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Multi-Cycle Cesium Ion Exchange Testing Using Spherical Resorcinol-Formaldehyde Resin with Diluted Hanford Tank Waste 241-AP-105

April 2018

SK Fiskum	AM Rovira
JR Allred	MR Smoot
HA Colburn	RA Peterson

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Executive Summary

Washington River Protection Solutions requested that at least 4 L of Hanford tank waste collected from tank 241-AP-105 and diluted to approximately 5.6 M Na (AP-105DF) be prepared for use in vitrification studies. Cesium removal was required to meet this objective and the waste pretreatment platform, established at Pacific Northwest National Laboratory, was used to create the vitrification feed. The Test Platform mimicked the Low-Activity Waste Pretreatment System (LAWPS) facility planned to pretreat Hanford tank waste supernate by removing solids in a cross flow filter apparatus and processing the supernate through ion exchange columns to remove cesium. At the time of testing, the ion exchange media was targeted to be spherical resorcinol-formaldehyde (SRF) resin.[1] The SRF resin has been tested with a wide array of simulants and process scales, but column performance testing with actual tank waste had been somewhat limited to two Hanford tank wastes (AP-101 and AN-102) in up to two process cycles. This report describes testing conducted in a total of six load elute cycles with the AP-105DF tank waste. The column system was first tested with simulant and was previously described[2,3]; this report describes the six process cycle results with the AP-105DF tank waste.

Column testing was conducted on SRF resin provided by Microbeads AS (Skedsmokorset, Norway, batch number 1F-370/1392), which was manufactured in August 2011. The column testing was prototypic to the intended LAWPS operations in a lead-lag column format, albeit on a small-scale basis with 10-mL resin beds. In this configuration, neither the length-to-diameter ratio nor the superficial flow velocity matched the full-scale design. In this process, the feed was directed downflow through the lead column and then through the lag column. Loading continued until the lag column reached 10% of the Hanford Tank Waste Treatment and Immobilization Plant contract limit for receiving supernatant waste for vitrification (a function of the Na and ^{137}Cs concentrations). As a result of this process condition, the lead column was nearly saturated with Cs. After loading, the feed was displaced with 0.1 M NaOH, and then the columns were rinsed with water in a downflow lead-to-lag configuration. Elution was conducted downflow from the lag to the lead column with 0.45 M HNO_3 followed by a water rinse. The resin was returned to the Na-form by processing 1 M NaOH downflow from the lag to the lead column, which deviated from the intended LAWPS process operation where the regeneration would occur in upflow to fluidize the resin beds. Variations to the feed flowrates, elution volumes, and elution flowrates were implemented to evaluate effects on the Cs load behavior and Cs leakage to the next process cycle.

Cs load and elution profiles were generated. From the load profile, the number of bed volumes (BVs) processed to reach 50% breakthrough on the lead column was determined along with the number of BVs processed before reaching 10% of the contract limit on the lag column. For AP-105DF, the 10% contract limit is set to ~0.015% of the influent ^{137}Cs concentration; this requires a Cs decontamination factor of 6620. Table ES.1 summarizes the observed column performance for the six process cycles.

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[1] The LAWPS system approach had been modified subsequent to the testing described in this report; test parameters were current at the testing time.

[2] Fiskum SK, HA Colburn, MR Smoot, JR Allred, RA Peterson. 2017. *Cesium Ion Exchange Using Spherical Resorcinol-Formaldehyde Resin in Support of Waste Qualification Testing for LAWPS*. PNNL-26837, RPT-DFTP-003, Rev. 0.0. Pacific Northwest National Laboratory, Richland, Washington.

[3] The first cycle AP-105 actual waste testing was also described by Fiskum et al. (2017).

Table ES.1. Column Performance Summary

Test Number	Flowrate, BV/h	Elution Volume, BVs	50% Cs Breakthrough, BVs	Mass Transfer Zone, BVs[(a)]	Cs Load Capacity, mg Cs/g H-form Resin	Contract Limit Breakthrough, BVs
1	1.80	15.9	206	95	6.64	275
2	3.05	29.9	205	124	6.38	245
3	4.53	23.0	189	149	6.09	189
4 (Take 1)	3.04	24.9	NA	NA	NA	NA
4 (Take 2)	2.95	29.5	182	112	6.06	193
5	3.17	29.1	181	128	5.53	202

(a) The mass transfer zone was the range between 1% and 90% C/C_0 breakthrough.
NA = not applicable; Test 4 Take 1 processing was short, with only 52.9 BVs.
One BV equals 9.9 mL.

Cs leakage from the lag column occurred from one process run to the next. Increased elution beyond 16 BVs was apparently required to keep the product effluent below the 10% contract limit. The leakage appeared to reduce as more feed was processed until the expected Cs breakthrough profile was established.

The repeated cycling resulted in significant resin swelling. The initial Na-form resin beds were 9.9 mL; after the final process cycle, the Na-form resin BVs were 10.9 mL (lead column) and 12.1 mL (lag column), expansions of 10% and 20%, respectively. Volume expansions of resin in the H-form were 106% and 112%, respectively.

The effluent and eluate were characterized to assess distribution of cations, anions, and radionuclides during the ion exchange process. In addition to Cs, small fractions of Fe, Ca, Cu, Pb, and Zn were measured in the eluate; all other cations and anions were quantitatively recovered in the Cs-decontaminated effluent. Within 10% analytical uncertainty, all ^{90}Sr, Pu, and Am reported to the Cs-decontaminated effluent. However, 66% of the Cm and 78% of the ^{99}Tc were found in the effluent; less than 4% was found in the eluate, indicating the difference may be held by the resin.

Acknowledgments

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Acronyms and Abbreviations

AP-105DF	Hanford tank waste from 241-AP-105, diluted to 5.7 M sodium
ASO	Analytical Support Operations
ASR	Analytical Service Request
AV	apparatus volume
BV	bed volume
DF	decontamination factor
DI	deionized (water)
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) or internal diameter, see context
KPA	kinetic phosphorescence analysis
LAW	low-activity waste
LAWPS	Low-Activity Waste Pretreatment System
MDL	method detection limit
NA	not applicable
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
R&D	research and development
SRF	spherical resorcinol-formaldehyde
TIC	total inorganic carbon
TOC	total organic carbon
WRPS	Washington River Protection Solutions
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WWFTP	WRPS Waste Form Testing Program

Contents

Figures

Tables

1.0 Introduction

Decanted tank waste supernatant will be pretreated in the Low-Activity Waste Pretreatment System (LAWPS) to meet the Hanford Tank Waste Treatment and Immobilization Plant (WTP) Low-Activity Waste (LAW) facility waste acceptance criteria in preparation for vitrification.[1] Washington River Protection Solutions (WRPS) is designing the LAWPS facility. The key process operations for treating the tank waste supernatants include solids filtration and cesium removal. At the time of this work (July 2017), the LAWPS design specified solids removal by cross flow filtration and cesium removal with spherical resorcinol-formaldehyde (SRF) ion exchange resin. Testing of actual tank waste processing on SRF resin has been conducted (Fiskum et al. 2006b, c; Duignan and Nash 2009) where up to two process cycles were employed. Duignan and Nash (2009) processed Savannah River National Laboratory tank waste up to 100% breakthrough in the lead column, whereas Fiskum et al. (2006a, b) processed the lead column to 50% breakthrough in the mode in which WTP plans to process tank waste.

Supernate waste delivered to the WTP LAW facility is required to be <3.18E-5 Ci ^{137}Cs/mole of Na (contract limit). In the LAWPS design at the time of this work, the SRF resin was to be loaded in two columns, ~300-gallon resin beds each, arranged in a lead-lag format.[2] Feed was to be processed until the lag column effluent reached 10% of the contract limit (i.e., 3.18E-6 Ci ^{137}Cs/mole of Na). The 10% contract limit was implemented by WRPS to manage lag column Cs loading and concomitant Cs leakage to the next process run. The Cs-decontaminated effluent was then to be forwarded to the WTP for vitrification.

The key functional parameters of the LAWPS and WTP LAW facilities were adapted for small-scale testing at Pacific Northwest National Laboratory (PNNL) and the designated Test Platform. The cesium ion exchange component of the Test Platform was constructed using small-scale ion exchange columns (each column contained 9.9 mL Na-form SRF resin in a 1.43-cm-diameter column). Other attributes of the ion exchange system were scaled to be generally prototypic of the LAWPS system at the time of this work. The ion exchange system was previously described by Fiskum et al. (2017).

Approximately 8 L of Hanford tank waste were collected from tank 241-AP-105 (hereafter called AP-105) to process through the Test Platform. WRPS requested that the tank waste be diluted to a target of 5.6 M Na and then filtered, and that cesium be removed to support vitrification testing. To complete this effort, 10 L of diluted AP-105 (AP-105DF) were processed through the ion exchange system, requiring a total of six load-elute cycles.

This report discusses results of multi-cycle testing using SRF resin with AP-105DF. Cesium capacity as a function of process cycle; cesium leakage into subsequent runs; and radionuclide, cation, and anion mass balances (with special emphasis on Cs, Pu, Na, K, Tc, transition metals) were determined.

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[1] 24590-WTP-ICD-MG-01-030, Rev. 0. 2015. *ICD 30 – Interface Control Document for Direct LAW Feed*. Bechtel National, Inc., Richland, Washington.

[2] After processing described herein, WRPS staff are considering alternative processing and ion exchange media.

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 69832. All research and development (R&D) work at PNNL is performed in accordance with PNNL's Laboratory-Level Quality Management Program to R&D activities, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications* (ASME 2000). 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 SRF resin, diluted AP-105 tank waste, 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 SRF Resin

The SRF ion exchange resin (Spheromers® RF 380) was provided by Microbeads AS, Skedsmokorset, Norway, batch number 1F-370/1392, and was manufactured in August 2011. A full description of the resin retrieval and pretreatment was previously provided by Fiskum et al. (2017).

The SRF resin was pretreated according to established protocol.[2] The SRF resin loaded in the column assembly was exposed to a process cycle before use with the AP-105DF as previously described (Fiskum et al. 2017). This process cycle used a simple simulant (Russell et al. 2017) spiked with 60 µg/mL Cs and tracer 0.1 µCi/mL ^{137}Cs.

2.2 AP-105 Tank Waste

As previously reported (Fiskum et al. 2017), multiple samples (32 each at nominally 250 mL) were collected at four different depths (91 in., 172 in., 253 in., and 334 in.) from the AP-105 Hanford tank. The first sample collected, 5AP-16-01, was subsampled for a limited analysis suite. The density was 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) 0272; results are provided in Table 2.1.

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[1] TP-DFTP-001, Rev.0.2. *DFLAW Test Platform Cesium Ion Exchange Testing with AP-105 Tank Waste with Spherical Resorcinol-Formaldehyde Resin*. Pacific Northwest National Laboratory, Richland Washington. 2017.

[2] WTP doc. no. 097893. CA Nash and CE Duffey. August 17, 2004. *Hanford RPP-WTP Alternate Resin Program - Protocol P1-RF: Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column*. SRNL-RPP-2004-00058, Savannah River Technology Center, Aiken, South Carolina.

Table 2.1. Characterization of Sample 5AP-16-01 Collected from Hanford Tank AP-105 (ASR 0272)

Analyte	Result	Result Units	Analysis Method
Al	1.02	M	ICP-OES
K	0.141	M	ICP-OES
Na	8.53	M	ICP-OES
OH^-	1.75(a)	M	Titration
^{133}Cs	5.7E-5	M	ICP-MS
^{137}Cs	180 ± 2%(b)	µCi/mL	GEA
^{137}Cs	2.07(b)	µg/mL	GEA
Density	1.405(c)	g/mL	Volumetric flask

(a) Based on first inflection point; assumed to be the free (unbound) hydroxide.
(b) Reference date is 4/26/17.
(c) Measured at 27.5 °C.
ASR 0272, sample 17-0868
GEA = gamma energy analysis; ICP-MS = inductively coupled plasma mass spectrometry; ICP-OES = inductively coupled plasma optical emission spectrometry

Samples of the AP-105 tank waste were combined and then diluted to ~5.7 M Na with 0.01 M NaOH before filtration through the cross flow filter system (Geeting et al. 2017). The sample/diluent volume in the filtration system was limited to 4 L; therefore, multiple compositing/diluting batches were prepared. The diluted and filtered AP-105 tank waste is termed AP-105DF throughout this report. The AP-105DF was 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 cross flow filter apparatus. Densities ranged from 1.25 to 1.30 g/mL. Due to the large volume (12 L) of AP-105DF, the multiple samples were not combined into one single container for homogenization and dilution. 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.

Because the feed solutions were to be kept in separate containers, it was desired to make the density (and by inference all other chemical/physical properties) the same. To that end, samples with high density were diluted or combined with samples with low density, thus normalizing all feed to around 1.27 g/mL.

The Cs isotopic composition of the AP-105DF was determined on a peak elution sample aliquot from the first ion exchange process run. It was assumed that all samples had the same cesium isotopic distribution; Cs isotopic distribution is shown in Table 2.2. The Cs isotopic ratio was measured by ICP-MS per ASR 0329. The advantage of using the elution sample for the isotopic ratio determination is that it is relatively free from the AP-105DF high salt matrix and contains the highest Cs concentration, and thus is less likely to be affected by isobaric interferences. The total Cs concentration was calculated from the measured ^{133}Cs and ^{137}Cs and the isotopic composition.

Table 2.2. AP-105DF Cs Isotopic Composition (ASR 0329)

Analyte[(a)]	Analysis Method	Results	Units
Cs isotopic mass ratio[(a,b,c)]	ICP-MS	64.0	wt% ^{133}Cs
		19.1	wt% ^{135}Cs
		16.8	wt% ^{137}Cs
Total Cs	ICP-MS	7.92	µg/mL Cs

(a) The peak Cs column eluate sample (TI014-E8-A) from the first AP-105DF ion exchange process cycle was analyzed for the Cs isotopic mass distribution by ICP-MS per ASR 0329 sample 17-1224. The quantity of hold-over Cs from the shakedown testing was assumed to result in negligible ^{133}Cs contribution compared to the Cs eluted from AP-105DF processing.

(b) Reference date is August 9, 2017.

(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.

2.3 Ion Exchange Process Testing

The ion exchange process system has been previously described (Fiskum et al. 2017); a system schematic for processing downflow lead column to lag column is reproduced in Figure 2.1. The quick disconnect valves were realigned to alter the fluid flow path downflow from lag column to lead column. Lead column samples were collected at valve 2 and lag column samples were collected from valve 3 during the AP-105DF loading process as well as the feed displacement (FD), water rinse, elution, and elution rinse (Tests 1-3). For Tests 4-5, the FD, water rinse, and elution were collected at the effluent line, bypassing the valve 3 sample position; the elution rinse was collected from the valve 3 sample position. Tests 4-5 collected regeneration solution at the valve 3 sample position in discrete ~15-mL volumes. The valve 3 sample position was thus well rinsed before processing AP-105DF in a subsequent cycle, whereas the valve 2 sample position was not well rinsed.

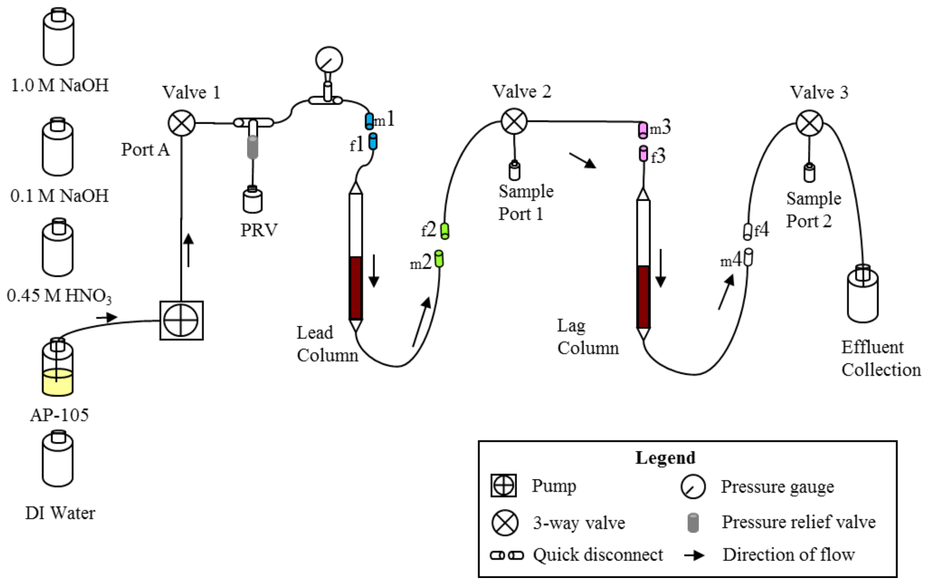

Figure 2.1. Cesium Ion Exchange Process Schematic Showing Downflow Lead-to-Lag Processing

Fiskum et al. (2017) also described the out-of-column and in-column SRF resin pretreatment steps. As a reminder, the bed volume (BV) corresponds to the initial settled Na-form resin BV as measured in a graduated cylinder prior to transferring the resin into the ion exchange column. The reference resin BVs are 9.9 mL for both the lead and lag columns.

The initial process cycle with 5.6 M Simple Simulant traced with ^{137}Cs was conducted in a radiologically controlled fume hood as previously described (Fiskum et al. 2017). The system was transferred to the hot cell for AP-105DF processing. A photograph of the in-cell system is shown in Figure 2.2.

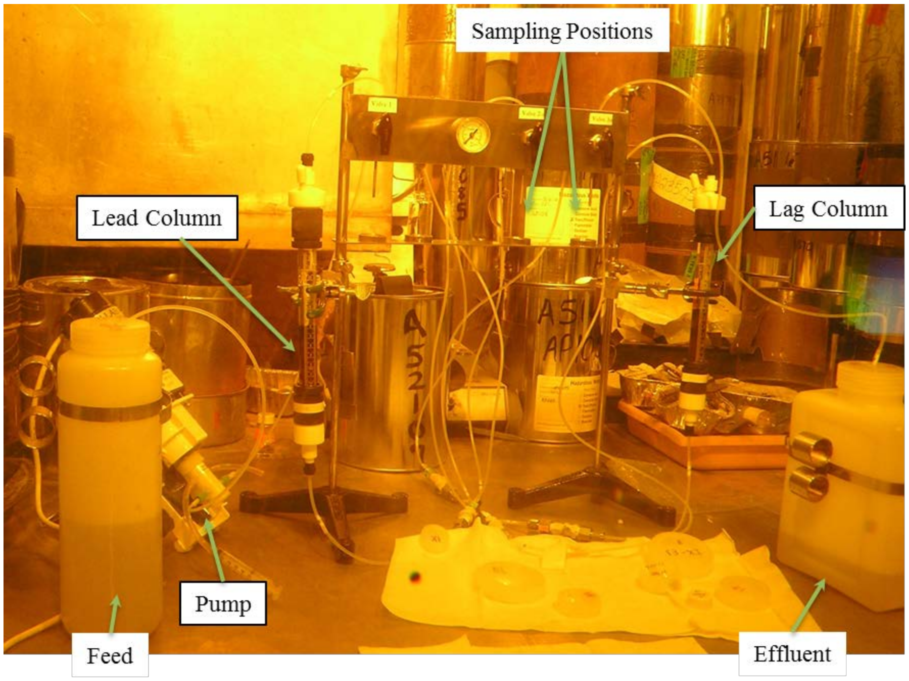

Figure 2.2. Column Assembly in the Hot Cell

The AP-105DF was processed through the ion exchange resin beds, lead to lag according to PNNL test instructions. A series of AP-105DF 1.5-L feed bottles were strategically processed to allow optimal feed volume management and support unattended, off-shift (graveyard) work. Effluent was collected in two to three different bottles to better manage the consequences of Cs breakthrough from the lag column. After the AP-105DF load step, 0.1 M NaOH FD, followed by water rinse, was passed through the system in the same lead-to-lag configuration. The flow orientation was switched by rearranging the quick disconnect connections as previously described (Fiskum et al. 2017), allowing elution to occur downflow from the lag column to the lead column. The columns were then rinsed with deionized (DI) water. The resin was converted to the Na-form by passing 1.0 M NaOH downflow from the lag column to the lead column. Once the lead column resin bed was fully converted based on visual examination of the bottom resin bed color, the flow direction was again changed back to the lead-to-lag direction.

All processing was conducted at ambient cell temperature conditions, nominally 26 to 30 °C. Test parameters, including process volumes, flowrates, and contact times, are summarized in Table 2.3 to Table 2.8. The AP-105DF flowrate and 0.45 M HNO_3 eluent volume and flowrate were adjusted from one test to the next to test process condition effects. The first AP-105DF process cycle mimics as best as possible the process flows anticipated at the LAWPS facility in terms of BV/h and total BVs. It is understood that the feed linear velocity cannot be matched in this small column configuration. As previously reported (Fiskum et al. 2017), increasing the linear velocity decreases the transition zone and sharpens the breakthrough curve. Therefore, the load curves developed from the small-scale system are likely worst-case bounding with respect to the transition zone.

Table 2.3. Test 1 Experimental Conditions for AP-105DF Column Processing, July 10-21, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	9.6	2.4	95	4.77	0.789	2.0
Loading (lead)	AP-105DF	294	NA	2921	1.80	0.298	239(b)
Loading (lag)(a)	AP-105DF	274	NA	2724	1.80	0.298	239(b)
Feed displacement	0.1 M NaOH	6.65	1.7	66.1	3.05	0.505	2.2
Water rinse	DI water	4.17	1.04	41.4	3.08	0.511	1.4
Elution	0.45 M HNO_3	15.9	3.98	158	1.40	0.231	11.7
Water rinse	DI water	5.79	1.44	57.5	1.47	0.243	4.0
Extended water rinse	DI water	8.74	2.18	86.8	3.14	0.520	2.8

(a) The feed volume through the lag column is reduced because of sampling from the lead column.
(b) Time includes the standby time over the weekend; see Section 2.3.1.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-014.

Table 2.4. Test 2 Experimental Conditions for AP-105DF Column Processing, July 31-August 5, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	7.4	1.85	73.9	2.94	0.486	2.5
Loading (lead)	AP-105DF	254	NA	2523	3.05	0.506	84
Loading (lag)(a)	AP-105DF	251	NA	2489	3.05	0.506	84
Feed displacement	0.1 M NaOH	6.4	0.79	63.6	3.00	0.497	2.2
Water rinse	DI water	4.7	1.18	47.1	2.94	0.486	1.7
Elution	0.45 M HNO_3	29.9	NA	297.0	1.98	0.327	15.6
Water rinse	DI water	5.6	NA	55.7	1.99	0.330	2.8
Extended water rinse	DI water	8.7	NA	86.4	3.07	0.508	2.8

(a) The feed volume through the lag column is reduced because of sampling from the lead column.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-015.

Table 2.5. Test 3 Experimental Conditions for AP-105DF Column Processing, August 21-25, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	8.0	2.01	79.9	2.84	0.470	2.80
Loading (lead)	AP-105DF	248	NA	2459	4.53	0.746	56
Loading (lag)(a)	AP-105DF	241	NA	2393	4.53	0.746	56
Feed displacement	0.1 M NaOH	6.3	1.57	62.5	3.08	0.510	2.1
Water rinse	DI water	4.2	1.04	41.3	3.02	0.500	1.4
Elution	0.45 M HNO_3	23.0	NA	228.8	1.92	0.318	12.25
Water rinse	DI water	6.3	NA	62.5	2.13	0.352	3.1
Extended water rinse	DI water	6.3	NA	89.5	2.86	0.474	3.1

(a) The feed volume through the lag column is reduced because of sampling from the lead column.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-016.

Table 2.6. Test 4 (Take 1) Experimental Conditions for AP-105DF Column Processing, September 12-15, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	9.4	2.4	93.6	2.76	0.456	3.4225
Loading (lead)	AP-105DF	53	NA	526	3.04	0.503	19.2(b)
Loading (lag)(a)	AP-105DF	52	NA	517	3.04	0.503	19.2(b)
Feed displacement	0.1 M NaOH	4.6	1.1	45.5	1.96	0.325	2.3
Water rinse	DI water	3.7	0.93	36.9	2.82	0.467	1.3
Elution(c)	0.45 M HNO_3	24.9	NA	247.5	2.31	0.382	10.8
Extended water rinse	DI water	12.8	NA	127.0	2.80	0.463	4.5

(a) The feed volume through the lag column is reduced because of sampling from the lead column.
(b) The columns remained partially loaded and in contact with AP-105DF for ~27 hours; see Section 2.3.4.
(c) Erratic flow conditions occurred during elution; see Section 2.3.4.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-020.

Table 2.7. Test 4 (Take 2) Experimental Conditions for AP-105DF Column Processing, September 18-22, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	8.3	2.06	82.1	2.66	0.441	3.1
Loading (lead)[a,b]	AP-105DF	238	NA	2370	2.95/3.6	0.489/0.597	77
Loading (lag)[a]	AP-105DF	235	NA	2332	2.95/3.6	0.489/0.597	77
Feed displacement	0.1 M NaOH	5.7	1.43	56.8	3.27	0.541	1.8
Water rinse	DI water	5.2	1.30	51.6	3.18	0.527	1.6
Elution	0.45 M HNO_3	29.5	NA	292.7	2.74	0.453	10.8
Water rinse	DI water	8.1	NA	80.5	3.04	0.503	2.8
Extended water rinse	DI water	9.8	NA	97.1	3.04	0.503	3.2

(a) An overcorrection in the flowrate was implemented after processing 162 BVs through the lead column (159 BVs through the lag column).
(b) The feed volume through the lag column is reduced because of sampling from the lead column.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-020.

Table 2.8. Test 5 Experimental Conditions for AP-105DF Column Processing, October 9-13, 2017

		Total Volume			Flowrate		Duration
Process Step	Solution	BV	AV	mL	BV/h	mL/min	h
Regeneration	1.0 M NaOH	9.1	2.41	90.8	3.09	0.512	3.0
Loading (lead)	AP-105DF	218	NA	2168	3.17	0.524	75[b]
Loading (lag)[a]	AP-105DF	213	NA	2117	3.17	0.524	75[b]
Feed displacement	0.1 M NaOH	7.8	2.05	77.2	4.40	0.728	1.8
Water rinse	DI water	5.1	1.34	50.4	4.12	0.682	1.2
Elution	0.45 M HNO_3	29.1	NA	289.4	2.56	0.424	11.4
Water rinse	DI water	7.9	NA	78.9	3.10	0.514	2.7
Extended water rinse	DI water	8.6	NA	85.3	3.05	0.505	2.8

(a) The feed volume through the lag column is reduced because of sampling from the lead column.
(b) Time includes the 6-h standby; see Section 2.3.5.
BV = bed volume (9.9 mL in the Na-form as loaded in the column).
AV = apparatus volume (nominally 40 mL).
NA = not applicable.
Processing was conducted according to PNNL test instruction TI-DFTP-021.

During the loading phase, nominal 2-mL samples were collected from both the lead and lag columns at the sample collection ports. The solution in the lag column remained static during the lead column sampling time of about 4 min. Samples were collected after the first ~4 BVs were processed and again at nominal 10- to 20-BV increments. Feed displacement, water rinse, and elution were collected sequentially in nominal 1.1-BV increments (Tests 1-3). Bulk collection of the FD and water rinse and bulk collection of the eluate were performed in Tests 4-5. The water rinse following elution was collected in nominal 1.1-BV increments. Finally, the bulk water rinse was conducted to better clear the system from acidic matrix, implemented to accommodate the resin idle time between tests and reduce exposure to residual acidic fluid. Aliquots of each solution were removed for GEA.

Cesium load and elution performance was determined from the ^{137}Cs in the collected samples relative to the native ^{137}Cs in AP-105DF feed. The collected samples were analyzed directly to determine the ^{137}Cs concentration using GEA (constant reference date of July 10, 2017). Cesium breakthrough and elution curves were generated as previously described (Fiskum et al. 2017).

Some unexpected issues occurred during processing. These are described in the following sections.

2.3.1 Test 1 Process Notes

After processing ~144 BVs of feed, one polyethylene line between the lead and lag columns broke. The break occurred off-shift and was not found until the next morning, after an estimated 147 mL of feed was calculated to have leaked from the system (passing through the lead column but not through the lag column). The broken line was replaced; the air in the replacement line and hardware displaced 3.7 mL of the fluid above the lag column. The replacement line also broke after processing another ~12 BVs of AP-105DF. At this point, the system (partially loaded) was placed in standby mode (all valves closed) over the weekend (Friday, July 14, at 5:00 p.m. until the following Monday, July 17, at 1:30 p.m.). The broken line was replaced with stainless steel tubing before resuming feed processing. The resin was in contact with AP-105DF for 239 hours, including the weekend standby period. Refer to Fiskum et al. 2017.

2.3.2 Test 2 Process Notes

Regeneration with 1 M NaOH was extended beyond the nominal 6 BV. Conversion of the lead column resin bed from H-form (orange) to Na-form (black) was slow (visually observed color change). It was clear that some channeling was occurring in the conversion process.

2.3.3 Test 3 Process Notes

Samples (15-mL) were collected from the lead column to support technetium studies (separate study) at 22, 200, and 247 BVs. The volume of AP-105DF that was processed through the lag column was proportionately lower.

2.3.4 Test 4 Process Notes

The AP-105DF feed ceased flowing after processing 52.9 BVs. The system pump head had failed. The pump was replaced along with the associated process tubing. The lead and lag columns were in static contact with the AP-105DF for 27 h while the pump was replaced. Once the pump was repaired, the AP-105DF feed process was aborted and FD, water rinse, elution, and water rinse were processed through the columns. During elution, some leakages were observed in the tubing connections. Fluid above the lead column resin bed dropped very close to the top of the bed. Corrective measures were implemented (tightened Swagelok connections, added fluid to the top of the resin bed from the column top access port).

A rapid fluid drop (5.7 mL) occurred through the lead column after establishing the fluid bed height. The knurled nut at the access port was retightened to correct this. After corrective measures, small gaps were observed at 1.0 and 1.5 cm from the bottom of the lag column resin bed, indicating that a vacuum pull from the top of the bed or upflow fluid movement through the bottom of the bed occurred. Processing continued, and the gap, as shown in Figure 2.3, was eventually no longer visible.

Figure 2.3. Gaps in Lag Column during Test 4 Take 1 Elution Processing

The test was restarted from the regeneration process step. The initial (short) Test 4 processing was termed “Take 1.” The subsequent processing was termed “Take 2.” After processing 162 BVs through the lead column (159 BVs through the lag column), an attempt was made to increase the AP-105DF flowrate from 0.489 to 0.50 BV/h. The correction was too high and the flowrate jumped to ~0.60 BV/h until the end of the feed processing.

2.3.5 Test 5 Process Notes

The remaining AP-105DF (2.05 L) was composited into a single, 2-L, high-density polyethylene bottle, which simplified the feed loading (no switching from one feed bottle to another feed bottle). After processing 195 BVs, the feed tube apparently was above the feed liquid and the lead column fluid headspace was displaced with air. The resin bed did not go dry, but some air contact with the top few millimeters of resin bed was likely. The feed processing was suspended for 6 h.

Lead column samples from previous process runs containing significant ^{137}Cs activity had been combined to form a 150-mL volume composite in a smaller, low-density polyethylene bottle (where the feed line position was more easily distinguished). This composite solution was used to re-establish the fluid height above the lead column to the more optimum full BV above the resin bed. Because the AP-105DF comprising this solution had been previously processed through the SRF column, it was depleted in Cs (27.6 µCi/mL ^{137}Cs). A 108-mL aliquot of this depleted solution was processed (combined head space adjustment and feed flow) before it was combined with the remaining AP-105DF. This final mixture (150 mL) contained an estimated 99 µCi/mL ^{137}Cs. The fluid processed after 195 BVs were fed through the lead column (190 BVs through the lag column) contained variable Cs concentrations, and examinations of the load curve will need to take this into account.

2.4 Sample Analysis

A summary of the sample collections and analyses from the various tests and process steps is provided in this section along with the cross references to ASR and Radiochemical Processing Laboratory sample identifications (IDs).

2.4.1 Cesium Load and Elution Sample Analysis

Aliquots of the various process samples' (regeneration, feed, effluent, FD, water rinse, elution, and water rinse) ^{137}Cs concentrations were determined by the ASO on calibrated gamma detectors. To support this analysis, all samples and sub-samples were collected and packaged in 10-mL and/or 2-mL volumes to accommodate the calibrated detector geometries. Several samples of feed material were provided in 1-mL counting geometries to better accommodate handling of the high sample activity; these were counted far away from the detector face, where geometry differences become insignificant.

Exact effluent volumes were calculated from the measured net sample mass and the known solution densities. Densities were measured using volumetric flasks and net weight or by measured mass of a known pipetted volume. Because of the high dose rate from ^{137}Cs, many eluate samples required dilution before removal from the hot cell. The peak eluate samples were diluted by a factor of about 10,000x in 0.45 M HNO_3. The GEA count times were adjusted to accommodate the specific sample ^{137}Cs concentration to target $\leq$1% count uncertainty.

2.4.2 Composite Feed, Effluent, and Eluate Sample Analysis

The composite feed, effluent, and elution samples were submitted for chemical and radiochemical analyses to the ASO. The Test 1 samples underwent extensive analysis; the Tests 2-5 samples were subject to a more limited suite of analytes. The analyte concentrations are expected to duplicate from one test to the next and extensive analyses were not considered critical to all tests. An aliquot of the peak elution sample from Test 1 only was submitted for ICP-MS to determine the Cs isotopic distribution. The Cs isotopic distribution is expected to be constant for all samples. Table 2.9 provides the analysis summary for Tests 1-5 inclusive of the cross references to the project sample ID, ASO ASRs, ASO sample IDs, and analysis scope.

All analyses were conducted by the ASO according to standard operating procedures, the ASO QA Plan, and special instructions attached to the ASR. The ASO was responsible for the preparation and analysis of appropriate analytical batch and instrument quality control samples and to provide any additional processing to the sub-samples that might be required (e.g., acid digestion). Preparation by direct dilution (e.g., GEA) did not require preparative blanks and matrix spikes.

Table 2.9. Analytical Scope

AP-105DF Test	Sample Description	Sample ID	ASR	ASO Sample ID	Analysis Scope
1	Composite feed	TI014-FEED	0335	17-1240	GEA, IC, ICP-MS, ICP-OES, total alpha/beta, U-KPA, ^{90}Sr, ^{99}Tc, Np, Pu, Am/Cm, TOC/TIC, free OH
	Composite effluent	TI014-EFF Comp		17-1241	GEA, IC, ICP-MS, ICP-OES, total alpha/beta, U-KPA, ^{90}Sr, ^{99}Tc, Np, Pu, Am/Cm, TOC/TIC, free OH
	Composite eluate	TI014-ELComp		17-1242	GEA, IC, ICP-MS, ICP-OES, total alpha/beta, U-KPA, ^{90}Sr, ^{99}Tc, Np, Pu, Am/Cm, TOC/TIC, $H^{+(a)}$
	Peak elution sample	TI014-E8-A	0329	17-1224	Cs isotopic
2	Composite feed	TI015-FEED	0372	18-0001	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite effluent	TI015-EFFComp		18-0002	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite eluate	TI015-ELComp		18-0003	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
3	Composite feed	TI016-FEED		18-0004	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite effluent	TI016-EFFComp		18-0005	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite eluate	TI016-ELComp		18-0006	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
4 Take 1	Composite feed	TI020-FEED-1-A		18-0007	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite effluent	TI020-EFFComp		18-0008	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite eluate	TI020-ELComp		18-0009	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
4 Take 2	Composite feed	TI020-FEED-Take2		18-0010	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite effluent	TI020-EFFComp-Take2		18-0011	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite eluate	TI020-EComp		18-0012	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
5	Composite feed	TI021-FEED		18-0013	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite effluent	TI021-EFFComp		18-0014	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS
	Composite eluate	TI021-EComp		18-0015	GEA, ICP-OES, Pu, ^{99}Tc, U ICP-MS

(a) The large sample dilution rendered the H^+ analysis too inaccurate for meaningful evaluation.

IC = ion chromatography; KPA = kinetic phosphorescence analysis; TIC = total inorganic carbon; TOC = total organic carbon.

3.0 Column Test Results with AP-105DF

The Cs load and elution behavior was evaluated on all AP-105DF tank waste process cycles. This section discusses the load and elution results for all tests. Raw data are provided in Appendix A.

3.1 Cs Load, Feed Displacement, and Water Rinse Results

The cesium load profiles for the Tests 1-5 are shown in Figure 3.1 through Figure 3.6. Each figure is shown with the ordinate %C/C_0 on a probability scale versus the abscissa BV on a linear scale. The probability scale has two advantages over a linear scale and log scale: 1) Cs breakthrough profile appears linear, allowing for backward and forward extrapolations, and 2) more detail can be observed at the onset of breakthrough and the high breakthrough. Also provided are the FD and water rinses following AP-105DF loading for Tests 1-3; FD and water rinses were not collected as individual samples for Tests 4-5. Also shown is the 10% contract limit as nominally 0.015 % C/C_0.[1] Tests 4-5 incorporate the regeneration effluent solution %C/C_0 concentrations that were collected in discrete ~15-mL increments.

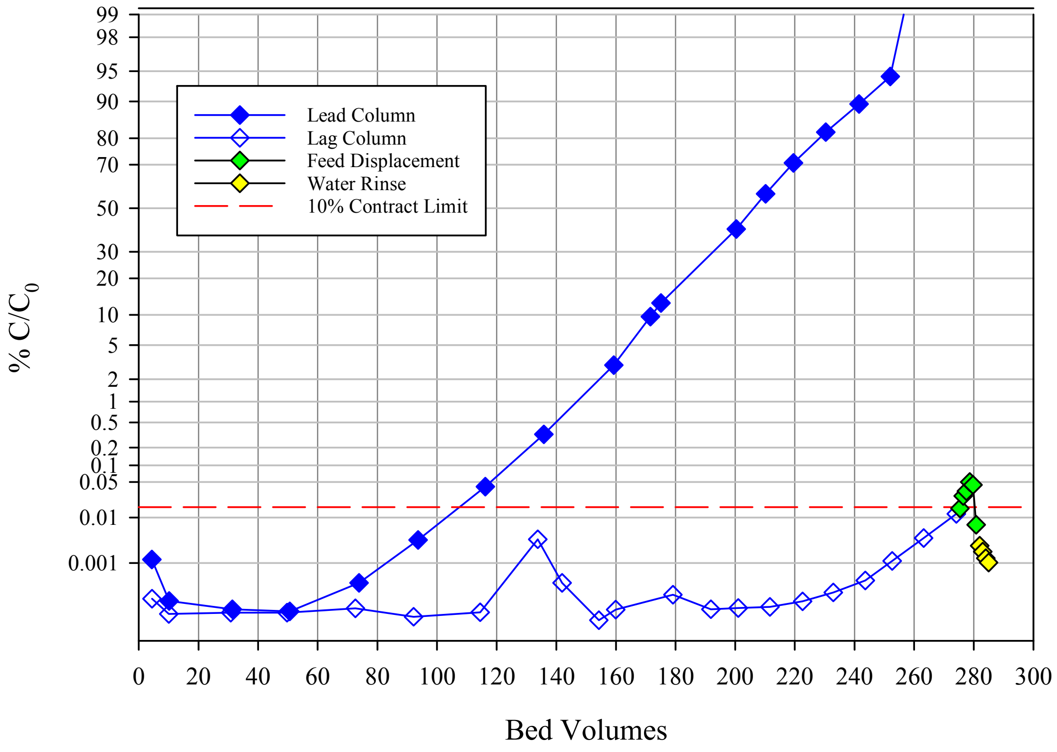

Figure 3.1. Test 1 Cesium Load Curve for AP-101DF, 1.80 BV/h

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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 5.7 M Na and 120 µCi ^{137}Cs/mL in the feed, the contract limit is 1.5E-3 C/C_0; 10% of this value is 0.015% C/C_0.

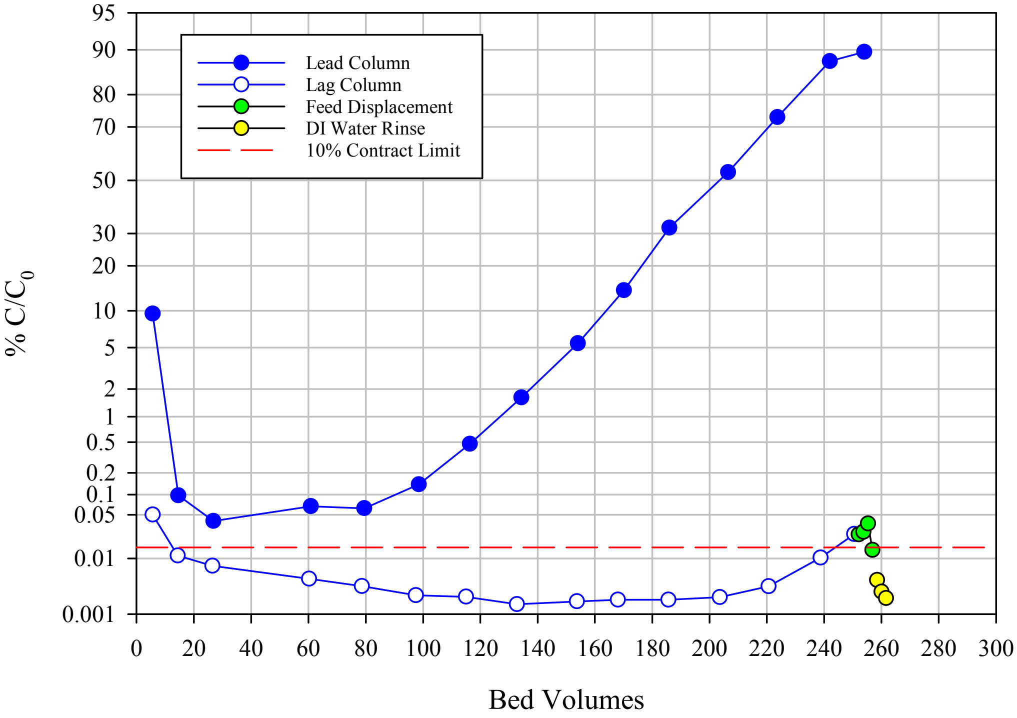

Figure 3.2. Test 2 Cesium Load Curve for AP-101DF, 3.05 BV/h

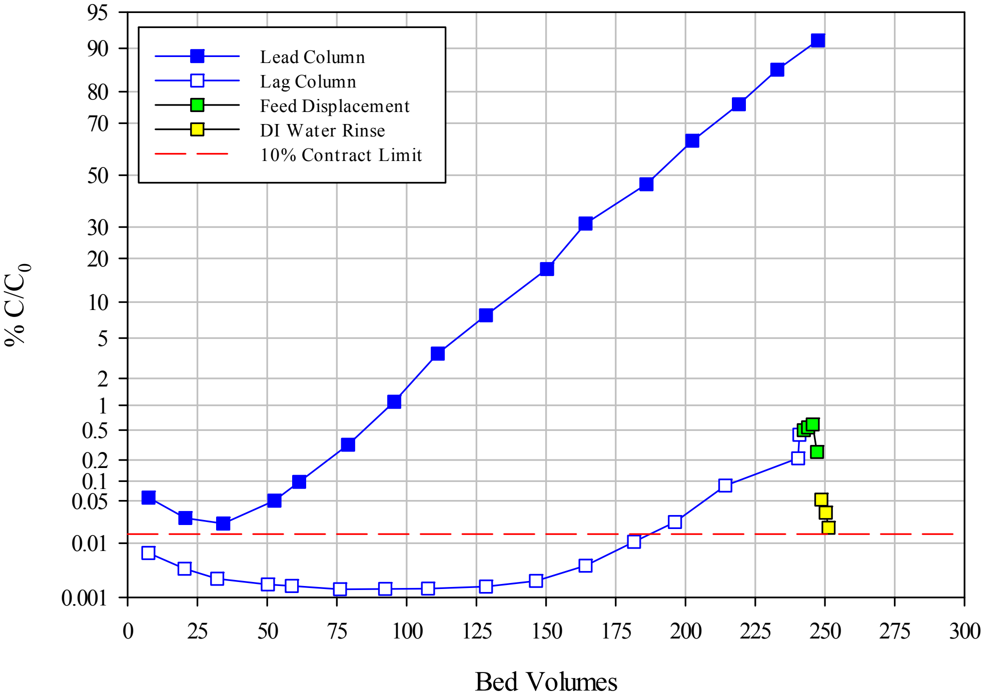

Figure 3.3. Test 3 Cesium Load Curve for AP-101DF, 4.53 BV/h

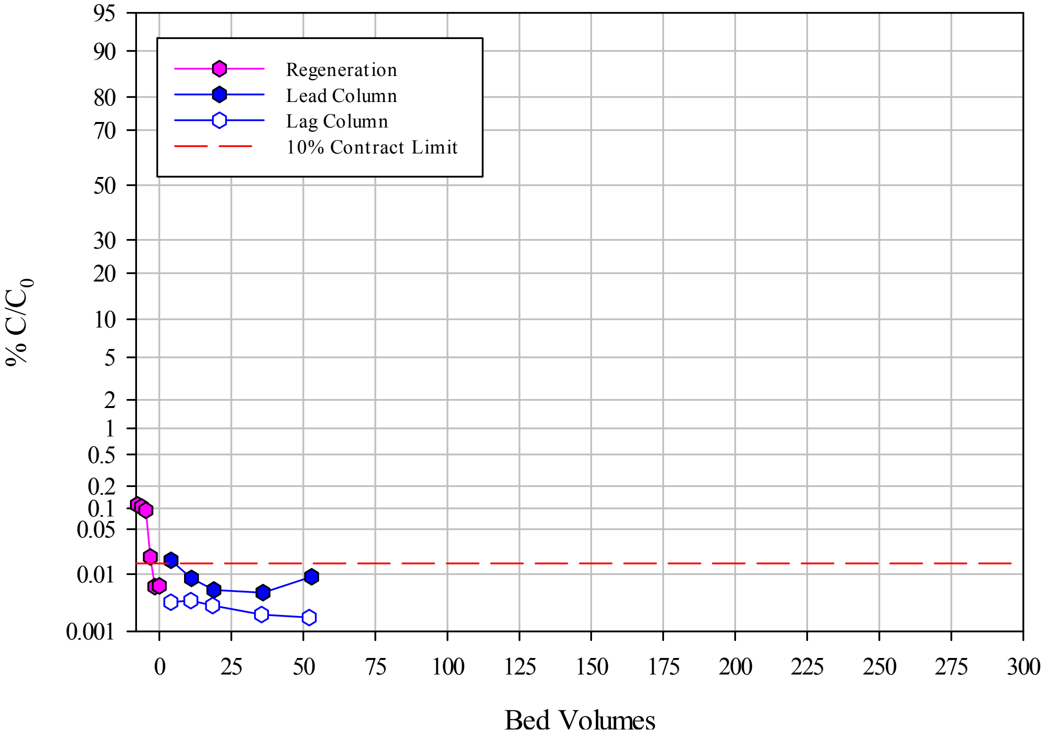

Figure 3.4. Test 4 Take 1 Cesium Load Curve for AP-101DF, 3.04 BV/h

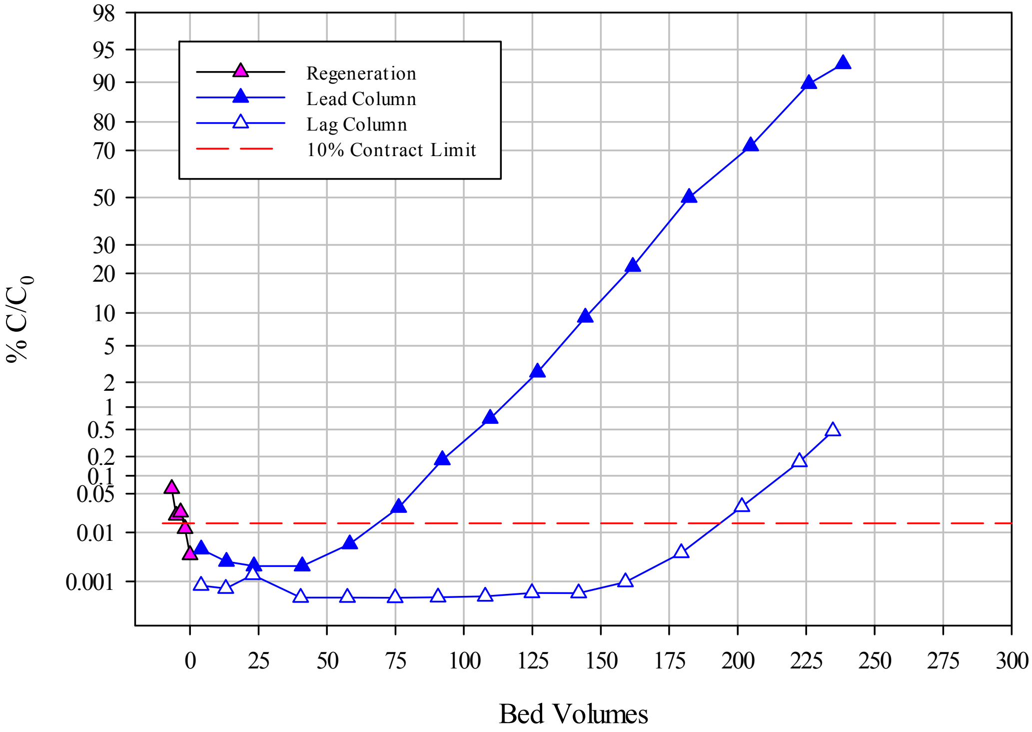

Figure 3.5. Test 4 Take 2 Cesium Load Curve for AP-101DF, 2.95 BV/h to 162 BVs, then 3.60 BV/h

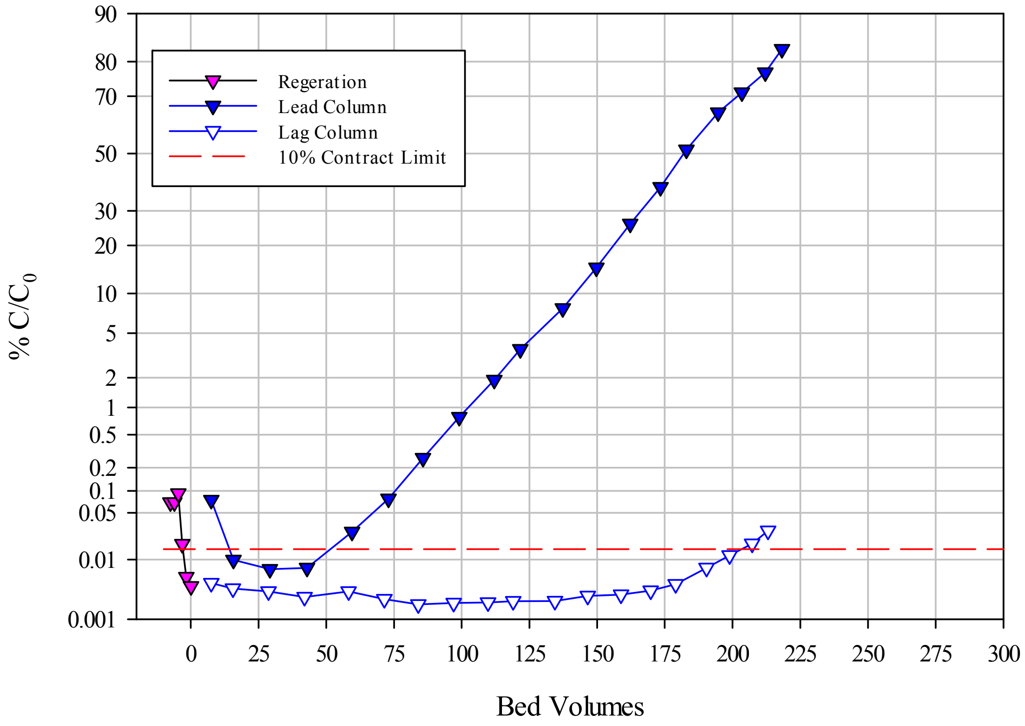

Figure 3.6. Test 5 Cesium Load Curve for AP-101DF, 3.17 BV/h

The first three BVs of FD solution continued the Cs breakthrough profile, as was expected because AP-105DF was still in the system. Starting with the fourth BV of FD, the Cs concentration in the effluent started to drop. The Cs concentration in the effluent continued to drop as water flow started through the system. Thus, the resin was shown to continue holding the Cs well as the fluid Cs, Na, and hydroxide concentrations decreased.

The regeneration solution showed a marked drop in Cs concentration as a function of BV processed. A similar trend can be observed for the first sample collected from the lead column. This is likely associated with a combination of two factors: 1) the rinsing of the lead column sample port from the last sample collected from the previous run and 2) some Cs leakage that developed over the interim standby period between process runs.

Table 3.1 provides the Cs-decontaminated effluent composite results in terms of ^{137}Cs concentration and overall decontamination factor (DF). A DF of 6620 was needed to meet the 10% contract limit. Three of the effluent composites exceeded this threshold and required batch contact processing to remove additional Cs in support of follow-on vitrification work (not reported herein).

Table 3.1. AP-105DF Effluent Composites ^{137}Cs Content and Decontamination Factor

Test	Effluent Container	^{137}Cs (µCi/mL)	Decontamination Factor
1	Effluent-1 (0-159 BVs)	7.07E-5	1.58E+06
	Effluent-2 (160-294 BVs)	7.25E-4	1.54E+05
2	Effluent-1 (0-154 BVs)	5.53E-3	2.14E+04
	Effluent-2 (154-254 BVs)	4.06E-3	2.91E+04
3	Effluent-1 (0-128 BVs)	2.21E-3	5.69E+04
	Effluent-2 (129-248 BVs)	**7.75E-2**	**1.62E+03**
4 Take1	Effluent-1 (0-53 BVs)	3.52E-3	3.45E+04
4 Take 2	Effluent-1 (0-144 BVs)	7.26E-4	1.68E+05
	Effluent-2 (144-238 BVs)	**1.05E-1**	**1.16E+03**
5	Effluent-1 (0-99 BVs)	3.87E-3	3.23E+04
	Effluent-2 (99-183BVs)	7.12E-3	1.75E+04
	Effluent-3 (183-218 BVs)	**2.12E-2**	**5.90E+03**

Bolded effluents exceeded the 6620 DF (10% of the waste acceptance criteria).

3.2 Cesium Elution Results

The elution profiles for Tests 1-3 are provided in Figure 3.7. (Tests 4-5 did not assess elution profiles.) The 0.45 M HNO_3 solution flow is shown in solid symbols; the water rinse following elution is shown in open symbols. There was no substantial difference in the elution profiles for the two flowrates tested (1.4 and 2.0 BV/h). Peak Cs removal occurred between 10 and 13 BVs. Tailing was similar. Tailing is an important attribute for the next process cycle as residual Cs on the lag column can greatly influence the subsequent load cycle effluent DF. Although 99+% of Cs removal can be attained, the residual Cs can contaminate the next cycle product.

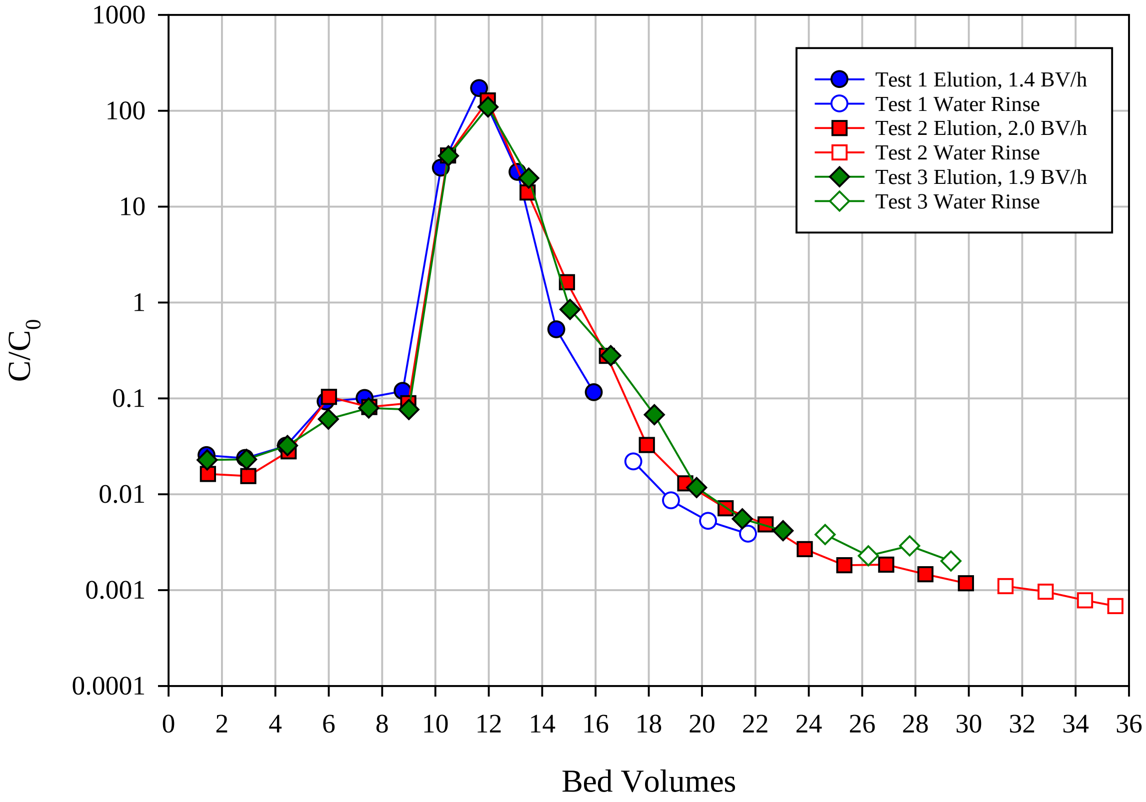

Figure 3.7. Elution Profiles for Tests 1-3

3.3 Activity Balance

The ^{137}Cs fractionation was determined between the effluents (collected in two to three different collection bottles), samples collected during the load processing, FD, water rinse, elution, and the final water rinses. The ^{137}Cs fractionation in the final extended water rinses was conducted on samples from Tests 4-5. The microcuries of ^{137}Cs loaded onto the lead and lag columns were calculated. Table 3.2 through Table 3.7 summarize the ^{137}Cs fractionations found in the various effluents as well as the Cs column loading for each process test. Overall sample handling and analytical uncertainty were estimated at ±3-5% and ±2%, respectively. Therefore, a Cs activity balance within 95% to 105% was considered excellent recovery.

Table 3.2. ^{137}Cs Activity Balance for AP-105DF Test 1

Input	µCi	%
Feed Sample	328,834	100
Output		
Effluent-1 (0-159 BVs)	0.108	3.29E-05
Effluent-2 (159-294 BVs)	0.938	2.85E-04
Load samples	1740	0.529
Loss (spill)	271	0.082
Feed displacement	2.16	6.58E-04
Water rinse	0.0775	2.36E-05
Elution	351,558	107
Water rinse	4.40	1.34E-03
Total ^{137}Cs recovery	353,577	108
Total ^{137}Cs Column Loading		
Lead column	229,270	69.7
Lag column	97,552	29.7

Table 3.3. ^{137}Cs Activity Balance for AP-105DF Test 2

Input	µCi	%
Feed Sample	294,285	100
Output		
Effluent-1 (0-154 BVs)	8.32	2.83E-03
Effluent-2 (154-254 BVs)	3.83	1.30E-03
Load samples	911	0.310
Feed displacement	1.944	6.61E-04
Water rinse	0.163	5.55E-05
Elution	313,584	107
Water rinse	0.958	3.25E-04
Total ^{137}Cs recovery	314,510	107
Total ^{137}Cs Column Loading		
Lead column	233,663	79.4
Lag column	59,699	20.3

Table 3.4. ^{137}Cs Activity Balance for AP-105DF Test 3

Input	µCi	%
Feed Sample	312,539	100
Output		
Effluent-1 (0-128 BVs)	2.76	8.84E-04
Effluent-2 (128-248 BVs)	85.4	2.73E-02
Load samples	3775	1.21
Feed displacement	37.2	1.19E-02
Water rinse	1.89	6.05E-04
Elution	307,268	98.3
Water rinse	2.74	8.76E-04
Total ^{137}Cs recovery	311,173	100
Total ^{137}Cs Column Loading		
Lead column	236,739	75.7
Lag column	71,936	23.0

Table 3.5. ^{137}Cs Activity Balance for AP-105DF Test 4, Take 1

Input	µCi	%
Feed Sample	66,520	100
Output		
Effluent-1 (0-53 BVs)	1.78	2.67E-03
Load samples	0.20	3.02E-04
Elution	68,063	102
Total ^{137}Cs recovery	68,067	102
Total ^{137}Cs Column Loading		
Lead column	66,515	100
Lag column	3.14	0.00008

Table 3.6. ^{137}Cs Activity Balance for AP-105DF Test 4, Take 2

Input	µCi	%
Feed Sample	294,050	100
Output		
Effluent-1 (0-144 BVs)	1.01	3.45E-04
Effluent-2 (144-238 BVs)	95.8	3.26E-02
Load samples	1286	0.437
Feed displacement and water rinse	0.5	1.81E-04
Elution	302,697	103
Water rinse	15.2	5.16E-03
Extended water rinse	3.1	1.04E-03
Total ^{137}Cs recovery	304,091	103
Total ^{137}Cs Column Loading		
Lead column	229,186	77.9
Lag column	64,194	21.8

Table 3.7. ^{137}Cs Activity Balance for AP-105DF Test 5

Input	µCi	%
Feed Sample	259,960	100
Output		
Effluent-1 (0-99 BVs)	3.64	1.40E-03
Effluent-2 (99-183BVs)	5.67	2.18E-03
Effluent-3 (183-218 BVs)	6.97	2.68E-03
Load samples	1942	0.747
Feed displacement and water rinse	3.8	1.46E-03
Elution	258,380	99.4
Water rinse	15.4	5.94E-03
Extended water rinse	4.1	1.56E-03
Total ^{137}Cs recovery	260,357	100
Total ^{137}Cs Column Loading		
Lead column	213,597	82.2
Lag column	44,867	17.3

3.4 Ion Exchange Column Absorbed Dose Calculation

The isotope ^{137}Cs decays to the metastable state of ^{137}Ba with the emission of a beta particle that has an average energy of 169.6 keV, and the metastable state de-excites to the ground state with the emission of a 662-keV photon. As the ^{137}Cs loads onto the resin bed, the resin is exposed to increasing decay energy (beta and gamma source terms). The accumulated dose to the SRF resin was determined from the calculated ^{137}Cs load and was assumed to be distributed over the entire volume of the resin bed. More realistically, the Cs load is more concentrated at the top of the bed.

Absorbed dose was calculated on a time step basis using the grab samples taken at intervals while the ion exchange system was operated. This involved determining a volumetric ^{137}Cs activity concentration, C_v, in each column after each grab sample. This concentration was derived from the GEA data for each of these grab samples. To first determine the gamma ray contribution to the column's absorbed dose, an exposure rate based on the volumetric geometry of the column was calculated using a volume source formula from Stabin (2007):

$$Exposure\ rate\left[\frac{C}{kg\ h}\right] = \dot{X} = \pi\, \Gamma \left(\frac{C_v}{\mu}\right)(1 - e^{-\mu t}) \ln\left(\frac{r^2 + h^2}{h^2}\right) \tag{3.1}$$

where Γ is the ^{137}Cs gamma constant 0.33 $\frac{R*m^2}{hr*Ci}$ (Saenger et al. 1972); μ is the linear attenuation coefficient of the material (assumed to be water) based on ICRU Report 44 (ICRU 1989):

$$\mu = \frac{\mu}{\rho_m} * \rho \tag{3.2}$$

where $\frac{\mu}{\rho_m}$ is the mass attenuation coefficient; ρ is the density of the material; t is the thickness of the source (ID of column 1.43 cm); r is the radius of the source $\left(\frac{t}{2}\right)$; h is the height of the source (6.3 cm).

Exposure rate was converted to exposure based on the duration of the time step. The column's exposure is converted to absorbed dose via Stabin (2007):

$$D\ [rad] = \frac{d\varepsilon}{dm} = 0.88\ X\ \frac{\left(\frac{\mu_{en}}{\rho}\right)_{material}}{\left(\frac{\mu_{en}}{\rho}\right)_{air}} \tag{3.3}$$

where $d\varepsilon$ is the mean energy imparted by ionizing radiation to matter in a volume element of mass dm; X is the exposure (R); $\left(\frac{\mu_{en}}{\rho}\right)_{material}$ is the mass energy absorption coefficient for the specified material at the photon energy of interest (assumed to be water, from ICRU Report 44 [ICRU 1989]); while $\left(\frac{\mu_{en}}{\rho}\right)_{air}$ is the mass energy absorption coefficient for dry air at the photon energy of interest (ICRU 1989).

The mass energy absorption coefficient for the 662 keV was interpolated from a log-log plot using the following linear interpolation formula:

$$\frac{[ln(Y) - ln(Y_1)]}{[ln(X) - ln(X_1)]} = \frac{[ln(Y_2) - ln(Y_1)]}{[ln(X_2) - ln(X_1)]} \tag{3.4}$$

where Y is the desired mass energy absorption coefficient component; X is the gamma energy that Y is being solved for; and $(X_1, Y_1), (X_2, Y_2)$ are the known data points.

Solving for Y leads to:

$$Y = \exp\left(\frac{[ln(X) - ln(X_1)] * [ln(Y_2) - ln(Y_1)]}{[ln(X_2) - ln(X_1)]} + ln(Y_1)\right) \tag{3.5}$$

For the beta dose contribution, it is assumed that all beta particle energy from the ^{137}Cs is deposited into the resin material (Slaback and Schlein 1998):

$$\begin{gathered} \dot{D}\left[\frac{rad}{s}\right] = (1.6E - 8)\ A_c\ \bar{E} \\ \bar{E}\ (\beta^-) \approx \frac{1}{3}\ E_{max} \end{gathered} \tag{3.6}$$

where $\bar{E}$ is the average energy of the beta particle (MeV) (0.169 MeV for ^{137}Cs [Browne and Tuli 2007]); A_c is the radionuclide activity concentration in the source (Bq/g); the mass of the source was based on water at 30 °C for the volume of the source.

Figure 3.8 through Figure 3.19 show the cumulative column dose for each test as a function of time. In each of these figures, the last data point for Cs loading is marked with a solid vertical black line while the final data point for the FD data is noted by a vertical dashed black line. Column elution represents the data after the FD line.

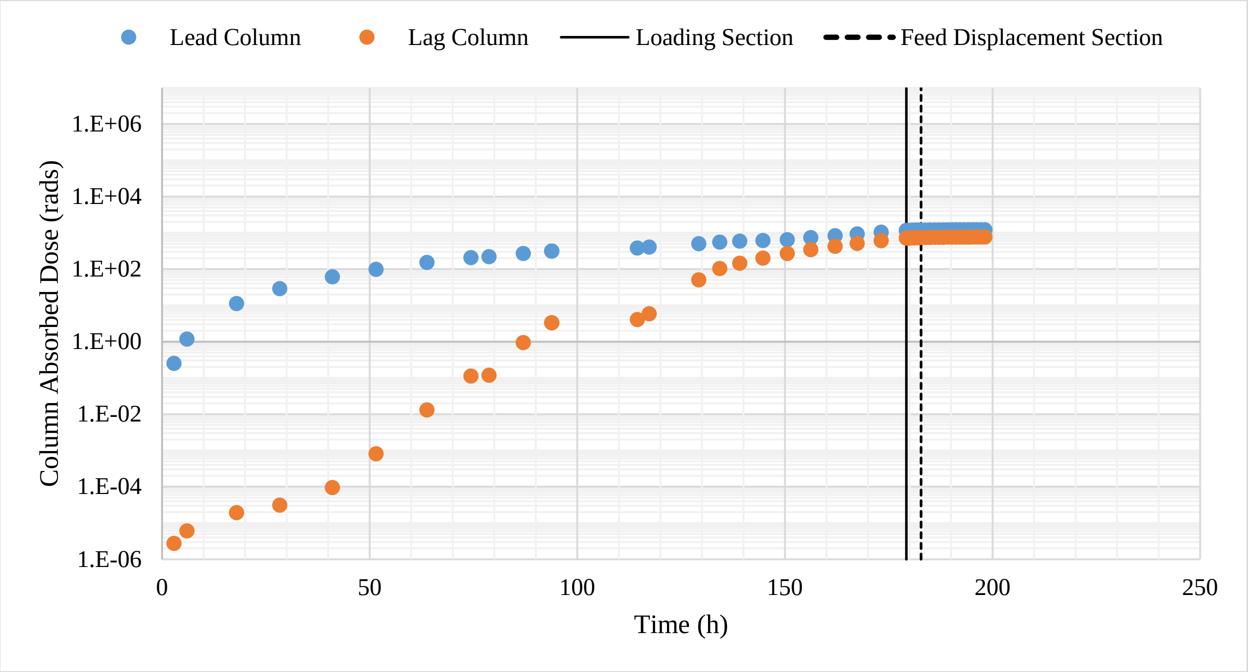

Figure 3.8. Test 1 Column Absorbed Gamma Dose as a Function of Time

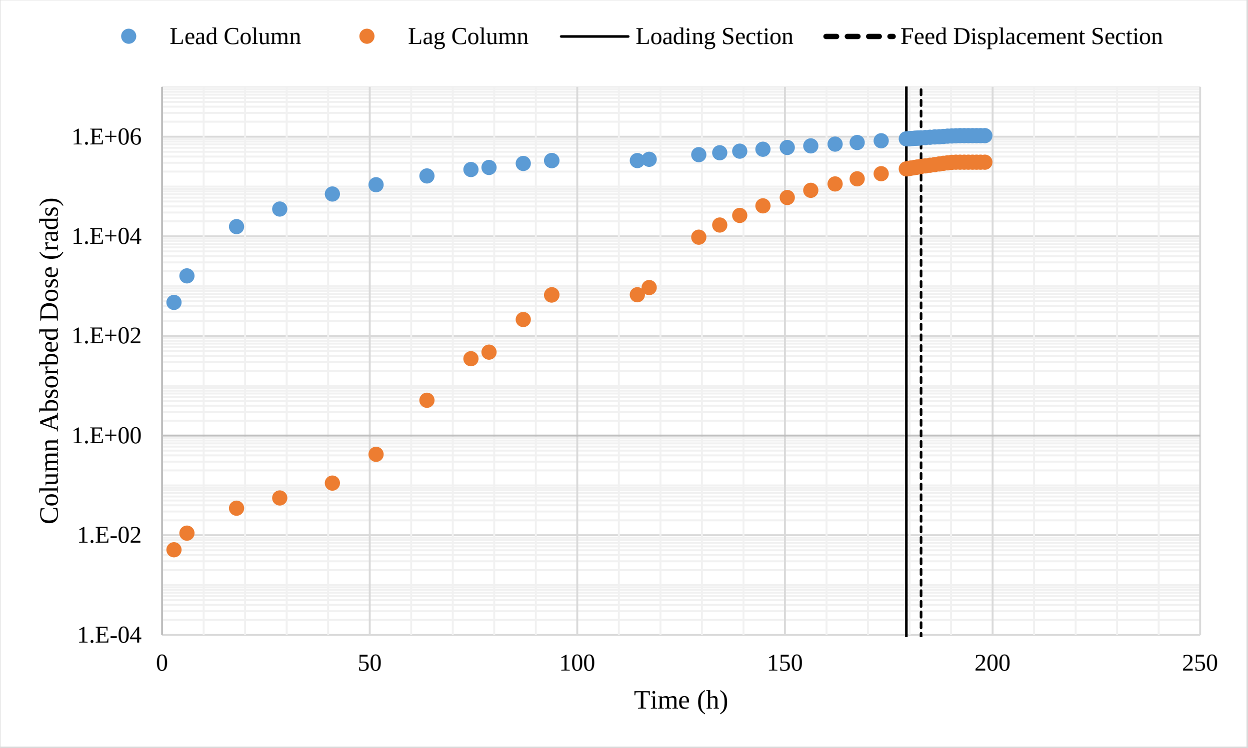

Figure 3.9. Test 1 Column Absorbed Beta Dose as a Function of Time

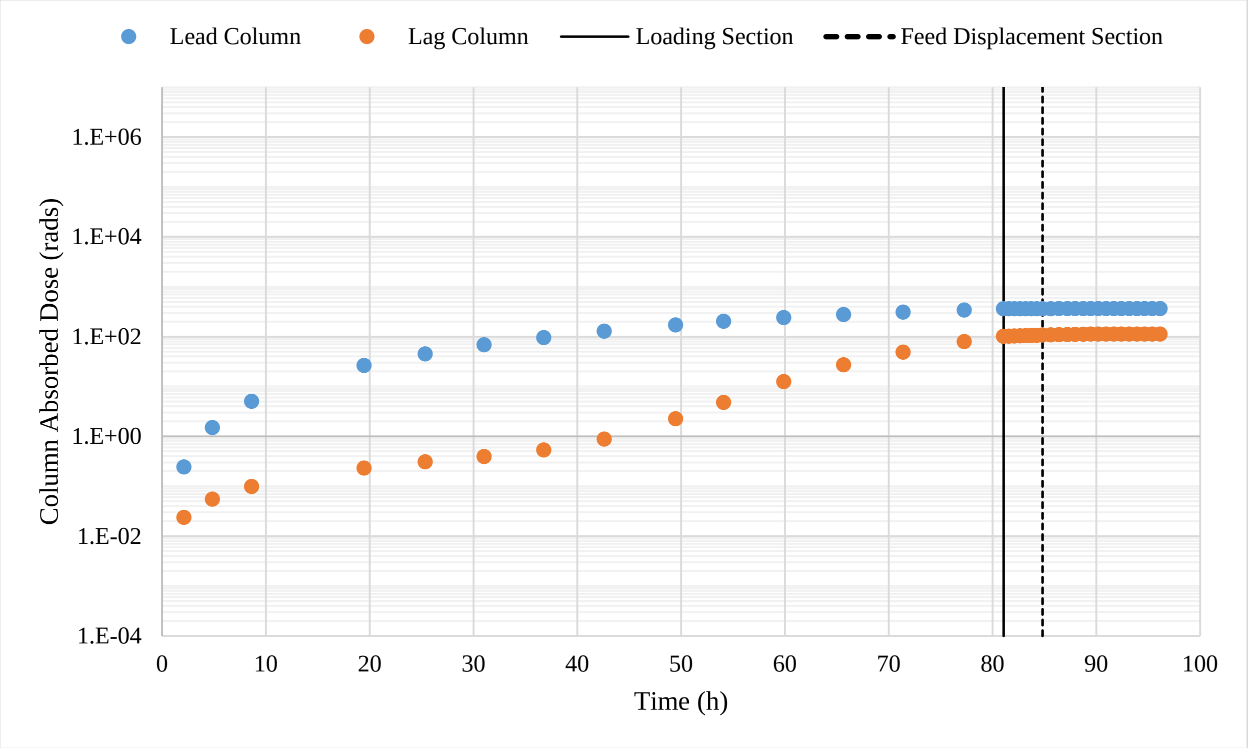

Figure 3.10. Test 2 Column Absorbed Gamma Dose as a Function of Time

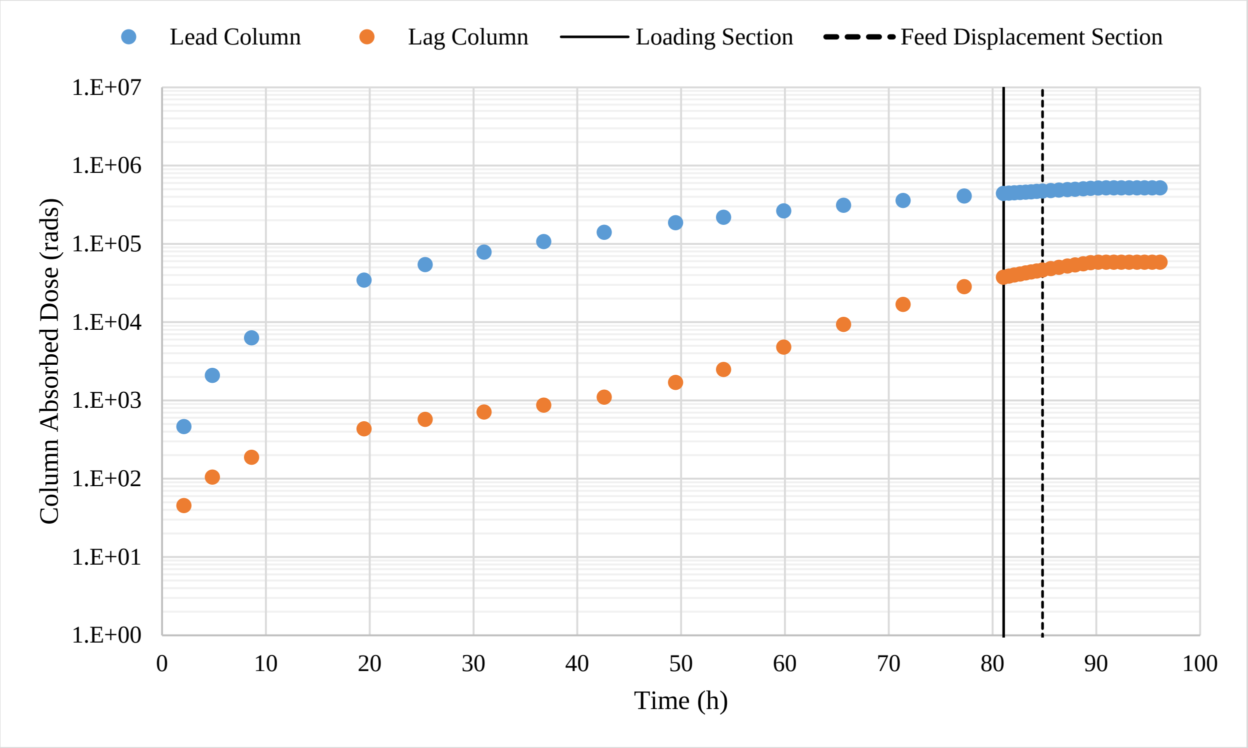

Figure 3.11. Test 2 Column Absorbed Beta Dose as a Function of Time

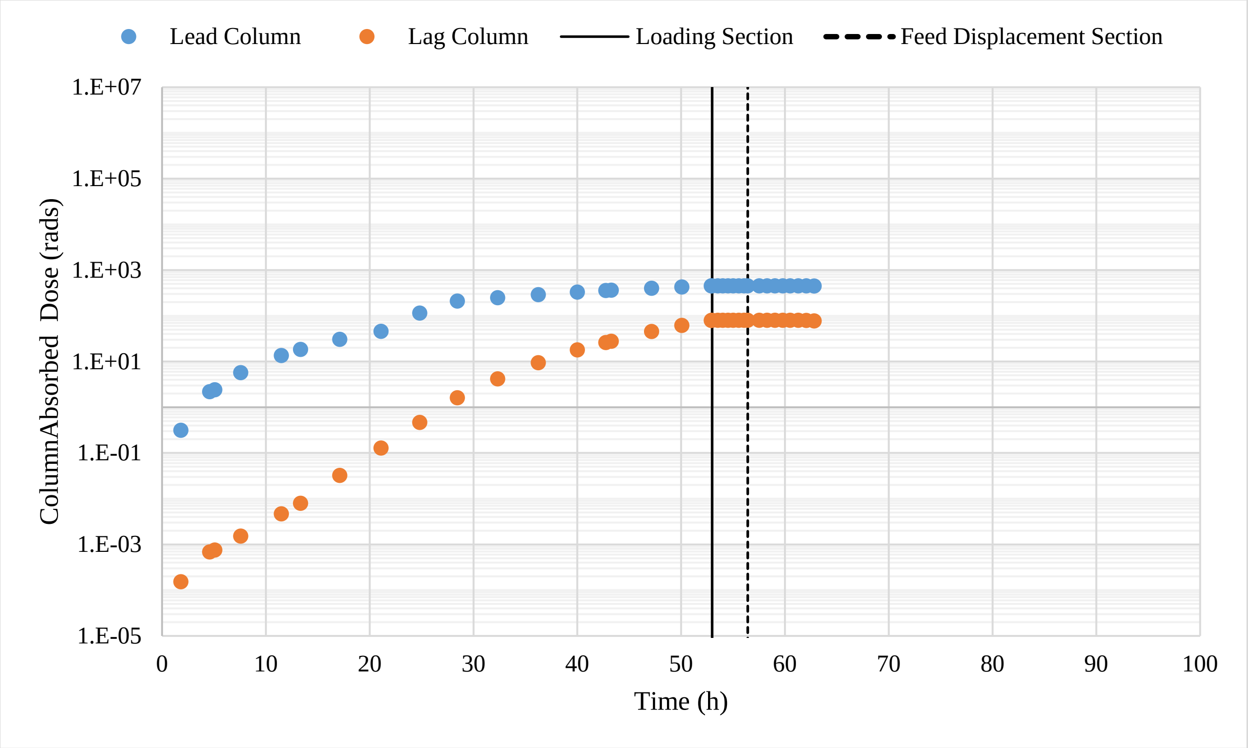

Figure 3.12. Test 3 Column Absorbed Gamma Dose as a Function of Time

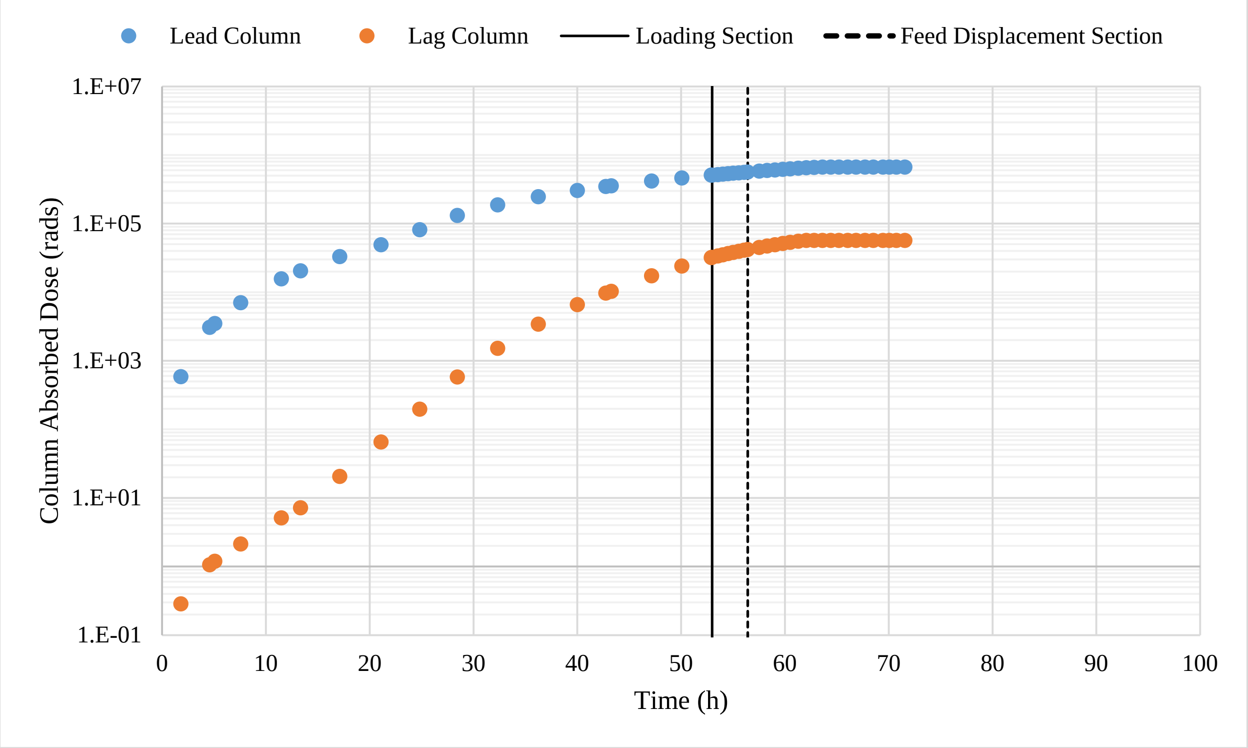

Figure 3.13. Test 3 Column Absorbed Beta Dose as a Function of Time

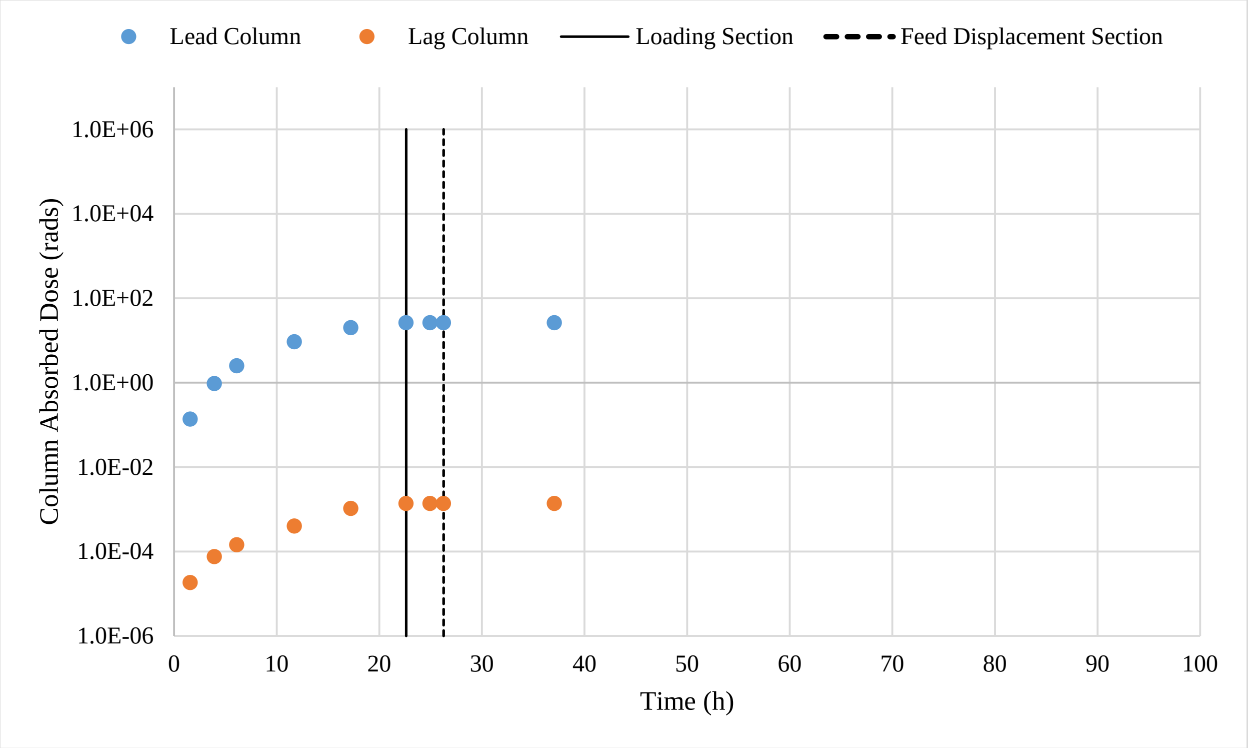

Figure 3.14. Test 4 Take 1 Column Absorbed Gamma Dose as a Function of Time

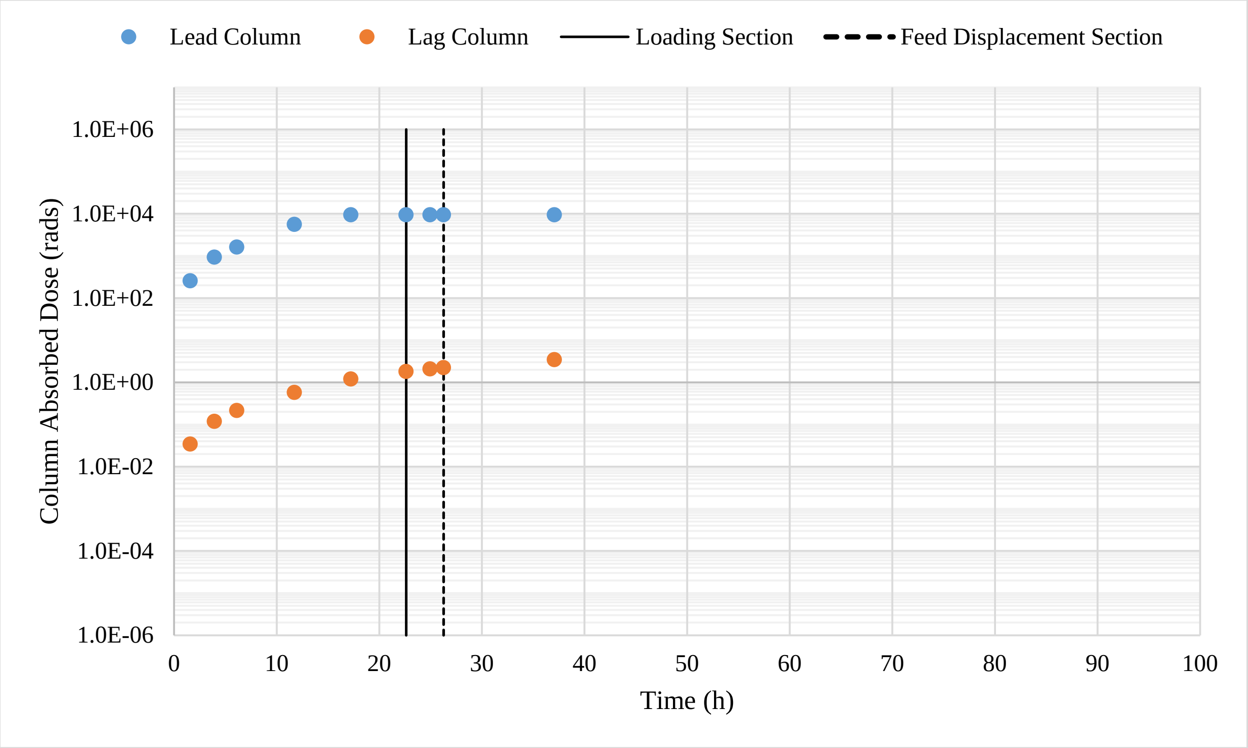

Figure 3.15. Test 4 Take 1 Column Absorbed Beta Dose as a Function of Time

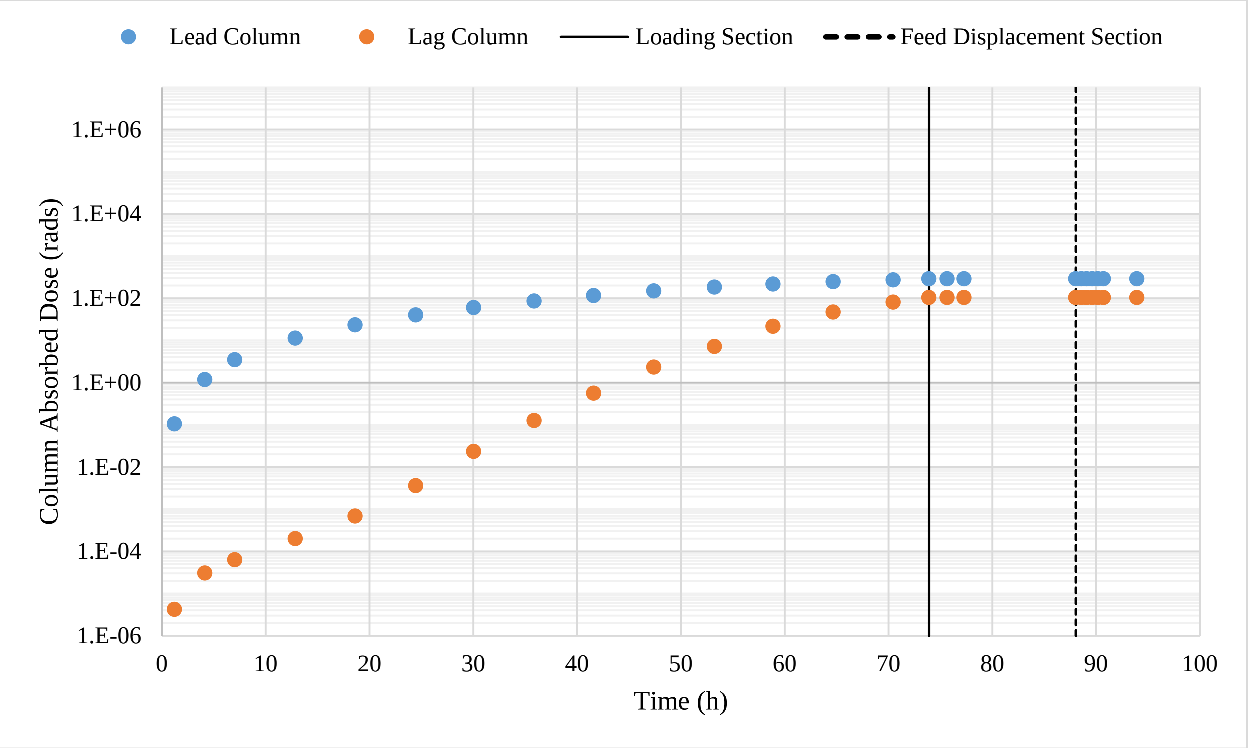

Figure 3.16. Test 4 Take 2 Column Absorbed Gamma Dose as a Function of Time

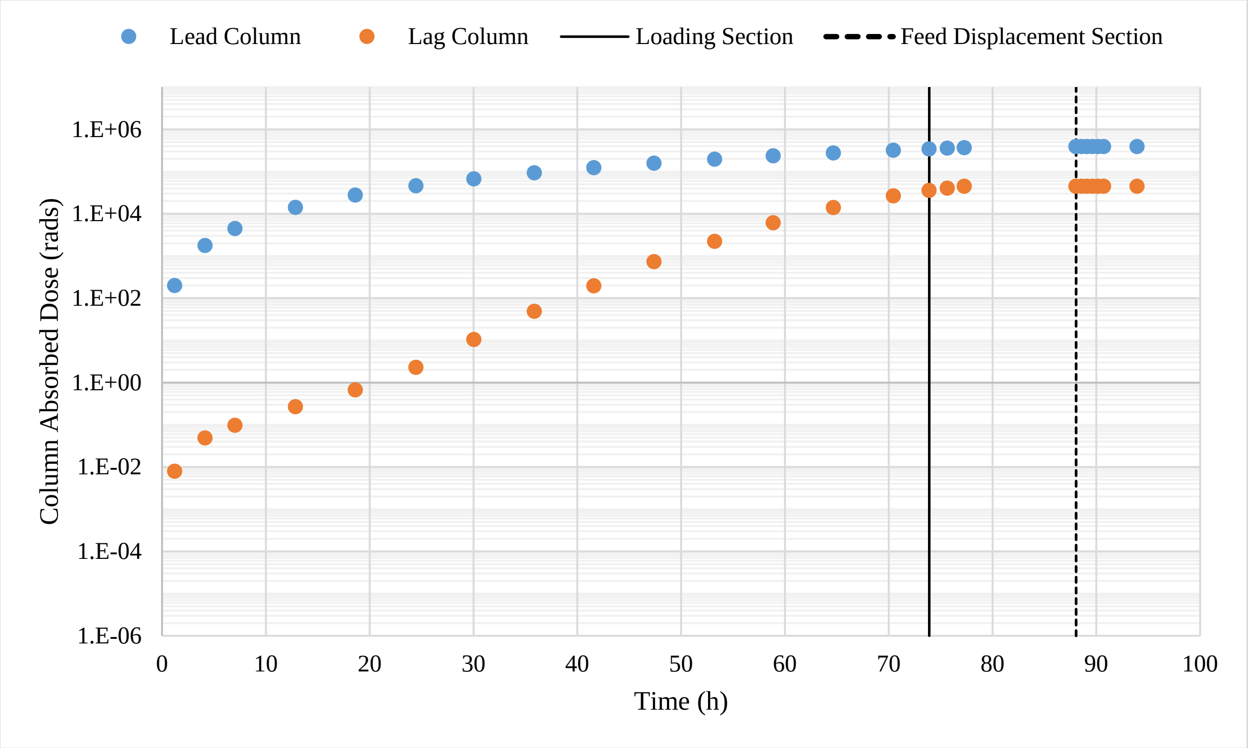

Figure 3.17. Test 4 Take 2 Column Absorbed Beta Dose as a Function of Time

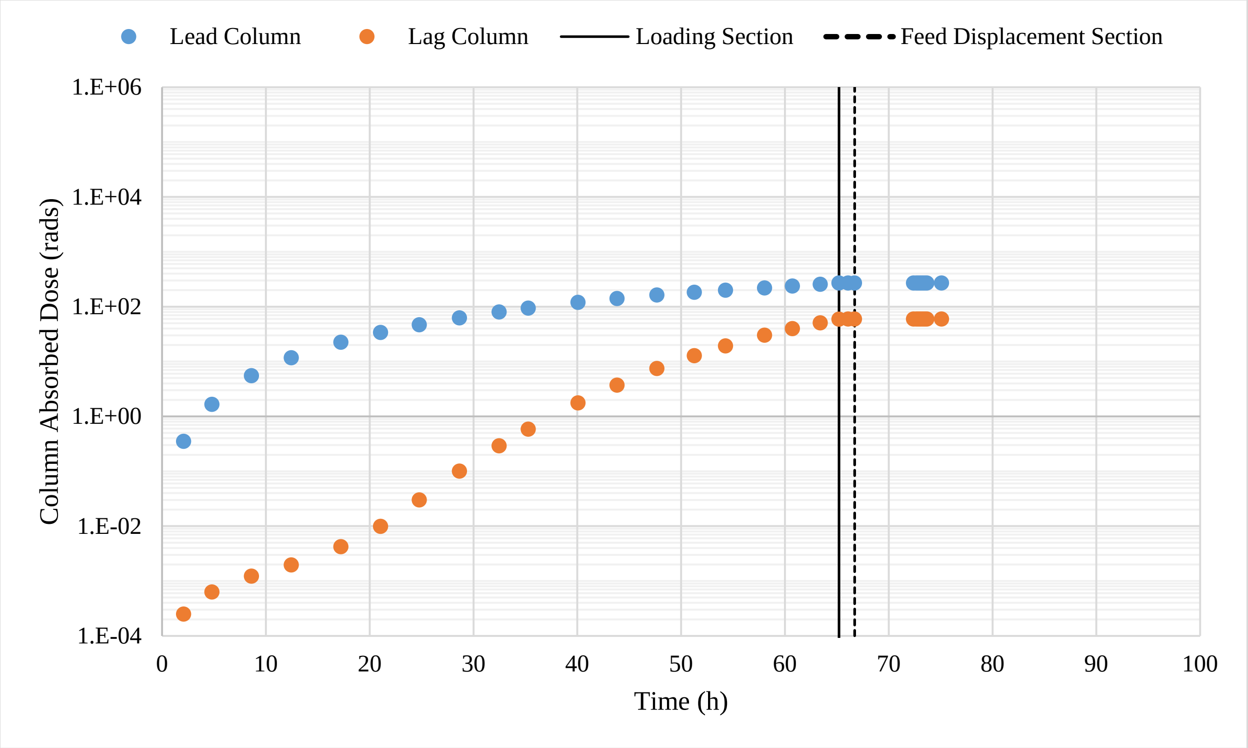

Figure 3.18. Test 5 Column Absorbed Gamma Dose as a Function of Time

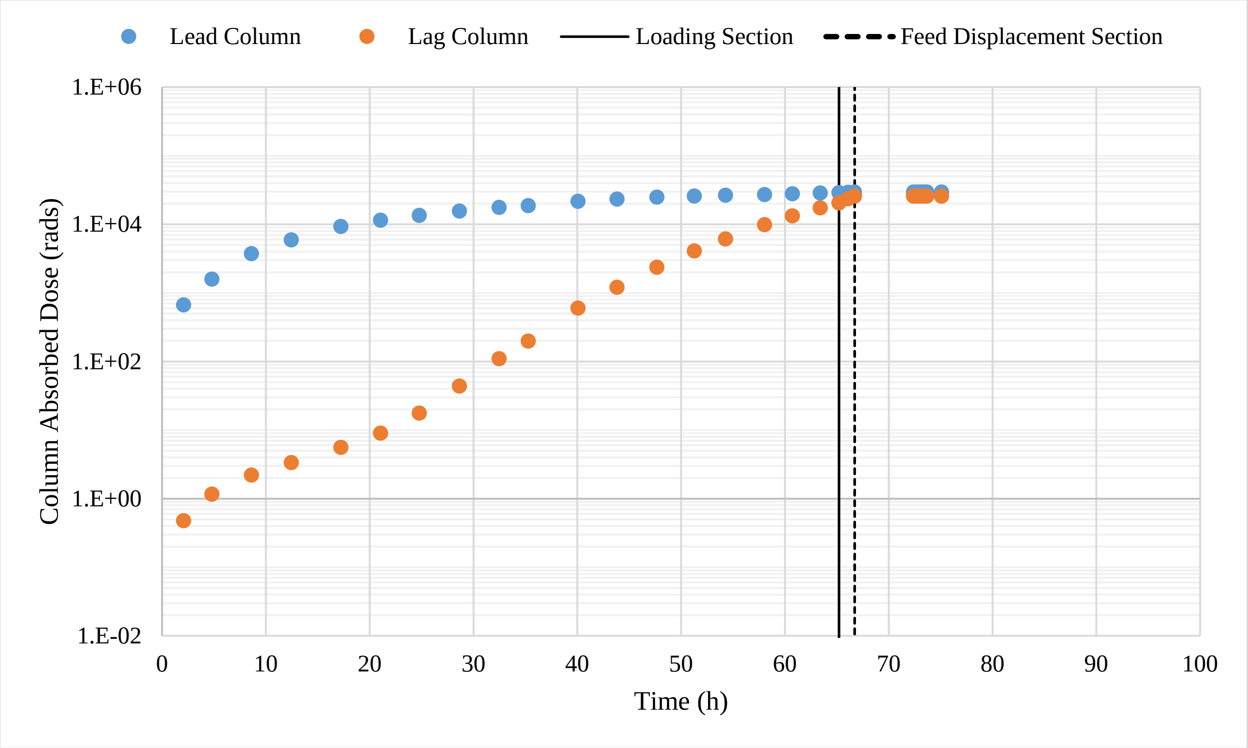

Figure 3.19. Test 5 Column Absorbed Beta Dose as a Function of Time

An exposure rate measurement of the Shielded Analytical Laboratory hot cell was taken at the location of the ion exchange system with an MGP Instruments (Mirion) AMP-100 energy compensated GM-tube. This detector had a ±10% error associated with it and could measure from 0.5 mR/h to 1000 R/h. The background exposure rate was measured to be 14.5 R/h inside the hot cell in the location of the ion exchange system. Figure 3.20 displays this rate from the time that ion exchange system began Test 1 to the completion of Test 5.

This high background resulted in substantial dose contribution when compared to the internal column dose as a result of ^{137}Cs loading into the system while operating inside the hot cell. Table 3.8 summarizes the cumulative absorbed dose each column received at the end of each test due to the ^{137}Cs loading as well as the hot cell background radiation. The final cumulative absorbed dose the ion exchange system received from background was on the order of 2.26×10^5 rads while the final cumulative absorbed dose due to ^{137}Cs was 2.64×10^6 rads to the lead column and 4.92×10^5 rads to the lag column.

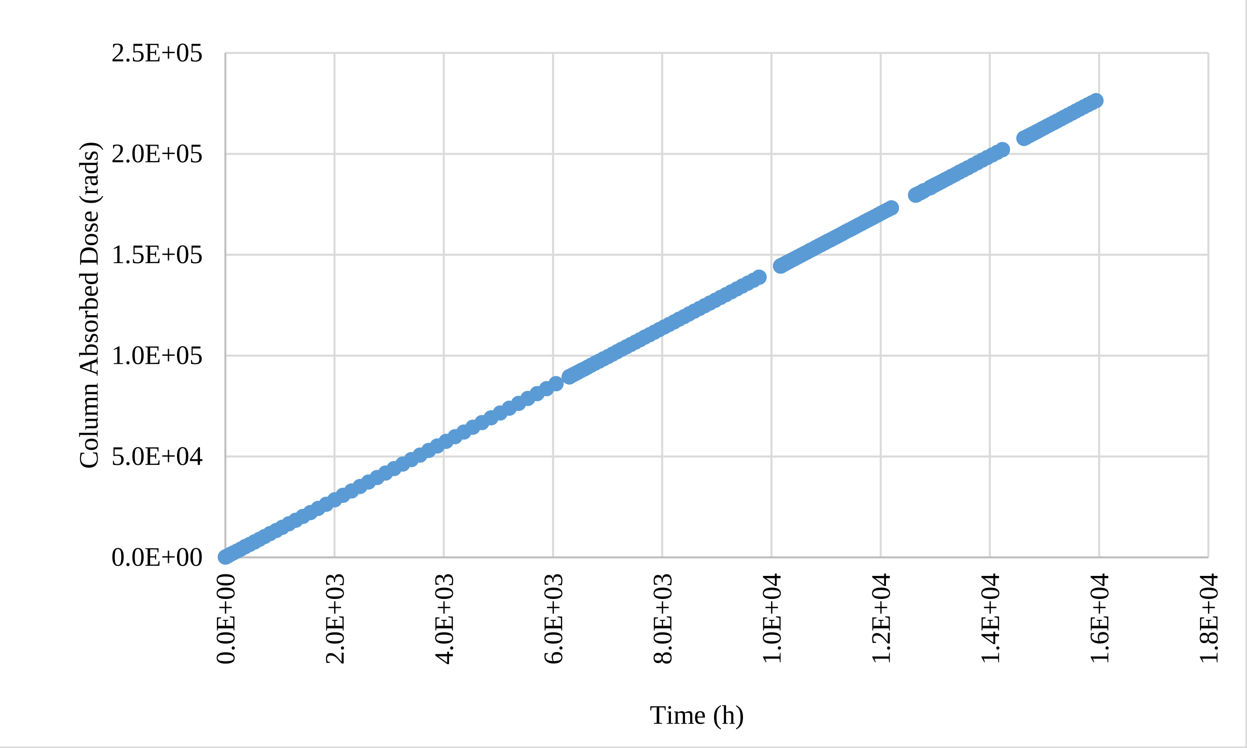

Figure 3.20. Column Absorbed Dose as a Function of Time during Tests 1-5 (including background from hot cell)

Table 3.8. Absorbed Dose Summary for Tests1-5

	Lead Column	Lag Column	Both Columns
Test	Cumulative Absorbed Dose to Column from ^{137}Cs Loading, rads[(a)]	Cumulative Absorbed Dose to Column from ^{137}Cs Loading, rads[(a)]	Cumulative Absorbed Dose to Columns Due to Background, rads[(a)]
1	1.04E+06	3.07E+05	8.94E+04
2	5.17E+05	5.82E+04	5.49E+04
3	6.61E+05	5.65E+04	3.51E+04
4 Take 1	9.42E+03	3.44E+00	3.70E+03
4 Take 2	3.87E+05	4.45E+04	2.45E+04
5	2.97E+04	2.57E+04	1.87E+04

(a) Cumulative column adsorbed dose from AP-105DF processing at the end of each test.

Fully loaded, full-scale columns at the LAWPS were modeled to see 300 MRad over the resin lifetime (i.e., 30 process cycles) or 10 MRad per process cycle. The gamma dose from the hot cell operations was estimated to be 2.87 MRad by the conclusion of the fifth process cycle. This was a substantial dose to SRF relative to a single process cycle at full scale, but it was a small fraction (1/100th) of the estimated full-scale resin lifetime dose. With the limited dose applied to this resin, extrapolation of synergistic effects from dose, chemical, and physical degradation mechanisms at full scale is difficult.

3.5 Chemical and Radiochemical Composition

The compositions of the AP-105DF ion exchange tests feeds, effluents, and eluates were evaluated to determine analyte fractionation. Test 1 feed, effluent, and eluate underwent extensive characterization to support follow-on work for vitrification and to understand analyte mass fractionation through the SRF ion exchange process. Table 3.9 summarizes the radioisotopic concentrations and fractionations and Table 3.10 summarizes the metals, anions, inorganic and organic carbon concentrations, and fractionations from Test 1. The analyte fractionations were calculated as ratios of the total analyte measured in the feed processed through the column and the total analyte collected in the Cs-decontaminated effluent and the Cs-bearing eluate according to Eqs. (3.7) and (3.8).

$$\frac{C_{Da} \times V_D}{C_{Fa} \times V_F} = F_{Da} \tag{3.7}$$

$$\frac{C_{Ea} \times V_E}{C_{Fa} \times V_F} = F_{Ea} \tag{3.8}$$

where C_{Da} = concentration of analyte (a) in the Cs-decontaminated effluent
V_D = volume of Cs-decontaminated effluent
C_{Fa} = concentration of analyte (a) in the AP-105DF feed
V_F = volume of AP-105DF feed
F_{Da} = fraction of analyte (a) in the Cs-decontaminated effluent
C_{Ea} = concentration of analyte (a) in the Cs-bearing eluate

V_E = volume of Cs-bearing eluate
F_{Ea} = fraction of analyte (a) in the Cs-bearing eluate

Analyses of the feed effluents and eluates for the remaining tests (Tests 2-5) were more limited in scope: GEA, ^{99}Tc, $^{239+240}$Pu, metals by ICP-OES, and U (ICP-MS). Ion exchange results for anions, free hydroxide, TIC, TOC, ^{90}Sr, and ^{237}Np were expected to be equivalent to those found in Test 1, i.e., no exchange whatsoever. It was noted that the $^{243+244}$Cm results were indeterminate in Test 1; however, the Cm and Am chemistry should mirror each other and Cm activity recovery was not further followed. Table 3.11 through Table 3.13 provide results for Tests 2-5 along with the percent recovery in the effluent and the eluate for each test.

Some analyte results are shown in brackets; this indicates that the analytical result was less than the estimated quantitation limit 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 value(s) to highlight the higher uncertainty. The opportunistic analyte results measured by ICP-OES are also shown in Table 3.10; these analytes are part of the data output but have not been fully evaluated for quality control performance.

Table 3.9. AP-105DF Feed, Effluent, and Eluate Compositions (Test 1) ASR 0335, Radionuclides

Analysis Method	Analyte	TI014-FEED µCi/mL[(a)]	TI014-EFF-Comp µCi/mL[(a)]	TI014-ELComp µCi/mL[(a)]	Fraction in Effluent %	Fraction in Eluate %
Gamma Energy Analysis (GEA)	^{60}Co	<3.2E-3	5.93E-4	<8.6E-2	--	--
	^{126}Sn/Sb	<1.1E-2	1.75E-5	<1.5E-1	--	--
	^{137}Cs	1.22E+2	5.30E-4	2.43E+3	0.00043%	107%
	^{152}Eu	<6.3E-3	6.07E-6	<3.3E-1	--	--
	^{154}Eu	<1.2E-2	1.35E-4	<2.4E-1	--	---
	^{241}Am	<5.4E-1	3.72E-4	<1.3E+1	--	--
Separations/ Alpha Energy Analysis (AEA)	^{237}Np	9.27E-6	8.57E-6	<MDL	92%	--
	^{238}Pu	1.93E-5	1.39E-5	1.39E-5	72%	3.9%
	$^{239+240}$Pu	5.18E-5	4.85E-5	1.97E-5	94%	2.0%
	^{241}Am	2.53E-4	2.55E-4	5.92E-5	101%	1.3%
	^{242}Cm	[8.11E-7]	[6.04E-7]	[3.87E-7]	[74%]	[2.6%]
	$^{243+244}$Cm	1.74E-5	1.15E-5	1.00E-5	66%	3.1%
Separations/ Beta Counting	^{90}Sr	5.78E-1	5.68E-1	6.79E-2	98%	0.63%
	^{99}Tc	9.54E-2	7.47E-2	1.05E-3	78%	0.059%
Proportional Counting	Total Alpha	<MDL	<MDL	<MDL	--	--
	Total Beta	1.05E+2	1.40E+0	2.58E+3	1.3%	132%

(a) Reference date is August 25, 2017.
"--" = not applicable.
<MDL = less than method detection limit.
Values in brackets have high uncertainty.

Table 3.10. AP-105DF Feed, Effluent, and Eluate Compositions (Test 1) ASR 0335, Inorganic and Carbon Analytes

Analysis Method	Analyte	TI014-FEED	TI014-EFF-Comp	TI014-ELComp	Fraction in Effluent	Fraction in Eluate
		µg/mL	µg/mL	µg/mL	%	%
ICP-MS	Hg	<8.1E-5	<8.1E-5	<8.1E-5	NA	NA
ICP-OES	Ag(a)	<1.8	<1.8	<0.3	--	--
	Al	14,550	14,500	50.4	100%	0.019%
	As	<98	<98	<16	--	--
	B	82.1	[55]	[8.8]	[67%]	[0.58%]
	Ba	[0.41]	[0.36]	[0.46]	[87%]	[6.0%]
	Ca	[41]	[51]	24.3	[123%]	3.2%
	Cd	<1.7	<1.7	<0.3	--	--
	Cr	362	362	[2.8]	100%	[0.042%]
	Fe	[4.9]	[6.25]	3.35	[128%]	3.7%
	K	3975	3920	558	99%	0.75%
	Li	<1.8	<1.8	<0.3	--	--
	Na	143,000	140,000	4770	98%	0.18%
	Ni	[30]	[33]	[1.6]	[108%]	[0.29%]
	P	[510]	[520]	10.5	[102%]	0.11%
	Pb	<25	<25	205	--	--
	Se	<141	[200]	<23	--	--
	Th	<7.4	<7.4	<1.2	--	--
	Ti	<0.9	<0.9	<0.1	--	--
	U (total)	<42	<42	<6.9	--	--
	Zn	[7.2]	[11]	8.33	[147%]	[6.3%]
	Zr	<1.3	<1.3	<0.2	100%	0.019%
KPA	U (total)	4.70	4.39	3.71	93%	4.2%
IC	Cl$^-$	4385	4355	NA	99%	--
	NO$_2^-$	63,600	61,800	NA	97%	--
	SO$_4^{2-}$	1800	1950	NA	108%	--
	C$_2$O$_4^{2-}$	195	178	NA	91%	--
	NO$_3^-$	113,500	112,500	NA	99%	--
	PO$_4^{3-}$	1018	967	NA	95%	--
Titration	Free Hydroxide	1.05 M	1.05 M	NA	100	--
Hot Persulfate Oxidation	TOC	2600	2770	NA	107%	--
	TIC	5845	5430	NA	93%	--

Table 3.10 (cont.)

Analysis Method	Analyte	TI014-FEED	TI014-EFF-Comp	TI014-ELComp	Fraction in Effluent	Fraction in Eluate
		µg/mL	µg/mL	µg/mL	%	%
ICP-OES Opportunistic Analytes	Be	[0.18]	[0.18]	[0.041]	[100%]	[1.2%]
	Bi	<34	<34	[6.5]	--	--
	Ce	<28	<28	<4.6	--	--
	Co	<4.4	<4.4	<0.7	--	--
	Cu	[4.3]	<2.3	11.2	--	[14]
	Dy	<2.1	<2.1	<0.3	--	--
	Eu	<0.6	<0.6	<0.1	--	--
	La	<1.8	<1.8	<0.3	--	--
	Mg	<1.4	<1.4	[1.4]	--	--
	Mn	<0.5	<0.5	[0.19]	--	--
	Mo	[54]	58.4	<1.0	[108]	--
	Nd	<12	<12	<1.9	--	--
	Pd	<11	<11	<1.7	--	--
	Rh	<10	<10	<1.7	--	--
	Ru	<9.8	<9.8	<1.6	--	--
	S	1390	1360	[67]	98%	[0.26%]
	Sb	<57	<57	<9.4	--	--
	Si	104	[71]	18.5	[68%]	0.96%
	Sn	[34]	<27.6	[6.1]	--	--
	Sr	<0.2	[0.23]	[0.10]	--	--
	Ta	<15	<15	<2.5	--	--
	Te	<26	<26	<4.2	--	--
	Tl	<55	<55	<9.1	--	--
	V	<2.0	<2.0	[0.33]	--	--
	W	[90]	[95]	<2.5	--	--
	Y	<0.4	<0.4	<0.1	--	--

(a) The Ag blank spike and matrix spike recoveries were 41% and 46%, respectively, indicating a low bias in Ag analysis.

Bracketed values indicate the associated sample results were less than the estimated quantitation limit but greater than the MDL. Analytical uncertainty for these analytes is >±15%.

NA = not analyzed.

“--” = not applicable.

Table 3.11. Test 2 and Test 3 Analytical Results Summary (ASR 372)

	Test 2					Test 3				
Analyte	TI015-FEED	TI015-EFFComp	TI015-ELComp	Fraction in Effluent	Fraction in Eluate	TI016-FEED	TI016-EFFComp	TI016-ELComp	Fraction in Effluent	Fraction in Eluate
Radionuclide	µCi/mL	µCi/mL	µCi/mL	%	%	µCi/mL	µCi/mL	µCi/mL	%	%
^{137}Cs	109	0.00471	1110	0.0044%	121%	129	0.0401	1510	0.031%	108%
$^{239+240}$Pu	5.82E-05	5.68E-05	<MDL	98	--	5.77E-05	5.68E-5	<MDL	99	--
^{99}Tc	1.03E-01	8.02E-02	[7.9E-06]	79%	[0.00091%]	8.92E-02	7.92E-02	7.58E-06	89%	0.00078%
Inorganic	µg/mL	µg/mL	µg/mL	%	%	µg/mL	µg/mL	µg/mL	%	%
Al	13,500	14,000	69.2	105%	0.061%	14,700	13,900	78.0	95%	0.049%
Cd	[0.85]	[1.5]	<0.17	[178%]	--	[1.5]	<0.79	<0.17	--	--
Cr	332	324	1.86	98%	0.067%	344	323	2.56	94%	0.068%
Cu	[3.0]	[2.4]	11.7	[81%]	[47%]	[2.7]	[1.6]	9.53	[59%]	32%
Fe	[5.6]	[3.6]	4.58	[65%]	[10%]	[5.9]	[3.5]	4.14	[59%]	6.4%
K	3735	3750	282	101%	0.90%	4130	3880	369	94%	0.82%
Mo	50.8	49.8	<0.51	99%	--	50.5	47.0	<0.51	93%	--
Na	136,500	132,000	3150	98%	0.28%	142,000	134,000	4110	95%	0.27%
Ni	30.1	30.2	[2.5]	101%	[1.0%]	30.3	28.3	4.35	94%	1.3%
Pb	[17]	[8.9]	87.6	54%	63%	[20]	<7.0	111	--	[51%]
S	1130	1090	[27]	97%	[0.29%]	1080	1020	[35]	95%	[0.30%]
Si	125	133	739	107%	71%	103	120	625	117%	56%
Sr	[0.081]	[0.11]	0.211	[137%]	[31%]	[0.080]	[0.056]	0.414	[70%]	[0.30%]
Zn	[3.85]	[5.1]	17.6	[134%]	[55%]	<1.1	<1.1	10.2	--	--
U ICP-MS	6.03	5.51	1.65	92%	3.3%	6.29	5.71	2.47	91%	3.6%

"--" = not applicable.

<MDL = less than method detection limit.

Bracketed values indicate the associated sample results were less than the estimated quantitation limit but greater than the MDL. Analytical uncertainty for these analytes is >±15%.

Table 3.12. Test 4 Take 1 and Test 4 Take 2 Analytical Results Summary (ASR 372)

	Test 4 Take 1					Test 4 Take 2				
Analyte	TI020-FEED-1-A	TI020-EFFComp	TI020-ELComp	Fraction in Effluent	Fraction in Eluate	TI020-FEED-Take 2	TI020-EFF Comp-Take 2	TI020-EComp	Fraction in Effluent	Fraction in Eluate
Radionuclide	µCi/mL	µCi/mL	µCi/mL	%	%	µCi/mL	µCi/mL	µCi/mL	%	%
^{137}Cs	111	0.00352	260	0.0031%	106%	130	0.0466	1060	0.035%	99%
$^{239+240}$Pu	7.39E-05	4.39E-05	<MDL	57%	--	5.94E-05	5.18E-05	<MDL	86%	--
^{99}Tc	0.0862	0.0767	<MDL	86%	--	0.0832	0.0772	[5.2E-6]	92%	[0.00075%]
Inorganic	µg/mL	µg/mL	µg/mL	%	%	µg/mL	µg/mL	µg/mL	%	%
Al	13,300	13,200	93.5	96%	0.32%	14,000	14,200	49.8	100%	0.043%
Cd	[1.3]	[1.1]	<0.17	[82%]	--	[1.3]	[1.3]	<0.17	[99%]	--
Cr	337	301	2.63	86%	0.35%	341	334	2.50	97%	0.089%
Cu	[2.9]	<1.1	5.32	--	83%	[2.4]	[1.2]	11.3	[49%]	[57%]
Fe	[4.1]	[15]	[3.4]	[353%]	[37%]	[8.1]	[5.4]	4.22	[66%]	[6.3%]
K	3930	3460	289	85%	3.3%	4020	3950	268	97%	0.81%
Mo	48.8	43.9	<0.51	87%	--	48.5	49.4	<0.51	101%	--
Na	140,000	128,000	3150	88%	1.0%	140,000	139,000	3020	98%	0.26%
Ni	30.5	24.8	5.86	78%	8.7%	29.8	29.0	5.48	96%	2.2%
Pb	[15]	<7.0	29.9	--	[90%]	[15]	<7.0	95.6	--	[77%]
S	1060	924	[23]	84%	[1.0%]	1050	1040	[20]	98%	[0.23%]
Si	127	152	546	115%	194%	145	172	373	117%	31%
Sr	[0.096]	[0.049]	0.148	[49%]	[70%]	[0.074]	[0.12]	0.115	[160%]	[19%]
Zn	<1.1	<1.1	13.7	--	--	[2.0]	<1.1	10.4	--	[63%]
U ICP-MS	5.87	4.54	1.46	75%	11%	6.08	5.52	2.22	90%	4.4%

"--" = not applicable.

<MDL = less than method detection limit.

Bracketed values indicate the associated sample results were less than the estimated quantitation limit but greater than the MDL. Analytical uncertainty for these analytes is >±15%.

Table 3.13. Test 5 Analytical Results Summary (ASR 372)

	Test 5				
Analyte	TI021-FEED	TI021-EFFComp	TI021-EComp	Fraction in Effluent	Fraction in Eluate
Radionuclide	µCi/mL	µCi/mL	µCi/mL	%	%
^{137}Cs	127	0.0052	860	0.0041%	89%
$^{239+240}$Pu	8.01E-05	6.42E-05	<MDL	76%	--
^{99}Tc	0.0862	7.88E-02	7.87E-06	86%	0.0012%
Inorganic	µg/mL	µg/mL	µg/mL	%	%
Al	14,900	14,650	75.6	97%	0.067%
Cd	[1.1]	[0.95]	<0.17	[85%]	--
Cr	341	334	3.09	97%	0.12%
Cu	[2.4]	[1.4]	12.5	[58%]	69%
Fe	[3.4]	[2.8]	4.89	[81%]	19%
K	4120	4010	287	96%	0.92%
Mo	49.9	48.4	<0.51	96%	--
Na	142,000	139,000	3500	97%	0.33%
Ni	31.1	28.8	6.93	91%	2.94%
Pb	[16]	<7.08	89.1	--	[73%]
S	1050	1024	[22]	96%	[0.28%]
Si	94.8	77.5	718	81%	100%
Sr	[0.082]	[0.106]	0.220	[127%]	35%
Zn	<1.12	<1.96	13.1	--	--
U ICP-MS	5.80	5.64	2.28	96%	5.2%

"--" = not applicable.
<MDL = less than method detection limit.
Bracketed values indicate the associated sample results were less than the estimated quantitation limit but greater than the MDL. Analytical uncertainty for these analytes is >±15%.

In Test 1, the Pu partitioned mostly to the effluent (94%), with 2% to the eluate and possibly 4% remaining on the ion exchange column. However, analysis uncertainty for $^{239/240}$Pu was approximately 4% (1-sigma),[1] and the possibility that all Pu could be accounted for in the effluent and eluate cannot be discounted. In subsequent tests, the Pu recovery in the effluent varied from 99% (Test 3) to 57% (Test 4 Take 1). The Test 4 Take 1 test only partially loaded the SRF with Cs because it was a short run. It is possible that Pu has some affinity for the SRF and as more Cs exchanges onto the resin, it displaces the Pu into the effluent. More studies would be required to assess this possibility. The Pu recovery in the later cycles (Test 4 Take 2 and Test 5) demonstrated reduced Pu recovery (86% and 76%, respectively) in the effluent relative to Tests 1, 2, and 3 (94%, 98%, and 99% respectively). The Pu was not detected in the eluate (Tests 2-5), indicating that some fraction of Pu may be accumulating onto the SRF from one cycle to the next.

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[1] All counting uncertainty was reported as 1-sigma.

The ^{237}Np (Test 1) results were similar to the Pu results where 92% of the loaded ^{237}Np was accounted for in the effluent; ^{237}Np was not detected in the eluate. The $^{243+244}$Cm chemistry behavior normally follows that of ^{241}Am ; therefore, the low $^{243+244}$Cm recovery (66%) in the Test 1 effluent is at odds with the quantitative recovery for ^{241}Am (101%) in the Test 1 effluent. Analytical uncertainties for ^{241}Am and $^{243+244}$Cm were 2% and 8%, respectively; these uncertainties were well below the missing 34% $^{243+244}$Cm. Virtually all ^{90}Sr reported to the effluent (analysis uncertainty was 2%). Nominally 78% of the ^{99}Tc was recovered in the effluent product; <0.1% was found in the eluate. This indicates that the ^{99}Tc may have reacted with the SRF resin and was retained. Analysis of the spent resin is intended to be conducted in the future.

The ^{99}Tc recovered similarly in all test cycles at about 79% to 92% with no up or down trend. A small amount of ^{99}Tc ($\leq$0.013%) was recovered in the eluate. Thus, the remaining ^{99}Tc mass balance could remain on the column.

The ICP-OES results for metals showed that virtually all analytes reported to the effluent. In some cases, the analyte percent recovery could not be calculated because the analyte was not detected in the feed, the effluent, and/or the eluate. Three transition metals (Fe, Cu, Zn), Pb, and U (U as measured by KPA or ICP-MS) recovered at >3% in the eluate composite. In the case of Test 1, Pb recovery could not be estimated because Pb was not detected in the feed. Over 50% of the Pb was detected in the eluate from Tests 2-5. These results indicate that a high proportion of Pb and some Fe, Cu, Zn, and U exchanged from the feed onto the ion exchanger during the loading phase and were eluted at least to some extent with 0.45 M HNO_3.

Nominally 100% of all anions were accounted for in the effluent. TIC is generally ascribed to carbonate and thus is ascribed as an anion. TOC includes oxalate and other organic carbon forms, usually complexants. The oxalate measured by IC, 2.2 E-3 M, was a small fraction of the TOC (0.22 M as C). The TOC recovery in the effluent was quantitative at 107%.

4.0 Discussion

This section summarizes processing trends associated with the number of cycles, flowrates, and elution parameters.

4.1 Cs Load Capacity

The Cs load capacity was calculated from the total Cs loaded onto the lead column, which was assumed to be fully saturated under the load conditions, and the dry H-form resin mass loaded into the lead column according to Eq. (4.1):

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

where A_{Cs} = total activity of ^{137}Cs, µCi loaded onto the lead column

CF = conversion factor, 6.6E-5 mg Cs/µCi ^{137}Cs (based the nominal 7.9 µg Cs/mL Cs and 120 µCi ^{137}Cs/mL in the AP-105DF)

M_R = mass of dry, H-form resin, 2.45 g

C = capacity, mg Cs/g H-form resin

Table 4.1 provides the Cs capacity found on the lead column with each process test. It is clear that the capacity decreased with each process cycle. After the six load/elute cycles, the Cs capacity dropped 17% (~2.8% per process cycle). It is noted that the Cs capacity change between Test 3 and Test 4 Take 2 was minimal. A short load cycle, Test 4 Take 1, was conducted between these two runs with minimal additional Cs loading and concomitant less chemical and dose exposure from AP-105DF loading. Therefore, these sources of resin degradation (chemical and radiological) would be substantially reduced for the Test 4 Take 1 process cycle. Previous testing with SRF in simulant using the WTP-designated process conditions showed a reduction of 7% Cs capacity after processing 16 full load/elute cycles (Fiskum et al. 2006a). This corresponds to a Cs capacity reduction of ~0.4% per process cycle. The observed increased rate of SRF degradation may be attributed to the LAWPS-designated process conditions (more Cs loading) and concomitant MRad radiolytic exposure.

Table 4.1. Cesium Capacity as a Function of Process Cycle

Test Number	Cs Capacity, mg Cs/g H-form Resin
1	6.64
2	6.38
3	6.09
4 Take 1	NA[(a)]
4 Take 2	6.06
5	5.53

(a) This test did not fully load the lead column.

4.2 Column Performance

The column performance parameters can be further evaluated in terms of the following:

a. 50% breakthrough
b. Mass transfer zone
c. Leakage of Cs from the lag column to the product

A reduction in the 50% Cs breakthrough point indicates a reduction in the Cs load capacity of the resin bed. Increasing the mass transfer zone reduces the feed volume that can ultimately be processed before reaching the effluent contract limit. Leakage of the Cs into the effluent product will affect whether or not the product will meet the contract specification or decrease the volume that can be processed before reaching the contract specification.

4.2.1 50% Breakthrough

Table 4.2 shows the lead column Cs load values for all tests. It is clear that the 50% breakthrough was similar for Tests 1 and 2. The 50% breakthrough point decreased moderately (8%) between Tests 2 and 3. The subsequent Test 4 Take 2 dropped an additional 4% and was equivalent to Test 5. The 50% breakthrough is expected to be independent of flowrate. The decrease in the 50% breakthrough is attributed to resin degradation from chemical, radiolytic, and mechanical means.

Table 4.2. 50% Breakthrough as a Function of Process Cycle

Test Number	50% Cs Breakthrough, BV	% Difference from Test 1
1	206	0.00%
2	205	0.49%
3	189	8.3%
4 Take 1	NA[(a)]	
4 Take 2	182	12%
5	181	12%

(a) This test did not fully load the lead column.

4.2.2 Mass Transfer Zone

Figure 4.1 shows how the Cs breakthrough curve changes with increasing flowrate on the lead column (Tests 1-3). Test 1 processed at the slowest flowrate; it showed the least Cs leakage early in the process run because the prior run processed feed at nominally 0.1 µC/mL ^{137}Cs (Fiskum et al. 2017). Examining the load curve from about 0.05% to 50% breakthrough shows that increasing flowrate decreases the breakthrough slope and thus increases the mass transfer zone.

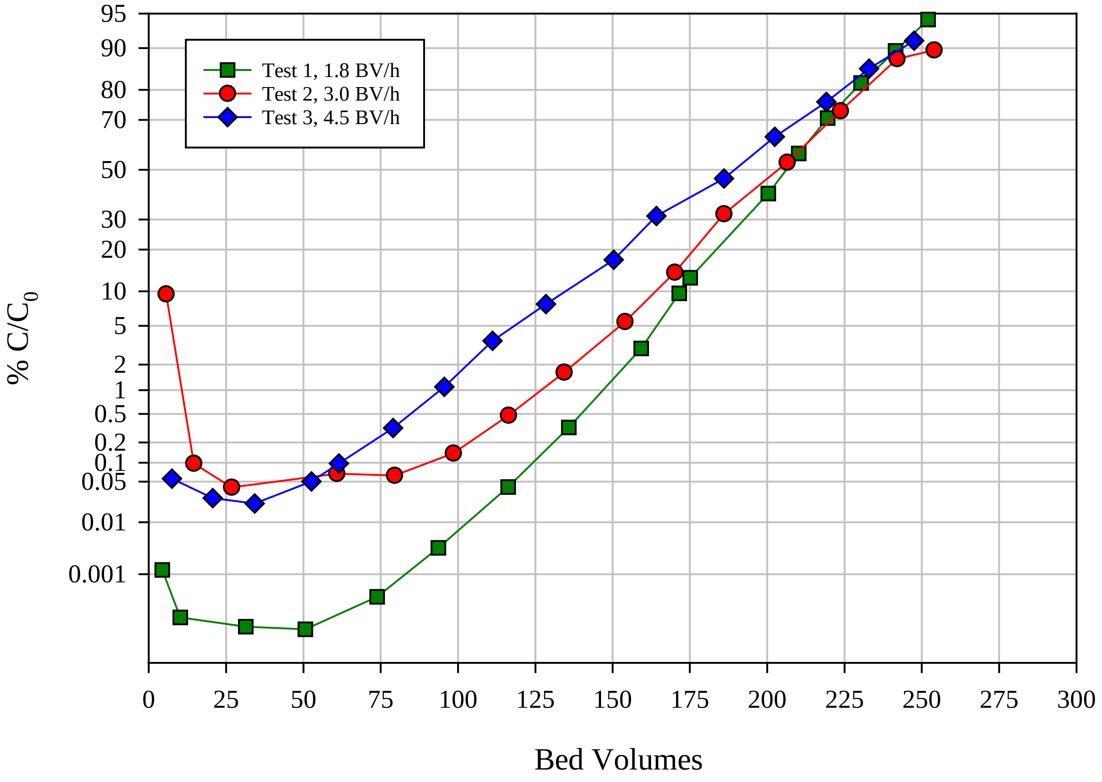

Figure 4.1. Cesium Breakthrough Curves as a Function of Flowrate on SRF Resin

The mass transfer zone was evaluated between 1% and 90% C/C_0 for all tests (see Table 4.3). The 90% breakthrough point remained fairly constant (4% standard deviation) for all tests regardless of the flowrate and process cycle. The 1% breakthrough point varied significantly, occurring sooner with increasing flowrate as can be discerned by examination of Figure 4.1. At constant flowrate (~3.0 BV/h), the mass transfer zone had a narrow range from 112 to 130 BVs. Figure 4.2 compares the breakthrough profiles for tests processed at ~3 BV/h (Test 2, Test 4 Take 2, and Test 5). Profiles from Tests 4 and 5 are shifted left relative to Test 2, indicative of the decreased Cs loading capacity.

Table 4.3. Mass Transfer Zone (1% to 90% C/C_0)

Test Number	Flowrate, BV/h	1% Breakthrough, BVs	90% Breakthrough, BVs	Transition Zone, BVs 1% to 90%
1	1.80	147	242	95
2	3.05	127	251	124
3	4.53	95	244	144
4 Take 1	3.04	NA(a)	NA(a)	NA(a)
4 Take 2	2.95	114	226	112
5	3.17	102	~230(b)	130

(a) This test did not fully load the lead column; only 53 BVs of AP-105DF were processed.
(b) Extrapolated from 83.1% breakthrough.

Increasing flowrate while maintaining residence time (i.e., constant BV/h) was shown to sharpen the mass transfer zone by overcoming film diffusion limitations (Fiskum et al. 2017). The increased flowrate in these tests was not sufficient to overcome the film diffusion limitation.

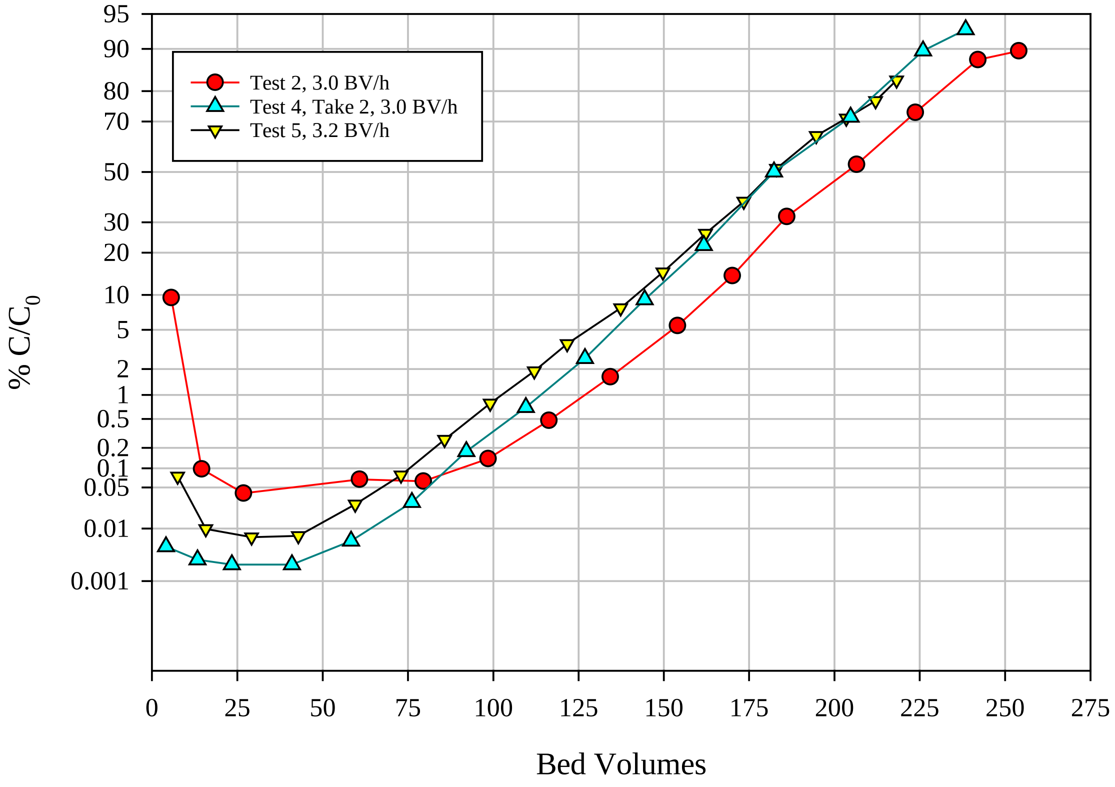

Figure 4.2. Lead Column Cesium Breakthrough Profiles at Constant Flowrate

4.2.3 Cs Leakage

Cesium leakage from the completion of one process cycle to the next process cycle was evaluated. Cs leakage stems from residual Cs on the lag column SRF resin exchanging into the product effluent and contaminating the product. Cs leakage may interfere with the ability to process large volumes of waste and maintain the effluent Cs concentration below the 10% contract limit.

Figure 4.3 shows all lag column load curves. Prior to Test 1 with AP-105DF, one cycle had been processed with simulant spiked with 60 µg/mL Cs and 0.1 µCi/mL ^{137}Cs (Fiskum et al. 2017). The lag column effluent ^{137}Cs concentration was proportionately low, resulting in low initial C/C_0 values. The Test 2 lag column effluent was the highest of the group; it followed the shortest elution (16 BVs) from Test 1. Test 3, Test 4 Take 1, and Test 5 had similar lag column Cs effluent concentrations following previous cycle elution of 30, 23, and 30 BVs, respectively. Test 4 Take 2 Cs effluent concentration started markedly lower than the others; this is attributed to the low total Cs loading associated with Test 4 Take 1, which gave the sparingly loaded lag column a chance to cycle (Na-form to H-form) once before the next load. The extra cycle is believed to remove more residual Cs from the resin bed.

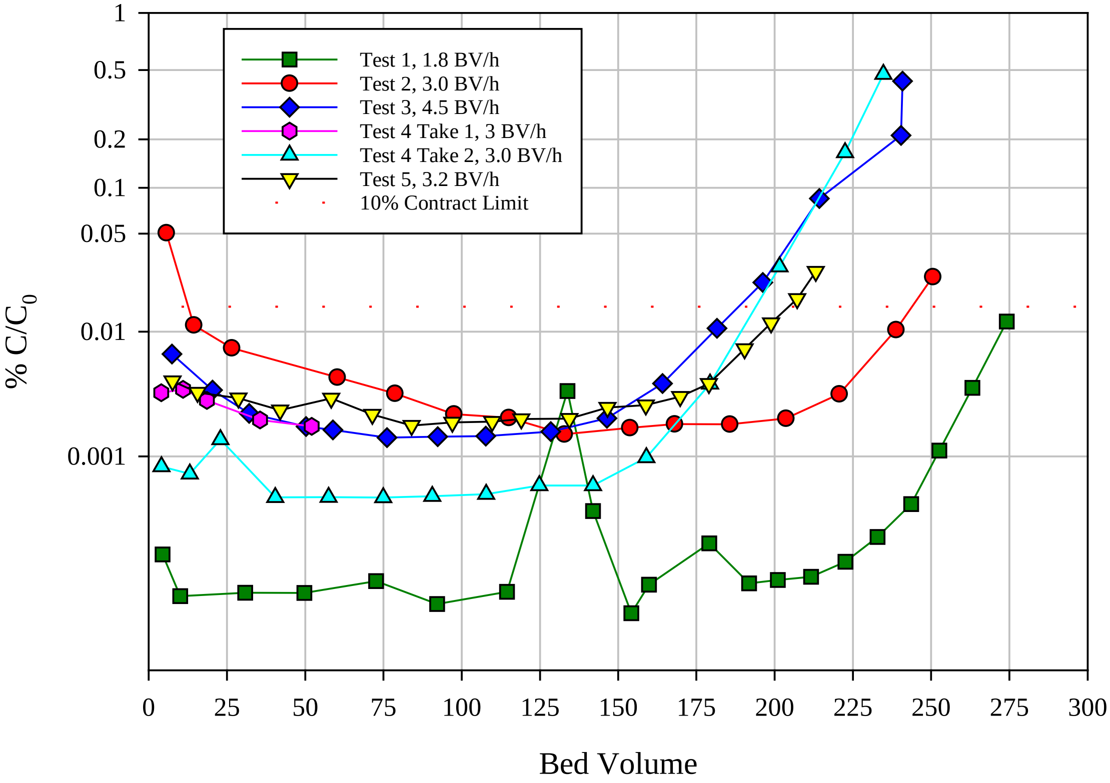

Figure 4.3. Comparison of Lag Column Cs Breakthrough

The elution with 0.45 M HNO_3 varied for the different process cycles from 16 to 30 BVs. The water rinse volumes following elution were relatively constant. The relationship of the Cs concentration in the final water rinse sample following elution was evaluated relative to the following process run Cs leakage from the lag column. Table 4.4 summarizes these salient attributes. The final water rinse Cs concentration decreases approximately linearly as a function of the BVs (range 15 to 30 BVs) of eluent processed (Figure 4.4).

It is noted that the onset of Cs breakthrough from the lag column did not influence appreciably where the contract limit was crossed later in the load cycle. Increasing flowrate resulted in earlier Cs breakthrough (Tests 1-3) as expected; increasing cycles between Tests 2 and 5 resulted in a reduction of 43 BVs to reach the 10% contract limit.

The relationship between the lag column initial Cs breakthrough with respect to elution volume and water rinsing was evaluated. Table 4.4 summarizes the initial feed sample Cs % C/C_0 from the lag column for each test along with the elution volume, water rinse volume, and final 15-mL water rinse sample Cs $\%C/C_0$. The extended water rinse was not included because the Cs concentration was not evaluated for the last segment of this rinse. The final water rinse sample Cs concentration was clearly a function of the BVs processed, as shown in Figure 4.4, where increasing the elution BVs resulted in a decreased Cs $\%C/C_0$ in the final water rinse sample.

Table 4.4. Lag Column Initial Cs Leakage

Test ID	Initial Lag Column Sample Cs Conc., % C/C_0	Elution, BVs	Water Rinse, BVs	Final Water Rinse Cs Conc., % C/C_0
Test 0[(a)]	<1.85E-4	16.4	5.3	4.73E-1
Test 1	1.34E-4	15.9	5.8	3.84E-1
Test 2	5.06E-2	29.9	5.6	6.81E-2
Test 3	6.77E-3	23.0	6.3	2.01E-1
Test 4a	3.35E-3	24.9	NA	NA
Test 4b	8.20E-4	29.5	8.1	7.96E-2
Test 5	4.17E-3	29.1	7.9	9.61E-2

(a) The initial test was conducted with simulant spiked to 60 µg/mL Cs and 0.1 µCi/mL ^{137}Cs (Fiskum et al. 2017). This was the virgin run for the lag column and no effluent Cs in the initial sample was expected.

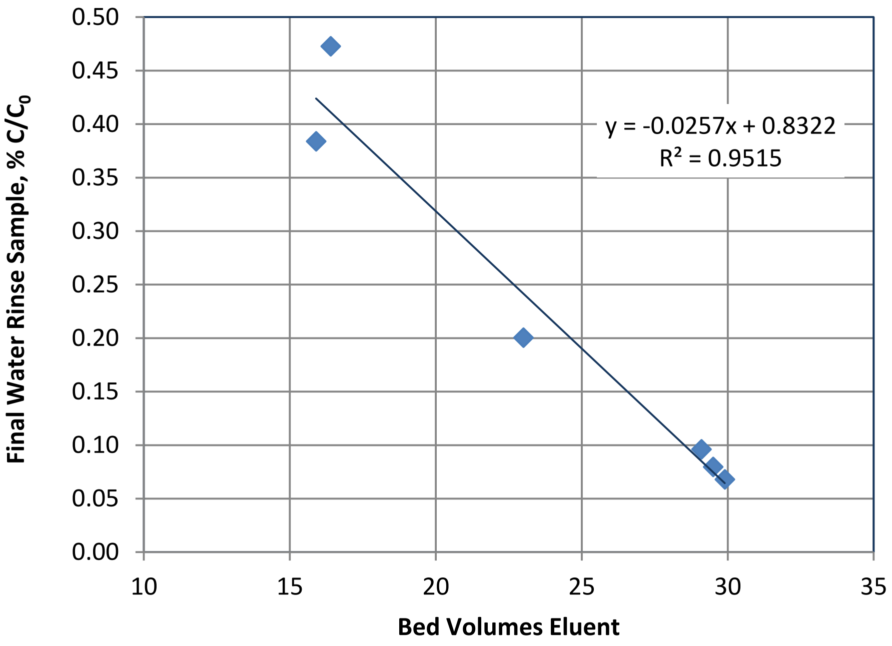

Figure 4.4. Final Water Rinse Sample Cs Concentration as a Function of the Elution BVs Processed, Tests 0 to 5

The relationship between effluent Cs coming off the lag column and eluent BVs processed in the previous ion exchange run was evaluated. Figure 4.5 shows the relationship of the initial lag column Cs effluent concentration (%C/C_0) as a function of the elution volume in the previous process cycle. It was clear that the 16 BV elution was insufficient to reduce the effluent product in the next process cycle enough to meet the 10% contract limit. Other efforts to equate Cs leakage into the subsequent process cycle were confounded by the changing elution conditions and changing Cs loading on the lag column.

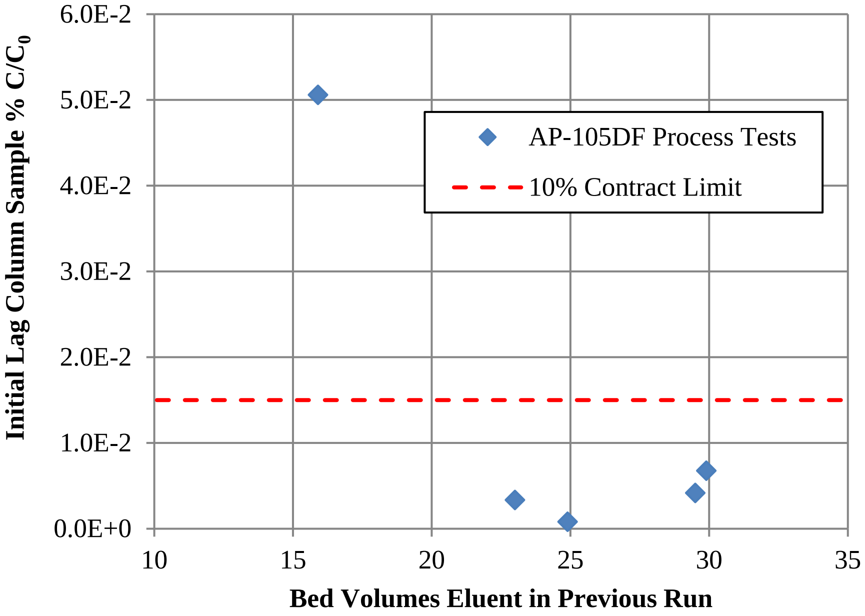

Figure 4.5. Initial Cs Leakage versus Eluent BVs Processed in Previous Cycle

5.0 Resin Bed Physical Properties

The ion exchange resin bed contracts as it converts to H-form and expands when it converts to Na-form. Figure 5.1 shows the in-column contraction/expansion history starting with the in-column pretreatment operations (Fiskum et al. 2017) and continuing through the final water rinse following the Test 5 AP-105DF processing cycle. Typical 20% shrink/swell behavior was observed. The lead column appeared to increase in volume approximately 10% from the first input volume (9.9-mL Na-form) to the final volume (10.9-mL Na-form), indicating that the resin beads were likely relaxing and expanding more with the repeated process cycles. The lag column appeared to shrink and swell differently from the lead column. Starting at the Test 4 Take 1 process cycle, the resin volume increased 20% (Na-form) relative to the initial packed column. Further, the lag column Na-form resin bed was approximately 10% larger in volume relative to the lead column resin bed. It is not clear what drove the difference in shrink/swell behavior between the two column runs. The gap observed in the lag column during elution in Test 4 Take 1 was not observed later during processing in Test 4 Take 2 and Test 5.

Oxygen is known to attack the SRF resin (Fiskum et al. 2006a). The AP-105DF feed is not expected to contain much dissolved oxygen due to its high salt content. The feed displacement (0.1 M NaOH), water, regeneration solution (1.0 M NaOH), and eluent (0.45 M HNO_3) are anticipated to contain significantly higher dissolved oxygen concentrations. Dependent on the process step, both the lead and lag columns receive first reagent exposure of dissolved oxygen. The divergent swell behavior of the lag column seemed to start with the observed gap in the resin bed. But later cycles did not show this gap and the lag column swelling was much larger than the lead column swelling. The repeated cycling may be irreversibly leading to expanded polystyrene bead.

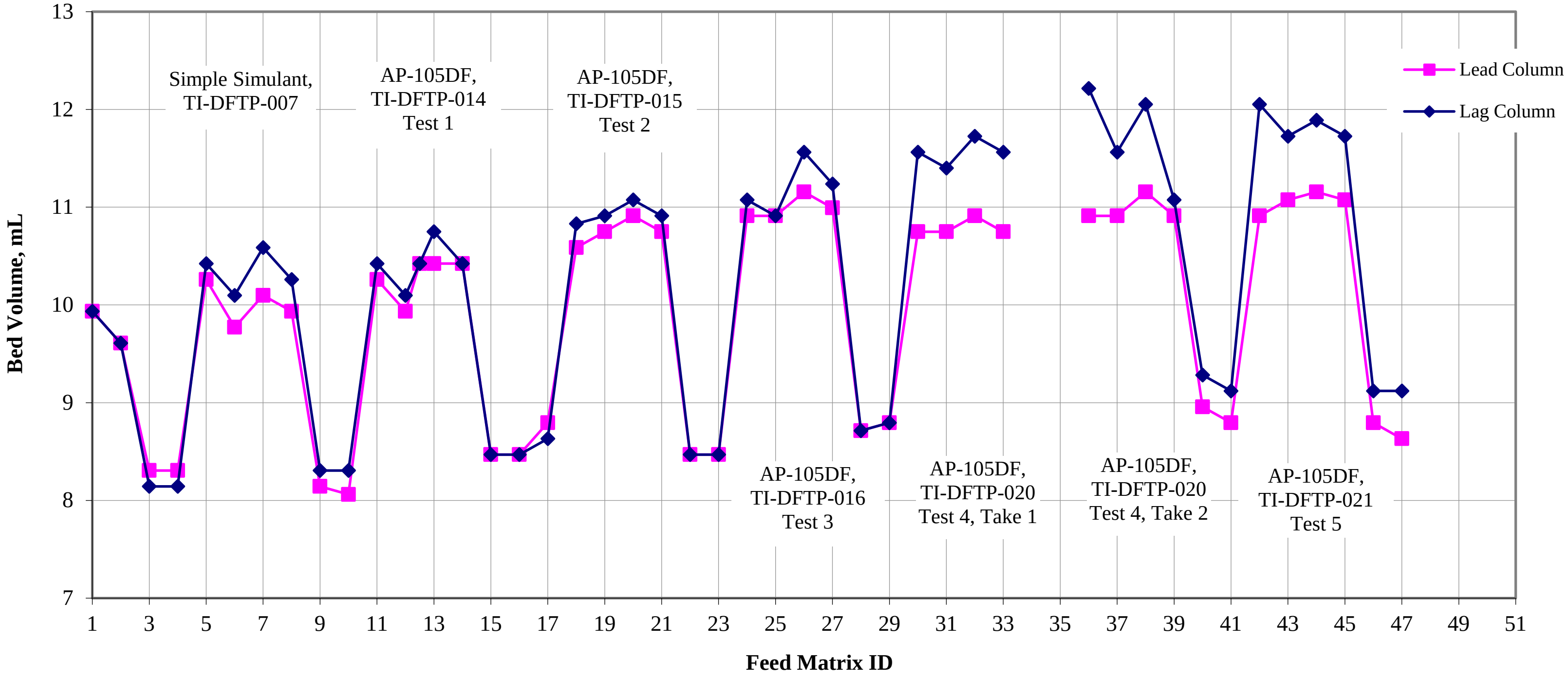

Figure 5.1. Resin Bed Expansion and Contraction History

Feed ID	Matrix	Feed ID	Matrix	Feed ID	Matrix	Feed ID	Matrix	Feed ID	Matrix
1	1.0 M NaOH soak	11	1.0 M NaOH	20	0.1 M NaOH FD	30	1.0 M NaOH	40	0.45 M HNO_3
2	DI water	12	AP-105DF Test 1	21	DI water	31	AP-105DF Test 4 Take 1	41	DI water
3	0.45 M HNO_3	12.5	AP-105DF Test 1 end	22	0.45 M HNO_3	32	0.1 M NaOH FD	42	1.0 M NaOH
4	DI water	13	0.1 M NaOH FD	23	DI water	33	DI water	43	AP-105DF Test 5
5	1.0 M NaOH	14	DI water	24	1.0 M NaOH	34	0.45 M HNO_3	44	0.1 M NaOH FD
6	Simple Simulant	15	0.45 M HNO_3	25	AP-105DF Test 3	35	DI water	45	DI water
7	0.1 M NaOH FD	16	DI water	26	0.1 M NaOH FD	36	1.0 M NaOH	46	0.45 M HNO_3
8	DI water	17	DI water	27	DI water	37	AP-105DF Test 4 Take 2	47	DI water
9	0.45 M HNO_3	18	1.0 M NaOH	28	0.45 M HNO_3	38	0.1 M NaOH FD		
10	DI water	19	AP-105DF Test 2	29	DI water	39	DI water		

6.0 Multi-Cycle Testing with AP-105DF Conclusions

A radioactive waste feed Test Platform was constructed at PNNL to support tank waste filtration[1] and Cs removal by ion exchange to meet near-term waste pretreatment for delivery of feed to the WTP LAW facility. 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 9.9 mL of SRF resin (Na-form). At the time of testing, process parameters were prototypic of the expected LAWPS process with respect to the ion exchange media. The goal was to process feed until the ^{137}Cs concentration in the effluent reached 10% of the LAW contract limit. To this end, effluent samples were collected periodically during the load process and measured for ^{137}Cs and the load curve was developed. The combined effect of Cs leakage and the delay between sampling and analysis resulted in the processing past the 10% LAW contract limit in three of the five cycles. The processing parameters resulted in fully loading the lead column and having a significant fraction of Cs loaded onto the lag column. Regeneration solutions consisted of FD (0.1 M NaOH), water rinse, eluent (0.45 M HNO_3), and post-elution water rinse. The flowrates in terms of BV/h were also matched to the LAWPS process for the first test, but because of the small column size, 9.9-mL lab-scale testing versus 297-gallon full-scale, linear flow velocities were fundamentally different.[2] The results are comparable in that the small column testing provides a worst-case bounding load profile. The higher linear flow rate with constant residence time sharpens the Cs load curve. However, Cs leakage to the next process run is not likely to improve.

A total of 12.3 L of AP-105DF tank waste, consisting of 5.7 M Na and 120 µCi/mL ^{137}Cs, was processed through the Cs ion exchange system in six process cycles. This processing exposed the SRF resin to chemical exposure (process solution and residence time in the resin bed) and physical changes (shrink/swell) similar to what would be experienced at the WRPS LAWPS (and to some extent, the WTP). Radiolytic exposure of the small column SRF test was about 1/100th of the resin lifetime exposure expected at full scale plant conditions and about a third of the exposure expected on a single process cycle at full scale plant conditions. Feed flowrates and elution volumes were modified slightly from one test to the next to evaluate effects on the Cs load profiles. Flowrates ranged from 1.8 to 4.5 BV/h and elution flowrates ranged from 1.4 to 2.7 BV/h. The following conclusions were made as a result of this work.

1. A quantity of 275 BVs of AP-105DF was processed at 1.8 BV/h through the first cycle before reaching the 10% contract limit. As flowrate increased to 4.5 BV/h, the volume processed before the limit was reached decreased to 190 BVs. Subsequent processing at ~3.0 BV/h maintained the ~200 BVs processed before reaching the 10% contract limit.

2. The lead column Cs capacity decreased from 6.64 (first process cycle) to 5.53 (sixth process cycle) mg Cs per g dry H-form resin, indicating ~2.8 % capacity loss per cycle. The Cs capacity decrease was attributed to combined chemical, physical, and radiolytic degradation. This degradation rate exceeded previously reported degradation of 7% over 16 process cycles (0.44% per cycle) from chemical and physical exposure.

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[1] The filtration component was reported by Geeting et al. (2017).

[2] Increasing linear flow velocity enhances film diffusion and thus ion exchange onto the SRF resin. Small columns with lower linear flowrates have poorer film diffusion.

3. The Cs mass transfer zone increased with increasing flowrate, ranging from 95 BVs at 1.8 BV/h to 144 BVs at 4.5 BV/h. The increased mass transfer zone was attributed to the reduction in residence/equilibration time and the increased flowrate did not overcome limitations due to film diffusion (Fiskum et al. 2017).

4. The 50% Cs breakthrough generally shifted left with successive cycles, ranging from 206 BVs (first process cycle) to 181 BVs (sixth process cycle), commensurate with the decreased capacity associated with resin degradation (Fiskum et al 2006a).

5. Within analytical uncertainty, typically >99% of the Cs processed through the ion exchange system was collected in the eluate. However, trace Cs remained on the resin beds.

6. After an initial loading with AP-105DF, Cs leakage from the lag column to the effluent was a significant factor in the system's ability to satisfy the effluent decontamination target. Although virtually all Cs was accounted for in the eluate, a very small Cs fraction remained on the lag column and leaked into the next cycle product. Approximately 30 BVs of 0.45 M HNO_3 eluent were required to meet the target DF in the next process cycle.

7. Small fractions (3% to 15%) of Fe, Cu, U, and Zn were found in the eluate; an indeterminate fraction of Pb was found in the eluate. This indicates that some fraction of these analytes exchanged onto the resin and was eluted with the acid. Less than 1% of detectable metals were found in the eluate. All other metals and anions were found with the effluent product.

8. Radionuclides (^{90}Sr, ^{237}Np, $^{239+240}$Pu, and ^{241}Am) were largely recovered in the effluent. About 2% of the Pu was found in the eluate and 4% was not accounted for. Only 66% of the $^{243+244}$Cm and 78% of the ^{99}Tc were recovered in in the effluent product. Less than 0.1% of the ^{99}Tc was recovered in the eluate, indicating that ~20% remained on the SRF. Only the first process cycle eluate was measured for $^{243+244}$Cm, where 3.1% was recovered.

9. The SRF resin continued to expand from one cycle to the next, increasing in volume by an additional 10% to 20% after six process cycles.

10. The SRF resin in two 10-mL beds maintained the Cs exchange efficacy after processing 12.3 L of AP-105DF in six cycles with reasonable efficiency despite the synergistic resin degradation effects from the combined radiological dose and chemical and physical (shrink/swell) exposures. These results support test observations and conclusions previously described in SRF literature reviews (King 2007; Brown 2014).

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Appendix A

Column Load and Elution Data

Appendix A

Column Load and Elution Data

The AP-105DF column loading and elution raw data for Tests 1-5 are provided in Table A.1 through Table A.12. The Test 4 cycles also include the regeneration sample data following the elution step.

Table A.1. AP-105DF 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
4.4	1.36E-3	1.22E-3	82,238	4.4	1.50E-4	1.34E-4	743,720	FD			
10.2	1.30E-4	1.17E-4	856,213	10.0	6.05E-5	5.43E-5	1,843,199	275.3	1.71E-2	1.54E-2	6507
31.4	8.05E-5	7.21E-5	1,386,420	30.8	6.53E-5	5.86E-5	1,707,607	276.4	3.03E-2	2.72E-2	3682
50.6	7.04E-5	6.32E-5	1,583,472	49.7	6.50E-5	5.83E-5	1,716,546	277.6	3.74E-2	3.35E-2	2985
73.9	3.71E-4	3.33E-4	300,640	72.6	8.41E-5	7.54E-5	1,325,798	278.7	5.55E-2	4.97E-2	2010
93.6	3.72E-3	3.34E-3	29,978	92.1	5.09E-5	4.56E-5	2,192,986	279.7	4.91E-2	4.40E-2	2273
116.2	4.56E-2	4.09E-2	2446	114.5	6.65E-5	5.96E-5	1,676,868	280.8	7.95E-3	7.12E-3	14,040
135.8	3.65E-1	3.28E-1	305	133.8	3.85E-3	3.45E-3	28,946	DI rinse			
159.3	3.32E+0	2.98	34	141.9	3.72E-4	3.34E-4	299,567	281.9	2.77E-3	2.48E-3	40,332
171.6	1.07E+1	9.63	10	154.2	4.13E-5	3.70E-5	2,702,566	283.0	2.04E-3	1.83E-3	54,722
175.1	1.42E+1	12.73	7.9	159.9	7.80E-5	7.00E-5	1,429,522	284.0	1.44E-3	1.29E-3	77,221
200.4	4.47E+1	40.07	2.5	179.2	1.90E-4	1.70E-4	587,351	285.0	1.15E-3	1.03E-3	97,250
210.2	6.34E+1	56.8	1.8	191.9	8.02E-5	7.19E-5	1,390,128				
219.6	7.89E+1	70.7	1.41	201.0	8.63E-5	7.73E-5	1,293,150				
230.4	9.14E+1	82.0	1.22	211.7	9.25E-5	8.30E-5	1,205,396				
241.5	9.98E+1	89.5	1.12	222.6	1.28E-4	1.15E-4	870,556				
252.1	1.05E+2	94.3	1.06	232.9	2.17E-4	1.95E-4	513,854				
262.9	1.12E+2	100	1.00	243.6	4.30E-4	3.85E-4	259,499				
272.1	1.17E+2	105	0.95	252.6	1.25E-3	1.12E-3	89,109				
282.9	1.20E+2	107	0.93	263.2	4.11E-3	3.68E-3	27,169				
294.0	1.19E+2	107	0.93	274.2	1.33E-2	1.19E-2	8382				

BV = bed volume; DI = deionized; DF = decontamination factor; FD = feed displacement; C$_0$ = 112 µCi ^{137}Cs/ mL.

Table A.2. Elution and Water Rinse Results Following AP-105DF Processing Test 1

Elution			Water Rinse		
BV	µCi ^{137}Cs/ mL	C/C_0	BV	µCi ^{137}Cs/ mL	C/C_0
1.42	2.84E+0	2.54E-2	17.42	2.43E+0	2.18E-2
2.87	2.65E+0	2.37E-2	18.84	9.56E-1	8.57E-3
4.40	3.55E+0	3.18E-2	20.23	5.85E-1	5.25E-3
5.89	1.03E+1	9.21E-2	21.73	4.28E-1	3.84E-3
7.35	1.12E+1	1.01E-1			
8.77	1.33E+1	1.20E-1			
10.21	2.83E+3	2.54E+1			
11.64	1.92E+4	1.72E+2			
13.08	2.56E+3	2.29E+1			
14.53	5.84E+1	5.24E-1			
15.94	1.29E+1	1.16E-1			

BV = bed volume; C_0 = 112 µCi ^{137}Cs/ mL.

Table A.3. AP-105DF Test 2 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
5.6	1.12E+1	9.49E+0	11	5.6	5.98E-2	5.06E-2	1977	FD			
14.5	1.15E-1	9.71E-2	1030	14.4	1.32E-2	1.12E-2	8934	252.1	2.94E-2	2.49E-2	4015
26.8	4.78E-2	4.04E-2	2474	26.5	8.89E-3	7.52E-3	13,300	253.7	3.23E-2	2.73E-2	3663
60.8	7.98E-2	6.75E-2	1481	60.2	5.26E-3	4.45E-3	22,456	255.4	4.34E-2	3.67E-2	2722
79.5	7.43E-2	6.29E-2	1591	78.6	3.90E-3	3.30E-3	30,288	256.9	1.63E-2	1.38E-2	7242
98.5	1.65E-1	1.39E-1	717	97.5	2.66E-3	2.25E-3	44,412	DI rinse			
116.3	5.65E-1	4.78E-1	209	115.0	2.49E-3	2.11E-3	47,476	258.5	5.00E-3	4.23E-3	23,666
134.3	1.93E+0	1.63E+0	61	132.7	1.82E-3	1.54E-3	65,121	260.0	3.07E-3	2.60E-3	38,505
154.0	6.45E+0	5.5	18	153.8	2.05E-3	1.73E-3	57,660	261.7	2.36E-3	1.99E-3	50,196
170.1	1.65E+1	14.0	7	168.0	2.20E-3	1.86E-3	53,620				
186.0	3.79E+1	32.0	3.1	185.7	2.20E-3	1.86E-3	53,784				
206.5	6.28E+1	53.1	1.9	203.6	2.45E-3	2.07E-3	48,204				
223.7	8.65E+1	73.2	1.4	220.7	3.87E-3	3.27E-3	30,549				
242.0	1.04E+2	87.9	1.14	238.8	1.22E-2	1.03E-2	9684				
254.0	1.06E+2	89.6	1.12	250.5	2.97E-2	2.51E-2	3976				

BV = bed volume; DI = deionized; DF = decontamination factor; FD = feed displacement; C$_0$ = 118 µCi ^{137}Cs/ mL/

Table A.4. Elution and Water Rinse Results Following AP-105DF Processing Test 2

Elution			Water Rinse		
BV	µCi ^{137}Cs/ mL	C/C_0	BV	µCi ^{137}Cs/ mL	C/C_0
1.47	1.92E+0	1.63E-2	31.37	1.30E-1	1.10E-3
2.99	1.83E+0	1.55E-2	32.88	1.14E-1	9.62E-4
4.50	3.31E+0	2.80E-2	34.36	9.24E-2	7.82E-4
6.01	1.23E+1	1.04E-1	35.50	8.05E-2	6.81E-4
7.52	9.61E+0	8.13E-2			
8.98	1.05E+1	8.89E-2			
10.47	4.04E+3	3.42E+1			
11.96	1.52E+4	1.29E+2			
13.46	1.66E+3	1.41E+1			
14.93	1.93E+2	1.63E+0			
16.43	3.29E+1	2.79E-1			
17.93	3.86E+0	3.27E-2			
19.37	1.53E+0	1.29E-2			
20.89	8.41E-1	7.11E-3			
22.38	5.72E-1	4.84E-3			
23.85	3.16E-1	2.67E-3			
25.33	2.15E-1	1.82E-3			
26.90	2.18E-1	1.84E-3			
28.37	1.73E-1	1.46E-3			
29.89	1.39E-1	1.18E-3			

BV = bed volume; C_0 = 118 µCi ^{137}Cs/ mL.

Table A.5. AP-105DF Test 3 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
7.5	7.04E-2	5.61E-2	1783	7.4	8.50E-3	6.77E-3	14,767	FD			
20.7	3.33E-2	2.66E-2	3764	20.4	4.42E-3	3.52E-3	28,416	242.5	6.25E-1	4.98E-1	201
34.2	2.71E-2	2.16E-2	4636	32.1	2.86E-3	2.28E-3	43,880	244.0	6.80E-1	5.42E-1	185
52.6	6.31E-2	5.03E-2	1988	50.2	2.24E-3	1.78E-3	56,115	245.6	7.40E-1	5.90E-1	170
61.5	1.22E-1	9.71E-2	1030	58.8	2.10E-3	1.67E-3	59,854	247.2	3.25E-1	2.59E-1	387
79.0	4.04E-1	0.32	311	76.1	1.81E-3	1.44E-3	69,413	DI rinse			
95.6	1.38E+0	1.10	91	92.3	1.84E-3	1.46E-3	68,300	248.7	6.55E-2	5.22E-2	1916
111.1	4.47E+0	3.56	28	107.7	1.86E-3	1.48E-3	67,386	250.3	4.09E-2	3.26E-2	3068
128.4	9.83E+0	7.83	13	128.5	2.03E-3	1.62E-3	61,788	251.3	2.32E-2	1.84E-2	5421
150.4	2.15E+1	17.15	6	146.4	2.61E-3	2.08E-3	48,015				
164.2	3.92E+1	31.26	3.2	164.2	5.00E-3	3.99E-3	25,075				
186.0	5.81E+1	46.33	2.2	181.6	1.33E-2	1.06E-2	9429				
202.5	7.98E+1	63.60	1.6	196.3	2.88E-2	2.30E-2	4355				
219.2	9.57E+1	76.27	1.31	214.3	1.07E-1	8.55E-2	1170				
232.9	1.07E+2	85.65	1.17	240.4	2.65E-1	2.11E-1	473				
247.6	1.15E+2	91.31	1.10	240.9	5.46E-1	4.35E-1	230				

BV = bed volume; DI = deionized; DF = decontamination factor; FD = feed displacement; C_0 = 125 µCi ^{137}Cs/ mL.

Table A.6. Elution and Water Rinse Results Following AP-105DF Processing Test 3

Elution			Water Rinse			Regeneration		
BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$
1.44	2.85E+0	2.27E-2	24.61	4.77E-1	3.80E-3	1.8	1.36E-1	1.12E-1
2.92	2.89E+0	2.30E-2	26.23	2.86E-1	2.28E-3	3.2	1.27E-1	1.04E-1
4.45	4.03E+0	3.21E-2	27.78	3.63E-1	2.89E-3	4.7	1.14E-1	9.39E-2
5.99	7.62E+0	6.07E-2	29.33	2.52E-1	2.01E-3	6.3	2.29E-2	1.89E-2
7.50	9.95E+0	7.93E-2				7.9	7.51E-3	6.18E-3
9.00	9.57E+0	7.62E-2				9.4	7.73E-3	6.37E-3
10.49	4.26E+3	3.39E+1						
11.97	1.38E+4	1.10E+2						
13.50	2.50E+3	1.99E+1						
15.05	1.07E+2	8.51E-1						
16.57	3.51E+1	2.80E-1						
18.21	8.46E+0	6.74E-2						
19.79	1.47E+0	1.17E-2						
21.51	6.94E-1	5.53E-3						
23.03	5.22E-1	4.16E-3						

BV = bed volume; C$_0$ = 125 µCi ^{137}Cs/ mL.

Table A.7. AP-105DF Test 4 Take 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
4.0	2.03E-2	1.67E-2	5982	4.0	4.07E-3	3.35E-3	29,815	NA			
11.2	1.04E-2	8.53E-3	11,724	11.0	4.33E-3	3.57E-3	28,017				
18.9	6.61E-3	5.44E-3	18,379	18.5	3.53E-3	2.91E-3	34,364				
36.0	5.93E-3	4.89E-3	20,467	35.5	2.53E-3	2.08E-3	48,084				
52.9	1.10E-2	9.04E-3	11,059	52.0	2.36E-3	1.94E-3	51,503				

BV = bed volume; DF = decontamination factor; FD = feed displacement; C$_0$ = 121 µCi ^{137}Cs/ mL

Table A.8. Elution Results Following AP-105DF Processing Test 4 Take 1

Elution			Water Rinse			Regeneration		
BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$
24.92	2.75E+2	2.26E+0	NA	NA	NA	1.6	7.44E-2	6.09E-2
						3.3	2.53E-2	2.07E-2
						4.8	2.90E-2	2.38E-2
						6.4	1.43E-2	1.17E-2
						8.3	4.43E-3	3.62E-3

BV = bed volume; C$_0$ = 121 µCi ^{137}Cs/ mL.

Table A.9. AP-105DF Test 4 Take 2 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
4.1	5.73E-3	4.69E-3	21,324	4.1	1.00E-3	8.20E-4	121,922	240.5	4.91E-3	4.02E-3	24,866
13.3	3.23E-3	2.64E-3	37,837	13.1	8.66E-4	7.09E-4	141,082				
23.4	2.59E-3	2.12E-3	47,111	22.9	1.70E-3	1.39E-3	71,981				
41.0	2.60E-3	2.13E-3	46,961	40.4	5.58E-4	4.57E-4	218,925				
58.3	7.27E-3	5.95E-3	16,797	57.5	5.64E-4	4.62E-4	216,596				
76.1	3.50E-2	0.03	3492	74.9	5.39E-4	4.41E-4	226,611				
92.1	2.18E-1	0.18	560	90.5	5.62E-4	4.60E-4	217,423				
109.5	8.61E-1	0.70	142	107.8	5.98E-4	4.89E-4	204,353				
126.8	3.19E+0	2.61	38	124.8	7.11E-4	5.82E-4	171,674				
144.4	1.12E+1	9.18	11	142.0	7.18E-4	5.88E-4	170,002				
161.8	2.71E+1	22.20	4.5	159.0	1.22E-3	1.00E-3	99,943				
182.3	6.11E+1	50.05	2.0	179.4	5.01E-3	4.10E-3	24,375				
204.8	8.75E+1	71.64	1.4	201.5	3.80E-2	3.11E-2	3212				
226.0	1.10E+2	89.68	1.12	222.5	2.14E-1	1.75E-1	572				
238.5	1.14E+2	93.11	1.07	234.7	6.13E-1	5.02E-1	199				

BV = bed volume; DF = decontamination factor; FD = feed displacement; C$_0$ = 122 μCi ^{137}Cs/ mL.

Table A.10. Elution and Water Rinse Results Following AP-105DF Processing Test 4 Take 2

Elution			Water Rinse			Regeneration		
BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$
29.46	1.03E+3	8.47E+0	EDi			1.5	8.71E-2	6.98E-2
			31.04	1.78E-1	1.46E-3	3.0	8.72E-2	6.98E-2
			32.67	1.15E-1	9.42E-4	4.5	1.15E-1	9.23E-2
			34.29	1.48E-1	1.21E-3	6.0	2.16E-2	1.73E-2
			35.93	2.13E-1	1.74E-3	7.5	6.53E-3	5.23E-3
			37.57	9.72E-2	7.96E-4	9.1	4.59E-3	3.67E-3
			EDiF					
			29.46	3.16E-2	2.59E-4			

BV = bed volume; C$_0$ = 122 µCi ^{137}Cs/ mL.

Table A.11. AP-105DF Test 5 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
7.5	9.38E-2	7.52E-2	1331	7.6	5.20E-3	4.17E-3	23,998	186.8	2.98E-2	2.38E-2	4196
15.8	1.24E-2	9.90E-3	10,099	15.6	4.20E-3	3.37E-3	29,711				
29.2	8.78E-3	7.03E-3	14,223	28.7	3.79E-3	3.04E-3	32,942				
42.9	9.21E-3	7.38E-3	13,549	42.0	3.03E-3	2.43E-3	41,199				
59.5	3.30E-2	2.65E-2	3778	58.3	3.77E-3	3.02E-3	33,067				
72.9	9.76E-2	0.08	1279	71.4	2.79E-3	2.23E-3	44,774				
85.7	3.28E-1	0.26	380	84.0	2.26E-3	1.81E-3	55,171				
99.1	9.84E-1	0.79	127	97.0	2.41E-3	1.93E-3	51,864				
112.0	2.37E+0	1.90	53	109.7	2.44E-3	1.96E-3	51,136				
121.6	4.56E+0	3.66	27	119.0	2.57E-3	2.06E-3	48,637				
137.3	9.76E+0	7.82	13	134.4	2.58E-3	2.07E-3	48,420				
149.7	1.85E+1	14.80	7	146.6	3.18E-3	2.54E-3	39,310				
162.2	3.25E+1	26.03	4	158.8	3.32E-3	2.66E-3	37,582				
173.4	4.73E+1	37.93	3	169.8	3.89E-3	3.12E-3	32,063				
183.0	6.41E+1	51.39	2	179.0	4.95E-3	3.97E-3	25,204				
194.7	8.07E+1	64.66	2	190.4	9.22E-3	7.39E-3	13,534				
203.5	8.89E+1	71.20	1	198.9	1.45E-2	1.16E-2	8617				
212.1	9.62E+1	77.08	1	207.2	2.18E-2	1.75E-2	5719				
218.2	1.04E+2	83.05	1	213.1	3.42E-2	2.74E-2	3650				

BV = bed volume; DF = decontamination factor; FD = feed displacement; C_0 = 125 μCi ^{137}Cs/ mL

Table A.12. Elution and Water Rinse Results Following AP-105DF Processing Test 5

Elution			Water Rinse		
BV	µCi ^{137}Cs/ mL	C/C$_0$	BV	µCi ^{137}Cs/ mL	C/C$_0$
29.13	8.93E+2	7.15E+0	EDi		
			30.83	1.83E-1	1.46E-3
			32.42	1.45E-1	1.16E-3
			33.95	1.31E-1	1.05E-3
			35.53	1.39E-1	1.11E-3
			37.08	1.20E-1	9.61E-4
			EDiF		
			29.13	4.76E-2	3.82E-4

BV = bed volume; C$_0$ = 125 µCi ^{137}Cs/ mL.

Appendix B

Analytical Reports

Appendix B

Analytical Reports

Analytical reports provided by the Analytical Support Operations (ASO) 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 Services Request (ASR) number.

Table of Contents

ASR 0272, Initial Characterization of AP-105

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 5-5677 MSIN P7-25

Project Number: 69833
Work Package: N78228

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:**
  4/28/17
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:**
  5/5/17
  (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?
☑ **No** ☐ **Yes**

Send Report To: Sandra Fiskum, Reid Peterson, **MSIN** P7-25, P7-22
John Geeting, Jarrod Allred **MSIN** K9-89, P7-25
Additional or Special Instructions __________

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

Date Delivered: 4/25/17
Delivered By (optional) J. Condray
Time Delivered: 09:40
Group ID (optional) __________
CMC Waste Sample? ☒ No ☐ Yes

Received By: L. Darnell @ 09:40
ASR Number: 0272 Rev.: 00
RPL Numbers: 17-0868
~~(first and last)~~

ASO Work Accepted By: KN Pool **Signature/Date:** 4/25/17

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**
17-0868	SAP-16-01	AP-105 as-sampled from the Hanford	ICP-OES (Al, Na, K)		
		tank	Free hydroxide		
			GEA (*Cs-137, and any other detectable*		
			Cs isotopes)		
			ICP-MS (Cs-133)		

AP-105 is expected to be 8.5 M Na
All analytes are expected to be well above instrument detection limits. Therefore required MDLs not provided.

ASR # 0272 Rev.: 00

Page 1 of 1

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 #: 69833
Point of Contact (name. telephone#): SK Fiskum 375-5677

Comments:

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

Sample Collection Date: ______________ Sample Collection Time: ______________

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

Comments *(list known isotopes)*: AP-105 tank waste, Cs-137 ~ 200 µCi/mL

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
Cr	~460 µg/mL	TWINS
hydroxide	~1.9 M	TWINS

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

Checklist Prepared By:
Sandra Fiskum Sandra Fiskum 4/14/17
Printed Name Signature Date

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

Project / WP#:	69833 / N78228
ASR#:	0272
Client:	S. Fiskum
Total Samples:	1 (liquid)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
17-0868	5AP-16-01	AP-105 as sampled from the Hanford Tank	NA

Sample Preparation: Client prepared.

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:**	05-01-2017	**ICP File:**	C0714
See Chemical Measurement Center 98620 file:		ICP-325-405-3 (Calibration and Maintenance Records)			

M&TE:	☒	PerkinElmer 5300DV ICP-OES	SN: 077N5122002
	☐	Sartorius ME414S Balance	SN: 22406373
	☒	Sartorius ME414S Balance	SN: 22006647
	☒	Sartorius R200D Balance	SN: 39080042
	☐	Mettler AT201 Balance	SN: 192720-92

Chris Thomps
Report Preparer

5/19/17
Date

Karl N. Pool
Review and Concurrence

5/19/17
Date

One liquid sample submitted under Analytical Service Request (ASR) 0272 was analyzed by ICP-OES. The sample was analyzed after dilution of the "as received" liquid in 5% v/v HNO_3. Neither the sample nor any of the dilutions were filtered.

All 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 document was 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 spikes, duplicate, blank spike, and serial dilution were conducted during the analysis run.

Preparation Blank (PB):

No preparation blank was required. The ICP-OES laboratory diluent (5% HNO_3) was analyzed as a blank. All AOI 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):

The MCVA solution was analyzed as the blank spike. Recovery values are listed for all analytes included in the BS that were measured at or above the EQL. All AOI were present in the MCVA solution. Recovery values for the AOI meeting this requirement ranged from 97% to 101%, and were within the acceptance criterion of 80% to 120%.

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

A replicate of sample 17-0868 was 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 0.1% to 1.3% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

No matrix spike was required.

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.

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.

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 17-0868. 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 2.2% to 5.1% and were within the acceptance criterion of ≤10%.

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

Because no MS sample was prepared, a post-digestion spike (A Component) was conducted on sample 17-0868. 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 100% to 107%, and were within the acceptance criterion of 80% to 120%.

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

Because no MS sample was prepared, a post-digestion spike (B Component) was conducted on sample 17-0868. 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. 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) Absolute precision, bias and detection limits may be determined on each sample if required by the client. The maximum number of significant figures for all ICP measurements is two.
5) 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 >	5/1/2017	5/1/2017	5/1/2017
		Process Factor >	1.0	500.0	500.0
		RPL/LAB >	405 diluent	17-0868 @ 500x	17-0868 @ 500x rep
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	SAP-16-01	
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)
0.0276	0.276	Al	--	20,600	20,900
0.0500	0.500	K	--	5,520	5,530
0.0171	0.171	Na	--	195,000	197,000
Other Analytes					
0.0030	0.030	Ag	--	--	--
0.1576	1.576	As	--	--	--
0.0120	0.120	B	[0.032]	71.7	68.0
0.0005	0.005	Ba	--	[0.60]	[0.52]
0.0002	0.002	Be	--	[0.19]	[0.32]
0.0546	0.546	Bi	[0.065]	[29]	--
0.0233	0.233	Ca	--	[90]	[99]
0.0027	0.027	Cd	--	[1.8]	[1.9]
0.0448	0.448	Ce	--	--	--
0.0071	0.071	Co	--	--	--
0.0047	0.047	Cr	--	505	505
0.0036	0.036	Cu	--	[3.9]	[3.2]
0.0034	0.034	Dy	--	[1.9]	--
0.0010	0.010	Eu	--	--	--
0.0017	0.017	Fe	[0.0045]	[5.9]	[6.2]
0.0030	0.030	La	--	--	--
0.0028	0.028	Li	--	--	--
0.0023	0.023	Mg	--	--	--
0.0009	0.009	Mn	--	--	--
0.0094	0.094	Mo	[0.021]	83.0	84.0
0.0188	0.188	Nd	--	--	--
0.0074	0.074	Ni	--	45.8	45.3
0.1021	1.021	P	--	752	715
0.0399	0.399	Pb	--	[25]	[31]
0.0170	0.170	Pd	--	--	--
0.0162	0.162	Rh	--	--	--
0.0158	0.158	Ru	--	[13]	[11]
0.1773	1.773	S	--	1,820	1,800
0.0919	0.919	Sb	--	--	--
0.2271	2.271	Se	--	--	[230]
0.0136	0.136	Si	--	[67]	[61]
0.0446	0.446	Sn	--	--	--
0.0003	0.003	Sr	--	[0.28]	[0.29]
0.0242	0.242	Ta	--	--	--
0.0412	0.412	Te	--	--	--
0.0120	0.120	Th	--	--	--
0.0014	0.014	Ti	--	--	--
0.0885	0.885	Tl	--	--	--
0.0676	0.676	U	--	--	--
0.0032	0.032	V	--	--	--
0.0249	0.249	W	--	141	154
0.0006	0.006	Y	[0.0008]	[0.35]	[0.42]
0.0066	0.066	Zn	--	[28]	[27]
0.0022	0.022	Zr	--	[1.2]	--

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "
near the top of each column. The estimated sample quantitation limit = EQL (in Colum
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%.

ASR 0272 Final from C0714 Fiskum ASR-0272 Tank Waste.xls

QC Performance 05/01/2017

Criteria >	**≤ 20%**	**80%-120%**	**75%-125%**	**80%-120%**	**80%-120%**	**≤ 10%**
QC ID >	**17-0868 Rep**	**LCS/BS**	**MS (none)**	**17-0868 + AS-A**	**17-0868 + AS-B**	**17-0868 5-fold Serial Dil**
Analytes	**RPD (%)**	**%Rec**	**%Rec**	**%Rec**	**%Rec**	**%Diff**
Al	1.3	101		104		3.3
K	0.1	100		100		2.2
Na	1.3	97		107		5.1
Other Analytes						
Ag		93		93		
As		99		101		
B	5.4	101		101		
Ba		98		97		
Be		99		99		
Bi		95		94		
Ca		102		101		
Cd		102		102		
Ce					92	
Co		99		99		
Cr	0.0	100		100		0.7
Cu		99		101		
Dy					99	
Eu					100	
Fe		99		98		
La					99	
Li		102		102		
Mg		99		98		
Mn		101		101		
Mo	1.1	98		98		
Nd					99	
Ni	1.1	98		97		
P	5.0	102		101		
Pb		99		99		
Pd					91	
Rh					93	
Ru					95	
S	1.0				99	
Sb		96		98		
Se		103		105		
Si		99		106		
Sn		95		95		
Sr		99		98		
Ta				98		
Te					99	
Th					101	
Ti		98		97		
Tl		91		92		
U					100	
V		99		98		
W	8.8	101		100		
Y		100		100		
Zn		101		102		
Zr		99		98		

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#:	69833 / N78228
ASR#:	0272
Client:	S. Fiskum
Total Samples:	1 (liquid)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
17-0868	5AP-16-01	AP-105 as sampled from Hanford tank	NA

Sample Preparation: Dilution of "as received" samples in 0.5 M HNO_3 and 2% v/v HNO_3 was performed by L. Darnell on 4/26/17 and G. Brown on 7/7/17, respectively.

Procedure: RPG-CMC-292, Rev. 0, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)".

Analyst:	G. Brown	**Analysis Date:**	07-10-2017	**ICP File:**	M0025

See Chemical Measurement Center 98620 file: ICP-MS-325-405-1 (Calibration and Maintenance Records)

M&TE:	☒	PerkinElmer NexION™ 350X ICP-MS	SN: 85VN4070702
	☐	Sartorius ME414S Balance	SN: 22406373
	☒	Mettler AT400 Balance	SN: 1113292667
	☒	Sartorius R200D Balance	SN: 39080042
	☒	Mettler AT201 Balance	SN: 192720-92

Garrett N. Brown
Report Preparer

7/24/17
Date

For C. Thompson
Review and Concurrence

7/25/17
Date

PNNL ASO ICP-MS Analysis Results

RPL ID	Client ID	Run Date	Process Factor	Sample	Replicate	Triplicate	Units
17-0868	5AP-16-01	07/10/17	1,934,970	[8230]	[8800]	[7730]	µg/L Cs-133
17-0868	5AP-16-01	07/10/17	389,931	[8070]	[7100]	nm	µg/L Cs-133
					Average:	8,000	µg/L Cs-133

QC Performance 07/10/2017

Criteria >	≤ 20%	80%-120%	75%-125%	80%-120%	≤ 10%			
QC ID >	17-0868 Trip	LCS/BS (None)	MS (None)	17-0868 + PS	17-0868 5-fold Serial Dil	Analyte IDL	Analye EQL	Lab Diluent
Analytes	RSD (%)	%Rec	%Rec	%Rec	%Diff	(µg/L)	(µg/L)	(µg/L)
Cs-133	6.44%	95.3%	na	95.0%	3.38%	0.0030	0.030	--

1) "--" indicates the value is < MDL. The method detection limit (MDL) = IDL times the "Process Factor" for each sample analysis. The estimated sample quantitation limit = EQL times the "Process Factor." 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%.

Matrix effects can be assessed from the serial dilution. Shaded results are outside the acceptance criteria.

nr = spike concentration less than 25% of sample concentration.

na = not applicable (e.g., LCS, BS or MS may not be required for liquid samples).

nm = not measured (e.g., triplicate analysis may not be required by procedure).

One liquid sample submitted under Analytical Service Request (ASR) 0272 was analyzed by ICP-MS for Cs-133. The sample was analyzed after dilution of the "as received" liquid in 0.5 M HNO_3 by L. Darnell on 4/26/17 and 2% v/v HNO_3 by G. Brown on 7/17/17. The sample and dilutions were not filtered.

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

The analyte of interest (AOI), Cs-133, was specified in the ASR. Results for Cs-133 are listed in the upper section of the attached ICP-MS Data Report. The quality control (QC) results for Cs-133 have been evaluated and are presented below. Results for other analytes are not included in the report.

Calibration of the ICP-MS was done following the manufacturer's recommended procedures using 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 the analytical procedure and ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) Quality Assurance Plan.* Instrument calibration, QC checks and blanks (e.g., ICV/ICB, CCV/CCB, LLS, ICS), post-digestion spike, triplicates, 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 10 ppb Rb-85 as an internal standard. All data were normalized using the Rb-85 mass. The IS recovery ranged from 96.8% to 103.5%, which is within the acceptance criterion of 30% to 120%.

Preparation Blank (PB) Sample:

No preparation blank (PB) sample was required to be prepared since the client sample was received as a liquid and only simple dilution was required prior to analysis. The ICP-MS laboratory diluent (2% v/v HNO_3) was analyzed in place of a PB sample as the initial and continuing calibration blanks (described below).

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

A blank spike (BS) sample was prepared by spiking a 2% HNO_3 blank with an equivalent volume of the 2 ppb Cs-133 CCV standard (1:1 ratio). The Cs-133 recovery was 95.3% and was within the acceptance criteria of 80% to 120% recovery.

Triplicate/Relative Standard Deviation (RSD):

A triplicate analysis was performed on sample 17-0868. The RSD is listed for analytes that were measured at or above the EQL. The RSD for Cs-133 was 6.44% and was within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

No matrix spike (MS) sample was required.

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 Cs-133 recoveries ranged from 94.9% to 97.4% and 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 concentrations of Cs-133 in all ICB/CCBs were within the acceptance criteria of <EQL.

Low-Level Standard (LLS):

The LLS solution was analyzed immediately after the first CCB. The recovery for Cs-133 (97.6%) was within the acceptance criteria of 75% to 125%.

Interference Check Standard (ICS):

The ICS solution was analyzed immediately after the LLS solution and immediately after the final CCV solution. The recoveries for Cs-133 (98.7% and 99.9%) were within the acceptance criteria of 80% to 120%.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 17-0868. Percent differences (%D) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %D for Cs-133 was 3.38% and was within the acceptance criterion of ≤10%.

Post-Digestion Spike (PS)/Analytical Spike (AS) - Sample (Cs-133 Component):

Because no MS sample was prepared, a post-digestion spike (Cs-133 Component) was conducted on sample 17-0868. 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 for Cs-133 was 95.0% which is 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 500 µg/mL (0.05 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) Absolute precision, bias and detection limits may be determined on each sample if required by the client. The maximum number of significant figures for all ICP-MS measurements is three.

Pacific Northwest
NATIONAL LABORATORY

Client:	S. Fiskum	Report Date:	5/10/2017
		Analysis Date:	5/1/2017

Subject: Hydroxide Analyses for: **Aqueous Samples**
Project: 69832 WP # N79888
69833 WP # N78228
ASR: **0262 & 0272** Rev-0 Procedure: RPG-CMC-228-Rev 0.1
Sample ID. 17-0791 and 17-0868

Diluted sample aliquots of 2 aqueous samples (see above assigned RPL Sample #'s), provided on ASR's 0262 and 0272 were analyzed by manual titration for the base constituents content following procedure RPG-CMC-228. Each sample was diluted by a factor of 26x prior to titration (0.100 mL of sample added to 2.5 mL of DI water). The titrant used was 0.0957 M HCl (Standardized HCl was prepared and documented on Chem Rec 232, prepared on 3/15/2017). 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 (the 1st inflection point at pH of 11.0 - 11.1) was very weak but discernable. A 2nd inflection point was determined for both samples at pH 7.6 to 7.7. The third inflection point (at pH 4.8 to 5.0) was easily identified and reportable.

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: ______________________ Date: 5/10/17

Reviewed by: ______________________ Date: 5/10/17

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

ASR # **0262 & 0272**

WP# N79888 & N78228

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

Equip # Beckman Coulter 560, SN#110650046

Report Summary for ASR # -- 0262 & 0272
Revision # **Rev-0**

Report Date: 5/10/2017
Analysis Date: 5/1/2017

				Concentration, moles / Liter		
				First Point	Second Point	Third Point
RPG #	Client ID	Diluted Initial pH	OH conc ug/mL	Molarity	Molarity	Molarity
17-0791	SS07Feed A	12.57	2.51E+04	1.47	0.74	0.42
17-0868	5AP-16-01	12.50	2.98E+04	1.75	1.49	0.73

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, 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 5.10.17

Reviewer: Karl N. Pool

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev #	0262 & 0272	Rev-0	
Client:	S. Fiskum		
Project:	69832	WP#	N79888
	69833		N78228

Report Date: **5/10/2017**
Analysis Date: **5/1/2017**

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: 5.10.17

Titrant	Molarity	Chem Rec#
HCl	**0.0957**	**232**

									OH			
RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	**Diluted** Initial pH reading	**1st Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
17-0791	SS07Feed A		NA	0.100	NA	NA	NA	12.570	1.540	11.000	0.147	1.474
17-0868	5AP-16-01		NA	0.100	NA	NA	NA	12.500	1.830	11.100	0.175	1.751

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	1-Jan-18

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

Slope
95.2%

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev # 0262 & 0272

WP# N79888
N78228

Procedure:
Alkalinity of Aqueous Solutions, Leachates and Supernates

By Manual Titration

Equip # Beckman Coulter 560, SN#110650046

Titrant	Molarity
HCl	0.0957

RPG #		Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
17-0791		0.100	2.310	7.700	0.074	0.737
17-0868		0.100	3.390	7.600	0.149	1.493

3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
2.750	4.800	0.042	0.421
4.150	5.000	0.073	0.727

Gamma Energy Analysis (GEA)

Revision 1 (This report was revised to correct the sample preparation information)

Project / WP#:	69833/ N78228
ASR#:	0272.00
Client:	SK Fiskum
Total Samples:	1

RPL ID	Client Sample ID
17-0868	SAP-16-01

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:	Not Applicable
Analysis Date or Date Range:	April 26, 2017
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*17-0868 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 T*

T Trang-le / 7/11/17
Prepare **Date**

LR Greenwood / 7/11/17
Reviewer **Date**

Page 1 of 3

SAMPLE RESULTS

Activities for all gamma emitters detected in this sample are presented in an attached Excel spreadsheet for ASR 0272.00. All sample results for all target isotopes are reported in units of µCi/sample with estimates of the total propagated uncertainty reported at the 1-sigma level.

ASO Project File, ASR 0272 has been created for this report including all appropriate supporting records which may include the Pipette Performance Verification form, Standard Certificates, Laboratory Bench Record, 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

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

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

The sample was sent to the counting room with 0.5ml of sample that was diluted to a total volume of 2.0ml prior to the GEA count. There is no process blank for this ASR.

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):

There are no duplicate samples for this ASR.

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. 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 0272.

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

filename 17-0868 Fiskum
4/26/2017

Client: Fiskum
ASR 0272

Project: 69833
WP: N78228

Prepared by: T Trang-le 4/26/2017

Technical Reviewer: 4/28/2017

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

M&TE: Gamma detectors T
Count dates: 26-Apr-17

	Measured Activity, µCi per mL ± 1s		
Sample	Lab ID	**Cs-137**	
SAP-16-01	17-0868	1.80E+02	± 2%

ASR 0329, Cs Isotopic Results

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. Fisk
Print Name SANDRA FISKUM
Phone 5-5677 MSIN ____

Project Number: 69832
Work Package: N82456

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:**
  August 9, 2017
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:**
  8/9/17
  (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?
☒ **No** ☐ **Yes**

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

Additional or Special Instructions ____

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

Date Delivered: 7/26/17
Delivered By (optional) ____
Time Delivered: 8:45am
Group ID (optional) ____
CMC Waste Sample? ☒ No ☐ Yes

Received By: T Trang-Le
ASR Number: 0329 Rev.: 00
RPL Numbers: (17-1224) (first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** 7/26/17

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**
17-1224	T1014-E8-A	Cs from AP-105	TIMS mass distribution of		
			Cs-133		
			Cs-134		
			Cs-137		
			Cs-135		
	Analyze in duplicate,	report duplicate results.			
	sample contains	1.8 µCi ^{137}Cs/mL solution	= 0.02 µg ^{137}Cs/mL		
	^{137}Cs should be about	20-25 % of the total Cs	content.		

ASR # 0329 Rev.: 00

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 #: 69832
Point of Contact (name telephone#): Sandra Fiskum 5-5677

Comments:

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

Sample Collection Date: 7/20/17 Sample Collection Time: 23:16

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

Comments *(list known isotopes)*: Cs-137
Cs-134

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

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

matrix is 0.45 M HNO_3

Checklist Prepared By:

Sandra Fiskum Sandra Fiskum 7/26/17
Printed Name Signature Date

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

Project / WP#:	69832 / N82456
ASR#:	0329
Client:	S. Fiskum
Total Samples:	1 (Aqueous)

ASO Sample ID	**Client Sample ID**	**Client Sample Description**	**Sample Weight (g)**
17-1224	TI014-E8-A	Cs from AP-105	NA

Sample Preparation: Dilution of "as received" samples in 2% v/v HNO_3 was performed by G. Brown on 8/9/17.

Procedure: RPG-CMC-292, Rev. 0, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."

Analyst:	G. Brown	**Analysis Date:**	08/09/2017	**ICP File:**	M0026

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

M&TE:	☒	PerkinElmer 5300DV ICP-MS	SN: 077N5122002	RPL 405 Bench
	☐	Sartorius ME414S Balance	SN: 22406373	RPL 405 Bench
	☐	Sartorius R200D Balance	SN: 39080042	RPL 405 Bench
	☒	Mettler AT400 Balance	SN: 192720-92	RPL 405 FH #3
	☐	Mettler AT400 Balance	SN: 1113292667	RPL 420 FH #13
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH #9

8/15/17

Report Preparer — Date

8/31/17

Review and Concurrence — Date

One aqueous sample submitted under Analytical Service Request (ASR) 0329 was analyzed by ICP-MS for Cs-133, Cs-135, and Cs-137. The sample was analyzed after dilution of the "as received" liquid in 2% v/v HNO_3 by G. Brown on 08/09/17. The sample and dilutions were not 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 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-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 0, *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 Cs-133, Cs-135, and Cs-137 (AOI) data were normalized using the In-115 mass as the IS. The In-115 IS recoveries ranged from 99% to 103%, which were within the acceptance criterion of 30% to 120%. An anomalously high value (130%) for the Sc-45 IS recovery was observed for sample 17-1224, likely caused by >8000 ng/mL boron in the sample. Contamination of the sample with B, Na, Si and other components from the glass container was likely.

Preparation Blank (PB):

No preparation blank was required to be prepared. A diluent blank from the ICP-MS laboratory (2% HNO_3) was analyzed in place of the preparation blank. All AOI 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 were 95%, 94%, and 98% for masses 133, 135, and 137, respectively and were within the acceptance criteria of 80% to 120% recovery.

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

A replicate of sample 17-1224 was 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 1.5% to 2.7% and were 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. The concentrations of all AOI were within the acceptance criteria of 90% to 110% recovery.

Initial/Continuing Calibration Blank (ICB/CCB):

The ICB/CCB solutions (2% v/v HNO_3) were 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.

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 17-1224. 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 0.2% to 1.8% 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 sample 17-1224. 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 96% to 99%, 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.

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

		Run Date >	8/9/2017	8/9/2017	8/9/2017	
		Multiplier >	1.0	10.02	10.02	
		RPL/LAB >	Lab 405 Blank	17-1224 @ 10x	17-1224 @ 10x rep	
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab 405 Blank	TI014-E8-A		
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	
0.0021	0.021	Cs 133	[0.002]	81.7	83.0	
0.0003	0.003	Cs 135	--	24.4	24.8	
0.001	0.007	Cs 137	--	21.4	22.0	
Other Analytes						
0.0057	0.057	Be 9	--	--	--	
0.380	3.80	B 11	--	8,110	8,240	(3)
0.134	1.34	Mg 25	--	23.4	23.7	
0.0020	0.020	V 51	--	0.648	0.671	
0.0125	0.125	Cr 53	--	6.01	5.84	
0.0061	0.061	Mn 55	--	1.21	1.17	
0.0053	0.053	Co 59	--	[0.07]	[0.10]	
0.0082	0.082	Ni 60	--	2.29	2.68	
0.0179	0.179	Cu 65	--	16.0	15.9	
0.570	5.70	Zn 66	--	37.1	36.6	
0.0021	0.021	Ga 69	--	1.16	1.10	
0.0070	0.070	As 75	--	[0.28]	[0.23]	
0.184	1.84	Se 77	--	--	--	
0.0027	0.027	Rb 85	--	1.90	1.75	
0.0052	0.052	Sr 88	--	2.46	2.34	
0.0113	0.113	Ag 107	--	--	--	
0.0030	0.030	Cd 111	[0.003]	[0.07]	[0.06]	
0.0039	0.039	Ba 138	--	18.0	17.9	
0.0023	0.023	La 139	--	[0.24]	[0.25]	
0.0027	0.027	Ce 140	--	0.613	0.636	
0.0023	0.023	Pr 141	--	[0.09]	[0.08]	
0.0027	0.027	Nd 143	--	[0.23]	[0.26]	
0.0031	0.031	Sm 149	--	[0.04]	[0.04]	
0.0025	0.025	Eu 151	--	--	--	
0.0026	0.026	Gd 157	--	[0.04]	[0.05]	
0.0025	0.025	Dy 161	--	[0.06]	[0.05]	
0.0023	0.023	Ho 165	--	--	--	
0.0024	0.024	Er 166	--	[0.04]	[0.04]	
0.0025	0.025	Tm 169	--	--	--	
0.0023	0.023	Yb 173	--	[0.05]	[0.04]	
0.0040	0.040	Lu 175	--	--	--	
0.0029	0.029	Tl 205	--	--	--	
0.0051	0.051	Pb 208	--	98.0	99.6	
0.0039	0.039	Th 232	--	[0.063]	[0.055]	
0.0026	0.026	U 238	--	2.27	2.26	
		Internal Standard % Recovery				
		Li 6 (IS)	106%	111%	107%	
		Sc 45 (IS)	104%	130%	128%	
		Y 89 (IS)	101%	104%	106%	
		In 115 (IS)	99%	103%	99%	
		Tb 159 (IS)	102%	106%	105%	
		Bi 209 (IS)	101%	106%	105%	

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) Boron-11 results were nearly 100x above the maximum calibration and may not be valid

M0026-71A ASR-0329 Fiskum 170809Re.xlsx

QC Performance 08/09/2017

Criteria >	≤ 20%	80%-120%	75%-125%	75%-125%	≤ 10%
QC ID >	17-1224 Dup	LCS/BS	MS (None)	17-1224 + CCV-71A	17-1224 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Diff
Cs 133	1.5	95		96	1.2
Cs 135	1.5	94		97	0.2
Cs 137	2.7	98		99	1.8
Other Analytes					
Be 9		96		91	
B 11	1.5			nr	4.5
Mg 25	1.4				
V 51	3.5	90		90	
Cr 53	2.8	92		87	
Mn 55	3.4	92		92	
Co 59		95		91	
Ni 60	15.6	92		96	
Cu 65	0.2	99		96	
Zn 66					
Ga 69	5.5	95		90	
As 75		99		87	
Se 77					
Rb 85	8.4	93		90	
Sr 88	5.0	96		92	
Ag 107		188		196	
Cd 111		96		96	
Ba 138	0.4	96		89	
La 139	1.9	95		92	
Ce 140	3.6	94		90	
Pr 141		94		91	
Nd 143		95		92	
Sm 149		97		92	
Eu 151		95		92	
Gd 157		95		91	
Dy 161		97		92	
Ho 165		94		90	
Er 166		97		90	
Tm 169		95		97	
Yb 173		95		90	
Lu 175		93		91	
Tl 205		95		93	
Pb 208	1.6	95		95	2.0
Th 232		97		94	
U 238	0.4	94		95	
Internal Standard % Recovery					
Li 6 (IS)	106%	101%		104%	111%
Sc 45 (IS)	116%	107%		110%	130%
Y 89 (IS)	108%	104%		108%	104%
In 115 (IS)	102%	99%		102%	103%
Tb 159 (IS)	105%	102%		108%	106%
Bi 209 (IS)	107%	103%		106%	106%

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.

ASR 0335, Test 1 Results

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 Fiskum
Print Name SANDRA FISKUM
Phone ______ MSIN ______

Project Number: 69833
Work Package: N82456

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***
  <u>If Yes, Contact ASO Lead before submitting Samples</u>
  ☐ 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:** 8/24/17
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:** 9/30/17
  (To be completed by ASO Lead)

Waste Designation Information

- **ASO Sample Information Check List Attached?** ☒ No ☐ Yes
  If no, Reference Doc Attached: see attachment
  or, Previous ASR Number: 272
  or, Previous RPL Number: ______

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

Send Report To: Sandra Fiskum **MSIN** P7-25
______ **MSIN** ______

Additional or Special Instructions ______

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

Date Delivered: 8/21/17
Delivered By (optional) ______
Time Delivered: 9:30am
Group ID (optional) ______
CMC Waste Sample? ☒ No ☐ Yes

Received By: Trang-Le
ASR Number: 0335 Rev.: 0
RPL Numbers: 17-1240 → 17-1242
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** 8/21/17

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
17-1240	TI014-FEED	AP-105 Direct Feed (Caustic)	1) GEA - As received solutions. (Co-60, Cs-137 and Eu-154) 2) IC- Anions (17-1240 and 17-1241 only) Cl, NO2, NO3, PO4, C2O4, SO4 3) Acid Digestion - 128 (17-1240 and 17-1241 Only) 4) ICP/MS - Hg (all 3 samples - Use acid digest solutions and diluted 17-1242) 5) ICP/OES - Ag, Al, As, B, Ba, Ca, Cd, Cr, Fe, K, Li, Na, Ni, P, Pb, Se, Th, Tl, U, Zn and Zr (all 3 samples - Use acid digest solutions and diluted 17-1242). 6) Total Alpha/Total Beta (all 3 samples) 7) U-KPA (all 3 samples) 8) Sr-90 (all 3 samples) 9) Tc-99 (all 3 samples) 10) Np-AEA (all 3 samples) 11) Pu-AEA (all 3 samples) 12) Am/Cm-AEA (all 3 samples) 13) TOC/TIC - (17-1240 and 17-1241 only) 14) Free OH - (17-1240 and 17-1241 only) 15) H+ (sample 17-1242 only)		
17-1241	TI014-EFF Comp	AP-105 - Cs Removed (Caustic)			
17-1242	TI014-ELComp	AP-105 - Cs Eluant (Acidic)			

Note: All 3 samples are to be run in duplicate. The Cs eluant sample will be pre-diluted by a factor of approximately 100x prior to loading out of the Hot Cell. Special instructions from the client are attached to the ASR. If there any questions, please contact K. Pool prior to starting analytical work.

ASR # 0335 Rev: 00

Special Instructions for ASR 0335

Analysis of AP-105 diluted feed, effluent, and eluate from Test 1 ion exchange process run

Use the ASO QA Plan current revision

Client ID	ASO Sample ID	Location for pickup	Matrix
TI014-FEED	17-1240	SAL cell 3	Caustic/5.6 M Na, high salt
TI014-EFFComp	17-1241	Lab 305	Caustic/5.6 M Na, high salt
TI014-ELComp	17-1242	SAL cell 3	Acidic/~0.2 M HNO_3

Please prepare each sample in duplicate so that I can have a duplicate analysis of each.

Because the TI014-ELComp sample is already acidified, acid digestion may not be needed. The high salt samples are assumed to require acid digestion. However if the analysis technique sufficiently dilutes the sample into required acid matrix, then acid digestion may be omitted.

Preparative QC samples expected for this suite include:

1. Process blank (digestion blank or diluent blank)
2. Duplicate (all samples processed in duplicate)
3. Blank spike or reagent spike
    a. not applicable to GEA
4. Matrix spike
    a. ICP-OES: not needed for major analytes, e.g., Na, for guidance on spiking needs see ICP-OES results from ASR 0316 report serial dilution instead per ASO QA Plan
    b. IC: not needed for major analytes, e.g., nitrate, report serial dilution instead per ASO QA Plan
    c. not applicable to GEA
    d. not applicable to hydroxide analysis

Please analyze **TI014-FEED** and **TI014-EFFComp** for the following:

Analyte	Analysis Method
^{60}Co ^{137}Cs ^{154}Eu	Gamma Energy Analysis (GEA) (report any other observed gamma emitting isotopes)
^{237}Np ^{238}Pu $^{239+240}$Pu ^{241}Am ^{242}Cm $^{243+244}$Cm	Acid Digestion Separations/Alpha Energy Analysis (AEA)
^{90}Sr	Acid Digestion / Separations/Beta Scintillation Counting
^{99}Tc	Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Separations/Beta Counting
Total Alpha	Acid Digestion / Alpha Scintillation or Gas Proportional Counting
Total Beta	Acid Digestion / Beta Scintillation or Gas Proportional Counting
Hg	Acid Digestion / ICP-MS
Ag Al As B Ba Ca Cd Cr Fe K Li Na Ni P Pb Se Th Ti U (total) Zn Zr	Acid Digestion Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
U (total)	Acid Digestion / Kinetic Phosphorescence Analysis (KPA)
$Cl^{-(d)}$ $NO_2^{-(d)}$ $NO_3^{-(d)}$ $PO_4^{3-(d)}$ $C_2O_4^{2-(d)}$ $SO_4^{2-(d)}$	Dilution Ion Chromatography (IC)
Total organic carbon (TOC) Total inorganic carbon (TIC)	Hot Persulfate Oxidation
Free Hydroxide	Titration

Analyze the **TI014-ELComp** for the following

Analyte	Analysis Method
^{60}Co	Gamma Energy Analysis (GEA) (report any other observed gamma emitting isotopes)
^{137}Cs	
^{154}Eu	
^{237}Np	Acid Digestion Separations/Alpha Energy Analysis (AEA)
^{238}Pu	
$^{239+240}$Pu	
^{241}Am	
^{242}Cm	
$^{243+244}$Cm	
^{90}Sr	Acid Digestion / Separations/Beta Scintillation Counting
^{99}Tc	Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Separations/Beta Counting
Total Alpha	Acid Digestion / Alpha Scintillation or Gas Proportional Counting
Total Beta	Acid Digestion / Beta Scintillation or Gas Proportional Counting
Hg	Acid Digestion / ICP-MS
Ag	Acid Digestion Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
Al	
As	
B	
Ba	
Ca	
Cd	
Cr	
Fe	
K	
Li	
Na	
Ni	
P	
Pb	
Se	
Th	
Ti	
U (total)	
Zn	
Zr	
U (total)	Acid Digestion / Kinetic Phosphorescence Analysis (KPA)
H^+	Titration

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

Project / WP#: 69833 / N82456
ASR#: 0335
Client: S. Fiskum
Total Samples: 3 (liquids)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
17-1240	TI014-FEED	AP-105 Direct Feed (Caustic)	1.2794
DUP-1240	TI014-FEED	AP-105 Direct Feed (Caustic)	1.2778
17-1241	TI014-EFF Comp	AP-105 – Cs Removed (Caustic)	1.2789
DUP-1241	TI014-EFF Comp	AP-105 – Cs Removed (Caustic)	1.2804
17-1242	TI014-ELComp	AP-105 – Cs Eluant (Acidic)	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 8/28/17, dilution of Sample 17-1242 in deionized water performed by L. Grow on 8/17/17, and dilution of "as received" samples in 5% v/v HNO_3 performed by D. Cherkasov on 8/31/17, respectively.

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

Analyst:	D. Cherkasov/ G. Brown	**Analysis Date:** **Revision Date:**	08/31/2017 10/02/2017	**ICP File:**	C0721

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

M&TE:	☒	PerkinElmer 5300DV ICP-OES	SN: 077N5122002
	☐	Sartorius ME414S Balance	SN: 22406373
	☒	Mettler AT400 Balance	SN: 1113292667
	☒	Sartorius R200D Balance	SN: 39080042
	☐	Mettler AT201 Balance	SN: 192720-92
	☒	SAL Cell 2 Balance	SN: 8033311209
	☒	Lab 305 Balance	SN: N04143
	☐	Lab 309 Balance	SN: 10803210

Garrett N. Brown
Report Preparer

10/02/17 Revision
Date

Review and Concurrence

10/4/17
Date

Three aqueous samples submitted under Analytical Service Request (ASR) 0335 were analyzed by ICP-OES. Samples 17-1240 and 17-1241 were prepared in duplicate following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Sample 17-1242 was diluted with DI water in the hot cell. 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, 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. With the exception of iron (0.43 µg/mL) and zinc (1.63 µg/mL), 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. The iron and zinc concentrations were significant compared to the concentrations in all samples.

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 41% to 101%, and, with the exception of silver (41%), were within the acceptance criterion of 80% to 120%. 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):

The ASR requested duplicate analyses of all samples. Duplicates of samples 17-1240 and 17-1241 were prepared and analyzed. Due to limited sample volume, a single preparation of sample 17-1242 was analyzed once undiluted and twice at a 5X dilution. 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 4.6% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike (MS) sample (sample 17-1240 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. Recovery values for the AOI meeting this requirement ranged from 46% to 108%, and, with the exception of silver (46%), were within the acceptance criterion of 75% to 125%. 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.

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.

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.

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 17-1240. 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 0.4% to 8.7% and were within the acceptance criterion of ≤10%.

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

In addition to the MS sample, a post-digestion spike (A Component) was conducted on sample 17-1240. 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 92% to 106%, 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 17-1240. 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 94% to 103%, 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.

Report Revision (10/02/2017):

A revision to the original data report was initiated on 10/02/2017 to include the 102.4X dilution of sample 17-1242 that was performed by L. Grow on 08/17/2017 in the hot cell prior to analysis. No other sample results were revised.

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 >	8/31/2017	8/31/2017	8/31/2017	8/31/2017	8/31/2017	8/31/2017	8/31/2017	8/31/2017	8/31/2017
		Process Factor >	1.0	24.7	620.0	620.5	619.3	610.8	102.4	512.0	512.0
		RPL/LAB >	405 diluent	BLK-1240	17-1240 @25x	DUP-1240 @25x	17-1241 @25x	DUP-1241 @25x	17-1242	17-1242 @5x	17-1242 @5x rep
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	BLK-1240	TI014-FEED		TI014-EFF Comp		TI014-EL Comp		
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0030	0.030	Ag	--	--	--	--	--	--	--	--	--
0.0276	0.276	Al	--	--	14,300	14,800	14,600	14,400	50.4	[44]	[47]
0.1576	1.576	As	--	--	--	--	--	--	--	--	--
0.0120	0.120	B	--	--	82.5	81.7	[56]	[54]	[8.8]	[14]	[8.7]
0.0005	0.005	Ba	--	[0.032]	[0.39]	[0.43]	[0.33]	[0.38]	[0.46]	[0.44]	[0.33]
0.0233	0.233	Ca	--	[1.2]	[39]	[43]	[52]	[49]	24.3	[22]	[15]
0.0027	0.027	Cd	--	--	--	[1.8]	--	[2.8]	--	--	--
0.0047	0.047	Cr	--	--	353	370	364	360	[2.8]	--	[4.2]
0.0017	0.017	Fe	--	0.430	[5.8]	[4.0]	[5.8]	[6.7]	3.35	[3.9]	[4.0]
0.0500	0.500	K	--	[6.1]	3,930	4,020	3,940	3,900	558	557	537
0.0028	0.028	Li	--	--	--	--	--	--	--	--	--
0.0171	0.171	Na	--	[0.75]	141,000	145,000	141,000	139,000	4,770	4,710	4,610
0.0074	0.074	Ni	--	--	[31]	[29]	[35]	[30]	[1.6]	--	--
0.1021	1.021	P	--	--	[490]	[530]	[520]	[520]	--	--	--
0.0399	0.399	Pb	--	--	--	--	--	--	205	[200]	[200]
0.2271	2.271	Se	--	--	--	--	[200]	--	--	--	--
0.0120	0.120	Th	--	--	--	--	--	--	--	--	[7.0]
0.0014	0.014	Ti	--	--	--	--	--	--	--	--	--
0.0676	0.676	U	--	--	--	--	--	--	--	--	--
0.0066	0.066	Zn	--	1.63	[7.0]	[7.3]	[14]	[7.0]	8.33	[7.7]	[4.4]
0.0022	0.022	Zr	--	--	--	--	--	--	--	--	--
Other Analytes											
0.0002	0.002	Be	--	--	[0.18]	--	[0.17]	[0.19]	[0.041]	--	--
0.0546	0.546	Bi	--	--	--	--	--	--	[6.5]	--	--
0.0448	0.448	Ce	--	--	--	--	--	--	--	--	--
0.0071	0.071	Co	--	--	--	--	--	--	--	--	--
0.0036	0.036	Cu	--	[0.098]	[4.0]	[4.6]	--	--	11.2	[11]	[10]
0.0034	0.034	Dy	--	--	--	--	--	--	--	--	--
0.0010	0.010	Eu	--	--	--	--	--	--	--	--	--
0.0030	0.030	La	--	--	--	--	--	--	--	--	--
0.0023	0.023	Mg	--	--	--	--	--	--	[1.4]	--	--
0.0009	0.009	Mn	--	--	--	--	--	--	[0.19]	--	--
0.0094	0.094	Mo	--	--	[53]	[55]	58.4	[52]	--	--	--
0.0188	0.188	Nd	--	--	--	--	--	--	--	--	--
0.0170	0.170	Pd	--	--	--	--	--	--	--	--	--
0.0162	0.162	Rh	--	--	--	--	--	--	--	--	--
0.0158	0.158	Ru	--	--	--	--	--	--	--	--	--
0.1773	1.773	S	--	--	1,350	1,430	1,280	1,440	[67]	[92]	--
0.0919	0.919	Sb	--	--	--	--	--	--	--	--	--
0.0136	0.136	Si	--	--	99.1	109	[70]	[72]	18.5	[16]	[12]
0.0446	0.446	Sn	--	--	[34]	--	--	[30]	[6.1]	--	--
0.0003	0.003	Sr	--	--	--	[0.19]	[0.22]	[0.23]	[0.096]	--	--
0.0242	0.242	Ta	--	--	--	--	--	--	--	--	--
0.0412	0.412	Te	--	--	--	--	--	--	--	--	--
0.0885	0.885	Tl	--	--	--	--	--	--	--	--	--
0.0032	0.032	V	--	--	--	--	--	--	[0.33]	--	--
0.0249	0.249	W	--	--	[85]	[94]	[96]	[94]	--	--	--
0.0006	0.006	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%.

ASR 0335 Final from C0721R Fiskum ASR-0335 Normal BS & MS.xls

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

QC Performance 08/31/2017

Criteria >	≤ 20%	≤ 20%	≤ 20%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	17-1240 Dup	17-1241 Dup	17-1242 @5x rep	LCS/BS	17-1240 MS	17-1240 + AS-A	17-1240 + AS-B	17-1240 5-fold Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Ag				41	46	92		
Al	3.2	0.9		96	nr	105		0.4
As						103		
B	1.0			97	105	102		
Ba				96	101	103		
Ca				98	102	105		
Cd				93	107	102		
Cr	4.6	1.0		94	nr	104		0.3
Fe				96	102	105		
K	2.4	1.1	3.6	96	100	102		8.7
Li				101	104	105		
Na	3.0	1.2	2.1	96	nr	103		0.8
Ni				96	106	104		
P				97	97	103		
Pb				96	108	105		
Se						106		
Th							94	
Ti				96	101	103		
U				98	104		103	
Zn				95	103	106		
Zr				97	102	103		
Other Analytes								
Be				89	97	97		
Bi				80		96		
Ce				86			93	
Co						103		
Cu				97	107	105		
Dy							99	
Eu							93	
La				92	97		99	
Mg				96	103	104		
Mn				97	105	103		
Mo				95	97	101		
Nd				92	96		98	
Pd							88	
Rh							95	
Ru							94	
S	5.3	11.2		97	106		100	
Sb						99		
Si	9.2			88	105	105		
Sn						97		
Sr				99	107	106		
Ta						102		
Te							95	
Tl						92		
V				91	96	97		
W				96		103		
Y						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.

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

Project / WP#:	69833 / N82456
ASR#:	0335
Client:	S. Fiskum
Total Samples:	3 (Aqueous)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
17-1240	TI014-Feed	AP-105 Direct Feed (Caustic)	NA
DUP-1240	TI014-Feed	AP-105 Direct Feed (Caustic)	NA
17-1241	TI014-EFF Comp	AP-105 Cs Removed (Caustic)	NA
DUP-1241	TI014-EFF Comp	AP-105 Cs Removed (Caustic)	NA
17-1242	TI014-EL Comp	AP-105 Cs Eluant (Acidic)	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 8/28/17, dilution of Sample 17-1242 in deionized water performed by L. Grow on 8/17/17, and ICP-MS bench dilution in 2% v/v HNO_3 or 2% HCl performed by D. Cherkasov on 9/15/17, respectively.

Procedure: RPG-CMC-292, Rev. 0, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."

Analyst:	D. Cherkasov G. Brown	**Analysis Date:**	09/29/2017	**ICP File:**	M0030

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

10/28/17
Date

Review and Concurrence

10/28/17
Date

Three aqueous samples submitted under Analytical Service Request (ASR) 0335 were analyzed by ICP-MS. Samples 17-1240 and 17-1241 were prepared in duplicate following RPL procedure RPG-CMC-128 by diluting 1 mL to approximately 25 mL. Approximately 0.2 mL of sample 17-1242 was diluted with approximately 20 mL DI water in the hot cell to reduce radiation dose. Prior to analysis all samples were further diluted in 2% HNO_3 or 2% HCl containing 100 ppb Au to reduce Hg carryover in the ICP-MS system. 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. 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 0, *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 (Hg-202) data were normalized using the data for the closest IS mass (e.g., Bi-209). The Bi-209 IS recoveries ranged from 94.5% to 108%, 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 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 (2% HCl and 100 ppb Au) was also analyzed. Results for the diluent blank were also within the acceptance criteria.

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

A blank spike (BS) sample was prepared by spiking a 2% HCl and 100 ppb Au blank with an equivalent volume of the CCV-Hg-2ppb standard (1:1 ratio). The recovery for Hg-202 was 90.6%, which is within the acceptance criteria of 80% to 120% recovery.

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

Duplicates of samples 17-1240 and 17-1241, as well as a replicate of sample 17-1242, 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.

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. 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 HCl and 100 ppb Au) 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.

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 17-1240. 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):

Because no MS sample was required to be prepared, a post-digestion spike (PS-Hg) was conducted on sample 17-1240. 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 90.4%, 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 >	9/29/2017	9/29/2017	9/29/2017	9/29/2017	9/29/2017	9/29/2017	9/29/2017	9/29/2017
		Process Factor >	4.96	1.0	6.067E+06	2.983E+07	5.960E+06	2.894E+07	1.013E+04	1.015E+04
		RPL/LAB >	BLK-1240	Lab 405 Blank	17-1240 @ 6.067E+06x	Dup-1240 @ 2.983E+07x	17-1241 @ 5.960E+06x	Dup-1241 @ 2.894E+07x	17-1242 @ 1.013E+04x	Rep-1242 @ 1.015E+04x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	PB	Lab 405 Blank	TI014-Feed		TI014-EFF Comp		TI014-EL Comp	
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0081	0.081	Hg 202	--	--	--	--	--	--	--	--

Internal Standard % Recovery

Bi 209 (IS)	108%	97.7%	107%	105%	105%	105%	104%	103%

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 09/29/2017

Criteria >	≤ 20%	≤ 20%	≤ 20%	80%-120%	75%-125%	75%-125%	≤ 10%
QC ID >	17-1240 Dup	17-1241 Dup	17-1242 Rep	LCS/BS	MS (None)	17-1240 + PS-Hg	17-1240 5-fold Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Diff
Hg 202	--	--	--	90.6%		90.4%	--

Internal Standard % Recovery

Bi 209 (IS)	105%	105%	103%	101%		108%	107%

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.

Pacific Northwest
NATIONAL LABORATORY

Client:	S. Fiskum	Report Date:	10/9/2017
		Analysis Date:	9/26/2017
			9/28/2017

Subject: Hydroxide and Hydrogen Ion Analyses for: Aqueous Samples
Project: 69833 WP # N79882

ASR: **0335** Rev-0 Procedure: RPG-CMC-228, Rev 0.1
Sample ID. 17-1240 thru 17-1242

Hydroxide analysis was performed for diluted sample aliquots of 2 aqueous samples (17-1240 and 17-1241), provided on ASR 0335. 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 0.0957 M HCl (Standardized HCl was prepared and documented on Chem Rec 232, prepared on 3/15/2017). 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. Samples were analyzed in duplicate therefore two pH measuresments are given per sample for each inflection point.

Sample ID	1st Inflection point	2nd Inflection point	3rd Inflection point
17-1240	11.2	7.1	3.8
17-1240 Dup	11.1	7.2	3.7
17-1241	11.3	7.7	3.9
17-1241 Dup	11.1	7.2	3.9
17-1242	9.8	NA	NA
17-1242 Dup	10.0	NA	NA

Hydrogen Ion analysis was performed using a diluted sample aliquot of 17-1242. The sample was diluted by 102.4x to lower the dose to a level that could be handled in a CA fume hood. The samples were also analyzed by manual titration for the acid constituents content following procedure RPG-CMC-228. Sample size was 0.100 mL of sample added to 2.5mL DI water. The titrant used was 0.0101M NaOH (Standardized NaOH was prepared and documented on Chem Rec 232, prepared on 3/15/17 and diluted on 9/26/17--restandardized on 9/26/17). The final H^+ molarity result has been corrected for the Hot Cell dilution.

The only inflection point for 17-1242 was at pH 9.8 and 10.0

Following is the report summary and the sample results calculated from the raw data. A copy of the titration

Prepared by: Date: 10/30/17
Reviewed by: Karl N. Pool Date: 10/30/17

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

ASR # **0335**

WP# N82456

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

Equip # Beckman Coulter 560, SN#110650046

Report Summary for ASR # -- 0335
Revision # **Rev-0**

Report Date: 10/9/2017
Analysis Date: 9/26/2017
9/28/2017

				Concentration, moles / Liter		
		Diluted		First Point	Second Point	Third Point
RPG #	**Client ID**	**Initial pH**	**OH conc ug/mL**	**Molarity**	**Molarity**	**Molarity**
17-1240	**TI014-FEED**	**12.27**	1.79E+04	**1.05**	**1.34**	**0.77**
17-1240 Dup	**TI014-FEED**	**12.19**		**1.05**	**1.24**	**0.77**
17-1241	**TI014-EFF Comp**	**12.18**	1.79E+04	**1.05**	**1.15**	**0.67**
17-1241 Dup	**TI014-EFF Comp**	**12.16**		**1.05**	**1.15**	**0.77**
17-1242	**TI014-ELComp**	**8.10**	NA	**1.03**		
17-1242 Dup	**TI014-ELComp**	**7.99**	NA	**1.03**		

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: 10·30·17
Reviewer: Karl N. Pool 10/30/17

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev # 0335 Rev-0
Client: S. Fiskum
Project: 69833 WP# N82456

Report Date: **10/9/2017**
Analysis Date: **9/26/2017**
9/28/2017

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: 10/30/17

Titrant	Molarity	Chem Rec#
HCl	**0.0957**	**232**

OH

RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	**Diluted** Initial pH reading	**1st Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
17-1240	TI014-FEED		NA	0.100	NA	NA	NA	12.27	1.10	11.17	0.105	1.053
17-1240 Dup	TI014-FEED		NA	0.100	NA	NA	NA	12.19	1.10	11.08	0.105	1.053
17-1241	TI014-EFF Comp		NA	0.100	NA	NA	NA	12.18	1.10	11.25	0.105	1.053
17-1241 Dup	TI014-EFF Comp		NA	0.100	NA	NA	NA	12.16	1.10	11.11	0.105	1.053

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	1-Jan-18

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

Slope
96.4%

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev # 0335 Rev-0

WP# N82456

Procedure:

Alkalinity of Aqueous Solutions, Leachates and Supernates

By Manual Titration

Equip # Beckman Coulter 560, SN#110650046

Titrant	Molarity
HCl	0.0957

RPG #		Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base
17-1240		0.100	2.500	7.090	0.134	1.340	3.300	3.770	0.077	0.766
17-1240 Dup		0.100	2.400	7.170	0.124	1.244	3.200	3.720	0.077	0.766
17-1241		0.100	2.300	7.690	0.115	1.148	3.000	3.920	0.067	0.670
17-1241 Dup		0.100	2.300	7.160	0.115	1.148	3.100	3.860	0.077	0.766

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev # 0335 Rev-0
Client: S. Fiskum
Project: 69833 WP# N82456

Report Date: **10/9/2017**
Analysis Date: **9/26/2017**
9/28/2017

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: 10/30/17

Titrant	Molarity	Chem Rec#
NaOH	**0.0101**	**232**

RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	**Diluted** Initial pH reading	H+ **1st Equivalence** Point Titrant Vol. (mL)	pH	Found millimoles acid	Molarity acid
17-1242	TI014-ELComp		102.4	0.100	NA	NA	NA	8.07	0.10	9.77	0.001	1.034
17-1242 Dup	TI014-ELComp		102.4	0.100	NA	NA	NA	7.99	0.10	9.95	0.001	1.034

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	1-Jan-18

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

Slope
96.4%

Battelle Pacific Northwest Laboratory
Nuclear Chemistry and Engineering Group

ASR # and Rev # 0335 Rev-0

WP# N82456
0

Procedure:

Alkalinity of Aqueous Solutions, Leachates and Supernates

By Manual Titration

Equip # Beckman Coulter 560, SN#110650046

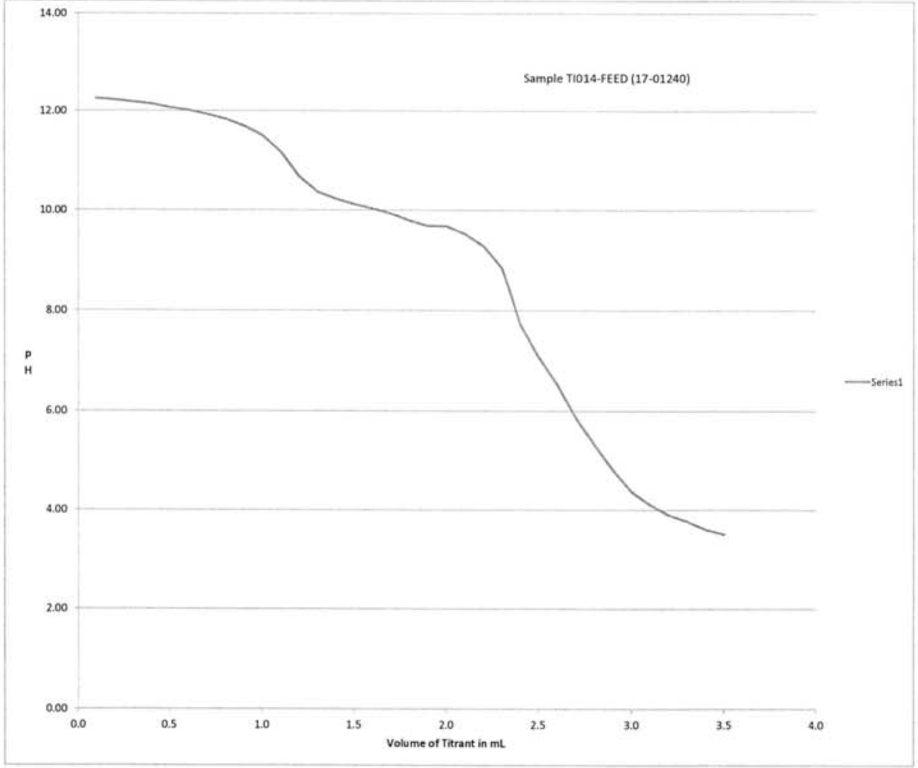
Titrant	Molarity
NaOH	0.0101

RPG #		Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles acid	Molarity acid

3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles acid	Molarity acid

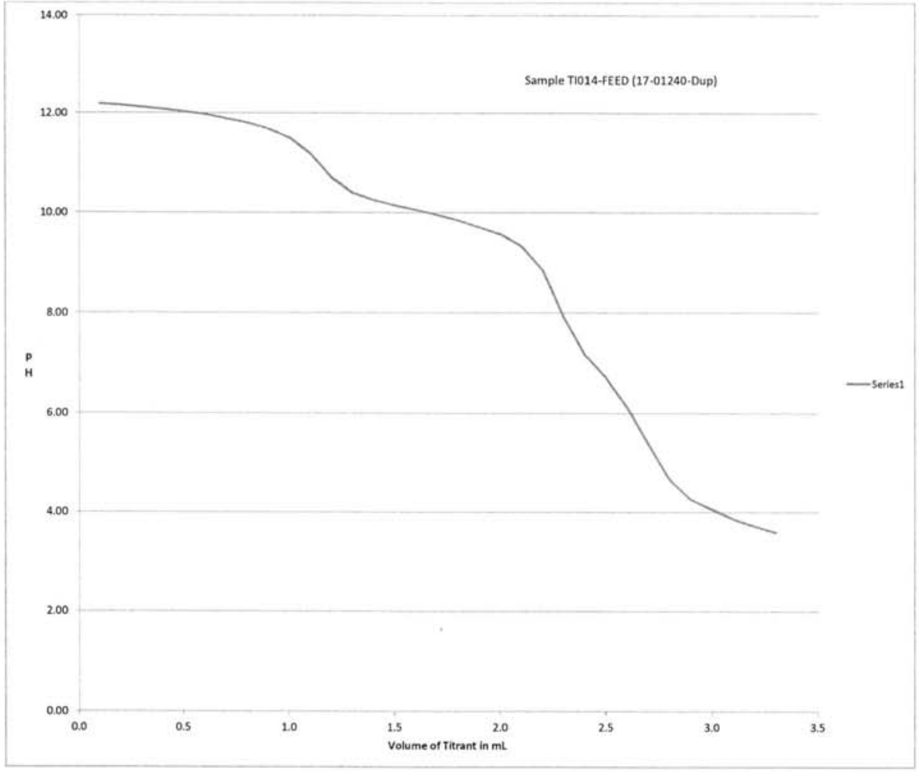
17-1240
TI014-FEED

Titrant Volume (mL)	pH
0.1	12.25
0.2	12.22
0.3	12.18
0.4	12.14
0.5	12.07
0.6	12.02
0.7	11.94
0.8	11.84
0.9	11.70
1.0	11.51
1.1	11.17
1.2	10.67
1.3	10.37
1.4	10.23
1.5	10.12
1.6	10.03
1.7	9.93
1.8	9.80
1.9	9.69
2.0	9.68
2.1	9.53
2.2	9.29
2.3	8.85
2.4	7.73
2.5	7.09
2.6	6.53
2.7	5.87
2.8	5.31
2.9	4.81
3.0	4.37
3.1	4.10
3.2	3.90
3.3	3.77
3.4	3.61
3.5	3.51

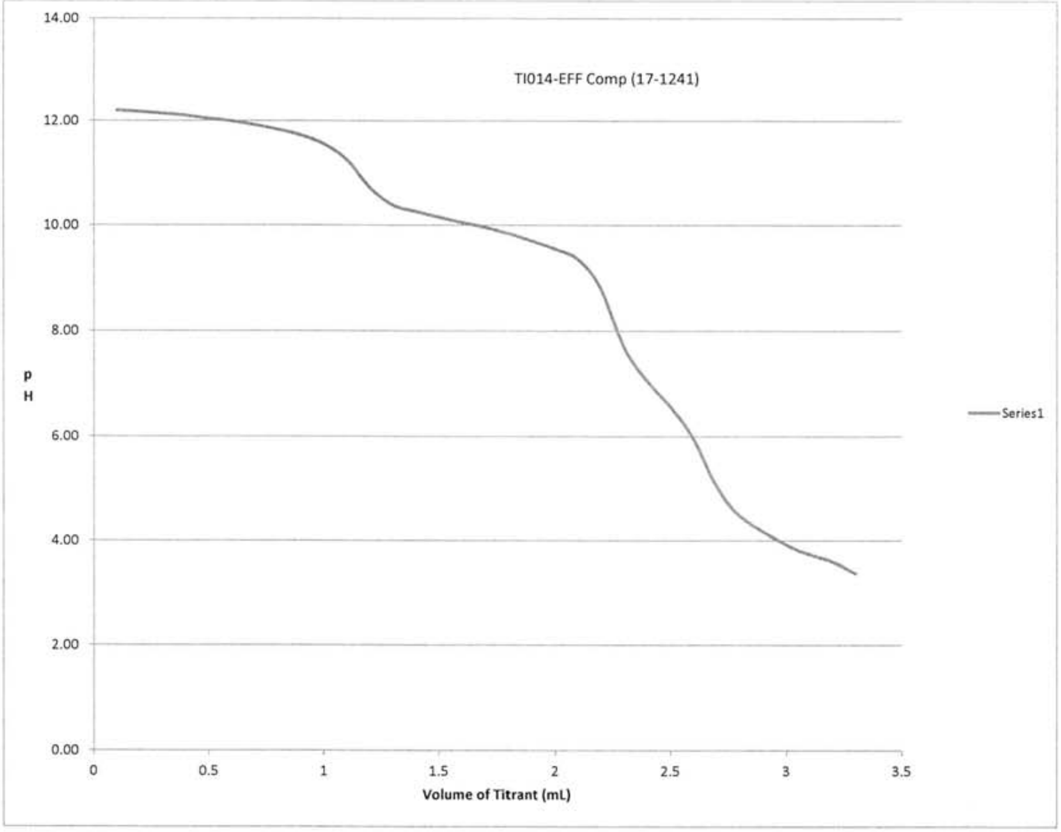


17-1240 Dup
TI014-FEED

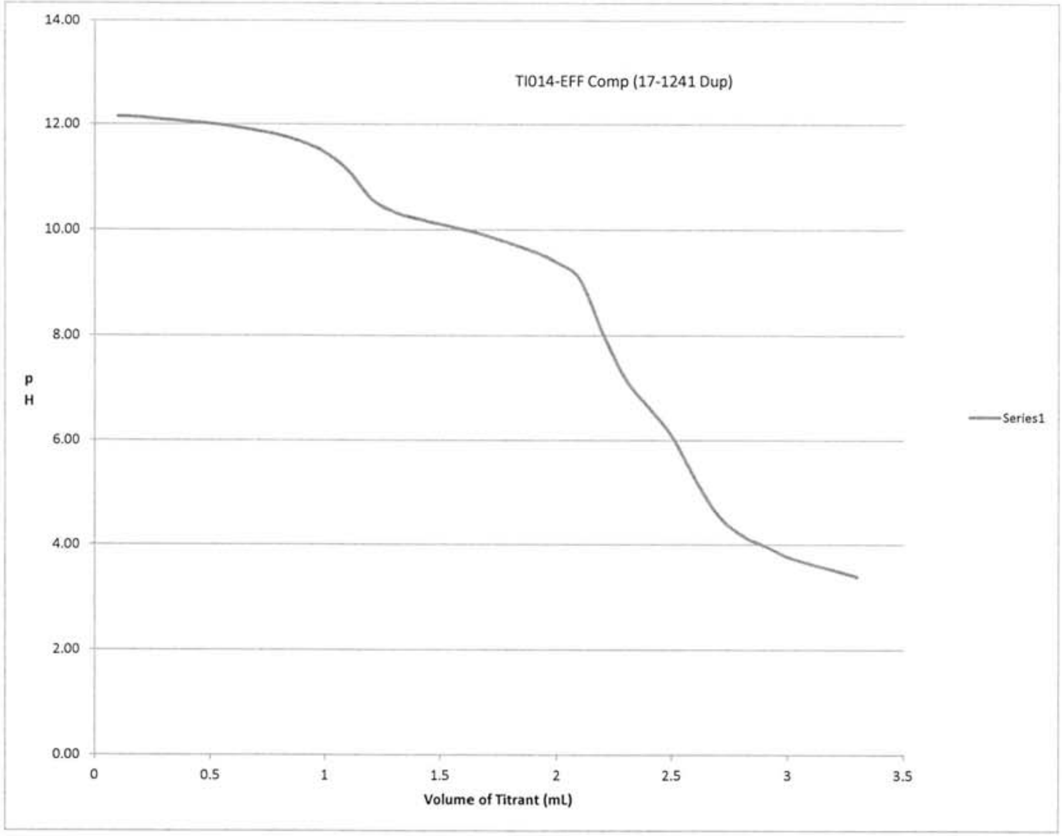
Titrant Volume (mL)	pH
0.1	12.19
0.2	12.16
0.3	12.12
0.4	12.08
0.5	12.03
0.6	11.98
0.7	11.89
0.8	11.81
0.9	11.69
1.0	11.50
1.1	11.18
1.2	10.70
1.3	10.39
1.4	10.25
1.5	10.15
1.6	10.06
1.7	9.96
1.8	9.85
1.9	9.71
2.0	9.57
2.1	9.33
2.2	8.86
2.3	7.91
2.4	7.17
2.5	6.71
2.6	6.11
2.7	5.37
2.8	4.66
2.9	4.26
3.1	3.86
3.2	3.72
3.3	3.59



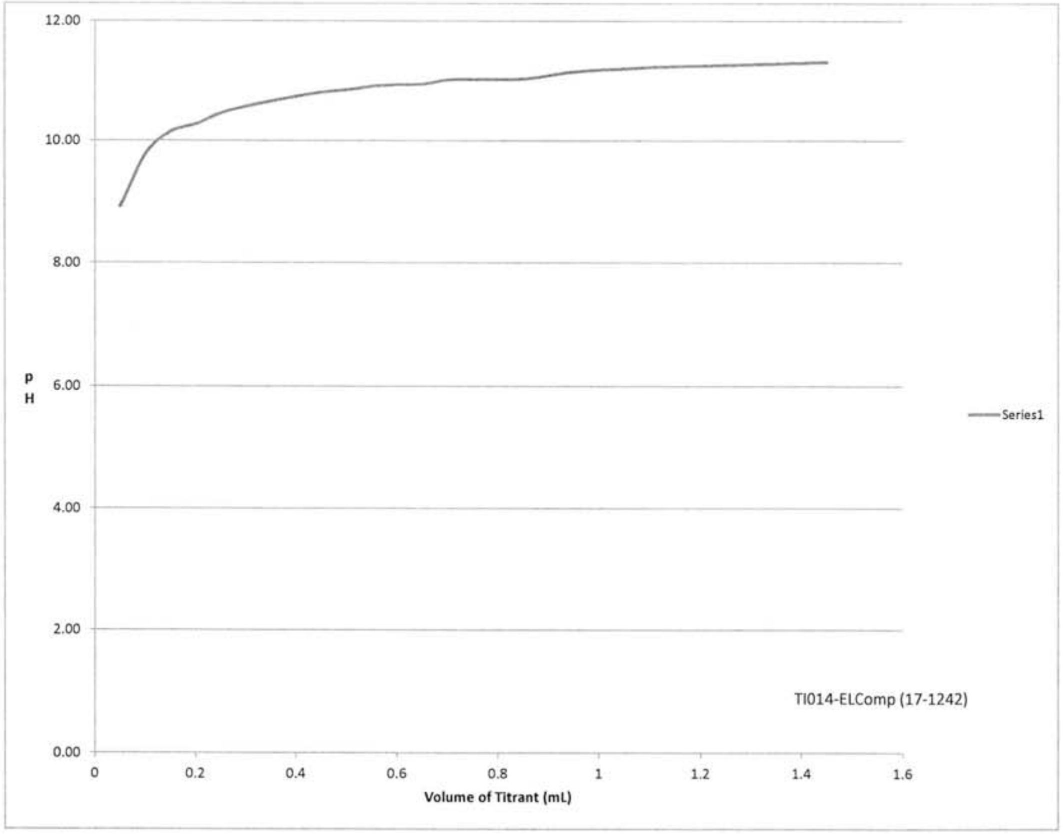
Titrant Volume (mL)	pH
0.1	12.20
0.2	12.17
0.3	12.14
0.4	12.10
0.5	12.04
0.6	11.99
0.7	11.92
0.8	11.83
0.9	11.72
1	11.55
1.1	11.25
1.2	10.71
1.3	10.38
1.4	10.26
1.5	10.15
1.6	10.05
1.7	9.96
1.8	9.84
1.9	9.70
2	9.55
2.1	9.35
2.2	8.79
2.3	7.69
2.4	7.05
2.5	6.57
2.6	5.95
2.7	5.04
2.8	4.47
3	3.92
3.1	3.74
3.2	3.60
3.3	3.37



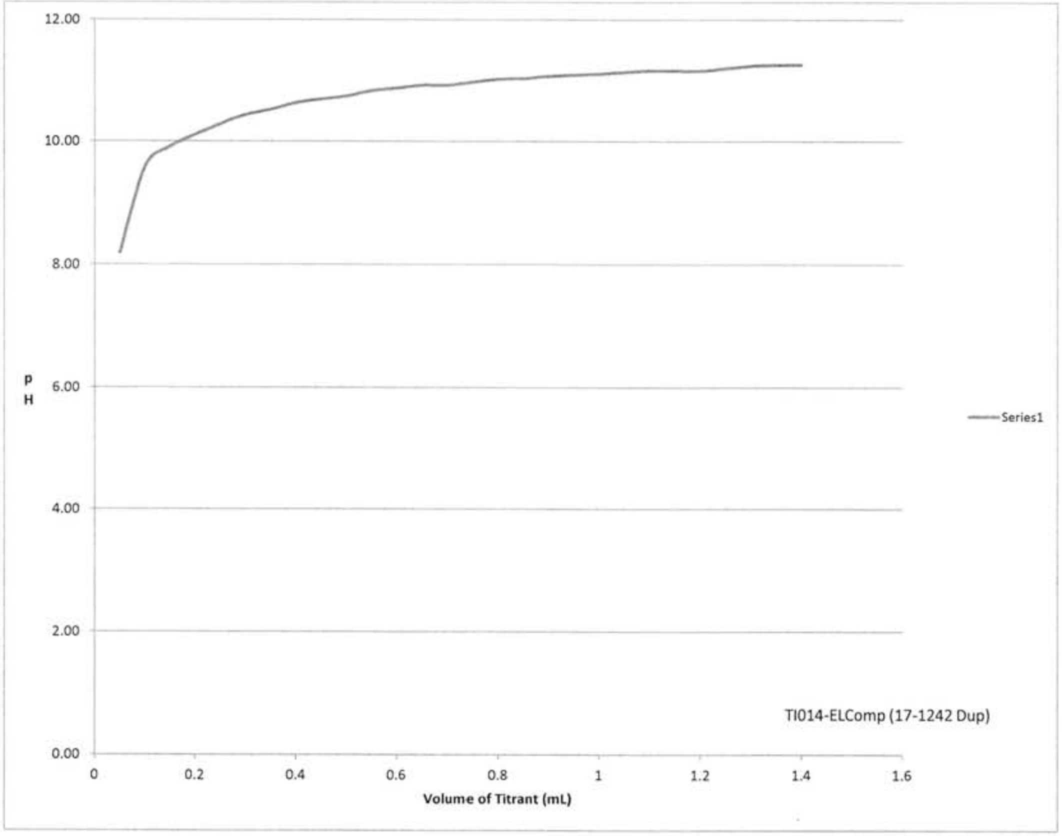
Titrant Volume (mL)	pH
0.1	12.15
0.2	12.13
0.3	12.09
0.4	12.05
0.5	12.01
0.6	11.95
0.7	11.88
0.8	11.79
0.9	11.66
1	11.46
1.1	11.11
1.2	10.58
1.3	10.33
1.4	10.20
1.5	10.10
1.6	10.00
1.7	9.88
1.9	9.59
2	9.38
2.1	9.06
2.2	8.03
2.3	7.16
2.4	6.62
2.5	6.08
2.6	5.25
2.7	4.56
2.8	4.19
2.9	3.97
3	3.76
3.1	3.63
3.2	3.51
3.3	3.39



Titrant Volume (mL)	pH
0.05	8.91
0.1	9.77
0.15	10.14
0.2	10.27
0.25	10.45
0.3	10.56
0.35	10.65
0.4	10.73
0.45	10.80
0.5	10.84
0.55	10.90
0.6	10.93
0.65	10.94
0.7	11.01
0.75	11.02
0.85	11.03
0.95	11.15
1.05	11.20
1.15	11.24
1.25	11.26
1.45	11.31



Titrant Volume (mL)	pH
0.05	8.19
0.1	9.59
0.15	9.91
0.25	10.28
0.3	10.43
0.35	10.52
0.4	10.63
0.45	10.69
0.5	10.74
0.55	10.83
0.6	10.87
0.65	10.92
0.7	10.92
0.75	10.97
0.8	11.02
0.85	11.03
0.9	11.07
1	11.11
1.1	11.16
1.2	11.16
1.3	11.24
1.4	11.26



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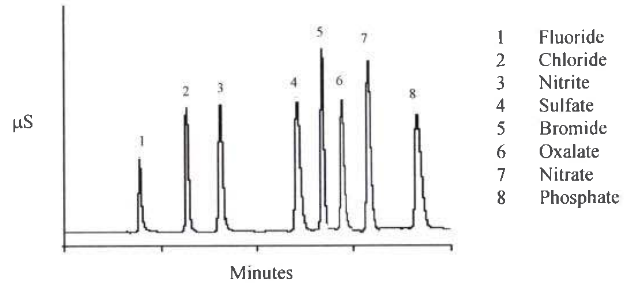
Common Inorganic Anions
Dionex AS18 Column; Hydroxide Gradient



Client: S. Fiskum ***ASR #:*** 0335
Project #: 69833 ***# Samples:*** 2 Liquids
Charge Code: N82456

*** RPL Numbers: 17-1240 & 17-1241 ***

Revision 2

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	9-7-17, 9-8-17, and 9-13-17
Calibration Date	08-31-17
Cal/Ver Stds Prep Date	08-29-17
Excel Data File	ASR-0335 Fiskum IC Results-Rev1.xls
M&TE Numbers	IC System (M&TE) SN 09080421
	Balance: 22406373
All Analysis Records	Chemical Measurement Center 98620: RIDS IC System File (IC-0237)

Prepared By — Date 2·28·18

Reviewed By — Date 2/28/18

IC Report

Sample Results

See Attachment: Sample Results ASR 0335

Revision 1

The report has been revised to correct the sulfate results. Sulfate results were originally reported non-detect but measurable quantities of sulfate are present in the samples.

Revision 2

The report has been revised to correctly report the analytical spike results.

Sample Analysis/Results Discussion

Two liquid samples were analyzed. The sample data is reported in µg/mL and samples required dilutions of up to 29,282 fold in order to obtain valid anion results within the calibration range. All results have been adjusted for all analytical dilutions. The analytes of interest for these samples include chloride, nitrite, nitrate, phosphate, oxalate and sulfate. The estimated method detection limits (MDL) are provided, and are based on the estimated quantitation limit (EQL), which is one-tenth of the lowest calibration standard (adjusted for the dilutions used for reporting the results).

Data Limitations

There are no limitations regarding this data. All QC sample requirements were met.

Quality Control Discussion

The method performance is evaluated against the acceptance criteria established by the analysis procedure and the Analytical Support Operations QA Plan, ASO-QAP-001.

IC Workstation QC Results

Dilution Blank: One dilution blank was analyzed with the sample batch. No anions were detected above the MDL.

Duplicate: Requested per the client, duplicate analysis was performed on both samples. The relative percent difference (RPD) is reported for all analytes which were measured at or above the EQL. The reported RPDs ranged from 0.63%-6.4%, which meets the ASO's QA Plan acceptance criteria of <20%.

Instrument LCS: The routine instrument LCS analyzed with the run met the acceptance criteria of 80% to 120% recovery (See attached sample results).

Analytical Spike (AS) (Accuracy): Analytical spikes were prepared, as no sample preparation was required for this analysis. Aqueous samples were analyzed as received. Results are reported using the 14641x dilution of the two liquid samples by adding a known concentration of mid-range multi-mix standard, "CCV 082917". The analytical spike recoveries ranged from 98% to 105%, meeting the method acceptance criteria of 75% to 125%.

Continuing Calibration Verification (CCV) / Continuing Calibration Blank (CCB): Numerous CCVs and CCBs were analyzed throughout the analysis run and the ICV/ICB are included in these results(See attachment "QC Sample Results and Performance"). All CCVs and CCBs affecting reported data produced results within the acceptance criteria of the QA Plan (i.e., 90% to 110% recovery for the CCV and results less than estimated quantitation limit or less than 5% of reported sample result for the CCB).

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 0335

		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
17-1240	TI014-FEED	100	4,340		1,500	63,400		200	1,800		12	192		2,900	113,000		15	1,050	
17-1241	TI014-EFF Comp	100	4,380		2,900	61,300		200	1,900		12	176		5,900	114,000		15	971	
Dilution Blank	Dilution Blank (9/7/17)	0.08	0.08	U	0.10	0.10	U	0.15	0.15	U	0.10	0.10	U	0.20	0.20	U	0.12	0.12	U
Dilution Blank	Dilution Blank (09/13/17)	0.08	0.08	U	0.10	0.10	U	0.15	0.15	U	0.10	0.10	U	0.20	0.20	U	0.12	0.12	U

Sample QC Results ASR 0335

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
17-1240	Sample	4,340	--	63,400	--	1,800	--	192	--	113,000	--	1,050	--
	Duplicate | RPD	4,430	2.1	63,800	0.63	1,800	0	197	2.6	114,000	0.9	985	6.4
17-1241	Sample	4,380	--	61,300	--	1,900	--	176	--	114,000	--	971	--
	Duplicate | RPD	4,330	1.1	62,300	1.6	2,000	5.1	179	1.7	111,000	2.7	962	0.93

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
17-1240	Sample	4,340	--	63,400	--	1,800	--	192	--	113,000	--	1,050	--
	AS Sample *	1.65	100	4.12	98	3.11	101	2.05	103	7.47	104	2.52	101
17-1241	Sample	4,380	--	61,300	--	1,900	--	176	--	114,000	--	971	--
	AS Sample *	1.68	102	4.11	101	3.15	105	2.09	105	7.33	99	2.52	101

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
9/7/2017 11:00	LCS 082917	98.9	96.3	102.8	104.2	96.5	103.8
9/13/2017 13:41	LCS 082917	99.5	95.8	101.5	NA	100.8	108.1

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
NA = Oxalate tends to fall out of solution after a short time period, no oxalate results are reported associated with this run date.
* = Dilution factor is not applied to this result.

Project Number: 69833
Charge Code: N79882
ASR Number: 0335
Client: S. Fiskum
Total Samples: 2 liquid

	Sample
RPL Numbers	17-1240 and 17-1241
Client IDs	TI014-FEED and TI014-EFF Comp

Analysis Procedure	RPG-CMC-386 Rev. 1, "Carbon Measured in Solids, Sludge, and Liquid Matrices"
Prep Procedure	None
Analyst	C. Rutherford
Analysis Date	October 18, 2017
CCV Standards	TIC/TOC CMS# 509806 and 521011
BS/LCS/MS Standards	TIC/TOC CMS# 520858 and 520712
Excel Data File	ASR 0335
M&TE Numbers	Carbon System (WD36639, RPL/701) Balance : Sartorius R200D, S/N 30809774
All Analysis Records	System File TC-18-1

Karl N. Pool For C. Rutherford 11/13/17
Prepared By Date

Karl N. Pool 11/13/17
Reviewed By Date

Table 1: TOC/TIC Results for ASR 0335

Results

TIC in Sample **17-1240** (mg C/L):	5800
MDL (mg C/L):	96
EQL:	480
TOC in Sample **17-1240** (mg C/L):	2610
MDL (mg C/L):	96
EQL:	480
TIC in Sample **17-1240-Dup** (mg C/L):	5890
MDL (mg C/L):	120
EQL:	601
TOC in Sample **17-1240** (mg C/L):	2590
MDL (mg C/L):	120
EQL:	601
17-1240 TIC RPD:	1.5%
17-1240 TOC RPD:	0.8%

TIC in Sample **17-1241** (mg C/L):	5260
MDL (mg C/L):	240
EQL:	1200
TOC in Sample **17-1241** (mg C/L):	1810*
MDL (mg C/L):	240
EQL:	1200
TIC in Sample **17-1241-Rep** (mg C/L):	5600
MDL (mg C/L):	240
EQL:	1200
TOC in Sample **17-1241-Rep** (mg C/L):	2770
MDL (mg C/L):	240
EQL:	1200
17-1241 TIC RPD (%)	6.3%
17-1241 TOC RPD (%)	**41.6%**

* This results is suspect - see narative for more detail

Sample Analysis/Results Discussion

Two liquid samples were submitted under Analytical Service Request (ASR) 0335 for total inorganic and total organic carbon analysis. The analysis was 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. The analyses were performed following procedure RPG-CMC-386, Rev. 1, *Carbon Analyses in Solids, Sludge and Liquid Matrices*

The two samples were analyzed in duplicate, with sample 17-1241 also run in replicate. Sample 17-1240 was selected for the analytical spike. For sample 17-1241 the original sample and the replicate sample are compared in Table 1 for RPD. The sample results are corrected for the contribution from the system blank, as per procedure RPG-CMC-386, Rev. 1. All data are reported as µg C/mL of sample.

Data Limitations

None

Quality Control Discussion

The calibration and QC sample standards for the TOC initial/continuing calibration verification check (ICV/CCV) sample is a 1000 µg/mL solution of total organic carbon standard. The calibration and QC sample standards for the TIC initial/continuing calibration verification check (ICV/CCV) sample is a 1000 µg/mL total inorganic 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 initial and continuing calibration verification samples (ICV/CCV), initial and continuing calibration blanks (ICB/CCB), laboratory duplicates for each sample, a laboratory control sample/blank spike (LCS/BS), and an analytical spike (AS). The work was performed in one batch.

Two blanks are run at the beginning of each batch and after ICV/CCV. The blanks must be <EQL. The banks run in the batch are <EQL.

Initial Calibration Check and Continuing Calibration Verification Standards:

> The calibration of the coulometer analysis system was checked by calibration verification standards analyzed at the beginning and end of the analysis run. TOC results for the two ICVs runs were 103.3% and 103.2% recovery, and for the two TIC ICVs runs the results were 97.2% and 99.7% recovery, within the acceptance criterion of 90% to 110%. The TOC result for the CCV run was 99.0% recovery and the TIC CCVs run was 102.1% 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 100.7% recovery and the TOC LCS/BS result was 100.1% recovery, meeting the acceptance criteria of 75% to 125%.

Duplicate/Replicate: Precision of the carbon measurements is demonstrated by the relative percent difference (RPD) between sample and duplicate/replicate. Sample 17-1240 TIC RPD was 1.5% and the TOC was 0.8%. The RPD for sample 17-1241 TIC was 6.3% and the TOC RPD was 41.6%. The RPD for 17-1241 TOC was the only RPD that did not meet the acceptance criteria of ≤20%. *Note: For sample 17-1241, the duplicate result (2715 mg/L) and replicate result (2769 mg/L) have an RPD of 2.0%. The result reported for 17-1241 is likely suspect. The result for 17-1241-rep reported in Table 1 is likely a more accurate result.*

Analytical Spike (AS): The accuracy of the carbon measurements can be estimated by the recovery from the AS. One AS sample was run with the batch. Sample 17-1240 was selected. The results for the analytical spike for TIC is 98.8% recovery and for the TOC, 98.2% recovery. The AS recovery for the TOC and TIC results meets 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#:	69833/ N82456
ASR#:	0335.00
Client:	SK Fiskum
Total Samples:	3

RPL ID	Client Sample ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

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:	August 25, 2017 @ 9:00 am
Analysis Date or Date Range:	August 25, 2017
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*17-1240 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 G, & T*

T Trang-Le / 9/15/17 LR Greenwood / 9/15/17

Prepare **Date** **Reviewer** **Date**

Page 1 of 3

SAMPLE RESULTS

Activities for all gamma emitters detected in this sample are presented in an attached Excel spreadsheet for ASR 0335.00. All sample results for all 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 0335 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

All three samples were received from the Shielded Analytical Laboratory (SAL).

Sample 17-1242 (TI014-EL-Comp) was prepared for dilution in the hot cell by filling the diluent vial with 4 aliquots of ~5mL each of DI water in lab 309. The diluent vial was loaded into the hot cell where ~0.2mL of AP-105 Cs Eluent was added to the diluent vial. The vial was then loaded out of the hot cell clean.

Samples 17-1240 and 17-1241 (TI014-FEED and TI014-EFF-Comp) were prepared by pipetting 2 mL of sample solutions provided to the ASO.

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. The most recent background is subtracted from all sample counts. Samples 17-1240 and 17-1242 had much higher dose rates than sample 17-1241. Consequently, these two samples were counted on a remote position on our track detector (T) while sample 17-1241 was counted on the face of detector G. We are not calibrated for a 2 mL geometry on detector T at the remote position (P20) used to count samples 17-1240 and 17-1242. However, this detector counts the glass scintillation vials through the side such that the height of liquid in the vial does not have any significant impact on the calibration geometry at such remote distances. Consequently, the calibration using 10 mL of liquid should be very close to that using 2 mL of liquid.

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 0335.

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

filename 17-1240 Fiskum.xls
9/15/2017

Client: Fiskum Project: 69833
ASR 0335 WP: N82456

Prepared by: T Trang-le 9/15/2017

Technical Reviewer: LRGreenwood 9/15/2017

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

M&TE: Gamma detectors G, T
Count dates: 25-Aug-17

		Measured Activity, µCi per ml ± 1s											
Sample	Lab ID	**Co-60**		**Sn/Sb-126**		**Cs-137**		**Eu-152**		**Eu-154**		**Am-241**	
TI014-FEED	17-1240	< 3.2E-3		< 1.1E-2		**1.22E+02**	**± 2%**	< 6.3E-3		< 1.2E-2		< 5.4E-1	
TI014-EFF-Comp	17-1241	**5.93E-04**	**± 2%**	**1.75E-05**	**± 2%**	**5.30E-04**	**± 2%**	**6.07E-06**	**± 9%**	**1.35E-04**	**± 2%**	**3.72E-04**	**± 5%**
TI014-ELComp	17-1242	< 8.6E-2		< 1.5E-1		**2.43E+03**	**± 2%**	< 3.3E-1		< 2.4E-1		< 1.3E+1	

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#:	69833/N82456/N79882
ASR#:	0335
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

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
Total Alpha and Beta Preparation Procedure:	RPG-CMC-4001, Rev. 1, *Source Preparation For Gross Alpha and Gross Beta Analyses.*
Technician/Analyst:	LP Darnell, (09/20/2017)
Spike Standard ID's	R-693-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	T. Trang-Le and CZ Soderquist, (09/21/2017)
Separation Procedure:	RPG-CMC-476, Rev. 0, *Strontium Separation Using Eichrom Strontium Resin*
Spike Standard ID:	R-693-b-2 (Sr-90)
Separation Date:	09/20/2017 @ 09:45 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:	09/20/2017
Analysis Date or Date Range:	09/21/17 & 09/29/17 (first count), 09/26/17 & 10/02/17 (second count)
Technician/Analyst:	T. Trang-Le & CZ Soderquist
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls
ASO Project 98620 File:	File Plan 5871: T 69833 0335: Sample preparation and analysis records; T-4.4 LSC 3100 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, Serial # DG08061340, RPL 425, Tri-Carb 2700TR software version 1.04 dated 9/99.

T Trang-le / 11/15/17
Preparer **Date**

/ 11/15/17
Reviewer **Date**

SAMPLE RESULTS

See attached data report, Sample Results for ASR 0335. All sample data are reported in µCi/mL with a 1-σ uncertainty (see Comments).

Sample preparation, separation, mounting, and counting

All three samples submitted under Analytical Service Request (ASR) 0335 were analyzed on duplicate for Sr-90 by chemical separation and liquid scintillation counting. All the samples were prepared in RPL/420. Aliquots of the acid digestions and pre-diluted TI014-EL-Comp were used for radioanalytical analyses; only Sr-90 data are included in this report.

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in samples prepared in RP/420 and include a lab blank (LB), and sample duplicate. Additional laboratory QC samples were prepared prior to separations; these include a laboratory separation blank, a reagent blank spike (RS), and a matrix spike (i.e., addition of Sr-90 standard to an aliquot of one of the samples).

Instrument Calibration Control

Laboratory Preparation Blank and Laboratory separations blank (LB):

There are no acceptance criteria for LB (see Comments).

Blank Spike (RS) – reagent spike:

The RS recovery of 101% and 96% meet the procedure acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 93% and 87% meet the acceptance criterion of 75% to 125% recovery. Note: the MS sample was prepared "after" digestion (see comments), by adding a known Sr-90 standard quantity to an aliquot of 17-1240 (TI014-FEED).

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 1-5% 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\17-1240 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 RS&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 0335

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

Plutonium 238, 239+240 Analysis

Project / WP#:	69833/N82456/N79882
ASR#:	0335.00
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

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, (08/31/2017)
Spike Standard ID's	R-687-a-2 (Pu-239), R-693-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le (09/01/2017)
Plutonium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*
Technician/Analyst:	LP Darnell, (10/16/2017)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, (10/16/2017)
Spike and Tracer Standard ID's:	R-687-a-2 (Pu-239), R-688-a-3 (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:	October 9-10, 2017
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 69833: 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 / 11/15/17
Preparer **Date**

/ 11/15/17
Reviewer **Date**

Page 1 of 3

Sample Results

See attached data report, Sample Results for ASR 0335. All data are reported in units of µCi per mL with a 1-σ uncertainty.

Sample Preparation, Separation, Mounting and Counting Methods

All three samples submitted under Analytical Service Request (ASR) 0335 were analyzed in duplicate for plutonium by Alpha Spectrometry. All the samples were prepared in RPL/420. Aliquots of the acid digestions and pre-diluted TI014-EL-Comp were used for radioanalytical analyses; only Pu-AEA data are included in this report.

Following the digestion process of samples TI014-FEED and TI014-EFF-Comp along with the pre-diluted TI014-EL-Comp, 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 October 9-10, 2017; 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. Gross alpha and gross 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 82% to 98%.

Laboratory Preparation Blank (PB):

There is no PB blank for this batch of sample. There are no acceptance criteria for Laboratory blank.

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

The BS recovery of 98% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 94% (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 17-1240 (TI014-FEED) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

The Pu-238 sample 17-1241 and duplicate pair was 9%, within DQO. The duplicate pairs for sample 17-1240 (22% RPD) and 17-1242 (40% RPD) were outside of acceptance limits. The RPD for Pu-239+240 is 3-17%, 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 0335.

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

Neptunium 237 Analysis

Project / WP#:	69833/N82456/N79882
ASR#:	0335.00
Client:	SK Fiskum
Total # of Samples:	3

Client Sample ID	RPL ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

Analysis Type:	AEA – Np-237
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, Rev0, *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, (08/31/2017)
Spike Standard ID's	R-687-a-2 (Pu-239), R-693-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le (09/01/2017)
Neptunium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium- 90*
Technician/Analyst:	LP Darnell, (10/16/2017)
Spike Standard ID's:	R-686-a-39 (Np-237)
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, (10/16/2017)
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as counting dates
Analysis Date or Date Range:	October 16-17, 2017
Technician/Analyst:	T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 69833: 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 / 11/15/17
Preparer **Date**

/ 11/15/17
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0335. 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

All three samples submitted under Analytical Service Request (ASR) 0335 were analyzed in duplicate for neptunium by Alpha Spectrometry. All the samples were prepared in RPL/420. Aliquots of the acid digestions and pre-diluted TI014-EL-Comp were used for radioanalytical analyses; only Np-AEA is reported in this report.

Following the digestion process of samples TI014-FEED and TI014-EFF-Comp along with the pre-diluted TI014-EL-Comp, 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. The samples were counted on October 16-17, 2017; no decay corrections were made.

Alpha and beta analyses were performed on each sample for checking the internal consistency of the Np alpha isotopic data. Gross alpha and gross 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 a laboratory blank, sample duplicate. Additional QC samples were prepared prior to separations; these include a laboratory blank, and 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) and Laboratory separations blank (LB):

There is no PB blank for this batch of sample. There are no acceptance criteria for Laboratory blank.

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

The RS recovery of 96% meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 100% 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 17-1240 (TI014-FEED) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Duplicate results are required to agree within ≤20% RPD. Duplicate results were 1-4% RPD. The Np-237 activity detected in the samples all have 1-σ counting error of 10% or greater. 1-σ counting error at these levels indicates the activity measured in the samples is nearing the minimum detectable activity for the samples.

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

None.

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 Laboratory Blank (LB) is prepared using laboratory reagents and provides data on the cleanliness of the radiochemistry preparation/separation processes. LB results are not normalized to processing or dilution factors associated with the samples.

6. 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 0335.

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

Tc-99 Analysis

Project / WP#:	69833/N82456/N79882
ASR#:	0335.00
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

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-KNO₃ Fusion* ☒ Digested as per RPG-CMC-128, Rev.1, *HNO₃-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, 08/31/2017
Spike Standard ID's	R-687-a-2 (Pu-239), R-693-b-2 (Sr-90)
Analysis Procedure	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Technician/Analyst:	LP Darnell and T. Trang-Le 09/01/2017
RadioChemical Preparation Procedure:	RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*
Technician/Analyst:	LP Darnell, 11-17-2017
Spike and Tracer Standard ID's:	R-540-b-8 (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:	11/17/2017
Technician/Analyst:	CZ Soderquist
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 69833: 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 / 12/11/17
Preparer **Date**

/ 12/11/17
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0335. All data are reported in units of µCi/g with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

Three samples submitted under ASR 0335 were analyzed for Tc-99. All the samples were prepared in Laboratory 420. Aliquots of the acid digestions and pre-diluted 17-1242 and duplicate (TI014-EL-Comp) were used for radioanalytical analyses. After leaching, the leachate solution was filtered and the entire leachate was processed and analyzed using procedure RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*.

The samples were counted on November 17, 2017; no decay corrections were made.

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 Tc-99 data. The beta results are included in the data tables as supplemental information only. The beta activity was measured by evaporating small aliquots of the acid digestion 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 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 Preparation Blank (PB):

BLK-1240 was prepared as the PB for this batch of sample. There are no acceptance criteria for PB.

Reagent Spike (RS):

The RS recovery of 91% (Tc-99) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery of 92% (Tc-99) meets 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 17-1240 (TI014-FEED) was selected as the matrix spike sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

Duplicate results are required to agree within $\leq$20% RPD. Duplicate results were 6-11% RPD. The Tc-99 activity detected in the samples all have 1-σ counting error of 10% or greater. 1-σ counting error at these levels indicates the activity measured in the samples is nearing the minimum detectable activity for the samples.

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 0335.

Uranium by Kinetic Phosphorescence Analysis (KPA)

Project / WP#:	69833/N82456/N79882
ASR#:	0335.00
Client:	SK Fiskum
Total Samples:	3

Client Sample ID	RPL ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

Analysis Type:	Uranium by KPA
Sample Processing Prior to Radiochemical Processing/Analysis	☐ None ☒ Digested as per RPG-CMC-128, *HNO_3-HCl Acid Extraction of Liquids Using a Dry Block Heater*
Spike Standard ID:	R-691-b Expiration date: 11/18/2017
Analysis Procedures:	RPG-CMC-4014, Rev. 1, *Uranium by Kinetic Phosphorescence Analysis* RPG-CMC-4015, Rev. 0, *Analysis of Soils and Sediment Samples for Actinides and Sr-90*
Analysis Date:	11/14/17
Technician/Analyst:	LP Darnell and CZ Soderquist
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls; Worksheet and Worksheet KPA standards
ASO Project 98620 File:	File Plan 5871: T 52578 8480 R1: Sample preparation and analysis records; T-4.4 Uranium KPA control charts and maintenance records; and T3 standard certificates and preparation. Also, balance calibration and performance check records.
M&TE Number(s):	Chemcheck Instruments, Inc., Model: KPA 11-R, WD59770, RPL 525; KPAWIN Software, Version 1.2.8, Release 19 July 2000.

T Trang-le / 12/11/17
Preparer **Date**

/ 12/11/17
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0335. The acid digestion sample results are reported in µg/g. The pre-dilution sample from the hot cell is reported in µg/mL. All results are reported with a 1-σ uncertainty (see Comments).

Sample Preparation and Analysis

All three samples were received by the Analytical Support Operations (ASO) under Analytical Service Request (ASR) 0335 for total uranium analysis by kinetic phosphorescence (KPA). Aliquots of the acid digestions and pre-diluted TI014-EL-Comp (17-1242) sample were used for this analysis.

All acid digestion aliquots and pre-diluted TI014-EL-Comp were processed through chemical separation to remove elemental interferences. Samples were diluted approximately 5-fold to ensure sample concentrations were within the calibration range.

Quality control (QC) samples prepared with the acid digestion include a process blank (PB) and sample duplicates for each samples. QC samples were not prepared with the simple dilution samples from the hot cell. The pre-diluted sample was prepared in duplicate for this analysis. Additional laboratory QC samples were prepared prior to analysis by KPA; these include a laboratory blank, a matrix spike, and two reagent blank spikes (RS/BS).

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

QUALITY CONTROL RESULTS

Instrument Calibration Control

Two calibration verification check standards were analyzed at the beginning of the batch analysis. The Recovery of the high range calibration verification standard is 102% and the recovery of the low range calibration verification standard is 99%. Both recovery values are with the acceptance criterion of 90% to 110%. Two continuing calibration verification standards were run at the end of the analytical run. The Recovery of the high range calibration verification standard is 98% and the recovery of the low range calibration verification standard is 98%. Both recovery values are with the acceptance criterion of 90% to 110%.

Process Blank (PB):

Two process blanks were prepared with the analytical run. An acid digestion blank and a lab blank (clean reagents run through the separation process). The acid digestion blank had measurable U at 3.57E-2 µg/mL, the laboratory blank has measurable U at 1.77E-3 µg/mL. The lowest concentration of U detected in the sample group is 3.52E+0. The U present in the process blanks is less than 5% of the concentration present in the samples.

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

A LCS/BS is required as part of the KPA batch run; i.e., prepared following after the acid digestion. This post-digestion LCS/BS has an 80% to 120% recovery acceptance criterion. No LCS/BS was prepared or analyzed with this batch run. Two reagent spike samples were samples were analyzed in this batch run, one within the higher concentration range on one in the lower concentration range. The recovery of the high range reagent spike is 97% and recovery of the low range reagent spike is 105%. Both spike recoveries are within the acceptance criterion.

Matrix Spike (MS):

Per the ASR, a matrix spike is to be prepared as part of the KPA batch run; i.e., after the acid digestion. This post-digestion matrix spike has a 75% to 125% recovery acceptance criterion. A matrix spike was prepared from TI014-FEED and the spike recovery is 82%, which meets the acceptance criterion.

Duplicate -- Relative Percent Difference (RPD):

All three samples were prepared in duplicate as requested in the instructions in the ASR. Duplicate results are required to agree within <20% RPD. The duplicate RPD for sample TI014-FEED is 0.3%, for sample TI014-EFF Comp the duplicate RPD is 5% and for sample TI014-ELComp the duplicate RPD is 5%. All duplicate RPD results meet the acceptance criterion.

Assumption and Limitations of the Data

There are no assumptions or limitations for these data.

Comments

1. The results have been corrected for all dilution factors resulting from sample processing.
2. The stated 1-σ uncertainty represents the total propagated error associated with processing and counting operations and includes weighing errors, volume uncertainties, and counting errors.

Attachment: Data Report -- Sample Results for ASR 0335

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

Alpha and Beta

Project / WP#:	69833/N82456/N79882
ASR#:	0335.00
Client:	SK Fiskum
Total # of Samples:	3

RPL ID	Client Sample ID
17-1240	TI014-FEED
17-1241	TI014-EFF-Comp
17-1242	TI014-ELComp

Analysis Type:	Alpha and Beta
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, Rev.0 *Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion* ☐ Other:
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, (8/31/2017)
Spike Standard ID's:	R-687-a-2 (Pu-239), R-693-b-2 (Sr-90)
Analysis Procedure:	RPG-CMC-408, Rev. 2, *Total Alpha and Beta Analysis*
Reference Date:	Not Applicable
Analysis Date(s) or Date Range:	Alpha and Beta (9/1/2017); (9/13/2017)
Technician/Analyst:	LP Darnell and T. Trang-Le
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\17-1240 Fiskum.xls
CMC Project 98620 File:	File Plan 5871: T 69833: 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):	LB 4100 gas proportional counter – See attached M&TE list

T Trang-le / 11/5/17
Preparer **Date**

/ 11/15/17
Reviewer **Date**

Sample Results

See attached data report, sample results for ASR 0335. All data are reported in units of µCi/mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

All three samples were received from the Shielded Analytical Laboratory (SAL) and submitted under Analytical Service Request (ASR) 0335 were analyzed for alpha and beta. All samples were prepared in laboratory 420. Aliquots of the acid digestions and pre-diluted TI014-EL-Comp were mounted for alpha/beta counting using procedure RPG-CMC-4001, then counted using Ludlum, and alpha/beta gas proportional counters per procedure RPG-CMC-408.

Sample 17-1242 (TI014-EL-Comp) was prepared for dilution in the hot cell by filling the diluent vial with 4 aliquots of ~5mL each of DI water in lab 309. The diluent vial was loaded into the hot cell where ~0.2mL of AP-105 Cs Eluent was added to the diluent vial. The vial was then loaded out of the hot cell clean.

QUALITY CONTROL RESULTS

Quality control (QC) samples prepared in laboratory 420 include a laboratory duplicate sample and a preparation blank. Additional QC samples were prepared prior to alpha and beta counting including a laboratory blank, a reagent blank spike (RS, Pu-239 and Sr-90), and addition of Pu-239 and Sr-90 standard to a diluted aliquot of the sample selected as the matrix spike (MS).

A summary of the Alpha and 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) and Laboratory blank (LB):

The alpha activity measured in the LB is required to be within the acceptance criteria of less than 5% of the sample isotope concentration or less than sample minimum detectable activity (MDA). The alpha was counted for 60 minutes in Ludlum, and the beta was counted for 100 minutes in LB4100 alpha beta proportional counter. The LB alpha and beta activities are less than the MDA.

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

The RS (Pu-239) recovery of 105% and (Sr-90) recovery of 86% meet the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS (Pu-239) recovery of 98% meets the acceptance criterion of 75% to 125% recovery. The MS (Sr-90) recovery is too small for the spike, and did not meet the acceptance criterion of 75% to 125%. Note: the MS sample was prepared by adding a known Pu-239 and Sr-90 standard quantity to an aliquot of the digestate. Sample number 17-1240 (TI014-FEED) was selected as the matrix spike sample.

Duplicate -- Relative Percent Difference (RPD):

Sample and duplicate sample RPD result are 2-3% for beta. RPD results are within the acceptance criterion of ≤ 20%. Sample and duplicate RPD results are not calculated for gross alpha measures because the results are less than the detection limit.

Instrument Quality Control

LB4100 alpha and beta counters undergo initial calibration to determine the detector efficiency. The established efficiency for each detector is used in the final calculation of the sample alpha and 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 0335.

Radiochemistry M&TE List

Description	Manufacturer/ SW	Model	Serial Number/ Property Number	Location	M&TE Category	Calibration Frequency	Calibration Standards	SN/ Lot#	Acquisition Date
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 3.00/Build 5	3100TR	WD48466	RPL/425	2	With use	Blank, H-3, and C-14 sealed sources & Quench curves (efficiency) for H-3,C-14, and Ni-63		9/2006
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 5.00/Build 3	5110TR	WD56319	RPL/425	2	With use	Blank, H-3, C-14 instrument performance assessment (IPA) sources & Quench curves (efficiency) for H-3 & C-14		8/2015
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 4.00/Build 5	3110TR	PT27127	RPL/425	2	With use	Blank, H-3, C-14 instrument performance assessment (IPA) sources & Quench curves (efficiency) for H-3 & C-14		5/2011

~~Liquid Scintillation Counter~~	~~Packard Tri Carb Packard Tri-Carb 2700TR software version 1.04 dated 9/99. The software runs on a DOS-2000 platform on a PC computer~~	~~2550~~	~~401664/ WD06783~~	~~RPL/425~~					
Automated NaI	Perkin Elmer Wizard 2	2480	WD56320	RPL/93	2	With use	I-129 instrument performance assessment source (IPA)		8/2015
Uranium									
Uranium Analyzer/ Kinetic Phosphorescence Analyzer	Chemcheck Instruments, Inc. The software used to control the instrument is KPAWIN Software, Version 1.2.9 Release 1999	KPA-11R	Serial number: 1045050203 WD59770	RPL/525		With use	Too many to list		1994

Radiochemistry M&TE List

ID	Number of Detectors	Property #	Model or Serial #	Location, in Bldg. 325	Procedures using the M&TE	PM Contract?
Dual Alpha Beta Gas Proportional Counters						
Oxford LB4100	16	WD13066	L8285-O	425	Total alpha, total beta, Sr/Y90, Tc99	Y
Alpha Counters						
Ludlum	10	multiple	multiple	425	Total Alpha	N
AEA (Ortec)*	32	multiple	multiple	425	AEA (total alpha, Pu, Am/Cm,Np)	N
Liquid Scintillation						
Perkin Elmer 3100TR	1	WD48466	DG08061340	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Perkin Elmer 3110TR	1	PT27127	DG03117614	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Perkin Elmer 5110TR	1	WD56319	SGLO344150055	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
~~Packard 2550~~	~~1~~	~~WD06783~~	~~401664~~	~~425~~	~~H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90~~	~~Y~~
Gamma						
HpGe*	25	see list	see list	425/93	GEA	N
LEPS (x-rays)*	4	see list	see list	425	X-ray (Fe-55, Ni-59, Nb-93m)	N
Well HPGe*	3	see list	see list	93	GEA – In development	N
Anti-Compton*	2	see list	see list	93	GEA – In development	N
Perkin Elmer 2480 Wizard Automated NaI	1	WD56320	SGWZ28150290	93	In development	Y
Uranium						
Chemcheck	1	WD59770	KPA11R	525	Uranium KPA	N
***Spectral Analysis System**						
~~Canberra - VAX 3000~~	~~1~~	~~WD12890~~	~~PE42AJB~~	~~425~~	~~AEA, GEA~~	~~N~~
~~Canberra - VAX 3100~~	~~1~~	~~WC38624~~	~~KA235W0225~~	~~425~~	~~AEA, GEA (Backup VAX)~~	~~N~~

List of Gamma Detectors

Name	Property Tag	Vendor	Model	Serial #	Type	Eff. %	CMC?
C	WD25157	EG&G Ortec	GEM70200P	33TP40378A	Ge	70	Y
D	WD06582	Princeton	IGC7022SD	2542	Ge	70	Y
E	WD12833	EG&G Ortec	GEM100210	34P40535A	Ge	100	Y
G	WD35719	Canberra	GC4020	9923007	Ge	40	Y
H	PT00941	Tennelec	CPVS3040195	6028	Ge	40	Y
I	WD12076	EG&G Ortec	GLP3638510P	33TE187	LEPS	X-RAY	Y
J	WD12075	EG&G Ortec	GLP3638510P	33TE190	LEPS	X-RAY	Y
K	WD06581	Princeton	NIGC6022SD	3612	P-Ge	60	Y
L	WD36413	EG&G Ortec	GEM20P-Plus	44TP21884A	Ge	20	Y
M	WD73130	Canberra	GC10021	9722	Ge	100	Y
N	WD73129	Canberra	GC10021	9716	Ge	100	Y
~~P~~	~~WD59508~~	~~EG&G Ortec~~	~~GEM30210~~	~~24P80V~~	~~Ge~~	~~30~~	~~Y~~
T	WD81868	EG&G Ortec	GEM20180	35TP21056A	Ge	20	Y
~~W1~~	~~PT27133~~	~~Canberra~~	~~GC4018~~	~~9919~~	~~Ge~~	~~40~~	~~Y~~
F	PT16418	Canberra	GC10021	10182	Ge	100	Y
Q	PT16419	Canberra	GC10021	10188	Ge	100	Y
R	PT16420	Canberra	GC10021	10181	Ge	100	Y
S	PT16430	Canberra	GC10021	10101	Ge	100	Y
U	PT16429	Canberra	GC10021	10178	Ge	100	Y
V	PT16428	Canberra	GC10021	10150	Ge	100	Y
W	PT16394	Canberra	GC10021	10129	Ge	100	Y
X	PT16402	Canberra	GC10021	10104	Ge	100	Y
Y	PT16401	Canberra	GC10021	10145	Ge	100	Y
Z	PT16410	Canberra	GC10021	10212	Ge	100	Y
LEPS1	WB61769	Ortec	101351600S	20M102	Ge	X-RAY	Y
LEPS2	WD48418	Canberra	GL2015R	6068240	Ge	X-RAY	Y
WA	WD81452	Ortec	GEM100210	35-P-40642A	Ge	100	Y
WB	WD81454	Ortec	GEM100210	34-P-40481A	Ge	100	Y
WC	WD81453	Ortec	GEM100210	34-P-40522A	Ge	100	Y
Well1	WD81450	Ortec	GEM170230-S	34-P-30914B	Ge Well	N/A	Y
Well2	WD81449	Ortec	GEM170230-S	34-P-40544A	Ge Well	N/A	Y
Well3	WD81451	Canberra	GCW2523-S	11051527	Ge Well	N/A	Y
AC1	PT27479	Ortec	GEM4276LBCHJ	54P33125A	Ge-AC	45	Y
AC2	WD56318	Canberra	GX5020	5945022	Ge-AC	50	Y

ASR 0372 Test 2 through Test 5 Results

Unfortunately, ASR 0372 incorrectly mixed up the elution sample identifications following Test 4 Take 1 and Test 4 Take 2 as indicated in Table B.1. Therefore, the ASO-reported results for these two samples need to be switched for all analytes.

Table B.1. Elution Sample Identification for Test 4 Take 1 and Test 4 Take 2, ASR 0372

Test ID	Client ID on ASR	Assigned ASO Sample ID	Corrected Client ID	Corrected ASO Sample ID
Test 4 Take 1	TI020-ELComp	18-0009	TI020-EComp	18-0012
Test 4 Take 2	TI020-EComp	18-0012	TI020-ELComp	18-0009

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
Print Name S. K. Fiskum
Phone 375-5677 MSIN

Project Number: 69832
Work Package: N79882

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)

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

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

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**
  ☐ ASO-QAP-001 (Equivalent to HASQARD).
  ☐ Minimum data report.
  X 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:**
  3/15/18
  (*To be completed by ASO Lead*)

Waste Designation Information

- **ASO Sample Information Check List Attached?** X No ☐ Yes
  If no, Reference Doc Attached:
  or, Previous ASR Number: 0335
  or, Previous RPL Number:

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

Send Report To: **S. K. Fiskum** **MSIN**
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: 13:00
Group ID (optional)
CMC Waste Sample? X No ☐ Yes

Received By: T. Trang-Le
ASR Number: 0372 Rev.: 01
RPL Numbers: (18-0001 thru 18-0015)
(first and last)

ASO Work Accepted By: KN Pool **Signature/Date:** 3/1/18

Analytical Services Request (ASR)

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

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

Revision 1 - ASR revised to remove U-KPA and add U by ICP/MS

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**
18-0001	TI015-FEED	Caustic/5.7 M Na, high Salt	1) GEA - All samples (Co-60, Cs-137, Eu-154, Am-241 and any other observed gamma emitting isotopes) 2) Acid Digestion- 128 - ***Caustic matrix samples only*** 3) ICP/OES - All samples - (Al, Cd, Cr, Cu, Fe, K, Mo, Na, Ni, Pb, S, Si, Sr and Zn) 4) Pu-AEA - Pu-239/240 - All Samples 5) Tc-99 - All Samples 6) ICP/MS - U only		
18-0002	TI015-EFF Comp	Caustic/5.7 M Na, high Salt			
18-0003	TI015-ELComp	***Acidic/~0.2 M HNO $_3$ - Sample Pre-Diluted in the hot cell By ~ 100x***			
18-0004	TI016-FEED	Caustic/5.7 M Na, high Salt			
18-0005	TI016-EFF Comp	Caustic/5.7 M Na, high Salt			
18-0006	TI016-ELComp	***Acidic/~0.2 M HNO3 - Sample Pre-Diluted in the hot cell By ~ 100x***			
18-0007	TI020-FEED-1-A	Caustic/5.7 M Na, high Salt			
18-0008	TI020-EFF Comp-Take 1	Caustic/5.7 M Na, high Salt			
18-0009	TI020-ELComp	***Acidic/~0.2 M HNO3 - Sample Pre-Diluted in the hot cell By ~ 100x***			
18-0010	TI020-FEED-Take2	Caustic/5.7 M Na, high Salt			
18-0011	TI020-EFF Comp-Take2	Caustic/5.7 M Na, high Salt			
18-0012	TI020-EComp	***Acidic/~0.2 M HNO3 - Sample Pre-Diluted in the hot cell By ~ 100x***			
18-0013	TI021-FEED	Caustic/5.7 M Na, high Salt			
18-0014	TI021-EFF Comp	Caustic/5.7 M Na, high Salt			
18-0015	TI021-ELComp	***Acidic/~0.2 M HNO3 - Sample Pre-Diluted in the hot cell By ~ 100x***			

Note: All the acidid matrix samples were pre-diluted in the hot cell by ~ 100x due to the high Cs-137 content in the parent sample... Use the dilution factor from the SAL bench sheet in all sample result calculations.

ASR #____0372__________ Rev:__01___

Special Instructions for ASR 0372

Analysis of AP-105 diluted feed, effluent, and eluate from Tests 2-5 ion exchange process runs

Use the ASO QA Plan current revision to apply batch processing and instrument QC.

IX Test	Client ID	ASO Sample ID	Location for Pickup	Matrix
2	TI015-FEED	18-0001	SAL cell 2	Caustic/5.7 M Na, high salt
	TI015-EFFComp	18-0002	Lab 305	Caustic/5.7 M Na, high salt
	TI015-ELComp	18-0003	SAL cell 2	Acidic/~0.2 M HNO_3
3	TI016-FEED	18-0004	SAL cell 2	Caustic/5.7 M Na, high salt
	TI016-EFFComp	18-0005	Lab 305	Caustic/5.7 M Na, high salt
	TI016-ELComp	18-0006	SAL cell 2	Acidic/~0.2 M HNO_3
4 Take 1	TI020-FEED-1-A	18-0007	Lab 305	Caustic/5.7 M Na, high salt
	TI020-EFFComp	18-0008	Lab 305	Caustic/5.7 M Na, high salt
	TI020-ELComp	18-0009	SAL cell 2	Acidic/~0.2 M HNO_3
4 Take 2	TI020-FEED-Take2	18-0010	Lab 305	Caustic/5.7 M Na, high salt
	TI020-EFFComp-Take2	18-0011	Lab 305	Caustic/5.7 M Na, high salt
	TI020-EComp	18-0012	SAL cell 2	Acidic/~0.2 M HNO_3
5	TI021-FEED	18-0013	SAL cell 2	Caustic/5.7 M Na, high salt
	TI021-EFFComp	18-0014	Lab 305	Caustic/5.7 M Na, high salt
	TI021-EComp	18-0015	SAL cell 2	Acidic/~0.2 M HNO_3

Please prepare one sample in duplicate per preparative batch.

Because the TI0*xx*-ELComp samples are already acidified, acid digestion may not be needed. The high salt samples (FEED and EFFComp) are assumed to require acid digestion. However if the preparative technique sufficiently dilutes the sample into required acid matrix, then acid digestion may be omitted.

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

1. Process blank (digestion blank or diluent blank)
2. Duplicate (one duplicate per analytical batch; if dilution is the only preparation, please prepare a diluent duplicate)
3. Blank spike or reagent spike
    a. not applicable to GEA
4. Matrix spike

a. ICP-OES: not needed for major analytes (e.g., Na in the FEED and EFFComp samples); for guidance on spiking needs see ICP-OES results from ASR 0316. In the case where analyte spikes are not used, report serial dilution result instead per ASO QA Plan
b. not applicable to GEA

Please analyze **FEED, EFFComp,** and **ELComp (or EComp)** samples as defined in Table 1.

Table 1.Sample Analyte List

Analyte	Target MDL	Analysis Method
^{60}Co	1.00E-03 µCi/mL	Gamma Energy Analysis (GEA) (report any other observed gamma emitting isotopes)
^{137}Cs	1.00E-05 µCi/mL	
^{154}Eu	1.00E-05 µCi/mL	
^{241}Am	2.00E-03 µCi/mL	
$^{239+240}$Pu	1.00E-05 µCi/mL	Acid Digestion Coprecipitation/Alpha Energy Analysis (AEA)
^{99}Tc	1.00E-5 µCi/mL	Separations/Beta Counting
Al	1 µg/mL	Acid Digestion Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
Cd	0.5 µg/mL	
Cr	0.5 µg/mL	
Cu	0.5 µg/mL	
Fe	0.5 µg/mL	
K	10 µg/mL	
Mo	1 µg/mL	
Na	100 µg/mL	
Ni	1 µg/mL	
Pb	1 µg/mL	
S	1 µg/mL	
Si	1 µg/mL	
Sr	0.5 µg/mL	
Zn	0.5 µg/mL	
U (total)	0.5 µg/mL	Acid Digestion / Kinetic Phosphorescence Analysis (KPA)

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

Project / WP#: 69832 / N79882
ASR#: 0372
Client: S. Fiskum
Total Samples: 15 (liquids)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0001	TI015-FEED	Caustic/5.7 M Na, high Salt	NA
18-0002	TI015-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0003	TI015-ELComp	Acidic/~0.2 M HNO_3	NA
18-0004	TI016-FEED	Caustic/5.7 M Na, high Salt	NA
18-0005	TI016-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0006	TI016-ELComp	Acidic/~0.2 M HNO_3	NA
18-0007	TI020-FEED-1-A	Caustic/5.7 M Na, high Salt	NA
18-0008	TI020-EFF Comp-Take-1	Caustic/5.7 M Na, high Salt	NA
18-0009	TI020-ELComp	Acidic/~0.2 M HNO_3	NA
18-0010	TI020-FEED-Take2	Caustic/5.7 M Na, high Salt	NA
18-0011	TI020-EFF Comp-Take2	Caustic/5.7 M Na, high Salt	NA
18-0012	TI020-EComp	Acidic/~0.2 M HNO_3	NA
18-0013	TI021-FEED	Caustic/5.7 M Na, high Salt	NA
18-0014	TI021-EFF Comp	Caustic/5.7 M Na, high Salt	NA
DUP-0014	TI021-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0015	TI021-ELComp	Acidic/~0.2 M HNO_3	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 18-0001, 18-0002, 18-0004, 18-0005, 18-0007, 18-0008, 18-0010, 18-0011, 18-0013, and 18-0014 on 11/28/17. Simple dilution in 5% HNO_3 was performed on Samples 18-0003, 18-0006, 18-0009, 18-0012, and 18-0015 on 10/25/17. Simple dilution of "as received" samples in 5% v/v HNO_3 was performed by J. Carter on 12/07/17.

Report Preparer

1/26/18
Date

Review and Concurrence

1/26/18
Date

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:**	12/07/2017	**ICP File:**	C0738
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
	☒	Sartorius R200D Balance	SN: 39080042
	☒	SAL Cell 2 Balance	SN: 8033311209
	☒	Lab 201 Denver A-160 Balance	SN: 60568

Fifteen aqueous samples submitted under Analytical Service Request (ASR) 0372 were analyzed by ICP-OES. Samples 18-0001, 18-0002, 18-0004, 18-0005, 18-0007, 18-0008, 18-0010, 18-0011, 18-0013, and 18-0014 were prepared following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Sample 18-0014 was prepared in duplicate following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Samples 18-0003, 18-0006, 18-0009, 18-0012, and 18-0015 were diluted with 5% HNO_3 in the hot cell to approximately 105x. 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. The silicon (1.66 µg/mL) and zinc (0.652 µg/mL) concentrations were >EQL and were significant (>10%) relative to many of the samples after taking the process factors into account.

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 97% to 105%, with the exception of silicon (23%), and were within the acceptance criterion of 80% to 120%. The low silicon recovery is likely due to reagent instability or volatility.

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

The ASR requested duplicate analysis of one sample. A duplicate of sample 18-0014 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 1.6% to 5.6% and were within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike 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 91% to 116%, with the exception of silicon (51%), and were within the acceptance criterion of 75% to 125%. As noted above for the RS, the reason for the low silicon recovery may be reagent instability or volatility.

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 and sulfur (85-86%) in the last three 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 (0.11-0.17 µg/mL) 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 18-0001. 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 2.2% to 7.0% and were within the acceptance criterion of ≤10%.

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

In addition to the RS, BS, and MS samples, a post-digestion spike (A Component) was conducted on sample 18-0001. 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 94% to 104%, 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 RS, BS, and MS samples, a post-digestion spike (B Component) was conducted on sample 18-0001. 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 sulfur, the only AOI meeting this requirement, was 95%, and was 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 >	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017
		Process Factor >	1.0	48.9	504.8	504.8	496.0	104.8	491.0	487.6	104.8	487.4
			405 diluent	BLK-0001	18-0001 @ 10x	18-0001 @ 10x rep	18-0002 @ 10x	18-0003	18-0004 @ 10x	18-0005 @ 10x	18-0006	18-0007 @ 10x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	Lab Diluent	BLK-0001	TI015-Feed		TI015-EFF Comp	TI015-ELF Comp	TI016-Feed	TI016-EFF Comp	TI016-EL Comp	TI020-Feed-1-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)
0.0038	0.038	Al	--	--	13,500	13,500	14,000	69.2	14,700	13,900	78.0	13,300
0.0016	0.016	Cd	--	--	[0.83]	[0.87]	[1.5]	--	[1.5]	--	--	[1.3]
0.0016	0.016	Cr	--	--	335	329	324	1.86	344	323	2.56	337
0.0023	0.023	Cu	--	--	[2.5]	[3.5]	[2.4]	11.7	[2.7]	[1.6]	9.53	[2.9]
0.0033	0.033	Fe	--	[0.26]	[5.7]	[5.5]	[3.6]	4.58	[5.9]	[3.5]	4.14	[4.1]
0.0262	0.262	K	--	[4.6]	3,730	3,740	3,750	282	4,130	3,880	369	3,930
0.0048	0.048	Mo	--	--	51.1	50.4	49.8	--	50.5	47.0	--	48.8
0.0075	0.075	Na	--	[3.3]	138,000	135,000	132,000	3,150	142,000	134,000	4,110	140,000
0.0040	0.040	Ni	--	--	30.1	30.1	30.2	[2.5]	30.3	28.3	4.35	30.5
0.0144	0.144	Pb	--	--	[16]	[17]	[8.9]	87.6	[20]	--	111	[15]
0.0898	0.898	S	--	--	1,150	1,110	1,090	[27]	1,080	1,020	[35]	1,060
0.0043	0.043	Si	[0.0076]	3.33	125	125	133	739	103	120	625	127
0.0001	0.001	Sr	--	[0.020]	[0.083]	[0.079]	[0.11]	0.211	[0.080]	[0.056]	0.414	[0.096]
0.0023	0.023	Zn	--	1.30	[3.6]	[4.1]	[5.1]	17.6	--	--	10.2	--
Other Analytes												
0.0014	0.014	Ag	--	--	--	--	--	[0.38]	--	--	--	--
0.0383	0.383	As	--	--	--	--	--	--	--	--	--	--
0.0032	0.032	B	[0.020]	[0.90]	123	119	68.2	555	82.3	76.1	438	213
0.0001	0.001	Ba	[0.0001]	0.580	0.915	0.920	1.01	5.18	1.28	1.15	14.1	0.857
0.0001	0.001	Be	--	--	[0.19]	[0.15]	[0.16]	[0.038]	[0.20]	[0.17]	[0.046]	[0.19]
0.0220	0.220	Bi	--	--	--	--	--	--	--	--	--	--
0.0054	0.054	Ca	--	6.86	45.8	46.5	41.5	30.6	50.5	45.7	23.8	50.1
0.0052	0.052	Ce	--	--	--	[2.9]	--	--	--	--	--	--
0.0033	0.033	Co	--	--	--	--	--	--	--	--	--	--
0.0012	0.012	Dy	--	--	--	--	--	--	--	--	--	--
0.0006	0.006	Eu	--	--	--	--	--	--	--	--	--	--
0.0010	0.010	La	--	--	--	--	--	--	--	--	--	--
0.0010	0.010	Li	--	[0.084]	--	[1.0]	[1.2]	[0.80]	[0.66]	--	[0.68]	[0.67]
0.0014	0.014	Mg	--	[0.23]	--	--	--	2.38	--	--	2.35	--
0.0003	0.003	Mn	--	--	--	--	--	[0.14]	--	[0.13]	[0.080]	--
0.0095	0.095	Nd	--	--	[5.8]	[5.0]	--	[2.1]	[4.9]	--	--	[6.3]
0.0369	0.369	P	--	--	476	451	455	[3.9]	465	423	[5.4]	450
0.0082	0.082	Pd	--	--	--	--	--	--	--	[4.4]	--	--
0.0108	0.108	Rh	--	--	[5.8]	--	--	--	--	--	--	--
0.0068	0.068	Ru	--	--	[6.8]	[8.3]	[9.2]	--	[7.2]	[7.3]	--	[10]
0.0569	0.569	Sb	--	--	--	--	--	--	--	--	--	--
0.0876	0.876	Se	--	--	--	--	--	--	--	--	[11]	[45]
0.0195	0.195	Sn	--	--	--	--	--	--	--	--	--	--
0.0109	0.109	Ta	--	--	--	--	--	--	--	--	--	--
0.0155	0.155	Te	--	--	--	--	--	--	--	--	--	--
0.0057	0.057	Th	--	--	--	--	--	--	--	[2.9]	--	--
0.0004	0.004	Ti	--	[0.021]	--	--	[0.28]	0.474	[0.21]	--	0.465	[0.24]
0.0310	0.310	Tl	--	--	--	--	--	--	--	--	--	--
0.0312	0.312	U	--	--	--	--	--	--	--	--	--	[16]
0.0016	0.016	V	[0.0039]	[0.23]	[2.0]	[1.6]	[2.3]	[0.59]	[2.1]	[1.7]	[0.57]	[1.9]
0.0187	0.187	W	--	--	[84]	[85]	[80]	--	[85]	[74]	--	[81]
0.0003	0.003	Y	--	--	--	--	--	--	--	--	--	--
0.0013	0.013	Zr	--	--	--	--	--	[0.55]	--	--	[0.47]	--

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 >	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017	12/7/2017
		Process Factor >	485.2	104.8	490.2	486.4	104.8	493.2	489.4	492.2	104.8
			18-0008 @ 10x	18-0009	18-0010 @ 10x	18-0011 @ 10x	18-0012	18-0013 @ 10x	18-0014 @ 10x	Dup-0014 @ 10x	18-0015
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	TI020-EFF Comp-Take 1	TI020-EL Comp	TI020-Feed-Take2	TI020-EFF Comp-Take2	TI020-Ecomp	TI021-Feed	TI021-EFF Comp		TI021-EL Comp
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
0.0038	0.038	Al	13,200	49.8	14,000	14,200	93.5	14,900	14,500	14,800	75.6
0.0016	0.016	Cd	[1.1]	--	[1.3]	[1.3]	--	[1.1]	[1.1]	[0.80]	--
0.0016	0.016	Cr	301	2.50	341	334	2.63	341	328	339	3.09
0.0023	0.023	Cu	--	11.3	[2.4]	[1.2]	5.32	[2.4]	[1.2]	[1.6]	12.5
0.0033	0.033	Fe	[15]	4.22	[8.1]	[5.4]	[3.4]	[3.4]	[2.7]	[2.9]	4.89
0.0262	0.262	K	3,460	268	4,020	3,950	289	4,120	3,970	4,050	287
0.0048	0.048	Mo	43.9	--	48.5	49.4	--	49.9	49.0	47.8	--
0.0075	0.075	Na	128,000	3,020	140,000	139,000	3,150	142,000	137,000	141,000	3,500
0.0040	0.040	Ni	24.8	5.48	29.8	29.0	5.86	31.1	28.3	29.2	6.93
0.0144	0.144	Pb	--	95.6	[15]	--	29.9	[16]	--	--	89.1
0.0898	0.898	S	924	[20]	1,050	1,040	[23]	1,050	997	1,050	[22]
0.0043	0.043	Si	152	373	145	172	546	94.8	76.5	78.4	718
0.0001	0.001	Sr	[0.049]	0.115	[0.074]	[0.12]	0.148	[0.082]	[0.12]	[0.091]	0.220
0.0023	0.023	Zn	--	10.4	[2.0]	--	13.7	--	[2.8]	--	13.1
Other Analytes											
0.0014	0.014	Ag	--	[0.31]	--	--	--	--	--	--	--
0.0383	0.383	As	--	--	--	--	--	--	--	--	--
0.0032	0.032	B	73.1	283	167	103	409	52.7	46.4	47.5	530
0.0001	0.001	Ba	0.751	1.74	0.808	1.38	2.02	0.920	0.689	0.809	4.78
0.0001	0.001	Be	[0.14]	[0.050]	[0.20]	[0.18]	[0.037]	[0.20]	[0.17]	[0.18]	[0.060]
0.0220	0.220	Bi	--	--	--	--	--	--	--	--	--
0.0054	0.054	Ca	43.3	26.4	46.0	49.1	34.7	47.5	48.4	54.1	27.2
0.0052	0.052	Ce	--	--	--	--	--	--	--	--	--
0.0033	0.033	Co	--	--	--	--	--	--	--	--	--
0.0012	0.012	Dy	[0.73]	--	--	--	--	--	--	[1.0]	--
0.0006	0.006	Eu	--	--	--	--	--	--	--	--	--
0.0010	0.010	La	--	--	--	--	--	--	--	[0.55]	[0.12]
0.0010	0.010	Li	--	[0.57]	--	[0.87]	[0.75]	[0.99]	[0.66]	--	[0.82]
0.0014	0.014	Mg	--	2.13	--	--	3.96	--	--	--	2.20
0.0003	0.003	Mn	--	[0.091]	--	--	[0.089]	--	--	--	[0.12]
0.0095	0.095	Nd	--	--	--	--	[1.2]	--	--	--	--
0.0369	0.369	P	412	--	446	446	[4.8]	447	436	449	[4.6]
0.0082	0.082	Pd	--	--	--	--	--	--	--	--	--
0.0108	0.108	Rh	--	--	--	--	--	--	--	--	--
0.0068	0.068	Ru	[6.4]	--	[6.7]	[7.8]	--	[7.6]	[6.5]	[5.4]	--
0.0569	0.569	Sb	--	--	--	--	--	--	--	--	--
0.0876	0.876	Se	--	--	--	--	--	--	--	--	--
0.0195	0.195	Sn	--	--	--	--	--	--	--	--	--
0.0109	0.109	Ta	--	--	--	--	--	--	--	--	--
0.0155	0.155	Te	--	--	--	--	--	--	--	--	--
0.0057	0.057	Th	--	--	--	--	--	--	--	--	--
0.0004	0.004	Ti	--	[0.30]	--	[0.28]	[0.42]	--	--	--	0.485
0.0310	0.310	Tl	--	--	--	--	--	--	--	--	--
0.0312	0.312	U	--	--	--	--	--	--	--	--	--
0.0016	0.016	V	[1.9]	[0.61]	[2.4]	[2.4]	[0.52]	[2.6]	[2.5]	[2.6]	[0.59]
0.0187	0.187	W	[71]	--	[87]	[83]	--	[82]	[75]	[82]	--
0.0003	0.003	Y	--	--	--	--	--	--	--	--	--
0.0013	0.013	Zr	--	[0.39]	--	--	[0.48]	--	--	--	[0.58]

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 12/7/2017

Criteria >	≤ 20%	80%-120%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	18-0014 Dup	LCS/RS	18-0001 MS	18-0001 + AS-A	18-0001 + AS-B	18-0xxx 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	1.6	103	106	101		3.6
Cd		100	102	101		
Cr	3.1	97	92	98		2.4
Cu		104	107	104		
Fe		101	100	101		
K	1.9	104	116	102		2.4
Mo	2.6	98	94	98		
Na	2.7	107	nr	94		2.2
Ni	3.0	101	101	103		
Pb		98	99	98		
S	5.6	97	91		95	
Si	2.4	**23**	**51**	101		7.0
Sr		106	98	102		
Zn		97	98	99		
Other Analytes						
Ag				93		
As				101		
B	2.4	104	102	102		**16.1**
Ba	16.0	101	103	100		
Be		100	101	99		
Bi		80	82	94		
Ca	11.2	108	115	104		
Ce		99	98		97	
Co				99		
Dy					97	
Eu					96	
La		99	99		97	
Li		118	119	108		
Mg		103	105	104		
Mn		100	100	101		
Nd		99	97		97	
P	3.1	101	95	99		
Pd					91	
Rh					94	
Ru					94	
Sb				104		
Se				102		
Sn				93		
Ta				99		
Te					99	
Th					96	
Ti		103	104	102		
Tl				91		
U		102	100		101	
V		100	98	98		
W		97	94	98		
Y				98		
Zr		107	107	102		

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.

Gamma Energy Analysis (GEA)

Project / WP#: 69832/ N79882
ASR#: 0372.00
Client: SK Fiskum
Total Samples: 15

RPL ID	Client Sample ID
18-0001	TI015-FEED
18-0002	TI015-EFF-Comp
18-0003	TI015-ELComp
18-0004	TI016-FEED
18-0005	TI016-EFF-Comp
18-0006	TI016-ELComp
18-0007	TI020-FEED-1-A
18-0008	TI020-EFF Comp-Take 1
18-0009	TI020-ELComp
18-0010	TI020-FEED-Take2
18-0011	TI020-EFF Comp-Take2
18-0012	TI020-EComp
18-0013	TI021-FEED
18-0014	TI021-EFF Comp
18-0015	TI021-ELComp

Battelle PNNL/RSE/ASO Radiochemistry Analysis Report

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:	July 10, 2017 @ 10:00 am
Analysis Date or Date Range:	November 27, 2017
Technician/Analyst:	T Trang-Le
Rad Chem Electronic Data File:	*18-0001 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 C,G,L,M,N,R,T*

T Trang-le / 12/11/17
Prepare **Date**

LRGreenwood / 12/11/17
Reviewer **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in this sample are presented in an attached Excel spreadsheet for ASR 0372.00. All sample results for all 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 0372 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

Five Pre-diluted samples, 18-0003, 18-0006, 18-0009, 18-0012 and 18-0015 were received from the Shielded Analytical Laboratory (SAL). The samples were diluted ~105x by pipetting ~100µL of each sample into ~10 mL of 0.5M HNO_3.

All samples were prepared by pipetting 2 mL of each sample into a 22 mL glass scintillation vial (calibrated geometry) and sent to the counting room for GEA analysis.

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

Required detection limits for these samples are provided by the client in ASR0372. In most cases we were not able to meet these detection limits due to the high level of Cs-137 and remote counting geometries. However, we did report activities below the requested MDL values for the EFF Comp samples, which had much lower activity levels. For the samples with high Cs-137 activities, it is not possible to meet the requested MDL values with any reasonable counting times.

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. 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 0372.

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

filename 18-0001 Fiskum.xls
11/29/2017

Client: Fiskum
ASR 0372

Project: 69832
WP: N79882

Prepared by: T Trang-le 12-6-2017

Technical Reviewer: 12-6-17

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

M&TE: Gamma detectors C,G,L,M,N, R, T
Count dates: 27-Nov-17

		Measured Activity, µCi per ml ± 1s									
Sample	Lab ID	**Co-60**		**Cs-137**		**Eu-152**		**Eu-154**		**Am-241**	
TI015-FEED	18-0001	< 4.5E-3		**1.09E+02**	± 2%	< 1.3E-2		< 1.1E-2		< 4.8E-1	
TI015-EFF-Comp	18-0002	**6.22E-04**	± 2%	**4.71E-03**	± 2%	< 6.5E-6		**1.32E-04**	± 2%	**3.86E-04**	± 5%
TI015-ELComp	18-0003	< 1.0E-2		**1.11E+03**	± 2%	< 3.2E-2		< 2.8E-2		< 6.1E-1	
TI016-FEED	18-0004	< 8.5E-3		**1.29E+02**	± 2%	< 3.3E-2		< 1.8E-2		< 2.1E-1	
TI016-EFF-Comp	18-0005	**6.38E-04**	± 2%	**4.01E-02**	± 2%	**6.01E-06**	± 20%	**1.32E-04**	± 2%	**3.75E-04**	± 17%
TI016-ELComp	18-0006	< 1.5E-2		**1.51E+03**	± 2%	< 7.1E-2		< 7.3E-2		< 3.3E+0	
TI020-FEED-1-A	18-0007	< 4.7E-3		**1.11E+02**	± 2%	< 1.6E-2		< 1.2E-2		< 4.9E-1	
TI020-EFF Comp-Take 1	18-0008	**6.63E-04**	± 2%	**3.52E-03**	± 2%	**4.08E-06**	± 16%	**1.38E-04**	± 2%	**3.15E-04**	± 6%
TI020-ELComp	18-0009	< 3.5E-3		**1.06E+03**	± 2%	< 9.0E-3		< 8.9E-3		< 1.9E-1	
TI020-FEED-Take2	18-0010	< 7.3E-3		**1.30E+02**	± 2%	< 2.0E-2		< 1.4E-2		< 2.0E-1	
TI020-EFF Comp-Take2	18-0011	**5.66E-04**	± 2%	**4.66E-02**	± 2%	**7.12E-06**	± 11%	**1.22E-04**	± 2%	**3.54E-04**	± 8%
TI020-EComp	18-0012	< 4.7E-3		**2.60E+02**	± 2%	< 1.8E-2		< 1.7E-2		< 7.9E-1	
TI021-FEED	18-0013	< 1.4E-2		**1.27E+02**	± 2%	< 6.0E-2		< 3.0E-2		< 9.2E-1	
TI021-EFF Comp	18-0014	**6.55E-04**	± 2%	**5.20E-03**	± 2%	< 8.4E-6		**1.26E-04**	± 3%	**4.73E-04**	± 26%
TI021-ELComp	18-0015	< 3.0E-3		**8.60E+02**	± 2%	< 1.1E-2		< 1.2E-2		< 6.4E-1	

Plutonium 238, 239+240 Analysis

Project / WP#:	69832/N79882
ASR#:	0372.00
Client:	SK Fiskum
Total # of Samples:	15

RPL ID	Client Sample ID
18-0001	TI015-FEED
18-0002	TI015-EFF-Comp
18-0003	TI015-ELComp
18-0004	TI016-FEED
18-0005	TI016-EFF-Comp
18-0006	TI016-ELComp
18-0007	TI020-FEED-1-A
18-0008	TI020-EFF Comp-Take 1
18-0009	TI020-ELComp
18-0010	TI020-FEED-Take2
18-0011	TI020-EFF Comp-Take2
18-0012	TI020-EComp
18-0013	TI021-FEED
18-0014	TI021-EFF Comp
18-0015	TI021-ELComp

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 -- *All 5 EL&EComp samples were pre diluted by ~ 100x in the hot cell*
Co-Precipitation Procedure:	RPG-CMC-496, Rev. 1, *Coprecipitation Mounting of Actinides for Alpha Spectroscopy*
Technician/Analyst:	LP Darnell, (01/2/2018 and 01/19/2018)
Spike and Tracer Standard ID's:	R-687-a-2 (Pu-239), R-688-a-10 and R700-a (Pu-242 tracer)
Plutonium Separation Procedure:	RPG-CMC-4017, Rev. 0, *Analysis of Environmental Water Samples for Actinides and Strontium-90*
Technician/Analyst:	LP Darnell, 1/2/18 and 1/22/18
Analysis Procedure:	RPG-CMC-422, Rev. 2, *Solutions Analysis: Alpha Spectrometry*
Reference Date:	Same as analyses dates
Analysis Date or Date Range:	January 2-23, 2018
Technician/Analyst:	T. Trang-Le, CZ Soderquist
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\18-0001 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 69832: 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 instrument performance checks.
M&TE Number(s):	Ortec AEA counters – 32 counters – See attached M&TE list and Mettler AT400, Serial # 1113292667.

/ 2/14/18
Preparer Date

T Trang-Le / 2/14/18
Reviewer Date

Sample Results

See attached data report, Sample Results for ASR 0372. All data are reported in units of μCi/mL with a 1-σ uncertainty.

Sample Preparation, Separation, Mounting and Counting Methods

All fifteen samples submitted under Analytical Service Request (ASR) 0372 were analyzed for plutonium by Alpha Spectrometry. All the samples were prepared in RPL/420. Five of the 15 samples were pre-diluted by ~ 105x with 0.5 M HNO_3 in the hotcell due to high activity of the samples and were processed as received from the hotcell in lab 420. The remaining 10 caustic matrix samples were diluted by ~100x in lab 420 prior to the plutonium analyses.

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 January 2-23, 2018; no decay corrections were made.

QUALITY CONTROL RESULTS

Radioanalytical quality control (QC) samples prepared in RPL/420 include a lab separation blank, sample duplicate, matrix spike (i.e., addition of Pu-239 standard to an aliquot of one of the samples) and reagent spike (RS, Pu-239).

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 93% to 107%.

Laboratory Preparation Blank (PB):

The activity level of the plutonium alpha emitters present in the blank is well below 5% of the activity present in the sample, meeting the acceptance criteria of less than 5% of the sample activity or less than the sample MDA.

Reagent Spike (RS):

The RS recoveries of 96% and 99% (Pu-239) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recoveries of 96% and 91% (Pu-239) meet the acceptance criterion of 75% to 125% recovery. Note: The MS sample was prepared after initial dilution, by adding a known quantity of Pu-239 standard to a diluted aliquot of the diluted sample. Sample numbers 18-0005 (TI016-EFF-Comp) and 18-0014 (TI021-EFF Comp) were selected as the matrix spike samples.

Laboratory Duplicate - Relative Percent Difference (RPD):

Sample 18-0005 (TI016-EFF-Comp) and 18-0014 (TI021-EFF Comp) were selected as the duplicate samples. For sample 18-0005, the Pu-238 sample and duplicate RPD of 7% and the duplicate RPD for Pu-239+240 of 18% are within the acceptance limit of $\leq$ 20% RPD. For sample 18-0014, the Pu-238 sample and duplicate RPD of 5% and the duplicate RPD for Pu-239+240 of 8% are within the acceptance limit of $\leq$ 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 0372.

Tc-99 Analysis

Project / WP#:	69832/N79882
ASR#:	0372.00
Client:	SK Fiskum
Total # of Samples:	15

RPL ID	Client Sample ID
18-0001	TI015-FEED
18-0002	TI015-EFF-Comp
18-0003	TI015-ELComp
18-0004	TI016-FEED
18-0005	TI016-EFF-Comp
18-0006	TI016-ELComp
18-0007	TI020-FEED-1-A
18-0008	TI020-EFF Comp-Take 1
18-0009	TI020-ELComp
18-0010	TI020-FEED-Take2
18-0011	TI020-EFF Comp-Take2
18-0012	TI020-EComp
18-0013	TI021-FEED
18-0014	TI021-EFF Comp
18-0015	TI021-ELComp

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 -- *All 5 ELComp samples were pre diluted by ~ 100x in the hot cell*
RadioChemical Preparation Procedure:	RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*
Technician/Analyst:	LP Darnell, 01/10/2018
Spike and Tracer Standard ID's:	R-540-b-8 (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:	01/11/2018
Technician/Analyst:	CZ Soderquist
Analysis Data (File):	RPG-RC\PNL\Projects\Backup files\Backup 18\18-0001 Fiskum.xlsx
CMC Project 98620 File:	File Plan 5871: T 69832: 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 Tri-Carb 3100 TR, Serial # DG08061340, RPL 425, Tri-Carb 2700TR software version 1.04 dated 9/99, Mettler AT400, Serial # 1113292667

Karl N. Pool / 2/14/18
Preparer **Date**

T Trang-le / 2/14/18
Reviewer **Date**

Sample Results

See attached data report, Sample Results for ASR 0372. All data are reported in units of µCi/mL with a 1-σ uncertainty (see comments).

Sample Preparation, Separation, Mounting and Counting Methods

All fifteen samples submitted under Analytical Service Request (ASR) 0372 were analyzed for Tc-99 by chemical separation and beta counting. All the samples were prepared in RPL/420. Five of the 15 samples were pre-diluted by ~ 100x with 0.5 M HNO_3 in the hotcell due to high activity of the samples and were processed as received from the hotcell in lab 420. The remaining 10 caustic matrix samples were diluted by ~100x in lab 420 prior to Tc-99 analyses by procedure RPG-CMC-432, Rev. 0, *Technicium-99 Analysis*.

The samples were counted on January 11, 2018; no decay corrections were made.

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 Tc-99 standard to an aliquot of one of the samples) and reagent spike. (RS, Tc-99).

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 Preparation Blank (PB):

The activity level of the Tc-99 present in the lab is very near the instrument background and below the MDA for all samples. The lab blank meets the acceptance criteria for being below the MDA for all samples.

Reagent Spike (RS):

The RS recovery of 89% (Tc-99) meets the acceptance criteria of 80% to 120% recovery.

Matrix Spike (MS):

The MS recovery for Tc-99 could not be calculated. The activity of Tc-99 added to the matrix spike samples was too low relative to the activity present in the sample. Sample 18-0001 (TI015-FEED) was selected for the matrix spike analyses. Note: The MS sample was prepared after initial dilution, by adding a known quantity of Tc-99 standard to a an aliquot of the diluted sample.

Laboratory Duplicate - Relative Percent Difference (RPD):

> Sample 18-0014 (TI021-EFF Comp) was selected as the duplicate sample. The Tc-99 sample and duplicate RPD of 10% is within the acceptance limit of ≤ 20% RPD.

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 0372.

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

filename 18-0001 Fiskum.xls
1/24/2018
Partial Data; uranium results to follow later

Client: S. Fiskum
ASR 372

Project: 69832
WP: N79882

Prepared by: T Trang-le 2/13/18

Technical Reviewer: 2/13/18

Procedures:
RPG-CMC-432, Rev 0, Technetium-99 Analysis
RPG-CMC-474, Rev 1, Measurement of Alpha and Beta Activity by Liquid Scintillation Spectrometry (Tc-99, Sr-90)
RPG-CMC-496, Rev 1, Coprecipitation Mounting of Actinides for Alpha Spectrometry
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha Spectrometry (Np, Pu, Am, Cm)
RPG-CMC-4014, Rev 1, Uranium by Kinetic Phosphorescence Analysis

M&TE:
Perkin Elmer TriCarb model 3100TR liquid scintillation, Alpha Spectrometry counting system
Chem-Chek Instruments model KPA-11R kinetic phosphorescence analyzer

Count dates: January 11-23, 2018

		Measured activities, µCi/mL ± 1s uncertainty						
Sample	**Lab ID**	**Tc-99**		**Total U, µg/mL**	**Pu-238**		**Pu-239+240**	
TI015-FEED	18-0001	1.03E-1	± 2%		1.68E-5	± 14%	5.82E-5	± 8%
TI015-EFF-Comp	18-0002	8.02E-2	± 2%		2.80E-5	± 11%	5.68E-5	± 8%
TI015-ELComp	18-0003	7.87E-6	± 23%		5.84E-7	± 122%	2.33E-7	± 285%
TI016-FEED	18-0004	8.92E-2	± 2%		1.26E-5	± 17%	5.77E-5	± 8%
TI016-EFF-Comp	18-0005	7.92E-2	± 2%		1.39E-5	± 16%	6.20E-5	± 7%
	18-0005 Dup	--			1.30E-5	± 16%	5.16E-5	± 8%
	RPD				7%		18%	
TI016-ELComp	18-0006	7.58E-6	± 23%		-1.29E-7	± 725%	1.16E-6	± 81%
TI020-FEED-1-A	18-0007	8.62E-2	± 2%		2.01E-5	± 13%	7.39E-5	± 7%
TI020-EFF Comp-Take 1	18-0008	7.67E-2	± 2%		1.52E-5	± 16%	4.39E-5	± 9%
TI020-ELComp	18-0009	5.15E-6	± 33%		3.14E-7	± 224%	-6.28E-8	± 1025%
TI020-FEED-Take2	18-0010	8.32E-2	± 2%		2.16E-5	± 13%	5.94E-5	± 8%
TI020-EFF Comp-Take2	18-0011	7.72E-2	± 2%		1.65E-5	± 14%	5.18E-5	± 8%
TI020-EComp	18-0012	2.66E-5	± 49%		-1.25E-7	± 725%	8.12E-7	± 105%

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

filename 18-0001 Fiskum.xls
1/24/2018
Partial Data; uranium results to follow later

	Lab	**Measured activities, µCi/mL ± 1s uncertainty**						
Sample	ID	**Tc-99**		**Total U, µg/mL**	**Pu-238**		**Pu-239+240**	
TI021-FEED	18-0013	8.61E-2	± 2%		5.39E-5	± 8%	8.01E-5	± 7%
TI021-EFF Comp	18-0014	7.50E-2	± 2%		3.94E-5	± 9%	6.17E-5	± 8%
	18-0014 Dup	8.26E-2	± 2%		3.74E-5	± 9%	6.66E-5	± 7%
		10%			5%		8%	
TI021-ELComp	18-0015	7.87E-6	± 23%		0.00E+0		-8.57E-7	± 93%
	Reagent spike	89%			--		99%, 96%	
	Matrix spike 18-0014	--			--		96%	
	Matrix spike 18-0005						91%	
	Lab blank	3.74E-6	± 106%		-7.43E-7	± 65%	-1.55E-6	± 45%
					7.20E-7	± 115%	-6.60E-7	± 93%

Shaded results are at or below detection limit
Matrix spike for Tc-99 was too small for the sample activity

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

Project / WP#: 69832 / N79882
ASR#: 0372.01
Client: S. Fiskum
Total Samples: 15 (Aqueous)

ASO Sample ID	Client Sample ID	Client Sample Description	Sample Weight (g)
18-0001	TI015-FEED	Caustic/5.7 M Na, high Salt	NA
18-0002	TI015-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0003	TI015-ELComp	Acidic/~0.2 M HNO_3	NA
18-0004	TI016-FEED	Caustic/5.7 M Na, high Salt	NA
18-0005	TI016-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0006	TI016-ELComp	Acidic/~0.2 M HNO_3	NA
18-0007	TI020-FEED-1-A	Caustic/5.7 M Na, high Salt	NA
18-0008	TI020-EFF Comp-Take-1	Caustic/5.7 M Na, high Salt	NA
18-0009	TI020-ELComp	Acidic/~0.2 M HNO_3	NA
18-0010	TI020-FEED-Take2	Caustic/5.7 M Na, high Salt	NA
18-0011	TI020-EFF Comp-Take2	Caustic/5.7 M Na, high Salt	NA
18-0012	TI020-EComp	Acidic/~0.2 M HNO_3	NA
18-0013	TI021-FEED	Caustic/5.7 M Na, high Salt	NA
18-0014	TI021-EFF Comp	Caustic/5.7 M Na, high Salt	NA
18-0015	TI021-ELComp	Acidic/~0.2 M HNO_3	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 18-0001, 18-0002, 18-0004, 18-0005, 18-0007, 18-0008, 18-0010, 18-0011, 18-0013, and 18-0014 on 11/28/17. Simple dilution in 5% HNO_3 was performed on Samples 18-0003, 18-0006, 18-0009, 18-0012, and 18-0015 by J. Turner on 10/25/17. Prior to analysis all samples were further diluted in 2% v/v HNO_3 by G. Brown on 02/28/18.

Garrett N. Brown
Report Preparer

4/10/18
Date

Review and Concurrence

4/11/18
Date

Procedure:	RPG-CMC-292, Rev. 1, "Determination of Elemental Composition by Inductively Coupled Argon Plasma Mass Spectrometry (ICP-MS)."					
Analyst:	G. Brown	**Analysis Date:**	03/02/2018	**ICP File:**	M0058A	
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		
	☒	Denver A-160 Balance	SN: 60568	RPL 201		
	☐	Sartorius R200D Balance	SN: 39080058	RPL 525 FH		

Fifteen aqueous samples submitted under Analytical Service Request (ASR) 0372.01 were analyzed by ICP-MS. Samples 18-0001, 18-0002, 18-0004, 18-0005, 18-0007, 18-0008, 18-0010, 18-0011, 18-0013, and 18-0014 were prepared following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Sample 18-0014 was prepared in duplicate following RPL procedure RPG-CMC-128 and diluted to approximately 25 mL. Samples 18-0003, 18-0006, 18-0009, 18-0012, and 18-0015 were diluted with 5% HNO_3 in the hot cell to approximately 105x. 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 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. Uranium-238 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 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.

Detection Limit:

The ASR specified a target detection limit of 0.5 μg/mL for total uranium. The instrument detection limit (IDL) and estimated quantitation limit (EQL) were determined from replicate analyses of a 2% HNO_3 blank solution. Both values (IDL = 0.0001 ng/mL, EQL = 0.001 ng/mL) were significantly below the requested target detection limit.

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 (U-238) data were normalized using the data for the closest IS mass (i.e., Bi-209). With one exception, the Bi-209 IS recoveries ranged from 91% to 103% and were within the acceptance criterion of 30% to 120%. The second interference check standard had a Bi-209 IS recovery of 131% that was caused by insufficient volume of the ICS standard in the autosampler (no ICS standard was available to mix in-line with the IS solution, resulting in a higher IS recovery).

Preparation Blank (PB):

A preparation blank (reagents only) was prepared for the extraction process. Results for the PB was 1.18 ng/mL U-238, which, after accounting for process factors, exceeded the acceptance criteria of <EQL (estimated quantitation level), less than ≤10% of the concentration in the samples, or <50% regulatory decision level. The uranium concentration in the PB at 243x dilution was greater than 10% of the concentrations in several of the diluted samples with process factors of ~270,000 (18-0001, 18-0002, 18-0004, 18-0005, 18-0007, 18-0008, 18-0010, 18-0011, 18-0013, and 18-0014). Trace levels of various environmental contaminants (e.g., calcium, iron, uranium, zinc, etc.) 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 result was <EQL (<0.001 ng/mL U-238).

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

A reagent spike (RS) sample (reagent and spikes) was prepared for the extraction process. The recovery for the AOI was 90%, which is within the acceptance criteria of 80% to 120% recovery.

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

A blank spike (BS) sample (lab diluent and spikes) was prepared by spiking the 2% HNO_3 lab diluent with an equivalent volume of a 2ppb standard (1:1 ratio). The recovery for the AOI was 96%, which is within the acceptance criteria of 80% to 120% recovery.

Duplicate/Replicate Relative Percent Difference (RPD):

Duplicates of sample 18-0014 were prepared and analyzed. RPD are listed for all analytes that were measured at or above the EQL. The RPD for the AOI meeting this requirement was 12% and was within the acceptance criterion of ≤20% for liquid samples.

Matrix-Spike (MS) Sample:

A matrix spike (MS) sample (18-0001 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 90% and was within the acceptance criterion of 75% to 125%.

Initial/Continuing Calibration Verification (ICV/CCV):

The ICV/CCV solution was analyzed immediately after calibration, after each group of generally not more than ten samples, and at the end of the analytical run. Due to analyst oversight, twelve samples were analyzed between the last two groups of CCV solutions. 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 generally not more than ten samples and at the end of the analytical run). Due to analyst oversight, twelve samples were analyzed between the penultimate and final CCB solutions. 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 for the AOI (99.7%) was 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 concentration of the AOI in the first ICS corresponded to 98% recovery, which is within the acceptance criteria of 80% to 120%. However, U-238 recovery in the second ICS was 55% due to insufficient volume of solutions loaded into the autosampler.

Serial Dilution (SD):

Five-fold serial dilution was conducted on samples 18-0003. 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 0.6% and was within the acceptance criterion of ≤10%.

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

In addition to the MS sample, a post-digestion spike was conducted on samples 18-0003. 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 value for the AOI meeting this requirement was 96%, and was 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 >	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018
		Process Factor >	1	242.59	277,636	272,399	10,217	269,723	267,856	10,257	268,115	266,807	10,295
		RPL/LAB >	BLK-2% HNO3	BLK-0001 @ 5x	18-0001 @ 5625x	18-0002 @ 5625x	18-0003 @ 100x	18-0004 @ 5625x	18-0005 @ 5625x	18-0006 @ 100x	18-0007 @ 5625x	18-0008 @ 5625x	18-0009 @ 100x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	BLK-2% HNO3	Prep Reagents	TI015-FEED	TI015-EFF Comp	TI015-ELComp	TI016-FEED	TI016-EFF Comp	TI016-ELComp	TI020-FEED-1-A	TI020-EFF Comp-Take1	TI020-ELComp
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0001	0.001	U 238	--	1.18	6025	5511	1653	6294	5712	2470	5867	4535	2216

Internal Standard % Recovery

Li 6 (IS)	101%	125%	104%	102%	105%	100%	100%	102%	100%	101%	104%
Sc 45 (IS)	102%	107%	105%	105%	105%	104%	103%	102%	102%	101%	104%
Y 89 (IS)	100%	100%	101%	100%	99%	102%	101%	102%	100%	101%	97%
In 115 (IS)	101%	99%	101%	101%	97%	101%	100%	98%	99%	97%	100%
Tb 159 (IS)	96%	100%	98%	99%	98%	99%	99%	98%	98%	98%	99%
Bi 209 (IS)	99%	97%	96%	99%	98%	96%	98%	97%	97%	98%	96%

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.

		Run Date >	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018	3/2/2018
		Process Factor >	269,263	268,038	10,252	271,700	268,660	482,058	10,240
		RPL/LAB >	18-0010 @ 5625x	18-0011 @ 5625x	18-0012 @ 100x	18-0013 @ 5625x	18-0014 @ 5625x	Dup-0014 @ 10,000x	18-0015 @ 100x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	TI020-FEED-Take 2	TI020-EFF Comp-Take2	TI020-EComp	TI021-FEED	TI021-EFF Comp		TI021-ELComp
(ng/mL)	(ng/mL)	(Analyte)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)	(ng/mL)
0.0001	0.001	U 238	6077	5516	1457	5804	5305	5984	2276

Internal Stan							
Li 6 (IS)	99%	104%	104%	104%	99%	93%	105%
Sc 45 (IS)	100%	103%	104%	102%	106%	96%	107%
Y 89 (IS)	102%	97%	100%	99%	100%	90%	101%
In 115 (IS)	99%	98%	98%	100%	99%	92%	100%
Tb 159 (IS)	96%	98%	97%	99%	99%	94%	96%
Bi 209 (IS)	96%	96%	97%	97%	98%	91%	96%

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

QC Performance 03/02/2018

Criteria >	≤ 20%	80%-120%	80%-120%	75%-125%	75%-125%	≤ 10%
QC ID >	18-0014 Dup	LCS/RS	LCS/BS	18-0001 MS	18-0003 + CCV	18-0003 5-fold Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Diff
U 238	12%	90%	96%	90%	96%	0.6%

Internal Stan

Li 6 (IS)	93%	105%	104%	93%	103%	105%
Sc 45 (IS)	96%	103%	104%	95%	102%	105%
Y 89 (IS)	90%	98%	99%	89%	97%	99%
In 115 (IS)	92%	99%	98%	93%	97%	97%
Tb 159 (IS)	94%	95%	96%	93%	96%	98%
Bi 209 (IS)	91%	101%	94%	97%	94%	98%

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.

Radiochemistry M&TE List

Description	Manufacturer/ SW	Model	Serial Number/ Property Number	Location	M&TE Category	Calibration Frequency	Calibration Standards	SN/ Lot#	Acquisition Date
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 3.00/Build 5	3100TR	WD48466	RPL/425	2	With use	Blank, H-3, and C-14 sealed sources & Quench curves (efficiency) for H-3,C-14, and Ni-63		9/2006
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 5.00/Build 3	5110TR	WD56319	RPL/425	2	With use	Blank, H-3, C-14 instrument performance assessment (IPA) sources & Quench curves (efficiency) for H-3 & C-14		8/2015
Liquid Scintillation Counter	Perkin Elmer QuantaSmart 4.00/Build 5	3110TR	PT27127	RPL/425	2	With use	Blank, H-3, C-14 instrument performance assessment (IPA) sources & Quench curves (efficiency) for H-3 & C-14		5/2011

~~Liquid Scintillation Counter~~	~~Packard Tri Carb Packard Tri-Carb 2700TR software version 1.04 dated 9/99. The software runs on a DOS-2000 platform on a PC computer~~	~~2550~~	~~401664/ WD06783~~	~~RPL/425~~					
Automated NaI	Perkin Elmer Wizard 2	2480	WD56320	RPL/93	2	With use	I-129 instrument performance assessment source (IPA)		8/2015
Uranium									
Uranium Analyzer/ Kinetic Phosphorescence Analyzer	Chemcheck Instruments, Inc. The software used to control the instrument is KPAWIN Software, Version 1.2.9 Release 1999	KPA-11R	Serial number: 1045050203 WD59770	RPL/525		With use	Too many to list		1994

Radiochemistry M&TE List

ID	Number of Detectors	Property #	Model or Serial #	Location, in Bldg. 325	Procedures using the M&TE	PM Contract?
Dual Alpha Beta Gas Proportional Counters						
Oxford LB4100	16	WD13066	L8285-O	425	Total alpha, total beta, Sr/Y90, Tc99	Y
Alpha Counters						
Ludlum	10	multiple	multiple	425	Total Alpha	N
AEA (Ortec)*	32	multiple	multiple	425	AEA (total alpha, Pu, Am/Cm,Np)	N
Liquid Scintillation						
Perkin Elmer 3100TR	1	WD48466	DG08061340	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Perkin Elmer 3110TR	1	PT27127	DG03117614	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
Perkin Elmer 5110TR	1	WD56319	SGLO344150055	425	H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90	Y
~~Packard 2550~~	~~1~~	~~WD06783~~	~~401664~~	~~425~~	~~H-3, C-14, Ni-63, Se-79, Pu-241, Sr-90~~	~~Y~~
Gamma						
HpGe*	25	see list	see list	425/93	GEA	N
LEPS (x-rays)*	4	see list	see list	425	X-ray (Fe-55, Ni-59, Nb-93m)	N
Well HPGe*	3	see list	see list	93	GEA – In development	N
Anti-Compton*	2	see list	see list	93	GEA – In development	N
Perkin Elmer 2480 Wizard Automated NaI	1	WD56320	SGWZ28150290	93	In development	Y
Uranium						
Chemcheck	1	WD59770	KPA11R	525	Uranium KPA	N
***Spectral Analysis System**						
~~Canberra - VAX 3000~~	~~1~~	~~WD12890~~	~~PE42AJB~~	~~425~~	~~AEA, GEA~~	~~N~~
~~Canberra - VAX 3100~~	~~1~~	~~WC38624~~	~~KA235W0225~~	~~425~~	~~AEA, GEA (Backup VAX)~~	~~N~~

List of Gamma Detectors

Name	Property Tag	Vendor	Model	Serial #	Type	Eff. %	CMC?
C	WD25157	EG&G Ortec	GEM70200P	33TP40378A	Ge	70	Y
D	WD06582	Princeton	IGC7022SD	2542	Ge	70	Y
E	WD12833	EG&G Ortec	GEM100210	34P40535A	Ge	100	Y
G	WD35719	Canberra	GC4020	9923007	Ge	40	Y
H	PT00941	Tennelec	CPVS3040195	6028	Ge	40	Y
I	WD12076	EG&G Ortec	GLP3638510P	33TE187	LEPS	X-RAY	Y
J	WD12075	EG&G Ortec	GLP3638510P	33TE190	LEPS	X-RAY	Y
K	WD06581	Princeton	NIGC6022SD	3612	P-Ge	60	Y
L	WD36413	EG&G Ortec	GEM20P-Plus	44TP21884A	Ge	20	Y
M	WD73130	Canberra	GC10021	9722	Ge	100	Y
N	WD73129	Canberra	GC10021	9716	Ge	100	Y
~~P~~	~~WD59508~~	~~EG&G Ortec~~	~~GEM30210~~	~~24P80V~~	~~Ge~~	~~30~~	~~Y~~
T	WD81868	EG&G Ortec	GEM20180	35TP21056A	Ge	20	Y
~~W1~~	~~PT27133~~	~~Canberra~~	~~GC4018~~	~~9919~~	~~Ge~~	~~40~~	~~Y~~
F	PT16418	Canberra	GC10021	10182	Ge	100	Y
Q	PT16419	Canberra	GC10021	10188	Ge	100	Y
R	PT16420	Canberra	GC10021	10181	Ge	100	Y
S	PT16430	Canberra	GC10021	10101	Ge	100	Y
U	PT16429	Canberra	GC10021	10178	Ge	100	Y
V	PT16428	Canberra	GC10021	10150	Ge	100	Y
W	PT16394	Canberra	GC10021	10129	Ge	100	Y
X	PT16402	Canberra	GC10021	10104	Ge	100	Y
Y	PT16401	Canberra	GC10021	10145	Ge	100	Y
Z	PT16410	Canberra	GC10021	10212	Ge	100	Y
LEPS1	WB61769	Ortec	101351600S	20M102	Ge	X-RAY	Y
LEPS2	WD48418	Canberra	GL2015R	6068240	Ge	X-RAY	Y
WA	WD81452	Ortec	GEM100210	35-P-40642A	Ge	100	Y
WB	WD81454	Ortec	GEM100210	34-P-40481A	Ge	100	Y
WC	WD81453	Ortec	GEM100210	34-P-40522A	Ge	100	Y
Well1	WD81450	Ortec	GEM170230-S	34-P-30914B	Ge Well	N/A	Y
Well2	WD81449	Ortec	GEM170230-S	34-P-40544A	Ge Well	N/A	Y
Well3	WD81451	Canberra	GCW2523-S	11051527	Ge Well	N/A	Y
AC1	PT27479	Ortec	GEM4276LBCHJ	54P33125A	Ge-AC	45	Y
AC2	WD56318	Canberra	GX5020	5945022	Ge-AC	50	Y

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