of 1.00 g/mL, this K_d corresponded to a predicted 50% Cs breakthrough of 669 bed volumes (BVs). The Cs load capacity at the equilibrium feed condition was determined to be 7.5 mg Cs/g dry CST.

The column testing was prototypic to the intended LAWPS operations in a lead-lag column format, although on a small-scale basis with 10-mL CST beds. The feed was processed downflow through the lead column and then through the lag column at ~2.2 BV/h. Loading continued until the lag column reached the WTP waste acceptance criteria (WAC) for receiving supernatant waste for vitrification (a function of the Na and ^{137}Cs concentrations). For AP-107, the WAC is 0.114% of the influent ^{137}Cs concentration; this required a Cs decontamination factor of 876. The Cs effluent from the lag column reached the WAC after processing ~410 BVs. To keep the subsequent product effluent below the WAC, a replacement lag column was prepared, the lead column was removed from service (after processing a total of 471 BVs), the lag column was put into the lead column position, and the replacement lag column was installed. Feed processing continued and after another ~290 BVs the Cs effluent from the lag column again exceeded the WAC.

In both cases, the lead columns only reached 25% Cs breakthrough before removal. Although 50% Cs breakthrough was not reached, this value was estimated and averaged based on extrapolation of the loading curves (640 BVs) and agreed within 4% of the predicted 50% Cs breakthrough from batch contact test results (669 BVs). Table ES.1 summarizes the observed column performance and relevant Cs loading characteristics.

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[1] Fiskum SK, HA Colburn, RA Peterson, AM Rovira, and MR Smoot. 2018. *Cesium Ion Exchange Using Crystalline Silicotitanate with 5.6 M Sodium Simulant*. PNNL-27587, Rev. 0; RPT-DFTP-008, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington.

Table ES.1. AP-107 Column Performance Summary with CST

		Lead Column		Lag Column	
Loading Range (BVs)	Flowrate (BV/h)	Extrapolated 50% Cs Breakthrough (BVs)	µCi Loaded	Contract Limit Breakthrough (BVs)	µCi Loaded
0-470	2.20	615	680,600	410	46,400
470-890	2.19(a)	664(b)	640,900	753	58,300

(a) The average flowrate did not include 10 hours processing at 1.36 BV/h, 825-837 BVs.

(b) This was a normalized value, incorporating a 393 BV negative offset, to compare directly with the original lead column. The un-shifted extrapolated value was 1057 BVs.

The AP-107 feed and effluent were characterized. Resource Conservation and Recovery Act (RCRA) elements Cd and Cr partitioned almost exclusively to the effluent. Pb and As were detected in the feed (with concentration errors likely to exceed 15%) but were below the method detection limit (MDL) in the effluent. Ag and Se were below the MDL in both the feed and the effluent; therefore, partitioning could not be assessed. In addition to Cs removal, large fractions of Ba, Ca, Sr, U, ^{90}Sr, and Pu were also significantly removed by the CST. Ni partitioned almost exclusively to the effluent. Concentrations and recoveries of selected analytes are summarized in Table ES.2; those with low recovery were assumed to be adsorbed onto CST.

Table ES.2. Selected Analyte Recovery in the AP-107 Effluent

	Analyte	Feed Concentration (µg/mL)	Effluent Concentration (µg/mL)	Effluent Recovery
RCRA metals	Ag	--	--	--
	As	[11]	--	--
	Ba	0.836	[0.14]	[16%]
	Cd	6.62	5.14	77%
	Cr	498	480	95%
	Pb	[9.0]	--	--
	Se	--	--	--
Other analytes of interest	Ca	34.1	17.0	49%
	Ni	21.0	20.8	98%
	Sr	0.331	[0.039]	[12%]
	U	[48]	[13]	[26%]
	^{238}U	20.7	10.8	52%
		µCi/mL	µCi/mL	
	^{90}Sr	5.81E-1	2.72E-4	0.046%
	$^{239+240}$Pu	5.43E-4	1.90E-4	35%

Notes:
"--" indicates the value was < MDL and effluent recovery could not be calculated.
Values in brackets [ ] were ≥ MDL but < EQL, with errors likely to exceed 15%.
EQL = estimated quantitation limit.

Acknowledgments

The authors thank the Shielded Facilities Operations staff LaWanda Grow, Jarrod Turner, and Michael Rojas for in-cell sample handling and systems manipulations. We thank the Analytical Support Operations (ASO) count room staff Truc Trang-Le and Mike Cantaloub for rapid ^{137}Cs analysis for the column load and batch contact sample analyses. We thank the ASO staff Karl Pool, Lori Darnell, Crystal Rutherford, Jenn Carter, Garrett Brown, Chuck Soderquist, and Deborah Coffey for the sample analysis processing and reporting. We thank Renee Russell for reviewing the ion exchange calculation files, test data packages, and this technical report. We also thank Matt Wilburn for technical editing of this report.

Acronyms and Abbreviations

AEA	alpha energy analysis
ASO	Analytical Support Operations
ASR	Analytical Service Request
AV	apparatus volume
BT	breakthrough
BV	bed volume
CST	crystalline silicotitanate
DF	decontamination factor
DI	deionized (water)
DSSF	double-shell slurry feed
EQL	estimated quantitation limit
FD	feed displacement
GEA	gamma energy analysis
IC	ion chromatography
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
ID	identification (number)
LAW	low-activity waste
LAWPS	Low-Activity Waste Pretreatment System
MDL	method detection limit
PNNL	Pacific Northwest National Laboratory
PTFE	polytetrafluoroethylene
QA	quality assurance
R&D	research and development
RCRA	Resource Conservation and Recovery Act
SRF	spherical resorcinol-formaldehyde
TIC	total inorganic carbon
TOC	total organic carbon
UOP	Honeywell UOP LLC
WAC	waste acceptance criteria
WRPS	Washington River Protection Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WWFTP	WRPS Waste Form Testing Program

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1.0 Introduction

At the time of testing, the Low-Activity Waste Pretreatment System (LAWPS) facility was planned to pretreat Hanford tank waste supernate by filtering solids and processing through ion exchange columns to remove cesium (Cs). Removal of Cs from these wastes is an important processing step in preparing the waste for long-term safe storage. The LAWPS Project is experiencing many modifications from its previous design. Changes of particular interest for the work described herein include changing the ion exchange media from the elutable spherical resorcinol-formaldehyde (SRF) resin to the non-elutable, inorganic, ion exchanger crystalline silicotitanate (CST), manufactured in a spherical (engineered) form by Honeywell UOP LLC (UOP; Des Plaines, IL). The use of inorganic ion exchangers offers many advantages over the use of regenerable organic ion exchangers. The inorganic exchangers are generally more resistant to chemical, thermal, and radiation degradation (Pillay 1986; Fiskum et al. 2006; King 2007; Brown 2014).

CST use has been studied for defense waste Cs removal at the Savannah River Site, Melton Valley, and Hanford (King 2007; Walker et al. 1998; Hendrickson et al. 1996). However, since these earlier tests were conducted, UOP had modified the CST formulation to make the CST more robust to fines generation, column plugging, and Nb and Zr leaching. Further, only limited studies have been conducted on Hanford tank wastes. Recent testing of a 5.6 M Na simple simulant on the current CST media formulation was conducted (Fiskum et al. 2018a) where three column flow rates were employed and the lead column processed the simulant to ~80% Cs breakthrough.

The goal of this current study was to evaluate the effectiveness of CST at removing Cs from Hanford tank 241-AP-107 (hereafter called AP-107). Washington River Protection Solutions (WRPS) requested that Pacific Northwest National Laboratory (PNNL) process AP-107 tank waste using batch contact and column testing to determine Cs removal behavior of the CST.

This report discusses results of batch contact and column testing studies from AP-107 actual Hanford tank waste. This report also discusses the results of the batch contact test in relation to the column test and the effectiveness of using batch contacts to predict column performance. Further, the column effluent chemical composition was compared with the feed composition to assess ion exchange behavior of selected elements (such as U, Ba, Sr, Ca, K, Pb, and Pu).

1.1 Quality Assurance

The work described in this report was conducted with funding from WRPS contract 36437/212, *DFLAW Radioactive Waste Test Platform*. This contract was managed under PNNL Project 71274. All research and development (R&D) work at PNNL is performed in accordance with PNNL's Laboratory-Level Quality Management Program, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 2000), to R&D activities. To ensure that all client quality assurance (QA) expectations were addressed, the QA controls of the PNNL's WRPS Waste Form Testing Program (WWFTP) QA program were also implemented for this work. The WWFTP QA program implements the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 2008), and NQA-1a-2009, *Addenda to ASME NQA-1-2008* (ASME 2009), and consists of the *WWFTP Quality Assurance Plan* (QA-WWFTP-001) and associated QA-NSLW-numbered procedures that provide detailed instructions for implementing NQA-1 requirements for R&D work.

The work described in this report was assigned the technology level "Applied Research" and was planned, performed, documented, and reported in accordance with procedure QA-NSLW-1102, *Scientific Investigation for Applied Research*. All staff members contributing to the work received proper technical and QA training prior to performing quality-affecting work.

2.0 Test Conditions

This section describes the CST media, AP-107 tank waste, batch contact conditions, and column ion exchange conditions. All testing was conducted in accordance with a test plan prepared by PNNL and approved by WRPS.[1]

2.1 CST Media

The CST ion exchange media (provided to PNNL by WRPS) was obtained from Honeywell UOP LLC, Des Plaines, IL. A 3-kg sample of sodium form IONSIV R9140-B, Batch 2081000057, was received at PNNL on February 26, 2018, and used for testing. The CST was sieved through a 25-mesh sieve and collected on a 60-mesh sieve to remove both particles >710 µm and <250 µm. Then the CST was washed with deionized (DI) water. A full description of the CST sampling, pretreatment, and physical property measurement was previously provided by Fiskum et al. (2018a). Table 2.1 provides a summary of salient CST physical properties.

Table 2.1. Washed R9140-B, Batch 2081000057, CST Physical Properties (Fiskum et al. 2018a)

Parameter	Average Value
Bulk density, g/mL	1.00
CST bed density, g/mL	1.00
Settled bed void volume, %	65.6
Particle size, microns[(a)]	D10: 418, D50: 571, D90: 775

(a) Volume basis.

2.2 AP-107 Tank Waste

Multiple samples (32 each at nominally 250 mL) were collected from the AP-107 Hanford tank in October 2017. The first and last samples collected, 7AP-17-11 and 7AP-17-46, were subsampled for a limited analysis suite to confirm density, Na, K, OH, and Cs concentrations. The densities were measured in-cell using a 10-mL volumetric flask. All other measurements were conducted by the Analytical Support Operations (ASO) according to Analytical Service Request (ASR) 0395.01; results are provided in Table 2.2. The results of the two samples agreed well, indicating the 32 samples were likely homogenous.

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[1] Fiskum, SK. 2018. TP-DFTP-029, Rev.0.0. *DFLAW Test Platform Cesium Ion Exchange Testing with AP-107 Tank Waste and 5.6 M Na Simple Simulant*. Pacific Northwest National Laboratory, Richland Washington.

Table 2.2. Characterization of Samples 7AP-17-11 and 7AP-17-46 Collected from Hanford Tank AP-107 (ASR 0395.01)

Analyte	7AP-17-11 Result	7AP-17-46 Result	Result Units	Analysis Method
Al	0.363	0.365	M	ICP-OES
K	0.095	0.099	M	ICP-OES
Na	5.611	5.633	M	ICP-OES
OH^-	0.93[a]	0.90[a]	M	Titration
^{133}Cs	5.42	5.84	µg/mL	ICP-MS
^{137}Cs	154[b]	159[b]	µCi/mL	GEA
^{137}Cs	1.77[b]	1.83[b]	µg/mL	GEA
Density	1.2698[c]	1.2630[c]	g/mL	Volumetric flask

(a) Based on first inflection point; assumed to be the free (unbound) hydroxide.
(b) Reference date is 11/27/17.
(c) Measured at 26.6 °C using a 10-mL volumetric flask.
ASR 0395.01, sample 18-0117 and 18-0118, see Appendix B.
GEA = gamma energy analysis; ICP-MS = inductively coupled plasma mass spectrometry; ICP-OES = inductively coupled plasma optical emission spectrometry

The Cs isotopic composition of the AP-107 samples was measured to determine the total Cs concentration in the AP-107 tank waste. Except for ^{133}Cs, direct analysis of AP-107 for the ^{135}Cs and ^{137}Cs isotopes is fraught with isobaric interferences. Therefore, subsamples (first and last AP-107 tank samples collected, 7AP-17-11 and 7AP-17-46 of AP-107) were processed to isolate Cs. Aliquots (1.5 mL) of AP-107 were batch contacted with 2 mL Na-form SRF resin suspended in 8 mL 1 M NaOH. The slurries were mixed for 24 hours on a shaker at room temperature. The aqueous phase was decanted and the SRF was washed three times with 6 mL 0.1 M NaOH, then rinsed three times with 6 mL DI water. Cs was eluted from the SRF resin with 0.45 M HNO_3. Quantitative recovery was not required because only the Cs isotope ratios were needed, and isotope fractionation does not occur in Cs uptake to, or elution from, SRF resin. The elution aliquots were measured by ICP-MS for Cs isotopic distribution; results are provided in Table 2.3. The total Cs concentration was calculated from the GEA measured ^{137}Cs and the ICP-MS measured isotopic composition. The calculated ^{133}Cs concentration agreed within 6% of the ICP-MS measured ^{133}Cs concentration (shown in Table 2.3).

Table 2.3. 7AP-17-11 and 7AP-17-46 Average Cs Isotopic Composition (ASR 0457)

Analyte[a]	7AP-17-11 Results	7AP-17-46 Results	Units
Cs isotopic mass ratio[a,b,c]	61.7	62.0	wt% ^{133}Cs
	17.2	17.0	wt% ^{135}Cs
	21.0	20.9	wt% ^{137}Cs
Total Cs	8.57		µg/mL Cs

(a) The Cs eluate samples (7AP-17-11-Cs and 7AP-17-46-Cs) were analyzed for the Cs isotopic mass distribution by ICP-MS per ASR 0457 samples 18-0832 and 18-0833, see Appendix B.
(b) Reference date is February 16, 2018.
(c) ^{134}Cs, a fission product, was not detected by GEA; with a 2.065 year half-life, it was assumed to be decayed to extinction.

The AP-107 samples were first processed through a crossflow and/or dead end filtration system (Geeting et al. 2018). The AP-107 samples were filtered in batches and provided in multiple ~1-L increments for ion exchange. Each container of material was measured for density using 10-mL volumetric flasks or the Coriolis meter installed in the crossflow filter apparatus. Densities ranged from 1.26 to 1.28 g/mL. It was assumed that, within analytical uncertainty, all samples provided for ion exchange processing were equivalent. Due to the large volume (~9 L) of AP-107, the multiple samples were not combined into one single container for homogenization. Doing so builds risk into the process (should a container leak), and handling the massive composite would be problematic given the weight-lifting limitations of the manipulators in the hot cells.

2.3 Batch Contact Conditions

A primary metric of an ion exchanger's performance is its distribution coefficient (K_d) measured in a batch contact experiment. The distribution coefficient is a quantitative measure of a material's capability to remove an ion from solution, and is the ratio of the concentration of the ion sorbed on the ion exchange material to the concentration of the ion remaining in solution at equilibrium. Previous work done on a 5.6 M Na simple simulant was used to obtain fundamental information about the behavior of the CST for use in modeling the equilibrium performance of the material.

Batch contact solutions consisted of the AP-107 tank waste plus various amounts of added ^{133}Cs as $CsNO_3$ solution. The equilibrium Cs concentrations were determined after batch contacts to assess Cs loading capacity on the CST and the Cs K_d. The preparation and batch contacts were processed in accordance with test instruction TI-DFTP-036 (Fiskum et al. 2018b).

Aliquots of Cs spike solutions (140 mg/mL or 14.0 mg/mL) were added to three centrifuge tubes in small volumes (1.77, 10.5, and 50.9 mg added Cs). The Cs-spiked centrifuge tubes were transferred to the hot cell and approximately 33-mL aliquots of filtered AP-107 supernate were transferred to each of the Cs spike solution containers and shaken to mix thoroughly. All Cs spike transfers and AP-107 transfers were tracked by mass and actual volume deliveries calculated based on mass and solution density. Table 2.4 shows the calculated initial Cs concentrations in the batch contact stock solutions. The Cs spike was equilibrated with AP-107 matrix ~10 days.

Table 2.4. Initial Cs Concentrations Used for the AP-107 Tank Waste Batch-Contact Tests

Solution ID	Cs Concentration (mg/L)	Cs Concentration (M)
TI-036-S0	8.57	6.39E-5
TI-036-S1	63.7	4.79E-4
TI-036-S2	330	2.48E-3
TI-036-S3	1579	1.19E-2

The F-factor is the ratio of the dry mass of the exchanger to the initial mass of the exchanger. An aliquot of the washed CST was allowed to air-dry overnight at ambient temperature to a free-flowing form. A small fraction of the air-dried CST was removed for F-factor evaluation. The F-factor sample aliquot was dried at ~100 °C overnight to determine the nominal water content remaining in the partially dried CST.

This nominal F-factor was used to determine the target CST aliquot mass to collect for the batch contact samples. This partially dried CST contained 23% water by mass.

A precisely weighed quantity of the washed and dried CST (targeted to be 0.0972 g wet and 0.075 g dry) was aliquoted into a 20-mL scintillation vial for each batch contact sample. The partially dried CST mass was determined to an accuracy of ≤1%.

Three F-factor samples were also weighed, one at the beginning of CST aliquoting process, one at the end of CST aliquoting process, and one later in the day when a replacement batch contact vial was prepared. The initial mass was designated M_I. The F-factor samples were dried to constant mass at 100 °C. The final mass was designated M_F. The F-factor was calculated according to Eq. (2.1). The average of the two F-factor samples (first and last from series, 0.7484, 0.12% relative percent difference) was used to calculate the dry CST mass contacted with solution, as discussed in Section 2.3.1. The F-factor from the third sample (0.7508) was used to determine the replacement batch contact CST sample F-factor.

$$\frac{M_F}{M_I} = \text{F-Factor} \tag{2.1}$$

The CST aliquots were transferred to the hot cell and then contacted with 15 mL of the various contact solutions (see Table 2.4) in duplicate. The AP-107 volume was transferred by pipet, and the actual volume was determined by mass difference and solution density. The targeted phase ratio (liquid volume to dry exchanger mass) was 200 mL/g CST. The obtained ratio varied between 198 and 206 mL/g CST.

The primary batch contact vials (S0-S3) along with a temperature sentinel vial were placed upright onto a Thermo LP vortex mixer[1] initially set to ~1000 revolutions per minute. Four hours into shaking, the lid from TI036-S0-CST came off and approximately 5 mL of the solution spilled into the sample tray. The other vials and sample tray were rinsed of salt solution with DI water. The lids were taped onto the glass vial to mitigate the chance for loosening. The three remaining primary vials and the S0 duplicate, TI036-S0-CST-d, were added back to the mixer to maintain an evenly distributed weight. The rotation rate was reduced to ~500 revolutions per minute. A new TI036-S0-CST vial was prepared. A new F-factor (F-3) sample was also collected at this time. The re-prepared TI036-S0-CST primary vial was mixed with the duplicates batch at the conclusion of the primary batch contacts.

The first batch of CST samples was contacted for 52 hours, with the exception of TI036-S0-CST-d, which was contacted for 48 hours, and the second batch was contacted for 91 hours. The temperature in the hot cell was nominally 27 °C throughout testing. After mixing for 52 hours, the temperature sentinel was 26.9 °C; after 91 hours of shaking, the temperature sentinel was 27.4 °C. After contact, the CST was settled and ~5 mL of the aqueous fractions were removed from the hot cell and filtered through 0.45-µm pore size nylon-membrane syringe filters.

2.3.1 Batch Contact Analysis and Calculations

Filtered 0.1-mL aliquots were collected and mixed with 1.9-mL of 0.1 M NaOH for GEA to determine the ^{137}Cs concentrations according to ASR 0514. All Cs K_d measurements were determined by measuring

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[1] The Thermo LP vortex mixer was selected for hot cell use because of its small size (15.4 x 21.0 x 8.3 cm) and small mass (3.1 kg).

^{137}Cs on both the initial AP-107 solution (initial concentration C_0) and the contacted solution (final concentration C_1). The Cs batch K_d was determined using the relationship shown in Eq. (2.2):

$$\frac{(C_0 - C_1)}{C_1} \times \frac{V}{M \times F} = K_d \tag{2.2}$$

where C_0 = initial ^{137}Cs concentration (µCi/mL)
C_1 = final (equilibrium) ^{137}Cs concentration (µCi/mL)
V = volume of the batch contact liquid (mL)
M = measured mass CST (g)
F = F-factor, mass of the dried CST divided by the mass of the undried CST
K_d = batch-distribution coefficient (mL/g)

Final (equilibrium) Cs concentrations (Cs_{Eq}) were calculated relative to the ^{137}Cs recovered in the contacted samples (C_1) according to Eq. (2.3):

$$Cs_0 \times \left(\frac{C_1}{C_0}\right) = Cs_{Eq} \tag{2.3}$$

where Cs_0 = initial Cs concentration in solution (µg/mL or M)
C_1 = equilibrium ^{137}Cs concentration in solution (µCi/mL)
C_0 = initial ^{137}Cs concentration in solution (µCi/mL)
Cs_{Eq} = equilibrium Cs concentration in solution (µg/mL or M)

The equilibrium Cs concentrations loaded onto the CST (Cs_{IX} in units of mg Cs per gram of dry CST mass) were calculated according to Eq. (2.4):

$$\frac{Cs_0 \times V \times \left(1 - \frac{C_1}{C_0}\right)}{M \times F \times 1000} = Cs_{IX} \tag{2.4}$$

where Cs_{IX} = equilibrium Cs concentration in the CST (mg Cs/g CST)
Cs_0 = initial Cs concentration in solution (µg/mL)
V = volume of the batch contact liquid (mL)
C_1 = final ^{137}Cs concentration in solution (µCi/mL)
C_0 = initial ^{137}Cs concentration in solution (µCi/mL)
M = mass of CST (g)
F = F-factor, mass of the dried CST divided by the mass of the undried CST
1000 = conversion factor to convert µg to mg

2.4 Ion Exchange Process Testing

This section describes the ion exchange column system and the process conditions. The preparations and column testing were conducted in accordance with TI-DFTP-042.[1]

2.4.1 Ion Exchange Column System

The ion exchange process system has been previously described (Fiskum et al. 2017, 2018b); a system schematic for processing downflow lead column to lag column is reproduced in Figure 2.1. The quick disconnects that were previously available for switching the direction of flow were used to swap out columns during testing as CST is a non-regenerable media. Flow through the system was controlled with a positive displacement fluid metering pump. Lead column samples were collected at valve 2 and lag column samples were collected from valve 3 during the AP-107 loading process. The feed displacement (FD) and water rinse that followed were collected from valve 3.

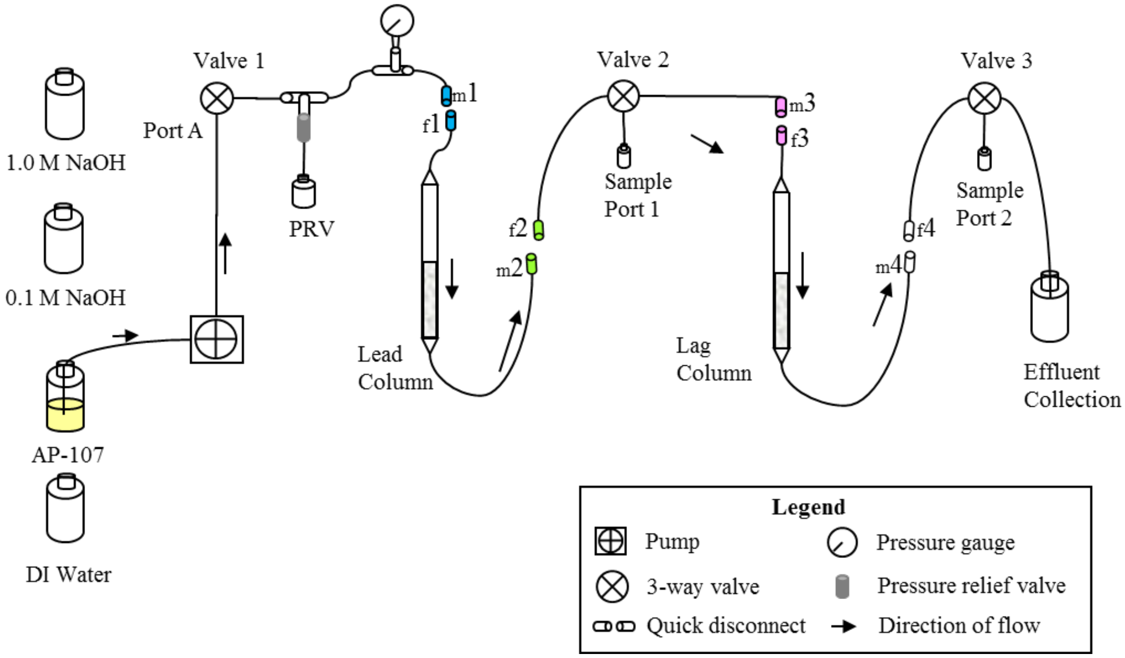

Figure 2.1. Cesium Ion Exchange Process Schematic Showing Downflow Lead-to-Lag Processing

Column assemblies were purchased from Spectrum Chromatography (Houston, TX), part number 125009. The column assembly included the column plus the top and bottom end fittings. Each column was made of borosilicate glass and was 20 cm tall with an inside diameter of 1.44 cm (corresponding to a CST volume of 1.6 mL/cm). Column fittings were composed of polytetrafluoroethylene (PTFE) and Teflon endplates and ferrule fittings for 1/8 in. outside diameter tubing.

As a reminder, the bed volume (BV) corresponded to the initial settled CST media BV as measured in a graduated cylinder prior to transferring the media into the ion exchange column. The reference CST BVs were 10.0 mL for both the lead and lag columns. A photograph of the AP-107 in-cell system 2 months

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[1] Fiskum SK. 2018. TI-DFTP-042, *Cesium Removal from AP-107 Using Crystalline Silicotitanate in Dual-Column Format.* Pacific Northwest National Laboratory, Richland, Washington. Implemented March 2018.

after processing concluded is shown in Figure 2.2. Note the lead column has a dark grey appearance; the darkening increased over time due to radiolytic damage from the ^{137}Cs on the glass column. The CST itself appeared white when it was later removed from the column.

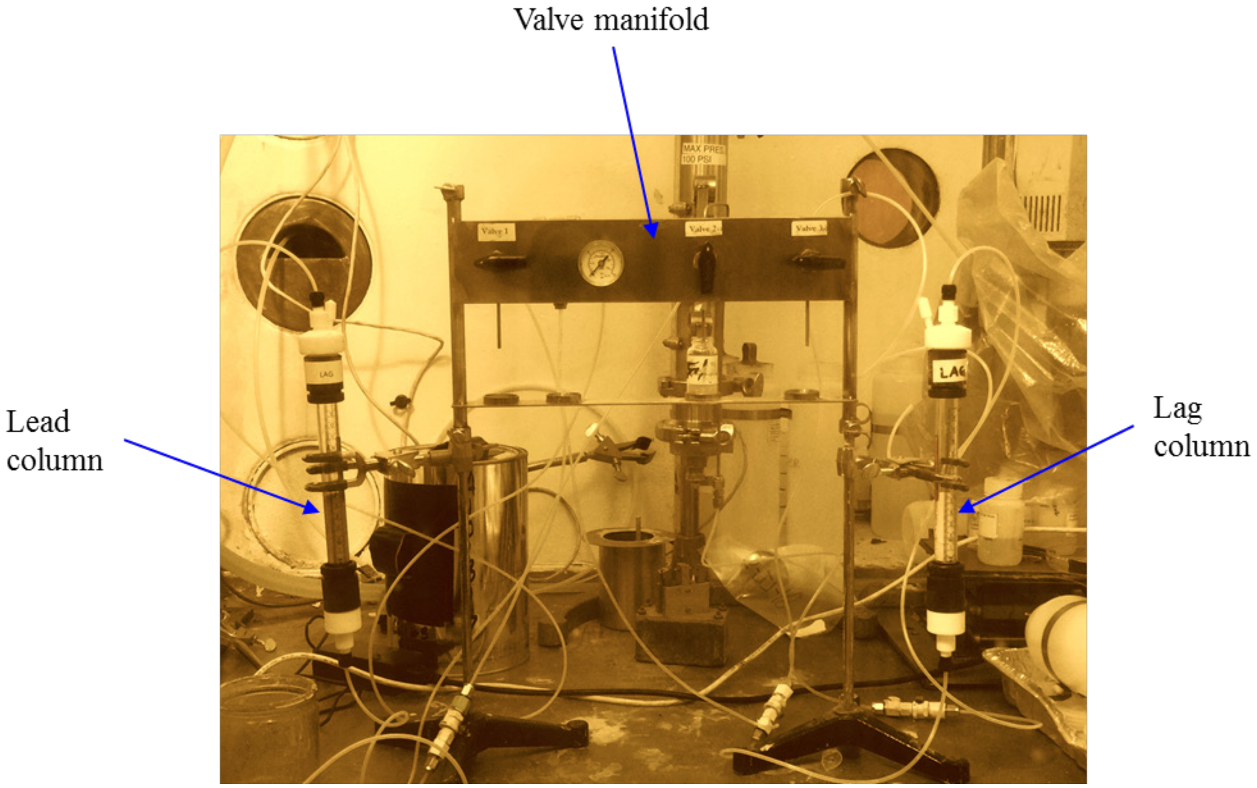

Figure 2.2. Column Assembly in the Hot Cell

2.4.2 AP-107 Tank Waste Process Conditions

A flow of DI water was used to verify the system integrity and calibrate the pump to flows of 0.50 and 0.38 mL/min (3.0 and 2.3 BV/h, respectively). The in-column CST pretreatment flowed 60 mL of 0.2 M NaOH as an initial column flush. The targeted concentration for this initial system flush was 1 M NaOH, but due to a preparation error the concentration did not reach its target. The conversion of the fluidic volume to strong base prevents the potential for Al in the AP-107 to precipitate out as $Al(OH)_3$ upon contact with water in the system. Use of 0.2 M NaOH (as opposed to 1 M NaOH defined in test plan TP-DFTP-029) was sufficient for this transition.

The AP-107 feed was processed through the ion exchange media beds, lead to lag. A series of AP-107 1.5-L feed bottles were strategically processed to allow optimal feed volume management and support unattended, off-shift (graveyard) operation. Effluent was collected in ~1.5-L increments. The volume limitation was intended to minimize impact of lag column Cs breakthrough exceeding the waste acceptance criteria (WAC). After the AP-107 loading, 6 BVs of 0.1 M NaOH FD followed by 6 BVs of DI water rinse were passed through the system to clean any residual feed out of the lines. In contrast to the AP-105 testing (Fiskum et al. 2018b), the flow direction was never changed as CST is non-elutable and led to one continuous process feed.

Halfway through feed processing, the lead column (column 1) reached about 25% Cs C/C_0 breakthrough and the lag column (column 2) effluent began exceeding the WAC. A new lag column (column 3) was prepared, the lead column was drained and removed from service, and the existing lag column was moved to the lead column position. The drained fluid from the original lead column was returned to the feed bottle for processing.

All processing was conducted at ambient cell temperature conditions, nominally 24 to 26 °C. Test parameters, including process volumes, flowrates, and contact times, are summarized in Table 2.5. The total cumulative volume of AP-107 processed was 8.91 L (891 BVs). The AP-107 process cycle mimicked, as best as possible, the process flow anticipated at the LAWPS facility in terms of BV/h and total BVs. It was understood that the feed linear velocity could not be matched in this small column configuration. As previously reported (Fiskum et al. 2018a), increasing the linear velocity decreases the transition zone and sharpens the breakthrough curve. Therefore, the load curves developed from this small-scale system are likely worst-case bounding with respect to the transition zone.

Table 2.5. Experimental Conditions for AP-107 Column Processing, March 2-19, 2018

Process Step	Solution	Volume (BV)	Volume (AV)	Volume (mL)	Flowrate (BV/h)	Flowrate (mL/min)	Duration (h)
Conditioning	0.2 M NaOH	6.0	1.4	60	3.1	0.52	1.9
Loading column 1 (lead position)	AP-107	471	NA	4712	2.2	0.37	213.5
Loading column 2 (lag position)(a)	AP-107	465	NA	4652	2.2	0.37	213.5
Loading column 2 (lead position)	AP-107	420	NA	4199	2.2(b)	0.37	193.0
Loading column 3 (lag position)(a)	AP-107	414	NA	4143	2.2(b)	0.37	193.0
Feed displacement	0.1 M NaOH	5.7	1.3	56.5	3.1	0.51	1.9
Water rinse	DI water	5.7	1.3	56.5	3.0	0.50	1.9

(a) The feed volume through the lag column was reduced relative to that of the lead column because of sampling from the lead column.

(b) Not including last 12 hours of loading, where flowrate was throttled back to 1.36 BV/h to avoid running the column dry overnight.

BV = bed volume (10.0 mL as measured in graduated column).

AV = apparatus volume (nominally 43.4 mL).

NA = not applicable.

During the loading phase, nominal 4-mL samples were collected from both the lead and lag columns at the sample collection ports (see Figure 2.1, valves 2 and 3). Sampling from the lead column necessitated a brief (10-minute) interruption of flow to the lag column. Samples were collected after the first ~10 BVs were processed and again at nominal 20- to 30-BV increments. Selected effluent samples from the lead column were measured for $^{239+240}$Pu, ^{90}Sr, Ba, Cr, and U in an effort to assess the load behavior for these analytes. The feed was processed for nearly 403 hours continuously. Feed displacement and water rinse were collected sequentially in nominal 1-BV increments from valve 3 and 2-mL aliquots of each of the samples were submitted for GEA.

Cesium load performance was determined from the ^{137}Cs in the collected samples relative to the native ^{137}Cs in AP-107 feed. The collected samples were analyzed directly to determine the ^{137}Cs concentration using GEA. Cesium loading breakthrough curves for both the lead and lag columns were generated based on the feed ^{137}Cs concentration (C_0) and the effluent Cs concentration (C) in terms of %C/C_0.

2.5 Sample Analysis

A composite feed sample was prepared by sampling 2 mL from each filtered sample bottle into one glass vial. Duplicate effluent composite samples were generated by collecting a pro-rated volume from each effluent bottle and combining in glass vials.

Table 2.6 summarizes the sample collections and analyses from the testing along with the cross references to ASR and Radiochemical Processing Laboratory sample identifications (IDs). The ASO was responsible for the preparation and analysis of appropriate analytical batch and instrument quality control samples and for providing any additional processing to the sub-samples that might be required (e.g., acid digestion, radiochemical separations, dilutions).

All analyses were conducted by the ASO according to standard operating procedures, the ASO QA Plan, and the ASR. Samples were analyzed directly (no preparation) for ^{137}Cs analysis by GEA.

Table 2.6. Analytical Scope

IX Test	Sample ID	ASR	ASO Sample ID	Analysis Scope
Column	TI042-Comp-Feed	0521.01	18-1604	GEA, ^{3}H, ^{99}Tc, IC, TOC/TIC, free OH, ICP-MS, ICP-OES, total alpha/beta, ^{90}Sr, ^{237}Np, ^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm
	TI042-Comp-Eff		18-1605	GEA, ^{3}H, ^{99}Tc, IC, TOC/TIC, free OH, ICP-MS, ICP-OES, total alpha/beta, ^{90}Sr, ^{237}Np, ^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm
	TI042-Comp-Eff-Dup		18-1606	GEA, ^{3}H, ^{99}Tc, IC, TOC/TIC, free OH, ICP-MS, ICP-OES, total alpha/beta, ^{90}Sr, ^{237}Np, ^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm
Column	TI042-L-F2-A (47 BVs)		18-1607	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F3-A (57 BVs)		18-1608	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F4-A (101 BVs)		18-1609	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F5-A (124 BVs)		18-1610	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F7-A (178 BVs)		18-1611	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F9-A (231 BVs)		18-1612	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F11-A (284 BVs)		18-1613	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F13-A (335 BVs)		18-1614	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F15-A (389 BVs)		18-1615	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
	TI042-L-F18-A (471 BVs)		18-1616	GEA, ICP-OES, ^{90}Sr, ^{238}Pu, $^{239+240}$Pu
Batch Contact	TI036-S0-CST-F		18-1617	GEA, ICP-OES
	TI036-S2-CST-F		18-1618	GEA, ICP-OES
	TI036-S3-CST-F		18-1619	GEA, ICP-OES

IC = ion chromatography; TIC = total inorganic carbon; TOC = total organic carbon.

3.0 Batch Contact Results

This section discusses the batch contact results for the AP-107 filtered tank waste with CST.

3.1 AP-107 Batch Contact Results

Equilibrium Cs concentrations and K_d results for the batch contacts are provided in Table 3.1; the K_d values are plotted versus Cs concentrations in Figure 3.1 on a log-log scale. Note that the primary samples were contacted for 52 hours and the duplicates were contacted for 91 hours, with the exception of TI036-S0-CST-d and TI036-S1-CST-d, which were contacted for 48 hours. There was no real difference in K_d between the 52- and 91-hour contact times, which confirms that the CST equilibrium was reached within 52 hours.

Table 3.1. Equilibrium Results for Batch Contact Samples in AP-107

Sample ID	Initial [Cs] (µg/mL)	Final [Cs] (µg/mL)	Δ Time (h)	K_d (mL/g)	Equilibrium Cs in CST (mg Cs/g)
TI036-S0-CST	8.57	2.1	91	625	1.33
TI036-S1-CST	63.7	13.3	52	758	10.2
TI036-S2-CST	330	123	52	341	42.0
TI036-S3-CST	1580	1281	52	47	60.8
TI036-S0-CST-d	8.57	2.0	48	649	1.33
TI036-S1-CST-d	63.7	13.3	48	754	10.1
TI036-S2-CST-d	330	117	91	368	43.0
TI036-S3-CST-d	1580	1246	91	53	65.9

Note that the AP-107 tank waste also contained 0.097 M K and 0.92 M free hydroxide.

The K_d vs. the log of the Cs equilibrium concentration was fit to a second order polynomial equation to calculate the K_d at the feed concentration of 8.57 µg Cs/mL, 669 mL AP-107/g CST. The theoretical 50% Cs breakthrough on the ion exchange column (λ) can be predicted from the product of the K_d value and the ion exchanger bed density (ρ_b) according to Eq. (3.1). The CST bed density is the dry CST mass divided by the volume in the column. Assuming a constant CST bed density of 1.00 g/mL, the theoretical 50% breakthrough (λ) for AP-107 with 8.57 µg/mL Cs is 669 BVs.

$$K_d \times \rho_b = \lambda \tag{3.1}$$

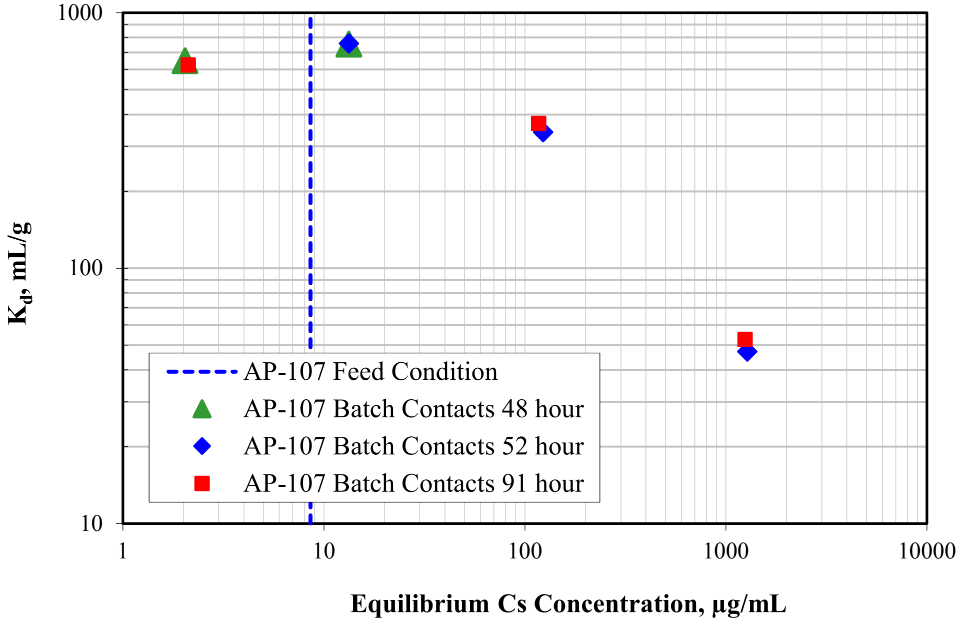

Figure 3.1. Equilibrium Cs K_d Curve for AP-107 with CST

Figure 3.2 compares the K_d values obtained with AP-107 batch contacts with the 24-hour 5.6 M Na simple simulant batch contacts (Fiskum et al. 2018a) and with those reported by Brown et al. (1996), who used CST batch 0739-38B, in two other materials (actual tank waste and 5 M Na simulant tank waste). The double-shell slurry feed (DSSF) tank waste was formulated from a blend of tank wastes (70% from AW-101, 20% from AP-106, and 10% from AP-102) and contained 5.0 M Na, 0.44 M K, and 2.0 M free hydroxide. The simulant DSSF tank waste was similarly constructed to contain 5.0 M Na, 0.475 M K, and 2.17 M free hydroxide. Overall, the K_d values as a function of the equilibrium Cs concentration agreed well between the different test matrices. A couple of exceptions were observed. The DSSF simulant resulted in a higher K_d value (1000 mL/g) at a low Cs concentration (~2 µg/mL) than those found for the other samples (625 to 785 mL/g). Brown et al. (1996) could not determine why the actual and simulant DSSF tank waste results diverged. Also, unlike the other tested matrices, the AP-107 matrix resulted in what appeared to be an apex in K_d values at ~13 µg/mL (756 mL/g) (see also Table 3.1 and Figure 3.2).

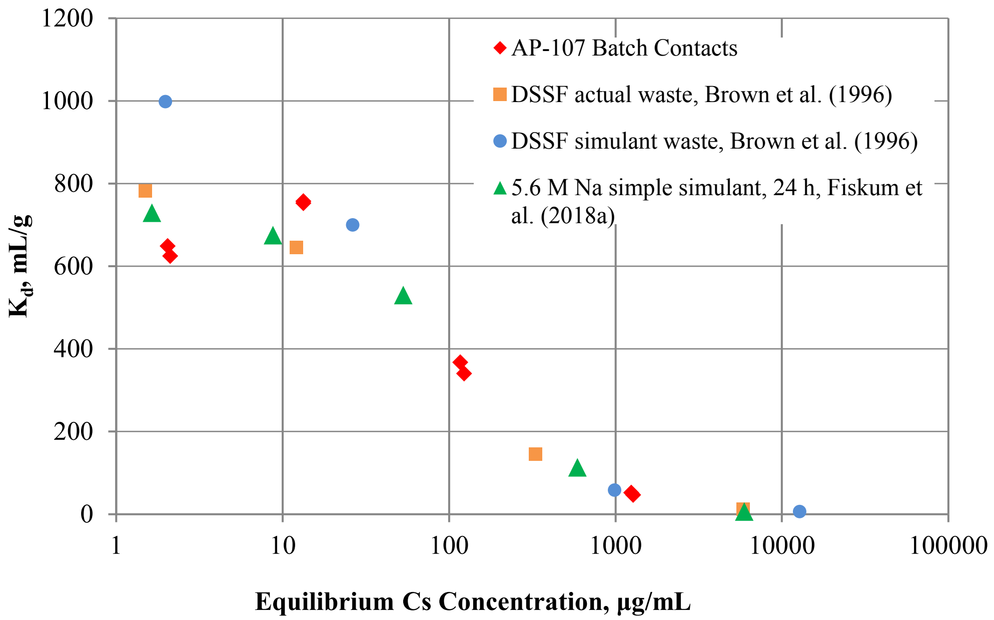

Figure 3.2. Comparison of AP-107 Test Data with 5.6 M Na Simple Simulant (Fiskum et al. 2018a) and Brown et al. (1996) Test Data

Figure 3.3 provides the isotherm for the AP-107 batch contact test samples. In this case, the equilibrium Cs concentration is expressed in terms of molarity (as opposed to µg/mL in Figure 3.1). The isotherm was fit to the Freundlich/Langmuir Hybrid equilibrium isotherm model (see Hamm et al. 2002) according to Eq. (3.2). The expected Cs loading onto the CST at a given Cs concentration can be determined from the isotherm.

$$\frac{a_i \times [\mathrm{Cs}]}{(\beta + [\mathrm{Cs}])} = \mathrm{Cs}_{\mathrm{IX}} \tag{3.2}$$

where [Cs] = equilibrium Cs concentration, mmole Cs per g CST

$\mathrm{Cs}_{\mathrm{IX}}$ = equilibrium Cs loading on the CST, mmole Cs per g CST

a_i = isotherm parameter constant (mmoles/mL)

β = isotherm parameter constant (dimensionless)

The parameter a_i has been computed by Hamm et al. (2002) by Eq. (3.3).

$$a_i = \rho_b \eta_{df} [\mathrm{Cs}] \tag{3.3}$$

An average CST density (ρ_b) of 1 g/mL, a total cesium capacity of 0.58 mmole/g CST for CST powder, and a dilution factor (η_{df}) of 0.68 for CST in its engineered form (to account for the added binder) were assumed. The dilution factor addresses which form of CST is being considered and is set to unity when CST is in its powder form. These values resulted in an a_i value of 0.39 mmoles/mL. The isotherm fit computed from AP-107 batch contact data resulted in an a_i parameter of 0.50 mmole/mL and a β value of 5.3E-4. The AP-107 tank waste a_i value falls between the Hamm calculated a_i value for the engineered form of CST (0.39 mmole/mL) and the CST powder value of 0.58 mmole/mL.

At the equilibrium Cs concentration of 8.57 µg Cs/mL (6.39E-5 M), the equilibrium Cs loading corresponded to 0.054 mmole Cs per g dry CST.

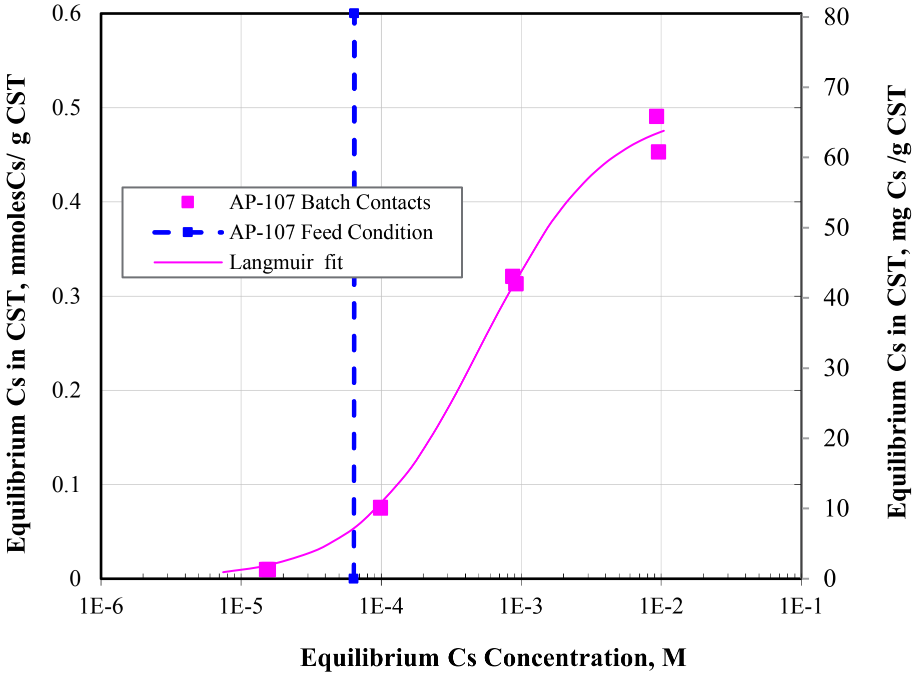

Figure 3.3. Isotherm for AP-107 Tank Waste with CST

4.0 Column Test Results

The Cs load behavior was evaluated with the AP-107 tank waste. This section discusses the load, FD, water rinse, and Cs mass balance results. Raw data are provided in Appendix A.

4.1 Cs Load, Feed Displacement, and Water Rinse Results

The AP-107 feed was processed at nominally 2.2 BV/h through the lead and lag columns. Figure 4.1 shows a linear-linear plot of the cesium load profile for feed processed through each column. The x-axis shows the BVs processed and the y-axis shows the effluent Cs concentration (C) relative to the feed concentration (C_0) in terms of % C/C_0. The C_0 value for ^{137}Cs was determined to be 156.5 µCi/mL. In this graphing layout, the Cs breakthrough from the lead column started at 125 BVs and continued to 470 BVs (a range of 345 BVs). By this point, the lag column effluent exceeded the WAC. The lead column (column 1) was removed from the system, the lag column (column 2) was moved to the lead position, and a new lag column (column 3) was installed, prototypic of proposed plant operations to keep the effluent compliant with the WAC. The Cs breakthrough from column 2 continued to 888 BVs. The column 1 50% Cs breakthrough did not occur due to the switching out of columns. Note that the lag column (columns 2 and 3) Cs breakthrough is not observable at this scale. The circled area in Figure 4.1 shows a leveling in the Cs load profile. This was attributed to the reduced flowrate from 2.2 to 1.4 BV/h to ensure the column would not run dry during unattended operations; the reduced flowrate allowed for greater Cs exchange onto the CST.

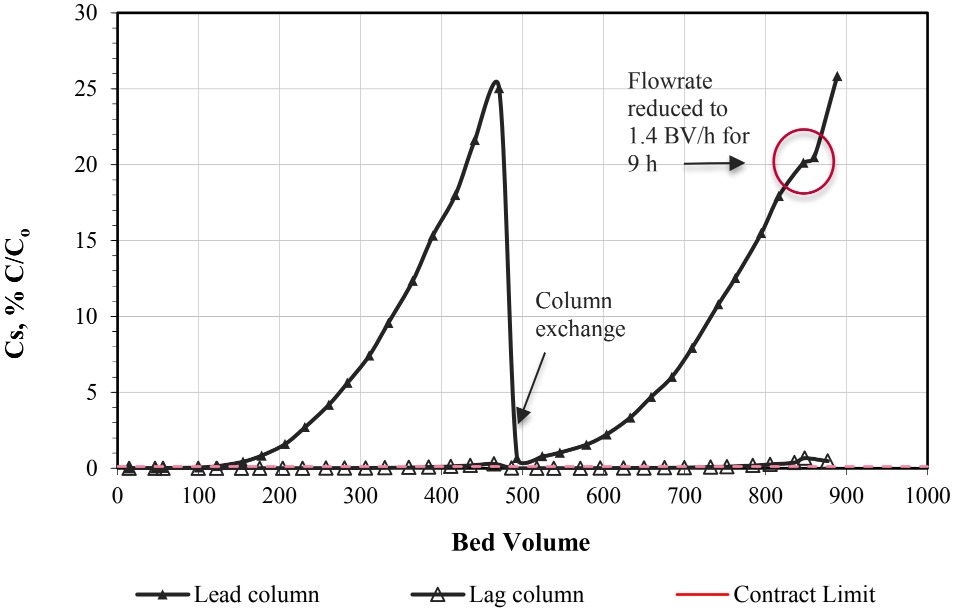

Figure 4.1. Lead and Lag Column Cs Load Profiles of AP-107 at 2.2 BV/h, Linear-Linear Plot

Figure 4.2 shows the same Cs load data provided in Figure 4.1, but with the ordinate % C/C_0 on a probability scale, as this scale tends to provide a relatively straight-line breakthrough curve under ideal load conditions and provides greater fidelity of load characteristics at low and high % C/C_0 values. In contrast to Figure 4.1, the Cs breakthrough from the lead column was observed nearly immediately (<57 BVs). Also provided are the FD and water rinse results following AP-107 loading and the WAC at 0.114 % C/C_0.[1] In this configuration, the WAC Cs breakthrough for the first cycle lead column (column 1) occurred at about 115 BVs, and 410 BVs for the lag (column 2). The second cycle lag (column 3) WAC Cs breakthrough occurred around 285 BVs after column change out (750 BVs total). The net BVs to WAC limit are different between these two columns because the second cycle lead column (column 2) was already partially loaded with Cs; thus, the second cycle lag column loaded faster than the first cycle lag column. Also worth noting, the column 2 Cs load curve in lag position matched the load curve almost exactly to when it was moved into the lead column position. This is interesting because the feed Cs to this column changed from 25% to 100% C/C_0 and is contrary to Walker et al. (1998, Fig. 30), where a significant step change was observed. It's also observed that the Cs load characteristics of the lead and lag columns did not mirror each other. There is significant curvature in the lead column (column 1) load profile and much less curvature in the lag column (column 2) load profile. This was also seen with the 5.6 M Na simulant tests conducted with CST by Fiskum et al. (2018a).

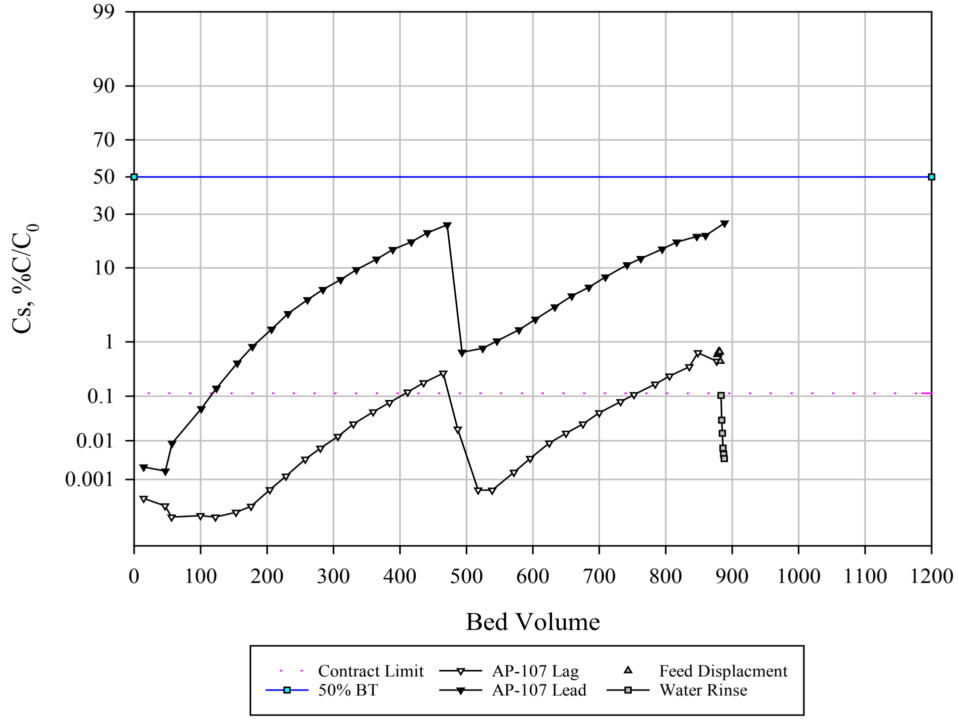

Figure 4.2. Lead and Lag Column Cs Load Profiles of AP-107 at 2.2 BV/h, Probability Plot

The first three BVs of FD solution continued the Cs breakthrough profile, as was expected because AP-107 was still in the system. By the fourth BV of FD and throughout the water rinse, the Cs concentration in the effluent dropped precipitously, which indicated the Cs was remaining firmly on the CST.

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[1] The contract limit was derived from the allowed curies of ^{137}Cs per mole of Na in the effluent to support contact handling of the final vitrified waste form—3.18E-5 Ci ^{137}Cs/mole Na. At 5.6 M Na and 156.5 µCi ^{137}Cs/mL in the feed, the contract limit is 1.14E-3 C/C_0; 0.114% C/C_0.

Table 4.1 provides the Cs-decontaminated effluent composite results in terms of ^{137}Cs concentration and overall decontamination factor (DF). A DF of 876 was needed to meet the WAC. Three of the effluent composites exceeded the WAC and require further column processing to remove additional Cs in support of follow-on vitrification work (not reported herein).

Table 4.1. AP-107 Effluent Composites ^{137}Cs Content and Decontamination Factor

Effluent Container	^{137}Cs (µCi/mL)	Decontamination Factor
Effluent-1 (0-115 BVs)	1.53E-4	1,024,049
Effluent-2 (116-261 BVs)	1.22E-3	128,606
Effluent-3 (262-389 BVs)	4.44E-2	3,529
Effluent-4 (390-471 BVs)	**2.73E-1**	**574**
Effluent-5 (472-579 BVs)	7.65E-3	20,458
Effluent-6 (580-742 BVs)	4.28E-2	3,661
Effluent-7 (743-847 BVs)	**3.17E-1**	**494**
Effluent-8 (848-889 BVs)	**7.00E-1**	**224**

Bolded effluents were below the 876 DF (the waste acceptance criteria).

4.2 Cesium Activity Balance

The ^{137}Cs fractionation was determined between the microCuries of ^{137}Cs calculated to be loaded onto the lead and lag columns and the effluents, samples collected during the load processing, FD, and water rinse. The quantities of Cs loaded onto the lead and lag columns were determined by subtracting the Cs recovered in the samples and effluents from the Cs fed to the column. Table 4.2 summarizes the ^{137}Cs fractions found in the various effluents as well as the calculated ^{137}Cs column loading. About 50% of the total Cs loaded onto column 1, 47% loaded onto column 2, and 4% loaded onto column 3. Sample and effluent collection amounted to less than 1% of the input Cs.

Table 4.2. ^{137}Cs Activity Balance for AP-107

Input	µCi	%
Feed sample	1.37E+06	100
Output		
Effluent-1 (0-115 BVs)	0.18	1.29E-05
Effluent-2 (116-261 BVs)	1.68	1.22E-04
Effluent-3 (262-389 BVs)	55.3	0.004
Effluent-4 (390-471 BVs)	218	0.016
Effluent-5 (472-579 BVs)	9.5	6.92E-04
Effluent-6 (580-742 BVs)	62.1	0.005
Effluent-7 (743-847 BVs)	309	0.023
Effluent-8 (848-889 BVs)	295	0.022
Load samples	1380	0.101
Feed displacement and water rinse	57.6	0.004
Total ^{137}Cs recovery	2,388	0.174
Total ^{137}Cs Column Loading		
Column 1 (Initial lead column Cs loading)	6.81E+05	49.7
Column 2 (Lag/Lead column Cs loading)	6.41E+05	46.8
Column 3 (Final lag column Cs loading)	5.83E+04	4.3
Column total	1.38E+06	100.7

The total Cs loaded per gram CST was calculated from the total Cs loaded onto the lead column, which was not assumed to be fully saturated under these load conditions, and the dry CST mass loaded into the lead column according to Eq. (4.1).

$$\frac{A_{Cs} \times CF}{M} = C \tag{4.1}$$

where A_{Cs} = activity of ^{137}Cs, µCi on the lead column
CF = conversion factor, µg Cs/µCi ^{137}Cs
M = mass of dry CST (9.7919 g)
C = capacity, mg Cs/g CST

The total Cs loaded was found to be 3.80 mg Cs/g CST (0.0283 mmoles Cs/g CST). This is ~50% less than the capacity found from batch contact testing with AP-107; however, this is expected because only ~50% of the available capacity was used due to early breakthrough.

4.3 Predicted 50% Cs Breakthrough

The 50% Cs breakthrough did not occur because column switching was required at ~25% Cs breakthrough for the effluent to meet the WAC. The 50% Cs breakthrough was estimated to compare with the Cs λ value predicted from batch-contact studies. The lead column load curve from 100 BVs to the column switch (470 BVs) and the next segment (500 to 888 BVs) were fit using second order polynomials to extrapolate the data to the 50% Cs breakthrough (shown on dashed lines in Figure 4.3). The column 2 load curve was adjusted 390 BVs to the left. This is the difference in BVs between where the two loading curves start and allows renormalization of the new lead column to the original lead column. The two 50% Cs breakthrough estimates were 615 and 664 BVs, respectively. Averaging the two values results in an extrapolated 50% Cs breakthrough value of 640 BVs. Batch contact testing cannot be used to determine the slope of the Cs load curve. Thus, it cannot predict the contract limit breakthrough in terms of BVs; however, recall the K_d vs. the log of the Cs equilibrium concentration was curve-fitted to calculate the K_d at the feed concentration during batch contact studies and resulted in 669 mL/g. The extrapolated column loading 50% Cs breakthrough value agreed within 4% of the batch contact results. This indicated batch contact testing was a good indicator of column performance. However, the Cs exchange onto CST is particle diffusion limited and the contact time with CST was not sufficient to reach equilibrium Cs loading before the lag column effluent exceeded the contract limit.

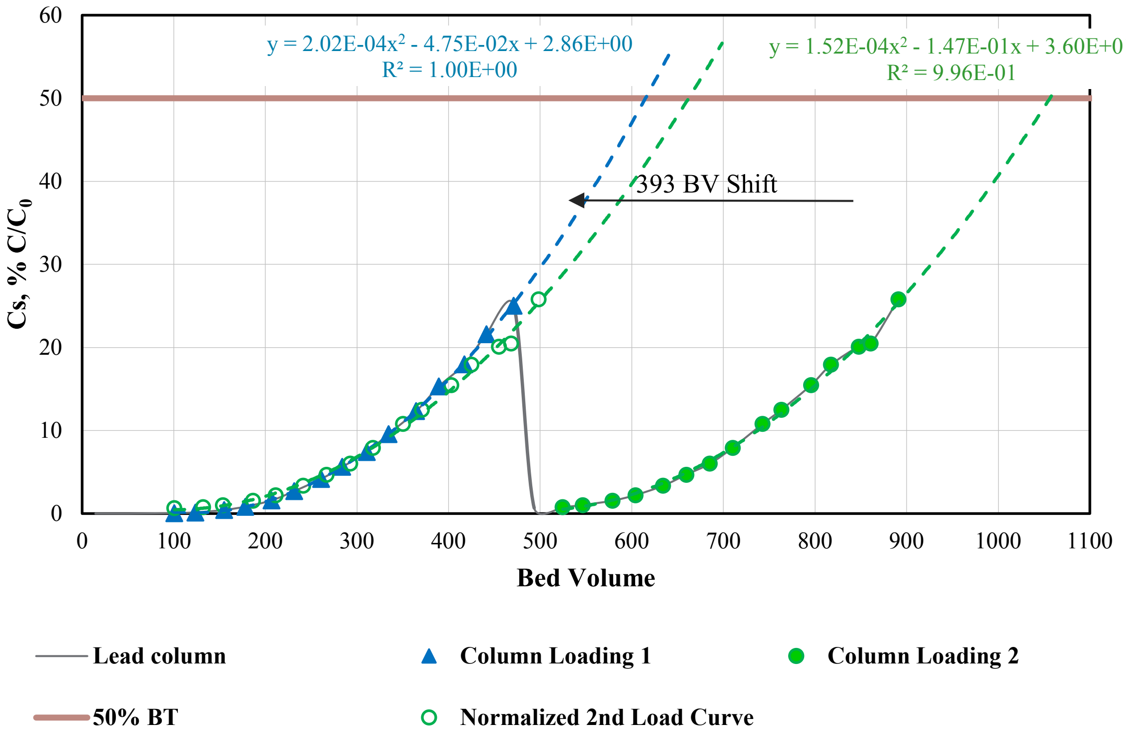

Figure 4.3. Extrapolated Cs Load Profiles

Figure 4.4 shows the lead and lag column Cs load profiles for the AP-107 and 5.6 M Na simple simulant column testing conducted by Fiskum et al. (2018a). During the simulant testing, three flowrates were conducted to examine the effect of flowrate on Cs load performance (4.56 BV/h for the Green test, 1.99 BV/h for the Blue test, and 1.19 BV/h for the Red test). Examining the breakthrough curves shows that increasing flowrate decreases the breakthrough slope and thus increases the mass transfer zone. Processing at 2.2 BV/h initially followed the trends seen with the simulant testing and Cs breakthrough curves remained between the Green and Blue simulant column runs, nearly fitting the load curve between the Green and Blue runs. However, early termination of the lead column processing only allowed side-by-side comparison up to 450 BVs. An alternative estimation of the 50% breakthrough can be performed using Figure 4.4. The 50% breakthrough for AP-107 can be inferred by bounding it between the Green and Blue simulant test 50% breakthrough points. In doing so, the breakthrough point would have been between 700 and 800 BVs which is slightly above the extrapolated value of 640 BVs for AP-107 tank waste. However, this is just an estimate and it is important to note that if Sr, Ca, Pb, etc. are high enough, the Cs exchange sites may be consumed by those elements and change the Cs load profile. In this case, it appeared that the early Cs load profile (50 to 200 BVs) matched that of the Blue simulant test closely, but seemed to diverge in the last half of the test (200 to 450 BVs).

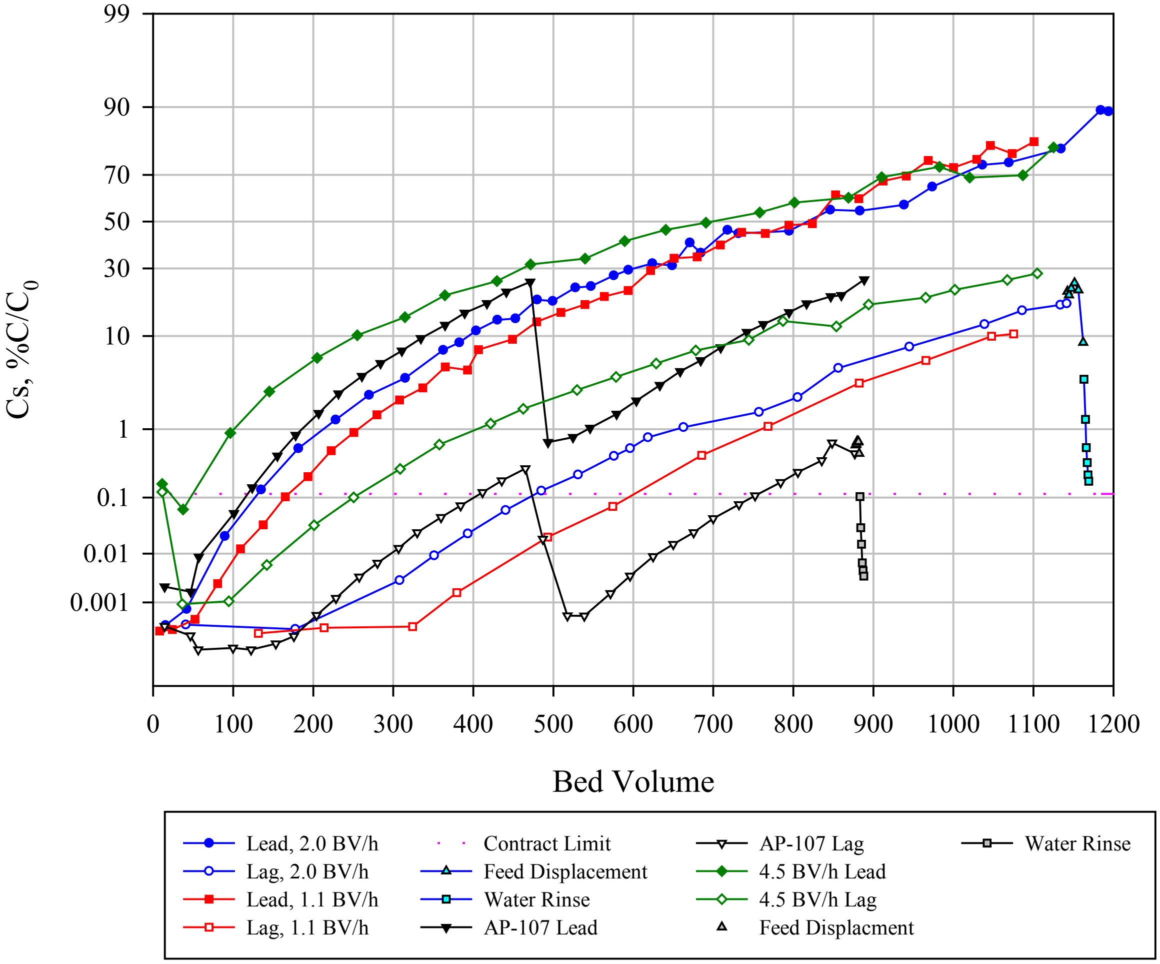

Figure 4.4. Lead and Lag Column Simulant (Fiskum et al. 2018) and AP-107 Tank Waste Cs Load Comparison

4.4 Contract Limit

The flowrate effect on the Cs exchange into CST was further examined by looking at the test lead columns individually and the combined lead/lag columns (as a combined system) for both the AP-107 and 5.6 M Na simulant data (Fiskum et al. 2018a). As previously described (Fiskum et al. 2018a), the combined system constitutes the lead/lag column volumes as one unit (20 mL CST bed total). This, in effect, halved the flowrate; for example, a 1.19-BV/h flowrate in a 10-mL CST bed would be halved to 0.60 BV/h in a 20-mL CST BV system. The BVs for the lag column 10% contract limits were halved.

This allowed for theoretical lead column data to be modeled at a greater number of flowrates. Table 4.3 shows the column systems, flowrates, and the BVs processed to reach the contract limit inclusive of simulant data (Fiskum et al. 2018a) and the AP-107 data. Figure 4.5 shows the relationship of the processed volume as a function of flowrate before reaching the contract limit. In this case, the data were fit to a logarithmic curve. The AP-107 data were comparable to the simulant data and help verify the legitimacy of the simulant performance in a dual column run. The current LAWPS design basis assumes 400 BVs are processed before reaching contract limit. The curve was backward-extrapolated to estimate the flowrate needed to reach this system volume and was found to be 0.21 BV/h; however, this flowrate was beyond the test range and would need to be confirmed with actual testing.

Table 4.3. Bed Volumes Processed to Reach Contract Limit

Test	Flowrate (BV/h)	BVs to Contract Limit
Simulant - Red lead column	1.19	162
Simulant - Blue lead column	1.99	129
AP-107 lead column	2.24	120
Simulant - Green lead column	4.56	48
Simulant - Red lead & lag columns	0.60	294
Simulant - Blue lead & lag columns	1.00	240
AP-107 lead & lag columns	1.12	200
Simulant - Green lead & lag columns	2.28	120

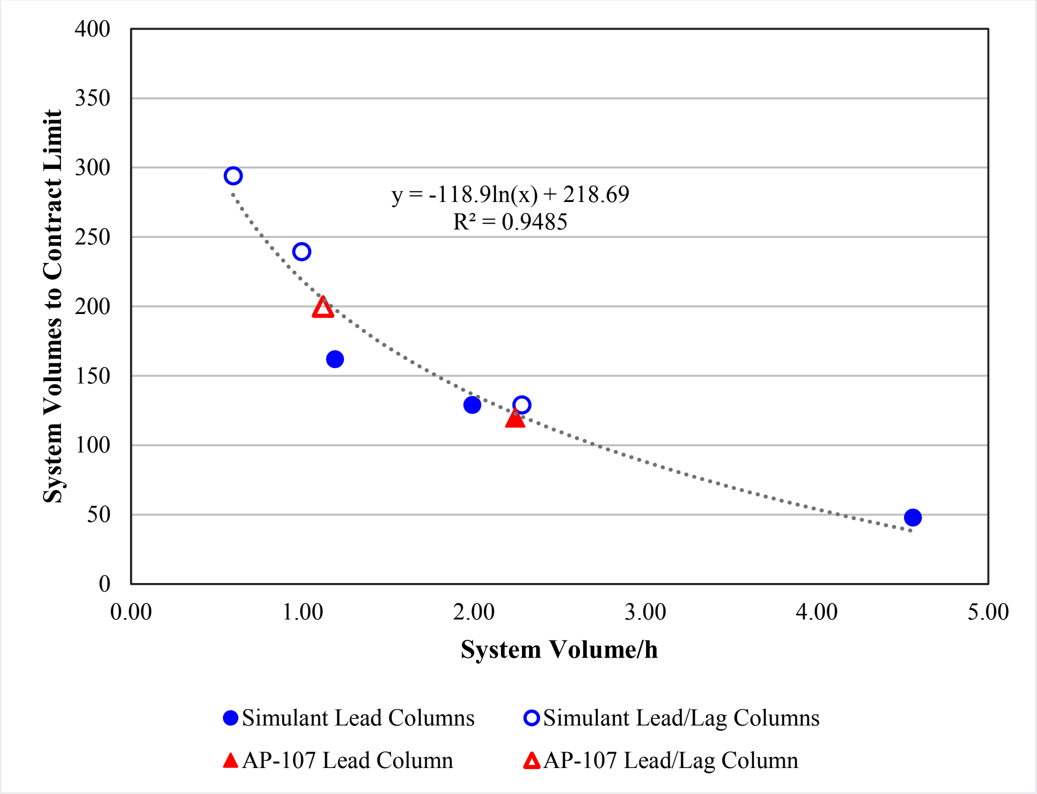

Figure 4.5. Volume Processed to Reach Contract Limit vs. Flowrate

4.5 Transition Zone

The transition (or exchange) zone is defined as the volume processed from the onset of Cs breakthrough to the full saturation of the ion exchanger where the effluent Cs concentration equals the influent Cs concentration. The 50% Cs breakthrough point is the inflection point around which the transition zone pivots. In the current study, the lead column was only loaded to 25% Cs breakthrough. The 50% breakthrough was modeled using extrapolation of the data as well as estimated using simulant data at bounding flowrates in Section 4.3. The 50% Cs breakthrough found for both these methods were 640 BVs and ranged between 700 and 800 BVs. Both methods have high associated errors and would need to be confirmed with additional testing.

5.0 Chemical and Radiochemical Composition

The AP-107 composite feed and composite effluent samples underwent extensive characterization to better define waste characteristics and assess analyte fractionation to the CST. Selected batch contact and lead column samples were analyzed to assess selected metal analyte load characteristics. The extensive characterization also helped support follow-on vitrification glass formulation.

Table 5.1 summarizes the feed and effluent radioisotope concentrations and fractionations to the effluent and Table 5.2 summarizes the feed and effluent metals, anions, inorganic and organic carbon concentrations, and fractionations to the effluent. By inference, the analytes not found in the effluent were assumed to be retained on the CST. Analyte fractionation was calculated as the ratio of the total analyte measured in the feed processed through the columns and the total analyte collected in the Cs-decontaminated effluent according to Eq. (5.1):

$$\frac{C_{Da} \times V_D}{C_{Fa} \times V_F} = F_{Da} \tag{5.1}$$

where C_{Da} = concentration of analyte *a* in the Cs-decontaminated effluent

V_D = volume of Cs-decontaminated effluent (8662 mL)

C_{Fa} = concentration of analyte *a* in the AP-107 feed

V_F = volume of AP-107 feed (8752 mL)

F_{Da} = fraction of analyte *a* in the Cs-decontaminated effluent

Some analyte results are shown in brackets; this indicates that the analytical result was less than the estimated quantitation limit (EQL) but greater than the method detection limit (MDL) and the associated analytical uncertainty could be higher than ±15%. The fractionation result was placed in brackets where it was calculated with one or more bracketed analytical values to highlight the higher uncertainty. The opportunistic analyte results measured by ICP-OES are also shown in Table 5.2; these analytes are part of the ICP-OES data output but have not been fully evaluated for quality control performance. The composite feed sample results in Table 5.1 and Table 5.2 were compared to the initial characterization sample results (Table 2.2); results agreed within analytical uncertainty.

Note that the total volume of AP-107 feed was slightly higher than the AP-107 effluent; the volume difference was associated with samples removed from the lead and lag columns and potential evaporation. The volume difference was ~1%; this has a very small effect on understanding the analyte fractionation.

Table 5.1. AP-107 Feed and Effluent ASR 0521.01, Radionuclides

Analysis Method	Analyte	TI042-Comp-Feed (µCi/mL)	TI042-Comp-Eff (µCi/mL)	Fraction in Effluent (%)
Gamma energy analysis (GEA)[(a)]	^{60}Co	<1.9E-3	5.92E-04	--
	^{137}Cs	1.49E+2	1.04E-01	0.04%
	^{154}Eu	<8.4E-3	3.49E-05	--
	^{241}Am	<4.1E-1	3.83E-04	--
Separations/ Alpha energy analysis (AEA)[(a)]	^{237}Np	3.93E-05	1.23E-05	30.8%
	^{238}Pu	1.14E-04	2.90E-05	25.2%
	$^{239+240}$Pu	5.43E-04	1.90E-04	34.6%
	^{241}Am	4.17E-04	3.86E-04	91.6%
	^{242}Cm	--	[1.15E-06]	--
	$^{243+244}$Cm	1.43E-05	1.68E-05	116%
Separations/ Beta counting[(a)]	^{90}Sr	5.81E-01	2.72E-04	0.05%
	^{99}Tc	9.20E-02	8.90E-02	95.7%
ICP-MS[(b)]	^{126}Sn	1.76E-01	1.74E-01	98.9%
	^{238}U	2.07E+01	1.08E+01	52.3%
Calculated from AEA results[(a)]	Sum of alpha[(c)]	1.13E-03	6.35E-04	55.7%
Proportional counting[(a)]	Total beta	1.50E+02	1.42E-01	0.09%

(a) Reference date is May 18, 2018.
(b) Reference date is July, 2018.
(c) This is the summation of alpha-emitting isotopes concentrations (Am, Cm, Np, and Pu isotopes) as measured by AEA.
"--" = not applicable; value not reported or fractionation cannot be calculated with a less-than value.

Table 5.2. AP-107 Feed and Effluent Compositions ASR 0521.01, Inorganic and Carbon Analytes

Analysis Method	Analyte	TI042-Comp-Feed (µg/mL)	TI042-Comp-Eff (µg/mL)	Fraction in Effluent (%)
ICP-MS	^{202}Hg	--	--	--
ICP-OES	Ag$^{(a)}$	--	--	--
	Al	9,850	9550	96%
	As	[11]	--	--
	B	34.6	29.1	83%
	Ba	0.836	[0.14]	[16%]
	Ca	34.1	17.0	49%
	Cd	6.6	5.1	77%
	Cr	498	480	95%
	Fe	19.4	12.7	65%
	K	3,910	3,800	96%
	Li	--	--	--
	Na	126,000	126,000	100%
	Ni	21.0	20.8	98%
	P	642	640	99%
	Pb	[9.0]	--	--
	Se	--	--	--
	Sr	0.331	[0.04]	[12%]
	Th	[12]	--	--
	Ti	[0.8]	[0.6]	[77%]
	U (total)	[48]	[13]	[26%]
	Zn	[1.8]	[1.3]	[69%]
	Zr	[1.4]	[1.5]	[104%]
IC	Cl^-	2,520	2,725	107%
	NO_2^-	52,600	56,000	105%
	SO_4^{2-}	1,530	1,840	119%
	$C_2O_4^{2-}$	460	500	108%
	NO_3^-	106,000	114,500	107%
	PO_4^{3-}	1,570	1,530	96%
Titration	Free Hydroxide	0.99 M	1.01 M	102%
Hot persulfate oxidation	TOC	2,290	2,310	100%
	TIC	7,630	7,970	103%

Table 5.2 (cont.)

Analysis Method	Analyte	TI042-Comp-Feed µg/mL	TI042-Comp-Eff µg/mL	Fraction in Effluent %
ICP-OES opportunistic analytes	Be	0.192	[0.11]	[58%]
	Bi	[7.4]	--	--
	Ce	--	--	--
	Co	--	--	--
	Cu	[0.84]	--	--
	Dy	[1.1]	--	--
	Eu	[1.2]	[0.19]	[16%]
	La	[1.0]	--	--
	Mg	6.85	--	--
	Mn	--	--	--
	Mo	43.0	41.3	95%
	Nd	--	--	--
	Pd	[8.4]	[3.3]	[39%]
	Rh	[6.1]	[5.3]	[85%]
	Ru	[12]	[8.8]	[73%]
	S	1,670	1,680	101%
	Sb	--	--	--
	Si	86.0	65.5	75%
	Sn	[21]	[7.2]	[34%]
	Ta	--	--	--
	Te	[6.1]	[5.6]	[91%]
	Tl	--	--	--
	V	[1.3]	[1.2]	[91%]
	W	68.6	66.7	96%
	Y	[0.39]	[0.09]	[23%]

Bracketed values indicate the associated sample results were less than the EQL but greater than the MDL. Analytical uncertainty for these analytes is > ±15%.
"--" = not detected or not applicable.

The CST appeared to sorb many transuranic elements. A large amount of the Pu was assumed to have partitioned to the CST (~70%), with 25% to 35% found in the effluent. The ^{237}Np results were similar to the Pu results where 31% of the processed ^{237}Np was accounted for in the effluent, resulting in 69% remaining on the CST. About 50% of the U fractionated to the CST. The Am and Cm were exceptions. The $^{243+244}$Cm chemistry behavior normally follows that of ^{241}Am; therefore, the $^{243+244}$Cm quantitative recovery (116%) in the effluent was complementary with the 92% recovery for ^{241}Am in the effluent.

Nominally all tritium reported to the effluent product. Virtually all ^{90}Sr remained on the CST (although analysis uncertainty was 12%). The ^{99}Tc recovered in the effluent product at 96%.

Nominally 100% of all anions were accounted for in the effluent. TIC is generally ascribed to carbonate. TOC includes oxalate and other organic carbon forms, usually complexants. The oxalate measured by IC, 0.01 M, was a small fraction of the TOC (0.19 M as C). The TOC recovery in the effluent was quantitative at 100%.

The feed composite, effluent composite, selected lead column 1 effluent samples, and three batch contact samples were also analyzed by ICP-OES. The results for metals showed that the majority of analytes remained in the effluent. (See Table 5.2 and Appendix B for analytical reports.) Al, Cr, K, Na, Ni, and P partitioned almost exclusively to the effluent. Pb and As were detected in the feed (with high errors likely exceeding 15%) but were below the MDL in the effluent composite. Ba, Ca, Sr, and U were also partially removed by the CST with only small percentages reported in the effluent.

The load behaviors of selected load samples were examined (Figure 5.1 to Figure 5.4) from samples collected from the lead column (47 to 471 BVs). Consistent with the Cs breakthrough load profile, the Pu breakthrough increased by 20% to 30% from 47 BVs of loading to 470 BVs. In this same loading interval, ^{137}Cs breakthrough increased by 26%. Figure 5.1 compares the Pu breakthrough (measured from ^{238}Pu and $^{239+240}$Pu) relative to Cs breakthrough. Figure 5.2 shows the Sr, Ti, and Zr breakthrough results. From ICP-OES results (high uncertainty), a nearly consistent 13% of Sr was removed by the CST. In contrast, ^{90}Sr C/C_0 was consistently below 1% (with the exception of the last sample analyzed at 471 BVs where 1.3% ^{90}Sr C/C_0 was measured). Based on ^{90}Sr results, a nominal Sr DF of ~700 was obtained for the first 178 BVs processed through the lead column. The Sr DF through the both the lead and lag columns was 2162 (based on the composite effluent result). Understanding the outcome of Zr and Ti was more complex as they were also CST components. The feed and effluent Zr and Ti concentrations were fundamentally equivalent, yet there was evidence of decreasing Zr and Ti concentrations in the lead column effluent samples. This indicated that the CST did not add Ti and Zr to the effluent and thus the CST was not chemically "decomposing."

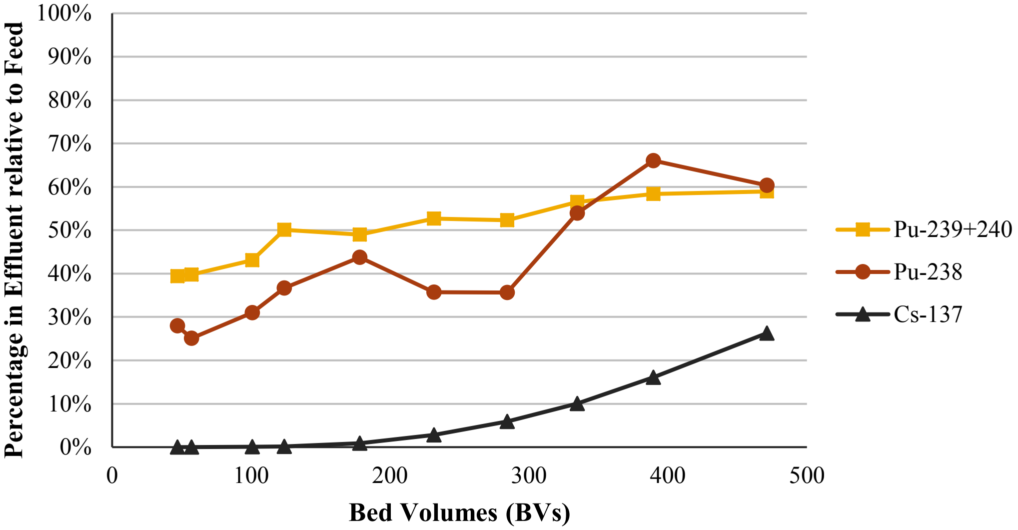

Figure 5.1. Increased Selected Radionuclide Percentage with Loading

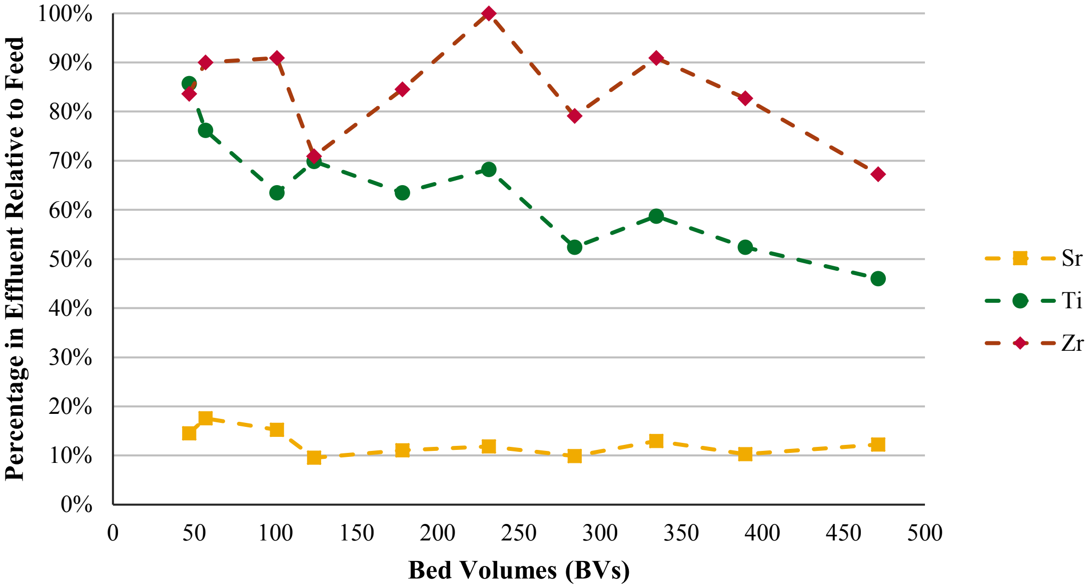

Figure 5.2. Decreased Selected Analyte Percentage with Loading
Dashed lines indicate data were ≥ MDL but < EQL, with errors likely to exceed 15%.

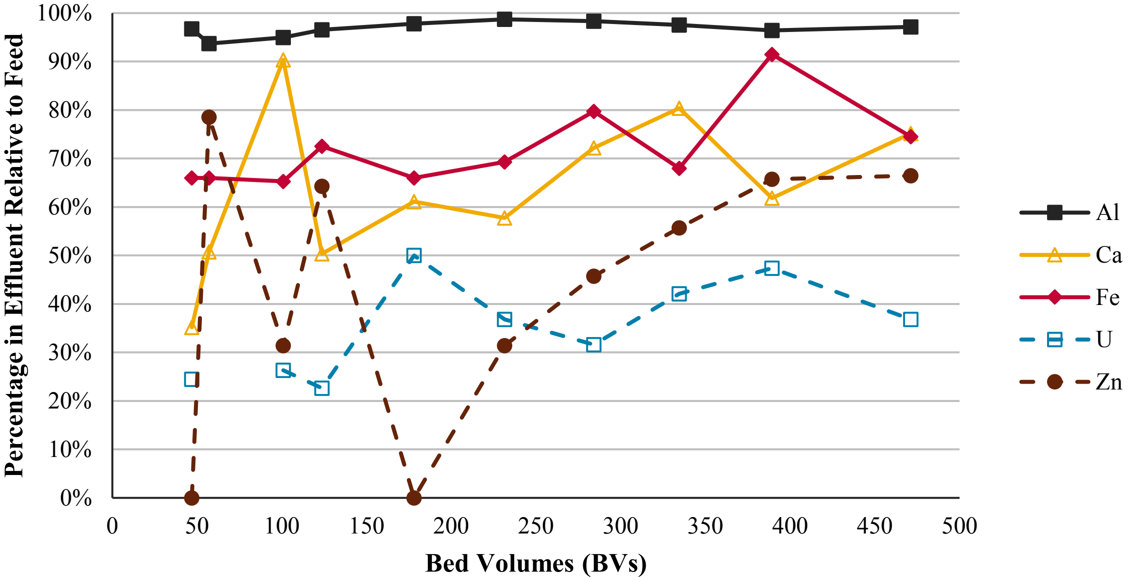

Figure 5.3. Increased or Erratic Selected Analyte Percentage with Loading
Analytes with dashed lines indicate data were ≥ MDL but < EQL, with errors likely to exceed 15%.

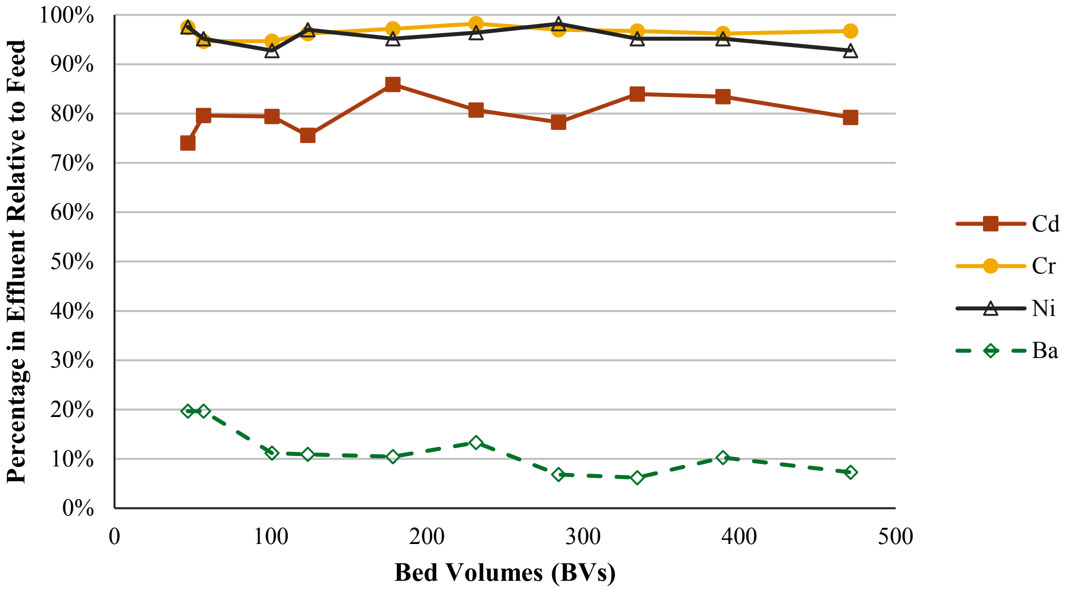

Figure 5.4. RCRA Element Percentages with Loading
Analytes with dashed line indicate data were ≥ MDL but < EQL, with errors likely to exceed 15%.

Analytes that showed increased or erratic percentages in the effluent with loading are shown in Figure 5.3. Zn (high analytical uncertainty) increased by 60% from 47 BVs of loading to 470 BVs. Ca and Fe appeared to manifest limited breakthrough profiles. U showed more variation in breakthrough but was fairly consistent around 35%. In contrast, the Al consistently recovered ~97%.

Detected Resource Conservation and Recovery Act (RCRA) elements were examined and are graphed in Figure 5.4. There seemed to be relatively no difference in any of the analyte percentages in the effluent with increased loading. Pb and As were detected in the feed (with errors likely to exceed 15%), but were not detected in the lead column effluent samples. Ba showed a slight decrease from 20% breakthrough to 7% breakthrough during loading; however, like Sr analysis by ICP-OES, the Ba result uncertainties are high. Cr and Ni, like Al, reported solely to the effluent. Cd averaged 80% recovery in the effluent over the loading duration. Data in Figure 5.1 through Figure 5.4 are shown in tabular form in Appendix C.

Batch contact samples at three different cesium concentrations (1.58E-05, 9.27E-04, and 9.64E-03 M Cs) were also analyzed to assess their exchange onto CST with increasing Cs concentration. Nominally all analyte exchange remained constant throughout the increase in Cs concentrations, with the exception of Ba, Ca, and Fe, which showed increases with increased Cs concentration. An increase in Ba percentage with increased Cs concentration contradicts what was found with column testing; however, both data sets had high errors associated with feed and sample concentrations. An increase in Ca and Fe was consistent with what was seen in column testing and could be attributed to Cs occupying iron and calcium exchange sites on the CST; as Cs loading increased, Cs appeared to displace these analytes. A graph of this information is shown in Figure 5.5.

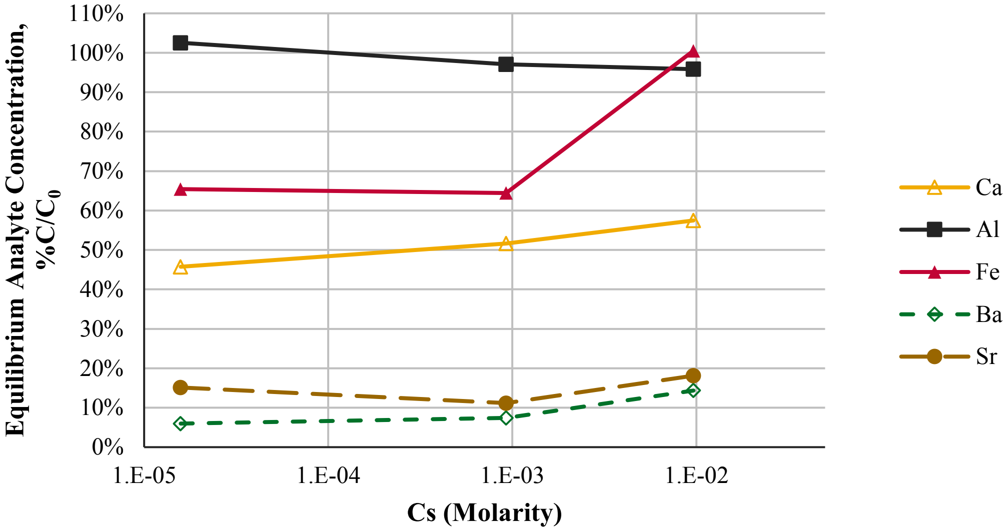

Figure 5.5. Cs Molarity vs. Equilibrium Analyte Concentration
Analytes with dashed line indicate data were ≥ MDL but < EQL, with errors likely to exceed 15%.

6.0 Conclusions

The objectives for CST testing were met: 1) conduct AP-107 tank waste testing to evaluate Cs load behavior on small column CST beds, 2) conduct batch contact testing with CST to determine the AP-107 Cs K_d factor and load capacity, and 3) decontaminate 9 L of AP-107 tank waste in preparation for vitrification processing. The AP-107 feed, column processing effluent, and batch contacts underwent characterization to better define waste characteristics and assess fractionation to the CST.

6.1 Batch Contact Testing

Batch contact testing with CST was conducted to determine the AP-107 Cs K_d values and load capacity. The following conclusions were made as a result of this work.

1. The calculated ^{137}Cs K_d of 669 mL AP-107/g CST at Cs equilibrium condition of 8.57 µg Cs/mL corresponded to a predicted 50% Cs breakthrough of 669 BVs. Although 50% Cs breakthrough was not achieved with column testing, this value was modeled using extrapolation and matched batch contact results within 6%.

2. The Cs load capacity at 8.57 µg Cs/mL equilibrium condition was 7.5 mg Cs/g dry CST. This value was over 50% higher than the column test results, but cannot be accurately compared because the column was not loaded to full capacity.

Batch contact testing cannot be used to determine the slope of the Cs load curve. Thus, it cannot predict the contract limit breakthrough in terms of BVs.

6.2 Column Testing

The ion exchange component of this Test Platform consisted of a small-scale column system with a pump and sampling points. Two columns were positioned in a lead-lag format, each filled with 10.0 mL of CST ion exchanger. The goal was to process feed until the ^{137}Cs concentration in the effluent reached the WAC. To this end, effluent samples were collected periodically during the load process and measured for ^{137}Cs and the load curve was developed. Early Cs breakthrough resulted in processing slightly past the WAC. As a result, a new lag column was needed to continue processing and the original lag column was moved to the lead position.

A total of 9 L of AP-107 tank waste, consisting of 5.6 M Na and 156 µCi/mL ^{137}Cs, was processed through the Cs ion exchange system. The feed flowrate was set to 2.2 BV/h with 0.1 M NaOH FD and water rinse flowrates set to 3 BV/h. The following conclusions were made as a result of this work.

1. A quantity of 410 BVs of AP-107 feed was processed at 2.2 BV/h before reaching the WAC. After switching columns, another 285 BVs were processed before the contract limit was again reached.

2. Extrapolation beyond the 25% lead column breakthrough indicated that a 50% breakthrough of 640 BVs would have been obtained if continuous loading had occurred. However, at 2.2 BV/h this cannot be obtained before Cs breakthrough above the WAC from the lag column occurs.

3. Actual waste results were in line with simulant test results with respect to the system flowrates vs. volume processed to contract limit.

4. Within analytical uncertainty, >99% of the Cs processed through the ion exchange system was collected on the CST in the columns. However, 0.82% of Cs leaked through the lag column into the effluent and required further processing to meet the WAC.

6.3 Sample Analysis

1. Al, Cr, K, Na, Ni, and P partitioned almost exclusively to the effluent. Pb and As were detected in the feed but not in the effluent. This indicates that some fraction of these analytes exchanged onto the CST. Small fractions (12% to 49%) of Ba, Ca, Sr, and U were found in the effluent, indicating they were partially removed by the CST from the feed. The ^{90}Sr analysis indicated nearly complete Sr removal with a DF of 2160. Only ~30% of the Pu partitioned to the effluent, indicating that ~70% sorbed onto the CST.

2. Selected lead column samples from before column change-out were analyzed to examine analyte loading as a function of process volume. The ^{90}Sr DFs started at ~700 and decreased to 76 through the first 471 BVs processed through the lead column. Ti and Zr decreased in percentage in the lead column samples with increasing load volume, yet the feed and effluent Zr and Ti concentrations were fundamentally equivalent. This indicated that the CST did not add Ti and Zr to the effluent and thus the CST was not chemically "decomposing." Zn, Ca, Fe, U, and Pu showed increasing breakthrough from the lead column with loading. Detected RCRA elements showed relatively no difference in percentage with loading.

3. Selected batch contact samples of varying Cs concentrations (6.39E-5 to 1.19E-2 M) showed nominally all analyte exchange remained constant through the increase in Cs concentration. However, Ba and Ca showed a slight decrease in analyte exchange with increased Cs concentration and Fe jumped from 65% to 100% C/C_0 equilibrium concentration when Cs increased from 9.27E-04 M to 9.64E-03 M.

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Appendix A

Column Load Data

The AP-107 column loading, feed displacement, and water rinse raw data are provided in Table A.1

Table A.1. AP-107 Test 1 Cs Load, Feed Displacement, and Water Rinse Results

Lead Column				Lag Column				Feed Displacement and Water Rinse			
BV	μCi ^{137}Cs/ mL	% C/C_0	DF	BV	μCi ^{137}Cs/ mL	% C/C_0	DF	BV	μCi ^{137}Cs/ mL	% C/C_0	DF
14.4	3.40E-3	2.17E-3	46,071	14.4	4.56E-4	2.91E-4	343,547	FD			
46.9*	2.64E-3	1.69E-3	59,238	46.5	2.76E-4	1.76E-4	567,124	880.5	9.62E-1	6.15E-1	163
57.0*	1.36E-2	8.68E-3	11,518	69.8	1.29E-4	8.21E-5	1,217,766	881.4	9.87E-1	6.31E-1	159
100.9*	8.49E-2	5.42E-2	1,844	99.8	1.41E-4	8.99E-5	1,112,153	882.3	1.08E+0	6.87E-1	146
123.8*	2.25E-1	1.44E-1	694	122.3	1.28E-4	8.19E-5	1,220,399	883.2	1.05E+0	6.73E-1	149
155.2	6.78E-1	0.433	231	153.3	1.78E-4	1.14E-4	880,862	884.1	1.07E+0	6.84E-1	146
178.1*	1.31E+0	0.839	119	175.9	2.68E-4	1.71E-4	584,293	885.1	7.31E-1	4.67E-1	214
206.9	2.49E+0	1.59	63	204.3	8.12E-4	5.19E-4	192,751	DI rinse			
231.5*	4.23E+0	2.70	37	228.5	1.93E-3	1.24E-3	80,955	886.0	1.62E-1	1.04E-1	966
260.9	6.53E+0	4.17	24	257.5	5.42E-3	3.46E-3	28,873	887.0	4.77E-2	3.04E-2	3,284
284.2*	8.81E+0	5.63	18	280.3	1.03E-2	6.57E-3	15,227	887.9	2.36E-2	1.50E-2	6,645
310.9	1.16E+1	7.42	13	306.7	1.99E-2	1.27E-2	7,881	888.8	1.04E-2	6.61E-3	15,121
334.6*	1.50E+1	9.58	10	330.0	3.88E-2	2.48E-2	4,034	889.8	7.25E-3	4.63E-3	21,586
364.8	1.93E+1	12.3	8	359.8	7.08E-2	4.53E-2	2,209	890.7	5.60E-3	3.58E-3	27,946
389.4*	2.40E+1	15.3	7	384.2	1.14E-1	7.32E-2	1,367				
417.3	2.82E+1	18.0	6	411.7	1.90E-1	1.21E-1	824				
441.4	3.38E+1	21.6	5	435.6	2.91E-1	1.86E-1	538				
471.2*	3.92E+1	25.0	4	465.2	4.42E-1	2.83E-1	354				
493.4	1.05E+0	0.67	149	486.9	2.95E-2	1.89E-2	5,297				
524.7	1.23E+0	0.79	127	517.8	7.93E-4	5.07E-4	197,365				
546.5	1.62E+0	1.03	97	539.2	7.85E-4	5.02E-4	199,254				
579.2	2.44E+0	1.56	64	571.6	2.46E-3	1.57E-3	63,733				
604.1	3.49E+0	2.23	45	596.1	5.64E-3	3.60E-3	27,759				
634.1	5.24E+0	3.35	30	625.8	1.37E-2	8.76E-3	11,412				
659.6	7.34E+0	4.69	21	651.0	2.36E-2	1.51E-2	6,632				
685.3	9.41E+0	6.01	17	676.4	3.87E-2	2.47E-2	4,042				
710.2	1.24E+1	7.93	13	700.9	6.91E-2	4.42E-2	2,264				
742.9	1.69E+1	10.8	9	733.2	1.20E-1	7.68E-2	1,302				
763.5	1.96E+1	12.5	8	753.3	1.70E-1	1.08E-1	922				
795.8	2.42E+1	15.5	6	785.2	2.71E-1	1.73E-1	577				
817.6	2.81E+1	17.9	6	806.7	3.93E-1	2.51E-1	398				
847.8	3.15E+1	20.1	5	836.6	5.46E-1	3.49E-1	286				
861.0	3.20E+1	20.5	5	849.6	5.81E-1	3.71E-1	269				
891.1	4.04E+1	25.8	4	879.4	9.27E-1	5.92E-1	169				

BV = bed volume; DI = deionized; DF = decontamination factor; FD = feed displacement; C_0 = 156.5 μCi ^{137}Cs/ mL; * = samples submitted for additional analysis.

Appendix B

Analytical Reports

Analytical reports provided by Analytical Support Operations are included in this appendix. In addition to the analyte results, they define the procedures used for chemical separations and analysis, as well as quality control sample results, observations during analysis, and overall estimated uncertainties. The analyses are grouped according to Analytical Service Request (ASR) number.

Table of Contents

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature For S. Fiskum
Print Name __Sandy Fiskum
Phone ___375-5677 MSIN ___P7-25

Project Number: 69832
Work Package: N96051

Matrix Type Information

- **Liquids:** X Aqueous ☐ Organic ☐ Multi-phase
- **Solids:** ☐ Soil ☐ Sludge ☐ Sediment
  ☐ Glass ☐ Filter ☐ Metal
  ☐ Smear ☐ Organic ☐ Other
- **Other:** ☐ Solid/Liquid Mixture, Slurry
  ☐ Gas ☐ Biological Specimen

(If sample matrices vary, specify on Request Page)

QA/Special Requirements

- **QA Plan:**
  X ASO-QAP-001 (Equivalent to HASQARD)
  ☐ Additional QA Requirements, List Document Below:
  Reference Doc Number:
- **Field COC Submitted?** X No ☐ Yes
- **Lab COC Required?** X No ☐ Yes
- **Sample/Container Inspection Documentation Required?**
  X No ☐ Yes
- **Hold Time:** X No ☐ *Yes*
  If *Yes*, Contact ASO Lead before submitting Samples
  ☐ Use SW 846 (PNL-ASO-071, identify analytes/methods where holding times apply)
  ☐ Other? Specify:
- **Special Storage Requirements:**
  X None ☐ Refrigerate ☐ Other, Specify:
- **Data Requires ASO Quality Engineer Review?** ☐ No X Yes

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc:
- **Disposition of Treated Samples:**
  X Dispose ☐ Return

Data Reporting Information

- **Is Work Associated with a Fee-Based Milestone?** X No ☐ Yes
  If yes, milestone due date:
- **Preliminary Results Requested, As Available?** ☐ No X Yes

- **Data Reporting Level**
  X ASO-QAP-001 (Equivalent to HASQARD).
  ☐ Minimum data report.
  ☐ Project Specific Requirements:
  Contact ASO Lead or List Reference Document:

- **Requested Analytical Work Completion Date:**
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:** 11/30/17
  (To be completed by ASO Lead)

Waste Designation Information

- **ASO Sample Information Check List Attached?** ☐ No ☐ Yes
  If no, Reference Doc Attached:
  or, Previous ASR Number:
  or, Previous RPL Number:

Does the Waste Designation Documentation Indicate Presence of PCBs?
X No ☐ Yes

Send Report To: __**S. Fiskum, H. Colburn** **MSIN**
__J. Geeting, J. Allred **MSIN**
Additional or Special Instructions

Receiving and Login Information *(to be completed by ASO staff)*

Date Delivered: 10/24/17
Delivered By (optional):
Time Delivered: 11:00 AM
Group ID (optional):
CMC Waste Sample? X No ☐ Yes

Received By: ___T. Trang-Le
ASR Number: 0395 Rev.: 01
RPL Numbers: 18-0117 thru 18-0118
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** 11/13/17

Analytical Services Request (ASR)

(REQUEST PAGE ---- Information Specific to Individual Samples)

Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**

ASO Staff Use Only **RPL Number**	Client Sample ID	Sample Description (& Matrix if varies)	Analyses Requested	ASO Staff Use Only **Test**	ASO Staff Use Only **Library**
Revision 1 - **1) Sr-90 Analyses and 2) Am-241 by GEA has been added to the scope of analyses.**					
18-0117	7AP-17-11	AP-107 Tank Supernate	1) GEA - As received solutions. (Cs-137, Am-241) 2) Acid Digestion - 128 a) ICP/OES - Al, K, and Na b) ICP/MS - Cs-133 (Isotopics) c) Sr-90 3) Free OH		
18-0118	7AP-17-46	AP-107 Tank Supernate			

ASR # 0395 Rev: 01

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 69832 / N79882
ASR#: 0395.01
Client: S. Fiskum
Total Samples: 2 (liquids)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0117	7AP-17-11	AP-107 Tank Supernate	NA
18-0118	7AP-17-46	AP-107 Tank Supernate	NA
DUP-0118	7AP-17-46	AP-107 Tank Supernate	NA

Sample Preparation: RPG-CMC-128, Rev. 1. "HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater", performed by L. Darnell on Samples 18-0117, 18-0118, and DUP-0118 on 11/28/17. Simple dilution of "as received" samples in 5% v/v HNO_3 performed by J. Carter on 12/07/17.

Procedure: <u>RPG-CMC-211, Rev. 4</u>, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)."

Analyst:	J. Carter	**Analysis Date:**	12/07/2017	**ICP File:**	C0738

See Chemical Measurement Center 98620 file: <u>ICP-325-405-3</u>
(Calibration and Maintenance Records)

M&TE:

☒	PerkinElmer 5300DV ICP-OES	SN: 077N5122002
☒	Mettler AT400 Balance	SN: 1113292667
☒	Sartorius R200D Balance	SN: 39080042
☒	SAL Cell 2 Balance	SN: 8033311209
☒	Lab 201 Denver A-160 Balance	SN: 60568

Garrett N. Brown
Report Preparer

1/26/18
Date

Review and Concurrence

1/26/18
Date

Two aqueous samples submitted under Analytical Service Request (ASR) 0395.01 were analyzed by ICP-OES. Samples 18-0117 and 18-0118 were prepared following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Sample 18-0118 was prepared in duplicate following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. All samples were further diluted in 5% HNO_3 prior to analysis. None of the samples were filtered.

All sample results are reported on a mass per unit volume basis (µg/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-OES Data Report. The quality control (QC) results for the AOI have been evaluated and are presented below. Analytes other than the AOI are reported in the bottom section of the report but have not been fully evaluated for QC performance.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte and for continuing calibration verification.

The controlling documents were procedures RPG-CMC-211, Rev. 4, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*, and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan.* Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), matrix spike, post-digestion spikes, duplicate, reagent spike, blank spike, and serial dilution were conducted during the analysis run.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentration of all AOI were within the acceptance criteria of <EQL (estimated quantitation level), ≤50% regulatory decision level, or ≤10% of the concentration in the samples.

Reagent Spike (RS)/Laboratory Control Sample (LCS):

A reagent spike (RS) sample (reagents and spikes) was prepared for the extraction process. Recovery values are listed for all analytes included in the RS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 103% to 107%, and were within the acceptance criterion of 80% to 120%.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

A duplicate of sample 18-0118 was prepared and analyzed. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for the AOI meeting this requirement ranged from 1.0% to 7.2% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike (MS) of sample 18-0001 was prepared for the extraction process. Recovery values are listed for all analytes included in the MS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 106% to 116%, and were within the acceptance criterion of 75% to 125%.

Initial/Continuing Calibration Verification (ICV/CCV):

MCVA and MCVB solutions were analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery, with the exception of potassium (110.1%) in the final CCV solution.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (5% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL, with the exception of sodium in the final three CCB solutions.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 70% to 130% recovery.

Interference Check Standard (ICS/SST):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-0018. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 3.5% to 6.9% and were within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS-A)/Analytical Spike (AS-A) - Sample (A Component):

In addition to the BS sample, a post-digestion spike (A Component) was conducted on sample 18-0017. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 97% to 101%, and were within the acceptance criterion of 80% to 120%.

Post-Digestion Spike (PS-B)/Analytical Spike (AS-B) - Sample (B Component):

In addition to the MS sample, a post-digestion spike (B Component) was conducted on sample 18-0117. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. There were no AOI included in the spike B Component.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 5% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017
		Process Factor >	1.0	48.9	242.6	242.6	244.5	243.3
			405 diluent	BLK-0001	18-0117 @ 5x	18-0117 @ 5x rep	18-0118 @ 5x	Dup-0018 @5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	BLK-0001	7AP-17-11		7AP-17-46	
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0038	0.038	Al	--	--	9,850	9,750	9,750	9,950
0.0262	0.262	K	--	[4.6]	3,720	3,710	3,730	4,010
0.0075	0.075	Na	--	[3.3]	129,000	129,000	129,000	130,000
Other Analytes								
0.0014	0.014	Ag	--	--	--	--	--	--
0.0383	0.383	As	--	--	--	--	--	--
0.0032	0.032	B	[0.020]	[0.90]	32.6	30.8	35.1	47.4
0.0001	0.001	Ba	[0.0001]	0.580	1.17	1.16	0.934	0.844
0.0001	0.001	Be	--	--	[0.14]	[0.13]	[0.14]	0.165
0.0220	0.220	Bi	--	--	--	--	--	--
0.0054	0.054	Ca	--	6.86	31.2	30.9	28.3	35.5
0.0016	0.016	Cd	--	--	6.62	6.50	6.85	6.82
0.0052	0.052	Ce	--	--	--	--	--	--
0.0033	0.033	Co	--	--	--	--	--	--
0.0016	0.016	Cr	--	--	496	492	491	494
0.0023	0.023	Cu	--	--	[1.4]	[1.3]	[1.1]	[0.93]
0.0012	0.012	Dy	--	--	--	--	--	--
0.0006	0.006	Eu	--	--	--	--	--	--
0.0033	0.033	Fe	--	[0.26]	16.4	16.0	14.8	14.7
0.0010	0.010	La	--	--	--	--	--	[0.47]
0.0010	0.010	Li	--	[0.084]	[0.65]	[0.73]	[0.98]	[0.77]
0.0014	0.014	Mg	--	[0.23]	--	--	--	[1.6]
0.0003	0.003	Mn	--	--	--	--	--	[0.18]
0.0048	0.048	Mo	--	--	41.7	40.2	41.6	41.0
0.0095	0.095	Nd	--	--	[3.0]	--	--	--
0.0040	0.040	Ni	--	--	22.1	21.8	22.6	22.4
0.0369	0.369	P	--	--	648	638	677	638
0.0144	0.144	Pb	--	--	[11]	[12]	[11]	[10]
0.0082	0.082	Pd	--	--	[2.4]	[3.1]	[4.1]	[3.5]
0.0108	0.108	Rh	--	--	[2.7]	[2.9]	--	--
0.0068	0.068	Ru	--	--	[6.8]	[6.6]	[6.4]	[5.6]
0.0898	0.898	S	--	--	1,720	1,690	1,740	1,640
0.0569	0.569	Sb	--	--	--	--	--	--
0.0876	0.876	Se	--	--	[31]	--	--	--
0.0043	0.043	Si	[0.0076]	3.33	38.3	38.6	42.4	42.0
0.0195	0.195	Sn	--	--	[5.4]	[6.1]	--	[6.3]
0.0001	0.001	Sr	--	[0.020]	[0.092]	[0.086]	[0.090]	[0.099]
0.0109	0.109	Ta	--	--	--	--	--	--
0.0155	0.155	Te	--	--	--	--	--	--
0.0057	0.057	Th	--	--	[1.7]	--	[1.8]	[2.1]
0.0004	0.004	Ti	--	[0.021]	--	--	--	[0.28]
0.0310	0.310	Tl	--	--	--	--	--	--
0.0312	0.312	U	--	--	[14]	[18]	[17]	[20]
0.0016	0.016	V	[0.0039]	[0.23]	[1.2]	[1.0]	[1.2]	[1.3]
0.0187	0.187	W	--	--	67.2	62.8	65.4	66.7
0.0003	0.003	Y	--	--	--	--	--	--
0.0023	0.023	Zn	--	1.30	[1.4]	[0.81]	[1.0]	[0.76]
0.0013	0.013	Zr	--	--	[0.60]	[0.33]	[0.41]	[0.44]

1) "–" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

QC Performance 12/7/2017

Criteria >	≤ 20%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	18-0018 Dup	LCS/RS	18-0001 MS	18-0117 + AS-A	18-0117 + AS-B	18-0117 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	2.0	103	106	97		3.5
K	7.2	104	116	101		3.6
Na	1.0	107	nr	nr		6.9
Other Analytes						
Ag				92		
As				102		
B	29.7	104	102	101		41.1
Ba	10.2	101	103	99		2.5
Be		100	101	97		
Bi		80	82	94		
Ca	22.5	108	115	103		
Cd	0.4	100	102	100		
Ce		99	98		96	
Co				99		
Cr	0.6	97	92	95		3.3
Cu		104	107	104		
Dy					96	
Eu					96	
Fe	0.3	101	100	100		
La		99	99		95	
Li		118	119	106		
Mg		103	105	102		
Mn		100	100	100		
Mo	1.3	98	94	97		
Nd		99	97		96	
Ni	0.9	101	101	102		
P	5.8	101	95	100		6.6
Pb		98	99	98		
Pd					91	
Rh					94	
Ru					94	
S	6.1	97	91		96	2.1
Sb				105		
Se				100		
Si	1.1	23	51	100		
Sn				94		
Sr		106	98	101		
Ta				98		
Te					98	
Th					95	
Ti		103	104	100		
Tl				91		
U		102	100		100	
V		100	98	97		
W	2.0	97	94	99		
Y				96		
Zn		97	98	99		
Zr		107	107	101		

Shaded results are outside the acceptance criteria

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

Battelle PNNL/RPL/Inorganic Analysis ... ICP-MS Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 69832 / N96051
ASR#: 0395.01
Client: S. Fiskum
Total Samples: 2 (Aqueous)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0117	7AP-17-11	AP-107 Tank Supernate	NA
18-0118	7AP-17-46	AP-107 Tank Supernate	NA
DUP-0118	7AP-17-46	AP-107 Tank Supernate	NA

Sample Preparation: RPG-CMC-128, Rev. 1. "HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater", performed by L. Darnell on 11/28/17 and ICP-MS bench dilution in 2% v/v HNO_3 performed by G. Brown on 12/07/17, respectively.

Procedure: RPG-CMC-292, Rev. 1, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."

Analyst:	G. Brown	**Analysis Date:**	12/07/2017	**ICP File:**	M0042

See Chemical Measurement Center 98620 file: ICP-325-405-3 (Calibration and Maintenance Records)

M&TE:	☒	PerkinElmer NexION™ 350X ICP-MS	SN: 85VN4070702	RPL 405
	☒	Sartorius R200D Balance	SN: 39080042	RPL 405
	☒	Mettler AT400 Balance	SN: M19445	RPL 405 FH
	☒	Mettler AT400 Balance	SN: 1113292667	RPL 420 FH
	☐	Ohaus EX324 Balance	SN: 8033311209	SAL Cell 2
	☐	Sartorius BA3105 Balance	SN: 10803210	RPL 309
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH
	☐			

Garrett N. Brown
Report Preparer

1/19/18
Date

Review and Concurrence

1/23/18
Date

Two aqueous samples submitted under Analytical Service Request (ASR) 0395.01 were analyzed by ICP-MS. Sample 18-0118 was prepared in duplicate following RPL procedure RPG-CMC-128 by diluting 0.5 mL to approximately 25 mL. Prior to analysis all samples were further diluted in 2% HNO_3. None of the samples were filtered.

All results are reported on a mass per unit volume basis (ng/mL or µg/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-MS Data Report. Cesium-133 was the only AOI. The quality control (QC) results for the AOI have been evaluated and are presented below.

Calibration of the ICP-MS was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards were used to verify acceptance of the six-point calibration curve and for initial and continuing calibration verification (ICV/CCV).

The controlling documents are procedure RPG-CMC-292, Rev 1, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)* and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan.* Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spike, duplicate, blank spike, and serial dilution were conducted during the analysis run.

Internal Standard (IS):

All solutions (blanks, standards, QC checks, and samples) were mixed in-line with a solution containing 10 ppb each of Li-6, Sc-45, Y-89, In-115, Tb-159, and Bi-209 as the internal standard (IS). The AOI (Cs-133) data were normalized using the data for the closest IS mass (e.g., In-115). The In-115 IS recoveries ranged from 94.5% to 104.8%, which were within the acceptance criterion of 30% to 120%.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. The concentration of Cs-133 in the PB (0.027 ng/mL) exceeded the acceptance criteria of <EQL (estimated quantitation level, 0.0016 ng/mL), <50% regulatory decision level, or less than ≤10% of the concentration in the samples. The PB also contained a number of environmental contaminants (Ba, Pb, Sr, Zn) that are consistent with the instrument sensitivity and background contamination levels that are ambient in the sample preparation and samples analyses laboratories. A diluent blank from the ICP-MS laboratory (2% HNO_3) was also analyzed. Results for the diluent blank were within the acceptance criteria.

Blank Spike (BS)/Laboratory Control Sample (LCS):

A blank spike (BS) sample was prepared by spiking a 2% HNO_3 blank with an equivalent volume of the CCV-71A-2ppb standard (1:1 ratio). The recovery for Cs-133 was 89.4%, which is within the acceptance criteria of 80% to 120% recovery.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

A duplicate of sample 18-0118 was prepared and analyzed. RPD are listed for all analytes that were measured at or above the EQL. RPD for the AOI (Cs-133) meeting this requirement was 8.5% and was within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

No matrix spike sample was required to be prepared.

Initial/Continuing Calibration Verification (ICV/CCV):

The ICV/CCV solution was analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. With the exception of the final CCV (88.6% Cs-133), the concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (2% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). With the exception of the penultimate CCB (0.022 ng/mL Cs-133), the concentration of all AOI were within the acceptance criteria of <EQL. Following an additional rinse time, the final CCB met the acceptance criteria.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The recovery of the AOI was 94% which is within the acceptance criteria of 70% to 130%.

Interference Check Standard (ICS):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The recoveries of the AOI were 87% and 97% which is within the acceptance criteria of 80% to 120%.

Serial Dilution (SD):

Five-fold serial dilution was conducted on samples 18-0117 and 18-0118. Percent differences (%D) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 1.5% to 3.1% and were within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS-71A)/Analytical Spike (AS-71A) - Sample (71A Component):

Because no MS sample was required to be prepared, a post-digestion spike (PS-71A) was conducted on samples 18-0117 and 18-0118. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement were 93.1% and 93.3%, respectively, and were within the acceptance criterion of 80% to 120%.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike 71A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, K, La, Lu, Mg, Mn, Na, Nd, Ni, P, Pb, Pr, Rb, S, Se, Sm, Sr, Th, Tl, Tm, U, V, Yb, and Zn. Analytes included in the spike 71B component are; Ge, Hf, Mo, Nb, Sb, Si, Sn, Ta, Te, Ti, W, and Zr. Analytes included in the spike 71C component are; Ir, Os, Pd, Pt, Re, Rh, and Ru. Analytes included in the spike 71D component are; Bi, In, Li, Sc, Tb, and Y. Analytes included in the spike Hg component are; Hg.

Battelle PNNL/RPG/Inorganic Analysis ... ICP-MS Data Report

		Run Date >	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017
		Process Factor >	1.0	10.04	481,142	488,711	487,008
		RPL/LAB >	Lab 405 Blank	BLK-0001 @ 10x	18-0117 @ 10,000x	18-0118 @ 10,000x	DUP-0118 @ 10,000x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab 405 Blank	BLK-0001 @ 10x	7AP-17-11	7AP-17-46	
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0002	0.002	Cs 133	--	0.0274	5.42	6.09	5.59

Internal Standard % Recovery

Li 6 (IS)	97%	103%	99%	101%	98%
Sc 45 (IS)	95%	105%	100%	99%	98%
Y 89 (IS)	94%	111%	98%	98%	91%
In 115 (IS)	97%	100%	99%	98%	100%
Tb 159 (IS)	97%	108%	100%	99%	102%
Bi 209 (IS)	98%	96%	95%	89%	101%

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

3) Values for IDL, EQL, laboratory and process blanks are listed as ng/mL, while samples are listed as µg/mL.

(*) The isotope measured exhibited significant molecular interferences due to HCl in the sample preparation.

QC Performance12/07/2017

Criteria >	≤ 20%	80%-120%	75%-125%	75%-125%	75%-125%	≤ 10%	≤ 10%
QC ID >	18-0118 Dup	LCS/BS	MS (None)	18-0117 + CCV-71A	18-0118 + CCV-71A	18-0117 5-fold Serial Dil	18-0118 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff
Cs 133	8.5%	89%		93%	93%	3.1%	1.5%
Internal Standard % Recovery							
Bi 209 (IS)	98%	101%		102%	103%	97%	98%
In 115 (IS)	98%	102%		103%	101%	98%	97%
Li 6 (IS)	91%	95%		99%	101%	98%	96%
Sc 45 (IS)	100%	97%		102%	100%	98%	95%
Tb 159 (IS)	102%	102%		102%	100%	99%	98%
Y 89 (IS)	101%	90%		94%	89%	92%	91%

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na2O2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Sr-90 by Liquid Scintillation Spectrometry

Project / WP#:	69832/N96051
ASR#:	0395.01
Client:	SK Fiskum
Total # of Samples:	2

RPL ID	Client Sample ID
18-0117	7AP-17-11
18-0118	7AP-17-46

Analysis Type:	Sr-90
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None ☐ Digested as per RPG-CMC-128, Rev.1, *HNO_3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other:
Pre-dilution Prior to Radiochemical Processing?	☐ No ☒ Yes
Separation Procedure:	RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin*
Spike Standard ID:	R-693-a-1 (Sr-90)
Separation Date:	11/18/2018 @11:25 a.m.
Technician/Analyst:	L. Darnell
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	NA
Analysis Date or Date Range:	01/19/2018 (first count), 01/25/2018 (second count)
Technician/Analyst:	LP Darnell & CZ Soderquist
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 18\18-0117 Fiskum.xls
ASO Project 98620 File:	File Plan 5871: T 69832 0351: Sample preparation and analysis records; T-4.4 LSC 3100 TR calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Perkin Elmer Tri-Carb 3100 TR, Serial # DG08061340, RPL 425, Tri-Carb 2700TR software version 1.04 dated 9/99, Mettler AT400, Serial # 1113292667

T Trang-le / 2/13/18
Preparer **Date**

/ 2/13/18
Reviewer **Date**

SAMPLE RESULTS

See attached data report, Sample Results for ASR 0395.01. All sample data are reported in µCi/mL with a 1-σ uncertainty (see Comments).

ASO Project File, ASR 0395.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, standard certificates, laboratory bench records, and Liquid Scintillation Counter Analysis printouts. Detector calibration records, control charts and balance calibration records can be found in the ASO Records.

Sample preparation, separation, mounting, and counting

Two samples submitted under Analytical Service Request (ASR) 0395.01 were analyzed for Sr-90 by chemical separation and liquid scintillation counting. The samples were prepared by simple dilution of the AP-107 tank liquid. The samples were diluted ~ 100x prior to chemical separations. All the samples were prepared in RPL/420.

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in RPL/420 include a lab separation blank (LB), sample duplicate, matrix spike (i.e., addition of Sr-90 standard to an aliquot of one of the samples) and reagent spike.

Instrument Calibration Control

Laboratory Preparation Blank and Laboratory separations blank (LB):

In this case, sample preparation was not performed prior to chemical separation other than simple dilution. A laboratory separation blank was run with the sample batch, however there are no acceptance criteria for laboratory separation blank (see Comments).

Blank Spike (BS) – Reagent Spike (RS):

The RS recovery of 100% meet the procedure acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 99% meet the procedure acceptance criteria of 80% to 120% recovery. Sample 7AP-17-46 (18-0118) was used for the matrix spike analyses.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤ 20% RPD. The ASO QAP further specifies that the two results need to be > 5 times the MDA or have individual uncertainties < 20%. Duplicate results were 1% RPD; thus meeting the ≤ 20% requirement.

Instrument Quality Control

The liquid scintillation counter is calibrated for tritium and C-14 using quenched standard sets that are purchased from the vendor. Daily control counts are then performed using a tritium, C-14, and a background count sample. The instrument software assesses the performance of the control counts and provides control charts to ensure the continuing calibration of the instrument. If the daily performance check fails, then the instrument is not used. Preventative maintenance and repairs are performed by the vendor under our service contract. The counting efficiency for Sr-90 is assumed to be 100%; therefore no specific Sr-90 calibration is performed. The LSC system calibration and performance is verified by assessing the recovery of a reagent spike and a matrix spike that are included in every batch of samples. A preparation blank (i.e., digestion blank) and a laboratory separations blank are also included with every batch of samples; the instrument background is subtracted from all results and the preparation and separation blanks are used to assess sample contamination during sample processing steps.

Assumption and Limitations of the Data

The 1-σ uncertainty reported for each Sr-90 result has been set at 2%. Although the calculated uncertainty values are less than 2% for all samples, the radiochemistry convention is to not report calculated uncertainties less than 2%, but to provide a more realistic estimate of the uncertainty in view of systematic uncertainties that are not fully accounted for in the uncertainty calculations.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radio-analytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the Sr-90 spike was not added prior to the digestion process.
3. Radiochemistry Electronic Systems File "RPG-RC\PNL\Projects\Backup files\Backup 18\ 18-0117 Fiskum.xls " has been created for this report. Supporting records such as Pipette Performance Verification forms, Laboratory Bench Record, Laboratory Sample Preparation Bench Sheet, Standards Certifications and preparation records, and balance calibration and performance check records are maintained per NC&E Group ASO File Plan 5871.
4. Sample results are compared to the process blank results to evaluate if the blank contains 5% or more of the measured isotope; the process blank results have been adjusted for all processing factors for the evaluation of the 5% criterion.
5. The stated 1-σ uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting error.

Attachment: Data Report -- Sample Results for ASR 0395.01.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Radiochemical Sciences and Engineering Group

filename 18-0117 Fiskum.xls
1/30/2018

Client: Fiskum
ASR 0395.01

Project: 69832
WP: N96051

Prepared by: T Trang-le 1-30-2018

Technical Reviewer: C. Soderquist 1-30-2018

Procedure: RPG-CMC-474, Rev 1, Measurement of Alpha and Beta Activity by Liquid Scintillation
M&TE: Perkin Elmer Tri Carb model 3100 TR liquid scintillation spectrometer

Count dates: January 19 & 25, 2018

		Measured Activity, µCi per ml ± 1s	
Sample	Lab ID	**Sr-90**	
7AP-17-11	18-0117	**5.97E-01**	**± 2%**
7AP-17-46	18-0118	**5.93E-01**	**± 2%**
	18-0118 DUP	**5.99E-01**	**± 2%**
	RPD	1%	
	Reagent Spike	100%	
	Matrix Spike	99%	
	Lab Blank	1.09E-04	± 44%

The lab blank is below detection limit

Pacific Northwest
NATIONAL LABORATORY

Client:	S. Fiskum	Report Date:	1/15/2018
		Analysis Date:	1/10/2018

Subject: Hydroxide Analyses for: Aqueous Samples
Project: 69832 WP # N96051

ASR: **0395.01** Procedure: RPG-CMC-228, Rev 0.1
RPL Sample ID 18-0117 thru 18-0118
Clinet Sample II 7AP-17-11 7AP-17-46

Hydroxide analysis was performed for diluted sample aliquots of 2 aqueous samples (18-0117 and 18-0118), provided on ASR 0395.01. Both client samples are undiluted AP-107 tank supernate. Samples were analyzed by manual titration for the base constituents content following procedure RPG-CMC-228, Rev. 0.1, *Determination of Hydroxyl (OH^-) and Alkalinity of Aqueous Solutions, Leachates & Supernates* . Sample size was 0.100 mL of sample added to 2.5 mL of DI water. The titrant used was 0.0902 M HCl (Standardized HCl was prepared and documented on Chem Rec 234, prepared on 7/11/2017). pH measurements were obtained using a Beckman Coulter 560 pH meter, SN 110650046. The pH meter was calibrated using 3 buffers, pH 4,7 and 10 and the calibration verified using an independent pH 7 buffer.

The initial diluted pH is reported on attached Report Summary along with the free hydroxide molarity.

Sample ID	1st Inflection point	2nd Inflection point	3rd Inflection point
18-0117	11.1	7.1	4.0
18-0118	10.8	7.0	4.4

Following is the report summary and the sample results calculated from the raw data. A copy of the titration curve data for each sample is also included with this report.

Prepared by: Crystal Rutherford Date: 1/15/18

Reviewed by: Date: 1/24/18

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group
Chemical Measurements Center

ASR # 0395.01

WP# N96051

Hydroxide and Alkalinity Determination
Procedure: RPG-CMC-228, Rev 0.1

Equip # Beckman Coulter 560, SN#110650046

Report Summary for ASR # -- 0395.01

Report Date: 1/15/2018
Analysis Date: 1/10/2018

				Concentration, moles / Liter		
				First Point	Second Point	Third Point
RPG #	Client ID	Diluted Initial pH	OH conc ug/mL	Molarity	Molarity	Molarity
18-0117	7AP-17-11	12.75	1.58E+04	0.93	1.25	0.54
18-0118	7AP-17-46	12.74	1.53E+04	0.90	1.34	0.42

OH conc (mg/L) = M (g/L) * 17,000

Note: Results are presented for the first, second, and third inflection points on the titration curves for the hydroxide titration, as applicable. The first inflection point is generally associated with the free hydroxide concentration. The second inflection point generally represents total hydroxide, or carbonate or a combination of aluminate and carbonate. The third inflection point is usually indicative of bicarbonate or other weak acids or possibly the continued protonation of alumina.

Analyst: Crystal Rutherford 1-15-18

Reviewer: Karl N. Pool 1/23/18

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev #	0395.01	Rev-0
Client:	S. Fiskum	
Project: 69832	WP#	N96051

Report Date: **1/15/2018**
Analysis Date: **1/10/2018**

Procedure: **RPG-CMC-228, Rev 0.1** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates

Spreadsheet: OH-TemplateLocked07.xls 4/1/2007

By Manual Titration

Equip # 110650046 Lab Loc. 301

Analyst: 1-15-18

Titrant	Molarity	Chem Rec#
HCl	**0.0902**	**234**

RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	**Diluted** Initial pH reading	**OH** **1st Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
18-0117	7AP-17-11		NA	0.100	NA	NA	NA	12.75	1.03	11.13	0.093	0.929
18-0118	7AP-17-46		NA	0.100	NA	NA	NA	12.74	1.00	10.75	0.090	0.897

Instrument Calibration

Buffer	Vendor	Lot Number	Expire Date
4	Inorganic Ventures	J2-WCS02116	1-Jul-18
7	Inorganic Ventures	J2-WCS02097	1-Mar-18
10	Inorganic Ventures	H2-WCS02089	14-Jan-18

Slope
95.6%

2-nd Verif	Vendor	Lot Number	Expire Date
7	Ricca	1603E33	1-Mar-18

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev #	0395.01	Rev-0
WP#	N96051	

Procedure:

Alkalinity of Aqueous Solutions, Leachates and Supernates

By Manual Titration

Equip # Beckman Coulter 560, SN#110650046

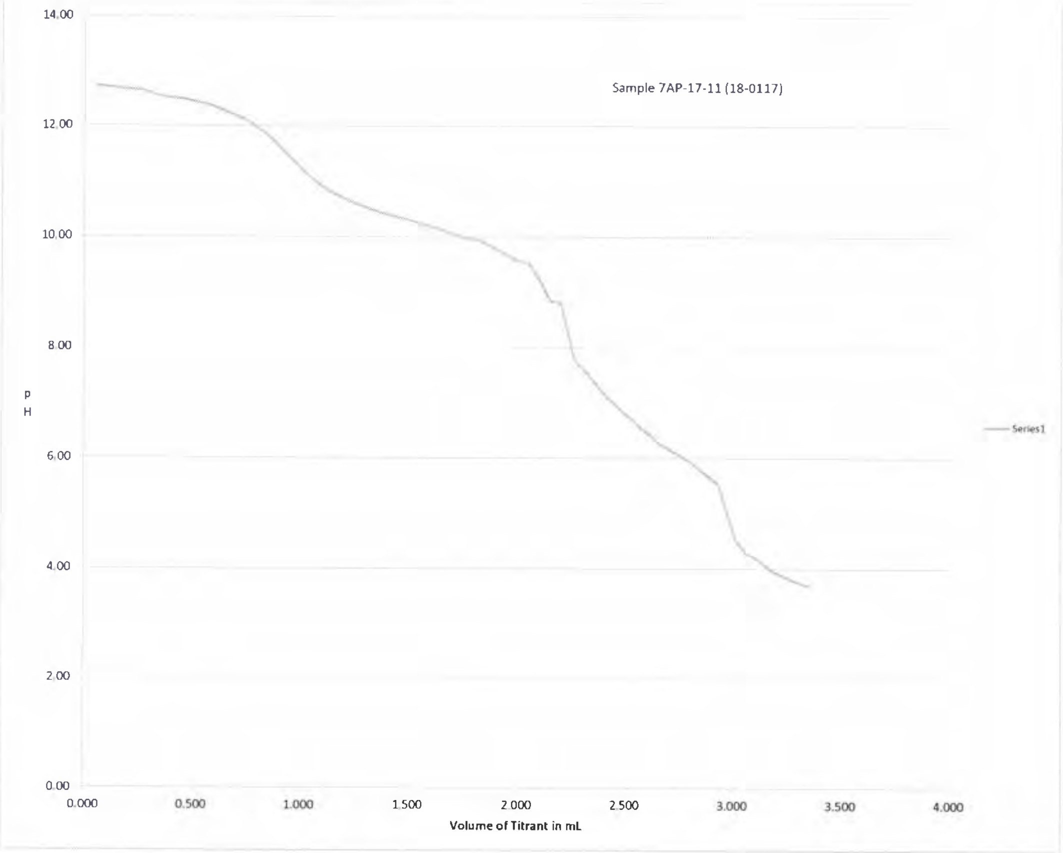
Titrant	Molarity
HCl	0.0902

RPG #		Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
18-0117		0.100	2.420	7.090	0.125	1.254
18-0118		0.100	2.480	6.960	0.134	1.339

3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
3.015	3.990	0.054	0.537
2.950	4.410	0.042	0.424

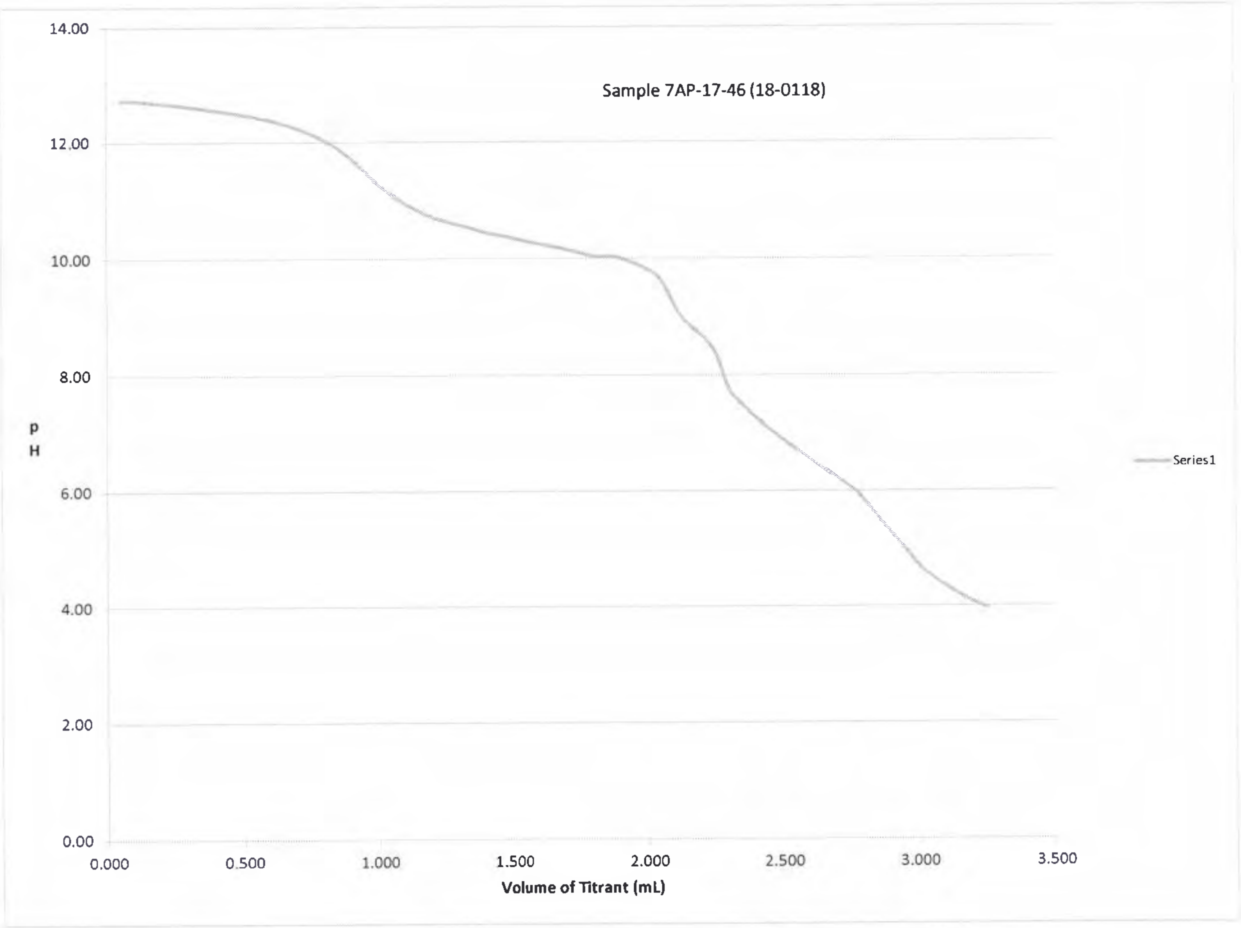
18-0117
7AP-17-11

Titrant Volume (mL)	pH
0.055	12.73
0.130	12.70
0.185	12.67
0.260	12.66
0.320	12.57
0.385	12.52
0.450	12.49
0.515	12.44
0.575	12.39
0.620	12.32
0.655	12.26
0.730	12.14
0.795	11.99
0.870	11.77
0.940	11.47
1.030	11.13
1.090	10.93
1.130	10.82
1.170	10.75
1.215	10.66
1.285	10.55
1.320	10.50
1.380	10.42
1.465	10.34
1.555	10.24
1.615	10.17
1.675	10.09
1.770	9.96
1.820	9.95
2.000	9.56
2.050	9.53
2.105	9.21
2.150	8.84
2.200	8.81
2.265	7.78
2.320	7.55
2.420	7.08
2.495	6.83
2.570	6.58
2.665	6.26
2.800	5.95
2.935	5.52
3.015	4.52
3.065	4.28
3.120	4.16
3.170	3.99
3.245	3.84
3.345	3.69



18-0118
7AP-17-46

Titrant Volume (mL)	pH
0.055	12.73
0.105	12.72
0.155	12.70
0.240	12.66
0.310	12.62
0.380	12.57
0.440	12.53
0.505	12.48
0.595	12.39
0.685	12.27
0.755	12.14
0.820	11.99
0.860	11.87
0.900	11.72
0.955	11.48
0.995	11.30
1.050	11.11
1.100	10.95
1.150	10.83
1.185	10.75
1.230	10.67
1.300	10.58
1.355	10.51
1.390	10.46
1.455	10.40
1.535	10.31
1.615	10.24
1.660	10.20
1.750	10.10
1.810	10.03
1.890	10.02
2.000	9.81
2.055	9.60
2.125	9.00
2.215	8.61
2.260	8.30
2.305	7.73
2.365	7.43
2.410	7.23
2.480	6.96
2.535	6.76
2.605	6.53
2.650	6.38
2.720	6.16
2.785	5.91
2.860	5.49
2.950	5.00
3.010	4.66
3.080	4.41
3.175	4.13
3.250	3.97



Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Gamma Energy Analysis (GEA)

Project / WP#: 69832/ N96051
ASR#: 0395.01
Client: SK Fiskum
Total Samples: 2

RPL ID	Client Sample ID
18-0117	7AP-17-11
18-0118	7AP-17-46

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per RPG-CMC-129, Rev. 0 *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☒ Other: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 3 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	None
Analysis Date or Date Range:	November 11, 2017
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*18-0117 Fiskum.xls*
ASO Project 98620 File:	*File Plan 5871, T4.4 Technical (Radiochemistry), Gamma Calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.*
M&TE Number(s):	*Detectors E & L*

T Trang-le / 12/7/2017
Prepare **Date**

LRGreenwood / 12/7/2017
Reviewer **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in this sample are presented in an attached Excel spreadsheet for ASR 0395.01. All sample results for target isotopes are reported in units of µCi/mL with estimates of the total propagated uncertainty reported at the 1-sigma level.

ASO Project File, ASR 0395.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, standard certificates, laboratory bench records, Shielded Analytical Laboratory Bench Sheet, and Gamma Energy Analysis printouts. Detector calibration records, control charts and balance calibration records can be found in the ASO Records.

Sample Preparation, Separation, Mounting and Counting Methods

Both samples were prepared for gamma counting by dilution 0.1 mL of parent sample with 10 mL of diluent. 2 mL of each dilution was sent to the counting room for GEA.

The quality control (QC) steps for direct GEA are discussed below.

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

No process blank was prepared for gamma counting.

Required Detection Limits

There are no required detection limits for these samples.

Blank Spike (BS)/Laboratory Control Sample (LCS)/ Matrix Spike (MS):

There are no BS, LCS or MS samples analyzed for ASO GEA analyses. Instrument performance is assessed by the analyses of daily control counts and weekly background counts, as discussed below.

Duplicate Relative Percent Difference (RPD):

No duplicate samples were provided for gamma counting.

Instrument Calibration and Quality Control

Gamma detectors are calibrated using multi-isotope standards that are NIST-traceable and prepared in the identical counting geometry to all samples and detectors. Counter control sources containing Am-241, Cs-137 and Co-60 are analyzed daily before the use of each detector. Procedure RPG-CMC-450 requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count.

Assumptions and Limitations of the Data

None

Interferences/Resolution

None.

Uncertainty

For gamma counting, the uncertainty in the counting data, photon abundance and the nuclear half-life, and efficiency are included in the calculation of the total uncertainty along with a systematic uncertainty for sample prep. The Canberra Genie software includes both random and systematic uncertainties in the calculation of the total uncertainties which are listed on the report. We conservatively estimate that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

Comments

None

Attachment: Data Report Sample Results for ASR 0395.01.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Radiochemical Sciences and Engineering Group

filename 18-0117 Fiskum.xls
12/7/2017

Client: Fiskum
ASR 0395.01

Project: 69832
WP: N96051

Prepared by: T Trang-le 12-7-2017

Technical Reviewer: LRGreenwood 12-7-2017

Procedures: RPG-CMC-450, Rev. 3, Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)

M&TE: Gamma detectors E,L
Count dates: 27-Nov-17

		Measured Activity, µCi per ml ± 1s				
Sample	Lab ID	**Cs-137**		**Co-60**		**Am-241**
7AP-17-11	18-0117	**1.54E+02**	**± 2%**	**4.16E-03**	**± 20%**	<3.5E-01
7AP-17-46	18-0118	**1.59E+02**	**± 2%**	**8.70E-04**	**± 19%**	<3.2E-01

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature Sandra k Fiskum
Print Name Sandra Fiskum
Phone 375-5677 MSIN P7-25

Project Number: 71274
Work Package: N96051

Matrix Type Information

- **Liquids:** ☑ Aqueous ☐ Organic ☐ Multi-phase
- **Solids:** ☐ Soil ☐ Sludge ☐ Sediment
  ☐ Glass ☐ Filter ☐ Metal
  ☐ Smear ☐ Organic ☐ Other
- **Other:** ☐ Solid/Liquid Mixture, Slurry
  ☐ Gas ☐ Biological Specimen

(If sample matrices vary, specify on Request Page)

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc: ____
- **Disposition of Treated Samples:**
  ☑ Dispose ☐ Return

QA/Special Requirements

- **QA Plan:**
  ☑ ASO-QAP-001 (Equivalent to HASQARD)
  ☐ Additional QA Requirements, List Document Below:
  Reference Doc Number: ____
- **Field COC Submitted?** ☑ No ☐ Yes
- **Lab COC Required?** ☑ No ☐ Yes
- **Sample/Container Inspection Documentation Required?**
  ☑ No ☐ Yes
- **Hold Time:** ☑ No ☐ ***Yes***
  If *Yes*, Contact ASO Lead before submitting Samples
  ☐ Use SW 846 (PNL-ASO-071, identify analytes/methods where holding times apply)
  ☐ Other? Specify: ____
- **Special Storage Requirements:**
  ☑ None ☐ Refrigerate ☐ Other, Specify: ____
- **Data Requires ASO Quality Engineer Review?** ☐ No ☑ Yes

Data Reporting Information

- **Is Work Associated with a Fee-Based Milestone?** ☑ No ☐ Yes
  If yes, milestone due date: ____
- **Preliminary Results Requested, As Available?** ☐ No ☑ Yes

- **Data Reporting Level**
  ☑ ASO-QAP-001 (Equivalent to HASQARD).
  ☐ Minimum data report.
  ☐ Project Specific Requirements: Contact ASO Lead or List Reference Document: ____

- **Requested Analytical Work Completion Date:** 2/27/18
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:** 2/27/18
  (To be completed by ASO Lead)

Waste Designation Information

- **ASO Sample Information Check List Attached?** ☐ No ☑ Yes
  If no, Reference Doc Attached: ____
  or, Previous ASR Number: ____
  or, Previous RPL Number: ____

Does the Waste Designation Documentation Indicate Presence of PCBs?
☑ **No** ☐ **Yes**

Send Report To: SK Fiskum **MSIN** P7-25
MSIN ____

Additional or Special Instructions ____

Receiving and Login Information *(to be completed by ASO staff)*

Date Delivered: 2/15/18
Delivered By (optional) ____
Time Delivered: 2:50 pm
Group ID (optional) ____
CMC Waste Sample? ☑ No ☐ Yes

Received By: T Trang-Le
ASR Number: 0457 Rev.: 0
RPL Numbers: 18-0832 → 18-0834
(first and last)

ASO Work Accepted By: KNPool **Signature/Date:** K N Pool 2/15/18

Analytical Service Request (ASR)

(REQUEST PAGE ----- Information Specific to Individual Samples)

Provide Analytes of Interest and Required Detection limits - ☐ Below ☐ Attached

ASO Staff Use Only				ASO Staff Use Only	
RPL Number	Customer Sample ID	Sample Description (& Matrix, if it varies)	Analysis Requested	Test	Library
18-0832	7AP-17-11-Cs	Cs component from AP-107	ICP-MS mass fractionation		
		tank waste in 0.45 M	of Cs (^{133}Cs, ^{135}Cs, ^{137}Cs)		
18-0833	7AP-17-46-Cs	HNO_3	↓		
18-0834	C2891	5.6 M Na Simple simulant	ICP MS: Cs-133		
		with 8 ppm Cs			

ASR # 0457 Rev.: 0

Page 1 of 1

Exhibit 1. Example ASO Sample Information Checklist (SICL) Form

Sample Location, Owner and Hazard Description Information

To be completed by the sample custodian relinquishing the sample(s) and based on best available information

ASO Customer Information:

Company: PNNL

Project #: 71274

Point of Contact (name, telephone#): SK Fiskum 375-5677

Comments:

C2891

Sample Description (medium, collection location, known contaminants, purpose of sample collection):

Sample Collection Date: 2/14/18 Sample Collection Time: —

Is the sample known to be radioactive? ☐ Yes ☑ No

Comments *(list known isotopes)*:

Is the sample known to contain or have come in contact with PCBs? ☐ Yes ☑ No

List any hazardous sample constituents known to be present:

Constituent/Chemical	**Concentration**	**Comment**
hydroxide	1.41 M	
Na	5.6 M	
nitrate	1.78 M	
nitrite	1.02 M	
phosphate	0.043 M	
Sulfate	0.066 M	

Are any other comments applicable to sample receipt, storage, handling, or disposition?

No RCRA metals present

Checklist Prepared By:

SK Fiskum SK Fiskum 2/12/18

Printed Name Signature Date

Exhibit 1. Example ASO Sample Information Checklist (SICL) Form

Sample Location, Owner and Hazard Description Information

To be completed by the sample custodian relinquishing the sample(s) and based on best available information

ASO Customer Information:

Company: PNNL
Project #: 71274
Point of Contact (name telephone#): SK Fiskum 375-5677

Comments: TAP-17-11-Cs
TAP-17-46-Cs

Sample Description (medium, collection location, known contaminants, purpose of sample collection): 0.45M HNO_3

Sample Collection Date: 2/12/2018 Sample Collection Time: –

Is the sample known to be radioactive? ☑ Yes ☐ No

Comments *(list known isotopes)*: Cs-137, Cs-135

Is the sample known to contain or have come in contact with PCBs? ☐ Yes ☑ No

List any hazardous sample constituents known to be present:

Constituent/Chemical	**Concentration**	**Comment**
Not known		

Are any other comments applicable to sample receipt, storage, handling, or disposition?

Corrosive 0.45M HNO_3

Checklist Prepared By:

SK Fiskum Sandra k Fisk 2/12/18
Printed Name Signature Date

Battelle PNNL/RPL/Inorganic Analysis … ICP-MS Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	71274 / N96051
ASR#:	0457
Client:	S. Fiskum
Total Samples:	3 (Aqueous)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0832	7AP-17-11-Cs	Cs component from AP-107 tank waste	NA
18-0833	7AP-17-46-Cs	Cs component from AP-107 tank waste	NA
18-0834	C2891	5.6 M Na Simple Simulant with 8 ppm Cs	NA

Sample Preparation: Sample dilution in 2% v/v HNO_3 performed by G. Brown on 02/16/18.

Procedure: RPG-CMC-292, Rev. 1, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."

Analyst:	G. Brown	**Analysis Date:**	02/16/2018	**ICP File:**	M0056-71A

See Chemical Measurement Center 98620 file: ICP-325-405-3 (Calibration and Maintenance Records)

M&TE:	☒	PerkinElmer NexION^{TM} 350X ICP-MS	SN: 85VN4070702	RPL 405
	☒	Sartorius R200D Balance	SN: 39080042	RPL 405
	☒	Mettler AT400 Balance	SN: M19445	RPL 405 FH
	☐	Mettler AT400 Balance	SN: 1113292667	RPL 420 FH
	☐	Ohaus EX324 Balance	SN: 8033311209	SAL Cell 2
	☐	Sartorius BA3105 Balance	SN: 10803210	RPL 309
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH

Garrett N. Brown
Report Preparer

4/13/18
Date

Review and Concurrence

4/13/18
Date

Three aqueous samples submitted under Analytical Service Request (ASR) 0457 were analyzed by ICP-MS. Prior to analysis, all samples were diluted in 2% HNO_3. None of the samples were filtered.

All results are reported on a mass per unit volume basis (ng/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-MS Data Report. The first two samples for the ASR (18-0832 and 18-0833) requested cesium-133, cesium-135, and cesium-137. The third sample from this ASR (18-0834) requested cesium 133 only. The quality control (QC) results for the AOI have been evaluated and are presented below.

Calibration of the ICP-MS was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards were used to verify acceptance of the six-point calibration curves and for initial and continuing calibration verification (ICV/CCV).

The controlling document was procedure RPG-CMC-292, Rev. 1, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS).* Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spike, duplicate, blank spike, and serial dilution were conducted during the analysis run.

Internal Standard (IS):

All solutions (blanks, standards, QC checks, and samples) were mixed in-line with a solution containing 10 ppb each of Li-6, Sc-45, Y-89, In-115, Tb-159, and Bi-209 as the internal standard (IS). The AOI (Cs-133, Cs-135, Cs-137) data were normalized using the data for the closest IS mass (e.g., In-115). The In-115 IS recoveries ranged from 99.3% to 107%, which were within the acceptance criterion of 30% to 120%.

Preparation Blank (PB):

A diluent blank from the ICP-MS laboratory (2% HNO_3) was analyzed as a preparation blank. Results for the diluent blank were within the acceptance criteria of <EQL (estimated quantitation level), <50% regulatory decision level, or less than ≤10% of the concentration in the samples.

Blank Spike (BS)/Laboratory Control Sample (LCS):

A blank spike (BS) sample was prepared by spiking a 2% HNO_3 blank with an equivalent volume of the CCV-71A-2ppb standard (1:1 ratio). The recoveries for the AOI ranged from 100% to 103%, which were within the acceptance criteria of 80% to 120% recovery.

Replicate Relative Percent Difference (RPD):

Replicates of samples 18-0832, 18-0833, and 18-0834 were prepared and analyzed. RPD's are listed for all analytes that were measured at or above the EQL. RPD's for the AOI meeting this requirement ranged from 0.2% to 1.7% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

The samples were prepared by simple dilution as such a matrix spike sample is not prepared as simple dilution is not a sample preparation method. An analytical spike was run with the samples and recovery information noted below.

Initial/Continuing Calibration Verification (ICV/CCV):

The ICV/CCV solution was analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (2% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL. Following an additional rinse time, the final CCB met the acceptance criteria.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 70% to 130% recovery.

Interference Check Standard (ICS):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-0833. Percent differences (%D) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 0.3% to 1.9% and were within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS-71A)/Analytical Spike (AS-71A) - Sample (71A Component):

Because no MS sample was required to be prepared, a post-digestion spike (PS-71A) was conducted on samples 18-0834. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 99% to 102%, respectively, and were within the acceptance criterion of 80% to 120%.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike 71A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, K, La, Lu, Mg, Mn, Na, Nd, Ni, P, Pb, Pr, Rb, S, Se, Sm, Sr, Th, Tl, Tm, U, V, Yb, and Zn. Analytes included in the spike 71B component are; Ge, Hf, Mo, Nb, Sb, Si, Sn, Ta, Te, Ti, W, and Zr. Analytes included in the spike 71C component are; Ir, Os, Pd, Pt, Re, Rh, and Ru. Analytes included in the spike 71D component are; Bi, In, Li, Sc, Tb, and Y. Analytes included in the spike Hg component are; Hg.

Battelle PNNL/RPG/Inorganic Analysis ... ICP-MS Data Report

		Run Date >	2/16/2018	2/16/2018	2/16/2018	2/16/2018	2/16/2018	2/16/2018	2/16/2018
		Process Factor >	1.0000	1,956.8	1,956.8	1,951.8	1,951.8	95,010.4	95,010.4
		RPL/LAB >	2% HNO_3 Diluent	18-0832 @ 2,000x	18-0832 @ 2,000x rep	18-0833 @ 2,000x	18-0833 @ 2,000x rep	18-0834 @ 100,000x	18-0834 @ 100,000x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	2% HNO_3 Diluent	7AP-17-11-Cs		7AP-17-46-Cs		C2891	
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0003	0.003	Cs 133	--	588	578	576	577	8015	7916
0.0028	0.028	Cs 135	--	163	162	157	159	NR	NR
0.0016	0.016	Cs 137	--	201	197	193	196	NR	NR

Internal Standard % Recovery

Li 6 (IS)	101%	102%	98%	98%	95%	96%	99%
Sc 45 (IS)	104%	101%	103%	102%	100%	99%	103%
Y 89 (IS)	101%	100%	97%	99%	97%	102%	101%
In 115 (IS)	102%	100%	102%	102%	102%	100%	101%
Tb 159 (IS)	101%	98%	98%	100%	99%	98%	98%
Bi 209 (IS)	100%	99%	101%	97%	96%	99%	101%

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

NR = not requested on ASR.

QC Performance 2/16/2018

Criteria >	≤ 20%	≤ 20%	≤ 20%	80%-120%	75%-125%	≤ 10%
QC ID >	18-0832 Rep	18-0833 Rep	18-0834 Rep	LCS/BS	AS 18-0834 + CCV71A	18-0833 5-fold Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec	%Diff
Cs 133	1.7%	0.2%	1.2%	103%	99%	0.9%
Cs 135	0.2%	1.1%	NR	100%	102%	1.0%
Cs 137	1.7%	1.5%	NR	100%	102%	1.9%

Internal Standard % Recovery

Li 6 (IS)	98%	95%	99%	101%	99%	91%
Sc 45 (IS)	103%	100%	103%	101%	101%	103%
Y 89 (IS)	97%	97%	101%	102%	100%	95%
In 115 (IS)	102%	102%	101%	102%	102%	102%
Tb 159 (IS)	98%	99%	98%	101%	100%	101%
Bi 209 (IS)	101%	96%	101%	100%	99%	97%

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na2O2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

NR = not requested on ASR.

Analytical Service Request (ASR)

(Information on this COVER PAGE is applicable to all samples submitted under this ASR)

Requestor --- Complete all fields on this COVER PAGE, unless specified as optional or ASR is a revision

Requestor:
Signature Sandra k. Fiskum
Print Name SANDRA FISKUM
Phone 55677 MSIN

Project Number: 71274
Work Package: N96056

Matrix Type Information

- **Liquids:** ☑ Aqueous ☐ Organic ☐ Multi-phase
- **Solids:** ☐ Soil ☐ Sludge ☐ Sediment ☐ Glass ☐ Filter ☐ Metal ☐ Smear ☐ Organic ☐ Other
- **Other:** ☐ Solid/Liquid Mixture, Slurry ☐ Gas ☐ Biological Specimen

(If sample matrices vary, specify on Request Page)

Disposal Information

- **Disposition of Virgin Samples:**
  Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
  If archiving, provide:
  Archiving Reference Doc:
- **Disposition of Treated Samples:**
  ☑ Dispose ☐ Return

QA/Special Requirements

- **QA Plan:**
  ☑ ASO-QAP-001 (Equivalent to HASQARD)
  ☐ Additional QA Requirements, List Document Below:
  Reference Doc Number:
- **Field COC Submitted?** ☑ No ☐ Yes
- **Lab COC Required?** ☑ No ☐ Yes
- **Sample/Container Inspection Documentation Required?**
  ☑ No ☐ Yes
- **Hold Time:** ☑ No ☐ ***Yes***
  If *Yes*, Contact ASO Lead before submitting Samples
  ☐ Use SW 846 (PNL-ASO-071, identify analytes/methods where holding times apply)
  ☐ Other? Specify:
- **Special Storage Requirements:**
  ☑ None ☐ Refrigerate ☐ Other, Specify:
- **Data Requires ASO Quality Engineer Review?** ☐ No ☒ Yes

Data Reporting Information

- **Is Work Associated with a Fee-Based Milestone?** ☑ No ☐ Yes
  If yes, milestone due date:
- **Preliminary Results Requested, As Available?** ☐ No ☑ Yes
- **Data Reporting Level**
  ☑ ASO-QAP-001 (Equivalent to HASQARD).
  ☐ Minimum data report.
  ☐ Project Specific Requirements: Contact ASO Lead or List Reference Document:
- **Requested Analytical Work Completion Date:** June 22, 2018
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:** 6/29/18
  (To be completed by ASO Lead)

Waste Designation Information

- **ASO Sample Information Check List Attached?** ☐ No ☒ Yes
  If no, Reference Doc Attached:
  or, Previous ASR Number:
  or, Previous RPL Number:

Does the Waste Designation Documentation Indicate Presence of PCBs?
☑ **No** ☐ **Yes**

Send Report To: SK Fiskum, AM Rovira **MSIN**
MSIN
Additional or Special Instructions See attached special instructions

Receiving and Login Information *(to be completed by ASO staff)*

Date Delivered: 4/24/18
Delivered By (optional)
Time Delivered: 3:20 pm
Group ID (optional)
CMC Waste Sample? ☒ No ☐ Yes

Received By: T Trang-Le
ASR Number: 0521 Rev: 01
RPL Numbers: 18-1604 to 18-1619
(first and last)

ASO Work Accepted By: DL Blanchard **Signature/Date:** 4-24-2018

Analytical Services Request (ASR)

(REQUEST PAGE ---- Information Specific to Individual Samples)

Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**

ASO Staff Use Only **RPL Number**	Client Sample ID	Sample Description (& Matrix if varies)	Analyses Requested	ASO Staff Use Only **Test**	ASO Staff Use Only **Library**
	Revision 1 - ASR Revised to correct the client sample ID for 18-1616				
18-1604	TI042-COMP-FEED	All Samples are present in approximately 5.6 M Na solution of Hanford Tank Waste Matrix The analyte list and requested detection limits for these three samples is also detailed in Table 1 of the attached special instructions. *Note: I-129 will be performed on these samples at a later date.*	1) GEA - All samples (Co-60, Cs-137 and Eu-154 and any other observed gamma emitting isotopes) 2) Tritium 3) Tc-99 4) IC-Anions - Cl, NO_2, NO_3, PO_4, C_2O_4 and SO_4 5) TOC/TIC - Hot Pursulfate 6) OH 7) Acid Digestion- 128 - Prep Lab a) ICP/OES - Ag, Al, As, B, Ba, Ca, Cd, Cr, Fe, K, Li, Na, Ni, P, Pb, Se, Sr, Th, Ti, U, Zn, Zr b) ICP/MS - Hg, Sn-126, U-238 c) Total Alpha d) Total Beta e) Sr-90 f) Np-AEA, Np-237 g) Pu-AEA, Pu-238, Pu-239/240 h) Am/Cm-AEA-241, Cm-243+244		
18-1605	TI042-COMP-EFF				
18-1606	TI042-COMP-EFF-DUP				

Analytical Services Request (ASR)

(REQUEST PAGE ---- Information Specific to Individual Samples)

Provide Analytes of Interest and Required Detection limits - ☐ **Below** ☐ **Attached**

ASO Staff Use Only **RPL Number**	Client Sample ID	Sample Description (& Matrix if varies)	Analyses Requested	ASO Staff Use Only **Test**	**Library**
18-1607	TI042-L-F2-A	All Samples are present in approximately 5.6 M Na solution of Hanford Tank Waste Matrix The analyte list and requested detection limits for these three samples is also detailed in Table 2 of the attached special instructions.	1) Acid Digestion - 128 - Prep Lab a) ICP/OES - Ba, Cr, Fe, K, Ni, Pb, Sr, Ti, Zn, Zr b) Sr-90 c) Pu-AEA - Pu-239/240		
18-1608	TI042-L-F3-A				
18-1609	TI042-L-F4-A				
18-1610	TI042-L-F5-A				
18-1611	TI042-L-F7-A				
18-1612	TI042-L-F9-A				
18-1613	TI042-L-F11-A				
18-1614	TI042-L-F13-A				
18-1615	TI042-L-F15-A				
18-1616	TI042-L-F18-A				
18-1617	TI036-S0-CST-F	All Samples are present in approximately 5.6 M Na solution of Hanford Tank Waste Matrix The analyte list and requested detection limits for these three samples is also detailed in Table 3 of the attached special instructions.	1) Acid Digest - 128 - Prep Lab a) ICP/OES - Ba, Cr, Fe, K, Ni, Pb, Sr, Ti, Zn, Zr		
18-1618	TI036-S2-CST-F				
18-1619	TI036-S3-CST-F				

Special Instructions for ASR 0521

Analysis of AP-107 diluted feed, effluent, and selected samples from column and batch contact processing. All samples are caustic and have 5.6 M Na.

Use the ASO QA Plan, ASO-QAP-001, current revision to apply batch processing and instrument QC.

IX Test	Client ID	ASO Sample ID	Location for Pickup	Analysis
Column	TI042-COMP-FEED	18-1604	SAL cell 5	Table 1
	TI042-COMP-EFF	18-1605	Lab 305	
	TI042-COMP-EFF-DUP	18-1606	Lab 305	
Column	TI042-L-F2-A	18-1607	Lab 305	Table 2
	TI042-L-F3-A	18-1608		
	TI042-L-F4-A	18-1609		
	TI042-L-F5-A	18-1610		
	TI042-L-F7-A	18-1611		
	TI042-L-F9-A	18-1612		
	TI042-L-F11-A	18-1613		
	TI042-L-F13-A	18-1614		
	TI042-L-F15-A	18-1615		
	TI042-L-F18-A	18-1616		
Batch Contact	TI036-S0-CST-F	18-1617	Lab 305	Table 3
	TI036-S2-CST-F	18-1618		
	TI036-S3-CST-F	18-1619		

The high salt samples are assumed to require acid digestion for most analytes. However if the preparative technique sufficiently dilutes the sample into required acid matrix, then acid digestion may be omitted. Note that the MDLs listed in Tables 1-3 should generally not pose an issue as the analytes are anticipated to be in relative high concentrations.

Preparative QC samples per preparative batch expected for this suite include:

1. Process blank (digestion blank or diluent blank); not applicable to GEA
2. Duplicate (one duplicate per analytical batch; if dilution is the only preparation, please prepare a diluent duplicate); not applicable to GEA
3. Blank spike (BS), laboratory control sample (LCS), or reagent spike (RS); not applicable to GEA
4. Matrix spike
    a. Not needed for major analytes (e.g., Na and NO_3 in the TI042-COMP-FEED and TI042-COMP-EFF samples). In the case where analyte spikes are not used, measure post spikes instead per ASO QA Plan
    b. Not applicable to GEA

Table 1. Column Feed Composite and Effluent Composite Analyte List

Analyte	Target MDL	Analysis Method
^{60}Co	1E-5 µCi/mL	GEA
^{137}Cs	1E-5 µCi/mL	
^{154}Eu	1E-5 µCi/mL	
^{237}Np	1E-6 µCi/mL	Acid Digestion Separations/Alpha Energy Analysis (AEA)
^{238}Pu	1E-6 µCi/mL	
$^{239+240}$Pu	1E-6 µCi/mL	
^{241}Am	1E-6 µCi/mL	
$^{243+244}$Cm	1E-6 µCi/mL	
^{3}H	1E-5 µCi/mL	Separations/Beta Scintillation Counting
^{90}Sr	1E-4 µCi/mL	Acid Digestion Separations/Beta Scintillation Counting
^{99}Tc	1E-4 µCi/mL	ICP-MS or Separations/Beta Counting
^{129}I	*1E-4 µCi/mL*	*To be analyzed at a later date, pending method development*
Total Alpha	1E-4 µCi/mL	Acid Digestion Alpha Scintillation or Gas Proportional Counting
Total Beta	1E-4 µCi/mL	Acid Digestion Beta Scintillation or Gas Proportional Counting
^{126}Sn		Acid Digestion ICP-MS
Hg		
^{238}U		
Ag	100 µg/mL	Acid Digestion ICP-OES
Al	1000 µg/mL	
As	100 µg/mL	
B	100 µg/mL	
Ba	1 µg/mL	
Ca	10 µg/mL	
Cd	10 µg/mL	
Cr	10 µg/mL	
Fe	10 µg/mL	
K	100 µg/mL	
Li	10 µg/mL	
Na	1000 µg/mL	
Ni	10 µg/mL	
P	10 µg/mL	
Pb	50 µg/mL	
Se	100 µg/mL	
Sr	1 µg/mL	
Th	10 µg/mL	
Ti	10 µg/mL	
U (total)	10 µg/mL	
Zn	10 µg/mL	
Zr	10 µg/mL	

Table 1. Continued

Analyte	Target MDL	Analysis Method
Cl^-	200 µg/mL	Dilution Ion Chromatography (IC)
NO_2^-	200 µg/mL	
NO_3^-	200 µg/mL	
PO_4^{3-}	200 µg/mL	
$C_2O_4^{2-}$	200 µg/mL	
SO_4^{2-}	200 µg/mL	
Total organic carbon (TOC)	200 µg/mL	Hot Persulfate Oxidation
Total inorganic carbon (TIC)	200 µg/mL	
Free Hydroxide	0.1 M	Titration

Table 2. Column Sample Analyte List

Analyte	Target MDL	Analysis Method
$^{239+240}Pu$	1E-6 µCi/mL	Acid Digestion Separations/Alpha Energy Analysis (AEA)
^{90}Sr	1E-4 µCi/mL	Acid Digestion Separations/Beta Scintillation Counting
Ba	1 µg/mL	Acid Digestion Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
Cr	10 µg/mL	
Fe	10 µg/mL	
K	10 µg/mL	
Ni	10 µg/mL	
Pb	50 µg/mL	
Sr	1 µg/mL	
Ti	10 µg/mL	
Zn	10 µg/mL	
Zr	10 µg/mL	

Table 3. Batch Contact Sample Analyte List

Analyte	Target MDL	Analysis Method
Ba	1 µg/mL	Acid Digestion Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
Cr	1 µg/mL	
Fe	10 µg/mL	
K	10 µg/mL	
Ni	10 µg/mL	
Pb	50 µg/mL	
Sr	1 µg/mL	
Ti	10 µg/mL	
Zn	10 µg/mL	
Zr	10 µg/mL	

Exhibit 1. Example ASO Sample Information Checklist (SICL) Form

Sample Location, Owner and Hazard Description Information

To be completed by the sample custodian relinquishing the sample(s) and based on best available information

ASO Customer Information:

Company: PNNL
Project #: 71274
Point of Contact (name, telephone#): SK Fiskum

Comments:

Sample Description (medium, collection location, known contaminants, purpose of sample collection):

Sample Collection Date: April 2018 Sample Collection Time: NA

Is the sample known to be radioactive? ☒ Yes ☐ No

Comments *(list known isotopes)*:

AP-107 tank waste

Is the sample known to contain or have come in contact with PCBs? ☐ Yes ☒ No

List any hazardous sample constituents known to be present:

Constituent/Chemical	**Concentration**	**Comment**
CrO_4^-		oxidizer
OH^-		corrosive
NO_3^-		oxidizer
NO_2^-		oxidizer

Are any other comments applicable to sample receipt, storage, handling, or disposition?

Samples range from low ^{137}Cs concentrations to 154 µCi/mL.

Checklist Prepared By:

SK Fiskum SK Fiskum 4/24/2018
Printed Name Signature Date

Battelle PNNL/RPL/Inorganic Analysis ... ICP-OES Analysis Report

PO Box 999, Richland, Washington 99352

Project / WP#: 71274 / N96056
ASR#: 0521.01
Client: S. Fiskum
Total Samples: 16 (liquids)
Sample Description AP-107 Tank Waste

ASO Sample ID	Client Sample ID	Sample Weight (g)	ASO Sample ID	Client Sample ID	Sample Weight (g)
18-1604	TI042-COMP-FEED	NA	18-1612	TI042-L-F9-A	NA
18-1605	TI042-COMP-EFF	NA	18-1613	TI042-L-F11-A	NA
18-1606	TI042-COMP-EFF-DUP	NA	18-1614	TI042-L-F13-A	NA
18-1607	TI042-L-F2-A	NA	18-1615	TI042-L-F15-A	NA
18-1608	TI042-L-F3-A	NA	18-1616	TI042-L-F18-A	NA
18-1609	TI042-L-F4-A	NA	18-1617	TI036-S0-CST-F	NA
18-1610	TI042-L-F5-A	NA	18-1618	TI036-S2-CST-F	NA
18-1611	TI042-L-F7-A	NA	18-1619	TI036-S3-CST-F	NA

Sample Preparation: RPG-CMC-128, Rev. 1. "HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater", performed by L. Darnell on 05/11/18 and 05/14/18. Simple dilution of "as received" samples in 5% v/v HNO_3 performed by J. Carter on 05/18/18.

Procedure: RPG-CMC-211, Rev. 4, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)."

Analyst: J. Carter **Analysis Date:** 05/18/18 **ICP File:** C0755

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE:	☒	PerkinElmer 5300DV ICP-OES	SN: 077N5122002
	☒	Mettler AT400 Balance	SN: 1113292667
	☒	Ohaus PA224C Balance	SN: B725287790
	☐	Sartorius R200D Balance	SN: 39080042
	☐	SAL Cell 2 Balance	SN: 8033311209

Report Preparer 6-20-18 Date

Review and Concurrence 6/20/18 Date

Sixteen aqueous samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed by ICP-OES. The samples were prepared following RPL procedure RPG-CMC-128, Rev. 1, "*HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*". All samples were diluted in 5% HNO_3 prior to analysis. None of the samples were filtered.

All sample results are reported on a mass per unit volume basis (µg/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-OES Data Report. There were two analyte lists requested, one being a shortened version. Samples were reported using the most extensive analyte list. The quality control (QC) results for the AOI have been evaluated and are presented below. Analytes other than the AOI are reported in the bottom section of the report but have not been fully evaluated for QC performance.

Calibration of the ICP-OES was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards (MCVA and MCVB) were used to verify acceptance of the two-point calibration curves obtained for each analyte and for continuing calibration verification.

The controlling documents were procedures RPG-CMC-211, Rev. 4, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Optical Emission Spectrometry (ICP-OES)*, and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan*. Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), matrix spike, post-digestion spikes, duplicate, reagent spike, blank spike, and serial dilution were conducted during the analysis run.

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. Three analytes (Ba, Ca and Zn) were above EQL, but well below the target MDL requested for each. Remaining analytes were within the acceptance criteria of <EQL (estimated quantitation level), ≤50% regulatory decision level, or ≤10% of the concentration in the samples. The lab diluent passed these criteria.

Blank Spike (BS)/Laboratory Control Sample (LCS):

A reagent spike (RS) sample (reagents and spikes) was prepared for the extraction process. Recovery values are listed for all analytes included in the RS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 100% to 112%, and were within the acceptance criterion of 80% to 120%. Ag failed the recovery criteria at 30%. The reason for the low silver recovery is unknown. However, the silver spike is added separately from the remaining elements and may indicate reagent instability.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

A duplicate of sample 18-1606 was prepared and analyzed. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for the AOI meeting this requirement ranged from 0.9% to 5.4% and were within the acceptance criterion of ≤20% for liquid samples, with the exception of Ca (28.3%).

Matrix-Spike (MS) Sample:

A matrix spike (MS) of sample 18-1606 was prepared for the extraction process. Recovery values are listed for all analytes included in the MS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement ranged from 29% to 115%, and were within the acceptance criterion of 75% to 125%, with the exception of Ag (29%).

Initial/Continuing Calibration Verification (ICV/CCV):

MCVA and MCVB solutions were analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery, with the exception of lithium (114% and 115%) in the final two CCV solutions.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (5% v/v HNO_3) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than ten samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL, with the exception of sodium in the final three CCB solutions (0.141, 0.101, & 0.154ppm).

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 70% to 130% recovery.

Interference Check Standard (ICS/SST):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-1606. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 1.2% to 8.2% and were within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS-A) - Sample (A Component):

In addition to the BS sample, a post-digestion spike (A Component) was conducted on sample 18-1606. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 93% to 109%, and were within the acceptance criterion of 80% to 120%.

Post-Digestion Spike (PS-B) - Sample (B Component):

In addition to the MS sample, a post-digestion spike (B Component) was conducted on sample 18-1606. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement ranged from 95% to 100%, and were within the acceptance criterion of 80% to 120%.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 5% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ta, Ti, Tl, V, W, Y, Zn, and Zr. Analytes included in the spike B component are; Ce, Dy, Eu, La, Nd, Pd, Rh, Ru, S, Te, Th, and U.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018
		Process Factor >	1.0	49.8	246.9	247.7	253.3	249.4	247.0	249.3	239.7	243.0
			405 diluent	BLK-1604	18-1604 @ 5x	18-1605 @ 5x	18-1606 @ 5x	DUP-1606 @ 5x	18-1607 @ 5x	18-1608 @ 5x	18-1609 @ 5x	18-1610 @ 5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	Prep Blank	TI042-Comp-Feed	TI042-Comp-Eff	TI042-Comp-Eff-Dup		TI042-L-F2-A	TI042-L-F3-A	TI042-L-F4-A	TI042-L-F5-A
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0014	0.014	Ag	[0.0020]	--	--	--	--	--	--	--	--	--
0.0038	0.038	Al	[0.0038]	[0.79]	9,850	9,450	9,370	9,840	9,570	9,250	9,390	9,590
0.0383	0.383	As	[0.044]	--	[11]	--	--	--	--	--	[13]	--
0.0032	0.032	B	[0.011]	[1.2]	34.6	31.1	27.9	28.2	57.5	73.9	60.8	59.9
0.0001	0.001	Ba	[0.0001]	0.0584	0.836	[0.20]	[0.075]	[0.14]	[0.17]	[0.17]	[0.094]	[0.092]
0.0054	0.054	Ca	[0.0074]	4.36	34.1	16.1	15.0	19.9	[12]	17.4	30.9	17.3
0.0016	0.016	Cd	--	--	6.62	5.28	5.21	4.94	4.91	5.29	5.28	5.05
0.0016	0.016	Cr	--	--	498	475	471	494	487	472	474	483
0.0033	0.033	Fe	--	[0.50]	19.4	12.9	12.2	13.1	12.8	12.8	12.7	14.2
0.0262	0.262	K	--	15.1	3,910	3,730	3,750	3,930	3,950	3,840	3,850	3,930
0.0010	0.010	Li	--	--	--	--	--	--	--	--	--	--
0.0075	0.075	Na	--	6.79	126,000	124,000	126,000	129,000	123,000	121,000	121,000	123,000
0.0040	0.040	Ni	--	--	21.0	21.0	20.6	20.9	20.6	20.0	19.6	20.5
0.0369	0.369	P	--	--	642	630	631	659	619	602	622	651
0.0144	0.144	Pb	--	--	[9.0]	--	--	--	--	--	--	--
0.0876	0.876	Se	--	--	--	--	--	--	--	--	--	--
0.0001	0.001	Sr	[0.0001]	[0.015]	0.331	[0.044]	[0.034]	[0.040]	[0.048]	[0.058]	[0.050]	[0.032]
0.0057	0.057	Th	--	[0.41]	[12]	--	--	--	--	--	--	--
0.0004	0.004	Ti	--	[0.032]	[0.80]	[0.61]	[0.56]	[0.69]	[0.69]	[0.61]	[0.51]	[0.56]
0.0312	0.312	U	--	--	[48]	[13]	[11]	[14]	[12]	--	[13]	[11]
0.0023	0.023	Zn	--	1.40	[1.8]	[1.3]	[1.2]	--	--	[1.4]	[0.56]	[1.2]
0.0013	0.013	Zr	--	[0.078]	[1.4]	[1.4]	[1.4]	[1.6]	[1.2]	[1.3]	[1.3]	[0.99]
Other Analytes												
0.0001	0.001	Be	--	--	0.192	[0.11]	[0.11]	[0.12]	[0.11]	[0.11]	[0.12]	[0.12]
0.0220	0.220	Bi	--	--	[7.4]	--	--	--	[8.0]	[8.5]	[7.3]	[10]
0.0052	0.052	Ce	--	--	--	--	--	--	--	--	--	--
0.0033	0.033	Co	--	--	--	--	--	--	[1.4]	--	--	--
0.0023	0.023	Cu	--	--	[0.84]	--	--	--	--	--	[0.58]	--
0.0012	0.012	Dy	--	--	[1.1]	--	--	--	--	--	--	--
0.0006	0.006	Eu	--	[0.042]	[1.2]	[0.19]	[0.20]	[0.18]	--	--	--	[0.22]
0.0010	0.010	La	--	--	[1.0]	--	--	--	--	--	--	[0.26]
0.0014	0.014	Mg	--	--	6.85	--	--	--	--	--	--	--
0.0003	0.003	Mn	--	--	--	--	--	--	--	--	--	--
0.0048	0.048	Mo	[0.0055]	--	43.0	40.5	41.0	42.4	41.4	40.9	40.8	40.4
0.0095	0.095	Nd	--	--	--	--	--	--	--	--	--	--
0.0082	0.082	Pd	--	--	[8.4]	[3.3]	[3.0]	[3.6]	[3.5]	[3.1]	[2.1]	[3.1]
0.0108	0.108	Rh	--	--	[6.1]	--	[6.4]	[4.1]	[3.2]	[6.7]	[4.4]	[5.8]
0.0068	0.068	Ru	[0.0087]	--	[12]	[9.9]	[9.5]	[7.1]	[7.7]	[7.6]	[8.6]	[8.7]
0.0898	0.898	S	--	[8.0]	1,670	1,650	1,660	1,720	1,650	1,600	1,630	1,640
0.0569	0.569	Sb	--	--	--	--	--	--	--	--	--	--
0.0043	0.043	Si	[0.011]	2.74	86.0	66.5	64.3	65.8	132	122	123	126
0.0195	0.195	Sn	--	--	[21]	[8.2]	[8.1]	[5.4]	[8.9]	[7.8]	--	[7.9]
0.0109	0.109	Ta	--	--	--	--	--	--	--	--	--	--
0.0155	0.155	Te	--	[0.80]	[6.1]	[5.6]	--	--	--	--	--	--
0.0310	0.310	Tl	--	--	--	--	--	--	--	--	[9.7]	--
0.0016	0.016	V	--	--	[1.3]	[1.1]	[1.2]	[1.3]	[1.1]	[1.3]	[1.4]	[1.3]
0.0187	0.187	W	--	--	68.6	66.7	67.4	66.1	66.7	69.3	65.9	65.8
0.0003	0.003	Y	--	--	[0.39]	--	[0.090]	--	--	--	--	--

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

Battelle PNNL/RPG/Inorganic Analysis ... ICPOES Data Report

		Run Date >	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018	5/18/2018
		Process Factor >	245.9	244.7	248.5	247.9	243.9	247.5	246.7	248.1	246.3
			18-1611 @ 5x	18-1612 @ 5x	18-1613 @ 5x	18-1614 @ 5x	18-1615 @ 5x	18-1616 @ 5x	18-1617 @ 5x	18-1618 @ 5x	18-1619 @ 5x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	TI042-L-F7-A	TI042-L-F9-A	TI042-L-F11-A	TI042-L-F13-A	TI042-L-F15-A	TI042-L-F18-A	TI036-S0-CST-F	TI036-S2-CST-F	TI036-S3-CST-F
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0014	0.014	Ag	--	--	--	--	--	--	--	--	--
0.0038	0.038	Al	9,700	9,770	9,750	9,660	9,550	9,600	10,100	9,560	9,440
0.0383	0.383	As	--	--	--	--	--	--	--	--	--
0.0032	0.032	B	53.0	48.4	51.4	52.8	65.7	57.7	41.6	43.3	45.5
0.0001	0.001	Ba	[0.088]	[0.11]	[0.057]	[0.052]	[0.087]	[0.061]	[0.050]	[0.062]	[0.12]
0.0054	0.054	Ca	21.0	19.9	24.8	27.6	21.2	25.7	15.6	17.6	19.6
0.0016	0.016	Cd	5.73	5.38	5.22	5.59	5.56	5.27	5.20	5.34	5.04
0.0016	0.016	Cr	488	491	486	484	482	483	504	478	471
0.0033	0.033	Fe	12.9	13.5	15.5	13.2	17.9	14.5	12.7	12.5	19.5
0.0262	0.262	K	3,960	3,980	3,930	3,850	3,850	3,890	4,010	3,860	3,820
0.0010	0.010	Li	--	--	--	--	--	--	--	--	--
0.0075	0.075	Na	123,000	122,000	124,000	122,000	125,000	121,000	127,000	119,000	119,000
0.0040	0.040	Ni	20.1	20.4	20.8	20.1	20.1	19.5	19.7	20.5	19.8
0.0369	0.369	P	633	642	611	637	625	621	622	611	602
0.0144	0.144	Pb	--	--	--	--	[3.9]	--	--	--	--
0.0876	0.876	Se	--	--	--	[27]	--	--	--	--	--
0.0001	0.001	Sr	[0.037]	[0.039]	[0.033]	[0.043]	[0.035]	[0.040]	[0.050]	[0.037]	[0.060]
0.0057	0.057	Th	--	--	--	--	--	--	--	--	--
0.0004	0.004	Ti	[0.52]	[0.55]	[0.41]	[0.47]	[0.42]	[0.36]	[0.72]	[0.52]	[0.57]
0.0312	0.312	U	[24]	[18]	[15]	[20]	[24]	[18]	[17]	[13]	[19]
0.0023	0.023	Zn	--	[0.57]	[0.81]	[0.99]	[1.2]	[1.2]	--	--	--
0.0013	0.013	Zr	[1.2]	[1.4]	[1.1]	[1.3]	[1.2]	[0.94]	[1.1]	[0.98]	[1.4]
Other Analytes											
0.0001	0.001	Be	[0.12]	[0.13]	[0.13]	[0.12]	[0.13]	[0.12]	[0.11]	[0.10]	[0.100]
0.0220	0.220	Bi	--	[8.4]	[7.2]	[11]	--	[12]	--	--	--
0.0052	0.052	Ce	--	--	--	--	--	--	--	--	--
0.0033	0.033	Co	--	--	[0.94]	--	--	--	--	--	[0.87]
0.0023	0.023	Cu	--	--	--	--	--	--	--	--	--
0.0012	0.012	Dy	--	--	--	--	--	--	--	--	--
0.0006	0.006	Eu	--	[0.24]	[0.19]	[0.33]	[0.19]	[0.17]	--	--	[0.17]
0.0010	0.010	La	--	--	--	--	--	--	--	--	[0.39]
0.0014	0.014	Mg	--	--	--	--	--	--	--	--	--
0.0003	0.003	Mn	--	--	--	--	--	--	--	--	--
0.0048	0.048	Mo	40.6	40.7	40.6	42.1	41.7	40.4	41.3	40.2	39.7
0.0095	0.095	Nd	--	--	--	--	--	--	--	--	--
0.0082	0.082	Pd	[3.7]	[3.4]	[3.3]	[3.8]	[3.2]	[2.2]	[3.8]	[3.1]	[3.4]
0.0108	0.108	Rh	[7.6]	[6.3]	--	[5.0]	[7.2]	[3.5]	[3.1]	[4.6]	--
0.0068	0.068	Ru	[8.5]	[7.8]	[7.6]	[11]	[7.7]	[7.1]	[9.1]	[8.6]	[7.8]
0.0898	0.898	S	1,620	1,630	1,620	1,610	1,590	1,620	1,620	1,570	1,550
0.0569	0.569	Sb	--	--	--	--	--	--	--	--	--
0.0043	0.043	Si	118	109	107	120	133	120	95.9	91.8	90.5
0.0195	0.195	Sn	[9.5]	--	[6.6]	[11]	[8.3]	[6.9]	[5.3]	[6.0]	[11]
0.0109	0.109	Ta	--	--	--	--	--	--	--	--	--
0.0155	0.155	Te	--	--	--	--	--	--	--	--	--
0.0310	0.310	Tl	--	--	--	--	[7.9]	--	--	--	--
0.0016	0.016	V	[1.1]	[1.2]	[1.1]	[1.5]	[1.5]	[1.7]	[1.5]	[1.6]	[1.4]
0.0187	0.187	W	66.7	67.1	66.1	63.7	67.0	69.0	69.7	63.8	62.1
0.0003	0.003	Y	--	--	--	--	--	--	--	--	--

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

QC Performance 5/18/2018

Criteria >	≤ 20%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	18-1606 Dup	LCS/BS	18-1606 MS	18-1606 + PS-A	18-1606 + PS-B	18-1606 5-fold Serial Dil
Analytes	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Ag		30	30	93		
Al	4.8	105	109	105		7.6
As				102		
B	0.9	105	100	102		
Ba		101	97	95		
Ca	28.3	103	103	98		
Cd	5.4	105	100	100		
Cr	4.8	100	nr	101		7.1
Fe	7.2	100	99	97		
K	4.5	105	116	106		1.2
Li		112	117	109		
Na	2.4	103	nr	nr		3.6
Ni	1.1	103	101	100		
P	4.3	105	98	101		8.2
Pb		105	99	98		
Se				100		
Sr		102	101	95		
Th					95	
Ti		103	100	98		
U		104	101		100	
Zn		103	98	100		
Zr		106	101	99		
Other Analytes						
Be		97	98	94		
Bi		82	86	90		
Ce		97	93		91	
Co				98		
Cu		102	101	102		
Dy					97	
Eu					96	
La		102	101		97	
Mg		104	102	99		
Mn		103	99	96		
Mo	3.2	103	97	98		
Nd		100	98		95	
Pd					83	
Rh					93	
Ru					96	
S	3.8	103	98		94	0.4
Sb				102		
Si	2.3	51	72	94		9.2
Sn				95		
Ta				97		
Te					88	
Tl				87		
V		101	99	96		
W	1.9	102	94	98		
Y				97		

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilutio

Battelle PNNL/RPL/ASO Hydroxide Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Hydroxide Analysis by Titration

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Hydroxide
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per RPG-CMC-128, Rev.1, *HNO_3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☒ Other: A sample aliquot of 1.0 mL for hydroxide analysis was created from the submitted samples – 5/11/18
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes
Analysis Procedure:	RPG-CMC-228, Rev. 0.1, *Determination of Hydroxyl (OH^-) and Alkalinity of Aqueous Solutions, Leachates & Supernates*
Analysis Date or Date Range:	6/27/18
Technician/Analyst:	CE Rutherford
Rad Chem Electronic Data File:	ASR 0521 (Fiskum).xls
ASO Project 98620 File:	File Plan 5872: Sample preparation and analysis records; LSC 3100 TR calibration, daily checks, and maintenance records; and standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Beckman Coulter pH Meter, SN: 110650046

/ 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

SAMPLE RESULTS

See attached data report, Sample Results for ASR 0521.01. All sample data are reported in µg/mL.

ASO Project File, ASR 0521.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form and laboratory bench records, and records associated with hydroxide analysis. Standard certificates and balance calibration records can be found in the ASO Records.

Sample preparation and analysis

Hydroxide analysis was performed for diluted sample aliquots of 3 aqueous samples (18-1604 thru 18-1605), provided on ASR 0521.01. Samples were analyzed by manual titration for the base constituents content following procedure RPG-CMC-228, Rev. 0.1, *Determination of Hydroxyl (OH⁻) and Alkalinity of Aqueous Solutions, Leachates & Supernates*.

A sample size of 0.100 mL was added to 2.5 mL of DI water. The titrant used was 0.0902 M HCl. Standardized HCl was prepared on 7/11/17 and documented on Chem_Rec 234. Titration pH measurements were obtained using a Beckman Coulter 560 pH meter with serial number (SN) 110650046. The pH meter was calibrated using 3 buffers, pH 4, 7 and 10 and the calibration verified using an independent pH 7 buffer.

The customer requested target MDL of 0.1M was achieved for all samples. The titrant used for the sample analyses is 0.0902M HCl along with a pH meter accurate to within 0.05 pH units. The low concentration of the titrant along with the sensitivity of the pH meter provides the detection capability to meet this requirement.

The initial diluted pH is reported on the attached Report Summary along with the free hydroxide molarity. Samples were analyzed in duplicate; therefore two pH measurements are given per sample for each inflection point.

Sample ID	1st Inflection point	2nd Inflection point	3rd Inflection point
18-1604	10.8	7.0	3.6
18-1604 Dup	10.8	7.1	3.6
18-1605	10.7	6.7	3.4
18-1605 Dup	10.7	6.7	3.4
18-1606	10.8	6.7	3.6
18-1606 Dup	10.5	6.5	3.3

Included in the data package is a report summary and the sample results calculated from the raw data. A copy of the titration curve data for each sample is also included with this report.

QUALITY CONTROL RESULTS

This analysis has limited quality control (QC) samples that are prepared. There are no laboratory blanks (LB), matrix spikes or reagent spikes analyzed. Samples are analyzed in duplicate.

Instrument Calibration Control

The pH meter was calibrated using 3 buffers, pH 4, 7 and 10 and the calibration verified using an independent pH 7 buffer.

Duplicate -- Relative Percent Difference (RPD):

> A duplicate sample was analyzed for each of the three samples in this sample set. The only acceptance criterion provided in ASO-QAP-001, Rev. 11, Analytical Support Operations (ASO) QA Plan is that pH units for duplicate samples are expected to agree within ± 0.1 pH units. The product of the hydroxide analysis is titration curves showing inflection points. The initial pH of the diluted samples is reported and the duplicate samples agreed within ± 0.1 pH units meeting the recommended requirement.

Assumption and Limitations of the Data

Generally ASO analysis methods are associated with reported uncertainty. Titrations are not amenable to calculations of uncertainty.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.

Attachment: Data Report -- Sample Results for ASR 0521.01

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group
Chemical Measurements Center

ASR # **521.01**

WP# N96056

Hydroxide and Alkalinity Determination
Procedure: RPG-CMC-228, Rev 0.1

Equip # Beckman Coulter 560, SN#110650046

Report Summary for ASR # -- 521.01

Revision # **Rev-0**

Report Date: 6/28/2018
Analysis Date: 6/27/2018

				Concentration, moles / Liter		
				First Point	Second Point	Third Point
RPG #	Client ID	Diluted Initial pH	OH conc ug/mL	Molarity	Molarity	Molarity
18-1604	**TI042-COMP-FEED**	**12.39**	1.53E+04	**0.90**	**1.17**	**0.90**
18-1604 Dup	**TI042-COMP-FEED**	**12.42**		**0.90**	**1.17**	**0.90**
18-1605	**TI042-COMP-EFF**	**12.52**	1.53E+04	**0.90**	**1.17**	**0.90**
18-1605 Dup	**TI042-COMP-EFF**	**12.50**		**0.90**	**1.17**	**0.90**
18-1606	**TI042-COMP-EFF-DUP**	**12.50**	1.53E+04	**0.90**	**1.17**	**0.99**
18-1606 Dup	**TI042-COMP-EFF-DUP**	**12.49**	1.69E+04	**0.99**	**1.17**	**0.99**

OH conc (mg/L) = M (g/L) * 17,000

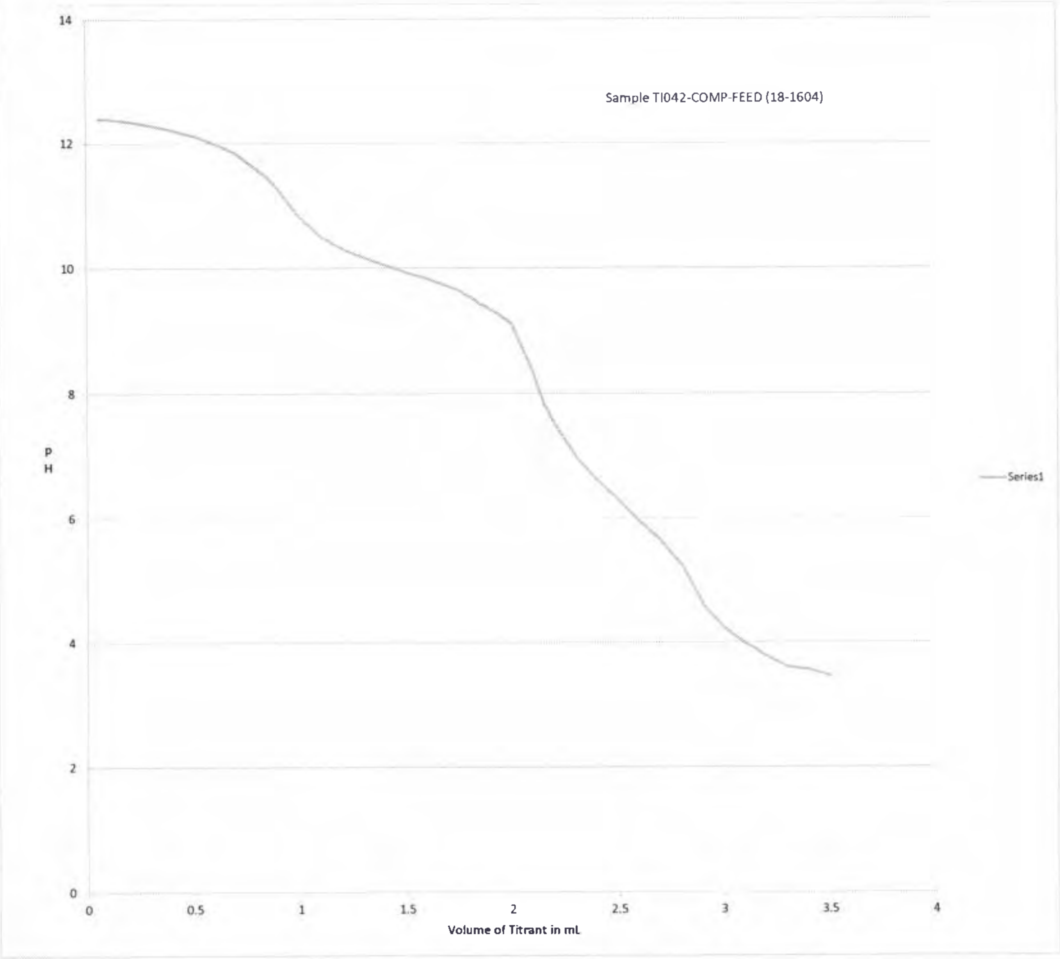
Note: Results are presented for the first, second, and third inflection points on the titration curves for the hydroxide titration, as applicable. The first inflection point is generally associated with the free hydroxide concentration. The second inflection point generally represents total hydroxide, or carbonate or a combination of aluminate and carbonate. The third inflection point is usually indicative of bicarbonate or other weak acids or possibly the continued protonation of alumina.

Analyst: 7/13/18

Reviewer: 7/13/18

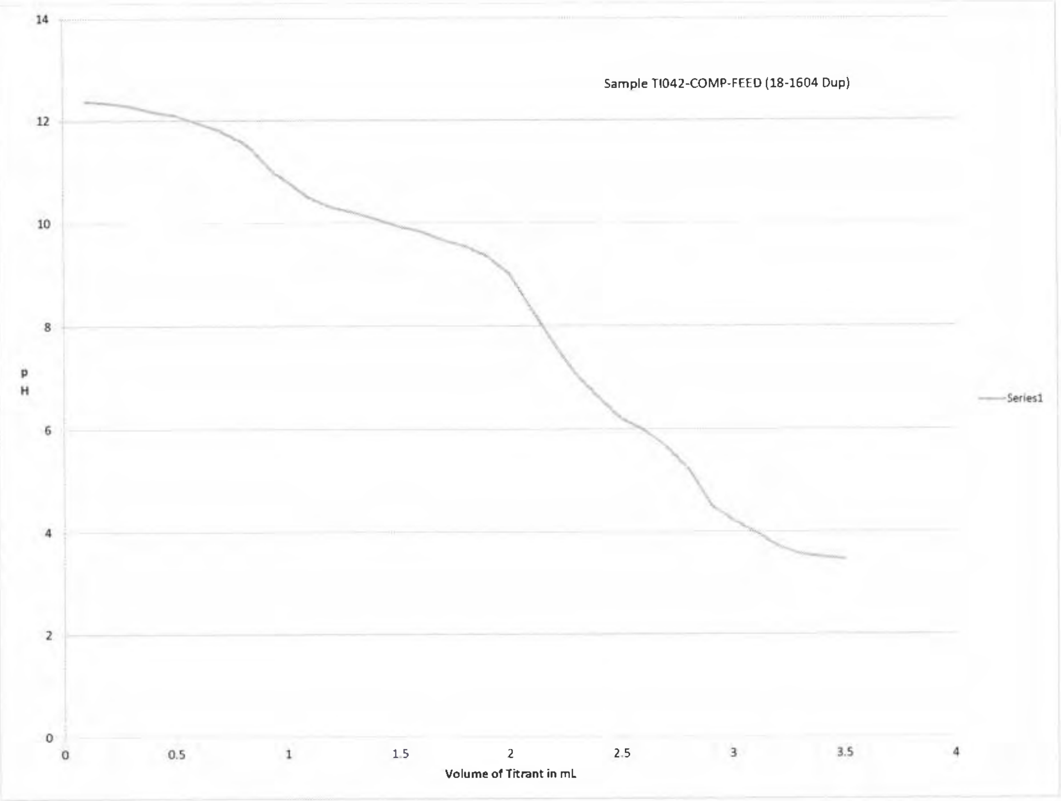
18-1604
TI042-COMP-FEED

Titrant Volume (mL)	pH
0.055	12.39
0.1	12.39
0.2	12.35
0.3	12.29
0.4	12.21
0.5	12.12
0.6	11.99
0.7	11.84
0.8	11.58
0.9	11.45
0.9	11.26
0.95	11.03
1.0	10.81
1.1	10.51
1.2	10.31
1.3	10.17
1.4	10.05
1.5	9.93
1.6	9.83
1.7	9.71
1.75	9.64
1.80	9.54
1.85	9.43
1.90	9.34
2.0	9.11
2.1	8.33
2.2	7.83
2.2	7.51
2.3	6.97
2.4	6.60
2.5	6.29
2.6	5.93
2.7	5.63
2.8	5.22
2.9	4.58
3.0	4.23
3.1	3.98
3.2	3.77
3.3	3.60
3.4	3.56
3.5	3.46



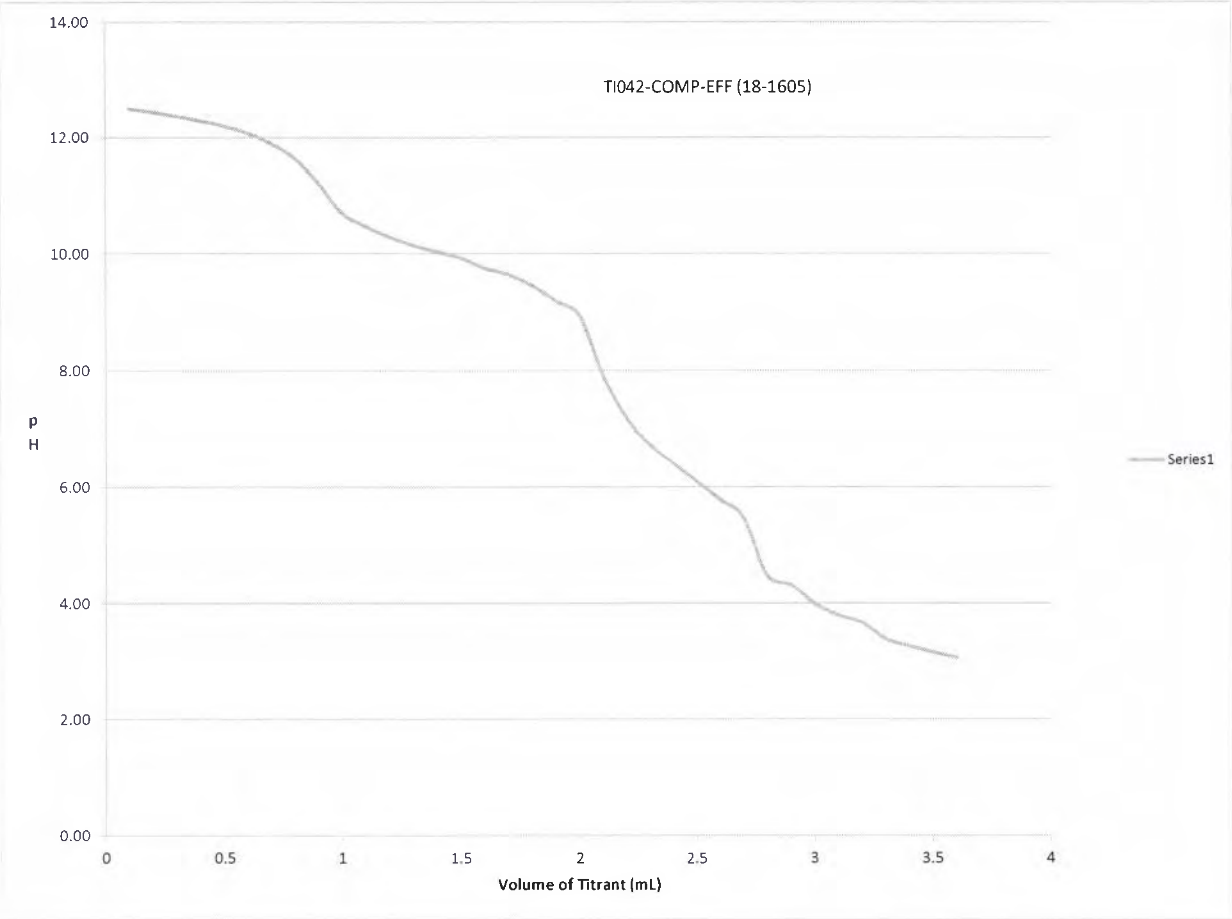
18-1604 Dup
TI042-COMP-FEED

Titrant Volume (mL)	pH
0.1	12.37
0.2	12.34
0.3	12.28
0.4	12.17
0.5	12.10
0.6	11.95
0.7	11.81
0.8	11.57
0.9	11.40
0.95	10.95
1.0	10.82
1.1	10.49
1.2	10.30
1.3	10.20
1.4	10.07
1.5	9.93
1.6	9.83
1.7	9.67
1.80	9.54
1.90	9.35
2.0	9.01
2.1	8.30
2.2	7.65
2.3	7.05
2.4	6.61
2.5	6.20
2.6	5.98
2.7	5.66
2.8	5.21
2.9	4.51
3.0	4.22
3.1	3.98
3.2	3.71
3.3	3.56
3.4	3.50
3.5	3.46



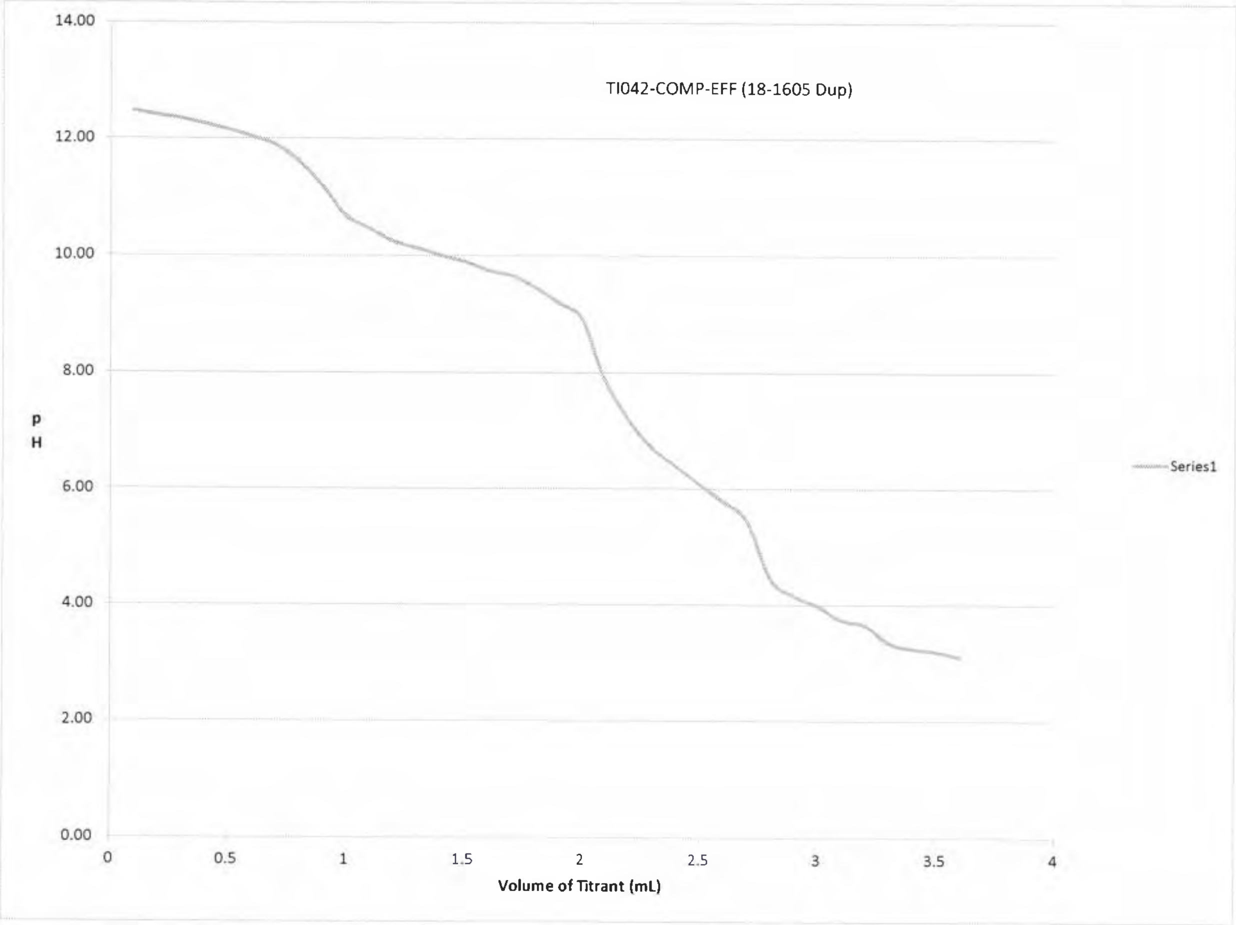
18-1605
TI042-COMP-EFF

Titrant Volume (mL)	pH
0.1	12.49
0.2	12.43
0.3	12.36
0.4	12.28
0.5	12.19
0.6	12.07
0.7	11.89
0.8	11.63
0.9	11.20
1	10.69
1.1	10.46
1.2	10.28
1.3	10.14
1.4	10.03
1.5	9.92
1.6	9.75
1.7	9.64
1.8	9.45
1.9	9.19
2	8.92
2.1	7.88
2.2	7.19
2.3	6.73
2.4	6.40
2.5	6.09
2.6	5.78
2.7	5.46
2.8	4.48
2.9	4.31
3	3.99
3.1	3.79
3.2	3.66
3.3	3.38
3.4	3.26
3.5	3.15
3.6	3.06



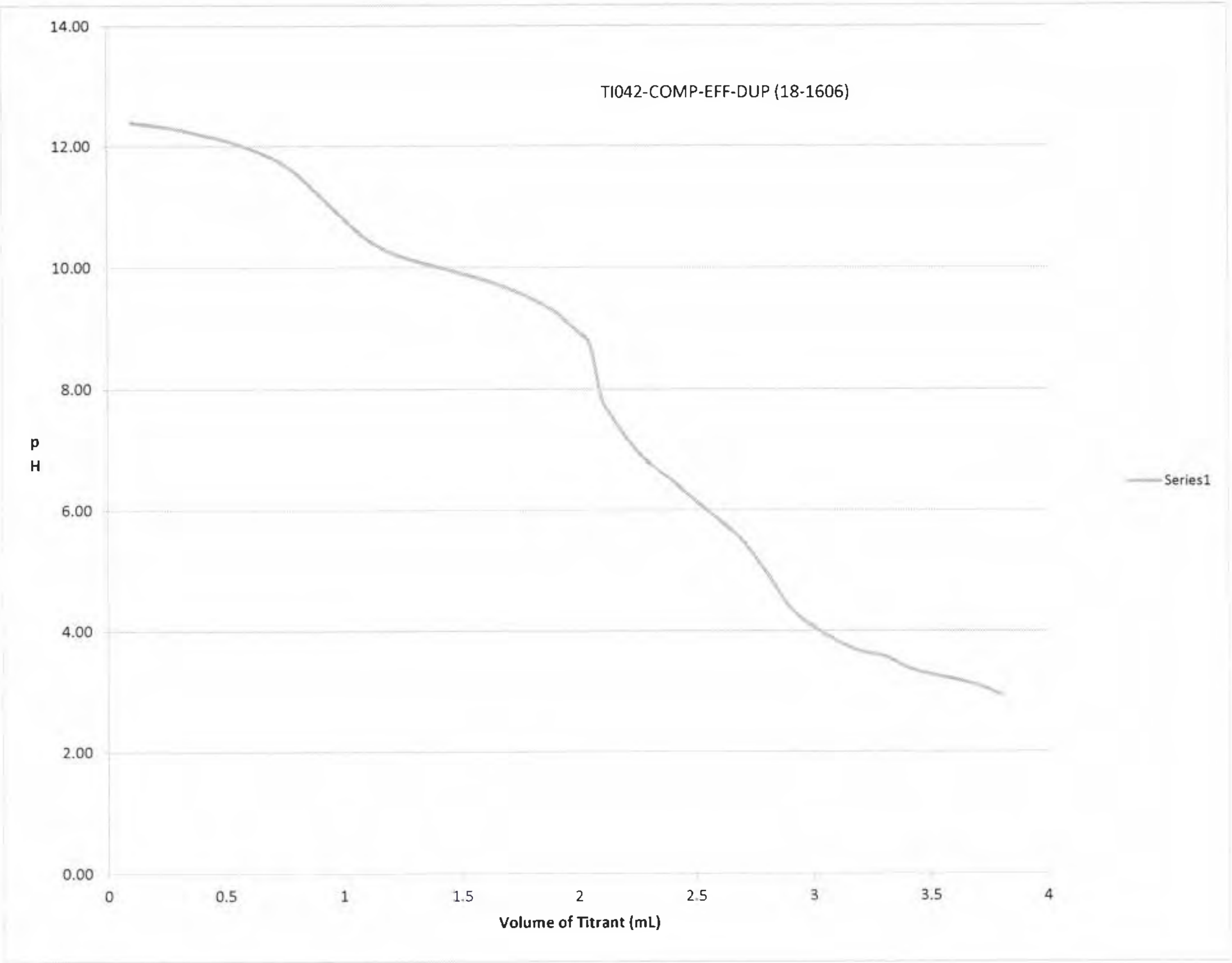
18-1605
TI042-COMP-EFF

Titrant Volume (mL)	pH
0.1	12.47
0.2	12.40
0.3	12.34
0.4	12.25
0.5	12.15
0.6	12.03
0.7	11.89
0.8	11.60
0.9	11.18
1	10.67
1.1	10.46
1.2	10.24
1.3	10.12
1.4	10.00
1.5	9.89
1.6	9.74
1.7	9.65
1.8	9.44
1.9	9.18
2	8.90
2.1	7.86
2.2	7.16
2.3	6.69
2.4	6.38
2.5	6.07
2.6	5.78
2.7	5.44
2.8	4.45
2.9	4.15
3	3.98
3.1	3.74
3.2	3.65
3.3	3.35
3.4	3.25
3.5	3.20
3.6	3.11



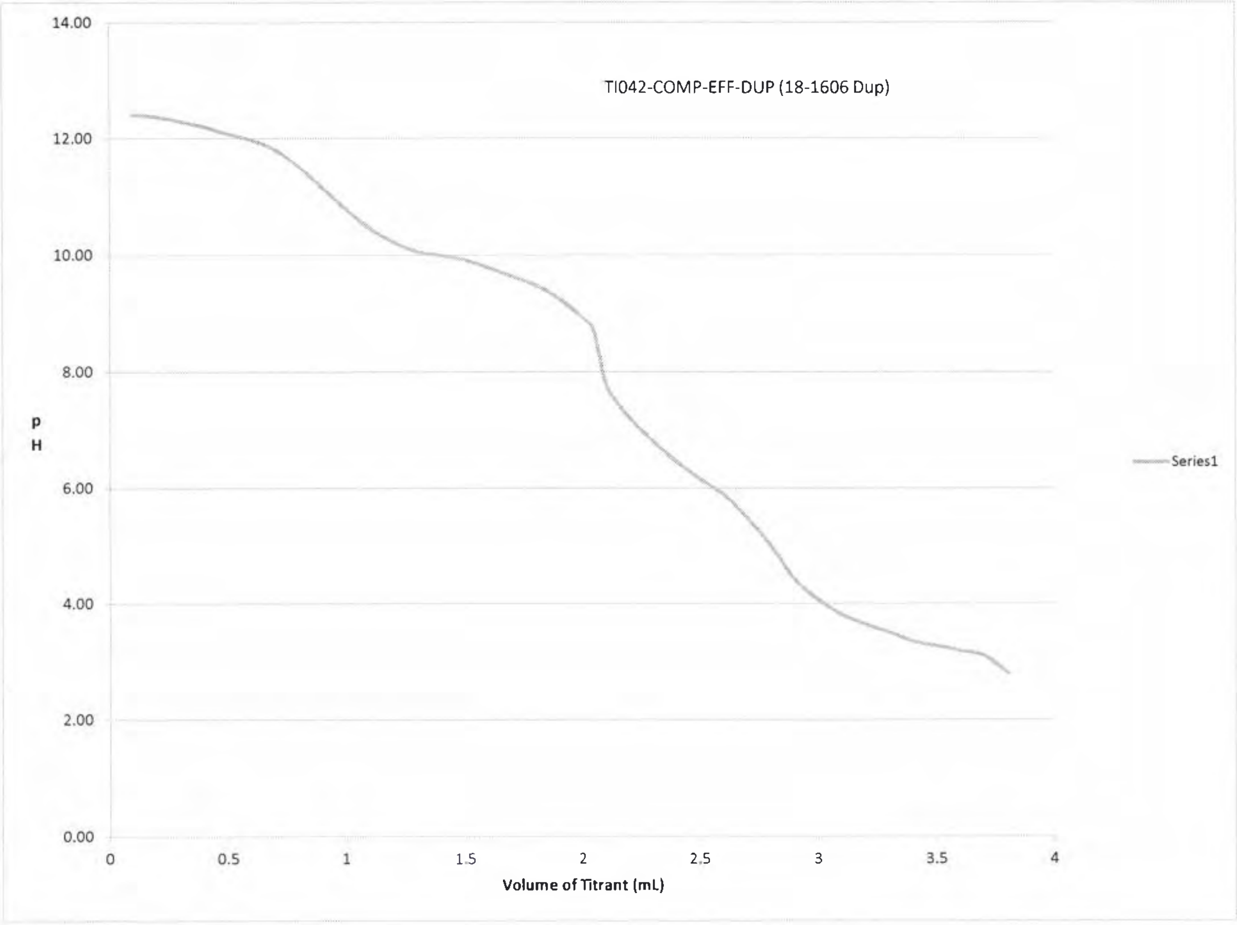
18-1606
TI042-COMP-EFF-Dup

Titrant Volume (mL)	pH
0.1	12.39
0.2	12.34
0.3	12.28
0.4	12.19
0.5	12.10
0.6	11.97
0.7	11.81
0.8	11.55
0.9	11.19
1	10.81
1.1	10.47
1.2	10.25
1.3	10.11
1.4	10.01
1.5	9.90
1.6	9.79
1.7	9.65
1.8	9.48
1.9	9.26
2	8.93
2.05	8.72
2.1	7.84
2.15	7.51
2.2	7.23
2.3	6.79
2.4	6.48
2.5	6.15
2.6	5.83
2.7	5.49
2.8	4.98
2.9	4.39
3	4.06
3.1	3.83
3.2	3.66
3.3	3.58
3.4	3.39
3.5	3.28
3.6	3.20
3.7	3.10
3.8	2.94



18-1606-Dup
TI042-COMP-EFF

Titrant Volume (mL)	pH
0.1	12.40
0.2	12.37
0.3	12.29
0.4	12.20
0.5	12.08
0.6	11.97
0.7	11.81
0.8	11.53
0.9	11.17
1	10.79
1.1	10.46
1.2	10.22
1.3	10.05
1.4	9.99
1.5	9.91
1.6	9.78
1.7	9.63
1.8	9.48
1.9	9.24
2	8.92
2.05	8.70
2.1	7.80
2.15	7.45
2.2	7.20
2.3	6.80
2.4	6.45
2.5	6.15
2.6	5.88
2.7	5.48
2.8	4.99
2.9	4.41
3	4.06
3.1	3.80
3.2	3.64
3.3	3.50
3.4	3.35
3.5	3.27
3.6	3.19
3.7	3.10
3.8	2.80

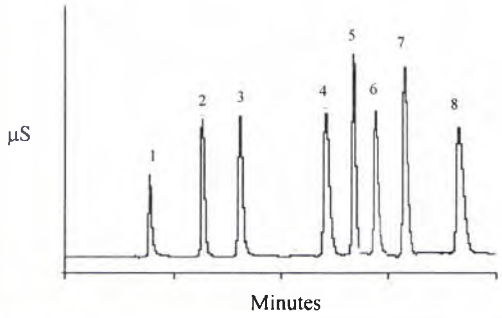


Battelle - Pacific Northwest National Laboratory
Analytical Support Operations – IC Report

PO Box 999, Richland, Washington 99352

Common Inorganic Anions

Dionex AS18 Column; Hydroxide Gradient



1	Fluoride
2	Chloride
3	Nitrite
4	Sulfate
5	Bromide
6	Oxalate
7	Nitrate
8	Phosphate

Client: S. Fiskum

ASR #: 0521.01

Project #: 71274

 # Samples: 3 liquids

Charge Code: N96056

*** RPL Number: 18-0604 and 18-0606 ***

Procedure, Analysis, System, and Records Information

Analysis Procedure	RPG-CMC-212 Rev.2, "Determination of Common Anions by Ion Chromatography"
Prep Procedure	NA
Analyst	JC Carter
Analysis Dates	06-18-18 through 06-20-18
Calibration Date	06-18-18
Cal/Ver Stds Prep Date	Cal 06-18-18 and Ver 06-18-18
Excel Data File	IC-0240 ASR-0521 Results.xls
M&TE Numbers	IC System (M&TE) WD81499
	Balance: B725287790
All Analysis Records	Chemical Measurement Center 98620: RIDS IC System File (IC-0240)

Prepared By — Date 6.26.18

Reviewed By — Date 7/5/18

IC Report

Sample Results

See Attachment: Sample Results ASR 0521.01

Sample Analysis/Results Discussion

Three liquid samples were submitted to the Analytical Support Operations (ASO) laboratory for ion chromatography analysis under ASR 0521.01. The results are discussed in this report. The analytes of interest include chloride, sulfate, nitrite, nitrate, oxalate and phosphate. The dilutions were prepared using deionized water and the water was analyzed as the 'Dilution Blank" sample. The sample results are reported in µg/mL and have been adjusted for analytical dilutions.

The estimated method detection limits (MDL) are provided for each analyte of interest measured and the MDLs have been adjusted for all analytical dilutions and processing factors. The MDLs are set at one-tenth the lowest calibration standard, which is defined as the estimated quantitation limit (EQL).

Data Limitations

The data reported for phosphate follows a CCV which failed for that analyte with a recovery of 80.5%. The phosphate results for the samples at the next higher dilution level and were within passing CCVs agree with those reported, however they are between the MDL and the EQL and are considered qualitative, not quantitative.

Sulfate was visible in the chromatograms for the highest dilution, but at that point was considered undetectable due to the elevated MDL. The data was manually entered into the report.

Client requested an MDL of ≤200µg/mL for all analytes. This was not met for both nitrite and nitrate due to dilution yet both were detected above the stated MDL.

Quality Control Discussion

The method performance is evaluated against the acceptance criteria established by the Analytical Support Operations QA Plan ASO-QAP-001 and in procedure, RPG-CMC-212 Rev.2.

IC Workstation QC Results

The method performance is evaluated against the acceptance criteria established by Analytical Support Operations QA Plan ASO-QAP-001 and in procedure, RPG-CMC-212 Rev.2.

Process Blank (Dilution Blank): No analytes of interested were detected, thus meeting the ASO's QA Plan acceptance criteria of all analytes being <EQL.

Duplicate: All samples were analyzed in duplicate. The relative percent difference (RPD) is reported for all analytes which were measured at or above the EQL. The reported RPDs ranged from 0 to 11% for all analytes of interest, which meets the ASO's QA Plan acceptance criteria of ≤20% for liquid samples.

Laboratory Control Sample (LCS): A routine instrument LCS was analyzed with recoveries ranging from 94% to 102%, meeting the QA Plan acceptance criteria of 80% to 120%.

Analytical Spike (AS) (Accuracy): Analytical spikes were prepared using all of the prepared dilutions of the two liquid samples by adding a known concentration of mid-range multi-mix standard, “CCV 061818”. The results for the spiked 67881x dilutions are reported. Where the spiking concentration exceeds 20% of the sample concentration, the AS recoveries meet the QA Plan acceptance criteria of 75% to 125%.

IC System QC Samples: Numerous calibration verification standards and calibration verification blanks were analyzed each run day. Results for the IC System QC samples (that bound the reported results for each analyte of interest) are within acceptance criteria of the ASO’s QA Plan (i.e., verification standard recoveries from 90% to 110% and verification blank results <EQL or ≤5% of reported sample result). An exception is the data reported for phosphate for all samples followed a CCV with a recovery of 80.5% but are followed by passing CCV at 94.6%. It is believed the data is unaffected due to the fact that the results for a higher dilution analyzed between passing CCVs agree with these reported values.

Deviations from Procedure

None

General Comments

- The reported "Final Results" have been corrected for all dilutions performed on the sample during processing or analysis.
- For each anion, the instrument EQL is defined as the concentration of the lowest calibration standard and the instrument MDL is set at one-tenth of the EQL. The MDLs and EQLs reported for each sample are adjusted for the sample dilution factors (processing and analysis) and assume non-complex aqueous matrices. Matrix-specific MDLs or EQLs may be determined, when requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference.

Sample Results ASR 521.01

		Cl			NO_2			SO_4			C_2O_4			NO_3			PO_4		
RPL Number	Client Sample ID	MDL µg/mL	Result µg/mL	DF	MDL µg/mL	Result µg/mL	DF	MDL µg/mL	Result µg/mL	DF	MDL µg/mL	Result µg/mL	DF	MDL µg/mL	Result µg/mL	DF	MDL µg/mL	Result µg/mL	DF
18-1604	TI042-COMP-FEED	42	2,520		620	52,600		10,000	1,532	*	56	460	J	1,200	106,000		67	1,570	
18-1605	TI042-COMP-EFF	42	2,700		620	54,800		10,000	1,957	*	56	500	J	1,200	113,000		67	1,590	
18-1606	TI042-COMP-EFF-DUP	42	2,750		620	57,200		10,000	1,715	*	56	500	J	1,200	116,000		67	1,470	
Dilution Blank	Dilution Blank	0.075	0.075	U	0.10	0.100	U	0.15	0.150	U	0.10	0.100	U	0.20	0.200	U	0.12	0.120	U

Sample QC Results ASR 521.01

Sample/Replicate Precision Results

		Cl		NO_2		SO_4		C_2O_4		NO_3		PO_4	
RPL Number	Sample ID	µg/mL	RPD	µg/mL	RPD	µg/mL	RPD	µg/mL	RPD	µg/mL	RPD	µg/mL	RPD
18-1604	Sample	2520	--	52600	--	U	--	J	--	106000	--	1570	--
	Duplicate | RPD	2550	1	52200	1	U	N/A	J	N/A	109000	3	1400	11
18-1605	Sample	2700	--	54800	--	U	--	J	--	113000	--	1590	--
	Duplicate | RPD	2720	1	55200	1	U	N/A	J	N/A	117000	3	1550	3
18-1606	Sample	2750	--	57200	--	U	--	J	--	116000	--	1470	--
	Duplicate | RPD	2800	2	57300	0	U	N/A	J	N/A	119000	3	1480	1

Sample Spike Results - At IC Workstation

		Cl		NO_2		SO_4		C_2O_4		NO_3		PO_4	
RPL Number	Sample ID	µg/mL	%Rec	µg/mL	%Rec	µg/mL	%Rec	µg/mL	%Rec	µg/mL	%Rec	µg/mL	%Rec
18-1604	Sample	2520	--	52600	--	U	--	460	--	106000	--	1570	--
	AS Sample	1.52	100	2.39	100	3.03	101	1.99	99	3.96	91	2.33	93
18-1605	Sample	2700	--	54800	--	U	--	500	--	113000	--	1590	--
	AS Sample	1.65	108	6.41	301	2.46	82	1.99	99	13.3	358	2.45	97
18-1606	Sample	2750	--	57200	--	U	--	500	--	116000	--	1470	--
	AS Sample	1.66	109	6.66	313	2.48	83	1.98	99	14.1	381	2.42	96

LCS/Blank Spike Results

Run ID	Sample ID	Cl % Rec	NO_2 % Rec	SO_4 % Rec	C_2O_4 % Rec	NO_3 % Rec	PO4 % Rec
18.06.18 22:56	LCS	99.5	100.7	100.6	101.5	94.1	95.2

AS = Analytical Spike: Spike performed at IC Workstation on Liquid Samples.
LCS = Laboratory Control Sample (or Blank Spike)
RPD = Relative Percent Difference
%Rec = Percent Recovery
DF = Data Quality Flag
U = Not Detected Above Method Detection Limit (MDL)
J = Detected, Result are Qualitative: Result >MDL but <EQL (Estimated Quantitation Limit)
-- = Value Not Calculated or Place Holder for Blank Cell
* = see report narrative "Data Limitations"

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	Fiskum
Total # of Samples:	3

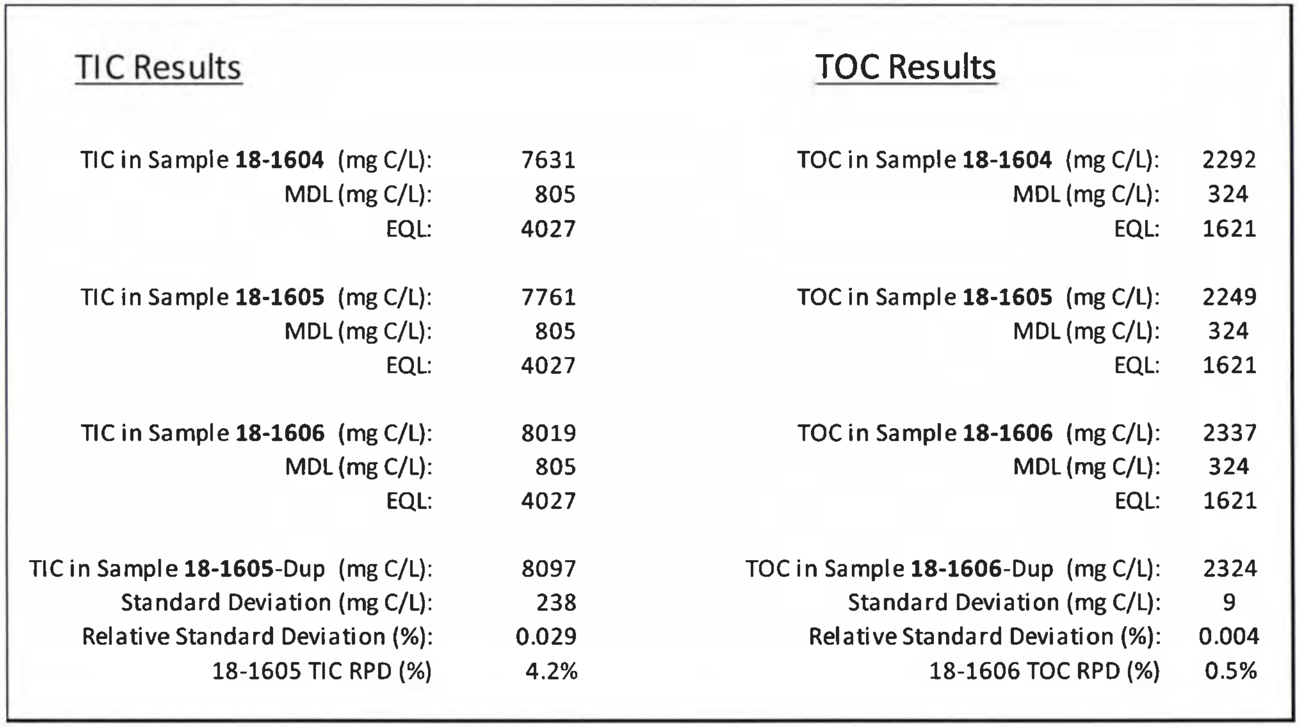
RPL ID	**Client Sample ID**
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Total Inorganic Carbon (TIC) Total Organic Carbon (TOC)
Analysis Procedure:	RPG-CMC-386, Rev. 1, "Carbon Analyses in Solids, Sludge, and Liquid Matrices"
Preparation Procedure:	None
Technician/Analyst:	C. Rutherford
Analysis Date(s):	May 22, 2018 (TOC) & June 18-19, 2018 (TIC)
CCV Standards:	TIC/TOC CMS# 533459 and 533458
BS/LCS/MS Standards:	TIC/TOC CMS# 533457 and 520858
Excel Data File:	ASR 0521.01
M&TE Numbers:	Carbon System (SN:115015013, RPL/701) Balance : Sartorius R200D, SN: 30809774
CMC Project 98620 File:	File Plan 5872: TC 18-4: Sample preparation and analysis records; calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.

Preparer 6/19/18
Date

Reviewer 7/13/18
Date

Table 1: TOC/TIC Results for ASR 0521



TIC Results		TOC Results	
TIC in Sample **18-1604** (mg C/L):	7631	TOC in Sample **18-1604** (mg C/L):	2292
MDL (mg C/L):	805	MDL (mg C/L):	324
EQL:	4027	EQL:	1621
TIC in Sample **18-1605** (mg C/L):	7761	TOC in Sample **18-1605** (mg C/L):	2249
MDL (mg C/L):	805	MDL (mg C/L):	324
EQL:	4027	EQL:	1621
TIC in Sample **18-1606** (mg C/L):	8019	TOC in Sample **18-1606** (mg C/L):	2337
MDL (mg C/L):	805	MDL (mg C/L):	324
EQL:	4027	EQL:	1621
TIC in Sample **18-1605**-Dup (mg C/L):	8097	TOC in Sample **18-1606**-Dup (mg C/L):	2324
Standard Deviation (mg C/L):	238	Standard Deviation (mg C/L):	9
Relative Standard Deviation (%):	0.029	Relative Standard Deviation (%):	0.004
18-1605 TIC RPD (%)	4.2%	18-1606 TOC RPD (%)	0.5%

Sample Analysis/Results Discussion

Three liquid samples were submitted under Analytical Service Request (ASR) 0521.01 for total inorganic carbon (TIC) and total organic carbon (TOC) analysis. The analyses were performed by the hot persulfate wet oxidation method, with the results summarized in Table 1. The TIC is measured first with additions of heated sulfuric acid followed by the addition of a silver-catalyzed acidic potassium persulfate solution for oxidation at 92-95 °C for TOC. In this case, two separate sample runs were performed, one for TOC only and the other for TIC only. The analyses were performed following procedure RPG-CMC-386, Rev. 1, *Carbon Analyses in Solids, Sludge and Liquid Matrices*

The samples were analyzed with one duplicate for each TIC and TOC. Sample 18-1604 was selected for the analytical spike for TIC, while 18-1606 was selected for the analytical spike for TOC. The sample results are corrected for the contribution from the system blank, as per procedure RPG-CMC-386. All data are reported as mg C/L of sample.

Data Limitations

None

Quality Control Discussion

The calibration and quality control (QC) sample standards for the TOC initial/continuing calibration verification check (ICV/CCV) samples wa a 1000 µg/mL solution of TOC standard.

The calibration and QC sample standards for the TIC ICV/CCV check sample is a 1000 µg/mL TIC standard. The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data bench sheets for traceability.

The QC samples analyzed as part of the method include ICV/CCV, initial and continuing calibration blank (ICB/CCB) samples, laboratory duplicates for each sample, a laboratory control sample/blank spike (LCS/BS), and an analytical spike (AS). The analysis was performed in one batch.

Two blanks are run at the beginning of each batch and after the ICV/CCV. The blanks must be < EQL. The blank results for the batch were < EQL (TOC EQL = 4027 µg/ml and TOC MDL=805 µg/ml; TIC EQL = 1621 µg//mL and TIC MDL= 324 µg/ml). There was a project-specific requirement that the MDL be less than 200 µg/ml; this requirement was not met.

Initial Calibration Check and Continuing Calibration Verification Standards:

> The calibration of the coulometer analysis system was checked by the analysis of calibration verification standards analyzed at the beginning and end of the analysis run. TOC results for the two ICVs were 98.2% and 98.8% recovery. Results for the two TIC ICV samples were 99.3% and 96.9% recovery. All were within the acceptance criterion of 90% to 110%. The TOC result for the CCV sample analyzed was 100.3% recovery and the TIC CCV sample result was 105.4% recovery within the acceptance criterion of 85% to 115%.

Laboratory Control Sample/Blank Spike: One TIC and TOC LCS/BS was analyzed. The TIC LCS/BS result was 104.0% recovery. The TOC LCS/BS result was 98.9% recovery. Bother results met the acceptance criteria of 75% to 125% recovery.

Duplicate/Replicate: Precision of the carbon measurements is demonstrated calculating the relative percent difference (RPD) between sample and duplicate/replicate. The RPD for the TIC sample, 18-1605, was 4.2%. The RPD for the TOC sample, 18-1606, was 0.5%. Both RPD results met the acceptance criteria of ≤ when the result is > 10 * the instrument detection limit (IDL) [which is also the method estimated quantitation limit (EQL)].

Analytical Spike (AS): The accuracy of the carbon measurements can be estimated by the recovery from the AS. Sample 18-1604 was selected for TIC and sample18-1606 was selected for TOC. The AS results for TIC was 95.3% recovery and for the TOC was 96.9% recovery. These recovery results met the acceptance criterion of 75% to 125%.

Deviation from Procedure:

None.

General Comments

1) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
2) For the TIC/TOC, the analysis MDL is calculated by dividing the batch IDL by the sample mass and is therefore dependent on sample size. The estimated quantitation limit (EQL) is defined as 5x the MDL. Results < 5x MDL have higher uncertainties and RPDs are not calculated if the results are < 5x MDL.
3) Where applicable, the reported "Final Results" have been corrected for any dilution performed on the sample prior to analysis.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Gamma Energy Analysis (GEA)

Project / WP#: 71274/ N96056
ASR#: 0521.01
Client: SK Fiskum
Total Samples: 3

RPL ID	Client Sample ID
18-1604	**TI042-COMP-FEED**
18-1605	**TI042-COMP-EFF**
18-1606	**TI042-COMP-EFF-DUP**

Analysis Type:	GEA- for all positively measured or non-detected isotopes
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None ☐ Digested as per RPG-CMC-129, Rev. 0 *HNO_3-HCl Acid Extraction of Solids Using a Dry Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry.
Analysis Procedure:	RPG-CMC-450, Rev. 3 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Reference Date:	None
Analysis Date or Date Range:	May 18, 2018
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*18-1604 Fiskum.xls*
ASO Project 98620 File:	*File Plan 5872, Technical (Radiochemistry), Gamma Calibration, daily checks, and maintenance records; and standard certificates and preparation. Also, balance calibration and performance check records.*
M&TE Number(s):	*Detectors D,E,T*

T Trang-le / 6/15/18
Prepare **Date**

/ 6/15/18
Reviewer **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in these aqueous samples are presented in an attached Excel spreadsheet for ASR 0521.01. All of the requested isotopes were detected and sample results are reported in units of µCi/mL with estimates of the total propagated uncertainty reported at the 1-sigma level. Am-241 was also detected and reported.

ASO Project File, ASR 0521.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, laboratory bench records, Shielded Analytical Laboratory Bench Sheet, and Gamma Energy Analysis printouts. Detector calibration records, standard certificates, control charts and balance calibration records can be found in the ASO Records.

Sample Preparation, Separation, Mounting and Counting Methods

The quality control (QC) steps for direct GEA are discussed below.

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

No process blank was prepared for gamma counting.

Required Detection Limits

A detection limit of 1E-5 µCi/mL was required for the detection of Co-60, Cs-137, and 154Eu. Activities for all of these nuclides were detected at levels significantly exceeding the requested detection limits.

Blank Spike (BS)/Laboratory Control Sample (LCS)/ Matrix Spike (MS):

There are no BS, LCS or MS samples analyzed for ASO GEA analyses. Instrument performance is assessed by the analyses of daily control counts and weekly background counts, as discussed below.

Duplicate Relative Percent Difference (RPD):

Sample TI042-COMP-EFF was analyzed in duplicate (18-1605 and 18-1606) and the relative percent differences (RPD) for the isotopes ranged from 3% to 9%, well within the acceptance criterion of < ± 20%.

Instrument Calibration and Quality Control

Gamma detectors are calibrated using multi-isotope standards that are NIST-traceable and prepared in the identical counting geometry to all samples and detectors, if possible. Counter control sources containing Am-241, Cs-137 and Co-60 are analyzed daily before the use of each detector. Procedure RPG-CMC-450 requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count. The most recent background is subtracted from all sample counts.

Assumptions and Limitations of the Data

None

Interferences/Resolution

None.

Uncertainty

For gamma counting, the uncertainty in the counting data, photon abundance and the nuclear half-life, and efficiency are included in the calculation of the total uncertainty along with a systematic uncertainty for sample prep. The Canberra Genie software includes both random and systematic uncertainties in the calculation of the total uncertainties which are listed on the report. We conservatively estimate that 2% is the lowest uncertainty possible for our GEA measurements taking into account systematic uncertainties in gamma calibration standards.

Comments

None.

Attachment: Data Report Sample Results for ASR 0521.01.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Radiochemical Sciences and Engineering Group

filename: 18-1604 Fiskum
6/13/2018

Client: Fiskum
ASR: 0521.01

Project: 71274
N96056

Prepared by: T Trang-le 6/13/18

Technical Reviewer: LRGreenwood 6/13/18

Procedures: RPG-CMC-450, Rev. 3 Gamma Energy Analysis and Low-Energy Photon Spectrometry
M&TE: Gamma detectors D,E,T
Count dates: 18-May-18

Sample	Lab ID	Measured Activity, µCi per mL ± 1s			
		Co-60	Cs-137	Eu-154	Am-241
TI042-COMP-FEED	18-1604	<1.9E-3	**1.49E+2 ± 2%**	<8.4E-3	<4.1E-1
TI042-COMP-EFF	18-1605	**5.84E-4 ± 2%**	**1.02E-1 ± 2%**	**6.86E-5 ± 2%**	**4.00E-4 ± 18%**
TI042-COMP-EFF-DUP	18-1606	**6.00E-4 ± 2%**	**1.05E-1 ± 2%**	**6.30E-5 ± 9%**	**3.65E-4 ± 6%**
	RPD	3%	3%	9%	9%

Total Alpha

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Total Alpha
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115, Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Radiochemical Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation for Gross Alpha and Gross Beta Analysis*
Technician/Analyst:	LP Darnell (05/15/2018)
Spike Standard ID's:	R-687-a-5 (Pu-239)
Analysis Procedure:	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Reference Date:	Not applicable
Analysis Date(s) or Date Range:	5/16/18
Technician/Analyst:	T Trang-Le
Analysis Data (File):	18-1604 Fiskum.xls
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ludlum alpha scintillation detectors – See attached M&TE list

T Trang-le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

Sample Results

See attached data report, sample results for Analytical Service Request (ASR) 0521.01. All data are reported in units of µCi/mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Three samples submitted under ASR 0521.01 requested analyses for Total Alpha. The three samples for total alpha were acid digested along with the other samples submitted on this ASR that required acid digestion. The acid digestion was performed in laboratory 420 utilizing a mixture nitric/hydrochloric acid heated in a temperature controlled heat block using procedure RPG-CMC-128. An aliquot of each acid digestate was mounted for total alpha counting using procedure RPG-CMC-4001, then counted using alpha gas proportional counters per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

QC samples prepared prior to alpha counting include a laboratory blank, laboratory duplicates, a reagent spike (RS, Pu-239), and addition of Pu-239 standard to a diluted aliquot of the sample selected as the matrix spike (MS).

A summary of the Total Alpha analysis results, including QC sample performance, is given in the attached data report.

Tracer:

Tracer is not used for this analysis.

Laboratory Preparation Blank (LB) and Digestion Process Blank (PB):

The laboratory digestion process blank (7.98E-6 ± 195% µCi/mL) was less than 5 % of the sample activity, meeting the acceptance criterion for the laboratory digestion process blank. The laboratory preparation blank (1.35E-7 ± 212% µCi/mL) was also less than 5% of the sample activities, meeting the acceptance criterion for the laboratory process blank.

Blank Spike (BS)/Reagent Spike (RS):

The RS (Pu-239) recovery of 97% meets the acceptance criterion of 80% to 120% recovery.

Matrix Spike (MS):

The MS (Pu-239) recovery of 97% meets the acceptance criterion of 75% to 125%. The MS sample was prepared by adding a known Pu-239 standard quantity to an aliquot of the sample. Sample number 18-1605 (TI042-COMP-EFF) was selected as the matrix spike sample.

Laboratory Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤ 20% RPD. The ASO QAP further specifies that the two results are statistically different at the 95% confidence level. When sample results are above detection but duplicate results exceed 20% RPD, the mean difference evaluation is performed to determine if the results are statistically different at the 95% confidence level. For sample 18-1605 (TI042-COMP-EFF), the duplicate results were 33% RPD and for sample 18-1606 (TI042-COMP-EFF) the duplicate results were 75%. Applying the mean difference calculation to sample 18-1605, the mean difference value is 0.39 and for sample 18-1606, the mean difference value is 1.31. Both mean difference results are below 1.96 thus the results are not statistically different at the 95% confidence level.

Instrument Quality Control

Alpha Ludlum detectors undergo initial calibration to determine the detector efficiency. The established efficiency for each detector is used in the final calculation of the sample total alpha activity. Continuing calibration verification checks are performed on the detectors once per day as the system is used.

Detector backgrounds are obtained once per day or as the system is used or per batch.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 0521.01.

Total Beta

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Total Beta
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115, Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Radio Chemical Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation for Gross Alpha and Gross Beta Analysis*
Technician/Analyst:	LP Darnell, (5/15/18)
Spike Standard ID's:	R-693-b-6 (Sr-90)
Analysis Procedure:	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Reference Date:	Not applicable
Analysis Date(s) or Date Range:	5/16/18
Technician/Analyst:	T Trang-Le
Analysis Data (File):	18-1604 Fiskum.xls
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	LB4100 detectors – See attached M&TE list

T Trang-le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

Sample Results

See attached data report, sample results for Analytical Service Request (ASR) 0521.01. All data are reported in units of µCi/mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Three samples submitted under ASR 0521.01 requested analyses for Total Beta. The three samples for total beta were acid digested along with the other samples submitted on this ASR that required acid digestion. The acid digestion was performed in laboratory 420 utilizing a mixture nitric/hydrochloric acid heated in a temperature controlled heat block using procedure RPG-CMC-128. An aliquot of each acid digestate was mounted for total beta counting using procedure RPG-CMC-4001, then counted using beta gas proportional counters per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

QC samples prepared prior to beta counting include a laboratory blank, laboratory duplicates, a reagent spike (RS, Sr-90), and addition of Sr-90 standard to a diluted aliquot of the sample selected as the matrix spike (MS).

A summary of the Gross Beta analysis results, including QC sample performance, is given in the attached data report.

Tracer:

Tracer is not used for this analysis.

Laboratory Preparation Blank (PB):

The laboratory digestion process blank (3.20E-7 ± 322% µCi/mL) was less than 5 % of the sample activity, meeting the acceptance criterion for the laboratory digestion process blank. The laboratory preparation blank (9.68E-5 ± 58%µCi/mL) was also less than 5% of the sample activities, meeting the acceptance criterion for the laboratory process blank.

Blank Spike (BS)/Reagent Spike (RS):

The RS (Sr-90) recovery of 112% meets the acceptance criterion of 80% to 120% recovery.

Matrix Spike (MS):

The MS (Sr-90) spike activity was too low to measure relative to the sample activity. Note: The MS sample was prepared by adding a known Sr-90 standard quantity to an aliquot of the leachate. Sample number 18-1605 (TI042-COMP-EFF) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

For sample 18-1605 (TI042-COMP-EFF), the duplicate results were 1% RPD and for sample 18-1606 (TI042-COMP-EFF) the duplicate results were 8%, both meeting the acceptance criterion of ≤ 20% RPD

Instrument Quality Control

Beta LB4100 detectors undergo initial calibration to determine the detector efficiency. The established efficiency for each detector is used in the final calculation of the sample total beta activity. Continuing calibration verification checks are performed on the detectors once per day as the system is used.

Detector backgrounds are obtained once per day or as the system is used or per batch.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 0521.01.

Tritium by Liquid Scintillation Spectrometry

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COM-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Tritium
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None ☐ Digested as per RPG-CMC-128, Rev.1, *HNO_3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Samples were ashed at 750°C for 24 hours, then residual ash was dissolved using mixtures of HNO_3 and HF acids
Pre-dilution Prior to Radiochemical Processing?	☐ No ☒ Yes
Procedure:	RPG-CMC-4019, Rev. 0, *Measurement of Tritium in Heavily Contaminated Samples*
Spike Standard ID:	R-505-a-20 (H-3)
Date:	06/22/2018
Technician/Analyst:	L. Darnell
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	NA
Analysis Date or Date Range:	June 22, 2018
Technician/Analyst:	LP Darnel, CZ Soderquist and TL Trang-Le
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 18\18-1604 Fiskum.xls
ASO Project 98620 File:	File Plan 5872: 71274 Sample preparation and analysis records; LSC 3100 TR calibration, daily checks, and maintenance records; and standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Perkin Elmer Tri-Carb 3100 TR, Serial # DG08061340, RPL 425, Mettler AT400, Serial # 111362654, RPL420 and Mettler AT400, Serial # 1113292667, RPL420

T Trang-Le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

SAMPLE RESULTS

See attached data report, Sample Results for ASR 0521.01. All sample data are reported in µCi/ml with a 1-σ uncertainty (see Comments).

ASO Project File, ASR 0521.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, laboratory bench records, and Liquid Scintillation Counter Analysis printouts. Detector calibration records, standard certificates, control charts and balance calibration records can be found in the ASO Records.

Sample preparation, separation, mounting, and counting

Three samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed for H-3 by chemical separation and liquid scintillation counting. A direct aliquot of the parent samples was processed through procedure RPG-CMC-4019, Rev. 0, *Measurement of Tritium in Heavily Contaminated Samples*. The tritium solutions were prepared for beta counting by liquid scintillation counting (RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry").*

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in RPL/420 include a laboratory separation blank (LB), sample duplicate, matrix spike (i.e., addition of H-3 standard to an aliquot of one of the samples) and reagent spike.

Instrument Calibration Control

Laboratory Preparation Blank (PB):

The activity level of H-3 present in the laboratory preparation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. The customer requested target MDL of 1E-5 µCi/ml was achieved for all samples with measurable activity above the sample specific MDC. Note: The samples were processed "As Received" from parent material, thus a sample preparation blank is not prepared.

Reagent Spike (RS):

The RS recovery of 94% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 98% meets the acceptance criteria of 75% to 125% recovery. Sample TI042-COMP-EFF (18-1605) was used for the matrix spike analyses.

Duplicate -- Relative Percent Difference (RPD):

> Duplicate results 18-1605 (TI042-COMP-EFF) are required to agree within ≤ 20% RPD. The ASO QAP further specifies that the two results need to be > 5 times the MDC or have individual uncertainties < 20%. Duplicate results were 7% RPD; thus meeting the ≤ 20% requirement.

Instrument Quality Control

The liquid scintillation counter (LSC) is calibrated for tritium and C-14 using quenched standard sets that are purchased from the vendor. Daily control counts are then performed using a tritium, C-14, and a background count sample. The instrument software assesses the performance of the control counts and provides control charts to ensure the continuing calibration of the instrument. If the daily performance check fails, then the instrument is not used. Preventative maintenance and repairs are performed by the vendor under our service contract. The counting efficiency for H-3 is assumed to be 100%; therefore no specific H-3 calibration is performed. The LSC system calibration and performance is verified by assessing the recovery of a reagent spike and a matrix spike that are included in every batch of samples. A preparation blank (i.e., digestion blank) and a laboratory separation blank are also included with every batch of samples; the instrument background is subtracted from all results and the preparation and separation blanks are used to assess sample contamination during sample processing steps.

Assumption and Limitations of the Data

The 1-σ uncertainty reported for each H-3 result has been set at 2%. Although the calculated uncertainty values are less than 2% for all samples, the radiochemistry convention is to not report calculated uncertainties less than 2%, but to provide a more realistic estimate of the uncertainty in view of systematic uncertainties that are not fully accounted for in the instrument uncertainty calculations based on counting statistics.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radio-analytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the H-3 spike was not added prior to the digestion process.
3. Radiochemistry Electronic Systems File "RPG-RC\PNL\Projects\Backup files\Backup 18\ 18-1604 Fiskum.xls " has been created for this report. Supporting records such as Pipette Performance Verification forms, Laboratory Bench Record, Laboratory Sample Preparation Bench Sheet, Standards Certifications and preparation records, and balance calibration and performance check records are maintained per NC&E Group ASO File Plan 5872.
4. Sample results are compared to the process blank results to evaluate if the blank contains 5% or more of the measured isotope; the process blank results have been adjusted for all processing factors for the evaluation of the 5% criterion.

5. The stated 1-σ uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting error.

Attachment: Data Report -- Sample Results for ASR 0521.01.

Sr-90 by Liquid Scintillation Spectrometry

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	Fiskum
Total # of Samples:	13

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COM-EFF
18-1606	TI042-COMP-EFF-DUP
18-1607	TI042-L-F2-A
18-1608	TI042-L-F3-A
18-1609	TI042-L-F4-A
18-1610	TI042-L-F5-A
18-1611	TI042-L-F7-A
18-1612	TI042-L-F9-A
18-1613	TI042-L-F11-A
18-1614	TI042-L-F13-A
18-1615	TI042-L-F15-A
18-1616	TI042-L-F18-A

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report

Analysis Type:	Sr-90
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-128, Rev.1, *HNO_3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other: Samples were ashed at 750°C for 24 hours, then residual ash was dissolved using mixtures of HNO_3 and HF acids
Pre-dilution Prior to Radiochemical Processing?	☐ No ☒ Yes
Separation Procedure:	RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin*
Spike Standard ID:	R-693-a-3 (Sr-90)
Separation Date:	06/07/2018 @11:45 a.m.
Technician/Analyst:	L. Darnell
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	NA
Analysis Date or Date Range:	June 7, 2018 (first count), June 11, 2018 (second count)
Technician/Analyst:	LP Darnel, CZ Soderquist and TL Trang-Le
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 18\18-1604 Fiskum.xls
ASO Project 98620 File:	File Plan 5872: 71274 Sample preparation and analysis records; LSC 3100 TR calibration, daily checks, and maintenance records; and standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Perkin Elmer Tri-Carb 3100 TR, Serial # DG08061340, RPL 425, Mettler AT400, Serial # 111362654, RPL420 and Mettler AT400, Serial # 1113292667, RPL420

T Trang-le / 6/28/18

Preparer **Date**

/ 6/28/18

Reviewer **Date**

SAMPLE RESULTS

See attached data report, Sample Results for ASR 0521.01. All sample data are reported in μCi/ml with a 1-σ uncertainty (see Comments).

ASO Project File, ASR 0521.01 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, laboratory bench records, and Liquid Scintillation Counter Analysis printouts. Detector calibration records, standard certificates, control charts and balance calibration records can be found in the ASO Records.

Sample preparation, separation, mounting, and counting

Thirteen samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed for Sr-90 by chemical separation and liquid scintillation counting. The samples were first acid digested in laboratory 420 utilizing a mixture nitric/hydrochloric acid heated in a temperature controlled heat block using procedure RPG-CMC-128, Rev.1, *HNO_3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater*. The samples were then run through the Sr-90 separation procedure (RPG-CMC-476, Rev. 0, *Strontium Separation using Eichrom Strontium Resin)* and the final Sr-90 solutions were prepared for beta counting by liquid scintillation counting (RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry").*

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in RPL/420 include a laboratory preparation blank (Lab Blnk-1604), sample duplicate, matrix spike (i.e., addition of Sr-90 standard to an aliquot of one of the samples) and reagent spike.

Instrument Calibration Control

Laboratory Preparation Blank (PB):

The activity level of Sr-90 present in the laboratory preparation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. Note: Samples 18-1605 thru 18-1616 the measureable activity of Sr-90 is very near the detection limit. The customer requested target MDL of 1E-4 μCi/ml was achieved for all samples with measurable activity above the sample specific MDC.

Reagent Spike (RS):

The RS recovery of 100% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 101% meets the acceptance criteria of 75% to 125% recovery. Sample TI042-COMP-FEED (18-1604) was used for the matrix spike analyses.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results 18-1604 (TI042-COMP-FEED) are required to agree within ≤ 20% RPD. The ASO QAP further specifies that the two results need to be > 5 times the MDC or have individual uncertainties < 20%. Duplicate results were 4% RPD; thus meeting the ≤ 20% requirement.

Instrument Quality Control

The liquid scintillation counter (LSC) is calibrated for tritium and C-14 using quenched standard sets that are purchased from the vendor. Daily control counts are then performed using a tritium, C-14, and a background count sample. The instrument software assesses the performance of the control counts and provides control charts to ensure the continuing calibration of the instrument. If the daily performance check fails, then the instrument is not used. Preventative maintenance and repairs are performed by the vendor under our service contract. The counting efficiency for Sr-90 is assumed to be 100%; therefore no specific Sr-90 calibration is performed. The LSC system calibration and performance is verified by assessing the recovery of a reagent spike and a matrix spike that are included in every batch of samples. A preparation blank (i.e., digestion blank) and a laboratory separation blank are also included with every batch of samples; the instrument background is subtracted from all results and the preparation and separation blanks are used to assess sample contamination during sample processing steps.

Assumption and Limitations of the Data

The 1-σ uncertainty reported for each Sr-90 result has been set at 2%. Although the calculated uncertainty values are less than 2% for all samples, the radiochemistry convention is to not report calculated uncertainties less than 2%, but to provide a more realistic estimate of the uncertainty in view of systematic uncertainties that are not fully accounted for in the instrument uncertainty calculations based on counting statistics.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radio-analytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the Sr-90 spike was not added prior to the digestion process.
3. Radiochemistry Electronic Systems File "RPG-RC\PNL\Projects\Backup files\Backup 18\ 18-1604 Fiskum.xls " has been created for this report. Supporting records such as Pipette Performance Verification forms, Laboratory Bench Record, Laboratory Sample Preparation Bench Sheet, Standards Certifications and preparation records, and balance calibration and performance check records are maintained per NC&E Group ASO File Plan 5872.
4. Sample results are compared to the process blank results to evaluate if the blank contains 5% or more of the measured isotope; the process blank results have been adjusted for all processing factors for the evaluation of the 5% criterion.

5. The stated 1-σ uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting error.

Attachment: Data Report -- Sample Results for ASR 0521.01.

Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Tc-99 Analysis

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	Tc-99
Sample Processing Prior to Radiochemical Processing/Analysis	☒ None ☐ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115 Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☐ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☐ No ☐ Yes
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta Analyses.*
Technician/Analyst:	LP Darnell, (05/15/2018)
Spike Standard ID's	R-687-a-5 (Pu-239), R-493-b-6 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le 05/16/2018
RadioChemical Preparation Procedure:	RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*
Technician/Analyst:	LP Darnell, (6/15/2018)
Spike and Tracer Standard ID's:	R-540-b-8 (6/15/2018) (Tc-99)
Analysis Procedure:	RPG-CMC-474, Rev. 1, *Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry*
Reference Date:	Not applicable
Analysis Date(s) or Date Range:	6/15/2018 and 6/16/2018
Technician/Analyst:	CZ Soderquist
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\18-1604 Fiskum.xls
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Perkin Elmer 3100 TR Liquid scintillation spectrometer– See attached M&TE list.

T Trang-Le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0521.01. All data are reported in units of µCi/mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Three samples submitted under ASR 0521.01 were analyzed for Tc-99. All the samples were prepared in Laboratory 420. A direct aliquot of the parent samples was processed using procedure RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*.

The samples were counted on June 15 and 16, 2018; no decay corrections were made.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a laboratory blank and sample duplicates, matrix spike, reagent spike and addition of Tc-99 standard to a separate aliquot of one of the samples.

The QC sample results for Tc-99 have been evaluated and are discussed below. A summary of the Tc-99 analysis results, including QC sample performance, is given in the attached data report.

Tracer:

There is no tracer for Tc-99 analysis.

Laboratory Separation Blank (LB):

The activity level of Tc-99 present in the laboratory separation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. The customer requested target MDL of 1E-4 µCi/ml was achieved for all samples with measurable activity above the sample specific MDC. Note: The samples were processed "As Received" from parent material, thus a sample preparation blank is not prepared.

Reagent Spike (RS):

The RS recovery of 93% (Tc-99) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 73% (Tc-99) does not meet the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after leaching, by adding a known quantity of Tc-99 standard to the leachate. Sample number 18-1606 (TI042-COMP-EFF-DUP) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

Duplicate results for 18-1606 (TI042-COMP-EFF-DUP) are required to agree within ≤20% RPD. The ASO QAP further specifies that the two results need to be > 5 times the MDC or have individual uncertainties < 20%. Duplicate results were 5% RPD, thus meeting the ≤ 20% requirement.

Instrument Quality Control

LSC counters receive initial calibration with NIST traceable sources to determine the counter efficiency. When internal tracers are not used, the counter efficiency is used in calculation of final results.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 0521.01.

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Battelle PNNL/RPL/ASO Radiochemistry Analysis Report
P.O. Box 999, 902 Battelle Blvd., Richland, Washington 99352

Am-241, Cm-242 and Cm-243+244 Analysis

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	AEA – Am-241
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115 Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell, (05/15/2018)
Spike Standard ID's	R-687-a-5 (Pu-239), R-493-b-6 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le (05/15 to 05/16/2018)
Americium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Sr-90*
Technician/Analyst:	LP Darnell, (05/30/2018)
Spike and Tracer Standard ID's:	R-437-b-500-8 (Am-241), R-628-a-9 (Am-243 tracer)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell (05/30/2018)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	June 4-5, 2018
Technician/Analyst:	T. Trang-Le & CZ Soderquist
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup18\18-1604 Fiskum.xls
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

T Trang-le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0521.01. All data are reported in units of µCi per mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

All three samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed for analyzed for americium and curium by Alpha Spectrometry. All the samples were prepared in laboratory 420. Aliquots of the acid digestions samples were used for radioanalytical analyses; only Am/Cm-AEA data are included in this report.

Following the digestion process of samples, the Am/Cm was separated from the leachate by anion exchange using procedure RPG-CMC-4017. The separated Am/Cm fraction was then mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted using alpha spectrometry using procedure RPC-CMC-422. The samples were counted on June 4-5, 2018; no decay corrections were made.

Alpha and beta analyses were performed on each sample for checking the internal consistency of the Am alpha isotopic data. Total alpha and total. beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a laboratory blank (LB) and sample duplicates. Additional QC samples were prepared prior to alpha counting including a reagent blank spike (RS, Am-241), and addition of Am-241 standard to an aliquot of the sample 18-1606 (TI042-COMP-EFF-DUP), selected as the matrix spike (MS). Cm spike and tracers are not used for this analyses method as the Am and Cm share the same chemistry through the separation processes.

The QC sample results for Am-AEA have been evaluated and are discussed below. A summary of the Alpha-AEA analysis results, including QC sample performance, is given in the attached data report.

Tracer:

> The Am-243 tracer is added to every sample after appropriate dilution and prior to americium separations. The Am-243 tracer corrects for radiochemical yield and mathematically removes the detector counting efficiency from the results calculations. Tracer recovery is required to be high enough to provide acceptable counting statistics. The Am-243 tracer counting statistics were acceptable for all samples. The tracer recoveries ranged from 88% to 101%.

Laboratory Preparation Blank (PB):

The activity level of Am-241 present in the laboratory preparation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. The customer requested target MDL of 1E-6 µCi/ml for Am-241 was not achieved for all samples with measurable activity above the sample specific MDC. The MDA values for the Am-241 ranged from 1.7E-6 µCi/ml to 1.9E-5 µCi/ml.

The activity level of Cm-242 present in the laboratory preparation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. The customer requested target MDL of 1E-6 µCi/ml for Cm-242 was not achieved for all samples with measurable activity above the sample specific MDC. The Cm-242 results are very near or below the method detection limit. The MDA values for the Cm-242 ranged from 9.7E-7 µCi/ml to 9.1E-6 µCi/ml.

The activity level of Cm-243+244 present in the laboratory preparation blank (-2.1E-7 µCi/ml) is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC. The customer requested target MDL of 1E-6 µCi/ml for Cm-243+244 was not achieved for all samples with measurable activity above the sample specific MDC. The MDA values for the Cm-243+244 ranged from 1.1E-6 µCi/ml to 9.6E-6 µCi/ml.

Blank Spike (BS)/Reagent Spike (RS):

The RS recovery of 95% (Am-241) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 90% (Am-241) meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared after digestion, by adding a known quantity of Am-241 standard to a diluted aliquot of the digestate. Sample number 18-1606 (TI042-COMP-EFF-DUP) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

The Am-241 sample and duplicate RPD range from 4% within the DQO of less than ≤ 20% RPD. As for Cm-243+244, sample and duplicate RPD range from 9% within the DQO of less than ≤ 20% RPD.

Instrument Quality Control

Alpha counters receive initial calibration with NIST traceable sources to determine the counter efficiency. When internal tracers are not used, the counter efficiency is used in calculation of final results.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A laboratory blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 0521.01.

Neptunium 237 Analysis

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COMP-EFF
18-1606	TI042-COMP-EFF-DUP

Analysis Type:	AEA – Np-237
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per RPG-CMC-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☒ No
	☐ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell (05/15/2018)
Spike Standard ID's	R-687-a-5 (Pu-239), R-493-b-6 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le (05/15 to 05/16-2018)
Neptunium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	LP Darnell, (06/19/2018)
Spike Standard ID's:	RS-686-a-1 (Np-237)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, (06/19/2018)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	June 21-22, 2018
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\18-1604 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

T Trang-Le / 7/13/18
Preparer **Date**

/ 7/13/18
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0521.01. All data are reported in units of uCi per mL with a 1-σ uncertainty unless noted otherwise (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Three samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed for neptunium by Alpha Spectrometry. The samples were acid digested using procedure RPG-CMC-128, "*HNO3-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater*". All the samples were prepared in laboratory 420. The leachate solution was used for radioanalytical analyses; only Np-AEA is reported in this report.

Following the digestion process, the Np was separated from the sludge leachate using anion exchange chromatography using procedure RPG-CMC-4017. The separated Np fraction was mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted by alpha spectrometry using procedure RPC-CMC-422.

Total alpha and total beta analyses were performed on each sample to obtain information to estimate aliquot sizes for other analyses and for checking the internal consistency of the Np alpha isotopic data. Total alpha and total beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples include an acid digestion blank and sample duplicate. Additional QC samples were prepared prior to separations; these include a laboratory separation blank, a reagent blank spike (BS), and a matrix spike (MS) made by adding Np-237 standard to a diluted sample.

Tracer:

Tracer is not used for analyses of Np.

Laboratory Preparation Blank (PB):

The Np-237 activity measured in the PB is required to be within the acceptance criteria of less than sample minimum detectable activity (MDA) or less than 5% of the sample isotope concentration. The Np-237 PB is <MDA; thus meeting the acceptance criteria. The customer requested target MDL of 1E-6 μCi/ml was achieved for all samples with measurable activity above the sample specific MDC.

Blank Spike (BS) – reagent spike (RS):

The RS recovery of 90% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 96% meets the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared "after" digestion, by adding a known Np-237 standard quantity to an aliquot of the leachate. Sample number 18-1604 (TI042-COMP-FEED) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results for 18-1604 (TI042-COMP-FEED) are required to agree within ≤20% RPD. The ASO QAP further specifies that the two results need to be > 5 times the MDC or have individual uncertainties < 20%. Duplicate results were 11% RPD, thus meeting the ≤ 20% requirement.

Instrument Quality Control

Alpha counters undergo calibration annually to determine the counter's efficiency over the normal calibration range of 3 to 6 MeV. The vendor software determines a constant detector efficiency for this energy range. Np samples are counted and results calculated using the established detector efficiency.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

Undissolved residue remained after the completion of the acid digestion/leach of all sludge samples.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. Post-Digestion Spike (PS) - A spike made after the initial sample preparation (e.g., fusion, digestion, or leach) is considered a PS. When extremely radioactive samples are analyzed, most of the radioanalytical spikes are made after the sample preparation (to avoid excessive consumption of spike and avoid creating unnecessary waste) and are post-digestion spikes. The MS prepared with this batch of sample is considered a PS, since the Np-237 spike was not added prior to the digestion process.

3. The 1-sigma uncertainty represents the total propagated error associated with processing and counting operations and include; weighing errors, volume uncertainties, and counting error.

4. The sample results are compared to the process blank to evaluate if the blank contains 5% or more of the measured isotope; the process blank result has been adjusted for all processing factors for evaluation of the 5% criterion.

5. The sample results are compared to the process blank to evaluate if the blank contains 5% or more of the measured isotope; the process blank result has been adjusted for all processing factors for evaluation of the 5% criterion.

Attachment: Data Report -- Sample Results for ASR 0521.01.

Plutonium 238, 239+240 Analysis

Project / WP#:	71274/N96056
ASR#:	0521.01
Client:	SK Fiskum
Total # of Samples:	13

RPL ID	Client Sample ID
18-1604	TI042-COMP-FEED
18-1605	TI042-COM-EFF
18-1606	TI042-COMP-EFF-DUP
18-1607	TI042-L-F2-A
18-1608	TI042-L-F3-A
18-1609	TI042-L-F4-A
18-1610	TI042-L-F5-A
18-1611	TI042-L-F7-A
18-1612	TI042-L-F9-A
18-1613	TI042-L-F11-A
18-1614	TI042-L-F13-A
18-1615	TI042-L-F15-A
18-1616	TI042-L-F18-A

Analysis Type:	AEA – Pu-238, Pu-239+240
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☐ Digested as per PNL-ALO-106, Rev. 1, *Acid Digestion of Waters, Soils, and Sludges for Subsequent Radiochemical Sample Analyses* ☐ Fusion as per RPG-CMC-115 Rev. 0, *Solubilization of Metals from Solids Using a KOH-KNO$_3$ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO$_3$-HCL Acid extraction of Liquids for Metals Analysis Using a Dry-Block Heater*
Pre-dilution Prior to Radiochemical Processing?	☐ No ☒ Yes -- *example 2 mL to 100 mL; 50x dilution*
Total Alpha Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta analyses.*
Technician/Analyst:	LP Darnell, (05/15/2018)
Spike Standard ID's	R-687-a-5 (Pu-239)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le (05/15 to 05/16-2018)
Plutonium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*
Technician/Analyst:	LP Darnell, (05/31/2018)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, (05/31/2018)
Spike and Tracer Standard ID's:	R-687-a-5 (Pu-239), R-700-1 (Pu-242 tracer)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	June 4-7, 2018
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\18-1604 Fiskum.xls
CMC Project 98620 File:	File Plan 5872: T 71274: Sample preparation and analysis records; T-4.4 Alpha Detector calibration, calibration verification checks, and maintenance records; and T3 Standard certificates and preparation. Also balance calibration and instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list

T Trang-le / 7/13/18

Preparer **Date**

/ 7/13/18

Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0521.01. All data are reported in units of µCi per mL with a 1-σ uncertainty.

Sample Preparation, Separation, Mounting and Counting Methods

Thirteen samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed for plutonium by Alpha Spectrometry. All the samples were prepared in RPL/420. Aliquots of the acid digestions were used for radioanalytical analyses; only Pu-AEA data are included in this report.

Following the digestion process of all samples, the Pu was separated by anion exchange using procedure RPG-CMC-4017. The separated Pu fraction was then mounted for alpha spectrometry by co-precipitation using procedure RPG-CMC-496, and then counted using alpha spectrometry using procedure RPC-CMC-422. The samples were counted on June 5-7, 2018; no decay corrections were made.

Alpha and beta analyses were performed on each sample for checking the internal consistency of the Pu alpha isotopic data. Total alpha and total beta activity were measured by evaporating small aliquots of leachate onto counting planchets per procedure RPG-CMC-4001 and counting per procedure RPG-CMC-408.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in RPL/420 include a laboratory blank (LB) and sample duplicates. Additional QC samples were prepared prior to alpha counting including a laboratory blank, a reagent blank spike (RS, Pu-239), and addition of Pu-239 standard to an aliquot of the sample digestate selected as the matrix spike (MS).

The QC sample results for Pu-AEA have been evaluated and are discussed below. A summary of the Pu-AEA analysis results, including QC sample performance, is given in the attached data report.

Tracer:

The Pu-242 tracer is added to every sample after appropriate dilution and prior to plutonium separations. The use of a Pu-242 tracer corrects for radiochemical yield and mathematically removes the detector counting efficiency from the results calculations. Tracer recovery is required to be high enough to provide acceptable counting statistics. The Pu-242 tracer counting statistics were acceptable for all samples. The tracer recoveries ranged from 95% to 110%.

Laboratory Preparation Blank (PB):

The activity level of Pu-239 present in the laboratory preparation blank is below the activity present in the samples with measurable activity meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDC.

Reagent Spike (RS):

The RS recovery of 96% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 93% (Pu-239) meets the acceptance criterion of 75% to 125% recovery. Note: The MS sample was prepared after digestion, by adding a known quantity of Pu-239 standard to a diluted aliquot of the digestate. Sample number 18-1606 (TI042-COMP-EFF-DUP) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

The Pu-238 sample 18-1606 (TI042-COMP-EFF-DUP) and duplicate pair was 5%, within DQO. The RPD for Pu-239+240 is 6%, within the acceptance limit of ≤ 20% RPD.

Instrument Quality Control

Alpha counters receive initial calibration with NIST traceable sources to determine the counter efficiency. When internal tracers are not used, the counter efficiency is used in calculation of final results.

Detector backgrounds are determined every 4 weeks or after the last analytical run, whichever is longer. Detector background counts are subtracted from all subsequent sample counts. A process blank is analyzed with each analytical batch to evaluate for contamination in the sample preparation process.

Assumption and Limitations of the Data

None

Attachment: Data Report -- Sample Results for ASR 0521.01.

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Nuclear Chemistry and Engineering Group

filename: 18-1604 Fiskum
6/25/2018

Client: Fiskum
ASR: 0521.01

Project: 71274
N96056

Prepared by: 7/13/18

Technical Reviewer: T Trang-Le 7/13/18

Procedures:	RPG-CMC-128, Rev 1, HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater RPG-CMC-4001, Rev 1, Source Preparation For Gross Alpha and Gross Beta Analysis RPG-CMC-408, Rev 2, Total Alpha and Beta Analysis RPG-CMC-476, Rev 0, Strontium Separation Using Eichrom Strontium Resin RPG-CMC-496, Rev 1, Coprecipitation Mounting of Actinides for Alpha Spectrometry RPG-CMC-432, Rev 0, Technetium-99 Analysis RPG-CMC-474, Rev 1, Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry RPG-CMC-4019, Rev 0, Measurement of Tritium in Heavily Contaminated Samples
M&TE:	Ludlum, Alpha AEA, Perkin Elmer Tri Carb Model 3100 TR Liquid scintillation spectrometer
Count dates:	5/16/2018 (total alpha, total beta), 6/11/2018 (Sr-90), 6/16/2018 (Tc-99), 6/22/2018 (tritium)

		Measured Activity, µCi per mL ± 1s				
Sample	Lab ID	**Total Alpha**	**Total Beta**	**Tritium**	**Sr-90**	**Tc-99**
TI042-COMP-FEED	18-1604	**1.01E-1 ± 51%**	**1.58E+2 ± 4%**	**8.81E-4 ± 4%**	**5.93E-1 ± 2%**	**9.20E-2 ± 2%**
	18-1604 DUP	-	-	-	**5.69E-1 ± 2%**	-
	RPD				4%	
TI042-COMP-EFF	18-1605	**5.80E-4 ± 28%**	**1.78E-1 ± 4%**	**8.28E-4 ± 4%**	**2.57E-4 ± 12%**	**8.16E-2 ± 2%**
	18-1605 DUP	**4.15E-4 ± 33%**	**1.77E-1 ± 4%**	**8.85E-4 ± 4%**	-	-
	RPD	33%	1%	7%		
TI042-COMP-EFF-DUP	18-1606	**1.32E-3 ± 17%**	**2.09E-1 ± 4%**	**8.78E-4 ± 4%**	**2.86E-4 ± 11%**	**9.05E-2 ± 2%**
	18-1606 DUP	**6.01E-4 ± 26%**	**1.92E-1 ± 4%**	-	-	**9.49E-2 ± 2%**
	RPD	75%	8%			5%

		Measured Activity, µCi per mL ± 1s				
Sample	Lab ID	**Total Alpha**	**Total Beta**	**Tritium**	**Sr-90**	**Tc-99**
TI042-L-F2-A	18-1607			-	**9.73E-4 ± 4%**	-
TI042-L-F3-A	18-1608			-	**8.44E-4 ± 4%**	-
TI042-L-F4-A	18-1609			-	**8.57E-4 ± 4%**	-
TI042-L-F5-A	18-1610			-	**7.99E-4 ± 4%**	-
TI042-L-F7-A	18-1611			-	7.25E-4 ± 74%	-
TI042-L-F9-A	18-1612			-	2.04E-3 ± 27%	-
TI042-L-F11-A	18-1613			-	2.40E-3 ± 24%	-
TI042-L-F13-A	18-1614			-	2.85E-3 ± 20%	-
TI042-L-F15-A	18-1615			-	**3.59E-3 ± 16%**	-
TI042-L-F18-A	18-1616			-	**7.61E-3 ± 8%**	-
	Reagent Spike	97%	112%	94%	100%	93%
	Matrix Spike	97%	--	98%	101%	73%
	Lab Blk-1604	1.35E-7 ± 212%	3.20E-7 ± 322%	4.21E-6 ± 15%	-5.09E-5 ± 51%	2.26E-6 ± 97.7%
	Acid digestion blank	7.98E-6 ± 195%	9.68E-5 ± 58%			

Shaded cells are below sample specific minimum detectable concentration
Gross beta matrix spike was too small for the sample activity

Pacific Northwest National Laboratory
PO Box 999, Richland, WA
Nuclear Chemistry and Engineering Group

filename: 18-1604 Fiskum
6/25/2018

Client: S. Fiskum
ASR: 0521.01

Project: 71274
N96056

Prepared by: Karl N. Pool 7/13/18

Technical Reviewer: T Trang-le 7/13/18

Procedures:
RPG-CMC-128, Rev 1, HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater
RPG-CMC-4001, Rev 1, Source Preparation For Gross Alpha and Gross Beta Analysis
RPG-CMC-4015, Rev 0, Analysis of Soil & Sediment Samples of Actinides and Sr-90
RPG-CMC-4017, Rev 0, Analysis of Environmental Water Samples for Actinides and Strontium-90
RPG-CMC-496, Rev 1, Coprecipitation Mounting of Actinides for Alpha Spectrometry
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha Spectrometry

M&TE: Ludlum, Alpha spectrometry counting system
Count dates: 5/16/2018 (total alpha), 6/4 to 6/7/2018 (Pu, Am), 6/21 & 6/22 (Np)

		Measured Activity, µCi per mL ± 1s						
Sample	Lab ID	**Total Alpha**	**Np-237**	**Pu-238**	**Pu-239+240**	**Am-241**	**Cm-243+244**	**Cm-242**
TI042-COMP-FEED	18-1604	**1.01E-1 ± 51%**	**3.72E-5 ± 5%**	**1.14E-4 ± 12%**	**5.43E-4 ± 6%**	**4.17E-4 ± 6%**	**1.43E-5 ± 35%**	0.00E+0
	18-1604 Dup	--	**4.14E-5 ± 4%**	--	--	--	--	--
			11%					
TI042-COMP-EFF	18-1605	**5.83E-4 ± 28%**	**1.24E-5 ± 7%**	**2.73E-5 ± 8%**	**1.91E-4 ± 3%**	**3.91E-4 ± 3%**	**1.66E-5 ± 10%**	9.86E-7 ± 43%
TI042-COMP-EFF-DUP	18-1606	**1.32E-3 ± 17%**	**1.21E-5 ± 7%**	**2.91E-5 ± 7%**	**1.83E-4 ± 3%**	**3.75E-4 ± 3%**	**1.76E-5 ± 10%**	1.77E-6 ± 32%
	18-1606 DUP	**6.01E-4 ± 26%**	--	**3.06E-5 ± 7%**	**1.95E-4 ± 3%**	**3.92E-4 ± 3%**	**1.61E-5 ± 10%**	7.02E-7 ± 51%
	RPD	75%		5%	6%	4%	9%	
TI042-L-F2-A	18-1607		--	**3.19E-5 ± 7%**	**2.14E-4 ± 3%**	--	--	--
TI042-L-F3-A	18-1608		--	**2.86E-5 ± 7%**	**2.16E-4 ± 3%**	--	--	--
TI042-L-F4-A	18-1609		--	**3.53E-5 ± 7%**	**2.34E-4 ± 3%**	--	--	--
TI042-L-F5-A	18-1610		--	**4.18E-5 ± 6%**	**2.72E-4 ± 3%**	--	--	--
TI042-L-F7-A	18-1611		--	**4.99E-5 ± 6%**	**2.66E-4 ± 3%**	--	--	--
TI042-L-F9-A	18-1612		--	**4.07E-5 ± 6%**	**2.86E-4 ± 3%**	--	--	--
TI042-L-F11-A	18-1613		--	**4.06E-5 ± 6%**	**2.84E-4 ± 3%**	--	--	--
TI042-L-F13-A	18-1614		--	**6.15E-5 ± 18%**	**3.07E-4 ± 7%**	--	--	--
TI042-L-F15-A	18-1615		--	**7.53E-5 ± 15%**	**3.17E-4 ± 7%**	--	--	--
TI042-L-F18-A	18-1616		--	**6.88E-5 ± 15%**	**3.20E-4 ± 7%**	--	--	--
	Reagent Spike	97%	90%	--	96%	95%	--	--
	Matrix Spike	97%	96%	--	93%	90%	--	--
	Acid digestion blank	7.98E-6 ± 195%	--	--	--	--	--	--
	Lab Blk-1604	1.35E-7 ± 212%	-4.91E-8 ± 371%	1.43E-6 ± 55%	1.85E-6 ± 47%	2.31E-6 ± 36%	-2.10E-7 ± 91%	1.40E-7 ± 137%

Shaded cells are below detection limit

Battelle PNNL/RPL/Inorganic Analysis ... ICP-MS Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	71274 / N96056
ASR#:	0521.01
Client:	S. Fiskum
Total Samples:	3 (Aqueous)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-1604	TI042-COMP-FEED	Hanford Tank Waste Matrix	
18-1605	TI042-COMP-EFF	Hanford Tank Waste Matrix	
18-1606	TI042-COMP-EFF-DUP	Hanford Tank Waste Matrix	

Sample Preparation: RPG-CMC-128, Rev. 1. "HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater performed by L. Darnell between 05/11/18 and 05/14/18. Prior to ICP-MS analysis all samples were further diluted in 2% v/v HNO_3 containing 100 ppb Au by D. Cherkasov on 07/20/18.

Procedure: RPG-CMC-292, Rev. 1, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."

Analyst:	D. Cherkasov G. Brown	**Analysis Date:**	07/20/2018	**ICP File:**	M0071

See Chemical Measurement Center 98620 file: ICP-325-405-3
(Calibration and Maintenance Records)

M&TE:				
	☒	PerkinElmer NexION™ 350X ICP-MS	SN: 85VN4070702	RPL 405
	☐	Sartorius R200D Balance	SN: 39080042	RPL 405
	☒	Ohaus Pioneer PA224C Balance	SN: B725287790	RPL 405
	☒	Mettler AT400 Balance	SN: M19445	RPL 405 FH
	☐	Mettler AT400 Balance	SN: 1113162654	RPL 420 FH
	☒	Mettler AT400 Balance	SN: 1113292667	RPL 420 FH
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH

Garrett N. Brown
Report Preparer

8/1/18
Date

Review and Concurrence

8/1/18
Date

Three liquid samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed by ICP-MS. Samples 18-1604, 18-1605, 18-1606, and DUP-1606 (0.5 mL) were prepared following RPL procedure RPG-CMC-128 and were diluted to approximately 25 mL. All samples were further diluted in 2% HNO_3 prior to analysis. None of the samples were filtered.

All results are reported on a mass per unit mass basis (ng/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-MS Data Report. Mercury was the only AOI; all results were reported for Hg-202. The quality control (QC) results for the AOI have been evaluated and are presented below.

Calibration of the ICP-MS was done following the manufacturer's recommended calibration procedure using multi-analyte custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards were used to verify acceptance of the six-point calibration curve and for initial and continuing calibration verification (ICV/CCV).

The controlling document was procedure RPG-CMC-292, Rev 1, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)* and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan.* Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spike, duplicate, blank spike, reagent spike, matrix spike, and serial dilution were conducted during the analysis run.

Mercury carryover between samples is a common problem encountered during ICP-MS analyses. Carryover is often mitigated by diluting all solutions in either 2% HNO_3 or 2% HCl with 100 ppb Au and keeping the concentration of mercury to a minimum (<10 ppb) when possible. In this data set, mercury carryover in the CCBs analyzed immediately after the CCVs averaged 0.062 ppb (2.52%). The analyses of sequential blanks after the analyses of the CCV showed continuing, but slowly reducing Hg carry over. Based on this carry over issue, the sample results are below detection based on best professional judgement.

Internal Standard (IS):

All solutions (blanks, standards, QC checks, and samples) were mixed in-line with a solution containing 10 ppb each of Li-6, Sc-45, Y-89, In-115, Tb-159, and Bi-209 as the internal standard (IS). The AOI (Hg-202) data were normalized using the data for the closest IS mass (e.g., Bi-209). The Bi-209 IS recoveries ranged from 97.2% to 101.8% for the entire analysis sequence, which were within the acceptance criterion of 30% to 120%.

Preparation Blank (PB):

A preparation blank sample, BLK-1604 (reagents only), was prepared for the extraction process. The concentration of Hg-202 was within the acceptance criteria of <EQL (estimated quantitation level), <50% regulatory decision level, or less than ≤10% of the concentration in the samples. A diluent blank from the ICP-MS laboratory (consisting of

2% HNO_3 and 100 ppb Au) was also analyzed. Results for the diluent blank were also within the acceptance criteria.

Matrix-Spike (MS) Sample:

A matrix spike (MS) sample (18-1606 and spikes) was prepared for the extraction process. The standard spike reagents used in the preparation lab do not contain mercury. Recovery values are listed for all analytes included in the MS that were measured at or above the EQL. The recovery value for the AOI was not reported since all matrix spike sample results were below EQL. The acceptance criterion is 75% to 125%. In addition to the MS sample, an additional post-digestion spike (PS) sample was analyzed as described below.

Reagent-Spike (RS) Sample:

A reagent spike (RS) sample (reagents and spikes) was prepared for the extraction process. The standard spike reagents used in the preparation lab do not contain mercury. Recovery values are listed for all analytes included in the RS that were measured at or above the EQL. The recovery value for the AOI was not reported since all RS sample results were below EQL. The acceptance criterion are 80% to 120%.

Blank Spike (BS)/Laboratory Control Sample (LCS):

A blank spike (BS) sample was prepared by spiking a 2% HNO_3 containing 100 ppb Au blank with an equivalent volume of the CCV-Hg-2ppb standard (1:1 ratio). The recovery for Hg-202 was 110%, which is within the acceptance criteria of 80% to 120% recovery.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

Duplicates of sample 18-1606 were prepared and analyzed. No RPD are listed for the AOI since all sample results were below EQL. The acceptance criterion is ≤20% RPD for liquid samples when the results are > EQL

Initial/Continuing Calibration Verification (ICV/CCV):

The ICV/CCV solution was analyzed immediately after calibration, after each group of not more than eleven samples, and at the end of the analytical run. The concentrations of all AOI ranged from 122.1% to 132.3% and were outside of the acceptance criteria of 90% to 110% recovery. The reason for this discrepancy is unknown, but is likely related to the instability of mercury in the standard solutions.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solution (2% v/v HNO_3 and 100 ppb Au) was analyzed immediately after the ICV solutions and after the CCV solutions (after each group of not more than eleven samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-1606. Percent differences (%D) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. No %Ds are listed for the AOI since all sample results were below EQL. The acceptance criterion of is ≤10%.

Post-Digestion Spike (PS-Hg)/Analytical Spike (AS-Hg) - Sample (Hg Component):

In addition to a MS sample, post-digestion spikes (PS-Hg) were conducted on the sample 18-1604. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. The recovery value for the AOI meeting this requirement was 196%, and was not within the acceptance criterion of 75% to 125%. The reason for this discrepancy is unknown, but is likely related to the instability of low levels of mercury in solution.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB solution. The LLS solution was also analyzed prior to the final CCV standard. The concentrations of all AOI were within the acceptance criteria of 75% to 125% recovery.

Interference Check Standard (ICS):

The ICS solution was analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike 71A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, K, La, Lu, Mg, Mn, Na, Nd, Ni, P, Pb, Pr, Rb, S, Se, Sm, Sr, Th, Tl, Tm, U, V, Yb, and Zn. Analytes included in the spike 71B component are; Ge, Hf, Mo, Nb, Sb, Si, Sn, Ta, Te, Ti, W, and Zr. Analytes included in the spike 71C component are; Ir, Os, Pd, Pt, Re, Rh, and Ru. Analytes included in the spike 71D component are; Bi, In, Li, Sc, Tb, and Y. Analytes included in the spike Hg component are; Hg.

Battelle PNNL/RPG/Inorganic Analysis ... ICP-MS Data Report

		Run Date >	7/20/2018	7/20/2018	7/20/2018	7/20/2018	7/20/2018	7/20/2018
		Process Factor >	1.00	5,018.161	9,725,402	9,878,120	10,136,322	9,883,187
		RPL/LAB >	2% HNO3 Lab Blank	BLK-1604 @100x	18-1604 @200,000x	18-1605 @200,000x	18-1606 @200,000x	DUP-1606 @200,000x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	2% HNO3 Lab Blank	Process Blank	TI042-COMP-FEED	TI042-COMP-EFF	TI042-COMP-EFF-DUP	TI042-COMP-EFF-DUP
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0247	0.247	Hg-202	--	--	--	--	--	--

Internal Standard % Recovery

Bi 209 (IS)	100%	98%	100%	100%	100%	102%

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

QC Performance 7/18/2018

Criteria >	≤ 20%	80%-120%	80%-120%	75%-125%	75%-125%	≤ 10%
QC ID >	18-1606 Duplicate	BS	RS	MS (18-1606)	18-1604 + PS Hg	18-1606 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Hg-202	--	110%	--	--	196%	--

Internal Standard % Recovery

Bi 209 (IS)	102%	99%	101%	100%	101%	100%

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na2O2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

NM = Not measured. The isotope was not measure due to method or molecular interference limitations.

Battelle PNNL/RPL/Inorganic Analysis ... ICP-MS Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	71274 / N96056	**ASR#:**	0521.01
Client Name:	S. Fiskum	**Total Samples:**	3 (Liquids)

Client Sample Description: Hanford Tank Waste Matrix

ASO Sample ID	Client Sample ID	Sample Weight (g)	ASO Sample ID	Client Sample ID	Sample Weight (g)
18-1604	TI042-COMP-FEED				
18-1605	TI042-COMP-EFF				
18-1606	TI042-COMP-EFF-DUP				

Sample Preparation: RPG-CMC-128, Rev. 1. “HNO3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater”, performed by L. Darnell between 05/11/18 and 05/14/18. Prior to ICP-MS analysis all samples were further diluted in 2% v/v HNO_3 by D. Cherkasov on 07/17/18.

Procedure: RPG-CMC-292, Rev. 1, “Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS).”

Analyst:	D. Cherkasov	**Analysis Date:**	07/18/2018	**ICP File:**	M0068-B

See Chemical Measurement Center 98620 file: ICP-MS-325-405-1 (Calibration and Maintenance Records)

M&TE:				
	☒	PerkinElmer NexION^{TM} 350X ICP-MS	SN: 85VN4070702	RPL 405
	☒	Sartorius R200D Balance	SN: 39080042	RPL 405
	☒	Ohaus Pioneer PA224C Balance	SN: B725287790	RPL 405
	☒	Mettler AT400 Balance	SN: M19445	RPL 405 FH
	☐	Mettler AT400 Balance	SN: 1113162654	RPL 420 FH
	☒	Mettler AT400 Balance	SN: 1113292667	RPL 420 FH
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH

Garrett N. Brown
Report Preparer

7/30/18
Date

Review and Concurrence

7/30/18
Date

Three liquid samples submitted under Analytical Service Request (ASR) 0521.01 were analyzed by ICP-MS. Samples 18-1604, 18-1605, 18-1606, and DUP-1606 (0.5 mL) were prepared following RPL procedure RPG-CMC-128 and were diluted to approximately 25 mL. All samples were further diluted in 2% HNO_3 prior to analysis. None of the samples were filtered.

All results are reported on a mass per unit mass basis (ng/mL) for each detected analyte. The data have been adjusted for instrument dilutions.

Analytes of interest (AOI) were specified in the ASR and are listed in the upper section of the attached ICP-MS Data Report (Sn-126 and U-238). Because the quadrupole ICP-MS cannot distinguish isobaric interferences (e.g., natural Te-126 and fission Sn-126), an additional mass (Te-125) was evaluated to confirm the absence of natural tellurium. The quality control (QC) results for the AOI have been evaluated and are presented below.

Calibration of the ICP-MS was done following the manufacturer's recommended calibration procedure using multi-analyte (natural abundance) custom standard solutions traceable to the National Institute of Standards and Technology (NIST). Midrange calibration verification standards were used to verify acceptance of the six-point calibration curves and for initial and continuing calibration verification (ICV/CCV). The data have been corrected from the natural abundance calibration solutions to report total isobaric results (ng/mL) at each m/z.

The controlling document was procedure RPG-CMC-292, Rev 1, *Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS).* Instrument calibrations, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spike, duplicate, matrix spike, blank spike, reagent spike, and serial dilution were conducted during the analysis run.

Internal Standard (IS):

All solutions (blanks, standards, QC checks, and samples) were mixed in-line with a solution containing 10 ppb each of Li-6, Sc-45, Y-89, In-115, Tb-159, and Bi-209 as the internal standard (IS). The Sn-126 data were normalized to the In-115 IS and the U-238 data were normalized to the Bi-209 IS. The In-115 IS recoveries ranged from 74% to 107% for the entire analysis sequence, with the exception of one unreported sample (201%) where solution was consumed prior to complete analysis. The Bi-209 IS recoveries ranged from 72% to 103% for the entire sequence, with the exception of the previously mentioned consumed sample and the matrix and reagent spike samples, which contained excess bismuth from the spike solution. All other recoveries were within the acceptance criterion of 30% to 120%.

Preparation Blank (PB):

A preparation blank sample BLK-1604 (reagents only) was prepared for the extraction process. The concentration of all AOI in the PB were within the acceptance criteria of <EQL (estimated quantitation level), ≤50% regulatory decision level, or ≤10% of the concentration in the samples. The PB contained 0.791 ng/mL U-238. Trace levels of various environmental contaminants are to be expected when analyzing highly diluted samples that have been prepared in a radiological laboratory. In addition to the PB, a

diluent blank from the ICP-MS laboratory (2% HNO_3) was analyzed, and the results were <EQL for all AOI.

Blank Spike (BS)/Laboratory Control Sample (LCS):

Three blank spike (BS) samples (71A, 71B) were prepared by separately spiking the 2% HNO_3 blank with an equivalent volume of each 2 ppb standard (1:1 ratio). The recovery for the AOI ranged from 89% to 101%, which is within the acceptance criteria of 80% to 120% recovery.

Duplicate/Replicate Relative Percent Difference (RPD)/Relative Standard Deviation (RSD):

Duplicates of sample 18-1606 were prepared and analyzed. RPD are listed for all analytes that were measured at or above the EQL. RPD for the AOI meeting this requirement ranged from 0.5% to 0.9% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike (MS) sample (18-1606 and spikes) was prepared for the extraction process. Recovery values are listed for all analytes included in the MS that were measured at or above the EQL. The recovery value for the AOI meeting this requirement was 96% and was within the acceptance criterion of 75% to 125%.

Reagent-Spike (RS) Sample:

A reagent spike (RS) sample (reagents and spikes) was prepared for the extraction process. Recovery values are listed for all analytes included in the RS that were measured at or above the EQL. The recovery value for the AOI meeting this requirement was 95% and was within the acceptance criterion of 80% to 120%.

Initial/Continuing Calibration Verification (ICV/CCV):

Three ICV/CCV solutions (71A, 71B) were analyzed immediately after calibration, after each group of not more than ten samples, and at the end of the analytical run. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery.

Initial/Continuing Calibration Blank (ICB/CCB):

Two different ICB/CCB solutions (2% v/v HNO_3, 2% v/v HNO_3 + tr. HF) were analyzed immediately after each respective ICV solution and after each respective CCV solution (after each group of not more than twelve samples and at the end of the analytical run). The concentration of all AOI were within the acceptance criteria of <EQL, with the exception of U-238 which exhibited significant carryover (ca. 5x EQL) from the prior CCV solution. This behavior is often observed during ultra-trace (sub-ppb) analysis and additional blanks or complete system cleaning may be necessary to return certain mass signals to the initial background levels.

Low-Level Standard (LLS):

The LLS solutions (71A, 71B) were analyzed immediately after the first CCB solution. The concentrations of all AOI were within the acceptance criteria of 75% to 125% recovery.

Interference Check Standard (ICS):

The ICS solutions (71A, 71B) were analyzed immediately after the first LLS solution and immediately prior to analyzing the final CCV solutions. The concentrations of all AOI were within the acceptance criteria of 80% to 120% recovery.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 18-1606. Percent differences (%D) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement was 3.4% and was within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS)/Analytical Spike (AS) - Sample (71A, 71B Components):

In addition to a MS sample, post-digestion spikes (PS-71A, PS-71B) were conducted on sample 18-1604. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement were 75% and 100%, which was within the acceptance criterion of 75% to 125%.

Other QC:

All other instrument-related QC tests for the AOI passed within their respective acceptance criteria.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) and estimated quantitation limits (EQL) shown are for acidified water and/or fusion flux matrices as applicable. Method detection limits (MDL) for individual samples can be estimated by multiplying the IDL by the "Process Factor" for that individual sample. The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Process Factor".
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations > EQL up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 50 µg/mL (0.005 per cent by weight). Note that bracketed values listed in the data report are within the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -". Note, that calibration and QC standard samples are validated to a precision of ±10%.
4) Analytes included in the spike 71A component (for the AS/PS) are; Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, K, La, Lu, Mg, Mn, Na, Nd, Ni, P, Pb, Pr, Rb, S, Se, Sm, Sr, Th, Tl, Tm, U, V, Yb, and Zn. Analytes included in the spike 71B component are; Ge, Hf, Mo, Nb, Sb, Si, Sn, Ta, Te, Ti, W, and Zr. Analytes included in the spike 71C component are; Ir, Os, Pd, Pt, Re, Rh, and Ru. Analytes included in the spike 71D component are; Bi, In, Li, Sc, Tb, and Y. Analytes included in the spike Hg component are; Hg.

Battelle PNNL/RPG/Inorganic Analysis ... ICP-MS Data Report

		Run Date >	7/18/2018	7/18/2018	7/18/2018	7/18/2018	7/18/2018	7/18/2018
		Process Factor >	1.00	1240.80	2954.74	2969.92	3034.61	2985.23
		RPL/LAB >	2% HNO3 Lab Blank	BLK-1604 @25x	18-1604 @60x	18-1605 @60x	18-1606 @60x	DUP-1606 @60x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	2% HNO3 Lab Blank	Process Blank	TI042-COMP-FEED	TI042-COMP-EFF	TI042-COMP-EFF-DUP	TI042-COMP-EFF-DUP
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0019	0.019	Sn-126	--	--	1.76E+02	1.73E+02	1.74E+02	1.75E+02
0.00006	0.00057	U-238	--	7.91E-01	2.07E+04	1.08E+04	1.08E+04	1.09E+04

Internal Standard % Recovery

In 115 (IS)	91%	103%	97%	97%	96%	96%
Bi 209 (IS)	90%	101%	86%	83%	83%	84%

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.

2) Values in brackets [ ] are ≥ MDL but < EQL, with errors likely to exceed 15%.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

QC Performance7/18/2018

Criteria >	≤ 20%	80%-120%	80%-120%	75%-125%	75%-125%	75%-125%	≤ 10%
QC ID >	18-1606 @60x DUP	BS	RS	MS (18-1606)	18-1604 + PS71A	18-1604 + PS71B	18-1606 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Sn-126	0.5%	101%		--	na	100%	--
U-238	0.9%	89%	95%	96%	75%	na	3.4%

Internal Standard % Recovery

In 115 (IS)	96%	102%	99%	99%	102%	103%	96%
Bi 209 (IS)	84%	96%	98%	98%	94%	97%	83%

Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration. Matrix effects can be assessed from the serial dilution.

na = not applicable; KOH flux and Ni crucible or Na2O2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

NM = Not measured. The isotope was not measure due to method or molecular interference limitations.

Appendix C

Analyte Concentrations with Loading

The AP-107 load behaviors of selected load samples from the lead column are provided in Table C.1 and from batch contact solution equilibrium concentrations in Table C.2.

Table C.1. Analyte Concentrations of Selected Samples from the Lead Column

	Feed	46.9 BV	57.0 BV	100.9 BV	123.8 BV	178.1 BV	231.5 BV	284.2 BV	334.6 BV	389.4 BV	471.2 BV
Sample ID>>	TI042-Comp-Feed	TI042-L-F2-A	TI042-L-F3-A	TI042-L-F4-A	TI042-L-F5-A	TI042-L-F7-A	TI042-L-F9-A	TI042-L-F11-A	TI042-L-F13-A	TI042-L-F15-A	TI042-L-F18-A
Analyte						**µg/mL**					
Al	9,850	9,570	9,250	9,390	9,590	9,700	9,770	9,750	9,660	9,550	9,600
Ba	0.836	[0.17]	[0.17]	[0.09]	[0.09]	[0.09]	[0.11]	[0.06]	[0.05]	[0.09]	[0.06]
Ca	34.1	[12]	17.4	30.9	17.3	21.0	19.9	24.8	27.6	21.2	25.7
Cd	6.62	4.91	5.29	5.28	5.05	5.73	5.38	5.22	5.59	5.56	5.27
Cr	498	487	472	474	483	488	491	486	484	482	483
Fe	19.4	12.8	12.8	12.70	14.2	12.9	13.5	15.5	13.2	17.9	14.5
Ni	21.0	20.6	20.0	19.6	20.5	20.1	20.4	20.8	20.1	20.1	19.5
Sr	0.33	[0.05]	[0.06]	[0.05]	[0.03]	[0.04]	[0.04]	[0.03]	[0.04]	[0.04]	[0.04]
Ti	[0.80]	[0.69]	[0.61]	[0.51]	[0.56]	[0.52]	[0.55]	[0.41]	[0.47]	[0.42]	[0.36]
U	[48]	[12]	--	[13]	[11]	[24]	[18]	[15]	[20]	[24]	[18]
Zn	[1.8]	--	[1.4]	[0.56]	[1.20]	--	[0.57]	[0.81]	[0.99]	[1.2]	[1.2]
Zr	[1.4]	[1.2]	[1.3]	[1.3]	[0.99]	[1.2]	[1.4]	[1.1]	[1.3]	[1.2]	[0.94]
Analyte						**µCi/mL**					
^{137}Cs	1.49E+2	2.64E-3	1.36E-2	8.49E-2	2.25E-1	1.31E+0	4.23E+0	8.81E+0	1.50E+1	2.40E+1	3.92E+1
^{238}Pu	1.14E-4	3.19E-5	2.86E-5	3.53E-5	4.18E-5	4.99E-5	4.07E-5	4.06E-5	6.15E-5	7.53E-5	6.88E-5
$^{239+240}$Pu	5.43E-4	2.14E-4	2.16E-4	2.34E-0	2.72E-4	2.66E-4	2.86E-4	2.84E-4	3.07E-4	3.17E-4	3.20E-4
^{90}Sr	5.81E-1	9.73E-4	8.44E-4	8.57E04	7.99E-4	[7.3E-4]	[2.0E-3]	[2.4E-3]	[2.9E-3]	3.59E-3	7.61E-3

BV = bed volume; Bracketed values indicate the associated sample results were less than the estimated quantitation limit but greater than the MDL. Analytical uncertainty for these analytes are > ±15%.

"--" = analyte was <MDL.

Additional analyte concentrations may be found in Appendix B.

Table C.2. Batch Contact Solutions Equilibrium Concentrations

Sample ID >> (Analyte)	TI042-Comp-Feed (µg/mL)	TI036-S0-CST-F (µg/mL)	TI036-S2-CST-F (µg/mL)	TI036-S3-CST-F (µg/mL)
Cs	8.57	2.11	123	1280
Ag	--	--	--	--
Al	9,850	10,100	9,560	9,440
As	[11.0]	--	--	--
B	34.6	41.6	43.3	45.5
Ba	0.836	[0.050]	[0.062]	[0.120]
Ca	34.1	15.6	17.6	19.6
Cd	6.62	5.20	5.34	5.04
Cr	498	504	478	471
Fe	19.4	12.70	12.50	19.5
K	3,910	4,010	3,860	3,820
Li	--	--	--	--
Na	126,000	127,000	119,000	119,000
Ni	21.0	19.7	20.5	19.8
P	642	622	611	602
Pb	[9.0]	--	--	--
Se	--	--	--	--
Sr	0.331	[0.050]	[0.037]	[0.060]
Th	[12]	--	--	--
Ti	[0.80]	[0.72]	[0.52]	[0.57]
U	[48]	[17]	[13]	[19]
Zn	[1.8]	--	--	--
Zr	[1.4]	[1.1]	[0.98]	[1.4]

"--" = analyte was <MDL.

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