PNNL-34395, Rev. 0
RPT-DFTP-037, Rev. 0

Cesium Removal from 5.5 and 7.0 M Na AP-105 Using Crystalline Silicotitanate

July 2023

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EL Campbell
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Prepared for the U.S. Department of Energy
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Pacific Northwest National Laboratory
Richland, Washington 99354

Summary

The Tank Side Cesium Removal (TSCR) system, currently operational by Washington River Protection Solutions, LLC, prepares initial low-activity Hanford waste tank supernate feeds for the Low-Activity Waste Facility at the Hanford Waste Treatment and Immobilization Plant (WTP). In addition to entrained solids removal from the supernate, the primary goal of TSCR is to remove cesium-137 (^{137}Cs) by ion exchange, allowing contact handling of the liquid effluent product at the WTP as governed by a waste acceptance criterion (WAC). Specific to ^{137}Cs, this requirement is <3.18E-5 Ci ^{137}Cs/mole of Na.[1] Crystalline silicotitanate (CST) manufactured by Honeywell UOP, LLC (product IONSIV R9140-B) has been selected as the ion exchange media for TSCR.

CST is a non-elutable inorganic material that has demonstrated robust chemical, physical, and radiation tolerance while maintaining functionality. However, testing to date on actual tank waste samples has been limited to Na concentrations between 5 and 6 M Na[2,3,4,5,6] while actual tank conditions can reach upwards of 9 M Na. Testing with feed from Hanford tank AP-105 incorporated testing at both 5.5 and 7 M Na in order to evaluate the impact of Na concentration on volume of waste processed before reaching the WAC. A 6-L volume of 5.5 M Na AP-105 and an 8-L volume of 7 M Na AP-105 were processed through the Radioactive Waste Test Platform system, established at Pacific Northwest National Laboratory to support small-scale waste qualification efforts. The columns consisted of 6- to 9-mL CST beds (CST lot 2002009604, sieved to screen out >30 mesh particles) placed in 1.5-cm-inner-diameter columns. Feed was processed at 1.9 bed volumes (BVs) per hour; the flowrate, in terms of contact time with the CST bed, matched the expected flowrate at TSCR. Table S.1 and Figure S.1 summarize the measured AP-105 Cs load performance for each feed condition.

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[1] 24590-WTP-ICD-MG-01-030, Rev. 1. 2017. *ICD 30 – Interface Control Document for Direct LAW Feed*. Bechtel National, Inc., Richland, Washington.

[2] Fiskum SK, AM Rovira, HA Colburn, AM Carney, and RA Peterson. 2019a. *Cesium Ion Exchange Testing Using a Three-Column System with Crystalline Silicotitanate and Hanford Tank Waste 241-AP-107*. PNNL-28958, Rev. 0; RPT-DFTP-013, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

[3] Fiskum SK, AM Westesen, AM Carney, TT Trang-Le, and RA Peterson. 2021b. *Ion Exchange Processing of AP-105 Hanford Tank Waste through Crystalline Silicotitanate in a Staged 2- then 3-Column System*. PNNL-30712, Rev. 0; RPT-DFTP-025, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

[4] Westesen AM, SK Fiskum, HA Colburn, JR Allred, MR Smoot, and RA Peterson. 2021a. *Cesium Ion Exchange Testing Using Crystalline Silicotitanate with Hanford Tank Waste 241-AP-107*. PNNL-27706, Rev, 1; RPT-DFTP-011, Rev. 1. Pacific Northwest National Laboratory, Richland, Washington.

[5] Westesen AM, SK Fiskum, AM Carney, EL Campbell, TT Trang-Le, and RA Peterson. 2021b. *Reduced Temperature Cesium Removal from AP-107 Using Crystalline Silicotitanate*. PNNL-31868, Rev. 0; RPT-DFTP-027, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

[6] Westesen AM, EL Campbell, AN Williams, AM Carney, TT Trang-Le, and RA Peterson. 2022. *Reduced Temperature Cesium Removal from AP-101 Using Crystalline Silicotitanate*. PNNL-32911 Rev. 0; RPT-DFTP-034, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

Table S.1. AP-105 5.5 and 7 M Na Column Performance Summary with CST

AP-105 Feed Condition	Column	WAC Limit Breakthrough (BVs)	50% Cs Breakthrough (BVs)	^{137}Cs Loaded (µCi/g)	Cs Loaded (mmoles/g CST)
5.5 M Na	Lead	172	888[(b)]	67,632	0.0384
	Lag	679[(b)]	NA	1942	0.0011
7 M Na	Lead	103	791	83,805	0.0476
	Lag/Middle	496	1332[(b)]	15,404	0.0088
	Polish[(a)]	885[(b)]	NA	569	0.0003

(a) The polish column was positioned in place after 470 BVs were processed through the lead-lag column system.
(b) Extrapolated value
BV = bed volume, 6 mL for 5.5 M Na and 9 mL for 7 M Na AP-105
The time weighted average flowrate was 1.9 BV/h.

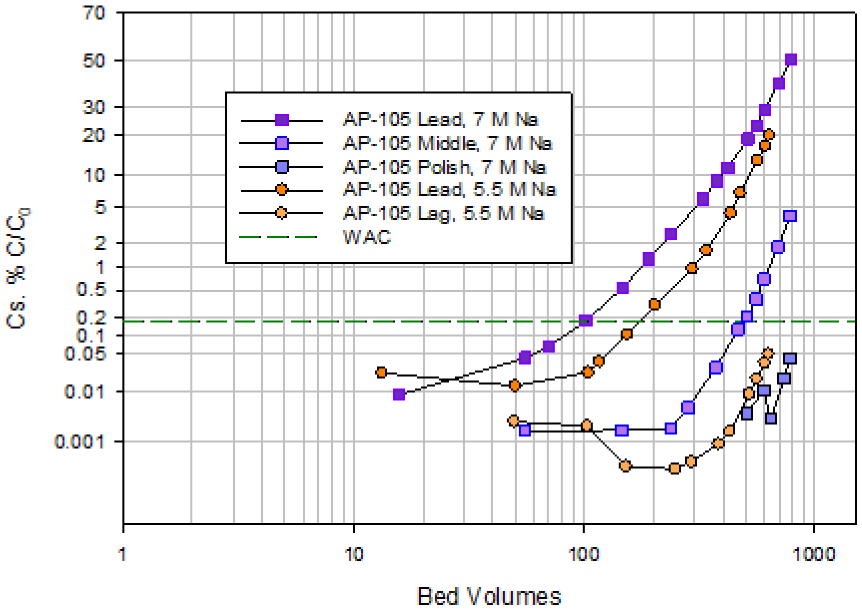

Figure S.1. Column Cs Load Profiles for 5.5 and 7 M Na AP-105

Batch contact tests were performed with the 7 M Na AP-105 tank waste at four Cs concentrations, each at a phase ratio of 200 (liquid volume to dry CST mass). The distribution coefficient (K_d) at the equilibrium condition of 7.16E-5 M Cs (7 M Na AP-105 feed condition) was 1101 mL AP-105/g CST. With a CST bed density of 1.00 g/mL (<30 mesh CST), this K_d corresponded to a predicted 50% Cs breakthrough of 1101 BVs. The observed column performance 50% Cs breakthrough (791 BVs) was ~32% short of the predicted performance (1101 BVs). The batch contact testing predicted a Cs load capacity of 0.079 mmoles Cs/g dry CST at the equilibrium Cs concentration. The Cs breakthrough from the lead column at the end of processing resulted in 0.048 mmoles Cs/ g CST – 60% of the maximum Cs loading at feed condition based on prediction from batch contact testing.

The AP-105 composite feed and composite effluent for each respective feed were characterized to understand the fractionation of selected metals and radionuclides. Concentrations and recoveries of the selected analytes are summarized in Table S.2; those with low recovery were assumed to be adsorbed onto CST. Large fractions of barium (Ba), cadmium (Cd), lead (Pb), plutonium (Pu), and strontium (Sr/^{90}Sr) significantly fractionated to the CST.

Table S.2. Recoveries of Analytes of Interest in the 5.5 and 7 M Na AP-105 Effluent

Metals / Non-metals	Analyte	Feed Concentration (M)	Effluent Concentration (M)	Fraction in Effluent
5.5 M Na AP-105	Al	5.05E-01	4.79E-01	94%
	Ba	3.69E-06	[2.9E-07]	8%
	Ca	1.29E-03	8.51E-04	65%
	Cd	[2.3E-05]	[9.9E-06]	43%
	Cr	6.21E-03	5.80E-03	92%
	Cu	1.13E-04	1.02E-04	88%
	K	9.59E-02	8.80E-02	91%
	Na	5.58E+00	5.33E+00	94%
	P	1.41E-02	1.15E-02	80%
	Pb	[1.9E-04]	[3.3E-05]	17%
	S	3.41E-02	3.24E-02	94%
7 M Na AP-105	Al	6.42E-01	6.09E-01	97%
	Ba	6.40E-06	[7.1E-07]	11%
	Ca	1.19E-03	9.30E-04	80%
	Cd	[4.1E-05]	1.72E-05	43%
	Cr	7.92E-03	7.52E-03	97%
	Fe	[8.9E-05]	[2.4E-05]	27%
	K	1.20E-01	1.17E-01	100%
	Na	7.18E+00	6.76E+00	97%
	P	1.70E-02	1.41E-02	85%
	Pb	[1.1E-04]	[5.0E-05]	47%
	S	4.36E-02	3.98E-02	94%
	Ti	[4.7E-05]	[3.3E-05]	72%
	U	[3.2E-04]	[4.9E-05]	16%
	Zn	[2.4E-05]	[1.1E-05]	46%

Radionuclides(a)	Analyte	Feed Concentration (µCi/mL)	Effluent Concentration (µCi/mL)	Fraction in Effluent
7 M Na AP-105	^{90}Sr	3.50E-01	1.69E-03	0.5%
	^{99}Tc	6.39E-02	5.31E-02	85%
	^{137}Cs	1.38E+02	2.76E+00	2%
	^{238}Pu	6.23E-06	5.21E-06	86%
	$^{239+240}$Pu	2.93E-05	2.15E-05	75%
	^{241}Am	1.81E-04	1.33E-04	75%

(a) Reference date is April 2023.

Notes:

Values in brackets [ ] were greater than or equal to the method detection limit but less than the estimated quantitation limit, with errors likely to exceed 15%.

The recovered fractions are calculated with values containing more significant figures than shown; using listed values may result in a slight difference due to rounding.

Acknowledgements

The authors thank Shielded Facility Operations staff Victor Aguilar, Hollan Brown, and Robert Cox for hot cell operations (ion exchange column system setup, processing, and sample removal). We thank the Analytical Support Operations (ASO) staff Leah Arrigo, Steven Baum, Lori Darnell, Sam Morrison, Christian Perez, Matthew Risenhuber, and Dave Blanchard for the sample analysis, data processing, and reporting. The authors thank Renee Russell for conducting the technical reviews of the calculation files and this report. The authors also thank David MacPherson for the quality review of the calculation files and this report and Matt Wilburn for his technical editing contribution to this report.

Acronyms and Abbreviations

ASO	Analytical Support Operations
ASR	Analytical Service Request
BV	bed volume
CST	crystalline silicotitanate
DF	decontamination factor
DI	deionized
EQL	estimated quantitation limit
erf	error function
FD	feed displacement
GEA	gamma energy analysis
IC	ion chromatography
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectroscopy
ID	identification
LAW	low-activity waste
MDL	method detection limit
NA	not applicable
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
R&D	research and development
SV	system volume
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
TSCR	Tank Side Cesium Removal
WAC	waste acceptance criteria
WRPS	Washington River Protection Solutions, LLC
WTP	Hanford Waste Treatment and Immobilization Plant
WWFTP	WRPS Waste Form Testing Program

Contents

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1.0 Introduction

The initial production of immobilized low-activity waste (LAW) is enabled by feeding tank waste supernate from the Hanford tank farms to the Tank Side Cesium Removal (TSCR) system and subsequent immobilization in the LAW Facility at the Hanford Waste Treatment and Immobilization Plant (WTP). Decanted tank waste supernate will be pretreated using TSCR to meet the WTP LAW Facility waste acceptance criteria (WAC). Specific to ^{137}Cs, this requirement is <3.18E-5 Ci ^{137}Cs/mole of Na.[7] The key process operations for treating the waste include solids filtration and cesium removal. The current TSCR design uses a dead-end filtration system for solids removal and crystalline silicotitanate (CST), a non-elutable ion exchange material, for cesium removal.

The treated LAW WAC requires supernate Na concentrations to fall between 5 and 8 M Na prior to WTP vitrification, with nominal TSCR operating conditions occurring between 5 and 6 M Na. Laboratory-scale ion exchange processing using TSCR prototypic unit operations continues to contribute toward Washington River Protection Solutions, LLC (WRPS) establishing accurate process flowsheets for the individual feed campaigns planned for TSCR. The Radioactive Waste Test Platform established at Pacific Northwest National Laboratory (PNNL) has been used to conduct laboratory-scale unit operation process steps on several wastes within the nominal operating bounds of TSCR processing (5 to 6 M Na) (Fiskum et al. 2019a, 2021b; Westesen et al. 2021a,b, 2022). In an effort to evaluate the impact of Na concentration on volume of waste processed before reaching the WAC, testing with AP-105 tank waste (reported herein) assessed ion exchange Cs removal at 5.5 and 7 M Na.

Figure 1.1 shows a temperature profile of the AP-105 tank waste supernate between January 2021 and September 2022; the temperature averaged 19.6 °C with a range of 17.1 to 23.1 °C. Zheng et al. (1997) showed that CST Cs capacity decreased as contact temperature increased. To match previous laboratory-scale ion exchange testing (Westesen et al. 2021b, 2022), an operating temperature of 16 °C was established for this AP-105 testing at 5.5 and 7 M Na. Operating at the lower range of temperatures will help to predict the maximum ^{137}Cs loading onto the CST in the TSCR system and guide the appropriate operating restrictions to ensure the column loading limit will not be exceeded.

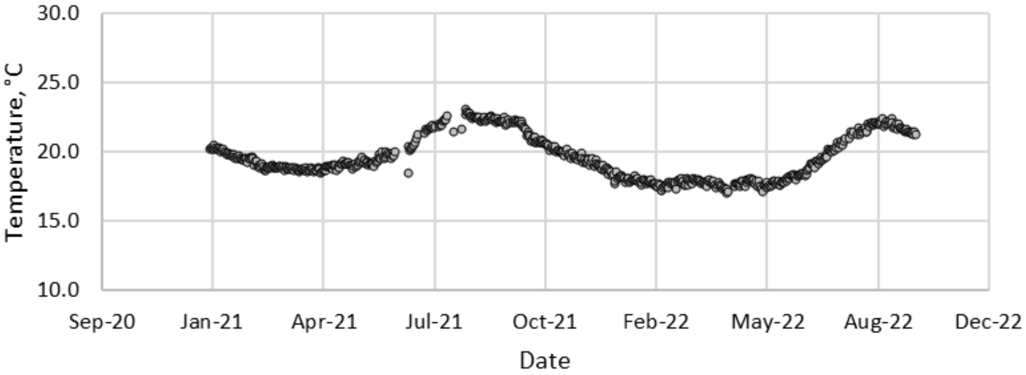

Figure notes: Data collected from 241-AP-105 Location Riser 4 18.
Data downloaded from Tank Waste Information Network System on December 15, 2022.

Figure 1.1. AP-105 Tank Waste Temperatures from January 2021 to December 2022

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[7] 24590-WTP-ICD-MG-01-030, Rev. 1. 2017. *ICD 30 – Interface Control Document for Direct LAW Feed*. Bechtel National, Inc., Richland, Washington.

The primary objective of the work described in this report was to test AP-105 Cs removal at two Na concentrations (5.5 and 7 M Na) using TSCR prototypic hybrid column processing at an operating temperature of 16 °C and establish Cs load profiles. For this testing, a lead-lag column system was used, and once the lag column effluent reached the WAC limit, a polish column was positioned after the lag column and processing continued in a lead-lag-polish configuration. Additional objectives of the current study are as follows:

1. Conduct batch contact testing with CST at 13, 16, 21, and 35 °C to determine the Cs load capacity of 7 M Na AP-105.
2. Conduct batch contact testing with CST at 16 °C and 5.5 M Na AP-105 and compare to previously reported capacity values (Fiskum et al. 2021b).
3. Compare the 16 °C 5.5 and 7 M Na AP-105 Cs load profiles.
4. Compare the 16 °C 5.5 M Na AP-105 Cs load profile to the previously reported 28 °C 5.5 M Na AP-105 load curve (Fiskum 2020).
5. Analyze the 5.5 and 7 M Na AP-105 ion exchange feed and effluent to derive the fates of key analytes (^{90}Sr, ^{99}Tc, ^{137}Cs, ^{238}U, $^{239+240}$Pu, ^{237}Np, ^{241}Am, Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Nb, Ni, P, Pb, S, Sr, Ti, Zn, Zr).
6. Provide 7 M Na Cs-decontaminated AP-105 for vitrification (conducted in spring 2023 and addressed in a separate report).

WRPS funded PNNL to conduct testing with AP-105 tank waste under the Task 2 of the WRPS statement of work *FY 2022 Radioactive Waste Test Platform Testing*, Rev. 2, Requisition 351656, dated July 21, 2022. There are no deviations from the statement of work.

2.0 Quality Assurance

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

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

3.0 Test Conditions

This section describes the CST media, AP-105 tank waste, column ion exchange conditions, sample analysis, and batch contact conditions. All testing was conducted in accordance with a test plan prepared by PNNL and approved by WRPS.[8]

3.1 CST Media

The CST used in this testing was procured by WRPS as ten 5-gallon buckets (149 kg total) of IONSIV R9140-B,[9] lot number 2002009604, from Honeywell UOP, LLC. The CST was transferred to PNNL for use in laboratory testing described herein. Details of the procurement and material properties can be found elsewhere (Fiskum et al. 2019b). Before use in column and batch contact testing, the <30-mesh CST fraction was first pretreated by contacting with 0.1 M NaOH successively until fines were no longer observed.

3.2 AP-105 Tank Waste Sample

WRPS collected multiple samples (36 each at nominally 250 mL) from the AP-105 Hanford tank in October 2022. As received, the AP-105 tank waste Na concentration measured 8.52 M Na with a density of 1.409 g/mL at an ambient cell temperature of 25.6 °C. Eleven of the 36 tank waste samples were composited and diluted to achieve a targeted 1.25 g/mL density and 5.5 M Na concentration. The remaining 25 jars were composited and diluted to target a 1.30 g/mL density and 7.0 M Na concentration. Dilution of the waste to 5.5 M Na combined nominally 1 L of AP-105 tank waste with 0.585 L of Columbia River process water. For the 7 M Na target, 0.246 L of Columbia River process water was combined for every 1 L of AP-105 tank waste. The AP-105 and water were mixed, and density was measured to verify the target dilution had been achieved. Density was measured for the 5.5 and 7 M Na solutions via 10-mL Class A volumetric flask and an analytical balance at an ambient cell temperature of 25.7 °C, and resulted in values of 1.263 and 1.312 g/mL, respectively. The diluted 5.5 and 7 M Na AP-105 samples were chilled to 16 °C before being filtered with a media grade 5 filter (Allred et al. 2023). After filtration, four bottles of 5.5 M Na AP-105 and seven bottles of 7 M Na AP-105, containing nominally 1 L each, were made available for ion exchange testing.

The densities and ^{137}Cs concentrations of each of the 11 bottles of diluted and filtered AP-105 were measured. The average relative standard deviations for both measurements on each feed campaign fell within 6% (see Table 3.1); therefore, the 5.5 and 7 M Na AP-105 feeds in respective containers were considered uniform. The total Cs concentration for each diluted waste was calculated from the respective ^{137}Cs concentration (in terms of µg/mL with unit conversion per the specific activity) and ^{137}Cs mass fraction (average 15.1 wt%). The total Cs concentration in the 5.5 M Na AP-105 sample was determined to be 8.31 µg/mL or 6.21E-5 M and 9.58 µg/mL or 7.16E-5 M for the 7 M Na AP-105 sample.

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[8] Westesen AM. 2022. Test Plan TP-DFTP-137, Rev. 0.0. *FY23 Cesium Ion Exchange Testing with 5.5 and 7.0 M Na AP-105 Tank Waste Using Crystalline Silicotitanate Media*. Pacific Northwest National Laboratory, Richland, Washington. Not publicly available.

[9] R9140-B is provided in the sodium form by the vendor.

Table 3.1. 5.5 and 7 M Na AP-105 Ion Exchange Feeds

AP-105 Na M	Bottle ID	Density	μCi ^{137}Cs/mL
5.5 M Na	IX-AP5-1	1.272	107.2
	IX-AP5-2	1.264	112.9
	IX-AP5-3	1.251	105.7
	IX-AP5-4	1.265	111.0
	Avg	1.263	109.2
	RPD	0.69	3.0
7.0 M Na	IX-AP5-5	1.297	130.8
	IX-AP5-6	1.316	129.6
	IX-AP5-7	1.304	110.2
	IX-AP5-8	1.308	126.4
	IX-AP5-9	1.316	131.9
	IX-AP5-10	1.322	126.6
	IX-AP5-11	1.322	129.7
	Avg	1.312	126.5
	RPD	0.70	5.9
RPD = relative percent standard deviation			

3.3 Ion Exchange Column Processing at 16 °C

This section describes the ion exchange column system and 5.5 and 7 M Na AP-105 process conditions. The preparations and column testing were conducted in accordance with a test instruction.[10,11]

3.3.1 Ion Exchange Column System

Figure 3.1 provides a piping and instrumentation diagram of the ion exchange process system used for the 5.5 and 7 M Na AP-105 testing. The columns were housed in a 12-inch × 6-inch × 15-inch (W×D×H) insulated box, previously used and described in Westesen et al. (2021b). Heat exchange was conducted with ethylene glycol from a chilled circulating bath flowing through copper tubing on the inner panels of the box. The internal temperature was monitored with a thermocouple seated inside a vial of water adjacent to the columns.

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[10] Westesen AM. 2022. Test Instruction TI-DFTP-138. *Cesium Removal from 5.5 M Na AP-105 Using Crystalline Silicotitanate at 16 °C.* Pacific Northwest National Laboratory, Richland, Washington. Implemented December 2022. Not publicly available.

[11] Westesen AM. 2022. Test Instruction TI-DFTP-139. *Cesium Removal from 7 M Na AP-105 Using Crystalline Silicotitanate at 16 °C.* Pacific Northwest National Laboratory, Richland, Washington. Implemented December 2022. Not publicly available.

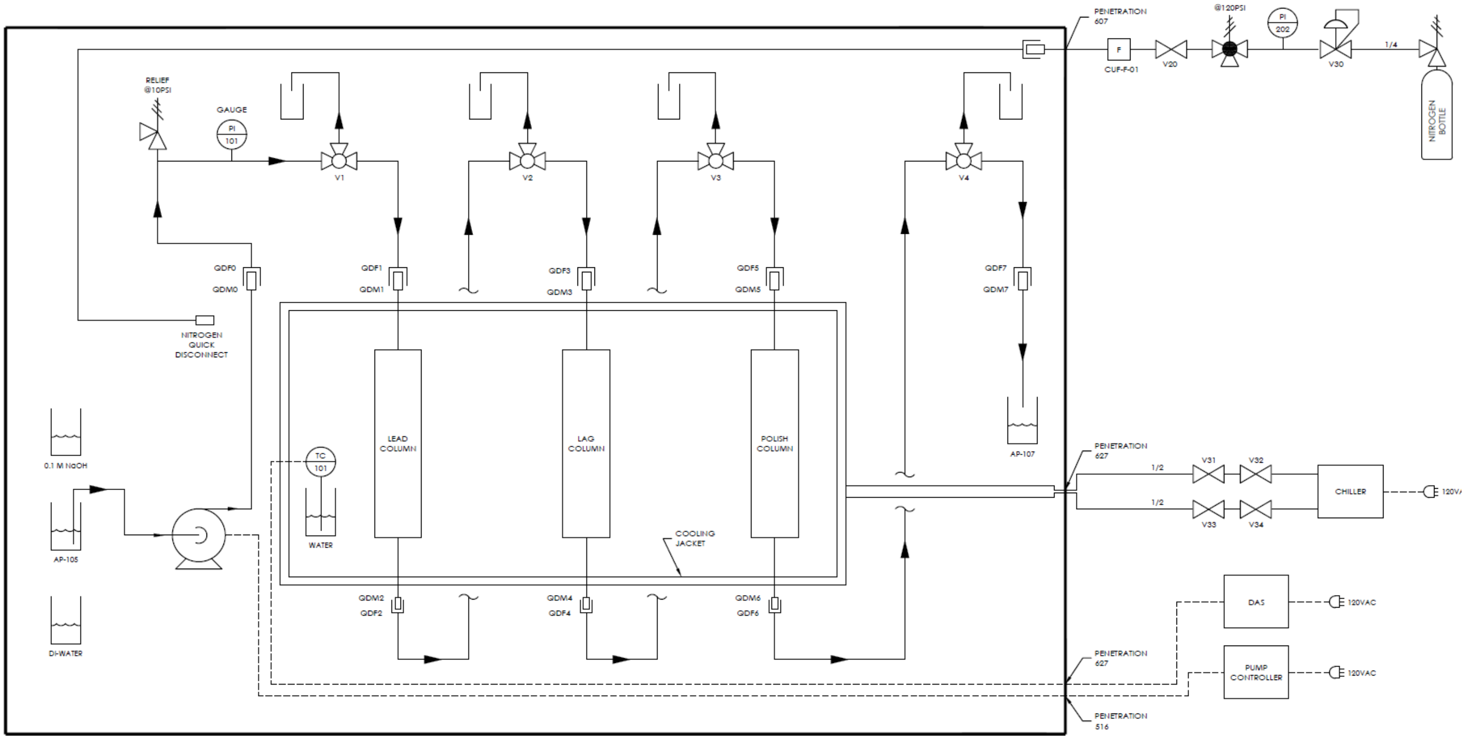

Figure 3.1. Chilled Ion Exchange Piping and Instrumentation Diagram

Chromaflex column assemblies (previously described by Westesen et al. 2022) made of borosilicate glass measured 9 cm tall with an inside diameter of 1.5 cm (corresponding to a CST volume of 1.77 mL/cm) were used. The CST was supported by an in-house-constructed support consisting of a 200-mesh stainless steel screen tack welded onto a stainless-steel O-ring. The flared cavity at the bottom of each column was filled to the extent possible with 4-mm-diameter glass beads to minimize the mixing volume below the CST bed. An adhesive centimeter scale with 1-mm divisions (Oregon Rule Co., Oregon City, OR) was affixed to each column with the 0-point coincident with the top of the support screen.

The valve manifold was the same as the one used previously for AP-107 and AP-101 processing reported in Westesen et al. (2021b and 2022). Four Swagelok valves (V1 through V4 in Figure 3.1) were installed on the valve manifold. Valve 1 was placed at the outlet of the pressure gage and used to isolate the columns from the pump (when in the closed position) and purge the tubing from the inlet to valve 1 (when placed in the sampling position). Lead column samples were collected at valve 2, the lag column samples were collected at valve 3, and the polish column samples were collected at valve 4. The gross AP-105 effluent, feed displacement (FD), water rinse, and flushed fluid were collected at the effluent line.

Aliquots of settled CST (pretreated, <30 mesh) were measured using a graduated cylinder and then quantitatively transferred to each individual column. Testing with 5.5 M Na AP-105 used 6.0-mL CST in each column while 7 M Na testing used 9 mL CST in each column. The CST was allowed to settle through the 0.1 M NaOH solution, thus mitigating gas bubble entrainment. The columns were tapped with a rubber bung until the CST height no longer changed. The CST bed volume (BV) corresponded to the settled CST media volume as measured in the graduated cylinder prior to transferring the media into the ion exchange column. The reference CST BV was 6.0 mL for 5.5 M Na AP-105 testing and 9.0 mL for 7.0 M Na AP-105 testing. Figure 3.2 is a photograph of the ion exchange system in-cell during AP-105 processing.

Figure 3.2. Ion Exchange Assembly in the Hot Cell

3.3.2 5.5 M Na AP-105 Tank Waste Process Conditions

Once the ion exchange columns were installed within the chiller box, a flow of 0.1 M NaOH was used to verify system integrity and calibrate the pump. The 5.5 M Na AP-105 contained in various 1.5-L polyethylene containers from the filtration process (Allred et al. 2023) was used as the first AP-105 ion exchange feed campaign. To provide stability, bottles were positioned in a bottle stand with the feed line inserted through the lid. When the contents in a feed bottle decreased to ~300 mL, the next bottle in line was moved to the feed position and the residual contents were poured into the new feed bottle. The 5.5 M Na AP-105 feed was processed downflow through the ion exchange media beds, lead to lag. Effluent was collected in ~1.3-L increments. This volume limitation allowed for safe transfer out of cell in 1.5-L polyethylene bottles. The lag column effluent Cs concentration was closely monitored but did not reach the WAC limit; thus, a polish column was not used during this feed campaign.

After the 5.5 M Na AP-105 processing (also “loading” in subsequent discussion) was completed, ~12 BVs (72 mL) of 0.1 M NaOH FD followed by ~12 BVs of deionized (DI) water were passed downflow through the system to rinse residual feed out of the columns and process lines. Twelve BVs is equivalent to ~1.7 times the fluid-filled system volume (SV).

Figure 3.3 provides daily temperature and flowrate profiles of the 5.5 M Na AP-105 processing as it went through the columns. Temperature was measured using a thermocouple placed inside a vial of water that sat within the exchanger. The exchanger temperature averaged 16.0 °C throughout the testing, with min/max temperatures of 15.6 and 16.3 °C, respectively. The pump head stroke length was close to the minimum at which it could be set. The stroke rate was toggled between 23.2 and 26.3 (maximum fidelity of 0.1 units) to maintain the flowrate between 1.8 and 2.2 BV/h. Deviations from the calibrated stroke rate setpoint caused a 4-day delay before dialing in the target 1.90 BV/h flowrate. Test parameters, including process volumes, flowrates, and CST contact times, are summarized in Table 3.2.

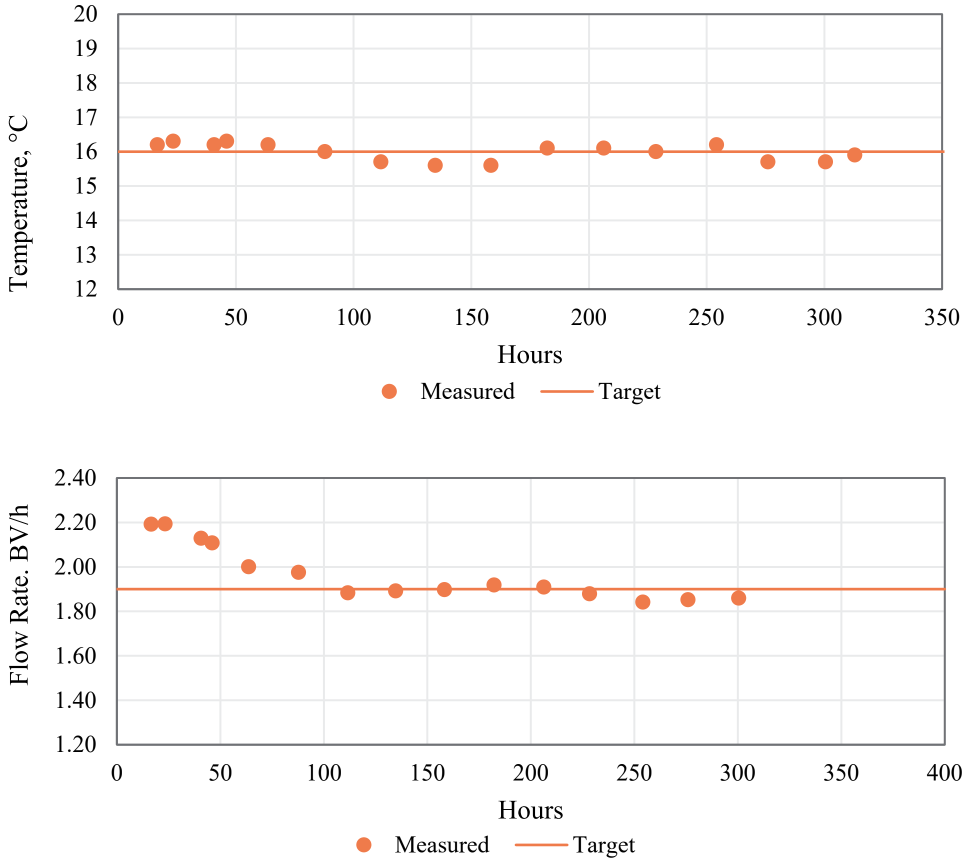

Figure 3.3. 5.5 M Na AP-105 Daily Column Temperature and Flowrate during Testing

Table 3.2. Experimental Conditions for 5.5 M Na AP-105 Column Processing at 16 °C, January 3-16, 2023

		Volume			Flowrate		Duration
Process Step	Solution	(BV)	(SV)	(mL)	(BV/h)	(mL/min)	(h)
Loading lead column	5.5 M Na AP-105	633. 7	NA	3802	1.96	0.198	324
Loading lag column[(a)]	5.5 M Na AP-105	611.9	NA	3804	1.96	0.198	324
Feed displacement	0.1 M NaOH	11.0	1.28	65.7	2.73	0.273	4.0
Water rinse	DI water	10.9	1.28	65.6	2.73	0.273	4.0
Flush with compressed air[(b)]	NA	7.8	0.91	46.6	NA	NA	NA

(a) The feed volume through the lag column was reduced relative to that of the lead column because samples collected from the lead column did not enter the lag column.
(b) The flush occurred on January 18, 2023, after the system sat in static contact with water rinse for 2 days.
BV = bed volume (6.0 mL as measured in graduated cylinder)
DI = deionized
SV = system volume (estimated 24 mL)
NA = not applicable

The total cumulative volume of 5.5 M Na AP-105 processed was 3.8 L (634 BVs). The 5.5 M Na AP-105 process cycle mimicked, as best as possible, the current process flow anticipated at the TSCR facility in terms of Na concentration, BV/h (i.e., contact time), FD, and water rinse as defined in the test plan. It was understood that the feed linear flow velocity in this small-column configuration (0.18 cm/min) could not begin to match that of the full-height processing configuration (7.3 cm/min, Fiskum et al. 2019b). The objective was to match contact time in the bed.

During the loading phase, nominal 2-mL samples were collected from the lead and lag columns at the sample collection ports (see Figure 3.1, valves 2 and 3). Sampling from the columns necessitated brief (~10-minute) interruptions of flow to the downstream columns. Samples were collected after the first 13 BVs were processed and again at nominal 12- to 92-BV increments. Only brief (~5-minute) interruptions were associated with changing the feed bottles.

The FD effluent was collected in bulk in a 125-mL polyethylene bottle. The water rinse was similarly collected. The fluid-filled volume was expelled with compressed air connected at the first quick disconnect in the system, *QDF0* (see Figure 3.1), in ~3 minutes. The collected volume (15.5 mL) did include the interstitial fluid space between the CST beads but was not expected to include fluid in the CST pore space. Hours of additional gas flow were required to dry the CST enough to be free-flowing such that it would effectively pour out of the columns into specially designed shielded containment for later examination (not addressed in this report). The recovered CST was 6.18 and 6.22 g for the lead and lag columns, respectively. With a CST bed density of 1.00 g/mL, quantitative recovery of the CST from the columns was estimated, with slight increases in mass on subsequent columns potentially due to CST fines carried over during air drying.

3.3.3 7 M Na AP-105 Tank Waste Process Conditions

After the conclusion of 5.5 M Na AP-105 testing, the system manifold was thoroughly rinsed with 0.1 M NaOH. Feed operations mimicked the 5.5 M Na AP-105 conditions detailed in Section 3.3.2. A 9.0-mL CST bed was used for the 7 M Na AP-105 testing; thus, the ~12 BV 0.1 M NaOH FD and DI water rinses following the loading phase had volumes of 108 mL each. The lag column reached the WAC after processing 464 BVs of feed, at which point the polish column was placed in line and processing continued until all available 7 M Na AP-105 had been treated.

Figure 3.4 provides daily temperature and flowrate profiles of the 7 M Na AP-105 processing as it went through the columns. Temperature was measured using a thermocouple placed inside a vial of water that sat within the exchanger. The exchanger temperature averaged 16.0 °C throughout the testing, with min/max temperatures of 15.6 and 16.3 °C, respectively. The stroke rate was adjusted throughout testing to maintain the flowrate at the targeted 1.9 BV/h. Test parameters, including process volumes, flowrates, and CST contact times, are summarized in Table 3.3.

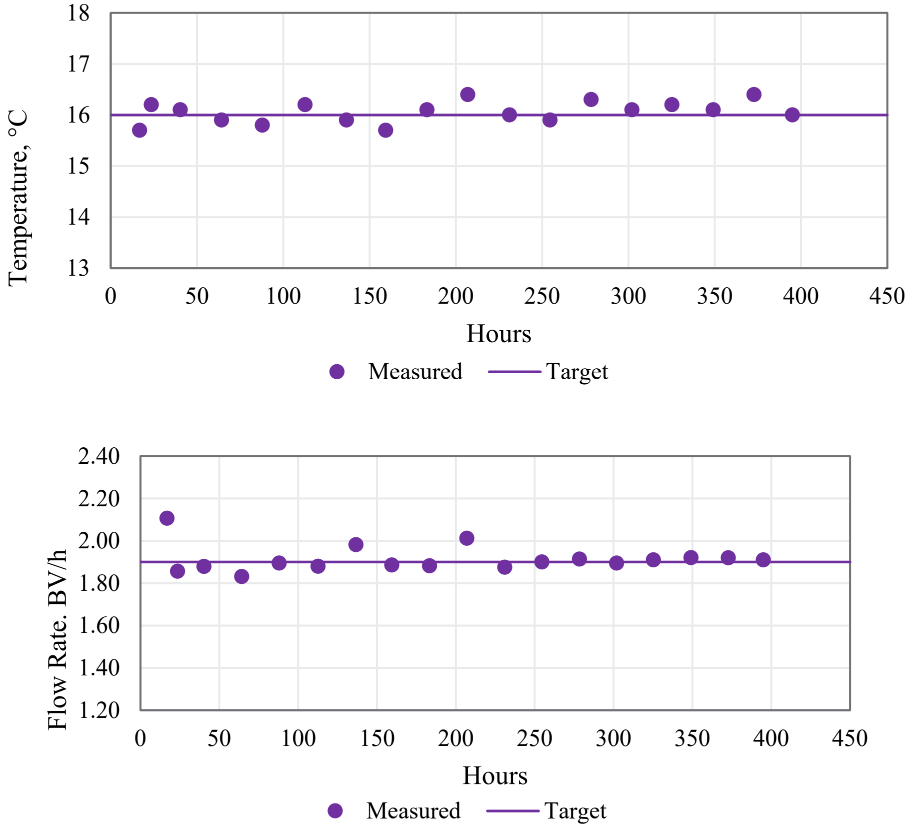

Figure 3.4. 7 M Na AP-105 Daily Column Temperature and Flowrate during Testing

Table 3.3. Experimental Conditions for 7 M Na AP-105 Column Processing at 16 °C, January 30 through February 16, 2023

		Volume			Flowrate		Duration
Process Step	Solution	(BV)	(SV)	(mL)	(BV/h)	(mL/min)	(h)
Loading lead column	7 M Na AP-105	796.2	NA	7166	1.95	0.292	414
Loading lag column[(a)]	7 M Na AP-105	792.4	NA	7207	1.95	0.292	414
Loading polish column[(b)]	7 M Na AP-105	332.5	NA	2992.4	1.95	0.292	198
Feed displacement[(c)]	0.1 M NaOH	10.8	1.34	97.1	2.73	0.410	4.0
Water rinse	DI water	10.1	1.25	90.5	2.85	0.427	3.5
Flush with compressed air	NA	4.4	0.58	39.8	NA	NA	NA

(a) The feed volume through the lag column was reduced relative to that of the lead column because samples collected from the lead column did not enter the lag column.
(b) The feed volume through the polish column was lower relative to that of the lead and lag columns because it was placed in position after 464 BVs were processed.
(c) The feed displacement occurred on February 20, 2023, after the system sat in static contact with AP-105 for 4 days.
BV = bed volume (9.0 mL as measured in graduated cylinder)
DI = deionized
SV = system volume (estimated 32 mL)
NA = not applicable

The total cumulative volume of 7 M Na AP-105 processed was 7.1 L (788 BVs). The recovered CST from each column was 8.73, 9.28, and 9.26 g for the lead, lag, and polish columns, respectively. With a CST bed density of 1.00 g/mL, quantitative recovery of the CST from the columns was estimated, with slight increases in mass on subsequent columns potentially due to CST fines carried over during air drying.

During processing of the final 7 M Na AP-105 feed bottle, dark solids were found to have accumulated in the bottom of the bottle (Figure 3.5). These solids passed through the media grade 5 backpulse dead end filter system (described in Allred et al. 2023). The solids were removed from the bottle, centrifuged to concentrate, and loaded out of the hot cell. The solids were centrifuged again to remove the bulk amount of the supernate and were washed twice with 10 mL of a 7 M Na simulant salt solution (1.1 M NaOH/5.9 M $NaNO_3$) in order to maintain pH. The clear supernate on the top of the solids was decanted and the remaining slurry was sent for low emission photon spectroscopy analysis for elemental characterization and was determined to be a complex matrix of Ni, Cr, Pb, Fe, Cd, and Cu.

Figure 3.5. 7 M Na Ion Exchange Solids

3.4 Batch Contact Conditions

Batch contact experiments with 7 M Na AP-105 effluent following ion exchange processing were conducted to evaluate Cs loading at four different temperatures. Stock solutions of 0.75 and 0.086 M $CsNO_3$ were prepared by dissolving the nitrate salt in a volumetric flask and diluted with DI water. Calculated volumes of Cs stock solutions were delivered to poly bottles and the mass of the spike was measured. The AP-105 effluent was spiked with ^{137}Cs, and nominally 120 mL was transferred into each poly bottle to achieve Cs concentrations of 1.2E-4, 3.3E-4, 8.5E-4, and 1.7E-2 M Cs. Solutions were prepared gravimetrically, and exact volumes were calculated from mass and density measurements. As a comparison, two stock solutions with Cs concentrations of 1.3E-4 and 3.3E-4 M Cs were prepared in the 5.5 M Na AP-105 effluent using the 0.086 M Cs spike solution. The 5.5 M Na AP-105 supernate contacts were only performed at 16 °C as corollary measurements to column testing with 5.5 M Na AP-105.

Nominal 0.075-g (dry mass basis) aliquots of CST were measured into 20-mL vials. F-factor samples were collected in duplicate, bracketing batch contact sample collection, and used to determine the dry mass of the exchanger. The F-factor was measured at nominally 105 °C with an average value of 0.918 for the 7 M contacts and 0.881 for the 5.5 M contacts. The F-factor at 105 °C measured at the time of the experiment was used to calculate the dry mass of CST for the 7 M and 5.5 M Na batch contact tests.

Aliquots (15-mL) of the AP-105 Cs stock solutions were added to the appropriate vials (in duplicate) and the exact solution volume transferred was calculated from net solution mass and density. The solution-to-mass phase ratio averaged 199 ± 4.

The 13 °C and ambient (25 ± 2 °C) batch contact tests were done concurrently. The 25 °C (ambient) samples were contacted on a Cole-Parmer (Vernon Hills, Illinois) large orbital shaker set to 240 rpm. The remaining three temperatures (13, 16, and 35 °C) were sequentially contacted in a Benchmark (Sayreville, New Jersey) Incu-Shaker refrigerated/heated orbital shaker set to 200 rpm. A vial of water co-located with each sample set was used to monitor the temperature over the ~ 240-hour contact time. The resulting temperature fluctuations are shown in Figure 3.6 with error bars representative of the 2.2 °C measurement uncertainty of a Type K thermocouple. The weighted mean temperature for each set of batch contacts is provided in Table 3.4.

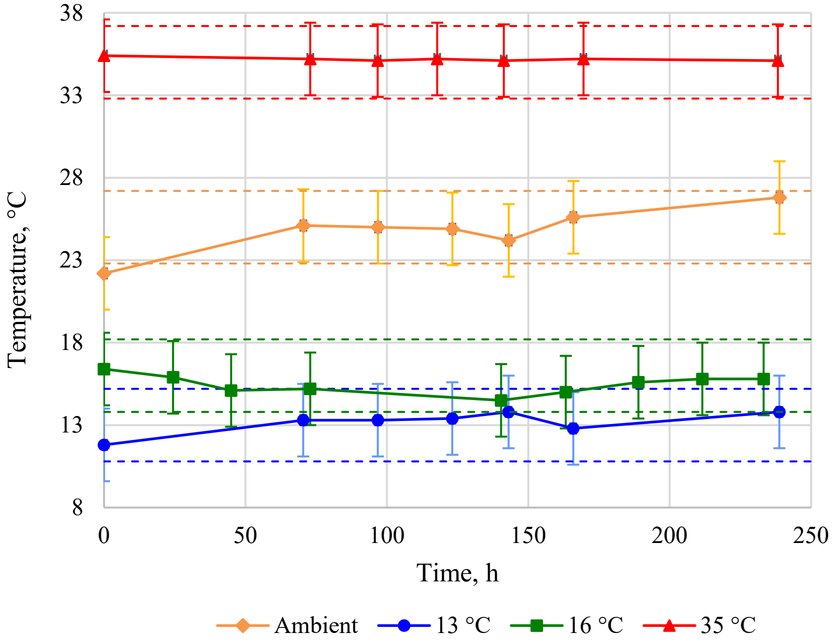

Figure 3.6. Temperature Profiles of Batch Contact Testing with AP-105 Tank Waste Supernate

Table 3.4. Average Contact Temperature

Target Temperature (°C)	Weighted Mean Temperature (°C)
13	13.1
16	15.3
21	24.9
35	35.2

After contact, 2 mL of the supernate was removed and filtered through a 0.45-micron pore size nylon syringe filter and transferred to a glass vial for gamma energy analysis (GEA). The ^{137}Cs activity measured by GEA in pre- and post-contacted solutions was used to determine the total Cs exchange. Analysis and data reduction were conducted using the methods previously reported (Fiskum et al. 2019a). The isotherm data were fitted to a Freundlich/Langmuir hybrid equilibrium fit (Hamm et al. 2002).

The batch distribution coefficients were calculated according to Eq (3.1).

$$\frac{(A_0 - A_1)}{A_1} \times \frac{V}{M \times F} = K_d \tag{3.1}$$

where A_0 = initial ^{137}Cs concentration (µCi/mL)
A_1 = final (equilibrium) ^{137}Cs concentration (µCi/mL)
V = volume of the batch contact liquid (mL)
M = measured mass of CST (g)
F = F-factor, mass of the 105 °C dried CST divided by the mass of the undried CST
K_d = batch-distribution coefficient (mL/g)

Final (equilibrium) Cs concentrations (C_{Eq}) were calculated relative to the tracer recovered in the contacted samples (A_1) and the initial metal concentration (C_0) according to Eq. (3.2)

$$C_0 \times \left(\frac{A_1}{A_0}\right) = C_{Eq} \tag{3.2}$$

where C_0 = initial Cs concentration in solution (µg/mL or M)
C_{Eq} = equilibrium Cs concentration in solution (µg/mL or M)

The equilibrium Cs concentrations loaded onto the CST (Q in units of mmoles Cs per gram of dry CST mass) were calculated according to Eq. (3.3)

$$\frac{C_0 \times V \times \left(1 - \frac{A_1}{A_0}\right)}{M \times F \times 1000 \times FW} = Q \tag{3.3}$$

where Q = equilibrium Cs concentration in the CST (mmole/g CST)
1000 = conversion factor to convert µg to mg
FW = Cs formula weight

3.5 Sample Analysis

Cesium load performance was determined from the ^{137}Cs measured in the collected samples relative to the native ^{137}Cs in each respective AP-105 feed. The collected samples were analyzed directly to determine the ^{137}Cs concentration using GEA. Cesium loading breakthrough curves for both the lead and lag columns were generated based on the feed ^{137}Cs concentration (C_0) and the effluent Cs concentration (C) in terms of % C/C_0.

A composite feed sample for each AP-105 feed (5.5 or 7 M Na) was prepared by collecting a pro-rated volume from each feed bottle and combining in a polyethylene vial; a composite effluent sample was similarly collected. Table 3.5 summarizes the specific sample collections and targeted analytes along with the cross-reference to the Analytical Support Operations (ASO) sample identification (ID).

The ASO was responsible for the preparation and analysis of appropriate analytical batch and instrument quality control samples and for providing any additional processing to the sub-samples that might be required (e.g., acid digestion, radiochemical separations, dilutions). All analyses were conducted by the ASO according to their standard operating procedures, the ASO QA Plan, and the Analytical Service Request (ASR). Samples were analyzed directly (no preparation) by GEA; longer count times were used to assess isotopes other than ^{137}Cs.

Table 3.5. Analytical Scope Supporting Column Processing, ASR 1672

Tank Waste Matrix	Sample ID	ASO Sample ID	Analysis Scope
5.5 M Na AP-105 tank waste	TI137-Comp-FEED	23-0325	ICP-OES (Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr)
5.5 M Na AP-105 tank waste (Cs decontaminated)	TI137-Comp-EFF	23-0326	ICP-OES (Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr)
7 M Na AP-105 tank waste	TI138-Comp-FEED	23-0327	GEA (^{137}Cs, ^{60}Co, ^{154}Eu) ICP-OES (Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr) Radioanalytical (^{90}Sr, ^{99}Tc, ^{237}Np, ^{238}Pu, $^{239+240}$Pu, ^{241}Am)
7 M Na AP-105 tank waste (Cs decontaminated)	TI138-Comp-EFF	23-0328	GEA (^{60}Co, ^{137}Cs, ^{154}Eu) IC anions (F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, $C_2O_4^{2-}$, SO_4^{2-}) Hot persulfate (TIC, TOC) Acid titration (free OH) ICP-OES (Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr) ICP-MS (Ba, Nb, Pb, Sr, ^{238}U) Radioanalytical (^{90}Sr, ^{99}Tc, ^{237}Np, ^{238}Pu, $^{239+240}$Pu, ^{241}Am)

ICP-OES = inductively coupled plasma optical emission spectroscopy
ICP-MS = inductively coupled plasma mass spectrometry
IC = ion chromatography
TIC = total inorganic carbon
TOC = total organic carbon

4.0 Results

This section discusses the Cs exchange behavior during batch contact and column testing with 5.5 and 7 M Na AP-105 tank waste. Batch contact raw data are provided in Appendix D. Ion exchange process raw data are provided in Appendix A.

4.1 Batch Contact Results

This section provides the K_d and isotherm curves for 7 M Na AP-105 tank waste at the four process test temperatures, and a comparison of AP-105 tank waste with AP-107 and AP-101 temperature-dependent isotherm results. Input data supporting the various isotherms and figures are provided in Appendix D.

4.1.1 K_d and Isotherm Results for 7 M Na AP-105

Figure 4.1 shows the K_d dependence on Cs concentration at 13, 16, 25, and 35 °C. The K_d increased with decreasing temperature, consistent with AP-107 and AP-101 tank waste batch contact testing (Fiskum et al. 2021a). There is negligible change in the K_d for the three lower Cs concentrations (1.2E-4, 3.3E-4, and 8.5E-4 M) measured. This behavior has been observed for AP-107 and AP-101 and suggests the K_d is unimpacted with small changes in Cs concentrations of <10^{-3} M Cs.

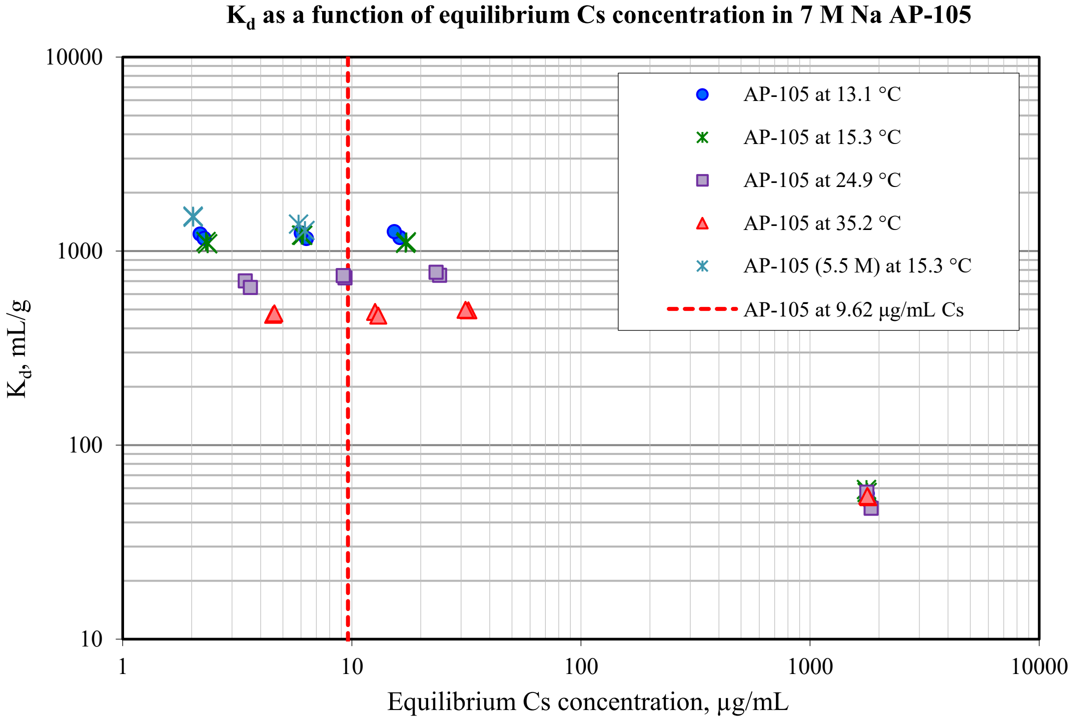

Figure 4.1. Cs K_d vs. Cs Concentration, AP-105 Tank Waste, Four Temperatures

Figure 4.2 shows the corresponding isotherms and Q (mmoles Cs/g dry CST) values vs. Cs molarity at all four test temperatures with 7 M Na AP-105 tank waste. It is important to note that the α_i, or total capacity in the matrix, was set to 0.68 mmoles Cs/g CST for this evolution of testing. Also provided are the curve fits to the Freundlich/Langmuir hybrid equilibrium model as given in Eq. (4.1) (Hamm et al. 2002).

$$Q = \frac{\alpha_I \times [Cs]}{(\beta + [Cs])} \tag{4.1}$$

where

$[Cs]$ = equilibrium Cs concentration, mmoles/mL or M

Q = equilibrium Cs loading on the CST, mmole Cs per g CST

α_i = isotherm parameter constant (mmoles/g), equivalent to total capacity in the matrix

β = isotherm parameter constant (mmoles/mL or M), selectivity coefficient, dependent on matrix and temperature; the larger the value, the less selective the CST is for Cs (Hamm et al. 2002)

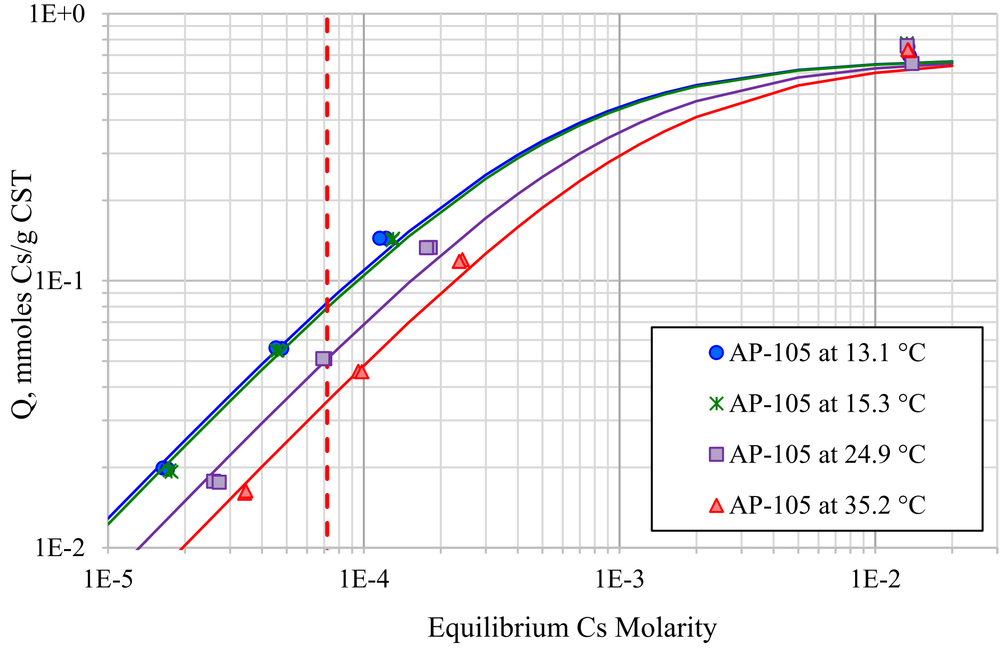

Figure 4.2. Q vs. Cs Equilibrium Concentration, 7 M Na AP-105 Tank Waste with Freundlich/Langmuir Hybrid Equilibrium Fits, Four Temperatures. The dashed red line represents the Cs concentration (7.16E-5 M) in AP-105 feed at 7 M Na.

The K_d and Q for the 13 and 16 °C results are nearly indistinguishable, hence the overlap of the Freundlich/Langmuir hybrid equilibrium model fit. A plot of Q (mmoles Cs/g CST) vs. temperature (Figure 4.3) indicates that the loading decreases linearly as temperature increases. This is consistent with the data collected for both AP-107 and AP-101 tank waste (Fiskum et al. 2021a). In fact, the slope of -0.0022 matches that of simple simulant (1 M NaOH/4.6 M $NaNO_3$) and AP-101 tank waste identically and is in excellent agreement with the slope obtained for AP-107 waste at -0.0025 (Fiskum et al. 2021a).

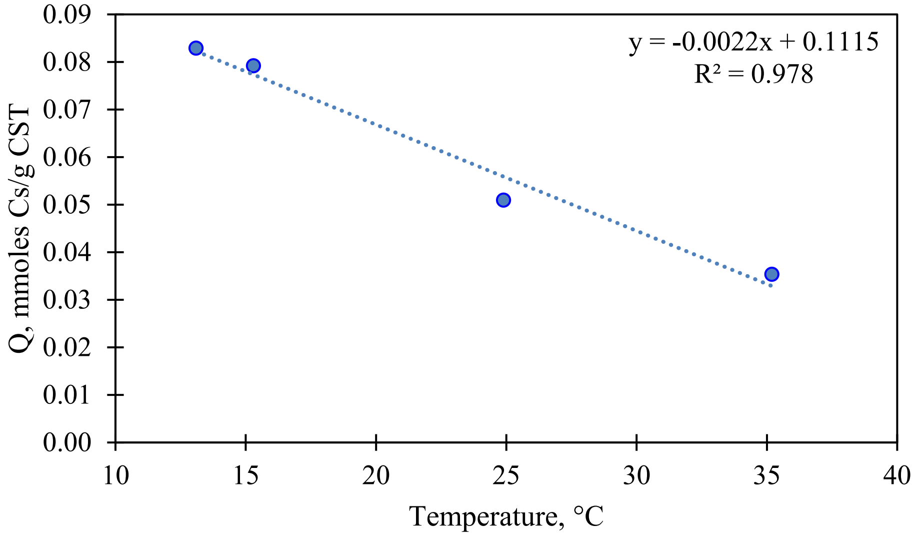

Figure 4.3. Q Dependence on Temperature for 7 M Na AP-105 Tank Waste

The experimental data for Cs loading (Q) at the three lower Cs concentrations (1.2E-4, 3.3E-4, and 8.5E-4 M) bracketing Cs near the feed condition are better represented by a linear Freundlich isotherm fit as shown in Figure 4.4. A comparison of the loading calculated using the Freundlich/Langmuir hybrid model and the linear Freundlich approach is shown in Table 4.1. The loadings predicted by both isotherms are in excellent agreement at the AP-105 feed condition of 7.16 E-5 M Cs; however, the Freundlich/Langmuir hybrid model overpredicts the loading at the lowest Cs concentration and underpredicts Cs loading at 8.8E-4 M Cs.

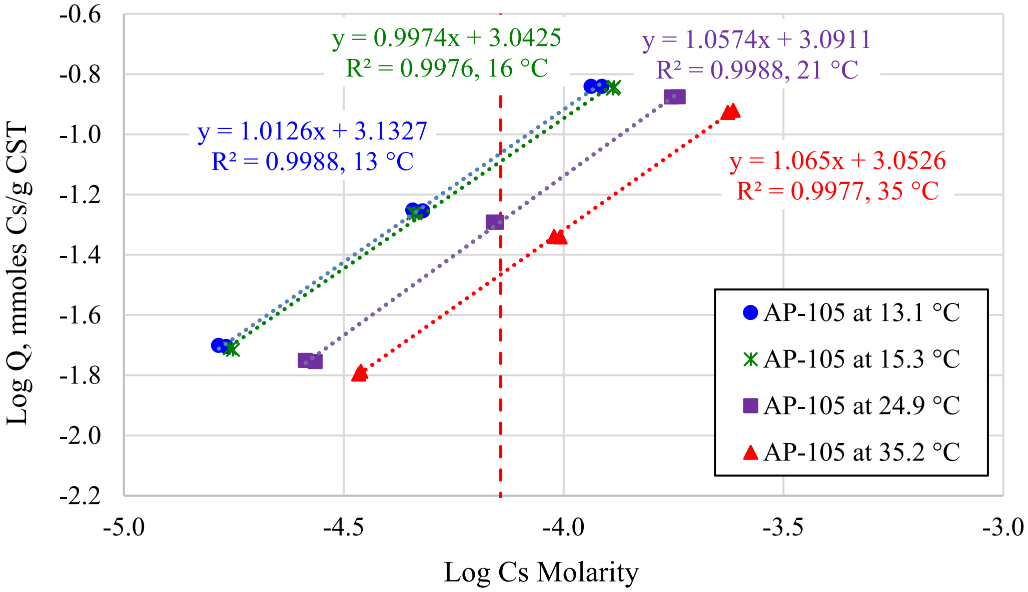

Figure 4.4. Linear Fits for Log Q vs. Log [Cs] at Four Test Temperatures

Table 4.1. Cs loading (Q, mmoles Cs/g CST) for the Freundlich/Langmuir Hybrid and Linear Freundlich Isotherm Model at 7 M Na AP-105 Feed Condition of 7.16E-5 M Cs

Process Temperature (°C)	Q (mmoles Cs/g) F/L Hybrid model	Q (mmoles Cs/g) Linear Freundlich model
13.1	0.083	0.087
15.3	0.079	0.081
24.9	0.051	0.051
35.2	0.035	0.034

4.1.2 Tank Waste Comparisons

The alpha parameter in the Freundlich/Langmuir hybrid model represents the maximum Cs loading that can be achieved under the corresponding matrix conditions. To compare the data across tank wastes, α_i (maximum Cs loading) was set to 0.68 mmoles Cs/g CST and Excel Solver was used to calculate the β parameters using a generalized reduced gradient nonlinear method. The calculated β parameters for AP-107, AP-101, AP-105, and AP-105 (7 M Na) are shown in Table 4.2. Note that AP-105 is unique in Na concentration of 7 M, whereas the Na concentration of the other wastes is nominally 5.6 M Na. The β values, or selectivity coefficient, can be used to compare Cs selectivity in the different tank waste matrices. The β values linearly increased with temperature, which is expected as increasing temperature inhibits Cs loading. The smaller the β value, the more favorable the exchange. The β values for AP-101 were the smallest of the waste series measured, which coincides with the ion exchange performance.

Table 4.2. Freundlich/Langmuir Hybrid Equilibrium β Parameter Summary for AP-107, AP-105, AP-101, and AP-105 (7 M Na) Tank Waste and Calculated Q and K_d

Matrix	Process Temperature (°C)	β, (Cs M)	K_d (mL/g)	Q (mmoles Cs/g CST)
AP-107 Tank Waste 6.91×10^{-5} M Cs	12.7	3.49E-04	1497	0.103
	15.9	4.43E-04	1249	0.086
	21.0	5.03E-04	1138	0.079
	34.5	9.74E-04	681	0.047
AP-101 Tank Waste 4.64×10^{-5} M Cs	12.9	3.85E-04	1721	0.0799
	15.7	4.76E-04	1391	0.0645
	21.7	5.28E-04	1237	0.0574
	34.3	9.29E-04	666	0.0309
AP-105 Tank Waste 5.65×10^{-5} M Cs	12.7	4.91E-04	1242	0.070
	15.9	6.11E-04	1019	0.058
	21.0	6.54E-04	956	0.054
	34.5	1.28E-03	510	0.029
AP-105 Tank Waste (7 M Na) 7.16×10^{-5} M Cs	13.1	5.18E-04	1153	0.0829
	15.3	5.46E-04	1101	0.0792
	24.9	8.88E-04	708	0.0509
	35.2	1.31E-03	491	0.0353

Figure 4.5 compares the K_d values vs. temperature for AP-107, AP-105 (5.5 and 7 M Na), and AP-101 tank waste. The K_d values from AP-105 FY20 and AP-105 7 M Na (this work) are nominally the same. However, the repeat 5.5 M Na AP-105 sample conducted in parallel with the 7 M Na AP-105 batch contacts shows the K_d increased from AP-105 FY20. The K_d values are calculated from the

Freundlich/Langmuir fit of the experimental data from the waste series with α_i = 0.68 mmoles Cs/g CST. This does use the initial Cs concentration from the tank waste supernate, which followed the order AP-101<AP-105 (FY20) < AP-105 (FY23) < AP-107, where AP-101 had the lowest Cs concentration; thus, AP-101 also had the highest K_d. A comparison of the K_d from 5.5 M Na AP-105 FY20 and FY23 is shown in Figure 4.5 and the FY23 data are greater than that of the older AP-105 and align much better with the hypothesized trend.

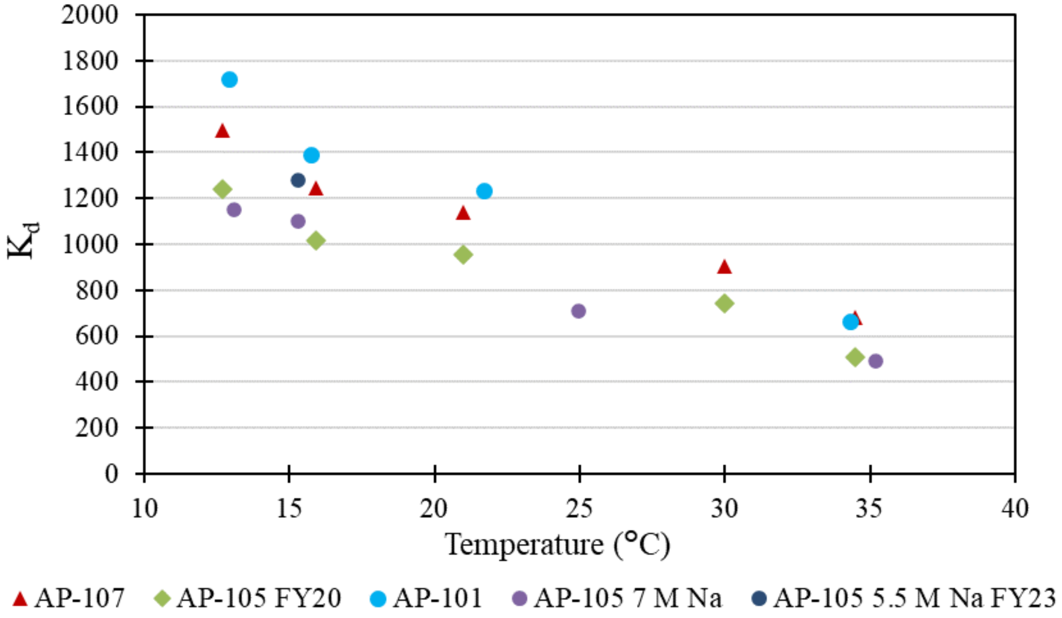

Figure 4.5. Cs K_d vs. Temperature for AP-107, AP-105 (5.5 and 7 M Na), and AP-101 Tank Waste at Corresponding Feed Conditions

Figure 4.6 compares the Q loading vs. temperature at Cs feed concentrations for AP-105 (7 M Na) to previous batch contact results from AP-107, AP-101, and AP-105. Q is related to the initial Cs concentration, and while the trend across temperatures is the same for all wastes analyzed, AP-107 had the highest initial Cs concentration (6.91×10^{-5}) and thus the largest Cs loading (Q). Interestingly, AP-101, with the lowest initial Cs concentration, was not the lowest Cs loading; instead, 5.5 M Na AP-105 (FY20) had the lowest Q. Also of note, the Cs loading for 7 M Na AP-105 was indistinguishable from the 5.5 M Na AP-105.

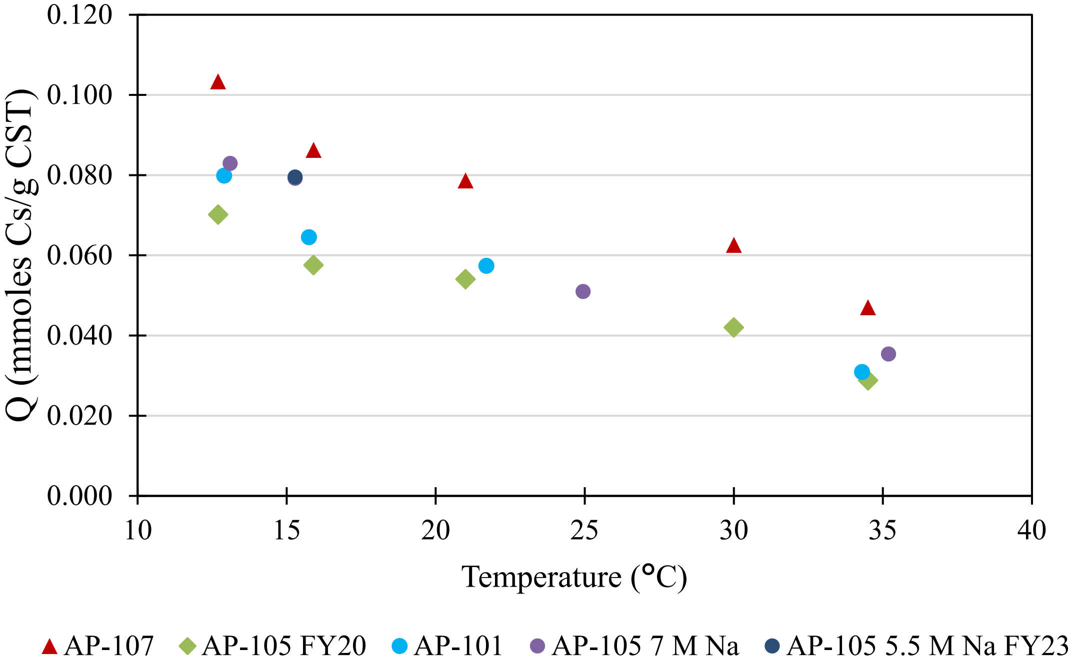

Figure 4.6. Q vs. Temperature for AP-107, AP-105 (5.5 and 7 M Na), and AP-101 Tank Waste at Corresponding Feed Conditions

4.2 Ion Exchange Processing

The Cs load behavior for AP-105 tank waste was evaluated at 5.5 and 7 M Na at 16° C. This section discusses the Cs load behavior for the two tests.

4.2.1 Cs Load Results

The 5.5 M Na AP-105 testing at 16 °C was considered to be the standard comparative reference point for evaluating the effects of increasing Na molarity on TSCR operations. The feed was processed at nominally 1.96 BV/h through the lead and lag columns for 625 BVs, at which time all the available 5.5 M Na feed had been treated prior to WAC breakthrough on the lag, so no installation of a polish column was conducted. Figure 4.7a shows a linear-linear plot of the cesium load profile for feed processed through each column. The x-axis shows the BVs processed and the y-axis shows the effluent Cs concentration (C) relative to the feed concentration (C_0) in terms of % C/C_0. The 5.5 M Na AP-105 C_0 value for ^{137}Cs was determined to be 109.2 µCi/mL (average of the four 5.5 M Na filter product bottle feeds). In this graphing layout, the Cs breakthrough from the lead column appeared to start at ~210 BVs and continued to 20% C/C_0 after processing 625 BVs when the last sample was collected from the lead column. The lag column Cs breakthrough performance is not discernable at this linear scale.

Figure 4.7b shows the same Cs load data provided in Figure 4.7a but with the ordinate % C/C_0 on a probability scale and the abscissa BVs processed on a log scale. Under normal load processing conditions, these scales provide a predictable straight-line Cs breakthrough curve and provide greater fidelity of load characteristics at low and high % C/C_0 values (Buckingham 1967). In contrast to Figure 4.7a, the Cs breakthrough from the lead column was observed to occur at around 100 BVs and breakthrough from the lag column started just before processing of 300 BVs. In addition to the 50% C/C_0 indication line, the

WAC limit, set at 0.162% C/C$_0$, is also apparent (dashed green line).[12] The WAC Cs breakthrough for the lead column occurred at 172 BVs. There was not enough volume of 5.5 M Na AP-105 to reach the WAC on the lag column, but this point can be extrapolated from the given breakthrough data and calculated to be 679 BVs.

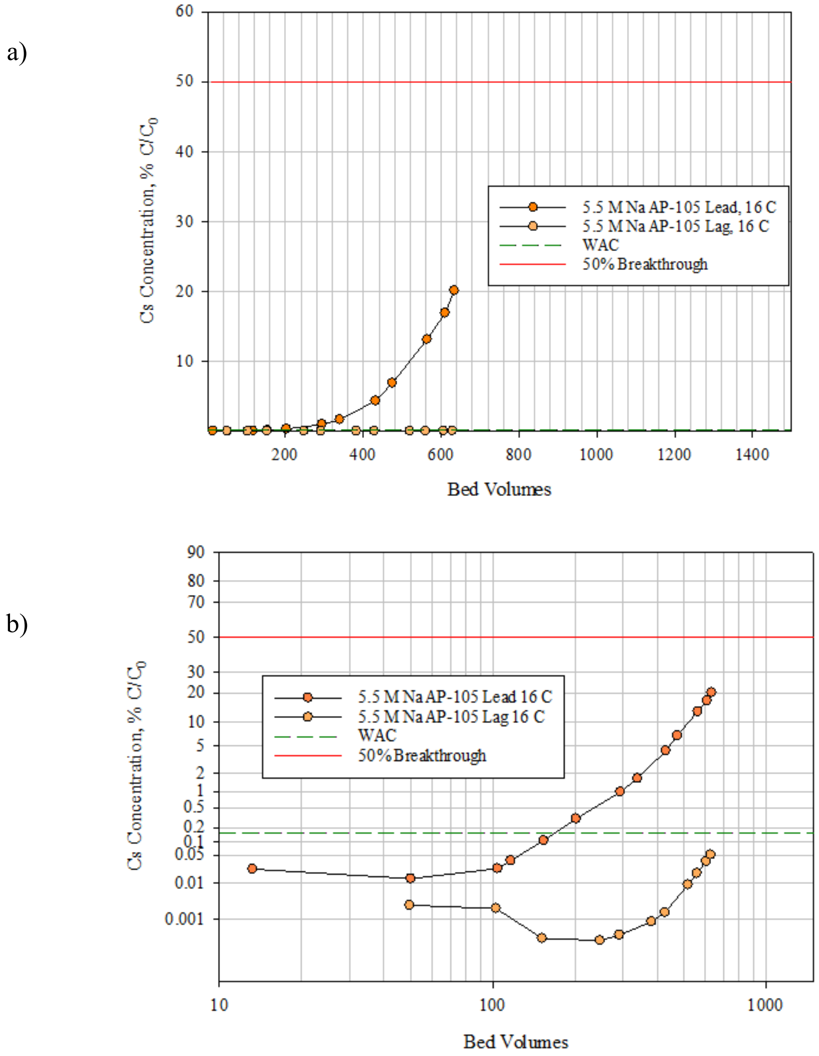

Figure 4.7. Lead and Lag Column Cs Load Profiles of 5.5 M Na AP-105 at 1.90 BV/h: a) Linear-Linear Plot; b) Probability-Log Plot

The 7 M Na AP-105 was processed at nominally 1.92 BV/h through the lead and lag columns for 470 BVs, at which time the lag column effluent approached the WAC limit. The polish column was then placed into position and processing continued for another 332 BVs. Figure 4.8 shows the Cs breakthrough profiles for the 7.0 M Na AP-105 columns using both scale presentations. The 7.0 M Na AP-105 C$_0$ value for ^{137}Cs was determined to be 126.5 µCi/mL (average of the seven 7.0 M Na filter product bottle feeds).

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[12] The WAC 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: 3.18×10^{-5} Ci ^{137}Cs/mole Na. At 5.58 M Na and 109 µCi ^{137}Cs/mL in the feed, the WAC limit translates to 0.162% C/C$_0$.

The Cs breakthrough from the lead column was observed to start at ~50 BVs and continued to 50% C/C_0 after processing 784 BVs when the last sample was collected from the lead column. Similarly, the lag column Cs breakthrough appeared to start at ~240 BVs and increased to 4% breakthrough when the last sample was collected from the column. Breakthrough on the polish column appeared after ~650 total BVs (186 net BVs) processed through the system. In addition to the 50% C/C_0 indication line, the WAC limit, set at 0.181% C/C_0, is also apparent (dashed green line).[13]

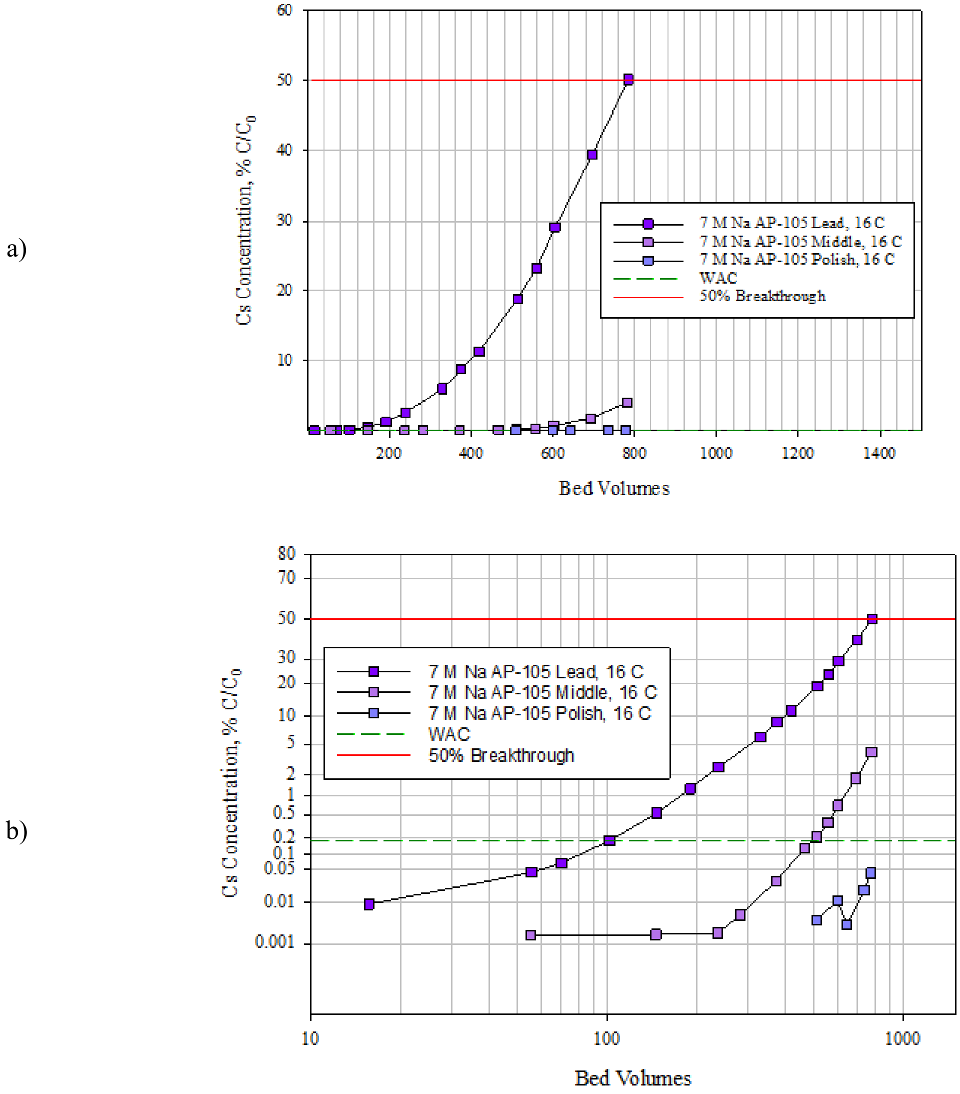

Figure 4.8. Lead, Lag, and Polish Column Cs Load Profiles of 7.0 M Na AP-105 at 1.90 BV/h: a) Linear-Linear Plot; b) Probability-Log Plot

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[13] The WAC 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 7.18 M Na and 126 µCi ^{137}Cs/mL in the feed, the WAC limit is 0.181% C/C_0.

The Cs breakthrough curves were modeled by the error function (erf) (Hougen and Marshall 1947; Klinkenberg 1948), as shown in Eq. (4.1):

$$\frac{C}{C_0} = \frac{1}{2}\left(1 + \mathrm{erf}\left(\sqrt{k_1 t} - \sqrt{k_2 z}\right)\right) \tag{4.1}$$

where:

k_1 and k_2 = parameters dependent on column conditions and ion exchange media performance
t = time (or BVs processed)
z = column length

Using this model, fits were generated to the 5.5 M Na (see Figure 4.9) and 7.0 M Na (see Figure 4.10) lead and lag column experimental data.

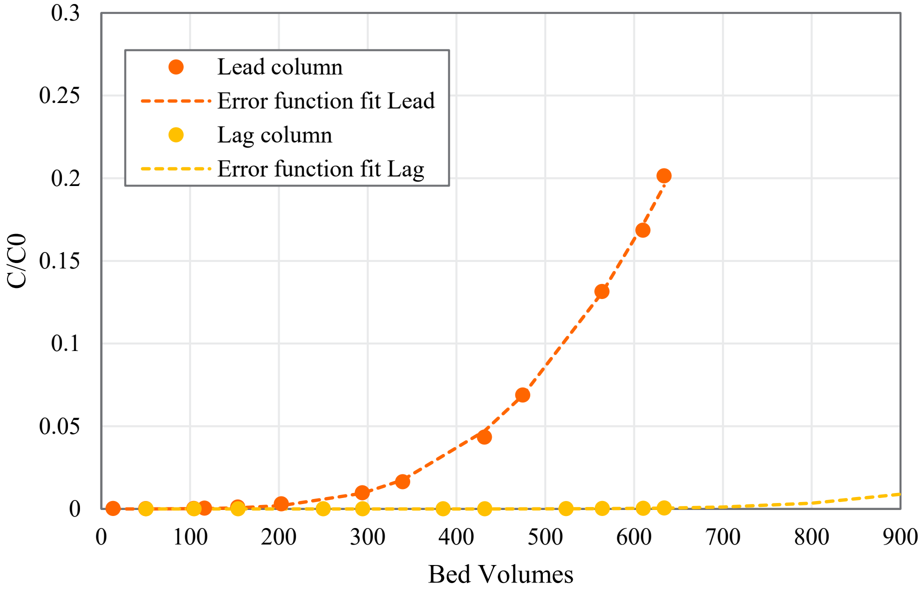


Figure 4.9. 5.5 M Na AP-105 Lead and Lag Column Cs Breakthrough with Error Function Fit

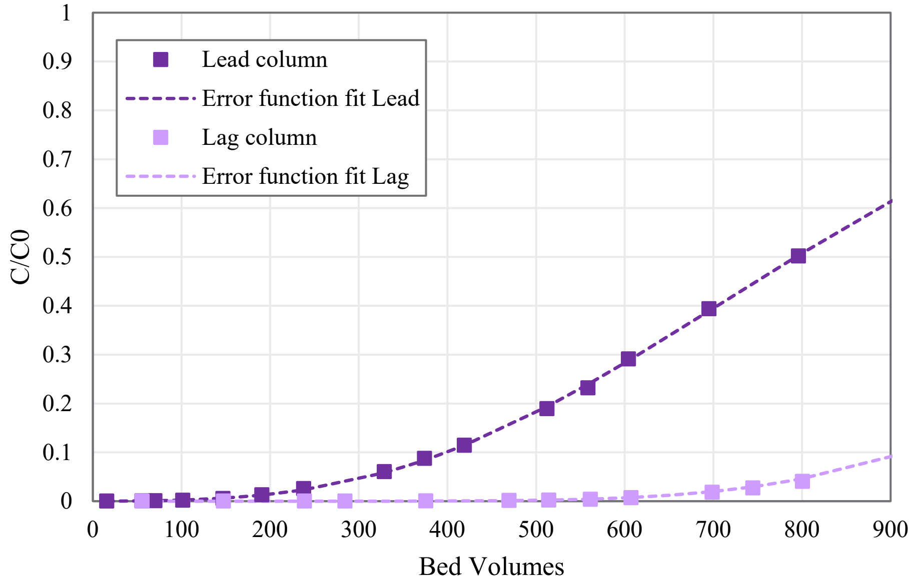


Figure 4.10. 7.0 M Na AP-105 Lead and Lag Column Cs Breakthrough with Error Function Fit

The 50% Cs breakthroughs for the 5.5 M Na AP-105 lead and lag columns were estimated from the error function fit at 888 and 1796 BVs, respectively, and the 7.0 M Na AP-105 lead and lag columns were estimated to be 791 and 1332 BVs, respectively. The reduced capacity observed during the 7.0 M Na AP-105 testing is consistent with batch contact results and is a direct result of increased sodium molarity in the feed.

The theoretical 50% Cs breakthrough on the ion exchange column (λ) can be predicted from the product of the K_d value and the ion exchanger bed density (ρ_b) according to Eq. (4.2) (Bray et al. 1993). The CST bed density is the dry CST mass divided by the volume in the column:

$$K_d \times \rho_b = \lambda \tag{4.2}$$

The lead column 50% Cs breakthrough values for both the 5.5 and 7 M Na feeds were over 25% lower than the Cs λ value predicted from the 15.3 °C batch contact studies (1280 and 1101 BVs, respectively). The increased value for the batch contact could be attributed to a lower processing temperature or competitive ion exchange species that are removed during column testing and are no longer present in the matrix for batch contacts.

The WAC limit Cs breakthroughs were interpolated for each column by curve-fitting the BVs processed as a function of the log % C/C_0 values (see Figure 4.11 and Figure 4.12). The curves were fitted to a second-order polynomial function ($R^2 \geq 0.99$) and the WAC limit breakthroughs were then calculated, resulting in the following:

- Lead column: 5.5 M Na, 172 BVs 7 M Na, 103 BVs
- Lag column: 5.5 M Na, 679 BVs 7 M Na, 496 BVs
- Polish column*: 7 M Na, 885 BVs (*=largely extrapolated)

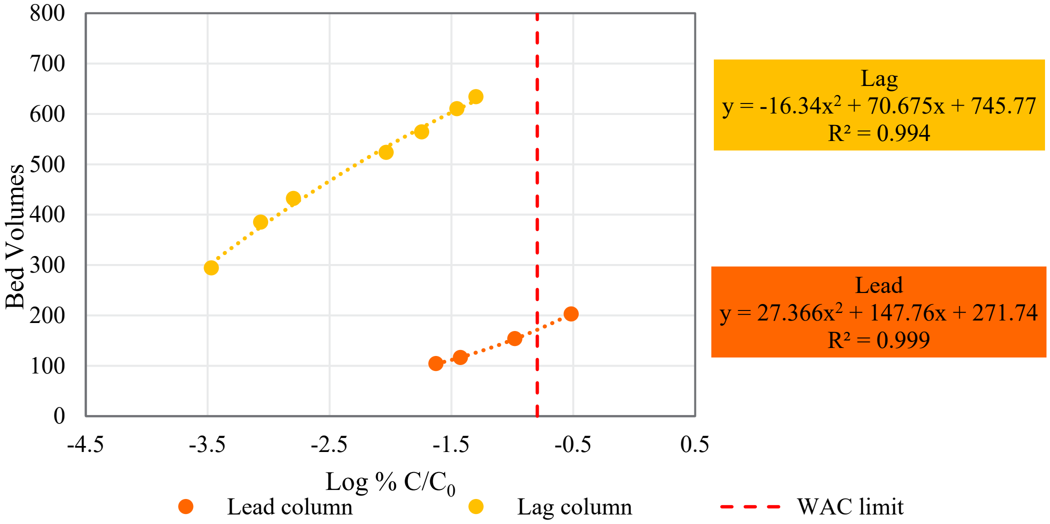


Figure 4.11. Curve Fits to Interpolate WAC Limit Breakthroughs from 5.5 M Na AP-105 Lead and Lag Columns

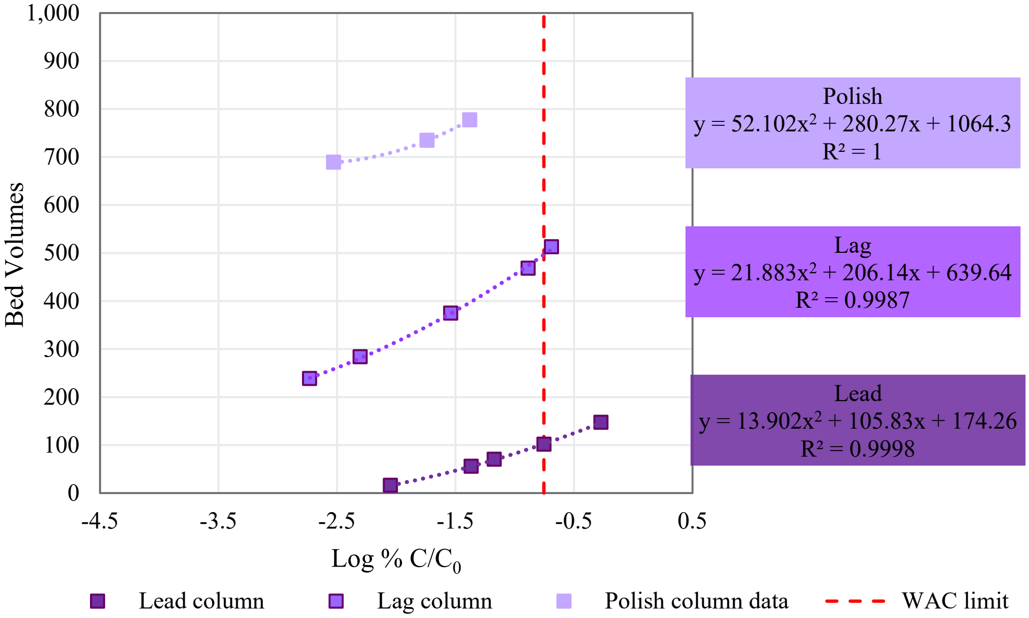


Figure 4.12. Curve Fits to Interpolate WAC Limit Breakthroughs from 7.0 M Na AP-105 Lead, Lag, and Polish Columns

4.2.2 Performance Comparison

Figure 4.13 provides the 5.5 and 7.0 M Na AP-105 column load profiles on one graph for direct comparison. The Cs exchange associated with the higher 7.0 M Na concentration resulted in earlier Cs breakthroughs and a slightly longer mass transfer zone. Decreasing the Na molarity proportionately decreases the Cs concentration and increases the loading onto the CST. Normalizing for the change in Na throughput (and subsequent curies of Cs) demonstrates the impact that solely changing the Na molarity has on CST usage under consistent temperature, cesium content, and competitive analyte concentrations. Figure 4.14 shows the normalized loading for the two conditions on a probability-log plot. As expected, the 5.5 M Na AP-105 test provides slightly (10-15%) better performance than the 7 M Na test. This primary impact appears to be due to the change in capacity as increasing the Na molarity has a distinct negative effect on Cs exchange kinetics, Cs load capacity, and Cs mass transfer zones.

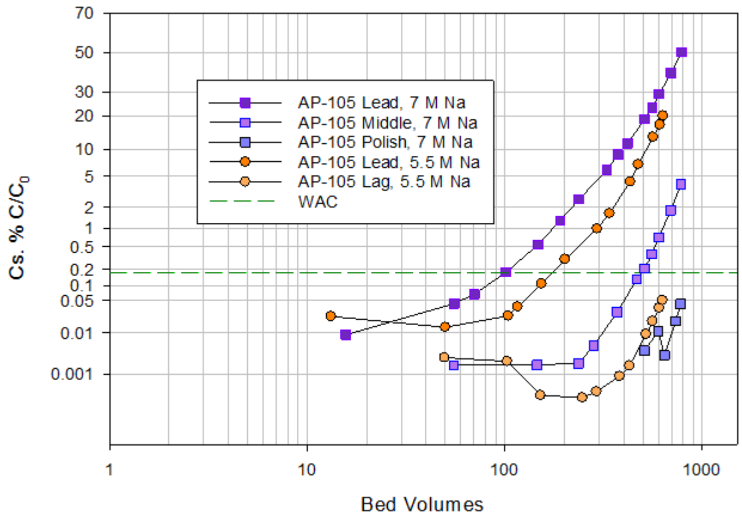


Figure 4.13. Comparative Cs Breakthrough Performance for 5.5 and 7 M Na AP-105, Probability-Log Plot

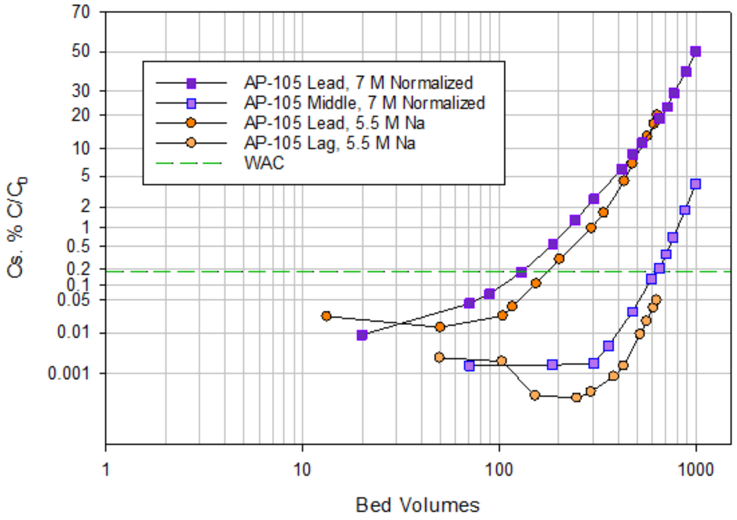


Figure 4.14. Normalized Comparative Cs Breakthrough Performance for 5.5 and 7 M Na AP-105, Probability-Log Plot

Table 4.3 summarizes the ^{137}Cs fractionation between the effluent, samples, and CST for each column test. The Cs fractionations to the effluents and the columns were determined based on the input ^{137}Cs and the measured ^{137}Cs in the various effluent streams. The quantities of Cs loaded onto the lead, lag, and polish columns were determined by subtracting the Cs recovered in the samples and effluents from the Cs fed to each column.

Table 4.3. ^{137}Cs Activity (Cs) Fractionation in the 5.5 and 7 M Na AP-105 Column Tests

AP-105 Test ID	Units	Input Feed	Effluent	Samples	Feed Disp. and Water Rinse	Lead CST Loading	Middle/Lag CST Loading	Polish CST Loading
5.5 M Na	µCi ^{137}Cs	4.18E+05	26.5	149	91.2	4.06E+05	1.17E+04	NA
	%	100	0.01	0.04	0.02	97.2	2.8	--
7 M Na	µCi ^{137}Cs	8.99E+05	133.4	620	145	7.54E+05	1.39E+05	5.12E+03
	%	100	0.01	7	0.02	83.9	15.4	0.57

The total Cs loaded per g CST (effective capacity) was calculated from the total ^{137}Cs loaded onto the column, at 50% Cs breakthrough, and the dry CST mass was loaded into the lead column. It is noted that Cs loading for the 5.5 M Na AP-105 only reached 20.1% C/C_0 Cs breakthrough, so the extrapolated 50% breakthrough value [Eq. (4.2)] was used to allow for gross comparisons in the Cs loading behavior between tests. The Cs mass loaded onto the CST bed was calculated according to Eq. (4.3):

$$\frac{A_{Cs} \times CF}{M} = C \tag{4.3}$$

where

A_{Cs} = activity of ^{137}Cs, µCi on the lead column
CF = conversion factor, mg Cs/µCi ^{137}Cs
M = mass of dry CST (6.0 or 9.0 g)
C = capacity, mg Cs/g CST

Table 4.4 summarizes the CST Cs loading performance for both column tests. Batch contact testing resulted in a measured Cs loading of 0.0791 mmoles Cs/g CST at 8.31 µg/mL Cs equilibrium condition and 0.0792 mmoles Cs/g CST at 9.62 µg/mL Cs equilibrium condition. In agreement with the compared K_d values, the column loading values for both the 5.5 and 7 M Na feeds were ~30% lower than the Cs loading values predicted from the 15.3 °C batch contact studies and may be attributed to a lower processing temperature or competitive ion exchange species that are removed during column testing and are no longer present in the matrix for batch contacts.

Table 4.4. CST Cs Load Performance Summary in the 5.5 and 7.0 M Na AP-105 Column Tests

Column Test	BV Processed	Flowrate (BV/h)	Initial Cs Concentration (µg Cs/mL)	Cs Loading (mg Cs/g CST)$^{(a)}$	Cs Loading (mmoles Cs/g CST)
5.5 M Na	625	1.96	8.31	7.38	0.0551
7.0 M Na	796	1.92	9.58	7.58	0.0566

(a) This value is predicted based on an extrapolated 50% breakthrough value

Figure 4.15 compares the 5.5 M Na AP-105 lead column Cs load profile at 16 °C with lead column breakthrough data from AP-105 from FY20 testing conducted at 28 °C (Fiskum et al. 2021b), AP-107 from FY21 testing conducted at 16 °C (Westesen et al. 2021b), and AP-101 from FY22 testing conducted at 16 °C (Westesen et al. 2022). CST lot 2002009604 sieved to <30-mesh was used in all tests. AP-105 processing at 16 and 28 °C and AP-107 at 16 °C all reached the WAC limit at nominally 190 BVs, while AP-101 at 16 °C reached the WAC at 275 BVs. The increased loading for AP-101 can be explained by the notably lower Na and Cs concentrations compared to the AP-105 and AP-107 wastes (see Table 4.5). The kinetics for all the 16 °C tests appear consistent, based on similar shapes in the load curves. Despite the similar BVs processed before reaching the WAC, the kinetics of the 28 °C AP-105 test are markedly faster as signified by the steeper breakthrough curve.

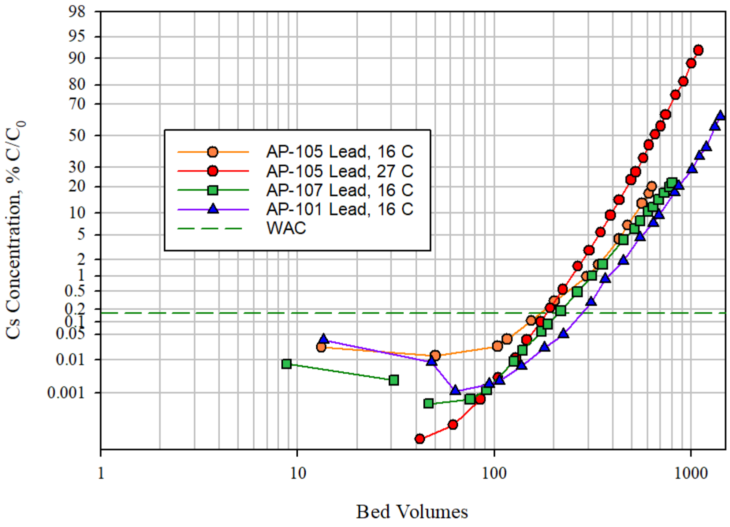


Figure 4.15. Load Profile Comparisons: AP-105 at 16 and 27 °C, AP-107 and AP-101 at 16 °C, CST Lot 2002009604

Table 4.5. AP-105 at 16 and 27 °C, AP-107 and AP-101 at 16 °C Testing Parameters

	AP-105 (Current)	AP-105 (FY20)	AP-107 (FY21)	AP-101 (FY22)
Configuration	Lead-Lag	Lead-Lag-Polish	Lead-Lag	Lead-Lag-Polish
Flowrate, BV/h	1.95	1.83	1.92	1.89
Process Temp. °C	16.0	27.0	16.0	16.0
Cs, M	6.21-05	5.66E-05	6.91E-05	4.64E-05
Na, M	5.5	6.1	5.8	5.2

4.2.3 Predicted TSCR Performance

Westesen et al. (2020) demonstrated that the impact of residence time (flowrate through the CST column in terms of BV/h or contact time) directly influenced the volume that can be processed before reaching the WAC limit or 50% breakthrough. An evaluation of 1-, 2-, and 3-column systems can be determined collectively in terms of SVs. The SV/h in the lead column was, by definition, equivalent to the BV/h flowrate. The combined lead-lag column system, with two sequential CST beds, corresponded to half this throughput. The 3-column system, used only in the 7 M Na AP-105 testing, corresponded to a third of this throughput. The AP-105 SVs, adjusted flowrate, and SVs to WAC limit are provided in Table 4.6. Using the SVs to WAC as a fraction of the 50% breakthrough, these data are then evaluated in terms of the square root of the fraction of the CST capacity used and the square root of SV/h to develop a linear relationship to project the volume of waste that can be processed through the TSCR facility before reaching the WAC limit. Figure 4.16 plots these data alongside data from 5.5 M Na AP-107 (Westesen et al. 2021b), 5.5 M Na AP-105 at 25 °C (Fiskum et al. 2021b), which was processed in a lead-lag-polish configuration, and two full-height column tests (Fiskum et al. 2019b) using tank waste simulant processed in a lead-lag configuration at 25 °C. Using these relationships, the volume projection for AP-105 tank waste processed under various temperature or Na molarities before WAC Cs breakthrough can be evaluated and are shown in Table 4.7.

The impact of kinetics on Cs exchange can be further evaluated by comparing the slopes of each data set. The slope modeled in Figure 4.16 is inversely proportional to the mass transfer coefficient and accentuates the impact on kinetics with varying temperature and Na molarity. The testing at 7 M Na and 16 °C shows a steeper slope compared to the room temperature AP-105 and simulant tests. This is due to the slower kinetics of the exchange as a result of the decreased temperature and increased viscosity. As expected, the 5.5 M Na test at 25 °C has the lowest slope of the three. This is expected as the mass transfer kinetics should increase with increasing temperature and a lower solution viscosity.

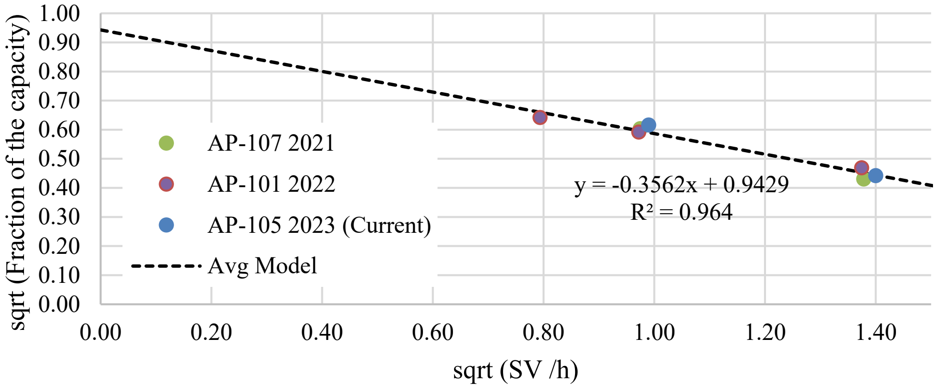
Table 4.6. Bed Volumes Processed to Reach WAC Limit for Cesium

AP-105 Systems	SV (mL)	Flowrate (SV/h)	SVs to WAC Limit
5.5 M Na lead column	6	1.96	173
5.5 M Na lead-lag columns	12	0.98	337
7 M Na lead column	9	1.92	103
7 M Na lead-lag columns	18	0.96	248
7 M Na lead-lag-polish columns	27	0.64	295[(a)]

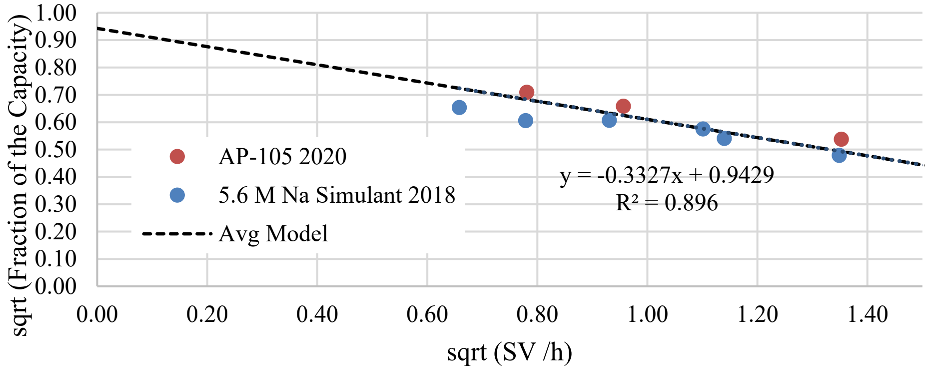
(a) The polish column was only in position during second half of processing interval from 470 to 802 BVs and did not reach the WAC limit. An extrapolated value is used here but may not be truly representative of the 27-mL CST bed (3-column system) configuration.

Table 4.7. AP-105 TSCR Processing Projection

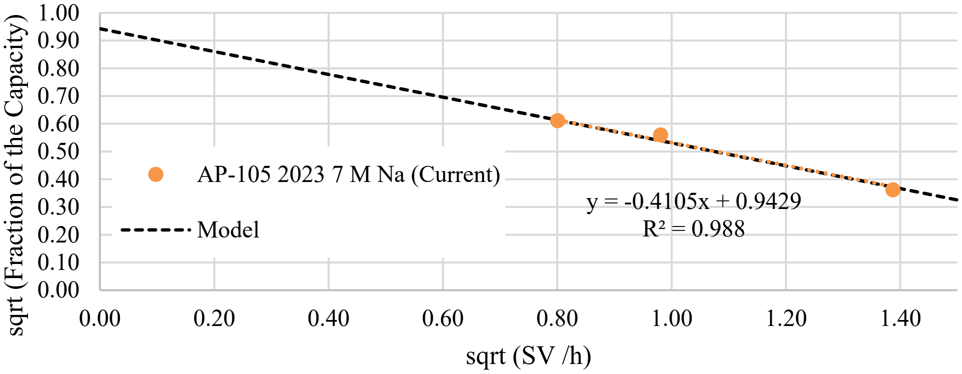
AP-105 Processing Condition	# of Columns	BVs through TSCR before WAC	Gallons through TSCR before WAC
5.5 M Na at 16 °C	1	180	84,900
	2	471	148,000
	3	1156	181,400
5.5 M Na at 25 °C	1	151	71,100
	2	370	116,300
	3	891	139,800
7 M Na at 16 °C	1	110	52,000
	2	346	108,800
	3	895	140,600



a) 5.5 M Na tank waste testing (AP-107, AP-101, and FY23 AP-105) at 16 °C



b) 5.5 M Na AP-105 and 5.6 M Na Simulant testing at 25 °C



c) FY23 7 M Na AP-105 testing at 16 °C

Figure 4.16. Breakthrough Results for AP-101, AP-107, AP-105, and 5.6 M Na Simulant Testing at Various Temperatures and Na Molarities

4.2.4 Metals and Radionuclide Analysis

The 5.5 and 7 M Na AP-105 composite feed and composite effluent samples underwent extensive characterization to better define waste characteristics and assess analyte fractionation to the CST.

Table 4.9 summarizes the feed and effluent metals concentrations and fractionations to the effluent. The anions, free hydroxide, inorganic carbon, and organic carbon concentrations in the effluent are provided in Table 4.10; they were not measured in the feed because it was shown that their concentrations were not affected by the CST processing (Westesen et al. 2021a). Further, bench handling of the effluent was safer for the analysts from a radiological dose perspective. Analytical reports along with result uncertainties and quality control discussions are provided in Appendix C.

By inference, the analytes present in the feed and not found in the effluent were assumed to be retained on the CST. Analyte fractionation was calculated as the ratio of the total analyte measured in the feed processed through the columns and the total analyte collected in the Cs-decontaminated effluent according to Eq. (4.4):

$$\frac{C_{Da} \times V_D}{C_{Fa} \times V_F} = F_{Da} \tag{4.4}$$

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-105 feed
V_F = volume of AP-105 feed
F_{Da} = fraction of analyte *a* in the Cs-decontaminated effluent

The analyte results shown in brackets indicate the result was less than the instrument estimated quantitation limit (EQL) but greater than or equal to the method detection limit (MDL); the associated analytical uncertainty could be higher than ±15%. The fractionation result was placed in brackets, where it was calculated with one or more bracketed analytical values to highlight the higher uncertainty. The opportunistic analyte results measured by ICP-OES are also shown in Table 4.9; these analytes are part of the ICP-OES data output but have not been fully evaluated for quality control performance.

Table 4.8. 7 M Na AP-105 Feed & Effluent Radionuclide Concentrations and Fractionations (ASR 1672)

Analysis Method	Analyte	Feed Conc. TI139-Comp-Feed (µCi/mL)	Effluent Conc. TI139-Comp-Eff (µCi/mL)	Fraction in Effluent (%)
Gamma energy analysis (GEA)[(a)]	^{60}Co	<8.3E-4	6.29E-02	--
	^{126}Sn	<8.6E+0	2.77E-02	--
	^{126}Sb	<6.0E-1	2.08E-02	--
	^{137}Cs	1.38E+02	2.76E+00	2%
	^{154}Eu	<7.1E-1	1.52E-02	--
Separations/ Alpha energy analysis (AEA)[(a)]	^{238}Pu	6.23E-06	5.21E-06	86%
	$^{239+240}$Pu	2.93E-05	2.15E-05	75%
	^{241}Am	1.81E-04	1.33E-04	75%
Separations/ Beta counting[(a)]	^{90}Sr	3.50E-01	1.69E-03	0.5%
	^{99}Tc	6.39E-02	5.31E-02	85%

(a) Reference date is April 2023.
"--" = not applicable; value not reported, or fractionation cannot be calculated with a less-than value.
The recovered fractions are calculated with values containing more significant figures than shown; using listed values may result in a slight difference due to rounding.

Table 4.9. 5.5 and 7 M Na AP-105 Feed and Effluent Inorganic Analyte Concentrations and Fractionation (ASR 1672)

ICP-OES	Analyte	Feed Concentration (M)	Effluent Concentration (M)	Fraction in Effluent
5.5 M Na AP-105	Al	5.05E-01	4.79E-01	94%
	Ba	3.69E-06	[2.9E-07]	8%
	Ca	1.29E-03	8.51E-04	65%
	Cd	[2.3E-05]	[9.9E-06]	43%
	Cr	6.21E-03	5.80E-03	92%
	Cu	1.13E-04	1.02E-04	88%
	K	9.59E-02	8.80E-02	91%
	Na	5.58E+00	5.33E+00	94%
	P	1.41E-02	1.15E-02	80%
	Pb	[1.9E-04]	[3.3E-05]	17%
	S	3.41E-02	3.24E-02	94%
7 M Na AP-105	Al	6.42E-01	6.09E-01	97%
	Ba	6.40E-06	[7.1E-07]	11%
	Ca	1.19E-03	9.30E-04	80%
	Cd	[4.1E-05]	1.72E-05	43%
	Cr	7.92E-03	7.52E-03	97%
	Cu	1.44E-04	1.26E-04	90%
	Fe	[8.9E-05]	[2.4E-05]	27%
	K	1.20E-01	1.17E-01	100%
	Na	7.18E+00	6.76E+00	97%
	P	1.70E-02	1.41E-02	85%
	Pb	[1.1E-04]	[5.0E-05]	47%
	S	4.36E-02	3.98E-02	94%
	Ti	[4.7E-05]	[3.3E-05]	72%
	U	[3.2E-04]	[4.9E-05]	16%
	Zn	[2.4E-05]	[1.1E-05]	46%
ICP-MS	**Analyte**	**Feed Concentration (µg/g)**	**Effluent Concentration (µg/g)**	**Fraction in Effluent**
7 M Na AP-105	Ba	1.90E-01	1.14E-01	60%
	Nb	2.24E-02	6.56E-01	>100%
	Pb	1.64E+01	4.15E+00	25%
	Sr	1.49E-01	<4.3E-02	--
	^{238}U	5.14E+00	4.00E+00	78%

Notes:
Values in brackets [ ] were ≥ MDL but < EQL, with errors likely to exceed 15%.
The recovered fractions are calculated with values containing more significant figures than shown; using listed values may result in a slight difference due to rounding.

Table 4.10. 7 M Na AP-105 Feed and Effluent Anions and Carbon Composition (ASR 1672)

Analysis Method	Analyte	Feed TI139-Comp-Feed (M)	Effluent TI139-Comp-Eff (M)
Titration	Free Hydroxide	NA	2.88
Ion Chromatography	F^-	NA	1.57E-03
	Cl^-	NA	9.90E-02
	NO_2^-	NA	9.74E-01
	NO_3^-	NA	1.88E+00
	PO_4^{3-}	NA	8.23E-03
	SO_4^{2-}	NA	2.46E-02
Hot persulfate oxidation*	Total organic C	NA	4.79E-01
	Total inorganic C[(a)]	NA	2.01E-01

(a) Assumed to be carbonate.
NA= not analyzed

In addition to Cs removal, the CST removed 99.5% of the ^{90}Sr with a ^{90}Sr decontamination factor of 202. Although over 99% of the ^{90}Sr was removed, this decontamination factor is significantly lower than previously observed with 5.5 M Na AP-105 at 25 °C (Fiskum et al. 2020). The reduced Sr decontamination may indicate a complexed version of Sr unable to be removed by the CST. About 20% of the Pu were also removed. About 25% of Am was calculated to be removed during processing; the chemistry involved in Am removal by CST is not known. Assuming the difference in total Am and Pu µCi content between the feed and effluent remained solely on the lead column CST (9 g), the CST would contain 41 nCi/g of transuranic (TRU) isotopes, which easily remains below the threshold 100 nCi/g defining TRU waste. Most of ^{99}Tc, 85% (likely present as anionic pertechnetate), was found in the effluent, showing semi-moderate Tc interaction with the CST.

The ICP-OES results for the feed composite and effluent composite showed that the majority of analytes remained in the effluent (see Table 4.9 and Appendix C for analytical reports). The Al, Cr, Cu, K, Na, and S (sulfate) partitioned exclusively to the effluent (>90% recovery) in both the 5.5 and 7 M Na testing. Analytes such as Ca, Fe, and Pb saw significantly more recovery in the 7 M Na AP-105 than in the 5.5 M Na AP-105.

Previous ICP-MS analysis from Fiskum et al. 2021b was used to determine the feed concentrations in the 7 M Na AP-105. Significant uptake of Pb (75%) was seen sorbed to the CST as well as fractions of the Ba and ^{238}U. An over recovery of Nb was seen with significantly higher amounts in the effluent as opposed to the calculated feed concentration. As a main constituent in CST, it's possible Nb is added to the effluent stream through CST fines that are pushed through the system during processing.

5.0 Conclusions

Cesium ion exchange column testing was conducted with CST lot 2002009604 sieved to <30 mesh to assess Cs ion exchange performance with AP-105 tank waste diluted to 5.5 and 7 M Na at 16 °C. Column testing was conducted at a small scale in PNNL's Radiochemical Processing Laboratory hot cells to accommodate the high radiological dose rate of the Hanford tank waste matrix. The results are summarized below.

5.1 Column Testing

AP-105 tank waste was diluted to produce 5.5 and 7 M Na feed conditions and processed through a two- or three-column format. The 7 M Na AP-105 testing necessitated the installation of a polish column after processing 470 BVs. A total of 6.3 L of 5.5 M Na and 7.9 L of 7 M Na AP-105 tank waste was processed through the Cs ion exchange system at 1.90 BV/h and 16 °C. Effluent samples were collected periodically from each column system during the load process and measured for ^{137}Cs to establish the Cs load curves. The flowrate was increased to 3.0 BV/h to process 12.0 BVs each of 0.1 M NaOH FD solution and water rinse. The following conclusions were drawn from the results of this work:

1. Testing showed that at 7 M Na and 16 °C, 791 BVs of AP-105 tank waste, processed at 1.90 BV/h, can be treated before reaching 50% Cs breakthrough on the lead column. The WAC limit was reached on the lag column when 496 BVs of 7 M Na AP-105 feed was processed. A polish column was installed and reached 0.04% breakthrough after processing 332 BVs of feed.
2. The WTP LAW WAC limit for the 7 M Na AP-105 lead and lag columns was reached nearly 100 BVs earlier than respective column breakthrough with 5.5 M Na AP-105 at 16 °C. Overall breakthrough slopes between the two tests indicated improved kinetic behavior in the 5.5 M Na AP-105 test due to the decreased concentrations of Na and Cs in the feed matrix.

5.1.1 Analyte Fractionation

1. Major components Al, K, Na, and S (sulfate) partitioned exclusively to the effluent in both 5.5 and 7 M Na AP-105 testing. Analytes such as Ca, Fe, and Pb saw significantly more recovery in the 7 M Na AP-105 effluent than in the 5.5 M Na AP-105 effluent.
2. The 7 M Na AP-105 effluent contained 55% of the feed Pu, and 77% of the feed Am. The balances of these isotopes were assumed to remain on the CST. Assuming the retained isotopes were bound only to the lead column CST bed, the CST would contain 41 nCi/g TRU, which is safely below the 100 nCi/g threshold defining TRU waste.
3. In addition to Cs removal, the 7 M Na AP-105 CST removed 99.5% of the ^{90}Sr but resulted in a significantly lower ^{90}Sr decontamination factor than has been observed previously. Previous removal with 5.5 M Na AP-105 at ambient temperature exceeded 99.9% ^{90}Sr and indicates temperature or matrix affects may likely be impacting ^{90}Sr removal.

5.2 Batch Contact Testing

Cs isotherms were developed for 7 M Na AP-105 tank waste at 13.1, 15.3, 24.9, and 35.2 °C using decontaminated effluent post ion exchange processing with nonradioactive Cs concentrations of 1.2E-4, 3.4E-4, 8.5E-4, and 1.7E-4 M. Batch contacts were conducted in duplicate with 0.075 g dry CST (lot 2002009604) per 15 mL of solution and agitated in a temperature-controlled box for ~240 hours. The isotherm data were fit to the Freundlich/Langmuir hybrid equilibrium model and the linear Freundlich model (for the lowest three Cs concentrations) to calculate K_d and Q values at AP-105 feed condition of 7.16E-5 M. Results of AP-105 batch contact testing were compared to AP-107, AP-105 (FY20), and AP-101 temperature studies. The following conclusions were made from this testing:

1. The Freundlich/Langmuir hybrid model overpredicts Cs loading at the lowest (1.2E-4 M) Cs concentration, underpredicts loading at 8.5E-4 M Cs, but accurately predicts the loading near the AP-101 feed condition. The linear Freundlich isotherm predicts loading with $R^2 > 0.99$ for the three lowest Cs concentrations at all temperatures.
2. The β values are largest for AP-105 when compared to AP-107 and AP-101, meaning the matrix is slightly less favorable for Cs uptake than the former two tanks.
3. The K_d values increase in the following order: AP-105 FY20 < 7 M Na AP-105 < 5.5 M Na AP-105 < AP-107 < AP-101.
4. The Cs loadings (Q) for 5.5 M and 7 M Na for AP-105 are indistinguishable at tank waste feed conditions.

6.0 References

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Appendix A – 5.5 M Na Column Load Data

The 5.5 M Na AP-105 lead and lag column loading raw data are provided in Table A.1. The raw data include the processed bed volumes (BVs) and corresponding ^{137}Cs concentration in the collected sample, % C/C_0, and the Cs decontamination factor (DF).

Table A.1. Lead and Lag Column Cs Breakthrough Results with 5.5 M Na AP-105

Lead Column				Lag Column			
BV	µCi ^{137}Cs/ mL	% C/C_0	DF	BV	µCi ^{137}Cs/ mL	% C/C_0	DF
13.2	2.51E-2	2.30E-2	4345	50.5	2.82E-3	2.58E-3	38,740
50.1	1.45E-2	1.33E-2	7524	104.6	2.27E-3	2.08E-3	48,072
104.2	2.59E-2	2.37E-2	4220	154.2	2.94E-4	2.69E-4	371,323
116.3	4.11E-2	3.76E-2	2656	250.1	2.53E-4	2.32E-4	431,417
153.8	1.15E-1	1.05E-1	949	294.3	3.71E-4	3.40E-4	294,007
202.8	3.33E-1	3.05E-1	328	385.1	9.45E-4	8.65E-4	115,593
293.9	1.06E+0	9.74E-1	103	432.0	1.75E-3	1.61E-3	62,258
339.4	1.79E+0	1.64E+0	61	523.4	1.01E-2	9.26E-3	10,794
431.6	4.73E+0	4.33E+0	23	564.3	1.98E-2	1.81E-2	5516
474.7	7.51E+0	6.88E+0	15	610.3	3.84E-2	3.52E-2	2844
563.9	1.44E+1	1.31E+1	8	634.1	5.51E-2	5.05E-2	1981
610.0	1.84E+1	1.69E+1	6				
633.7	2.20E+1	2.01E+1	5				

BV = bed volume, 6 mL/BV
DF = decontamination factor
C_0 = 109 µCi ^{137}Cs/ mL (reference date February 2023)

Appendix B – 7 M Na Column Load Data

The 7 M Na AP-105 lead and lag column loading raw data are provided in Table B.1 The raw data include the processed bed volumes (BVs) and corresponding ^{137}Cs concentration in the collected sample, % C/C0, and the Cs decontamination factor (DF).

Table B.1. Lead, Lag, and Polish Column Cs Breakthrough Results with 7 M Na AP-105

Lead Column				Lag Column				Polish Column			
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
15.7	1.13E-2	8.99E-3	11,121	55.8	2.09E-3	1.66E-3	60,180	514.8	4.82E-3	3.83E-3	26,103
55.5	5.44E-2	4.32E-2	2314	147.5	2.14E-3	1.70E-3	58,748	607.8	1.35E-2	1.07E-2	9325
70.2	8.51E-2	6.75E-2	1481	238.9	2.36E-3	1.87E-3	53,463	653.0	3.75E-3	2.98E-3	33,580
101.6	2.24E-1	1.78E-1	562	284.5	6.29E-3	5.00E-3	20,019	745.9	2.30E-2	1.83E-2	5464
146.9	6.78E-1	5.38E-1	186	375.7	3.66E-2	2.90E-2	3444	789.3	5.30E-2	4.21E-2	2376
190.9	1.64E+0	1.30E+0	77	469.6	1.65E-1	1.31E-1	765				
238.0	3.24E+0	2.58E+0	39	514.5	2.59E-1	2.06E-1	486				
328.9	7.61E+0	6.05E+0	17	561.4	4.66E-1	3.70E-1	270				
374.3	1.11E+1	8.80E+0	11	607.2	8.95E-1	7.11E-1	141				
419.3	1.44E+1	1.15E+1	9	698.8	2.29E+0	1.82E+0	55				
512.5	2.39E+1	1.90E+1	5	744.8	3.41E+0	2.71E+0	37				
558.8	2.93E+1	2.33E+1	4	800.7	5.09E+0	4.04E+0	25				
604.4	3.68E+1	2.92E+1	3								
695.2	4.98E+1	3.95E+1	3								
796.2	6.34E+1	5.04E+1	2								

BV = bed volume, 9 mL/BV
DF = decontamination factor
C$_0$ = 126 μCi ^{137}Cs/ mL (reference date March 2023)

Appendix C – Analytical Reports

This appendix includes analytical reports provided by Pacific Northwest National Laboratory's Analytical Support Operations (ASO) laboratory. In addition to the analyte results, these reports define the procedures used for chemical separations and analysis, as well as quality control sample results, observations during analysis, and overall estimated uncertainties. The analyses are grouped according to Analytical Service Request (ASR) number. Cross-references of ASO sample IDs to test description are provided in the body of the report (see Table 3.5 of the main report).

Appendix C Table of Contents

ASR 1420, 5.5 and 7 M Na AP-105 Ion Exchange Feed, Effluent, and Selected Lead Column Samples

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 Amy Westesen
Print Name Amy Westesen
Phone 371-7908 MSIN

Project Number: 79156
Work Package: NN2052

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)

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

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: NA**
  ☐ Dispose ☐ Return

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: see pg 2

- **Requested Analytical Work Completion Date:**
  (Note: Priority rate charge for < 10 business day turn-around time)
- **Negotiated Commitment Date:**
  (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: 1017
  or, Previous RPL Number:

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

Send Report To: **AM Westesen** **MSIN**
MSIN
Additional or Special Instructions

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

Date Delivered:
Delivered By (optional)
Time Delivered:
Group ID (optional)
CMC Waste Sample? X No ☐ Yes

Received By:
ASR Number: 1672 Rev.: 00
RPL Numbers: (23-0325) to (23-0328)
(first and last)

ASO Work Accepted By: **Signature/Date:**

ASO Staff Use Only	**Provide Analytes of Interest and Required Detection limits** - ☐ **Below** ☐ **Attached**			ASO Staff Use Only	
RPL Number	**Customer Sample ID**	**Sample Description (& Matrix, if it varies)**	**Analysis Requested**	**Test**	**Library**
23-0325	TI138-Comp-Feed	5.5 M Na AP-105 tank waste	1) Acid Digestion-128 Prep Lab 2) ICP/OES- Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr		
23-0326	TI138-Comp-Eff	5.5 M Na AP-105 tank waste- Cs removed	1) Acid Digestion-128 Prep Lab 2) ICP/OES- Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr		
23-0327	TI139-Comp-Feed	7 M Na AP-105 tank waste	1) GEA- All samples (Cs-137, Co-60 and Eu-154 and any other observed gamma emitting isotopes) 2) Tc-99 3) Sr-90 4) Np-AEA, Np-237 5) Pu-AEA, Pu-238, Pu-239/240 6) Am-AEA, Am-241 7) Acid Digestion-128 Prep Lab a) ICP/OES- Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr		
23-0328	TI139-Comp-Eff	7 M Na AP-105 tank waste- Cs removed	1) GEA- All samples (Cs-137, Co-60 and Eu-154 and any other observed gamma emitting isotopes) 2) IC-Anions- F, Cl, NO_2, NO_3, PO_4, C_2O_4, and SO_4 3) TIC/TOC- Hot Pursulfate 4) OH 5) Tc-99 6) Sr-90 7) Np-AEA, Np-237 8) Pu-AEA, Pu-238, Pu-239/240 9) Am-AEA, Am-241 10) Acid Digestion-128 Prep Lab a) ICP/OES- Al, As, Ba, Ca, Cd, Cr, Fe, K, Na, Ni, P, Pb, S, Sr, Ti, U, Zn, Zr b) ICP/MS- Ba, Nb, Pb, Sr, U-238		

ASR # ___1672________Rev.: __00____

Page ___1_____ of ______1_______

Gamma Energy Analysis (GEA)

Project / WP#:	79577/NN2052
ASR#:	1672
Client:	A. Westesen
Total # of Samples:	2

RPL ID	Client Sample ID
23-0327	TI139-Comp-Feed
23-0328	TI139-Comp-Eff

Analysis Type:	GEA
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*
Pre-dilution Prior to Radiochemical Processing?	☒ No ☐ Yes
Analysis Procedure:	Activity #4468 – *Gamma Energy Analysis (GEA)*
Reference Date:	02/22/2023 @ 9:00 am
Analysis Date or Date Range:	2/22/2023 & 4/07/2023 for GEA
Technician/Analyst:	TL Trang-Le and LR Greenwood for GEA
Rad Chem Electronic Data File:	RPG-RC\PNL\Projects\Backup files\Backup 23\23-0327 Westesen(AP-105).xlsx
ASO Project 98620 File:	File Plan 5872: T4.4 Technical (Radiochemistry), Gamma Calibration, daily checks, and maintenance records; and T3 standard certificates and preparation. ERecords CASE1830.150173A
M&TE Number(s):	Detector T for GEA

_______________ / _______________
Preparer **Date**

_______________ / _______________
Reviewer **Date**

_______________ / _______________
QE Review **Date**

SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheet for ASR 1672.00. All sample results for 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 1672 has been created for this report including all appropriate supporting records which includes the Pipette Performance Check Worksheet forms, laboratory bench records, and Liquid Scintillation Counter Analysis printouts. All supporting records are available upon request. Standard certificates, detector calibration records, control charts and balance calibration records can be found in the ASO Records.

Sample preparation and counting

Two samples were sent for gamma on February 22, 2023. Sample 23-0327 TI139-COMP-Feed was sent in 10mL geometry while the sample 23-0328 TI139-COMP-Eff was sent in the 20ml geometry. On April 7, 2023, the client asked the ASO staff from RPL/420 lab to re-prepare the 23-0327 TI139-COMP-Feed and sent with 0.133 g of sample in 2ml geometry for recount. The client requested the report on the re-prepare sample.

QUALITY CONTROL RESULTS

Tracer:

Tracers are not used for ASO GEA methods.

Process Blank (PB):

No process blank was prepared by ASO 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. Lab Assist Activity 4468, Gamma Energy Analysis, requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count.

Assumptions and Limitations of the Data

None

Interferences/Resolution

None

Uncertainty

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

Comments

None

Attachment: Data Report Sample Results for ASR 1672.00.

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

filename 23-0327 Westesen (AP-105)
4/21/2023

Client: A. Westesen
ASR 1672

Project: 79156
WP#: NN2052

Prepared by: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2023.04.24 11:20:04 -07'00'

Technical Reviewer: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2023.04.24 11:34:38 -07'00'

Procedures: Activity #4468- Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)

M&TE: Gamma detectors T
Count dates: Feb 22, 2023 & April 7, 2023

RPL ID: Sample ID: **Isotope**	23-0327 Re-prepare 2mL **TI139-Comp-Feed** **µCi/sample ± 1s**		23-0328 **TI139-Comp-Eff** **µCi/sample ± 1s**	
Co-60	< 8.30E-05		**6.29E-03**	**± 2%**
Cs-137	**1.38E+01**	**± 2%**	**2.76E-01**	**± 2%**
Eu-154	< 3.34E-04		**1.54E-03**	**± 7%**
Am-241	<1.94E-02		**5.67E-03**	**± 24%**
Sn-126	<4.61E-03		**2.77E-03**	**± 12%**
Sb-126	<2.55E-04		**2.08E-03**	**± 5%**

Sample 23-0327 (TI139-Comp-feed) is re-prepared in lab 420
with 1.33E-1 g in 2mL geometry.

MEMORANDUM

To:	**David Blanchard/Truc Trang-Le**	Project No.:	**79156/NN2052**
From:	**Veronika Vazquez**	Internal Distribution:	**ASR 1672 Files**
Subject:	**ASR 1672.00 Westessen (AP105) QE Review OH**		

QE Review for one ASO sample identified as 23-0328, was analyzed for hydroxide by R. Risenhuber on 04/04/2023. No preparation was required and sample was analyzed in triplicate following procedure RPG-CMC-228.

The QE has reviewed the hydroxide Analysis Report, the associated data, the associated quality control checks of the Balance Performance Check Log (Balance Sartorius ME414S, S/N: 21308482 and the Pipette Performance Check Worksheet. All the quality control checks met the required control limits for acceptance.

QC checks and blanks including reagent spike and lab prep blank were conducted during the analysis run and within acceptance criteria.

Pacific Northwest
NATIONAL LABORATORY

Client: Westesen

Report Date: 4/26/2023
Analysis Date: 4/4/2023

Subject: Hydroxide Analyses for: **TI139-Comp-Eff**

ASR: **1672** Rev-0
Sample ID. 23-0328

Procedure: RPG-CMC-228-Rev 1

Direct sample aliquots of 1 sample (see above assigned RPL Sample #'s, were analyzed in triplicate for the base constituents content following procedure RPG-CMC-228, and using manual titration. The titrant used was 0.1085 M HCl and the base standard, 0.1072 M NaOH was used for QC verification standards and matrix spike. -- See Standarization page of the result report.

The hydroxide Standard recovery was 106%, well within the allowed ± 20% recovery range. No hydroxide was detected in the reagent blank. Additional sample aliquots were weighed, thus density information is available -- see data pages following.

The best estimate of the MDL for this method is obtained from the reagent blank which did not show any inflection points and is consistent with a value of 0 within our measurement sensitivity. The results are accepted based on the QC data meeting normal acceptance criteria and the hydroxide RPD's @ <3% easily met the allowed ± 20% Relative Percent Deviation (RPD).

Following is the report summary, the sample results calculated from the raw data.

Prepared by: Date: 4/26/23

Reviewed by: Date: 4/27/23

Laboratory Bench Record

Client	**Westesen**				
Work Order #		**Procedure**	**RPG-CMC-228** OH-	**Balance #**	**see attached**
ASR #	**1672**			**Temperature**	**see attached**

Sample #'s	Tare (g)	+Sample (g)	+H2O (g)	Burette Start (mL)	Burette End (mL)	Sample mass (g)	Spike mass (g)	Total liquid mass titrated (g)	Total volume needed to titrate (mL)	Volume sample titrated (mL)
23-0328-1	16.5101	16.7572	21.7599	1.71	6.52	0.2471		5.2498	4.81	0.1831
23-0328-2	16.7528	17.0061	22.0088	6.61	11.57	0.2533		5.2560	4.96	0.1877
23-0328-3	16.8943	17.1494	22.1356	11.79	16.89	0.2551		5.2413	5.10	0.1890
23-0328 BS	16.6551	16.8516	21.8391	16.89	17.10		0.1965	5.1840	0.21	
23-0328 PB	16.6735	16.6735	21.6740	17.10	17.18			5.0005	0.08	

	M OH	Average	Stdev.S	%RSD
23-0328-1	2.8511	2.8824	0.0405136	1.4055
23-0328-2	2.8680			
23-0328-3	2.9282			
23-0328 BS	0.1139			

Sample Density Calculation

replicate	grams dispensed	g/mL	g/mL	
1	0.2677	1.3378	1.3497	Avg
2	0.2718	1.3583	0.008812	Stdev.s
3	0.2709	1.3538	0.653%	%RSD
4	0.2699	1.3488		

dispensing 0.2001 mL sample via pipette O42969I with balance 21308482

Notes: 23-0328 BS received 0.2 mL of 98620A-7-3 (0.1072 M NaOH) via pipette O42969I on balance 21308482 (exp 8/23). Samples were titrated with 98620A-7-4 (0.1085 M HCl)

Spike/Tracer ID	98620A-7-3			
Isotope	n/a	Vol Used (mL)		Vol Used (mL)
Concentration	0.1072 M NaOH	0.2001		0.2001
Reference Date				
Expiration Date				

Analyst/Date 4/26/23

Reviewer/Date 4/27/23

Client	Westesen	**Procedure**	RPG-CMC-228		**Balance #**	2670227
Work Ord			OH- Standardization		**Temperature**	N/A
ASR #	1672					

MW KHP (g/mol) **204.22**

NaOH standardization

sample	g KHP	mol KHP	mol NaOH needed	Volume at Start (mL)	Volume at End (mL)	Volume used for Titration (mL)	Volume Used for Titration (L)	Concentration OH (mol/L)	Concentration OH (mol/L)	
1	0.0665	3.256E-04	3.256E-04	2.00	5.05	3.05	3.05E-03	0.106764	0.1072	Avg
2	0.1492	7.306E-04	7.306E-04	5.23	12.03	6.80	6.80E-03	0.107438921	0.000281033	Stdev
3	0.1419	6.948E-04	6.948E-04	12.11	18.60	6.49	6.49E-03	0.107063005	0.262	%RSD
4	0.0693	3.393E-04	3.393E-04	10.15	13.31	3.16	3.16E-03	0.107386053		
5	0.1223	5.989E-04	5.989E-04	13.40	18.98	5.58	5.58E-03	0.107323292		

HCl Standarization

	mL HCl added	Volume OH at Start (mL)	Volume OH at End (mL)	Volume OH Used (mL)	Volume OH Used (mL)		Concentration HCl (mol/L)	
1	2.0072	9.73	11.79	2.06	2.03	Avg	0.1085	
2	2.0072	11.79	13.79	2.00	0.031144823	stdev	0.001688476	stdev
3	2.0072	13.81	15.88	2.07	1.533	%RSD	1.556	%RSD
4	2.0072	15.88	17.90	2.02				
5	2.0072	17.9	19.91	2.01				

KHP: TCI, lot RI7AG-DG
0.1072 M NaOH: Lot 98620A-7-3
0.1085 M HCl: Lot 98620A-7-4

Notes: KHP was dried for 2 days at 105.5°C and then stored under vacuum.

Analyst/Date 4/26/23

Reviewer/Date 4/27/23

PNNL - Analytical Support Operations

Pipette Performance Check Worksheet

Procedure: PNL-ASO-066

ASRs:	1672
System File (if applicable):	Westesen OH
Analyst Performed:	Matthew RisenHuber
Balance SN:	

Temperature (°C)	19.7
Water Density @ °C	0.9982063
Temperature Correction per Procedure PNL-ASO-066	

%RSD Acceptance Criterion [(a)] = 0.5 **%Vol Bias Acceptance Criterion [(a)] = 1%**

Balance ID	21308482, exp 8/23
Date Performed	4/4/2023
Pipette ID	O42969I
Selected/Fixed vol. (mL)	0.2
Aliquot 1 Wt. (g)	0.1994
Aliquot 2 Wt. (g)	0.1999
Aliquot 3 Wt. (g)	0.1997
Aliquot 4 Wt. (g)	0.1998
Aliquot 5 Wt. (g)	0.1998
Average Wt. (g)	0.19972
%RSD (1 σ)	0.1
Delivery Volume (mL)	0.2001
Delivery Vol %Bias (mL)	0.04%
Acceptable %RSD(Y/N)	Y
Acceptable %Bias (Y/N)	Y

Balance ID	
Date Performed	
Pipette ID	
Selected/Fixed vol. (mL)	
Aliquot 1 Wt. (g)	
Aliquot 2 Wt. (g)	
Aliquot 3 Wt. (g)	
Aliquot 4 Wt. (g)	
Aliquot 5 Wt. (g)	
Average Wt. (g)	
%RSD (1 σ)	
Delivery Volume (mL)	
Delivery Vol %Bias (mL)	
Acceptable %RSD(Y/N)	
Acceptable %Bias (Y/N)	

Balance ID	22006647
Date Performed	4/42023
Pipette ID	4831306
Selected/Fixed vol. (mL)	2
Aliquot 1 Wt. (g)	2.0044
Aliquot 2 Wt. (g)	2.0018
Aliquot 3 Wt. (g)	2.0030
Aliquot 4 Wt. (g)	2.0026
Aliquot 5 Wt. (g)	2.0042
Average Wt. (g)	2.0032
%RSD (1 σ)	0.055
Delivery Volume (mL)	2.0072
Delivery Vol %Bias (mL)	0.36%
Acceptable %RSD(Y/N)	Y
Acceptable %Bias (Y/N)	Y

Balance ID	
Date Performed	
Pipette ID	
Selected/Fixed vol. (mL)	
Aliquot 1 Wt. (g)	
Aliquot 2 Wt. (g)	
Aliquot 3 Wt. (g)	
Aliquot 4 Wt. (g)	
Aliquot 5 Wt. (g)	
Average Wt. (g)	
%RSD (1 σ)	
Delivery Volume (mL)	
Delivery Vol %Bias (mL)	
Acceptable %RSD(Y/N)	
Acceptable %Bias (Y/N)	

Balance ID	
Date Performed	
Pipette ID	
Selected/Fixed vol. (mL)	
Aliquot 1 Wt. (g)	
Aliquot 2 Wt. (g)	
Aliquot 3 Wt. (g)	
Aliquot 4 Wt. (g)	
Aliquot 5 Wt. (g)	
Average Wt. (g)	
%RSD (1 σ)	
Delivery Volume (mL)	
Delivery Vol %Bias (mL)	
Acceptable %RSD(Y/N)	
Acceptable %Bias (Y/N)	

Balance ID	
Date Performed	
Pipette ID	
Selected/Fixed vol. (mL)	
Aliquot 1 Wt. (g)	
Aliquot 2 Wt. (g)	
Aliquot 3 Wt. (g)	
Aliquot 4 Wt. (g)	
Aliquot 5 Wt. (g)	
Average Wt. (g)	
%RSD (1 σ)	
Delivery Volume (mL)	
Delivery Vol %Bias (mL)	
Acceptable %RSD(Y/N)	
Acceptable %Bias (Y/N)	

(a) For volumes greater than 100 uL, the %RSD Criterion is set at 0.5% and the Bias Criterion at 1%.

Analyst/Date Matt 4/26/23

Review/Date 4/27/23

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

filename 23-0327 Westesen (AP-105)
4/27/2023

Client: A. Westesen
ASR 1672

Project: 79156
WP#: NN2052

Prepared by: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2023.05.03 08:39:49 -07'00'

Technical Reviewer: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2023.05.03 08:38:38 -07'00'

Procedures: Activity #4416 Coprecipitation Mounting of Actinides for Alpha Spectroscopy
Activity #4417 Separation of Plutonium and Americium using Eichrom TRU resin
M&TE: Alpha AEA
Count dates: April 19-20, 2023

	lab	Measured Activity, µCi/g ± 1s						
Sample	ID	Pu-238		Pu-239+240		Am-241		Np-237
TI139-Comp-Feed	23-0327	**8.17E-06**	**± 3%**	**3.85E-05**	**± 2%**	**2.37E-04**	**± 2%**	+
TI139-Comp-Eff	23-0328	**6.94E-06**	**± 3%**	**2.90E-05**	**± 2%**	**1.93E-04**	**± 2%**	
	23-0328 DUP	**1.24E-05**	**± 2%**	**2.83E-05**	**± 2%**	**1.61E-04**	**± 2%**	
	Blank Spike**			97%		89%		
	Matrix Spike*			-		-		
	Process Blank	< 4.4E-8		< 2.8E-8		< 2.5E-8		

*The matrix spikes are too small relative to the sample activity to give reliable results.
** The blank spike yields were determined by the Pu-242 and Am-243 tracers.
+ The Np237 analyses have not been completed yet

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

filename 23-0327 Westesen (AP-105)
4/17/2023

Client: A. Westesen
ASR 1672

Project: 79156
WP#: NN2052

Prepared by: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2023.04.18 11:40:24 -07'00'

Technical Reviewer: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2023.04.18 11:52:10 -07'00'

Procedures: Activity #4672- Technicium Measurement

M&TE: Perkin Elmer TriCarb model 3100TR liquid scintillation spectrometer
Count dates: 11-Apr-23

Sample	lab ID	Measured Activity, µCi/g ± 1s Tc-99	
TI139-Comp-Feed	23-0327	**8.39E-02**	**± 2%**
TI139-Comp-Eff	23-0328	**7.07E-02**	**± 2%**
	23-0328 DUP	**7.08E-02**	**± 2%**
	Blank Spike	99%	
	Matrix Spike	133%*	
	Process Blank	< 2.9E-5	

*The matrix spike is too small relative to the large sample activity to give reliable results

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

filename 23-0327 Westesen (AP-105)
4/28/2023
Rev. 1

Client: A. Westesen
ASR 1672

Project: 79156
WP#: NN2052

Prepared by: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2023.04.28 09:54:49 -07'00'

Technical Reviewer: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2023.04.28 09:51:24 -07'00'

Procedures: Activity #4409- Strontium separation using Eichrom strontium resin

M&TE: Perkin Elmer TriCarb model 3100TR liquid scintillation spectrometer
Count dates: 21-Apr-23

Sample	lab ID	Measured Activity, µCi/g ± 1s Sr-90	
TI139-Comp-Feed	23-0327	**4.59E-01**	**± 2%**
TI139-Comp-Eff	23-0328	**2.22E-03**	**± 2%**
	23-0328 DUP	**2.28E-03**	**± 2%**
	Blank Spike	93%	
	Matrix Spike	59%*	
	Process Blank	< 2.3E-6	

*The matrix spike is too small relative to the sample activity to give reliable results. Sample 23-0327 was diluted and reanalyzed since the original count had too much activity for the liquid scintillation counter. The report was revised with the new data for this sample.

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

Project / WP#: 79156 / NN2052
ASR#: 1672
Client: A. Westesen
Total Samples: 4 (liquids)

ASO Sample ID	Client Sample ID	Client Sample Description
23-0325	TI138-Comp-Feed	5.5 M Na AP-105 tank waste
23-0326	TI138-Comp-Eff	5.5 M Na AP-105 tank waste – Cs removed
23-0327	TI139-Comp-Feed	7 M Na AP-105 tank waste
23-0328	TI139-Comp-Eff	7 M Na AP-105 tank waste – Cs removed

Sample Preparation: Simple dilution of "as received" samples in 5% v/v HNO3 performed by C. Perez.

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

Analyst:	C. Perez	**Analysis Date:**	3/23/2023	**ICP File:**	C0901

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: 21308482
	☐	Mettler AT400 Balance	SN: 1113162654
	☐	Sartorius R200D Balance	SN: 39080042
	☐	Mettler AT201 Balance	SN: 192720-92
	☒	Ohaus Pioneer PA224C	SN: B725287790
	☐	SAL Cell 2 Balance	SN: 8033311209

Report Preparer

Date

Review and Concurrence

Date

4 liquid samples were submitted under Analytical Service Request (ASR) 1672 and were analyzed by ICP-OES. The sample had an acid digestion performed and was done in combination with samples from ASR 1672.

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 highlighted section of the attached ICP-OES Data Report. The quality control (QC) results for the AOI have been evaluated and are presented below.

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), post-digestion spikes, duplicate, blank spike, and serial dilution were conducted during the analysis run.

<u>Preparation Blank (PB):</u>

A preparation blank was supplied with the samples. All AOI except for Barium (Ba) and Iron (Fe) were within the acceptance criteria of <EQL (estimated quantitation level), <50% regulatory decision level, or less than ≤10% of the concentration in the sample. Both were very low levels and just above the EQL.

<u>Blank Spike (BS)/Laboratory Control Sample (LCS):</u>

A 50:50 mixture of the MCVA and MCVB solutions 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 meeting this requirement were within the acceptance criterion of 80% to 120%.

<u>Duplicate/Replicate Relative Percent Difference (RPD):</u>

A Replicate of each sample was prepared and analyzed. RPD are listed for all analytes that were measured at or above the EQL. All AOI detected were within the acceptance criterion of ≤20% for liquid samples.

<u>Triplicate Relative Standard Deviation (RSD):</u>

No triplicate sample was analyzed.

Matrix-Spike (MS) Sample:

The samples were analyzed "as received" and diluted as appropriate for analyses of the target analytes. A matrix spike sample was generated due to the sample preparation beforehand. Aluminum (Al), Chromium (Cr) and Sodium (Na) shows as "nr" on the percentage recovered. This concludes that the spike concentration is <25% of the sample concentration which gives inconclusive results. All other AOI passed the recovery percentage.

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. All AOI were within the acceptance criteria of 90% to 110%.

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). All AOI passed on the ICB/CCBs ran except for Sodium (Na) on the ICP03.0-5 rerun and the ICP03.0-6 rerun. ICB/CCB failed on the Sodium, but the sample concentrations were 20X the EQL therefore the data is acceptable.

Low-Level Standard (LLS):

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

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. Recovery values are listed for all analytes included in the SST that were measured at or above the EQL. All AOI were within the acceptance criteria of 80% to 120%.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 23-0325. The percent difference (%D) for all AOI were withing the acceptance criteria of ≤ 10%.

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

A post-digestion spike (A Component) was conducted on sample 23-0327. All AOI were within the acceptance criterion of 80% to 120%.

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

A post-digestion spike (B Component) was conducted on sample 23-0328. All AOI were within the acceptance criterion of 80% to 120%.

Post-Digestion Spike (PS-Q3A) - Sample (A Tormont Component):

A post-digestion spike (A Tormont Component) was not conducted.

Post-Digestion Spike (PS-Q3B) - Sample (B Tormont Component):

A post-digestion spike (B Tormont Component) was not conducted.

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, Eu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sm, 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 >	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023
		Process Factor >	1.0	1.0	186.1	186.1	465.3	2326.5	185.8	464.4	2321.9	185.2	463.0
			405 Diluent	23-0326 PB @1x	23-0325 @10x	23-0325 @10x Replicate	23-0325 @25x	23-0325 @125x SRD	23-0326 @10x	23-0326 @25x	23-0326 @125x SRD	23-0326 Dup @10x	23-0326 Dup @25x
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >			T1138-Comp-Feed	T1138-Comp-Feed	T1138-Comp-Feed	T1138-Comp-Feed	T1138-Comp-Eff	T1138-Comp-Eff	T1138-Comp-Eff	T1138-Comp-Eff	T1138-Comp-Eff
(µg/mL)	(µg/mL)	(Analyte)	(µg/mL)	(µg/mL)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.0019	0.019	Ag	--	--	--	--	--	--	--	--	--	--	--
0.0101	0.101	Al	--	--	11,000	11,300	10,900	10,700	10,700	10,800	11,100	9,180	10,800
0.0619	0.619	As	--	--	--	--	--	--	--	--	--	--	--
0.0060	0.060	B	[0.038]	[0.033]	54.1	55.0	56.3	[120]	31.2	35.1	[86]	27.6	32.5
0.0001	0.001	Ba	--	0.0078	0.353	0.359	[0.40]	[0.52]	[0.039]	--	--	[0.026]	--
0.0001	0.001	Be	--	--	[0.058]	[0.056]	[0.080]	--	[0.051]	--	--	[0.037]	--
0.0245	0.245	Bi	--	--	--	--	--	--	--	--	--	--	--
0.0056	0.056	Ca	--	[0.038]	36.3	36.2	36.1	[58]	28.1	28.3	[24]	24.8	29.1
0.0014	0.014	Cd	--	[0.0015]	[0.83]	[1.2]	[1.1]	[5.1]	[0.68]	[1.5]	--	[0.52]	--
0.0103	0.103	Ce	--	--	--	--	--	--	--	--	--	--	--
0.0043	0.043	Co	--	--	--	--	--	--	--	--	--	--	--
0.0020	0.020	Cr	--	--	262	261	260	258	250	250	261	216	250
0.0023	0.023	Cu	--	--	6.02	5.83	[5.9]	[6.2]	5.77	[6.0]	[8.0]	5.00	[5.4]
0.0023	0.023	Dy	--	--	--	--	--	--	--	--	--	--	--
0.0006	0.006	Eu	--	--	--	--	--	--	--	--	--	--	--
0.0014	0.014	Fe	--	0.0373	[1.6]	[1.1]	[0.80]	--	[0.75]	[0.90]	--	[1.5]	[3.1]
0.0312	0.312	K	[0.041]	--	3,150	3,200	2,950	2,780	2,990	2,860	2,800	2,560	2,870
0.0019	0.019	La	--	--	--	--	--	--	--	--	--	--	--
0.0007	0.007	Li	--	--	[0.42]	[0.51]	--	--	[0.31]	--	--	[0.26]	--
0.0018	0.018	Mg	--	[0.0029]	[0.39]	--	--	[5.7]	--	--	--	--	--
0.0002	0.002	Mn	--	--	0.774	0.784	[0.71]	[0.76]	0.717	[0.66]	[1.0]	0.670	[0.64]
0.0044	0.044	Mo	--	--	40.5	40.7	43.4	[41]	38.0	40.4	[55]	30.2	36.5
0.0073	0.073	Na	[0.010]	[0.031]	103,000	103,000	103,000	104,000	98,600	102,000	107,000	86,300	103,000
0.0088	0.088	Nd	--	--	--	--	--	--	--	--	--	--	--
0.0022	0.022	Ni	--	[0.0033]	22.1	21.7	23.3	[23]	20.9	24.0	[28]	18.8	21.1
0.0905	0.905	P	--	--	335	319	[320]	[430]	306	[310]	[240]	269	[330]
0.0269	0.269	Pb	--	--	[15]	[14]	[16]	[85]	[5.9]	--	--	[5.1]	--
0.0054	0.054	Pd	--	--	--	--	--	--	--	--	--	--	--
0.0211	0.211	Rh	--	--	--	--	--	--	--	--	--	--	--
0.0063	0.063	Ru	--	--	[1.2]	--	--	--	[1.6]	[3.5]	--	--	--
0.1262	1.262	S	--	--	862	907	859	[890]	850	899	[910]	753	855
0.0598	0.598	Sb	--	--	--	--	--	--	--	--	--	--	--
0.1656	1.656	Se	--	--	--	--	--	--	--	--	--	--	--
0.0086	0.086	Si	[0.022]	[0.013]	[2.9]	[3.8]	[8.3]	[21]	[4.2]	--	[31]	[3.4]	--
0.0291	0.291	Sn	--	--	--	--	--	--	--	--	--	--	--
0.0001	0.001	Sr	--	[0.0004]	[0.15]	[0.15]	[0.12]	--	--	--	--	--	--
0.0246	0.246	Ta	--	--	--	--	--	--	--	--	--	--	--
0.0197	0.197	Te	--	--	--	--	--	--	--	[15]	--	[4.4]	[20]
0.0071	0.071	Th	--	--	--	--	--	--	--	--	--	--	--
0.0006	0.006	Ti	--	[0.0008]	--	--	--	--	[0.44]	[0.59]	--	[0.41]	[0.40]
0.0814	0.814	Tl	--	--	--	--	--	--	--	--	--	--	--
0.0410	0.410	U	--	[0.042]	[12]	[7.7]	--	--	[9.4]	--	[98]	[10]	--
0.0013	0.013	V	[0.0028]	[0.0031]	[0.77]	[0.90]	[1.4]	--	[0.73]	[1.0]	[3.6]	[0.76]	[1.3]
0.0161	0.161	W	--	--	[14]	[15]	[12]	--	[28]	[28]	--	[20]	[30]
0.0006	0.006	Y	--	--	--	--	--	--	--	--	--	--	--
0.0027	0.027	Zn	--	[0.019]	--	--	--	--	[1.1]	--	--	--	--
0.0014	0.014	Zr	--	--	--	--	--	--	[0.93]	[0.71]	--	[0.91]	[1.2]

Other Analytes

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%.

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assisted digests.

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

		Run Date >	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023	3/23/2023
		Process Factor >	2315.0	167.1	417.8	2088.9	2088.9	189.7	474.1	2370.7
			23-0326 Dup @125x SRD	23-0327 @10x	23-0327 @25x	23-0327 @125x SRD	23-0327 @125x SRD Rep.	23-0328 @10x	23-0328 @25x	23-0328 @125x SRD
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID >	T1138-Comp-Eff	T1139-Comp-Feed	T1139-Comp-Feed	T1139-Comp-Feed	T1139-Comp-Feed	T1139-Comp-Eff	T1139-Comp-Eff	T1139-Comp-Eff
(µg/mL)	(µg/mL)	(Analyte)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)	(µg/g)
0.0019	0.019	Ag	--	--	--	--	--	--	--	--
0.0101	0.101	Al	10,100	13,100	13,100	13,200	13,400	11,400	12,700	12,900
0.0619	0.619	As	--	--	--	--	--	--	--	--
0.0060	0.060	B	[75]	42.7	45.9	[79]	[73]	26.3	29.3	[66]
0.0001	0.001	Ba	--	0.635	0.705	[0.72]	[0.76]	[0.066]	[0.080]	--
0.0001	0.001	Be	--	[0.070]	[0.050]	--	--	[0.061]	[0.040]	--
0.0245	0.245	Bi	--	--	--	--	--	--	--	--
0.0056	0.056	Ca	[41]	35.5	37.4	[43]	[45]	26.0	29.9	[31]
0.0014	0.014	Cd	--	[1.2]	[1.2]	[8.2]	--	[1.2]	[1.7]	--
0.0103	0.103	Ce	--	--	--	--	--	--	--	--
0.0043	0.043	Co	--	--	--	--	--	--	--	--
0.0020	0.020	Cr	236	308	310	321	316	272	294	314
0.0023	0.023	Cu	[6.2]	7.20	[7.2]	[9.0]	[9.3]	6.22	[7.2]	[8.8]
0.0023	0.023	Dy	--	--	--	--	--	--	--	--
0.0006	0.006	Eu	--	--	--	--	--	--	--	--
0.0014	0.014	Fe	--	[2.0]	[1.6]	[7.8]	--	[1.0]	[0.98]	--
0.0312	0.312	K	2,620	3,880	3,530	3,460	3,450	3,270	3,380	3,630
0.0019	0.019	La	--	--	--	--	--	--	--	--
0.0007	0.007	Li	--	[0.46]	[0.46]	--	--	[0.23]	--	--
0.0018	0.018	Mg	--	--	[0.74]	--	--	--	--	--
0.0002	0.002	Mn	--	0.922	0.876	[0.81]	[0.48]	0.840	[0.82]	[0.67]
0.0044	0.044	Mo	[47]	47.0	48.0	[58]	[53]	39.5	41.9	[63]
0.0073	0.073	Na	97,600	121,000	126,000	127,000	129,000	107,000	120,000	123,000
0.0088	0.088	Nd	--	--	--	--	--	--	--	--
0.0022	0.022	Ni	[10]	26.1	28.0	[23]	[24]	24.5	27.1	[31]
0.0905	0.905	P	--	388	416	[410]	[310]	327	[340]	[370]
0.0269	0.269	Pb	--	[17]	[17]	--	--	[7.7]	--	--
0.0054	0.054	Pd	--	--	--	--	--	--	--	--
0.0211	0.211	Rh	--	--	--	--	--	--	--	--
0.0063	0.063	Ru	--	[1.1]	--	--	--	[1.9]	--	--
0.1262	1.262	S	[690]	1,070	1,060	[780]	[990]	929	987	[700]
0.0598	0.598	Sb	--	--	--	--	--	--	--	--
0.1656	1.656	Se	--	--	--	--	--	--	--	--
0.0086	0.086	Si	[45]	[2.7]	--	--	--	--	[6.6]	[40]
0.0291	0.291	Sn	--	--	--	--	--	--	--	--
0.0001	0.001	Sr	--	[0.15]	[0.15]	--	--	--	--	--
0.0246	0.246	Ta	--	--	--	--	--	--	--	--
0.0197	0.197	Te	--	[6.0]	[11]	--	--	[3.9]	[11]	--
0.0071	0.071	Th	--	--	--	--	--	--	--	--
0.0006	0.006	Ti	--	--	--	[1.7]	--	[0.69]	[0.94]	[1.9]
0.0814	0.814	Tl	--	--	--	--	--	--	--	--
0.0410	0.410	U	--	--	[20]	--	[97]	[8.8]	--	--
0.0013	0.013	V	[5.6]	[0.59]	[1.1]	[5.4]	[4.8]	[0.89]	[1.8]	[11]
0.0161	0.161	W	--	29.7	[33]	[37]	--	[28]	[31]	--
0.0006	0.006	Y	--	--	--	--	--	--	--	--
0.0027	0.027	Zn	--	[0.74]	[1.7]	--	--	[0.54]	--	--
0.0014	0.014	Zr	--	--	--	--	--	[1.6]	[2.2]	--
Other Analytes										

QC Performance 3/23/2023

Criteria >	≤ 20%	75%-125%	75%-125%	80%-120%	80%-120%	≤ 10%
QC ID >	**23-0327 @125x SRD Rep Dup**	**MS-23-0326 @25x A**	**MS-23-0326 @25x B**	**PSA-0327 @25x AS-A**	**PSB-0328 @25x AS-B**	**23-0325 @125x SRD 5-Fold Serial Dil**
Analytes	**RPD (%)**	**%Rec**	**%Rec**	**%Rec**	**%Rec**	**%Diff**
Ag				97		
Al	1.3	nr		107		1.3
As				99		
B			89	104		
Ba		95		99		
Be		101		103		
Bi				99		
Ca		99		101		
Cd		103		104		
Ce		94			99	
Co				102		
Cr	1.5	nr		99		0.8
Cu		100		105		
Dy					101	
Eu					100	
Fe		98		103		
K	0.2	103		103		5.9
La		96			100	
Li		101		106		
Mg		98		102		
Mn		101		104		
Mo			98	98		
Na	1.3	nr		98		0.7
Nd		93			99	
Ni		98		102		
P			116	97		
Pb		101		102		
Pd					95	
Rh					97	
Ru					98	
S			107		99	
Sb				99		
Se				107		
Si				103		
Sn				100		
Sr		97		106		
Ta				101		
Te					99	
Th		98			102	
Ti			101	100		
Tl				93		
U		97			106	
V		96		100		
W				102		
Y				101		
Zn		99		108		
Zr			105	101		
Other Analytes						

Shaded results are outside the acceptance criteria.

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

na = not applicable; KOH flux and Ni crucible or Na_2O_2 flux and Zr crucible for fusion preparations, or Si for HF assis

		Run Date >	**05/23/23**	**05/23/23**	**05/23/23**	
		Process Factor	**1.00**	**569**	**569**	
Units: ng/g		**RPL/LAB >**	**Blank Avg.**	**23-0328 (30x)**	**23-0328 (30x) Rep**	
Instr. Det. Limit (IDL)	**Est. Quant. Limit (EQL)**	**Client ID >**	**2% HNO3 Lab Blank**	**Tl139-Comp-Eff**		
0.002	0.019	**Sr 88**	**0.000**	**42.0**	**44.1**	Less than the lower calibration limit and <IDL
0.006	0.064	**Ba 137**	**-0.001**	**114.0**	**113.6**	
0.001	0.014	**Ba 138**	**-0.001**	**83.8**	**80.8**	Less than the lower calibration limit.
0.003	0.032	**Pb 206**	**0.000**	**4334.9**	**3896.3**	
0.002	0.016	**Pb 207**	**-0.001**	**4423.9**	**3952.3**	
0.002	0.020	**Pb 208**	**0.000**	**4371.3**	**3928.4**	
0.000	0.003	**U 238**	**0.000**	**4084.6**	**3916.0**	

Internal Standard % Recovery

Tb 159 (IS)	102%	98%	99%

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 times the "multiplier". Overall error for values ≥ EQL is estimated to be within ±15%.
IS = Internal Standard. The concentration of certain elements cannot be determined due to the presence of the IS in all solutions.

QC Performance 5/23/23

Criteria >	**≤ 35%**	**75%-125%**	**≤ 10%**	**75%-125%**	**75%-125%**
QC ID >	**23-0328 Rep**	**23-0328 Post Spike CCV71A**	**23-0328 5-fold Serial Dil**	**23-0326 MS**	**23-0326 BS**
Analytes	**RPD (%)**	**%Rec**	**%Diff**	**%Rec**	**%Rec**
Sr 88	4.9%	96.9%	36.9%	36.6%	283.5%
Ba 137	0.3%	108.2%	0.7%	93.5%	97.4%
Ba 138	3.7%	108.1%	0.9%	92.6%	98.9%
Pb 206	10.7%	85.2%	0.4%	96.1%	95.0%
Pb 207	11.3%	73.8%	0.0%	91.4%	92.6%
Pb 208	10.7%	72.7%	0.4%	92.7%	92.1%
U 238	4.2%	91.7%	0.5%	95.1%	92.4%

Internal Standard % Recovery

Tb 159 (IS)	99%	97%	98%

Conc. Is less than 10X the EQL for the Serial Dilution on Sr-88.

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.

		Run Date >	**05/23/23**	**05/23/23**	**05/23/23**
		Process Factor	**1.00**	**569**	**569**
ppb = ng/g		**RPL/LAB >**	**Blank Avg.**	**23-0328 (30x)**	**23-0328 (30x) Rep**
Instr. Det. Limit (IDL)	**Est. Quant. Limit (EQL)**	**Client ID >**	**2% HNO3 Lab Blank**	**TI139-Comp-Eff**	
0.0010	0.0070	**Nb 93**	**0.000**	**660.0**	**652.1**

Internal Standard % Recovery

Tb 159 (IS)	98%	96%	94%

QC Performance 5/23/23

Criteria >	**≤ 35%**	**75%-125%**	**≤ 10%**
QC ID >	**23-0328 Rep**	**23-0328 Post Spike CCV71B**	**23-0328 5-fold Serial Dil**
Analytes	**RPD (%)**	**%Rec**	**%Diff**
Nb 93	0.3%	107.1%	0.3%

Internal Standard % Recovery

Tb 159 (IS)	83%	104%	102%

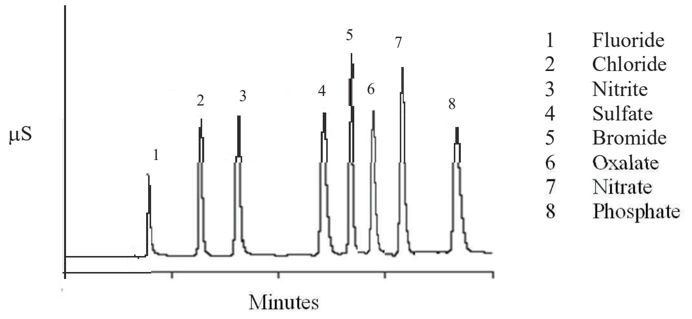
Matrix Spike and Blank spike were not spiked with Niobium.

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

PO Box 999, Richland, Washington 99352

Common Inorganic Anions

Dionex AS18 Column; Hydroxide Gradient



Client:	A. Westesen	***ASR #:***	1672
Project #:	79156	***# Samples:***	1 Liquid(s)
Charge Code:	NN2052		

Procedure, Analysis, System, and Records Information

Analysis Procedure	RPG-CMC-212 Rev.2, "Determination of Common Anions by Ion Chromatography"
Prep Procedure	None
Analyst	C Perez
Analysis Dates	03/29/2023
Calibration Date	03/7/2023
Cal/Ver Stds. Prep Date	03/7/2023
Excel Data File	IC-0266 ASR-1672 Westesen
M&TE Numbers	IC System (M&TE)
	Balance: B725287790
All Analysis Records	Chemical Measurement Center 98620: RIDS IC System File (IC-0266)

Christian Perez Digitally signed by Christian Perez
Date: 2023.07.13 07:06:13 -07'00'

Prepared By **Date**

David L Jr Blanchard Digitally signed by David L Jr Blanchard
Date: 2023.07.13 10:06:28 -07'00'

Reviewed By **Date**

IC Report

Sample Results

See Attachment: Sample Results ASR 1672

Sample Analysis/Results Discussion

One sample was submitted to the Analytical Support Operations (ASO) laboratory for ion chromatography analysis under ASR 1672, RPL Number 23-0328. The results are discussed in this report. The analytes of interest for sample TI139-Comp-Eff are Chloride (Cl-), Fluoride (F-), Nitrate (NO3-), Nitrite (NO2-), Sulfate (SO4-), Oxalate (C2O4) and Phosphate (PO4-). Different dilutions are reported due to allow for accurate representation of the analytes of interest. Nitrate and Nitrite are reported from the higher dilution numbers that the instrument could detect and keep in range for the samples. Oxalate will not be reported (see the Quality Control Discussion).

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

Data Limitations

N/A

Quality Control Discussion

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

Oxalate is not reported due to issues with calibration verification and QC failures. Oxalate can be difficult to detect accurately with this instrument and column used.

IC Workstation QC Results

Process Blank: A process blank was provided from the radiochemistry team and all AOIs passed at the 1x dilution.

Duplicate: The relative percent difference (RPD) is reported for all analytes which were measured at or above the EQL. The reported RPDs for all analytes of interest at or above the EQL at 100x dilution except for Nitrate and Nitrite since they were over range for instrument detection. The reported RPDs for Nitrate and Nitrite passed on the 1000x dilution.

Triplicate: No triplicate was provided/analyzed.

IC Report

Serial Dilution: The percent difference (%D) is reported for all analytes which were measured at or above the EQL. The reported %Ds for all analytes of interest at or above the EQL at 1000x and 5000x dilutions, met the acceptance criteria of ≤ 10%.

Analytical Spike (AS) (Accuracy): An analytical spike was prepared by adding a known concentration of a multi-mix standard to samples after preliminary preparation. All analytes of interest met recovery acceptance criteria of 80% to 120%.

Blank Spike): All analytes of interest met the acceptance criteria of 80% to 120% at the 1x dilution.

Low Calibration Verification Sample (LCV): All analytes of interest met the acceptance criteria of 75% to 125% except for Phosphate for being higher at 130%. The Phosphate sample results may be slightly biased high. The Analytical Spike for Phosphate passed so this brings more confidence in the sample results for Phosphate.

High Calibration Verification Sample (HCV): All analytes of interest met the acceptance criteria of 90% to 110% except for Nitrate which was 116%. The Blank spike concentration fell at a similar concentration to the sample which shows confidence in the calibration curve where the sample is reported. No Flagging necessary for Nitrate.

ICV/CCV & ICB/CCB Samples: Numerous calibration verification standards and calibration verification blanks were analyzed with the run. The results for the ICV/CCV & ICB/CCB samples (that bound the reported results for each analyte of interest) are within the acceptance criteria of the ASO's QA Plan (i.e., verification standard recoveries from 90% to 110% and verification blank results <EQL or <5% of reported sample result).

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.

IC Report

- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference.

Sample Results ASR-1672

Table Summary

🟨 = Reporting Result

RPL Number	Client Sample ID	Ext. Dil. And PF	F			Cl			NO_2			SO_4			NO_3			PO_4		
			MDL µg/g	Result µg/g	DF	MDL µg/g	Result µg/g	DF	MDL µg/g	Result µg/g	DF	MDL µg/g	Result µg/g	DF	MDL µg/g	Result µg/g	DF	MDL µg/g	Result µg/g	DF
23-0328 @100x	TI139-Comp-Eff	772	3.86	30.1	J	10.0	3512.6		19.3	45162.0	O.R.	29.3	2370.0		19.3	116803.6	O.R.	29.336	802.88	
23-0328 Dup @100x	TI139-Comp-Eff	760	3.8	29.6	J	9.9	3503.6		19	44460.0	O.R.	28.9	2363.6		19.0	116584.0	O.R.	28.88	760	
23-0328 @1000x	TI139-Comp-Eff	7720	193	38.6	U	100.4	3311.9		193	54117.2		293.4	2238.8	J	193	101132.0		293.4	293.4	U
23-0328 Dup @1000x	TI139-Comp-Eff	7600	38	38.0	U	98.8	3344.0		190	54492.0		288.8	2204.0	J	190.0	101080.0		288.8	288.8	U

Sample QC Results ASR-1672

Sample/Replicate Precision Results

RPL Number	Sample ID	F		Cl		NO_2		SO_4		NO_3		PO_4	
		µg/g	RPD	µg/g	RPD	µg/g	RPD	µg/g	RPD	µg/g	RPD	µg/g	RPD
23-0328 @100x	Sample	J	--	4.55	--	OvrRng	--	3.07	--	OvrRng	--	1.04	--
23-0328 Dup @100x	Duplicate | RPD	J	N/A	4.61	1.31	OvrRng	N/A	3.11	1.29	OvrRng	N/A	1	3.92
23-0328 @1000x	Sample	U	--	0.429	--	7.01	--	J	--	13.1	--	U	--
23-0328 Dup @1000x	Duplicate | RPD	U	N/A	0.44	2.53	7.17	2.3	J	N/A	13.3	1.5	U	N/A

Sample Spike Results - At IC Workstation

RPL Number	Sample ID	F		Cl		NO_2		SO_4		NO_3		PO_4	
		µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec
23-0328 @100x	Sample	0.039	--	4.55	--	OvrRng	--	3.07	--	OvrRng	--	1.04	--
23-0328 @100x +k	AS Sample	0.778	99%	6.6	109%	OvrRng	N/A	8.79	102%	OvrRng	N/A	6.1	90%
23-0328 @1000x	Sample	U	--	0.4	--	7.01	--	0.29	--	13.1	--	U	--
23-0328 @1000x +k	AS Sample	0.767	102%	2.4	103%	10.8	101%	5.84	99%	17.2	109%	5.44	97%

LCS/Blank Spike Results

Run ID	Sample ID	F		Cl		NO_2		SO_4		NO_3		PO_4	
		µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec	µg/g	%Rec
23-0328 PB @1x	Sample	U	--	0.046	--	U	--	U	--	U	--	U	--
23-0328 BS @1x	BS Sample	2.7	100.0	6.8	98.4	13.5	98.4	20.8	101.1	13.9	101.3	20.2	98.2

Run ID	F	Cl	NO_2	SO_4	NO_3	PO_4
	Rec %	Rec %	Rec %	Rec %	Rec %	Rec %
LCS 3/29/2023 1:11:38 PM	102.8	100.5	105.9	99.2	99.2	102.0

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
O.R. = Over-range

Project Number: 79156
Charge Code: NN2052
ASR Number: 1672
Client: A. Westesen
Total Samples: 1 liquid

	RPL Numbers	Client IDs
Sample(s)	23-0328	TI139-Comp-Eff

Analysis Procedure	RPG-CMC-386 Rev. 1, "Carbon Measured in Solids, Sludge, and Liquid Matrices"
Prep Procedure	None
Analyst	C. Perez
Analysis Date	June 6, 2023
CCV Standards	TIC/TOC CMS # 590751 and 590871
BS/LCS/MS Standards	TIC/TOC CMS # 590264 and Agilent Lot: 0006672667
Excel Data File	ASR-1740 and ASR-1672 Data Workup TIC-TOC.xlsx
M&TE Numbers	Carbon System (WD36639, RPL/701)
	Balance : Sartorius R200D, S/N 30809774
All Analysis Records	5015_06-06-2023-075031

Christian Perez
Digitally signed by Christian Perez
Date: 2023.06.26 08:34:06 -07'00'

Prepared By Date

Steven Baum
Digitally signed by Steven Baum
Date: 2023.06.27 11:06:02 -07'00'

Reviewed By Date

Table 1: TIC/TOC Results for ASR 1672 Westesen

TIC in Sample **23-0328** (mg C/L):	5389
TIC in Sample **23-0328 Duplicate** (mg C/L):	6097
TOC in Sample **23-0328** (mg C/L):	2383
TOC in Sample **23-0328 Duplicate** (mg C/L):	2448
23-0328 TIC RPD:	12%
23-0328 TOC RPD:	3%

Sample Analysis/Results Discussion

One liquid sample was submitted under Analytical Service Request (ASR) 1672 total organic carbon analysis and total inorganic analysis. The sample analyzed was at the dilution factor of 7.97. The analysis was performed by the hot persulfate wet oxidation method, with the results summarized in Table 1. The TIC is determined first by acidifying with heated sulfuric acid, converting inorganic carbonates to CO_2 (i.e., TIC analysis), then the persulfate solids and silver-catalyst solution are added, and the remaining organic carbon converted to CO_2 (i.e., TOC analysis). The analyses were performed following procedure RPG-CMC-386, Rev. 1, *Carbon Analyses in Solids, Sludge and Liquid Matrices*.

The sample was analyzed with one duplicate for each TIC and TOC. An analytical spike was also run for TIC and TOC on the sample. 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 mg C/L 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 duplicate for the 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 a blank is run after ICV/CCV. The blanks must be <EQL. The blanks run in the batch are all <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. The two TIC ICVs the results were 100% and 100% recovery. The two TOC ICVs the results were 94% and 95% recovery. Both TIC/TOC met the range of 90-110% recovery. The closing CCV results were 100% for TIC and 96% for TOC.

Laboratory Control Sample/Blank Spike: One TIC/TOC LCS/BS was analyzed. The TIC LCS/BS result was 99% recovery, meeting the acceptance criteria of 75% to 125%. The TOC LCS/BS result was 95% 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 23-0328 TIC was 12% RPD. Sample 23-0328 TOC was 3% RPD. Both TIC/TOC % RPD meet the acceptance criteria of ≤20%.

Analytical Spike (AS): The accuracy of the carbon measurements can be estimated by the recovery from the AS. The results for the analytical spike for the TIC is 97.9% recovery. The results for the analytical spike for the TOC is 95.7% recovery. The AS recovery for the TIC/TOC results meets the acceptance criterion of 75% to 125%.

Deviation from Procedure: No deviations from the procedure. The sample was run on the same batch as ASR-1740 Bauman.

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 volume 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.

Appendix D – Batch Contact Results

Table D.1 provides the experimental results used to produce the AP-105 Cs distribution coefficient (K_d) curves and isotherms at four contact temperatures (Figure 4.1 and Figure 4.2 in the main body of this report). The dry crystalline silicotitanate (CST) masses were based on F-factors of 0.918 and 0.881 for 7 M Na AP-105 and 5.5 M Na AP-105, respectively at the nominal 105 °C drying temperature.

Table D.1. AP-105 (5.5 and 7 M Na) Tank Waste Isotherm Data

Sample ID	Dry CST Mass (g)	AP-105 Vol. (mL)	Initial Cs Conc. (M)	Equil. Cs Conc. (M)	K_d (mL/g)	Q (mmoles Cs/g)
13.1 °C						
TI140-S1-13	0.0741	14.9098	1.15E-4	1.64E-05	1222	1.99E-02
TI140-S1-13-d	0.0741	14.8880	1.15E-4	1.71E-05	1162	1.97E-02
TI140-S2-13	0.0743	14.8563	3.26E-4	4.77E-05	1160	5.57E-02
TI140-S2-13-d	0.0749	14.9160	3.26E-4	4.53E-05	1238	5.60E-02
TI140-S3-13	0.0748	14.8956	8.46E-4	1.22E-04	1172	1.44E-01
TI140-S3-13-d	0.0753	14.8689	8.46E-4	1.15E-04	1258	1.44E-01
TI140-S4-13	0.0743	14.8356	1.72E-2	1.34E-02	56	7.52E-01
TI140-S4-13-d	0.0762	14.8271	1.72E-2	1.36E-02	51	6.96E-01
15.3 °C						
TI140-S1-16	0.0750	14.9441	1.15E-4	1.74E-05	1120	1.95E-02
TI140-S1-16-d	0.0758	14.9473	1.15E-4	1.77E-05	1090	1.92E-02
TI140-S2-16	0.0765	14.9419	3.26E-4	4.59E-05	1207	5.47E-02
TI140-S2-16-d	0.0767	14.9460	3.26E-4	4.54E-05	1209	5.47E-02
TI140-S3-16	0.0743	14.9331	8.46E-4	1.30E-04	1116	1.44E-01
TI140-S3-16-d	0.0753	14.8961	8.46E-4	1.29E-04	1098	1.42E-01
TI140-S4-16	0.0750	14.8900	1.72E-2	1.33E-02	59	7.76E-01
TI140-S4-16-d	0.0761	14.9098	1.72E-2	1.33E-02	57	7.49E-01
TI140-S5-16	0.0715	14.9797	1.25E-4	1.53E-05	1495	2.31E-02
TI140-S5-16d	0.0712	14.9898	1.25E-4	1.53E-05	1514	2.32E-02
TI140-S6-16	0.0714	14.9495	3.34E-4	4.40E-05	1377	6.06E-02
TI140-S6-16d	0.0714	15.0372	3.34E-4	4.70E-05	1277	6.04E-02
24.9 °C						
TI140-S1-21	0.0751	14.8945	1.15E-4	2.58E-05	701	1.77E-02
TI140-S1-21-d	0.0746	14.8981	1.15E-4	2.72E-05	649	1.76E-02
TI140-S2-21	0.0749	14.9029	3.26E-4	7.03E-05	727	5.09E-02
TI140-S2-21-d	0.0748	14.8516	3.26E-4	6.91E-05	746	5.11E-02
TI140-S3-21	0.0742	14.8735	8.46E-4	1.82E-04	750	1.33E-01
TI140-S3-21-d	0.0749	14.8550	8.46E-4	1.76E-04	777	1.33E-01
TI140-S4-21	0.0751	14.8575	1.72E-2	1.33E-02	57	7.59E-01
TI140-S4-21-d	0.0742	14.8219	1.72E-2	1.39E-02	47	6.51E-01
35.2 °C						
TI140-S1-35	0.0759	14.9643	1.15E-4	3.41E-05	470	1.60E-02
TI140-S1-35-d	0.0740	14.9806	1.15E-4	3.45E-05	477	1.63E-02
TI140-S2-35	0.0754	14.9501	3.26E-4	9.50E-05	486	4.58E-02
TI140-S2-35-d	0.0744	14.8900	3.26E-4	9.80E-05	465	4.56E-02
TI140-S3-35	0.0750	14.9376	8.46E-4	2.43E-04	495	1.20E-01
TI140-S3-35-d	0.0765	14.8185	8.46E-4	2.36E-04	498	1.18E-01
TI140-S4-35	0.0748	14.8565	1.72E-2	1.35E-02	54	7.27E-01
TI140-S4-35-d	0.0753	14.5973	1.72E-2	1.34E-02	55	7.35E-01

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