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# Reduced Temperature Cesium Removal from AP-107 Using Crystalline Silicotitanate

September 2021

AM Westesen
SK Fiskum
AM Carney
EL Campbell
TT Trang-Le
RA Peterson

U.S. DEPARTMENT OF
ENERGY

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

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# Summary

The Tank Side Cesium Removal (TSCR) system is currently being constructed to process Hanford tank waste supernates for vitrification. TSCR incorporates a filtration system and cesium (Cs) removal system using columns filled with crystalline silicotitanate (CST) ion exchanger, produced by Honeywell UOP, LLC (product IONSIV™ R9140-B).

Laboratory-scale ion exchange processing using TSCR prototypic unit operations continues to contribute toward Washington River Protection Solutions establishing accurate process flowsheets for the individual feed campaigns planned for TSCR. The Test Platform established at the Pacific Northwest National Laboratory Shielded Analytical Laboratory has been used to conduct laboratory-scale unit operation process steps on several Hanford tank wastes at ambient temperature.[1,2,3,4] This report describes the small-scale ion exchange testing with 8.0 L of filtered supernate from tank 241-AP-107 (AP-107) at 16°C (62°F) to demonstrate processing at temperature conditions that are more prototypic of what the TSCR system could experience during colder seasons of the year.

One of the waste acceptance criteria (WAC) for the Hanford Waste Treatment and Immobilization Plant (WTP) Low-Activity Waste Facility is that the waste must contain less than $3.18 \times 10^{-5}$ Ci $^{137}$Cs per mole of Na.[5] For the AP-107 tank waste to meet this criterion, only 0.114% of the influent $^{137}$Cs concentration may be delivered to the WTP; this requires a Cs decontamination factor of 878. Prototypic TSCR operations are intended to use a lead-lag column configuration until the lag column reaches the WAC limit, then a polish column will be brought online for a lead-lag-polish column configuration. However, for the testing reported herein, the lag column did not reach the WAC limit until all the feed had been processed, so no polish column was used. Flowrate was adjusted to match the CST contact time expected for the full-scale operation, i.e., matched bed volumes per hour (BV/h) flowrate. The feed was processed downflow through the lead column, then through the lag column at an average of 1.90 BV/h until the entire available AP-107 feed was processed. The Cs-decontaminated product was retained for vitrification testing (to be reported separately).

The lead column only reached 22% Cs breakthrough after processing 799 BVs of feed; the 50% Cs breakthrough was extrapolated to occur at ~1100 BVs. This extrapolated 50% Cs breakthrough value was lower than the batch contact estimate (1255 BVs[6]) by 11%. Given the extrapolation from column processing and the overall measurement uncertainties, the agreement within 11% was considered

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[1] Rovira AM, SK Fiskum, HA Colburn, JR Allred, MR Smoot, and RA Peterson. 2018. *Cesium Ion Exchange Testing Using Crystalline Silicotitanate with Hanford Tank Waste 241-AP-107*. PNNL-27706; RPT-DFTP-011, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

[2] Rovira AM, SK Fiskum, JR Allred, JGH Geeting, HA Colburn, AM Carney, TT Trang-Le, and RA Peterson. 2019. *Dead-End Filtration and Crystalline Silicotitanate Cesium Ion Exchange with Hanford Tank Waste AW-102*. PNNL-28783, Rev. 0; RPT-TCT-003, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

[3] Fiskum SK, AM Rovira, HA Colburn, AM Carney, and RA Peterson. 2019. *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.

[4] Fiskum SK, AM Westesen, AM Carney, TT Trang-Le, and RA Peterson. 2021. *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.

[5] 24590-WTP-ICD-MG-01-030, Rev. 0. 2015. *ICD 30 – Interface Control Document for Direct LAW Feed*. Bechtel National, Inc. (River Protection Project Waste Treatment Plant), Richland, Washington.

[6] Fiskum SK, AM Westesen, AM Carney, TT Trang-Le, and RA Peterson. 2021. *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.

reasonable. Testing confirmed that 200 more BVs can be processed to 50% breakthrough at 16°C than at 28°C, demonstrating improved operating performance (i.e., higher Cs capacity) at the lower temperature. The Cs effluent from the lag column reached the WAC limit after processing 799 BVs. Cs breakthrough from the lag column began at 400 BVs, reaching $1.82 \times 10^{-1}$ µCi/mL, or 0.112 % Cs breakthrough, after processing all 799 BVs of feed. Table S.1 and Figure S.1 summarize the observed column performance and relevant Cs loading characteristics.

Table S.1. AP-107 Column Performance Summary with CST at 16°C

| Column | WAC Limit Breakthrough (BVs) | Extrapolated 50% Cs Breakthrough (BVs) | $^{137}$Cs Loaded (µCi) | Cs Loaded (mg/g CST) |
|---|---|---|---|---|
| Lead | 201 | 1100[(a)] | 1.22E+6 | 7.08 |
| Lag | 791 | NA | 7.56E+4 | 0.44 |

(a) Extracted value.
BV = bed volume, 10.0 mL
The time-weighted average flowrate was 1.90 BV/h.

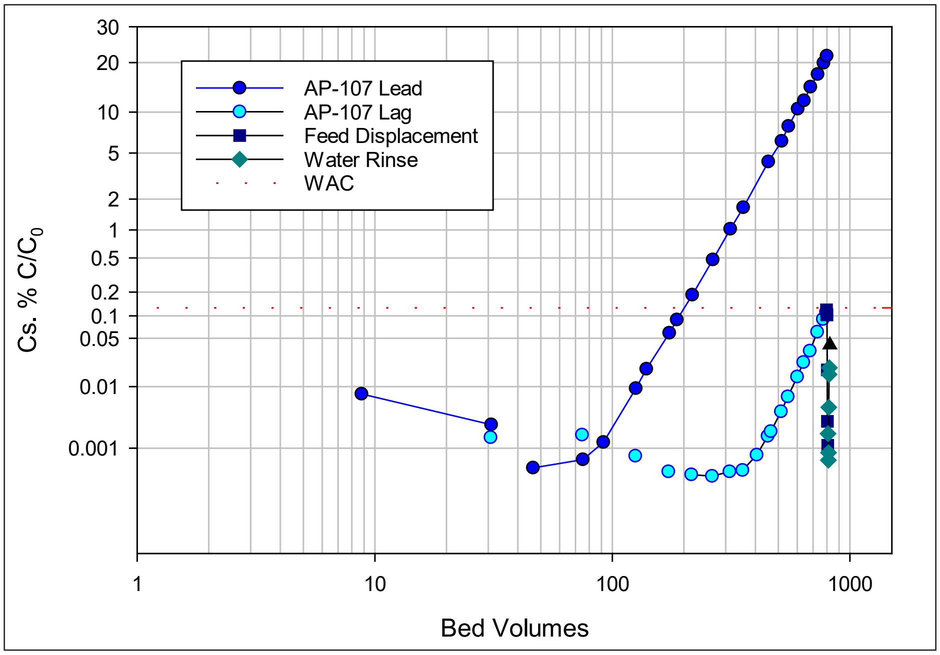

Figure S.1. Lead and Lag Column Cs Load Profiles for AP-107 at 16°C

The AP-107 composite feed and composite effluent were characterized to understand the fractionation of selected metals and radionuclides. Concentrations and recoveries of the selected analytes are summarized in Table ES.2; those with low recovery were assumed to be adsorbed onto CST. Lead (Pb), barium (Ba), and strontium (Sr) were detected in the feed (with concentration errors likely to exceed 15%) but were below the method detection limit (MDL) in the effluent; this was indicative of uptake by the CST. In addition to Cs removal, measurable fractions of cadmium (Cd), iron (Fe), $^{237}$Np, $^{238}$Pu, and $^{239+240}$Pu also partitioned to the CST.

Table S.2. Recoveries of Analytes of Interest in the AP-107 Effluent

| | Analyte | Feed Concentration (M) | Effluent Concentration (M) | Fraction in Effluent |
|---|---|---|---|---|
| Metals / Non-metals | Al | 3.78E-01 | 3.74E-01 | 98% |
| | Ba | [4.5E-06] | <8.2E-07 | -- |
| | Ca | [3.7E-04] | 2.41E-04 | -- |
| | Cd | 7.23E-05 | 5.96E-05 | 81% |
| | Fe | 3.38E-04 | 2.79E-04 | 82% |
| | K | 1.01E-01 | 9.54E-02 | 94% |
| | Na | 6.44E+00 | 6.20E+00 | 95% |
| | Nb | <7.8E-06 | [2.1E-05] | -- |
| | P | 2.87E-02 | 2.71E-02 | 93% |
| | Pb | [6.3E-05] | <4.8E-05 | -- |
| | S | 7.21E-02 | 6.94E-02 | 95% |
| | Sr | [1.1E-06] | <8.7E-07 | -- |
| | Th | <1.3E-05 | <1.3E-05 | -- |
| | U | <6.9E-05 | [8.4E-05] | -- |
| | Zn | [1.5E-04] | [3.1E-05] | -- |
| | Zr | <5.8E-06 | [2.1E-05] | -- |
| | Analyte | Feed Concentration (µCi/mL) | Effluent Concentration (µCi/mL) | Fraction in Effluent |
| Radioisotopes | $^{137}$Cs | 1.54E+02 | 2.17E-02 | 0.014% |
| | $^{237}$Np | 4.59E-05 | 3.70E-05 | 80% |
| | $^{238}$Pu | 8.67E-05 | 6.12E-05 | 70% |
| | $^{239+240}$Pu | 7.39E-04 | 4.98E-04 | 67% |

Notes:
"<" values were < MDL, sample-specific MDL provided in Appendix C.
"--" indicates effluent recovery could not be calculated.
Values in brackets [ ] were ≥ MDL but < EQL, with errors likely to exceed ±15%.
EQL = estimated quantitation limit.

# Acknowledgements

The authors thank Shielded Facility Operations staff Hollan Brown and Victor Aguilar for hot cell operations (ion exchange column system setup, processing, and sample removal). We thank the Analytical Support Operations (ASO) staff Jenn Carter, Chuck Soderquist, Sam Morrison, Lori Darnell, and Karl Pool 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 Bill Dey 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

| | |
|---|---|
| AEA | alpha energy analysis |
| ASO | Analytical Support Operations |
| ASR | Analytical Service Request |
| BV | bed volume |
| CST | crystalline silicotitanate |
| DF | decontamination factor |
| EQL | estimated quantitation limit |
| erf | error function |
| FD | feed displacement |
| GEA | gamma energy analysis |
| ICP-OES | inductively coupled plasma optical emission spectroscopy |
| ID | identification |
| ILAW | immobilized low-activity waste |
| LAW | low-activity waste |
| MDL | method detection limit |
| PNNL | Pacific Northwest National Laboratory |
| QA | quality assurance |
| R&D | research and development |
| RPD | relative percent difference |
| RPL | Radiochemical Processing Laboratory |
| RSD | relative standard deviation |
| SV | system volume |
| 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 |

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# 1.0 Introduction

The initial production of immobilized low-activity waste (ILAW) 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 Hanford Waste Treatment and Immobilization Plant (WTP) Low-Activity Waste (LAW) Facility. Decanted tank waste supernatant will be pretreated using TSCR to meet the WTP LAW Facility waste acceptance criteria (WAC).[7] The TSCR unit uses a filter to remove entrained solids and then a non-elutable crystalline silicotitanate (CST) ion exchanger capable of retaining up to 141.6 kCi of $^{137}$Cs on each column within the unit.[8] TSCR operation will pause for replacement of ion exchange columns when the output of the last column in the multiple-column sequence reaches the WTP LAW WAC limit. Spent ion exchange columns will be stored on a nearby concrete pad until the ion exchange media (and retained $^{137}$Cs) can be processed through WTP as high-level waste.

The TSCR WAC require that feed temperatures be less than 35°C (95°F). The average bulk supernate temperatures of the majority of double-shell tanks in the Hanford tank farms are currently below 35°C and average around 16°C (62°F) during the spring and winter months. Previous laboratory-scale ion exchange processing using TSCR prototypic unit operations has been conducted at ambient laboratory (hot cell) temperatures of ~28°C (Fiskum et al. 2019a,b). With a decrease in temperature, CST capacity for Cs increases while the kinetics of the exchange decrease. The primary objective of the work described herein was to test the impact of 16°C operating temperature (to represent seasonal changes in tank temperature) on ion exchange processes with AP-107 tank waste.

Cesium removal using CST, product IONSIV™ R9140-B, manufactured by Honeywell UOP, LLC (Des Plaines, IL), was conducted at 16°C in dual (lead-lag) column processing to establish Cs load profiles. A lead-lag-polish column system was to be used after the lag column effluent reached the WAC limit; however, the increased capacity of the CST at the lower operating temperature allowed for all the feed to be processed before the lag column effluent exceeded the WTP LAW WAC limit. Additional objectives of the current study were as follows:

1. Decontaminate ~500 mL of AP-107 tank waste from $^{137}$Cs/Cs by running the tank waste through a single column containing 10.0 mL of CST, in order to conduct batch contact testing to determine the Cs load capacities at 13°C, 16°C, 21°C, and 35°C (reported separately, Fiskum et al. 2021a).
2. Compare the 16°C AP-107 Cs load profile to the previously reported AP-107 load curve conducted at 28°C (Fiskum et al. 2019a).
3. Analyze the AP-107 ion exchange feed and effluent to derive the fates of key analytes ($^{137}$Cs, $^{239+240}$Pu, $^{237}$Np, Al, Ba, Ca, Cd, Fe, K, Na, Nb, P, Pb, S, Sr, U, Zn, Zr).
4. Provide Cs-decontaminated AP-107 for vitrification (to be conducted later and addressed in a separate report).

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[7] 24590-WTP-ICD-MG-01-030, Rev. 0. 2015. *ICD 30 – Interface Control Document for Direct LAW Feed*. Bechtel National, Inc. (River Protection Project Waste Treatment Plant), Richland, Washington.

[8] RPP-RPT-61030, Rev. 1. 2019. *TSCR Process Operation Description*, AVANTech Incorporated, Richland, Washington.

The efficacy of loading higher amounts of Cs onto the lead column CST while maintaining a product below the WTP LAW WAC limit from the polish column was of prime interest to support the evolving Washington River Protection Solutions (WRPS) TSCR design. The design of the tests reported herein exposed the CST to higher feed volumes through the individual column beds, allowing for a more representative assessment of the fractionations of analytes of interest.

WRPS funded Pacific Northwest National Laboratory (PNNL) to conduct testing with AP-107 tank waste under the statement of work presented in Requisition #340584, “FY 2021 Radioactive Waste Test Platform,” Rev. 0, dated November 9, 2020.

# 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-107 tank waste, and column ion exchange conditions. All testing was conducted in accordance with a test plan prepared by PNNL and approved by WRPS.[9]

## 3.1 CST Media

WRPS purchased ten 5-gallon buckets (149 kg total) of IONSIV™ R9140-B,[10] lot number 2002009604, material number 8056202-999, from Honeywell UOP, LLC. This CST production lot was screened by the manufacturer to achieve an 18 × 50 mesh size product. As requested by PNNL, the product was delivered to WRPS in a series of 5-gallon buckets (as opposed to a 50-gallon drum) to aid in material distribution, handling, and sampling at PNNL. The CST was transferred from WRPS to PNNL on September 20, 2018, under chain of custody. Once received, the CST was maintained at PNNL in environmentally controlled spaces. One of the 5-gallon buckets of CST was transferred to the PNNL Radiochemical Processing Laboratory (RPL). The handling and splitting of the CST in preparation for laboratory testing were previously described (Fiskum et al. 2019b). A 180-g subsample split was passed through a 30-mesh sieve (ASTM E11 specification). Of this starting mass, 61.9 g, or 34 wt%, passed through the sieve and was collected for column testing; this was similar to the 36% mass fraction achieved during AP-105 testing (Fiskum et al. 2021b). The <30-mesh CST fraction was pretreated by contacting with 200 mL of 0.1 M NaOH five successive times, during which the 0.1 M NaOH rinse solution and colloidal fines from the CST were decanted. The rinsed CST was maintained with an overburden of 0.1 M NaOH. Table 3.1 provides the physical properties of <30-mesh sieved CST (product R9140-B, Lot 2002009604) that had been washed and air dried (Westesen et al. 2020). These properties were expected to apply to the current test because CST processing was essentially identical. The CST particle number (28) across the 1.5-cm column diameter was close to the minimum ideal (≥30) defined by Helfferich (1962); this mitigated fluid channeling due to wall effects.

Table 3.1. Physical Properties of <30 Mesh, Washed CST Product R9140-B, Lot 2002009604 (Westesen et al. 2020)

| Parameter | Result | Units |
|---|---|---|
| Bulk density | 1.03 | g/mL |
| CST bed density | 1.00 | g/mL |
| Settled bed void volume | 68.2 | % |
| Cumulative particle undersize fractions[(a)] | $d_{10}$: 398<br>$d_{50}$: 541<br>$d_{90}$: 738 | microns |
| Column inner diameter | 1.5 | cm |
| Particle number across column diameter (based on $d_{50}$) | 28 | NA |
| (a) Volume basis, post-sonication | | |

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[9] Westesen AM. 2021. TP-DFTP-099, Rev. 0.0. *FY21 Cesium Ion Exchange Testing with AP-107 Tank Waste with Crystalline Silicotitanate*. Pacific Northwest National Laboratory, Richland, Washington. Not publicly available.

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

## 3.2 AP-107 Tank Waste Sample

Multiple samples (36 each at ~250 mL for a combined 8.8 L) were collected (by WRPS) in two sets from Hanford tank 241-AP-107 in November 2020. The samples were delivered to PNNL's RPL in two shipments of 16 jars each and were placed into the Shielded Analytical Laboratory hot cells. Two of the sample containers (7AP-20-16 and 7AP-20-34) were held for batch contact testing (Fiskum et al. 2021a), leaving 34 bottles to be filtered and subsequently ion exchanged at 16°C.

Analytical measurements were conducted by PNNL's Analytical Support Operations (ASO) laboratory according to an Analytical Service Request (ASR); results are provided in Table 3.2. The first and last samples from each shipment (7AP-20-16 and 7AP-20-33 from the first shipment, 7AP-20-34 and 7AP-20-51 from the second shipment) were subsampled and measured for the $^{137}$Cs concentration by gamma energy analysis (GEA) (ASR 1193). The AP-107 densities of these samples were measured at ambient temperature in-cell using 10-mL volumetric flasks. The results of the duplicate pairs agreed within 10% relative percent difference (RPD), and it was assumed that all 36 samples were essentially homogenous, within analytical uncertainty (±10% to 15%).

Table 3.2. Characterization of Samples Collected from Hanford Tank 241-AP-107 in November 2020 (ASR 1193)

| | 7AP-20-16 | 7AP-20-33 | 7AP-20-34 | 7AP-20-51 | Average | RPD, % | Analysis Method |
|---|---|---|---|---|---|---|---|
| $^{137}$Cs, µCi/mL | 160 | 151 | 177 | 168 | 164 | 6.8 | GEA |
| Density, g/mL | 1.281 | 1.272 | 1.291 | 1.289 | 1.283 | 0.67 | Volumetric flask |

The 34 jars of available AP-107 tank waste samples were composited into a series of 1.5-L polyethylene bottles and chilled to 16°C before being filtered with a media grade 5 filter, described by Allred et al. (2021). After filtration, six bottles of AP-107, containing nominally 1.3 L each, as well as ~400 mL of unfiltered feed from draining the filtration system were made available for ion exchange testing.

The densities and $^{137}$Cs concentrations of each of the bottles of AP-107 were measured. The density average was 1.271 g/mL (0.42% RSD) and the $^{137}$Cs average was 161.7 µCi/mL (4.1% RSD; reference date March 2021). Therefore, AP-107 feeds in all containers were considered uniform. The total Cs concentration was calculated from the $^{137}$Cs concentration (in terms of µg/mL with unit conversion per the specific activity) and $^{137}$Cs mass fraction from previous analysis reported in Fiskum et al. 2019a (average 19.9 wt%). The total Cs concentration in the AP-107 was 9.37 µg/mL (6.99E-05 M Cs).

## 3.3 Reduced Temperature Ion Exchange Processing

This section describes the ion exchange column system and AP-107 process conditions. The preparations and column testing were conducted in accordance with a test instruction.[11]

### 3.3.1 Ion Exchange Column System

Figure 3.1 provides a piping and instrumentation diagram of the ion exchange process system. The columns were housed in a 12-inch × 6-inch × 15-inch (W×D×H) insulated box constructed of Styrofoam and covered with aluminized mylar film. The walls and lid were removable, allowing easy access to the columns. Heat exchange was conducted with water from a chilled circulating bath flowing in serpentine fashion through copper tubing on inside front and back panels. The temperature within the insulated box was controlled by adjusting the water bath temperature; internal temperature was monitored with a thermocouple seated inside a vial of water adjacent to the columns.

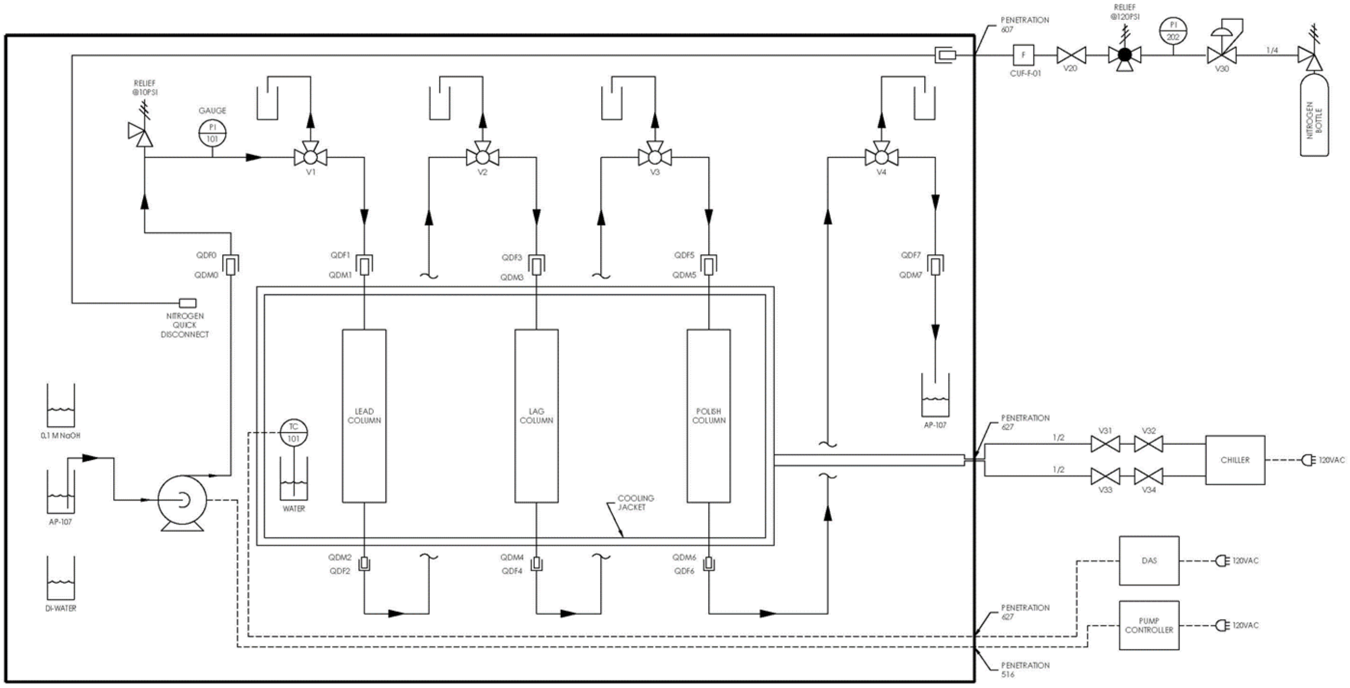

Figure 3.1. Chilled Ion Exchange Piping and Instrumentation Diagram

Figure 3.2 shows photographs of the system heat exchanger before installation in the hot cell. The heat exchanger housed all three columns. A 10-inch × 3-inch front window was installed for visual monitoring of the columns during processing. Tubing preceding each column was coiled within the heat exchanger to ensure the temperature of the feed entering the columns was within the operating range of 16°C ± 2.2°C.

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[11] Westesen AM. 2021. *Reduced Temperature Cesium Removal from AP-107 Using Crystalline Silicotitanate in a Two and Three-Column Format.* TI-DFTP-100. Pacific Northwest National Laboratory, Richland, Washington. Not publicly available. Implemented March 2021.

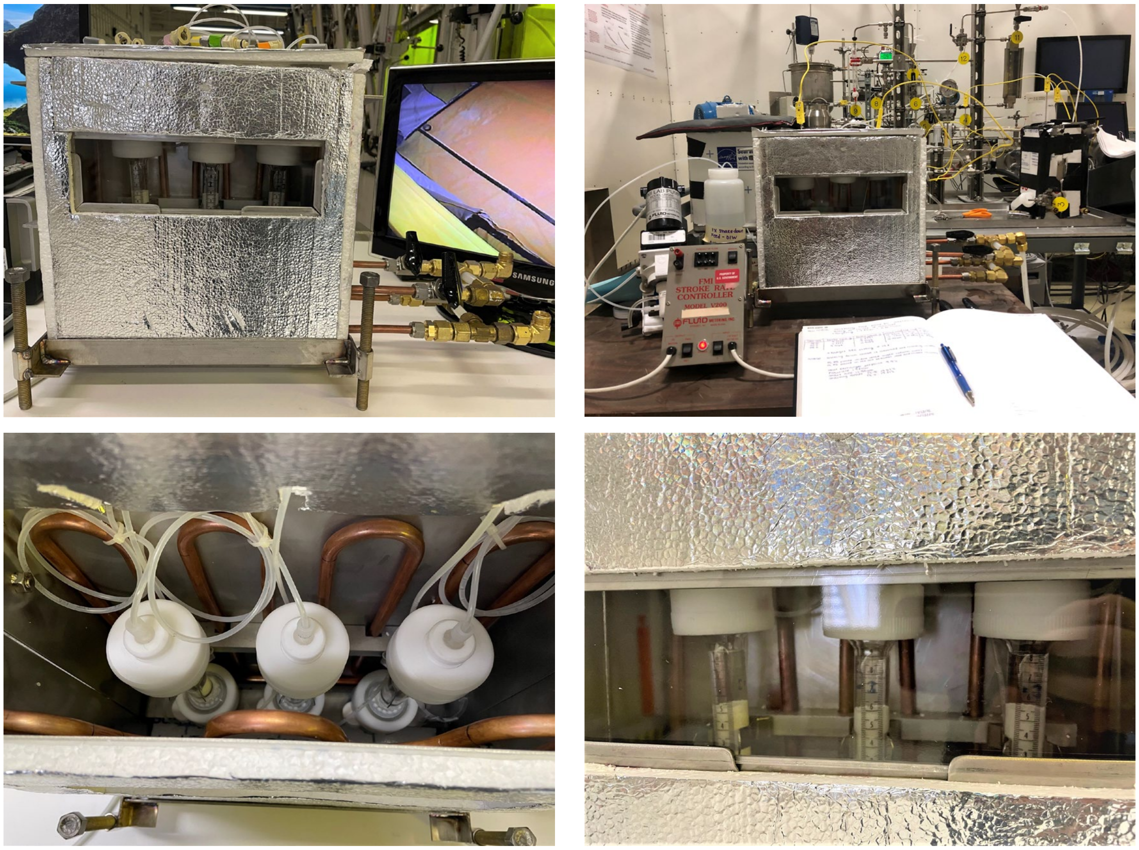

Figure 3.2. Photographs of Insulated Box and Ion Exchange System Outside of the Hot Cell

Flow through the system was controlled with a Fluid Metering Inc. positive displacement pump. Fluid was pumped past an Ashcroft pressure gage and a Swagelok pressure relief valve with a 10-psi trigger point. The 1/8-inch outside diameter / 1/16-inch inside diameter polyethylene tubing was purchased from Polyconn (Plymouth, MN). The 1/8-inch outside diameter / 1/16-inch inside diameter stainless steel tubing was used in conjunction with the valve manifold. Valved quick disconnects (QDM/QDF in Figure 3.1) were purchased from Cole Parmer (Vernon Hills, IL). Use of the quick disconnects enabled easy disassembly and re-assembly for installation in the hot cell. Multiple quick disconnects were used such that columns could be isolated (required for system install and reserved polish column) or replaced as needed. Also, recovery from upset conditions could be accommodated by allowing access to a column either downflow or upflow.

Chromaflex® column assemblies were custom ordered from Kimble Chase (www.kimble-chase.com). Each column assembly included the column plus the standard top and bottom end fittings. Each column was made of borosilicate glass; the straight portion of the column was 9 cm tall with an inside diameter of 1.5 cm (corresponding to a CST volume of 1.77 mL/cm). The 1.5-cm inside diameter columns are not commercial-off-the-shelf items. The columns are flared at each end to support the off-the-shelf column fittings and tubing connectors that were composed of polytetrafluoroethylene. 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. With a rubber O-ring, the bed support was snug-fitted into place in the column (as previously described by Fiskum et al. 2019b). 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 that had been used previously for AP-105DF (diluted and filtered supernate from Hanford waste tank 241-AP-105) processing reported in Fiskum et al. (2021b). 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 valve 4 would have been used to collect polish column samples had the polish column been needed. The gross AP-107 effluent, feed displacement (FD), water rinse, and flushed fluid were collected at the effluent line.

Three 10.0-mL aliquots of settled CST (pretreated, <30 mesh) were measured using a graduated cylinder and then quantitatively transferred, one aliquot each, to the three columns. 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 10.0 mL; each of the three columns contained 10.0 mL CST. The settled CST bed heights in the columns were nominally 5.5 cm. This small column bed height corresponded to 2.4% of the full-height TSCR column (234 cm or 92 inches) and the BV corresponded to 0.0017% of the full-scale column (596 L) (Siewert 2019).

The system was planned to run in a lead-lag configuration until the lag column reached the WAC limit, then the polishing column would come on-line. However, the lag column did not reach the WAC limit until the very end of feed processing, so the lead-lag-polish configuration was not used during this testing and feed was only processed in a lead-lag configuration.

The entire fluid-filled volume of the assembly was calculated for the two-column system at ~48 mL, and for the three-column system at ~68 mL. The bed void volume was assigned 66% (Westesen et al. 2020). Therefore, each CST bed held 6.6 mL of fluid and the CST only comprised ~30% of the fluid-filled bed volume. The TSCR system platform may have a much larger fluid fraction associated with the CST bed. The fluid-filled mixing space above each CST bed ranged from 3.5 to 4.5 mL. The fluid mixing volume below each CST bed ranged from 3.0 to 4.2 mL. Thus, ~60% of the total fluid holdup volume was unavoidably associated with the geometry of the two-column system. These scales of fluid mixing volume fractions are not likely to be representative of plant-scale operations. Figure 3.3 is a photograph of the chilled ion exchange system in-cell after processing with the AP-107 was completed. Not included in the picture is the effluent bottle.

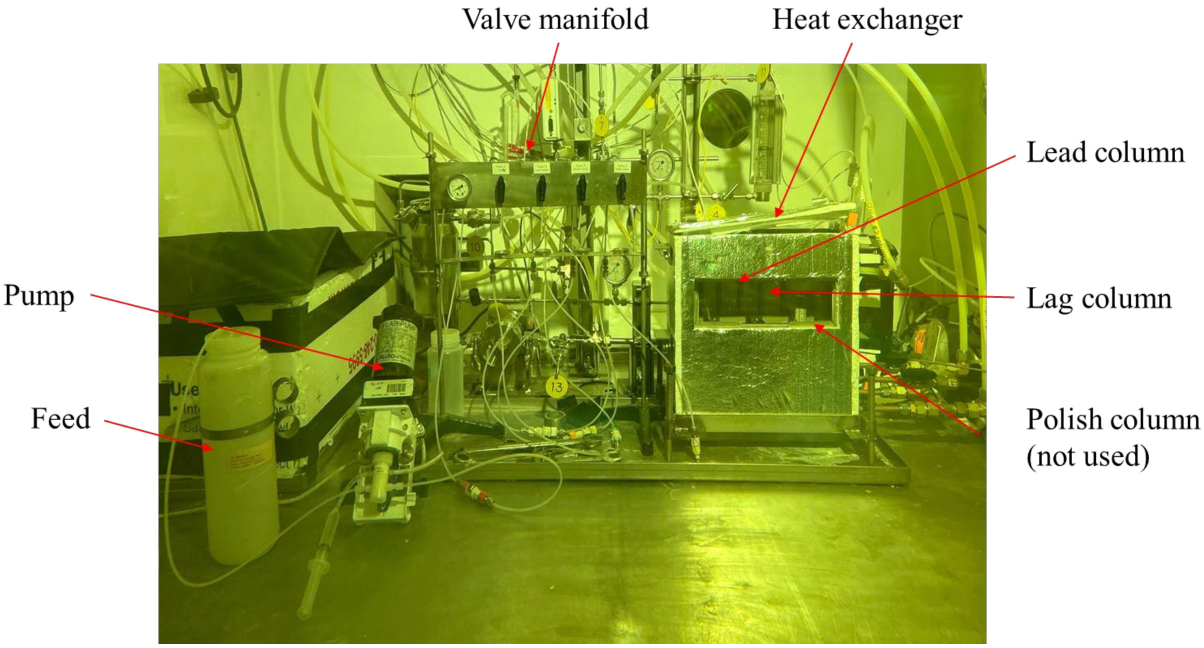

Figure 3.3. Ion Exchange Assembly in the Hot Cell Post Processing

### 3.3.2 AP-107 Tank Waste Process Conditions

Once the ion exchange assembly was installed in the hot cell, a flow of 0.1 M NaOH was used to verify system integrity and calibrate the pump. The AP-107 contained in various 1.5-L polyethylene containers from the filtration process (Allred et al. 2021) was used as the ion exchange feed. To provide stability, bottles were positioned in a bottle stand just before the feed line was inserted. When the contents in a feed bottle decreased to ~200 mL, the next bottle in line was moved to the feed position and the residual contents were poured into the new feed bottle. The AP-107 feed was processed downflow through the ion exchange media beds, lead to lag. Effluent was collected in ~1.0- to 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.

After the AP-107 processing (also “loading” in subsequent discussion) was completed, 11 BVs of 0.1 M NaOH FD followed by 11 BVs of deionized water were passed downflow through the system to rinse residual feed out of the columns and process lines. The 11 BVs was equivalent to ~1.7 times the fluid-filled system volume (SV).

Figure 3.4 provides the temperature profile of the AP-107 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 duration of testing with min/max temperatures of 14.0°C and 16.9°C, respectively. Test parameters, including process volumes, flowrates, and CST contact times, are summarized in Table 3.3. The pump head stroke length was close to the minimum at which it could be set. The stroke rate was toggled throughout testing to maintain the flowrate to the targeted 1.90 BV/h. Figure 3.4 shows the achieved flowrate as a function of time.

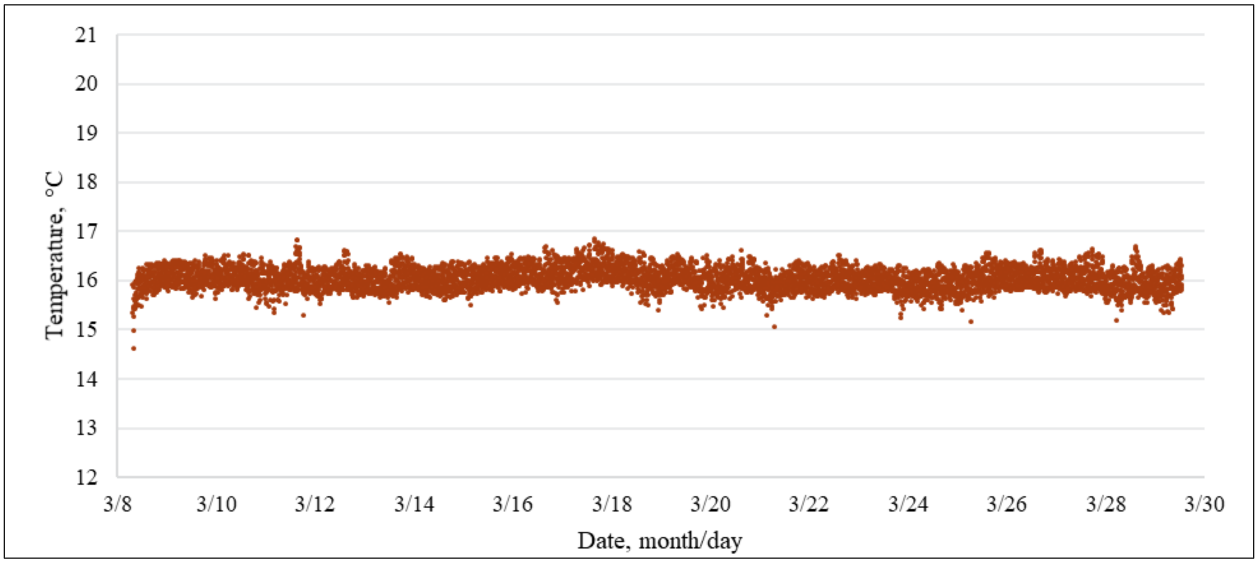

Figure 3.4 AP-107 Column Temperature during Testing

Table 3.3. Experimental Conditions for AP-107 Column Processing at 16°C, March 8 to March 29, 2021

| | | Volume | | | Flowrate | | Duration |
|---|---|---|---|---|---|---|---|
| Process Step | Solution | (BV) | (SV) | (mL) | (BV/h) | (mL/min) | (h) |
| Loading lead column | AP-107 | 798.9 | NA | 7989 | 1.90 | 0.316 | 422 |
| Loading lag column[(a)] | AP-107 | 794.4 | NA | 7944 | 1.90 | 0.316 | 422 |
| Feed displacement | 0.1 M NaOH | 11.9 | 2.48 | 119 | 3.01 | 0.502 | 4.0 |
| Water rinse | DI water | 12.6 | 2.63 | 126 | 3.28 | 0.547 | 3.9 |
| Flush with compressed air[(b)] | NA | 3.4 | 0.71 | 34.3 | 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 March 29, 2021, after the system sat in static contact with water rinse for 66 h (over the weekend).

BV = bed volume (10.0 mL as measured in graduated cylinder).
DI = deionized.
SV = system volume (estimated 48 mL).
NA = not applicable.

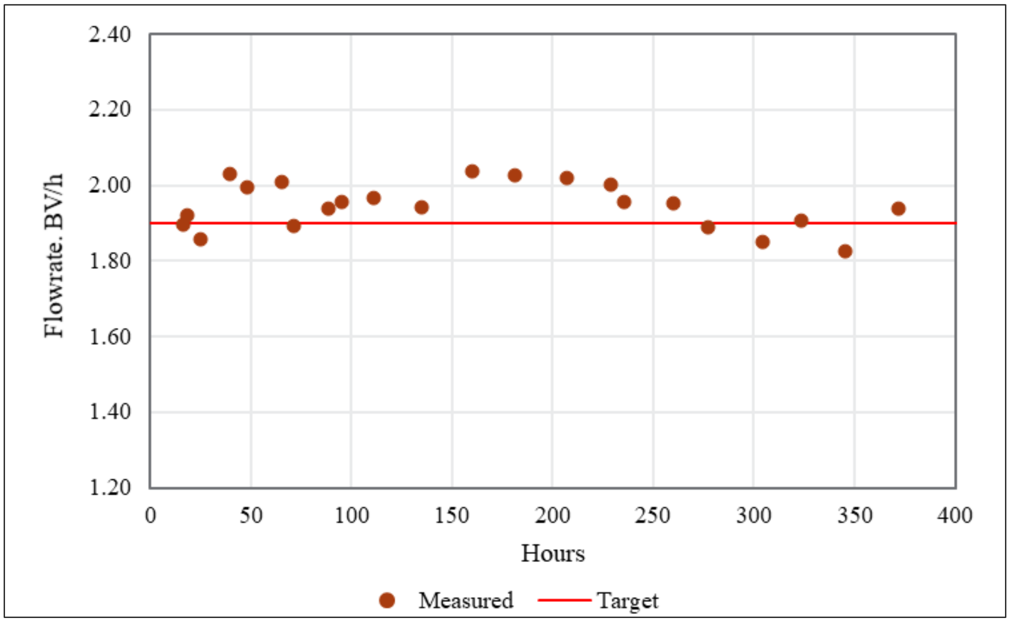

Figure 3.5. AP-107 Flowrate as a Function of Time

The total cumulative volume of AP-107 processed was 7.99 L (798.9 BVs). The AP-107 process cycle mimicked, as best as possible, the current process flow anticipated at the TSCR facility in terms of 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 point 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 lead column necessitated brief (~7-minute) interruption of flow to the lag column. Samples were collected after the first 9 BVs were processed and again at nominal 13- to 97-BV increments. Only brief (~5-min) interruptions were associated with changing the feed bottles.

The FD effluent was collected in a series of 6 vials in ~18-mL increments. 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 ~6 min. The collected volume (34.3 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).

## 3.4 Sample Analysis

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

A composite feed sample 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. Selected effluent samples from the lead column were measured for selected radionuclides and cations to assess the exchange behavior for these analytes. Table 3.4 summarizes the specific sample collections and targeted analytes along with the cross-reference to the 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 ASR. Samples were analyzed directly (no preparation) by GEA; longer count times were used to assess isotopes other than $^{137}$Cs.

Table 3.4. Analytical Scope Supporting Column Processing, ASR 1248

| Sample ID | ASO Sample ID | Analysis Scope |
|---|---|---|
| TI100-Comp-FEED | 21-0864 | GEA ($^{137}$Cs)<br>ICP-OES (Al, Ba, Ca, Cd, Fe, K, Na, Nb, P, Pb, S, Sr, U, Zn, Zr)<br>Radioanalytical ($^{237}$Np, $^{239+240}$Pu) |
| TI100-Comp-EFF | 21-0865 | |
| TI100-L-F2 | 21-1070 | |
| TI100-L-F4 | 21-1071 | |
| TI100-L-F6 | 21-1072 | |
| TI100-L-F8 | 21-1073 | |
| TI100-L-F10 | 21-1074 | |
| TI100-L-F12 | 21-1075 | |
| TI100-L-F14 | 21-1076 | |
| TI100-L-F17 | 21-1077 | |
| TI100-L-F22 | 21-1078 | |

ICP-OES = inductively coupled plasma optical emission spectroscopy

# 4.0 Results

This section discusses the Cs exchange behavior during the load, FD, water rinse, and final solution flush from the system. Raw data is provided in Appendix A.

## 4.1 Ion Exchange Processing

The AP-107 feed was processed at nominally 1.90 BV/h through the lead and lag columns. Figure 4.1 shows a linear-linear plot of the cesium load profile for feed processed through each column. The x-axis shows the BVs processed and the y-axis shows the effluent Cs concentration (C) relative to the feed concentration ($C_0$) in terms of % $C/C_0$. The $C_0$ value for $^{137}$Cs was determined to be 162 µCi/mL (average of the seven filter product bottle feeds, RSD of 4.1%). In this graphing layout, the Cs breakthrough from the lead column appeared to start at ~250 BVs and continued to 22% $C/C_0$ after processing 799 BVs when the last sample was collected from the lead column. It is obvious that the lag column Cs breakthrough performance is not discernable at this linear scale.

Figure 4.2 shows the same Cs load data provided in Figure 4.1, but with the ordinate % $C/C_0$ on a probability scale 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 4.1, the Cs breakthrough from the lead column was observed to occur around 90 BVs processed. The WAC at 0.114% $C/C_0$ is also apparent (dashed red line).[12] The WAC Cs breakthrough for the lead column occurred at 200 BVs. The lag column WAC Cs breakthrough occurred right at the end of processing ~791 BVs. The originally planned polish column was not used during this testing since the lag column did not reach the WAC limit until the very end of feed processing.

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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: $3.18\times10^{-5}$ Ci $^{137}$Cs/mole Na. At 5.6 M Na and 162 µCi $^{137}$Cs/mL in the feed, the WAC limit translates to 0.114% $C/C_0$.

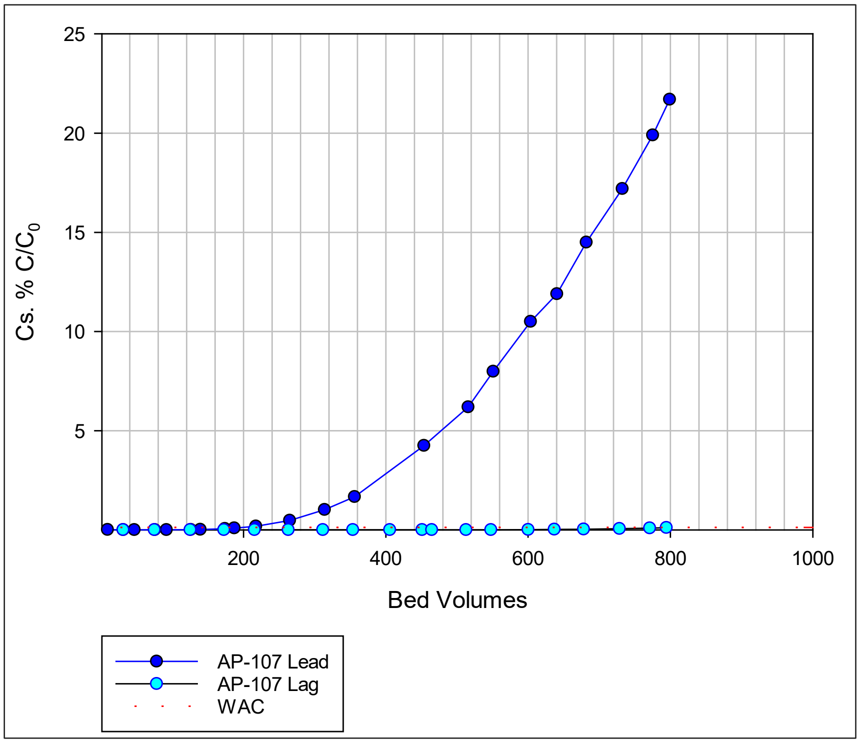

Figure 4.1. Lead and Lag Column Cs Load Profiles of AP-107 at 1.90 BV/h, Linear-Linear Plot

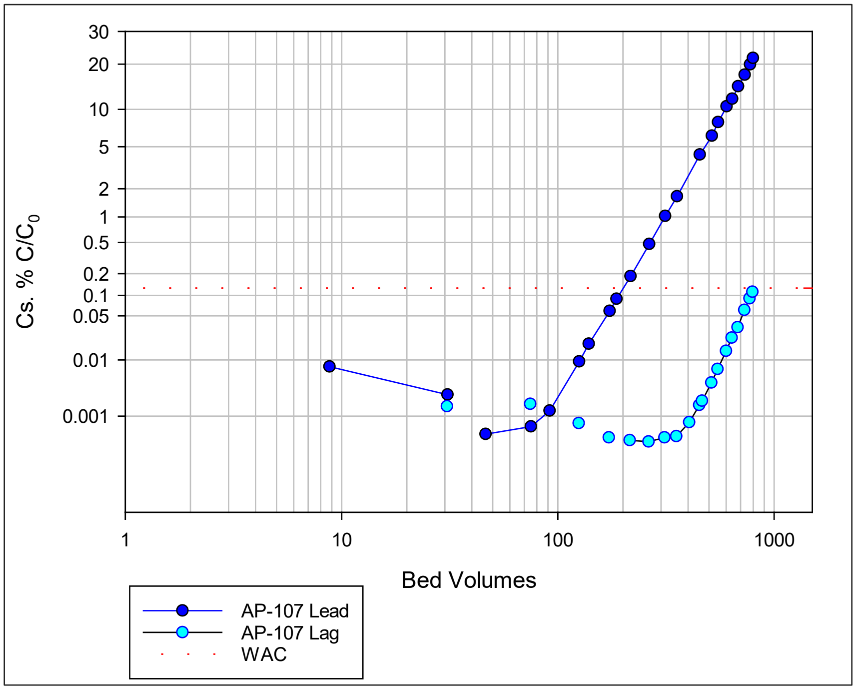

Figure 4.2 Lead and Lag Column Cs Load Profiles of AP-107 at 1.90 BV/h, Probability-Log Plot

The 50% Cs breakthrough on the 10.0-mL lead column was not reached because there was insufficient AP-107 feed available at the RPL for processing. However, the column data was evaluated to estimate the BVs to 50% breakthrough. The breakthrough curve can be estimated by the error function (erf) (Hougen and Marshall 1947; Klinkenberg 1948):

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

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 lead and lag column experimental data (see Figure 4.3 and Figure 4.4). The lead column breakthrough profile deviated below the model fit starting at ~750 BVs. This indicated non-ideal Cs loading and is consistent with what was seen with previous AP-105 testing (Fiskum et. al 2021b) and differences in capacity between batch contact testing and column testing (Fiskum et. al 2021a).

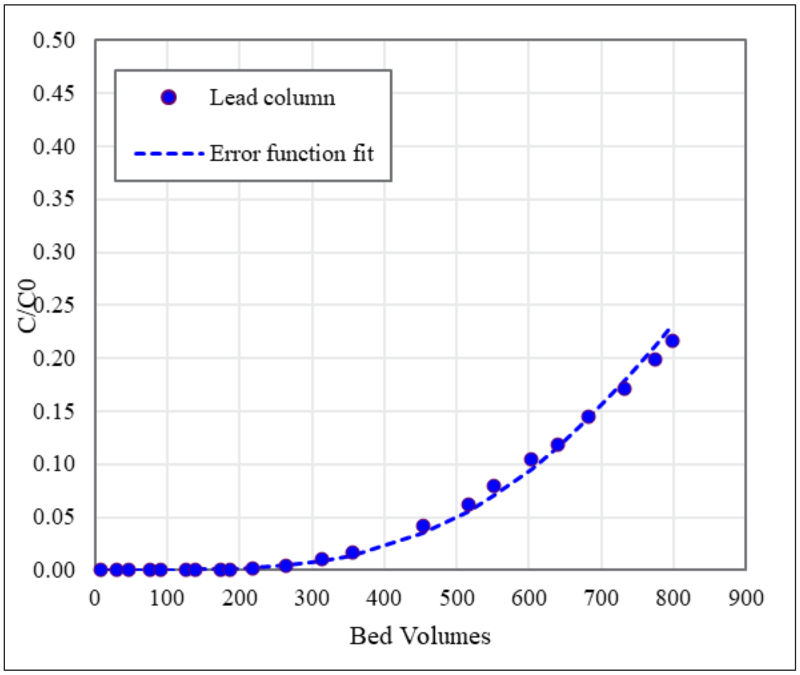

Figure 4.3 Lead Column Cs Breakthroughs with Error Function Fit

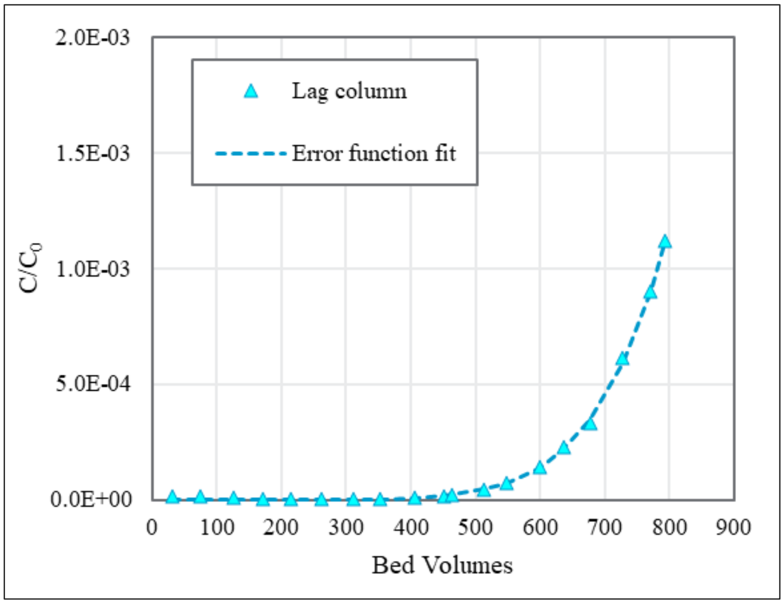

Figure 4.4 Lag Column Cs Breakthroughs with Error Function Fit

The 50% Cs breakthroughs for the lead and lag columns were estimated from the error function fit at 1087 BVs and 2271 BVs, respectively. The lead column breakthrough fit lands at ~200 BVs less than what was calculated from batch contact testing (Fiskum et al. 2021a). The difference in BVs between the error fit estimation and batch contact calculations is not seen as significant due to such a large extrapolation required from the column breakthrough. The 50% breakthrough likely occurs between 1100 and 1250 BVs and would require additional feed for column loading to better estimate. These values are nominally 40% higher than what was calculated for the AP-105 column breakthroughs (Fiskum et al. 2021b).

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 4.5). The curves were fitted to a second-order polynomial function ($R^2 = >0.99$) and the WAC limit breakthroughs were then easily calculated, resulting in the following:

- Lead column: 201 BVs
- Lag column: 791 BVs

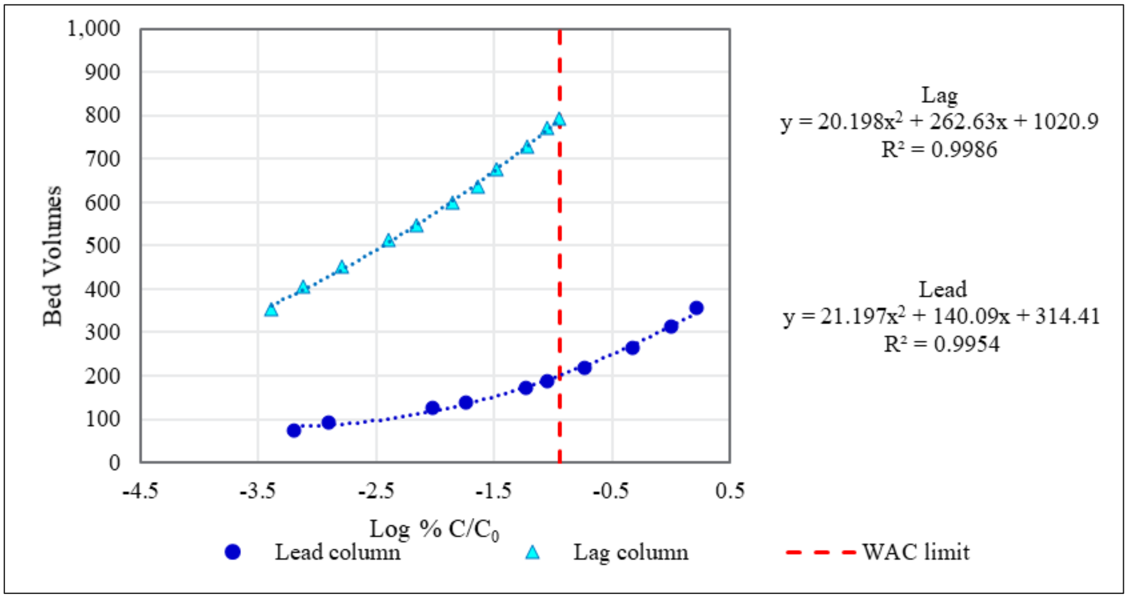

Figure 4.5. Curve Fits to Interpolate WAC Limit Breakthroughs from Lead and Lag Columns

Figure 4.6 compares the 16°C AP-107 Cs load profiles with previously tested (FY 2019) AP-107 loading conducted at 28°C (Fiskum et al. 2019a). CST Lot 2002009604 was used in both cases; however, a <25-mesh sieve fraction was used for AP-107 at 28°C and a <30-mesh sieve fraction was used for the 16°C testing. The smaller particle size used in the 16°C testing increased the kinetics of the Cs exchange; however, the decrease in temperature both slowed the kinetics and increased the capacity for Cs within the CST. This change in capacity resulted in a shift of the 50% breakthrough point to the right. This is seen when comparing the predicted 50% breakthrough points between the two tests, with the 16°C test (1087 BVs to 50% breakthrough) resulting in a nominal 200 BV increase over the testing at 28°C (900 BVs to 50% breakthrough). The lead column breakthroughs for both tests appear to reach the WAC limit after processing the same number of BVs (200). When comparing the overall slope of both lines, the steepness of the 28°C breakthrough slope is greater than that of the 16°C slope, which indicates that the temperature impact on kinetics is greater than the particle size impact. For the lag column, the reduced temperature testing shifted the load profile to the right and the WAC limit was reached at a significantly later process volume (791 BVs at 16°C vs. 590 BVs at 28°C), indicating that although the kinetics were slower, this was compensated by the increased capacity at 16°C and reduced CST particle size.

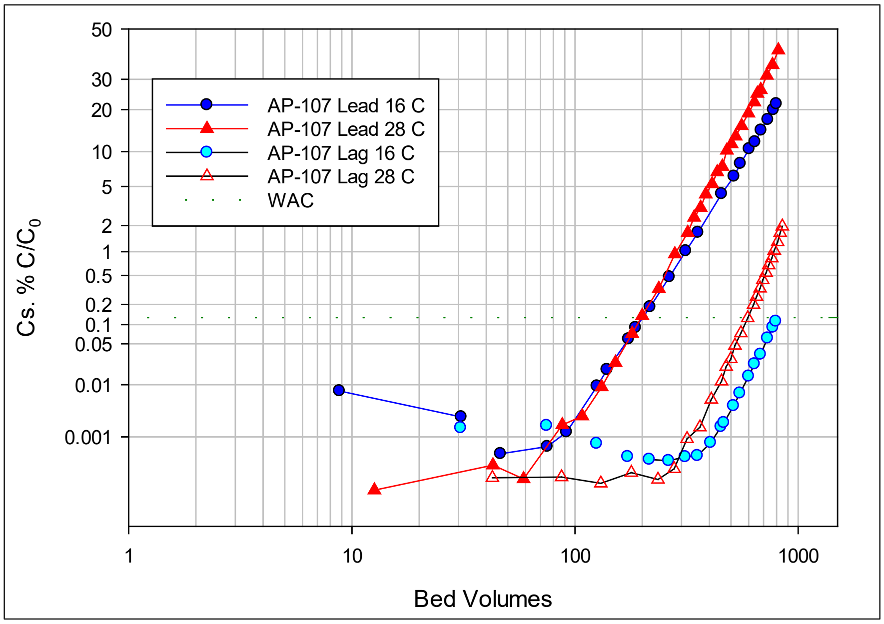

Figure 4.6. Load Profile Comparisons: AP-107 at 16°C and 28°C (Fiskum et al. 2019a), CST Lot 2002009604

## 4.2 TSCR WAC Limit

Using data from full-height simulant tests (Fiskum et al. 2019b) and the analytical model described in Section 4.1, Westesen et al. (2020) determined the impact of residence time on breakthrough by plotting, as shown in Figure 4.7, $\sqrt{BV_{x\%}}$ vs. $\sqrt{\frac{1}{Q}}$, where $BV_{x\%}$ represents the number of bed volumes to a target breakthrough and Q represents the flow rate in BVs per unit time. This demonstrates that the flowrate through the CST column (in terms of BV/h or contact time) directly influences the volume that can be processed before reaching a target breakthrough, in a linear relationship. The lines included in Figure 4.7 are based on capacity and have a slope of $\sqrt{\frac{(1-\epsilon_B)\rho_B K_d}{\varepsilon_B}}$, where $\varepsilon_B$ is the porosity of the bed, $\rho_B$ is the density of the ion exchange material, and $K_d$ represents the ion exchange capacity of the material.

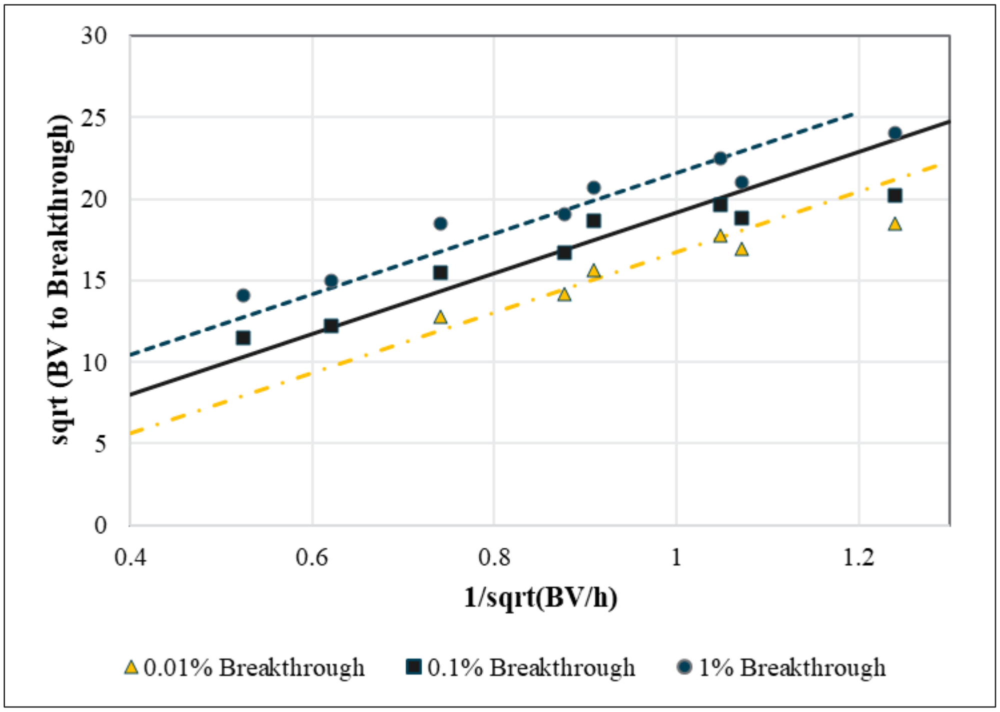

Figure 4.7. Impact of Residence Time on Breakthrough (Westesen et al. 2020)

Using this method, AP-107 results from the lead and lag columns can be evaluated to project the volume of waste that can be processed through the TSCR facility before reaching the WAC limit. Figure 4.8 plots this data alongside data for AP-105 (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. The maximum volume projection for AP-107 tank waste processed before WAC breakthrough on the polish column is 311,000 gallons. It is important to note that the lines included are not fits to the data; they are calculated slopes from the CST capacity, so a steeper slope represents a higher CST Cs capacity. Looking at the data in this manner helps to express the difference in BVs to breakthrough that was seen in the AP-107 chilled lag column. From the figure, we can see the lead column breaks through similarly, but with decreasing throughput the difference becomes more pronounced. Deviations from the theoretical model occur at throughputs below 1 SV/h. These deviations are attributed to dispersion within the column due to the low flowrate.

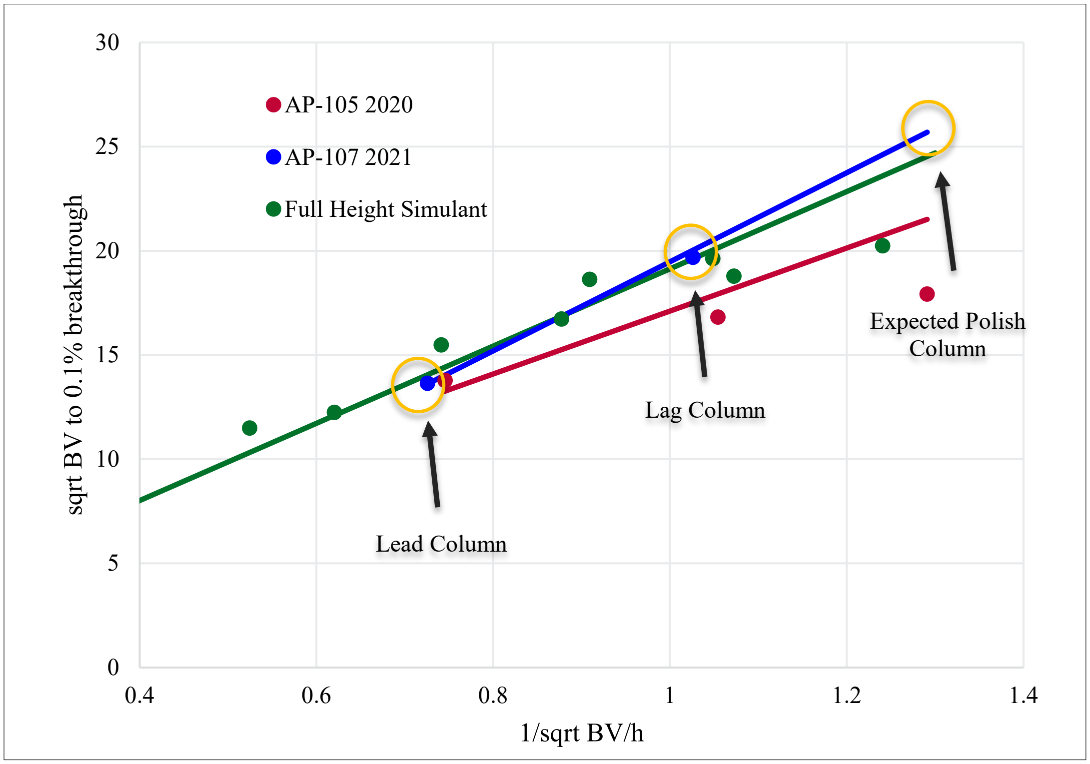

Figure 4.8. Projected Breakthrough Results of AP-105, AP-107, and 5.6 M Na Simulant

To further evaluate the impact of kinetics on Cs exchange, the same data from Figure 4.8 can be graphed as the percentage of capacity used vs. the residence time (BV/h) for each testing condition. Figure 4.9 shows reasonable linear fits over the range of interest and accentuates the impact on kinetics with varying temperature. The testing at 16°C shows a lower capacity use when 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. This analysis can also be used to estimate the BVs to breakthrough on the polish column: ~214,000 gallons.

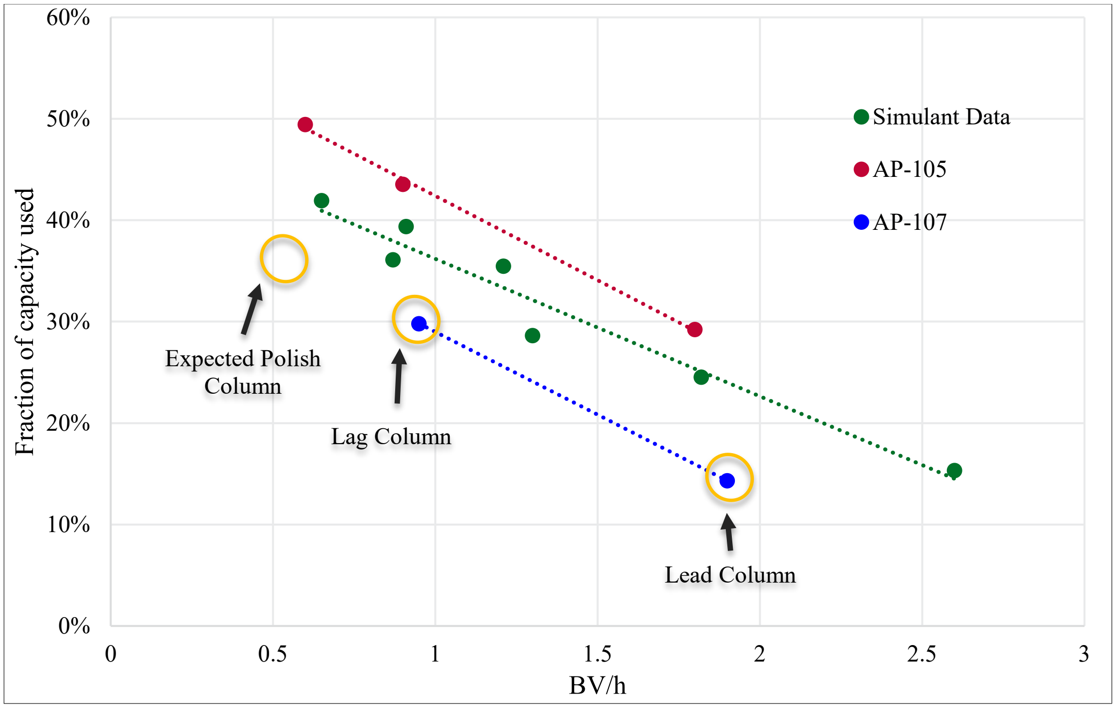

Figure 4.9. Percentage of CST Capacity Used vs. Residence Time for AP-105, AP-107, and 5.6 M Na Simulant

## 4.3 Cesium Activity Balance

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 and lag columns were determined by subtracting the Cs recovered in the samples and effluents from the Cs fed to each column. Table 4.1 summarizes the $^{137}$Cs fractions found in the various effluents as well as the calculated $^{137}$Cs column loadings. About 94% of the total Cs loaded onto the lead column (markedly higher than the 67% found with AP-105 processing, Fiskum et al. 2021b), and only 6% loaded onto the lag column. Sample and effluent collection amounted to ~0.05% of the input Cs.

Table 4.1. $^{137}$Cs Activity Balance for AP-107

| Input | µCi | % |
|---|---|---|
| Feed sample | 1.30E+06 | 100 |
| Output | | |
| Effluent-1 (0-91 BVs) | 2.181 | 1.68E-04 |
| Effluent-2 (91-214 BVs) | 0.264 | 2.03E-05 |
| Effluent-3 (214-310 BVs) | 0.222 | 1.71E-05 |
| Effluent-4 (310-404 BVs) | 0.5 | 3.74E-05 |
| Effluent-5 (404-510 BVs) | 3.4 | 2.65E-04 |
| Effluent-6 (510-634 BVs) | 21.708 | 1.67E-03 |
| Effluent-7 (634-725 BVs) | 59.50 | 4.59E-03 |
| Effluent-8 (725-791 BVs) | 95.4 | 7.35E-03 |
| Load samples | 409 | 3.15E-02 |
| Feed displacement, water rinse, and flush | 14.6 | 1.12E-03 |
| Total $^{137}$Cs recovered in effluents | 607 | 4.67E-02 |
| Total $^{137}$Cs column loading | | |
| Lead column Cs loading | 1.22E+06 | 94.1 |
| Lag column Cs loading | 7.56E+04 | 5.8 |
| Column total | 1.30E+06 | 100.0 |

The total Cs loaded per g CST was calculated from the total Cs loaded onto the lead column and the dry CST mass loaded into the lead column according to Eq. (4.2):

$$\frac{\mathrm{A_{Cs}} \times \mathrm{CF}}{\mathrm{M}} = \mathrm{C} \tag{4.2}$$

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 (10.0 g)
C = capacity, mg Cs/g CST

A total of 7.08 mg Cs/g CST (0.0528 mmoles Cs/g CST) was loaded onto the lead column. This value represents how much was loaded onto the lead column when the lag column reached the WAC limit and is consistent with previous AP-107 and 5.6 M Na simulant studies (see Table 4.2). The documented safety analysis developed for TSCR limits a single column curie loading to 141,600 Ci, which equates to 0.10 mmole Cs per g CST.

Table 4.2. Cs CST Column Loading Comparison

| Test | Sieve Fraction | CST Cs loading (mg Cs/g CST) | Reference |
|---|---|---|---|
| AP-107 chilled, 2.4% full height | <30 mesh | 7.08 | Current report |
| AP-105, 2.4% full height | <30 mesh | 5.39 | Fiskum et al. 2021b |
| AP-107, 2.4% full height | <25 mesh | 6.76 | Fiskum et al. 2019a |
| 5.6 M Na simulant, 2.5% full height | <25 mesh | 6.87 | Fiskum et al. 2019b |
| 5.6 M Na simulant, 2.5% full height | <30 mesh | 7.63 | Rovira et al. 2018 |
| 5.6 M Na simulant, 2.5% full height | <35 mesh | 7.04 | Fiskum et al. 2019b |
| 5.6 M Na simulant, 12% full height | <25 mesh | 6.95 | Fiskum et al. 2019b |
| 5.6 M Na simulant, 100% full height | As received | 6.60 | Fiskum et al. 2019b |

See Russell et al. (2017) for the 5.6 M Na simulant formulation.

## 4.4 Metals and Radionuclide Analysis

The AP-107 composite feed and composite effluent samples underwent extensive characterization to better define waste characteristics and assess analyte fractionation to the CST. Table 4.3 summarizes the GEA, alpha energy analysis (AEA), and fractionation results for radionuclides in the composite feed and effluent samples. Compositions and fractionations of inorganic analytes, determined by inductively coupled plasma optical emission spectroscopy (ICP-OES), are shown in Table 4.4.

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. (4.3):

$$\frac{C_{Da} \times V_D}{C_{Fa} \times V_F} = F_{Da} \tag{4.3}$$

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-107 feed
$V_F$ = volume of AP-107 feed
$F_{Da}$ = fraction of analyte *a* in the Cs-decontaminated effluent

Ten lead column samples (collected after processing 30.9, 75.2, 128, 174, 217, 314, 454, 604, 732, and 799 BVs) were also selected for metal and radionuclide analysis to assess analyte load characteristics. The opportunistic analyte results measured by ICP-OES are also shown in Table 4.4; these analytes are part of the ICP-OES data output but have not been fully evaluated for quality control performance. The analyte results shown in brackets indicate the result was less than the instrument 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. Complete results of the GEA, AEA, and ICP-OES analyses are given in Appendix C (ASR 1248).

Table 4.3. AP-107 Feed and Effluent Radionuclide Concentrations and Fractionations (ASR 1248)

| Analysis Method | Analyte | TI100-FEED-COMP[(a)] (µCi/mL) | TI100-EFF-COMP[(a)] (µCi/mL) | Percent in Effluent |
|---|---|---|---|---|
| Gamma energy analysis (GEA)[(b)] | $^{60}$Co | <8.5E-4 | 3.93E-04 | -- |
| | $^{126}$Sn | <3.5E-2 | 3.61E-04 | -- |
| | $^{126}$Sb | <4.2E-3 | 2.56E-04 | -- |
| | $^{137}$Cs | 1.54E+02[(c)] | 2.17E-02 | 0.014 |
| | $^{154}$Eu | <4.2E-3 | 2.79E-05 | -- |
| | $^{241}$Am | <2.1E-1 | 2.48E-04 | -- |
| Separations/ Alpha energy analysis (AEA)[(a)] | $^{237}$Np | 4.59E-05 | 3.70E-05 | 80 |
| | $^{238}$Pu | 8.67E-05 | 6.12E-05 | 70 |
| | $^{239+240}$Pu | 7.39E-04 | 4.98E-04 | 67 |
| | Total Alpha | 1.15E-03 | 7.59E-04 | 65 |

(a) In the test instruction identified in footnote 11, the samples were labeled “TI100-COMP-FEED” and “TI100-COMP-EFF”; however, they were inadvertently mislabeled as “TI100-FEED-COMP” and “TI100-EFF-COMP” in ASR 1248 (the laboratory analysis documents). The Sample IDs shown in ASR 1248 are used in this report to maintain traceability to the analysis results.
(b) Reference date is June 2021.
(c) $^{137}$Cs measured in the individual feed samples was 162 µCi/mL (see Section 3.2); the 154 µCi/mL value was 3.5% lower and was not considered statistically different given the overall experimental uncertainty.
“--” = 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 4.4. AP-107 Feed and Effluent Inorganic Analyte Concentrations and Fractionations (ASR 1248)

| Analysis Method | Analyte | TI100-FEED-COMP[(a)] (M) | TI100-EFF-COMP[(a)] (M) | Percent in Effluent |
|---|---|---|---|---|
| ICP-OES | Al | 3.78E-01 | 3.74E-01 | 98 |
| | Ba | [4.5E-06] | <8.2E-07 | -- |
| | Ca | [3.7E-04] | 2.41E-04 | 64 |
| | Cd | 7.23E-05 | 5.96E-05 | 81 |
| | Fe | 3.38E-04 | 2.79E-04 | 82 |
| | K | 1.01E-01 | 9.54E-02 | 94 |
| | Na | 6.44E+00 | 6.20E+00 | 95 |
| | Nb | <7.8E-06 | [2.1E-05] | NA |
| | P | 2.87E-02 | 2.71E-02 | 93 |
| | Pb | [6.3E-05] | <4.8E-05 | -- |
| | S | 7.21E-02 | 6.94E-02 | 95 |
| | Sr | [1.1E-06] | <8.7E-07 | -- |
| | Th | <1.3E-05 | <1.3E-05 | -- |
| | U | <6.9E-05 | [8.4E-05] | -- |
| | Zn | [1.5E-04] | [3.1E-05] | -- |
| | Zr | <5.8E-06 | [2.1E-05] | NA |

| Analysis Method | Analyte | TI100-FEED-COMP[(a)] (M) | TI100-EFF-COMP[(a)] (M) | Percent in Effluent |
|---|---|---|---|---|
| ICP-OES Opportunistic Analytes | Ag | [1.0E-05] | [1.2E-05] | -- |
| | As | <5.1E-04 | 5.22E-04 | -- |
| | Au | <9.7E-06 | 9.88E-06 | -- |
| | B | 7.38E-03 | 2.71E-03 | 36 |
| | Be | [6.4E-06] | 6.12E-06 | -- |
| | Bi | <6.4E-05 | <6.5E-05 | -- |
| | Ce | <7.8E-05 | <7.9E-05 | -- |
| | Co | <2.9E-05 | <3.0E-05 | -- |
| | Cr | 1.14E-02 | 1.09E-02 | 95 |
| | Cu | [1.2E-04] | [1.1E-04] | -- |
| | Dy | <5.1E-06 | <5.2E-06 | -- |
| | Eu | <1.7E-06 | <1.7E-06 | -- |
| | Ga | <5.3E-05 | 5.59E-05 | -- |
| | Hf | <5.8E-05 | 1.05E-04 | -- |
| | La | <5.2E-06 | 5.27E-06 | -- |
| | Li | 1.22E-04 | 1.02E-04 | 82 |
| | Lu | <1.1E-06 | 1.16E-06 | -- |
| | Mg | <2.3E-05 | [2.4E-05] | -- |
| | Mn | [2.2E-05] | [2.1E-05] | -- |
| | Mo | 4.95E-04 | 4.65E-04 | 93 |
| | Nd | <3.2E-05 | 3.24E-05 | -- |
| | Ni | 3.59E-04 | 3.30E-04 | 91 |
| | Pd | [5.3E-05] | [4.8E-05] | -- |
| | Rh | [4.2E-05] | [6.5E-05] | -- |
| | Ru | [6.9E-05] | [7.9E-05] | -- |
| | Sb | <1.8E-04 | <1.9E-04 | -- |
| | Sc | <3.6E-06 | <3.6E-06 | -- |
| | Se | <7.0E-04 | <7.1E-04 | -- |
| | Si | 4.56E-03 | 3.16E-03 | 68 |
| | Sn | [9.3E-05] | [1.6E-04] | -- |
| | Ti | <7.0E-06 | <7.1E-06 | NA |
| | W | 3.67E-04 | 3.80E-04 | 102 |

(a) In the test instruction identified in footnote 11, the samples were labeled "TI100-COMP-FEED" and "TI100-COMP-EFF"; however, they were inadvertently mislabeled as "TI100-FEED-COMP" and "TI100-EFF-COMP" in ASR 1248 (the laboratory analysis documents). The sample IDs shown in ASR 1248 are used in this report to maintain traceability to the analysis results.

Bracketed values indicate the associated sample results were less than the EQL but greater than or equal to the MDL. Analytical uncertainty for these analytes was > ±15%.

"--" indicates the recovery could not be calculated.

NA = not applicable; Nb, Ti, and Zr are components of CST.

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.

Lead (Pb), barium (Ba), and strontium (Sr) were detected in the feed (with concentration errors likely to exceed 15%) but were below the MDL in the effluent; this was indicative of uptake by the CST. The ICP-OES results for the feed composite and effluent composite showed that the majority of analytes remained in the effluent. The Al, Cr, K, Na, P, and S partitioned exclusively to the effluent (>90% recovery). In addition to Cs removal, nominally 40% of the Ca and 20% of the Cd and Fe also partitioned to the CST.

About 20% of the Np and 30% of the Pu were removed by the CST. The Np and Pu removal factors previously reported for AP-107 testing at 28°C were 57% and 63%, respectively (Fiskum et al. 2019a). Assuming the difference in total Pu µCi content between the feed and effluent remained with the lead column CST (10.0 g), the CST would contain 222 nCi/g transuranic (TRU) isotopes, which is over the threshold 100 nCi/g defining TRU waste.

The load behaviors of selected analytes were examined as a function of BVs processed through the lead column. Figure 4.10 shows the Al, Ba, Ca, Cd, and Fe breakthrough results along with the Cs breakthrough profile. The Al breakthrough serves as an "internal standard" for comparison of the ICP-OES analysis results; its breakthrough remained at 95% ±3% throughout the analytical run.

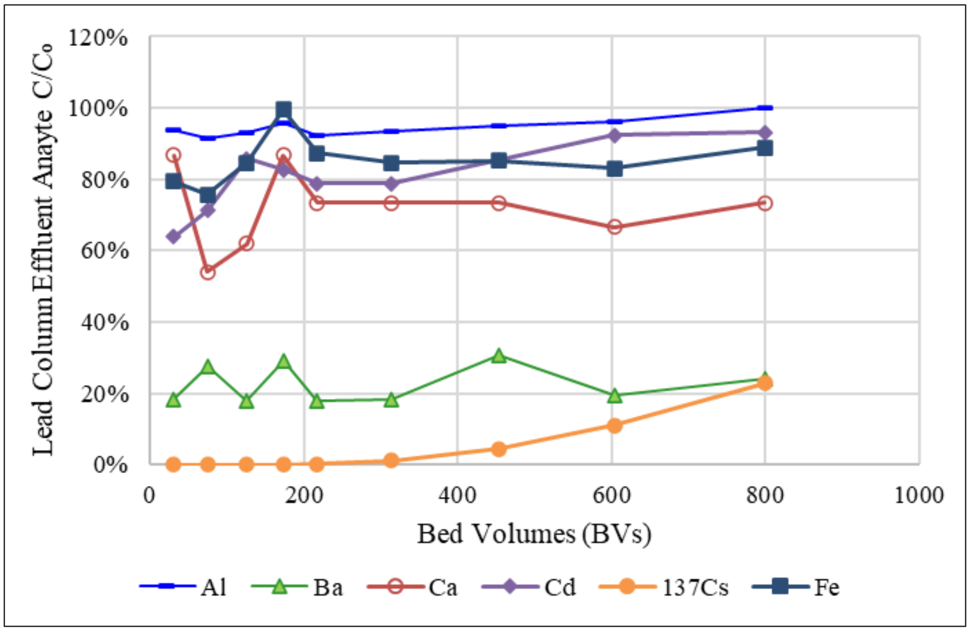

Figure 4.10. Al, Ba, Ca, Cd, Cs, and Fe Load Profiles from the Lead Column

Similarly, the selected lead column effluent samples were analyzed for $^{237}$Np, $^{238}$Pu, and $^{239+240}$Pu. Figure 4.11 shows the load profiles in comparison with that of $^{137}$Cs. The Np and Pu breakthrough profiles showed a large increase in effluent concentration from ~30% to ~85% in the 30 to 75 BVs range before resulting in steady-state around 90% between 75 and 800 BVs. Although the trend of the breakthrough is consistent with that measured from previous AP-107 processing (Fiskum et al. 2019b), the amount partitioned to CST was nominally 53% less for Np and 75% less for Pu.

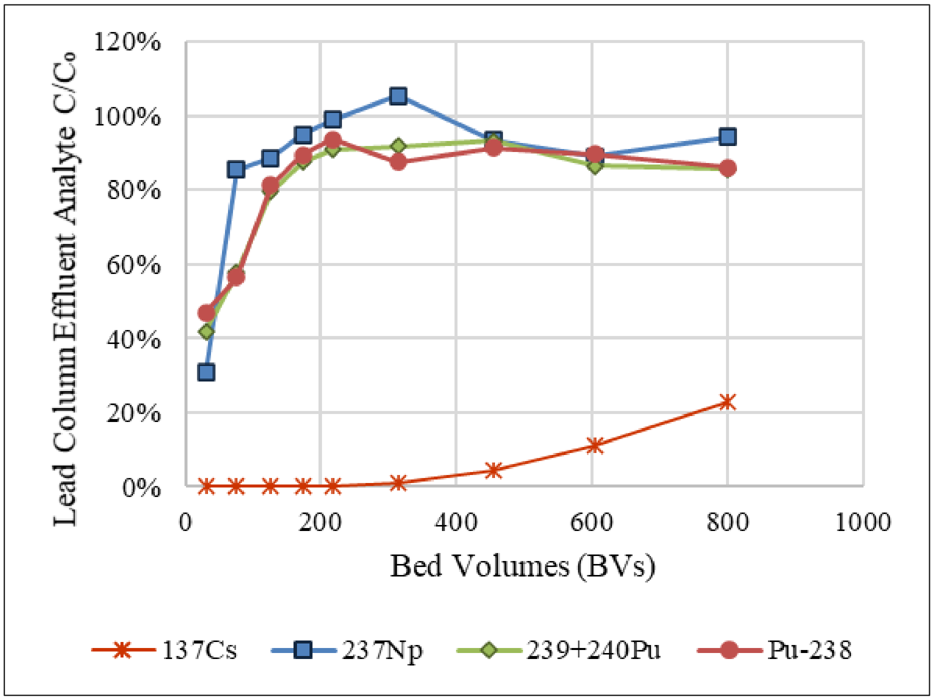

Figure 4.11. $^{137}$Cs, $^{237}$Np, $^{238}$Pu, and $^{239+240}$Pu Load Profiles onto the Lead Column

Neither Ti, Nb, nor Zr (components of CST) were detected in the feed; however, small concentrations (with errors likely to exceed 15%) of Nb and Zr were found in the effluent. This is indicative of small losses of CST components occurring 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-107 tank waste at 16°C. Column testing was conducted at a small scale in the RPL hot cells to accommodate the high radiological dose rate of the Hanford tank waste matrix. The results summary is provided below.

## 5.1 Column Testing

AP-107 tank waste was processed through two columns sequentially positioned in a lead-lag format; each column was filled with 10.0 mL of CST ion exchanger. A total of 8.0 L of AP-107 tank waste, consisting of 5.6 M Na and 162 µCi/mL $^{137}$Cs, was processed through the Cs ion exchange system at 1.90 BV/h and 16°C. Effluent samples were collected periodically from each column during the load process and measured for $^{137}$Cs to establish the Cs load curves. The flowrate was increase to 3.0 BV/h to process 12.0 BVs each of 0.1 M NaOH FD and water rinse. The following conclusions were drawn from the results of this work.

1. Testing showed that at 16°C, 800 BVs of AP-107 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 790 BVs of AP-107 feed (essentially the complete volume of available feed) was processed and therefore the polish column was never brought online.
2. The WTP LAW WAC limit for the lead column with <30-mesh CST was reached at the same time as lead column breakthrough with AP-107 and <25-mesh CST at 28°C (Fiskum et al. 2019a). However, the overall breakthrough slope of the 28°C test was greater than that of the 16°C test, indicating that temperature impacts on kinetics are greater than the particle size impact on kinetics.
3. Extrapolation of Cs effluent concentration data from the lead column showed the 50% Cs breakthrough would be reached after processing 1087 BVs.
4. The Cs load profile for the lag column at 16°C was offset to the right (later Cs breakthrough) of the lag column load profile generated with AP-107 at 28°C (Fiskum et al. 2019a). This indicates that although the kinetics were slower at the colder temperature, the reduction in kinetics was compensated by the increase in capacity at 16°C and smaller CST particle size used in the 16°C test (<30 mesh) compared to the 28°C test (<25 mesh).
5. For the lag column, the reduced temperature testing was shifted to the right and reached the WAC limit at a significantly later time than the test at higher temperature, indicating that although the kinetics were slower, again, this was compensated by the increase in capacity and reduced particle size.
6. The total Cs loading onto the lead column (7.08 mg Cs/g CST) was similar to that seen in previous AP-107 and simulant testing (6.76 and 7.63 mg Cs/g CST, respectively) at similar processing flowrates.

## 5.2 Analyte Fractionation

1. Major components Al, K, Na, P, and S partitioned exclusively to the effluent. Minor components, Cr, Mo, and Ni, also portioned to the effluent (>90% recovery).
2. Approximately 40% of the Ca and 20% of the Cd and Fe partitioned to the CST.
3. Nb and Zr, components of CST (below MDL in the feed), were detected in the composite effluent and the selected lead column effluent samples, indicating that a small amount of CST components leached into solution.
4. The effluent contained 80% of the feed Np and 70% of the feed Pu. 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 111 nCi/g TRU, which is above the 100 nCi/g threshold defining TRU waste.

# 6.0 References

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# Appendix A – Column Load Data

The AP-107 lead and lag column loading raw data are provided in Table A.1. The feed displacement, water rinse, and final fluid expulsion raw data are provided in Table A.2. 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 AP-107

| Lead Column | | | | Lag Column | | | |
|---|---|---|---|---|---|---|---|
| BV | µCi $^{137}$Cs/mL | % C/$C_0$ | DF | BV | µCi $^{137}$Cs/mL | % C/$C_0$ | DF |
| 8.8 | 1.25E-02 | 7.70E-03 | 1.30E+04 | 30.7 | 2.46E-03 | 1.52E-03 | 6.57E+04 |
| 30.9 | 4.02E-03 | 2.49E-03 | 4.02E+04 | 74.6 | 2.71E-03 | 1.68E-03 | 5.96E+04 |
| 46.4 | 7.17E-04 | 4.43E-04 | 2.26E+05 | 125.0 | 1.18E-03 | 7.32E-04 | 1.37E+05 |
| 75.2 | 1.02E-03 | 6.33E-04 | 1.58E+05 | 172.3 | 6.24E-04 | 3.86E-04 | 2.59E+05 |
| 91.7 | 2.04E-03 | 1.26E-03 | 7.94E+04 | 215.3 | 5.49E-04 | 3.39E-04 | 2.95E+05 |
| 125.7 | 1.52E-02 | 9.41E-03 | 1.06E+04 | 262.8 | 5.16E-04 | 3.19E-04 | 3.14E+05 |
| 139.1 | 2.97E-02 | 1.84E-02 | 5.44E+03 | 311.4 | 6.22E-04 | 3.85E-04 | 2.60E+05 |
| 173.7 | 9.57E-02 | 5.92E-02 | 1.69E+03 | 353.5 | 6.60E-04 | 4.08E-04 | 2.45E+05 |
| 186.9 | 1.44E-01 | 8.92E-02 | 1.12E+03 | 405.4 | 1.23E-03 | 7.61E-04 | 1.31E+05 |
| 217.2 | 2.97E-01 | 1.84E-01 | 5.44E+02 | 450.8 | 2.59E-03 | 1.60E-03 | 6.24E+04 |
| 264.8 | 7.74E-01 | 4.78E-01 | 2.09E+02 | 464.5 | 3.11E-03 | 1.92E-03 | 5.20E+04 |
| 313.7 | 1.65E+00 | 1.02E+00 | 9.80E+01 | 512.8 | 6.59E-03 | 4.07E-03 | 2.46E+04 |
| 356.0 | 2.69E+00 | 1.67E+00 | 6.00E+01 | 547.7 | 1.13E-02 | 7.01E-03 | 1.43E+04 |
| 453.5 | 6.83E+00 | 4.22E+00 | 2.37E+01 | 600.1 | 2.28E-02 | 1.41E-02 | 7.09E+03 |
| 515.8 | 1.00E+01 | 6.19E+00 | 1.61E+01 | 636.8 | 3.70E-02 | 2.29E-02 | 4.37E+03 |
| 550.9 | 1.29E+01 | 7.98E+00 | 1.25E+01 | 678.1 | 5.41E-02 | 3.34E-02 | 2.99E+03 |
| 603.5 | 1.70E+01 | 1.05E+01 | 9.51E+00 | 728.3 | 9.89E-02 | 6.11E-02 | 1.64E+03 |
| 640.4 | 1.92E+01 | 1.19E+01 | 8.41E+00 | 771.0 | 1.46E-01 | 9.02E-02 | 1.11E+03 |
| 681.9 | 2.35E+01 | 1.45E+01 | 6.88E+00 | 794.4 | 1.82E-01 | 1.12E-01 | 8.91E+02 |
| 732.4 | 2.77E+01 | 1.72E+01 | 5.83E+00 | | | | |
| 775.2 | 3.22E+01 | 1.99E+01 | 5.02E+00 | | | | |
| 798.9 | 3.51E+01 | 2.17E+01 | 4.61E+00 | | | | |

BV = bed volume, 10.0 mL
DF = decontamination factor
$C_0$ = 162 µCi $^{137}$Cs/mL

Table A.2. Feed Displacement, Water Rinse, and Final Flush Results Following AP-107 Processing

| Feed Displacement | | | | Water Rinse | | | | Final Fluid Flush | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 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 |
| 1.9 | 1.94E-1 | 1.20E-1 | 8.36E+2 | 2.0 | 2.85E-3 | 1.76E-3 | 5.68E+4 | 3.4 | 6.55E-2 | 4.05E-2 | 2.47E+3 |
| 3.9 | 1.71E-1 | 1.05E-1 | 9.49E+2 | 4.2 | 9.95E-4 | 6.15E-4 | 1.63E+5 | | | | |
| 5.7 | 1.67E-1 | 1.03E-1 | 9.71E+2 | 6.3 | 1.35E-3 | 8.35E-4 | 1.20E+5 | | | | |
| 8.0 | 2.88E-2 | 1.78E-2 | 5.63E+3 | 8.5 | 7.70E-3 | 4.76E-3 | 2.10E+4 | | | | |
| 9.9 | 4.57E-3 | 2.82E-3 | 3.54E+4 | 10.6 | 2.48E-2 | 1.53E-2 | 6.52E+3 | | | | |
| 11.9 | 1.81E-3 | 1.12E-3 | 8.96E+4 | 12.6 | 3.09E-2 | 1.91E-2 | 5.24E+3 | | | | |

BV = bed volume, 10.0 mL
DF = decontamination factor
$C_0$ = 162 µCi $^{137}$Cs/mL

# Appendix B – Analyte Concentrations as a Function of Loading

The load behaviors of selected analytes in AP-107 were evaluated from selected samples collected from the lead column. Analysis results of these samples are summarized in Table B.1.

Table B.1. Analyte Concentrations of Selected Samples from the Lead Column during AP-107 16°C Processing

| BV Processed> | NA | 30.9 | 75.2 | 125.7 | 173.7 | 217.2 | 313.7 | 453.5 | 603.5 | 798.9 |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample ID> | TI100-Feed-Comp | TI100-L-F2-A | TI100-L-F4-A | TI100-L-F6-A | TI100-L-F8-A | TI100-L-F10-A | TI100-L-F12-A | TI100-L-F14-A | TI100-L-F17-A | TI100-L-F22-A |
| Analyte | ICP-OES, M | | | | | | | | | |
| Al | 3.78E-01 | 3.54E-01 | 3.47E-01 | 3.52E-01 | 3.62E-01 | 3.49E-01 | 3.53E-01 | 3.58E-01 | 3.63E-01 | 3.78E-01 |
| Ba | [4.5E-06] | <8.3E-07 | [1.2E-6] | <8.2E-07 | [1.3E-6] | [8.0E-7] | <8.2E-07 | [1.4E-6] | [8.7E-7] | [1.1E-6] |
| Ca | [3.7E-04] | [3.2E-4] | [2.0E-4] | [2.3E-4] | [3.2E-4] | [2.7E-4] | [2.7E-4] | [2.7E-4] | [2.5E-4] | [2.7E-4] |
| Cd | 7.23E-05 | [4.6E-5] | 5.16E-05 | 6.20E-05 | [6.0E-5] | [5.7E-5] | [5.7E-5] | 6.17E-05 | 6.68E-05 | 6.73E-05 |
| Fe | 3.38E-04 | 2.69E-04 | 2.56E-04 | 2.86E-04 | 3.37E-04 | 2.95E-04 | 2.86E-04 | 2.88E-04 | 2.81E-04 | 3.01E-04 |
| K | 1.01E-01 | 9.46E-02 | 9.23E-02 | 9.57E-02 | 9.67E-02 | 9.39E-02 | 9.57E-02 | 9.46E-02 | 9.59E-02 | 9.92E-02 |
| Na | 6.44E+00 | 6.13E+0 | 6.00E+0 | 6.18E+00 | 6.26E+00 | 6.09E+00 | 6.18E+00 | 6.31E+00 | 6.22E+00 | 6.26E+00 |
| Nb | <7.8E-06 | [3.3E-5] | [3.2E-5] | [1.5E-5] | [1.1E-5] | [1.9E-5] | <8.0E-06 | [8.3E-6] | <7.8E-06 | [9.6E-6] |
| P | 2.87E-02 | 2.68E-02 | 2.61E-02 | 2.53E-02 | 2.66E-02 | 2.59E-02 | 2.55E-02 | 2.69E-02 | 2.65E-02 | 2.75E-02 |
| S | 7.21E-02 | 6.96E-02 | 6.58E-02 | 7.05E-02 | 7.24E-02 | 7.05E-02 | 6.86E-02 | 6.96E-02 | 6.67E-02 | 7.02E-02 |
| U | <6.9E-05 | <7.1E-05 | <7.0E-05 | [9.2E-5] | [7.6E-5] | [9.2E-5] | [8.8E-5] | [8.0E-5] | [7.1E-5] | [7.6E-5] |
| Zn | [1.5E-04] | [4.4E-5] | [2.6E-5] | <2.5E-05 | [2.4E-5] | [1.7E-4] | <2.5E-05 | [2.8E-5] | <2.5E-05 | [3.2E-5] |
| Zr | <5.8E-06 | [1.3E-5] | [1.3E-5] | [1.3E-5] | [1.5E-5] | [1.2E-5] | [1.3E-5] | [1.5E-5] | [1.1E-5] | [1.2E-5] |
| Analyte | Radiochemistry, µCi/mL[(a)] | | | | | | | | | |
| $^{137}$Cs | 1.54E+02 | 4.02E-03 | 1.02E-03 | 1.52E-02 | 9.57E-02 | 2.97E-01 | 1.65E+00 | 6.83E+00 | 1.70E+01 | 3.51E+01 |
| Total Alpha | 1.15E-03 | 4.10E-04 | 7.34E-04 | 8.44E-04 | 5.86E-04 | 6.62E-04 | 7.96E-04 | 1.08E-03 | 6.45E-04 | 8.05E-04 |
| $^{237}$Np | 4.59E-05 | 1.41E-05 | 3.92E-05 | 4.07E-05 | 4.36E-05 | 4.54E-05 | 4.84E-05 | 4.29E-05 | 4.09E-05 | 4.33E-05 |
| $^{238}$Pu | 8.67E-05 | 4.07E-05 | 4.89E-05 | 7.05E-05 | 7.76E-05 | 8.12E-05 | 7.60E-05 | 7.93E-05 | 7.77E-05 | 7.46E-05 |
| $^{239+240}$Pu | 7.39E-04 | 3.10E-04 | 4.26E-04 | 5.89E-04 | 6.47E-04 | 6.73E-04 | 6.78E-04 | 6.90E-04 | 6.40E-04 | 6.33E-04 |

(a) Reference date is June 2021.
BV = bed volume, 10.0 mL
Bracketed values indicate the associated sample results were less than the estimated quantitation limit (EQL) but greater than or equal to the method detection limit (MDL). Analytical uncertainties for these analytes are > ±15%.
Additional analyte concentrations may be found in Appendix C, ASR 1248.

# Appendix C – Analytical Reports

Analytical reports provided by the Analytical Support Operations (ASO) laboratory are included in this appendix. In addition to the analyte results, they define the procedures used for chemical separations and analysis, as well as quality control sample results, observations during analysis, and overall estimated uncertainties. The analyses are grouped according to Analytical Service Request (ASR) number. Cross-references of ASO sample IDs to test description are provided in the body of the report (see Table 3.2 and Table 3.4 of the main report).

## Appendix C Table of Contents

ASR 1193, Initial Characterization of AP-107, As-Received

ASR 1248, AP-107 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 Sandra K Fiskum
Print Name Sandra K Fiskum
Phone 375-5677 MSIN

**Project Number:** 77636
**Work Package:** NH0127

## Matrix Type Information

◆ **Liquids:** ☑ Aqueous ☐ Organic ☐ Multi-phase
◆ **Solids:** ☐ Soil ☐ Sludge ☐ Sediment
☐ Glass ☐ Filter ☐ Metal
☐ Smear ☐ Organic ☐ Other

◆ **Other:** ☐ Solid/Liquid Mixture, Slurry
☐ Gas ☐ Biological Specimen

(If sample matrices vary, specify on Request Page)

## Disposal Information

◆ **Disposition of Virgin Samples:**
Virgin samples are returned to requestor unless archiving provisions are made with receiving group!
**If archiving, provide:**
Archiving Reference Doc:

◆ **Disposition of Treated Samples:**
☐ Dispose ☐ Return NA

## QA/Special Requirements

◆ **QA Plan:**
☑ ASO-QAP-001 (Equivalent to HASQARD)
☐ Additional QA Requirements, List Document Below:
Reference Doc Number:

◆ **Field COC Submitted?** ☑ No ☐ Yes
◆ **Lab COC Required?** ☑ No ☐ Yes
◆ **Sample/Container Inspection Documentation Required?** ☑ No ☐ Yes

◆ **Hold Time:** ☑ No ☐ ***Yes***
***If Yes,*** **Contact ASO Lead before submitting Samples**
☐ Use SW 846 (PNL-ASO-071, identify analytes/methods where holding times apply)
☐ Other? Specify:

◆ **Special Storage Requirements:**
☑ None ☐ Refrigerate ☐ Other, Specify:

◆ **Data Requires ASO Quality Engineer Review?** ☐ No ☐ Yes

## Data Reporting Information

◆ **Is Work Associated with a Fee-Based Milestone?** ☑ No ☐ Yes
If yes, milestone due date:

◆ **Preliminary Results Requested, As Available?** ☐ No ☑ Yes

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

◆ **Requested Analytical Work Completion Date:** 12/18/20
(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?** ☑ No ☐ Yes
If no, Reference Doc Attached:
or, Previous ASR Number: 0747
or, Previous RPL Number:

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

**Send Report To:** SK Fiskum **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? ☐ No ☐ Yes

Received By:
ASR Number: 1193 Rev.:
RPL Numbers:
(first and last)

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

# Analytical Service Request (ASR)

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

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

| ASO Staff Use Only | | | | ASO Staff Use Only | |
|---|---|---|---|---|---|
| RPL Number | Customer Sample ID | Sample Description (& Matrix, if it varies) | Analysis Requested | Test | Library |
| | 7AP-20-16 | AP-107 tank waste | GEA Cs-137, Co-60 | | |
| | 7AP-20-33 | | | | |
| | 7AP-20-34 | ↓ | ↓ | | |
| | 7AP-20-51 | | | | |
| | | | | | |
| | | | | | |
| | 0.2 ml sample in | 1.8 ml 0.1M NaOH | | | |
| | glass LSC vials. | | | | |
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ASR # ____________ Rev.: ______

Page ______ of ______

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

filename 21-0262 Fiskum
12/11/2020

Client: SK Fiskum Project: 77636
ASR: 1193 WP: NH0127

Prepared by: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2020.12.11 11:57:59 -08'00'

Technical Reviewer: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2020.12.11 12:00:21 -08'00'

Procedure: RPG-CMC-450 Rev3, Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)
Count date: December 8-9, 2020
M & TE: G,L,T

| | Lab | Measured Gamma Emitters, μCi per sample ± 1s counting error | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Sample** | ID | **Co-60** | | **Cs-137** | | **Eu-155** | **U-235** | **U-238** | |
| 7AP-20-16 | 21-0262 | **9.58E-5** | **± 37%** | **3.16E+1** | **± 2%** | <4.83E-3 | <2.56E-3 | <2.89E-2 | |
| 7AP-20-33 | 21-0263 | **9.79E-5** | **± 10%** | **3.10E+1** | **± 2%** | <3.71E-3 | <2.76E-3 | <2.59E-2 | |
| 7AP-20-34 | 21-0264 | **9.57E-5** | **± 14%** | **3.50E+1** | **± 2%** | <6.52E-3 | <3.20E-3 | **1.34E-2** | **± 6%** |
| 7AP-20-51 | 21-0265 | **1.11E-4** | **± 8%** | **3.41E+1** | **± 2%** | <4.99E-3 | <2.80E-3 | <3.09E-2 | |

# 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 Karl N Pool for A. Westesen
Print Name ___Amy Westesen______________________
Phone ______371-7908________ MSIN ___________

**Project Number:** ______77636________________
**Work Package:** ________NH0135_____________

**Matrix Type Information**

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

(If sample matrices vary, specify on Request Page)

**Disposal Information**

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

**QA/Special Requirements**

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

**Data Reporting Information**

| | | |
|---|---|---|
| ◆ **Is Work Associated with a Fee-Based Milestone?** X No ☐ Yes<br>If yes, milestone due date:<br>_________________________<br>◆ **Preliminary Results Requested, As Available?** ☐ No **X** Yes | ◆ **Data Reporting Level**<br>X ASO-QAP-001 (Equivalent to HASQARD).<br>☐ Minimum data report.<br>☐ Project Specific Requirements: Contact ASO Lead or List Reference Document:____________________ | ◆ **Requested Analytical Work Completion Date:**<br>______________________________<br>**(Note: Priority rate charge for < 10 business day turn-around time)**<br>◆ **Negotiated Commitment Date:**<br>______7/10/21____________<br>**(*To be completed by ASO Lead*)** |

**Waste Designation Information**

| | |
|---|---|
| ◆ **ASO Sample Information Check List Attached?** **X** No ☐ Yes<br>If no, Reference Doc Attached: ________________________<br>or, Previous ASR Number: __________________________<br>or, Previous RPL Number: ____________________ | ***Does the Waste Designation Documentation Indicate Presence of PCBs?***<br>**X No ☐ Yes** |

**Send Report To:** ___**A. Westesen, S. Fiskum**_______ **MSIN** __________________
_______E. Campbell______________ **MSIN** __________________
**Additional or Special Instructions** ____________________________________________

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**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: _____1248______Rev.: ____01____
RPL Numbers: _21-0864 & 21-0865 and 21-1070 – 21-1078_
(first and last)

**ASO Work Accepted By:** ___KN Pool___ **Signature/Date:** Karl N Pool 6/4/21

# Analytical Services Request (ASR)

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

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

| ASO Staff Use Only<br>**RPL Number** | Client Sample ID | Sample Description (& Matrix if varies) | Analyses Requested | ASO Staff Use Only<br>**Test** | **Library** |
|---|---|---|---|---|---|
| | **Revision 1** - ASR revised to update client ID's and Remove GEA from Cs-IX Effluent Fractions | | | | |
| 21-0864 | TI100-FEED-COMP | AP-107 - Supernate Feed | 1) Direct Sample<br>a) GEA - Cs-137<br>b) Pu-AEA (Pu-239+240)<br>c) Np-AEA - Direct Sample<br>2) Acid Digest - 128<br>a) ICP/OES - Al, Ba, Ca, Cd, Fe, K, Na, Nb, P, Pb, S, Sr, Ti, U, Zn and Zr | | |
| 21-0865 | TI100-EFF-COMP | AP-107 - IX Effluent | | | |
| 21-1070 | TI100-L-F2-A | Cs-IX Effluent Fractions | 1) Direct Sample<br>a) Pu-AEA (Pu-239+240)<br>b) Np-AEA - Direct Sample<br>2) Acid Digest - 128<br>a) ICP/OES - Al, Ba, Ca, Cd, Fe, K, Na, Nb, P, Pb, S, Sr, Ti, U, Zn and Zr | | |
| 21-1071 | TI100-L-F4-A | | | | |
| 21-1072 | TI100-L-F6-A | | | | |
| 21-1073 | TI100-L-F8-A | | | | |
| 21-1074 | TI100-L-F10-A | | | | |
| 21-1075 | TI100-L-F12-A | | | | |
| 21-1076 | TI100-L-F14-A | | | | |
| 21-1077 | TI100-L-F17-A | | | | |
| 21-1078 | TI100-L-F22-A | | | | |
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ASR #_1248_________ Rev:__01___

Page 1 of 1

ASR 1248.01 Request Page

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

| | |
|---|---|
| **Project / WP#:** | 77636 / NH0135 |
| **ASR#:** | 1248.01 |
| **Client:** | A.Westesen |

| ASO Sample ID | Client Sample ID | Client Sample Description | Sample Weight (g) |
|---|---|---|---|
| 21-0864 | TI100-FEED-COMP | AP-107 – Supernate Feed | NA |
| 21-0865 | TI100-EFF-COMP | AP-107 IX Effluent | NA |
| 21-1070 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1071 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1072 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1073 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1074 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1075 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1076 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1077 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |
| 21-1078 | TI100-L-F2-A | CS-IX Effluent Fractions | NA |

**Sample Preparation**: Simple dilution of the dissolved sample in 5% v/v $HNO_3$ performed by J. Carter on 06/21/2021.

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

| | | | | | |
|---|---|---|---|---|---|
| **Analyst:** | J. Carter | **Analysis Date:** | 06/21/2021 | **ICP File:** | C0875 |

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

| **M&TE:** | | | | |
|---|---|---|---|---|
| | ☒ | PerkinElmer 5300DV ICP-OES | SN: 077N5122002 | RPL 405 Bench |
| | ☐ | Sartorius ME414S | SN: 21308482 | RPL 420 |
| | ☒ | Ohaus Pioneer PA224C | SN: B725287790 | RPL 405 Bench |
| | ☐ | Mettler AT201 Balance | SN: 1113043473 | RPL 302 FH #4 |
| | ☐ | Mettler AT400 Balance | SN: 1113292667 | RPL 420 FH #13 |
| | ☐ | Sartorius R200D Balance | SN: 39080058 | RPL 525 FH #9 |

Carter
Report Preparer

6/23/2021
Date

Samuel S Morrison
Review and Concurrence

7/7/2021
Date

Eleven liquid samples were submitted under Analytical Service Request (ASR) 1248.01 and analyzed by ICP-OES. The samples were analyzed as is and diluted in 5% HNO3 prior to analysis. Neither the sample nor any of the dilutions were filtered.

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

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

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

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

Preparation Blank (PB):

I blank was prepared with the samples containing only reagents. All AOI were within the acceptance criteria of <EQL (estimated quantitation level), <50% regulatory decision level, or less than ≤10% of the concentration in the samples.

Laboratory Control Sample (LCS):

An LCS was prepared along with the samples containing reagents and spike solutions. Recovery values are listed for all analytes included in the LCS that were measured at or above the EQL. Recovery values for the AOI meeting this requirement were within the acceptance criterion of 80% to 120%.

Replicate Relative Percent Difference (RPD):

A duplicate of sample 21-0865 was prepared and analyzed. RPDs are listed for all analytes that were measured at or above the EQL. RPDs for the AOI meeting this requirement ranged from 3.0% to 6.6% and were within the acceptance criterion of ≤20% for liquid samples.

Initial/Continuing Calibration Verification (ICV/CCV):

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

Initial/Continuing Calibration Blank (ICB/CCB):

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

Low-Level Standard (LLS):

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

Interference Check Standard (ICS/SST):

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

Analytical Spikes:

Analytical spikes were conducted on sample 21-0864. Recovery values are listed for all analytes in the spike that were measured at or above the EQL, and that had a spike concentration ≥25% of that in the sample. Recovery values for the AOI meeting this requirement were between 101% and 111% in the AS-A, 106%-107% in the AS-B, 103% in the AS-A(torr) and 101% in the AS-B(torr) which are all within the acceptance criterion of 80% to 120%.

Serial Dilution (SD):

Five-fold serial dilution was conducted on sample 21-0864. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds for the AOI meeting this requirement ranged from 0.3% to 10.0% and were within the acceptance criterion of ≤10%.

Other QC:

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

Comments:

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

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

| | | Run Date > | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 |
|---|---|---|---|---|---|---|---|---|---|
| | | Process Factor > | 1.0 | 49.4 | 244.5 | 244.8 | 248.2 | 251.1 | 247.7 |
| | | RPL/LAB > | 405 Diluent | BLK-0864 | 21-0864 | 21-0865 | | 21-1070 | 21-1071 |
| Instr. Det. Limit (IDL) | Est. Quant. Limit (EQL) | Client ID > | Lab Diluent | Reagents Only | TI100-FEED-COMP | TI100-EFF-COMP | | TI100-L-F2-A | TI100-L-F4-A |
| (µg/mL) | (µg/mL) | (Analyte) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) |
| 0.0276 | 0.276 | Al | -- | -- | 10,200 | 10,300 | 9,890 | 9,560 | 9,350 |
| 0.0005 | 0.005 | Ba | -- | [0.22] | [0.62] | -- | -- | -- | [0.17] |
| 0.0233 | 0.233 | Ca | -- | [1.5] | [15] | [12] | [7.3] | [13] | [8.1] |
| 0.0027 | 0.027 | Cd | [0.0035] | [0.16] | 8.13 | 6.79 | [6.6] | [5.2] | [5.8] |
| 0.0017 | 0.017 | Fe | [0.0025] | [0.69] | 18.9 | 16.1 | 15.1 | 15.0 | 14.3 |
| 0.0500 | 0.500 | K | [0.066] | [4.8] | 3,930 | 3,790 | 3,670 | 3,700 | 3,610 |
| 0.0171 | 0.171 | Na | [0.032] | [2.7] | 148,000 | 145,000 | 140,000 | 141,000 | 138,000 |
| 0.0030 | 0.030 | Nb | -- | -- | -- | [2.2] | [1.7] | [3.1] | [3.0] |
| 0.1021 | 1.021 | P | -- | -- | 889 | 860 | 817 | 829 | 808 |
| 0.0399 | 0.399 | Pb | -- | -- | [13] | -- | -- | -- | -- |
| 0.1773 | 1.773 | S | -- | -- | 2,310 | 2,260 | 2,190 | 2,230 | 2,110 |
| 0.0003 | 0.003 | Sr | -- | -- | [0.097] | -- | -- | -- | -- |
| 0.0120 | 0.120 | Th | -- | -- | -- | -- | -- | -- | -- |
| 0.0676 | 0.676 | U | -- | -- | -- | [21] | [19] | -- | -- |
| 0.0066 | 0.066 | Zn | -- | [2.2] | [9.6] | [2.4] | [1.6] | [2.9] | [1.7] |
| 0.0022 | 0.022 | Zr | -- | -- | -- | [1.9] | [1.9] | [1.2] | [1.2] |
| Other Analytes | | | | | | | | | |
| 0.0030 | 0.030 | Ag | -- | -- | [1.1] | [1.3] | [1.3] | [0.76] | [0.75] |
| 0.1576 | 1.576 | As | -- | -- | -- | -- | -- | -- | -- |
| 0.0078 | 0.078 | Au | -- | -- | -- | -- | -- | -- | -- |
| 0.0120 | 0.120 | B | [0.015] | -- | 79.8 | 30.5 | [28] | 84.0 | 82.4 |
| 0.0002 | 0.002 | Be | -- | -- | [0.058] | -- | -- | -- | -- |
| 0.0546 | 0.546 | Bi | -- | -- | -- | -- | -- | -- | -- |
| 0.0448 | 0.448 | Ce | -- | -- | -- | -- | -- | -- | -- |
| 0.0071 | 0.071 | Co | -- | -- | -- | -- | -- | -- | -- |
| 0.0047 | 0.047 | Cr | -- | -- | 592 | 582 | 553 | 557 | 534 |
| 0.0036 | 0.036 | Cu | -- | [0.18] | [8.1] | [7.2] | [7.1] | [6.1] | [6.5] |
| 0.0034 | 0.034 | Dy | -- | -- | -- | -- | -- | -- | -- |
| 0.0010 | 0.010 | Eu | -- | -- | -- | -- | -- | -- | -- |
| 0.0152 | 0.152 | Ga | -- | -- | -- | -- | [3.9] | -- | [4.2] |
| 0.0048 | 0.048 | Hf | -- | -- | -- | -- | [2.1] | -- | -- |
| 0.0030 | 0.030 | La | -- | -- | -- | -- | -- | -- | -- |
| 0.0028 | 0.028 | Li | -- | [0.15] | [0.85] | -- | -- | [0.80] | -- |
| 0.0008 | 0.008 | Lu | -- | -- | -- | -- | -- | -- | -- |
| 0.0023 | 0.023 | Mg | -- | -- | -- | [0.58] | -- | [1.7] | -- |
| 0.0009 | 0.009 | Mn | -- | -- | [1.2] | [1.2] | [1.1] | [1.2] | [1.0] |
| 0.0094 | 0.094 | Mo | -- | -- | 47.5 | 45.4 | 43.8 | 44.0 | 42.3 |
| 0.0188 | 0.188 | Nd | -- | -- | -- | -- | -- | -- | -- |
| 0.0074 | 0.074 | Ni | -- | [2.0] | 21.1 | 20.7 | [18] | 18.9 | 19.1 |
| 0.0170 | 0.170 | Pd | -- | -- | [5.6] | [4.5] | [5.7] | [4.9] | -- |
| 0.0162 | 0.162 | Rh | -- | -- | [4.3] | [6.7] | -- | [4.6] | [6.6] |
| 0.0158 | 0.158 | Ru | -- | -- | [7.0] | [8.0] | [8.0] | [7.6] | [7.1] |
| 0.0919 | 0.919 | Sb | -- | -- | -- | -- | -- | -- | -- |
| 0.0007 | 0.007 | Sc | -- | -- | -- | -- | -- | -- | -- |
| 0.2271 | 2.271 | Se | -- | -- | -- | -- | -- | -- | -- |
| 0.0136 | 0.136 | Si | [0.049] | [2.2] | 128 | 96.2 | 81.3 | 103 | 115 |
| 0.0212 | 0.212 | Sm | -- | -- | -- | -- | -- | -- | -- |
| 0.0446 | 0.446 | Sn | -- | -- | [11] | [23] | [15] | [11] | [13] |
| 0.0242 | 0.242 | Ta | -- | -- | -- | -- | -- | -- | -- |
| 0.0110 | 0.110 | Tb | -- | -- | -- | -- | -- | -- | -- |
| 0.0412 | 0.412 | Te | -- | -- | -- | -- | -- | -- | -- |
| 0.0014 | 0.014 | Ti | -- | -- | -- | -- | -- | -- | -- |
| 0.0885 | 0.885 | Tl | -- | -- | -- | -- | -- | -- | -- |
| 0.0046 | 0.046 | Tm | -- | -- | -- | -- | -- | -- | -- |
| 0.0032 | 0.032 | V | -- | -- | -- | -- | -- | -- | -- |
| 0.0249 | 0.249 | W | -- | -- | 67.5 | 71.7 | 68.0 | 63.0 | 65.1 |
| 0.0006 | 0.006 | Y | -- | -- | -- | -- | -- | -- | -- |

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

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

Results from C0875 ASR-1248.01 Westesen.xlsm

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

| | | Run Date > | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 | 6/21/2021 |
|---|---|---|---|---|---|---|---|---|---|
| | | Process Factor > | 247.2 | 241.8 | 241.8 | 249.6 | 249.8 | 244.9 | 247.7 |
| | | RPL/LAB > | 21-1072 | 21-1073 | 21-1074 | 21-1075 | 21-1076 | 21-1077 | 21-1078 |
| Instr. Det. Limit (IDL) | Est. Quant. Limit (EQL) | Client ID > | TI100-L-F6-A | TI100-L-F8-A | TI100-L-F10-A | TI100-L-F12-A | TI100-L-F14-A | TI100-L-F17-A | TI100-L-F22-A |
| (µg/mL) | (µg/mL) | (Analyte) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) | (µg/mL) |
| 0.0276 | 0.276 | Al | 9,500 | 9,770 | 9,420 | 9,530 | 9,670 | 9,790 | 10,200 |
| 0.0005 | 0.005 | Ba | -- | [0.18] | [0.11] | -- | [0.19] | [0.12] | [0.15] |
| 0.0233 | 0.233 | Ca | [9.3] | [13] | [11] | [11] | [11] | [10] | [11] |
| 0.0027 | 0.027 | Cd | 6.97 | 6.72 | [6.4] | [6.4] | 6.94 | 7.51 | 7.57 |
| 0.0017 | 0.017 | Fe | 16.0 | 18.8 | 16.5 | 16.0 | 16.1 | 15.7 | 16.8 |
| 0.0500 | 0.500 | K | 3,740 | 3,780 | 3,670 | 3,740 | 3,700 | 3,750 | 3,880 |
| 0.0171 | 0.171 | Na | 142,000 | 144,000 | 140,000 | 142,000 | 145,000 | 143,000 | 144,000 |
| 0.0030 | 0.030 | Nb | [1.4] | [1.0] | [1.8] | -- | [0.77] | -- | [0.89] |
| 0.1021 | 1.021 | P | 785 | 823 | 802 | 790 | 832 | 821 | 851 |
| 0.0399 | 0.399 | Pb | -- | -- | -- | -- | -- | -- | -- |
| 0.1773 | 1.773 | S | 2,260 | 2,320 | 2,260 | 2,200 | 2,230 | 2,140 | 2,250 |
| 0.0003 | 0.003 | Sr | -- | -- | -- | -- | -- | -- | -- |
| 0.0120 | 0.120 | Th | -- | -- | -- | -- | -- | -- | -- |
| 0.0676 | 0.676 | U | [22] | [18] | [22] | [21] | [19] | [17] | [18] |
| 0.0066 | 0.066 | Zn | -- | [1.6] | [11] | -- | [1.8] | -- | [2.1] |
| 0.0022 | 0.022 | Zr | [1.2] | [1.4] | [1.1] | [1.2] | [1.4] | [1.0] | [1.1] |
| Other Analytes | | | | | | | | | |
| 0.0030 | 0.030 | Ag | [1.8] | [1.7] | [1.7] | [1.6] | [1.0] | [1.2] | [0.76] |
| 0.1576 | 1.576 | As | -- | -- | -- | -- | -- | -- | -- |
| 0.0078 | 0.078 | Au | -- | -- | -- | -- | -- | -- | -- |
| 0.0120 | 0.120 | B | 89.3 | 64.1 | 83.2 | 80.0 | 70.0 | 68.5 | 65.4 |
| 0.0002 | 0.002 | Be | -- | -- | -- | -- | -- | -- | -- |
| 0.0546 | 0.546 | Bi | -- | -- | -- | -- | -- | -- | -- |
| 0.0448 | 0.448 | Ce | -- | -- | -- | -- | -- | -- | -- |
| 0.0071 | 0.071 | Co | -- | -- | -- | -- | -- | -- | -- |
| 0.0047 | 0.047 | Cr | 572 | 570 | 556 | 551 | 559 | 550 | 561 |
| 0.0036 | 0.036 | Cu | [6.8] | [7.3] | [7.3] | [7.4] | [7.5] | [7.4] | [7.6] |
| 0.0034 | 0.034 | Dy | -- | -- | -- | -- | -- | -- | -- |
| 0.0010 | 0.010 | Eu | -- | -- | -- | -- | -- | -- | -- |
| 0.0152 | 0.152 | Ga | -- | -- | [4.6] | -- | -- | [4.5] | -- |
| 0.0048 | 0.048 | Hf | -- | -- | -- | -- | -- | -- | -- |
| 0.0030 | 0.030 | La | -- | -- | -- | -- | -- | -- | -- |
| 0.0028 | 0.028 | Li | [0.76] | [0.84] | [0.80] | [0.84] | [0.77] | [0.78] | [0.76] |
| 0.0008 | 0.008 | Lu | -- | -- | -- | -- | -- | -- | -- |
| 0.0023 | 0.023 | Mg | -- | -- | -- | -- | -- | -- | -- |
| 0.0009 | 0.009 | Mn | [1.2] | [1.2] | [1.1] | [1.2] | [1.1] | [1.2] | [1.2] |
| 0.0094 | 0.094 | Mo | 44.5 | 45.2 | 43.5 | 42.8 | 43.8 | 42.8 | 43.9 |
| 0.0188 | 0.188 | Nd | -- | -- | -- | -- | -- | -- | -- |
| 0.0074 | 0.074 | Ni | [18] | 18.5 | 18.2 | 19.1 | 18.8 | 20.6 | 20.4 |
| 0.0170 | 0.170 | Pd | [5.0] | [4.7] | [4.6] | [5.2] | [5.9] | [4.5] | [6.4] |
| 0.0162 | 0.162 | Rh | -- | -- | [6.6] | [6.3] | [4.9] | [5.9] | [6.5] |
| 0.0158 | 0.158 | Ru | [7.0] | [7.5] | [6.6] | [6.1] | [6.8] | [6.6] | [8.9] |
| 0.0919 | 0.919 | Sb | -- | -- | -- | -- | -- | -- | -- |
| 0.0007 | 0.007 | Sc | -- | -- | -- | -- | -- | -- | -- |
| 0.2271 | 2.271 | Se | -- | -- | -- | -- | -- | -- | -- |
| 0.0136 | 0.136 | Si | 88.6 | 105 | 85.0 | 93.0 | 117 | 120 | 128 |
| 0.0212 | 0.212 | Sm | -- | -- | -- | -- | -- | -- | -- |
| 0.0446 | 0.446 | Sn | [18] | [13] | [17] | -- | [12] | -- | [15] |
| 0.0242 | 0.242 | Ta | -- | -- | -- | -- | -- | -- | -- |
| 0.0110 | 0.110 | Tb | -- | -- | -- | -- | -- | -- | -- |
| 0.0412 | 0.412 | Te | -- | -- | -- | -- | -- | -- | -- |
| 0.0014 | 0.014 | Ti | -- | -- | -- | -- | -- | -- | -- |
| 0.0885 | 0.885 | Tl | -- | -- | -- | -- | -- | -- | -- |
| 0.0046 | 0.046 | Tm | -- | -- | -- | -- | -- | -- | -- |
| 0.0032 | 0.032 | V | -- | -- | -- | -- | -- | -- | -- |
| 0.0249 | 0.249 | W | 66.8 | 73.4 | 67.6 | 66.7 | 69.9 | 72.9 | 69.1 |
| 0.0006 | 0.006 | Y | -- | -- | -- | -- | -- | -- | -- |

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

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

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

**QC Performance 06/21/2021**

| Criteria > | ≤ 20% | 80%-120% | 75%-125% | 80%-120% | 80%-120% | 80%-120% | 80%-120% | ≤ 10% |
|---|---|---|---|---|---|---|---|---|
| **QC ID >** | **DUP-0865** | **LCS/BS** | **MS-0865** | **21-0864 + AS-A** | **21-0864 + AS-B** | **21-0864 + AS-A (Tor)** | **21-0864 + AS-B (Tor)** | **21-0864 5-fold Serial Dil** |
| **Analytes** | **RPD (%)** | **%Rec** | **%Rec** | **%Rec** | **%Rec** | **%Rec** | **%Rec** | **%Diff** |
| **Al** | 3.6 | 110 | 89 | 106 | | | | 1.1 |
| **Ba** | | 113 | 102 | 110 | | | | |
| **Ca** | | 111 | 102 | 111 | | | | |
| **Cd** | | 117 | 103 | 107 | | | | |
| **Fe** | 6.6 | 115 | 101 | 111 | | | | |
| **K** | 3.2 | 106 | 106 | 111 | | | | 10.0 |
| **Na** | 3.4 | 117 | nr | nr | | | | 0.3 |
| **Nb** | | | | | | | 101 | |
| **P** | 5.2 | 112 | 97 | 107 | | | | |
| **Pb** | | 114 | 101 | 106 | | | | |
| **S** | 3.0 | 111 | 98 | | 106 | | | 2.7 |
| **Sr** | | 105 | 93 | 101 | | 103 | | |
| **Th** | | 112 | 101 | | 107 | | | |
| **U** | | | | | 107 | | | |
| **Zn** | | | | 110 | | | | |
| **Zr** | | | | 108 | | | | |
| **Other Analytes** | | | | | | | | |
| **Ag** | | | | 99 | | | | |
| **As** | | | | 112 | | | | |
| **Au** | | | | | | | 107 | |
| **B** | | 113 | 97 | 108 | | | | |
| **Be** | | 111 | 99 | 107 | | | | |
| **Bi** | | | | 101 | | | | |
| **Ce** | | 106 | 94 | | 100 | | | |
| **Co** | | | | 108 | | | | |
| **Cr** | 5.2 | 112 | nr | 106 | | | | 2.5 |
| **Cu** | | 119 | 108 | 116 | | | | |
| **Dy** | | | | | 105 | | | |
| **Eu** | | | | | 104 | 106 | | |
| **Ga** | | | | | | 110 | | |
| **Hf** | | | | | | | 111 | |
| **La** | | 108 | 97 | | 102 | | | |
| **Li** | | 112 | 108 | 111 | | | | |
| **Lu** | | | | | | 111 | | |
| **Mg** | | 114 | 102 | 110 | | | | |
| **Mn** | | 115 | 102 | 112 | | | | |
| **Mo** | 3.5 | 110 | 98 | 105 | | | | |
| **Nd** | | 110 | 99 | | 106 | | | |
| **Ni** | | 114 | 100 | 108 | | | | |
| **Pd** | | | | | 100 | | | |
| **Rh** | | | | | 102 | | | |
| **Ru** | | | | | 103 | | | |
| **Sb** | | | | 106 | | | | |
| **Sc** | | | | | | 108 | | |
| **Se** | | | | 109 | | | | |
| **Si** | 16.8 | 106 | 102 | 116 | | | | |
| **Sm** | | | | | | 109 | | |
| **Sn** | | | | 103 | | | | |
| **Ta** | | | | 107 | | | | |
| **Tb** | | | | | | 106 | | |
| **Te** | | | | | 105 | | | |
| **Ti** | | | | 110 | | | | |
| **Tl** | | | | 103 | | | | |
| **Tm** | | | | | | 110 | | |
| **V** | | | | 104 | | | | |
| **W** | 5.2 | | | 104 | | | | |
| **Y** | | | | 105 | | 105 | | |

*Shaded results are outside the acceptance criteria.*

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

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

filename 21-0864 Westesen
6/8/2021

Client: Westesen Project: 77636
ASR 1248 WP#: NH0135

Prepared by: Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2021.06.08 11:18:26 -07'00'

Technical Reviewer: Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2021.06.08 10:44:20 -07'00'

| | |
|---|---|
| Procedures: | RPG-CMC-450, Rev. 3 Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS) Spectrometry |
| M&TE: | Gamma detectors E,T |
| Count dates: | 3-Jun-21 |

**Measured Activity, µCi/mL ± 1s**

| RPL ID: | 21-0864 | | 21-0865 | |
|---|---|---|---|---|
| Sample ID: | TI100-FEED-COMP | | TI100-EFF-COMP | |
| **Isotope** | | | | |
| **Co-60** | <8.5E-04 | | **3.93E-04** | **± 2%** |
| **Sn-126** | <3.5E-02 | | **3.61E-04** | **± 7%** |
| **Sb-126** | <4.2E-03 | | **2.56E-04** | **± 2%** |
| **Cs-137** | **1.54E+02** | **± 2%** | **2.17E-02** | **± 2%** |
| **Eu-154** | <4.2E-03 | | **2.79E-05** | **± 12%** |
| **Am-241** | <2.1E-01 | | **2.48E-04** | **± 12%** |

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

filename 21-0864 Westesen
7/19/2021

Client: Westesen Project: 77636
ASR 1248

Prepared by: C Soderquist 7-21-2021

Technical Reviewer: T Trang-Le 7/21/21

Procedures:
RPG-CMC-408, Rev 2, Total Alpha and Beta Analysis
RPG-CMC-4001, Rev 1, Source Preparation for Gross Alpha and Gross Beta Analysis
RPG-CMC-4017, Rev 0, Measurement of Actinides and Sr-90 in Environmental Water Samples
RPG-CMC-496, Rev 1, Coprecipitation Mounting of Actinides for Alpha Spectroscopy
RPG-CMC-422, Rev 2, Solutions Analysis: Alpha spectrometry

M&TE: Ludlum alpha counters, alpha spectrometry counting system
Count dates: June 17 -July 14, 2021

| | | Measured Concentration, µCi/mL ± 1s | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Sample** | lab ID | **Gross Alpha** | | **Np-237** | | **Pu-238** | | **Pu-239+240** | | sum of alpha emitters |
| **TI100-FEED-COMP** | 21-0864 | 1.15E-3 | ± 13% | 4.59E-5 | ± 3% | 8.67E-5 | ± 3% | 7.39E-4 | ± 2% | 8.71E-4 |
| **TI100-EFF-COMP** | 21-0865 | 7.85E-4 | ± 16% | 3.66E-5 | ± 4% | 6.25E-5 | ± 3% | 4.99E-4 | ± 2% | 5.98E-4 |
| | 21-0865 Dup | 7.33E-4 | ± 17% | 3.74E-5 | ± 4% | 5.98E-5 | ± 4% | 4.96E-4 | ± 3% | 5.93E-4 |
| | RPD | 7% | | 2% | | 4% | | 1% | | |
| **TI100-L-F2-A** | 21-1070 | 4.10E-4 | ± 24% | 1.41E-5 | ± 5% | 4.07E-5 | ± 3% | 3.10E-4 | ± 2% | 3.65E-4 |
| **TI100-L-F4-A** | 21-1071 | 7.34E-4 | ± 17% | 3.92E-5 | ± 3% | 4.89E-5 | ± 3% | 4.26E-4 | ± 2% | 5.14E-4 |
| **TI100-L-F6-A** | 21-1072 | 8.44E-4 | ± 16% | 4.07E-5 | ± 3% | 7.05E-5 | ± 4% | 5.89E-4 | ± 3% | 7.00E-4 |
| **TI100-L-F8-A** | 21-1073 | 5.86E-4 | ± 20% | 4.36E-5 | ± 3% | 7.76E-5 | ± 3% | 6.47E-4 | ± 2% | 7.68E-4 |
| **TI100-L-F10-A** | 21-1074 | 6.62E-4 | ± 18% | 4.54E-5 | ± 3% | 8.12E-5 | ± 3% | 6.73E-4 | ± 2% | 8.00E-4 |
| **TI100-L-F12-A** | 21-1075 | 7.96E-4 | ± 16% | 4.84E-5 | ± 3% | 7.60E-5 | ± 4% | 6.78E-4 | ± 3% | 8.03E-4 |
| **TI100-L-F14-A** | 21-1076 | 1.08E-3 | ± 14% | 4.29E-5 | ± 3% | 7.93E-5 | ± 4% | 6.90E-4 | ± 3% | 8.12E-4 |
| **TI100-L-F17-A** | 21-1077 | 6.45E-4 | ± 19% | 4.09E-5 | ± 3% | 7.77E-5 | ± 4% | 6.40E-4 | ± 3% | 7.59E-4 |
| **TI100-L-F22-A** | 21-1078 | 8.05E-4 | ± 16% | 4.33E-5 | ± 3% | 7.46E-5 | ± 4% | 6.33E-4 | ± 3% | 7.50E-4 |

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

filename 21-0864 Westesen
7/19/2021

| Sample | lab ID | Measured Concentration, µCi/mL ± 1s | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | **Gross Alpha** | | **Np-237** | | **Pu-238** | | **Pu-239+240** | |
| | Reagent spike | 103% | | 92% | | -- | | 90% | |
| | Matrix spike | 77% | | 86% | | -- | | 93% | |
| | lab blank | -9.7E-7 | ± 40% | -8.2E-8 | ± 60% | 1.1E-6 | ± 27% | 1.2E-6 | ± 25% |

These samples were received at the analytical lab on April 5, 2021 for measurement of gamma emitters, Np-237, plutonium alpha, and metals by ICP-OES. Gamma emitters and ICP metals were reported separately.

The samples had high dissolved solids, and the volume of each sample was only a few mL. The gross alpha activity was measured, but the high dissolved solids limited the volume counted to only 0.1 mL, and consequently the counting error is high. The gross alpha results are probably biased a low by the high dissolved solids.

Neptunium and plutonium were chemically separated from each sample, then mounted for alpha spectrometry by coprecipitation. No other alpha emitters were measured. The sum of Np-237, Pu-239+240, and Pu-238 agrees reasonably well with the gross alpha, although the gross alpha data has high uncertainty.

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

Filename ASR 1248 alpha emitters narrative
July 21, 2021

Prepared by: C. Soderquist 7-21-2021

Concur: T Trang-Le 7/21/21

## Alpha Emitters, ASR 1248

Eleven samples under this ASR were delivered to the analytical lab on April 5, 2021. Two samples required gamma spectrometry. All of the others required measurement of neptunium-237, plutonium alpha, and metals by ICP-OES. Data for ICP-OES metals is reported separately. An amended ASR was issued on June 4, 2021 to include neptunium and plutonium measurement on all samples.

Each sample consisted of several mL of salty aqueous solution. Gamma emitters were measured in the raw sample, as received. Gamma emitters are reported separately.

Before neptunium and plutonium could be measured, the approximate alpha activity had to be known, so the gross alpha activity was measured. The samples were diluted (to reduce the dissolved solids) and a small volume of each dilution was evaporated onto a steel counting disk. The counting disks were counted for gross alpha emission (procedure RPG-CMC-408). The samples have low alpha activity and the gross alpha results are near the detection limit and have high uncertainty. Additionally, the gross alpha results are probably biased several percent low by the small amount of mass on each counting disk, which absorbs part of the alpha emission.

Neptunium and plutonium were chemically separated using anion exchange in hydrochloric acid (procedure RPG-CMC-4017, Measurement of actinides and Sr-90 in environmental water samples). Each raw sample was measured into a beaker and evaporated with concentrated nitric acid (to oxidize neptunium and plutonium), then the residue was dissolved in concentrated hydrochloric acid. Neptunium and plutonium were then separated by anion exchange in hydrochloric acid. The plutonium measurement uses a $^{242}$Pu tracer. No alpha emitting tracer is available for neptunium measurement. Spikes were run using $^{237}$Np and $^{239}$Pu. The samples were mounted for alpha spectrometry by coprecipitation on 70 µg of $NdF_3$ on a membrane filter (procedure RPG-CMC-496). The samples were counted on alpha spectrometers (procedure RPG-CMC-422). All of the alpha spectra had good resolution and were free of extraneous alpha peaks. Plutonium-239 and -240 have nearly identical alpha energies, too close to resolve, and we report their sum.

The samples have easily measurable neptunium and plutonium. The measured $^{237}$Np and plutonium alpha add up to the measured gross alpha, within the rather high uncertainty of the gross alpha measurement. All of the blanks had negligible activity compared to the samples, and were at or near the detection limit.

The $^{237}$Np reagent spikes have averaged 95%, both reagent and matrix spikes, over the last five years. Neptunium is measured without a tracer. The plutonium-239 spikes have averaged 96% (reagent spikes) and 95% (matrix spikes) over the last five years. Plutonium is measured against a $^{242}$Pu tracer. The tracer recoveries are typically high 90% or more.

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

# Gamma Energy Analysis (GEA)

**Project / WP#:** 77636/NH0135
**ASR#:** 1248.00
**Client:** A. Westesen
**Total Samples:** 2

| RPL ID | Client Sample ID |
|---|---|
| 21-0864 | TI100-FEED-COMP |
| 21-0865 | TI100-EFF-COMP |

| | |
|---|---|
| **Analysis Type:** | GEA- for all positively measured or non-detected isotopes |
| **Sample Processing Prior to Radiochemical Processing/Analysis** | ☒ None<br>☐ Digested as per RPG-CMC-129, Rev. 0 *$HNO_3$-HCl Acid Extraction of Solids Using a Dry Block Heater*<br>☐ Fusion as per RPG-CMC-115, *Solubilization of Metals from Solids Using a KOH-$KNO_3$ Fusion*<br>☐ Other:<br>Preparation may also involve attaining a GEA geometry that is compatible with the calibration geometry. |
| | |
| **Analysis Procedure:** | RPG-CMC-450, Rev. 3 *Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)* |
| **Reference Date:** | None |
| **Analysis Date or Date Range:** | June 3, 2021 |
| **Technician/Analyst:** | T Trang-Le |
| | |
| **Rad Chem Electronic Data File:** | *21-0864 Westesen.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. Also, balance calibration and performance check records.* |
| **M&TE Number(s):** | *Detectors E,T* |

Truc Trang-Le Digitally signed by Truc Trang-Le Date: 2021.06.08 12:52:28 -07'00' / 
**Prepare** **Date**

Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2021.06.08 12:59:01 -07'00' / 
**Reviewer** **Date**

## SAMPLE RESULTS

Activities for all gamma emitters detected in these samples are presented in an attached Excel spreadsheet for ASR 1248.00. All sample results for target isotopes are reported in units of µCi/mL with estimates of the total propagated uncertainty reported at the 1-sigma level.

ASO Project File, ASR 1248.00 has been created for this report including all appropriate supporting records which may include the Pipette Performance Check Worksheet form, standard certificates, laboratory bench records, Shielded Analytical Laboratory Bench Sheet, and Gamma Energy Analysis printouts. Detector calibration records, control charts and balance calibration records can be found in the ASO Records.

### Sample Preparation, Separation, Mounting and Counting Methods

2 mL samples were sent to the counting room for GEA.

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

## QUALITY CONTROL RESULTS

### Tracer:

Tracers are not used for ASO GEA methods.

### Process Blank (PB):

No process blank was prepared 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. Procedure RPG-CMC-450 requires that a counter control source is checked daily and must be within ±3 sigma or ±3% of the control value, whichever is greater. Gamma counting was not performed unless the control counts were within the required limits. Background counts are performed on all gamma detectors at least weekly for either an overnight or weekend count.

## Assumptions and Limitations of the Data

None

## Interferences/Resolution

None.

## Uncertainty

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

## Comments

None

Attachment: Data Report Sample Results for ASR 1248.00.

**Pacific Northwest National Laboratory**

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354
1-888-375-PNNL (7665)

***www.pnnl.gov***