PNWD-3751
WTP-RPT-135, Rev. 1

# Small Column Ion Exchange Testing of Spherical Resorcinol-Formaldehyde Resin for $^{137}Cs$ Removal from Pre-Treated Hanford Tank 241-AN-102 Waste (Envelope C)

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April 2006

Prepared for Bechtel National Inc.
under Contract No. 24590-101-TSA-W0000-00004

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ACCEPTED FOR
WTP PROJECT USE

April 2006

Test specification: 24590-PTF-TSP-RT-04-0005, Rev. 0
Test plan: TP-RPP-WTP-378, Rev. 0
Test exceptions: 24590-PTF-TEF-RT-05-00008
24590-PTF-TEF-RT-05-00011

R&T focus area: Pretreatment
Test Scoping Statement(s): A-212

Battelle—Pacific Northwest Division
Richland, Washington 99352

## COMPLETENESS OF TESTING

*This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-04-0005, Rev. 0 and Test Plan TP-RPP-WTP-378, Rev. 0 and Test Exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.*

**Approved:**

4/26/06

Gordon H. Beeman, Manager
WTP R&T Support Project

Date

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# Terms and Abbreviations

| | |
|---|---|
| AEA | alpha energy analysis |
| AP-101DF | Hanford Tank 241-AP-101 diluted feed |
| ASO | Analytical Support Operations |
| ASR | analytical services request |
| ASTM | American Society for Testing and Materials |
| AV | apparatus volume |
| BNI | Bechtel National, Inc. |
| BS | blank spike |
| BV | bed volume |
| $C/C_o$ | analyte concentration in column effluent (C) divided by analyte concentration in feed ($C_o$) |
| DF | decontamination factor |
| DI | deionized (water) |
| DOE | U.S. Department of Energy |
| DQO | data quality objectives |
| EQL | estimated quantitation limit |
| FMI | Fluid Metering, Inc., Syosset, NY |
| GEA | gamma energy analysis |
| HLW | high-level waste |
| IBC | IBC Advanced Technologies, Inc., American Fork, Utah |
| IC | ion chromatography |
| ICP-AES | inductively coupled plasma-atomic emission spectrometry |
| ICP-MS | inductively coupled plasma-mass spectrometry |
| ILAW | immobilized low-activity waste |
| KPA | kinetic phosphorescence analysis |
| LAW | low-activity waste |
| L/D | length to diameter (ratio) |
| M | molarity, moles/liter |
| MDL | method detection limit |

| | |
|---|---|
| MRQ | minimum reportable quantity |
| MS | matrix spike |
| M&TE | measuring and test equipment |
| NA | not applicable |
| ND | not detected |
| NM | not measured |
| ORP | Office of River Protection |
| PB | preparation blank |
| PNWD | Battelle—Pacific Northwest Division |
| QA | quality assurance |
| QARD | Quality Assurance Requirements and Descriptions (document) |
| QC | quality control |
| QAPjP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RF | resorcinol-formaldehyde |
| RPD | relative percent difference |
| RPP-WTP | River Protection Project-Waste Treatment Plant |
| RPL | Radiochemical Processing Laboratory (PNWD facility) |
| R&T | Research and Technology |
| SAL | Shielded Analytical Laboratory |
| SOW | statement of work |
| TRU | transuranics |
| WTP | Waste Treatment Plant |
| WTPSP | Waste Treatment Plant Support Project |

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# Testing Summary

Spherical resorcinol-formaldehyde (RF) resin, Microbeads Lot 5E-370/641, was selected for testing in a lead-lag column format on actual Hanford tank wastes. This report describes Hanford Tank 241-AN-102 (AN-102) waste pretreatment and Cs ion exchange performance testing with the spherical RF resin, satisfying the requirements of Technical Scoping Statement A-212.

## Objectives

The test objectives were to

- provide sufficient characterization data to evaluate ion exchange performance for spherical RF resin
- perform column testing with pre-treated AN-102 tank waste and determine the loading and elution performance under nominal Waste Treatment Plant (WTP) processing conditions.(a)

These test objectives are further discussed in Table S.1.

**Table S.1.** Test Objectives

| Test Objective | Objective Met? | Discussion |
|---|---|---|
| Provide sufficient characterization data to evaluate ion exchange performance for spherical RF resin. | Yes | The AN-102 tank waste feed, ion exchange effluent, and eluate were characterized for metals, inclusive of Na and Cs, anions, and selected actinides, to determine their fate during processing. The feed Na concentration was 4.94 M, and the Cs concentration was 5.92E-5 M (7.93 mg/L). Most Resource Conservation and Recovery Act (RCRA)-listed metals were also characterized. |
| Perform column testing on AN-102 tank waste and determine load and elution performance. | Yes | Lead- and lag-column Cs load and elution profiles were generated for the pretreated AN-102 tank waste. The lead column was partially loaded with Cs from prior testing with AP-101. The $^{137}$Cs maximum breakthrough was 10% after processing 202 bed volumes (BVs) at 3 BV/h. This loading test condition was more stringent than design conditions where only 100 BVs of Envelope C waste processing is planned at a flowrate of 1.5 BV/h. The lead-column load did not demonstrate a clear breakthrough profile; the Cs breakthrough gradually increased as a function of BV. A breakthrough profile for Cs from the lag column was not obtained because the Cs concentration in the lag-column effluent was essentially constant during the entire test. The composite effluent Cs removal (9.0E-4% $C/C_o$) was 2 orders of magnitude lower than the contract limit of 0.099% $C/C_o$. Both the lead and lag columns were eluted with 30 BVs of 0.5 M nitric acid. The elution proceeded normally with the peak Cs concentration captured in the fifth to sixth BV. Within experimental uncertainty, all Cs loaded on the lead and lag columns was recovered in the eluates. |

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(a) Column performance testing with Hanford tank waste AP-101 was documented in report WTP-RPT-134; spent resin analysis will be provided in a later report.

## Test Exceptions

Specific test details were modified in Test Exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. Table S.2 summarizes the test exceptions to the test plan and provides a discussion of the impacts on the tests.

**Table S.2.** Test Exceptions

| Test Exception ID | Test Exceptions | Discussion |
|---|---|---|
| 24590-PTF-TEF-RT-05-00008 | Modify elution volumes to include range from 15 to 30 BVs. The actual volume will be determined in consultation with the Research and Technology (R&T) lead. | The intent was to match processing conditions to those of SL-644 and to those likely in the WTP. The AP-101 testing on SL-644 used a 15-BV elution. The final SL-644 elution volumes were nominally 30 BVs. For the current test, 30 BVs of eluant were passed through each column. |
| | For the shakedown test only, increase the water rinse following elution to 8 BVs. The water rinse solutions, collected in 1- to 2-BV increments, are to be analyzed for pH using pH paper indicating strips. | This test was to determine how Cs elution was affected by water. This specific test did not apply to AN-102 processing. |
| 24590-PTF-TEF-RT-05-00011 | The interval for lag-column eluate sample collection after the last waste tested will be determined in consultation with the R&T lead. | Round-the-clock coverage for the AN-102 processing operation was estimated to last 1 week. However, the AN-102 load condition continued much longer than anticipated. The lag-column elution had to be conducted on the following week with more limited resources. Eluate samples were collected in 1.4-BV increments from 0 to 10 BVs, one composite from 10 to 22 BVs, and then again in 1.4-BV increments from 22 to 29 BVs processed. |

## Results and Performance Against Success Criteria

Success criteria for the RPP-WTP project were to demonstrate adequate Cs decontamination from the Hanford tank waste and to demonstrate adequate Cs elution to meet follow-on tank waste processing. These are further discussed in Table S.3.

**Table S.3.** Success Criteria

| Success Criteria | Discussion |
|---|---|
| Combined effluent from the second column after processing 60 BVs meets the low-activity waste (LAW) production specification of 0.3 Ci/m$^3$ of immobilized low-activity waste (ILAW). | The $^{137}$Cs concentration of ≤0.3 Ci/m$^3$ in glass requires a decontamination factor of 1160 in the AN-102 matrix. The first 60 BVs of effluent were collected as a composite. Because subsequent effluent collections were virtually identical in activity to the first 60 BVs, a composite of the total was prepared for analysis. The processing of 202 BVs of AN-102 tank waste resulted in a Cs decontamination factor of 111,000, nearly two orders of magnitude greater than that required to meet the contract limit. |
| Elution performance meets process requirements. | The lead column that previously was used to process AP-101 tank waste was eluted with 15 BVs of 0.5 M nitric acid. It was placed into the lag position for AN-102 processing. The Cs bleed from the lag column did not significantly add to the AN-102 effluent $^{137}$Cs concentration where a nearly constant 1.3E-3 µCi/mL $^{137}$Cs was obtained. Both lead and lag columns were eluted following AN-102 processing with 30 BVs of 0.5 M nitric acid. The measured and calculated Cs removed from the columns was >99%. |

## Quality Requirements

Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization and to the approved Test Plan, TP-RPP-WTP-378, Rev. 0.[a] This work was performed to the quality requirements of NQA-1-1989 Part I, "Basic and Supplementary Requirements," NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions* (QARD). These quality requirements were implemented through PNWD's *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements were implemented through WTPSP's Statement of Work (WTPSP-SOW-005) with the Radiochemical Processing Laboratory (RPL) Analytical Service Operations (ASO). PNWD addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNWD's procedure QA-RPP-WTP-604. This review verifies that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives. This review procedure is part of PNWD's WTPSP QA manual.

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(a) SK Fiskum. 2004. *Column Performance Testing of Actual Wastes from Tanks AP-101 and AN-102 Using Spherical Resorcinol Formaldehyde Ion Exchange Resin*, Battelle—Pacific Northwest Division, Richland, WA.

## R&T Test Conditions

This report summarizes the actual AN-102 waste preparation and ion exchange testing using Microbeads spherical RF resin, Lot 5E-370/641. The resin was subsampled and pretreated before testing. Resin pretreatment included washing in 0.5 M $HNO_3$, then converting it to the Na-form and back to the H-form. Subsamples of the pretreated resins were taken for testing. The dry resin mass in each column was determined from a duplicate volume dried under vacuum at $50^{o}C$ to constant mass.

The actual tank waste AN-102 feed preparation mimicked the pretreatment flowsheet at the WTP. Archived samples of AN-102 required compositing, dilution, Sr/TRU(a) precipitation, filtration, and dilution to 5 M Na.

The pretreated AN-102 was processed through the dual column system that had previously been used on simulant AP-101 and actual AP-101 diluted to 5 M Na. Resin bed volumes (BVs) were nominally 11 mL in a 1.5-cm ID glass column with a nominal length-to-diameter (L/D) ratio of 4.2 when the resin was in the Na-form and an L/D of 3.3 when the resin was contracted in the H-form. Load and elution processing was conducted according to nominal plant design and throughput. The AN-102 was processed at 3.0 BV/h; elution was conducted at 1.4 BV/h.

All test conditions delineated by the Test Plan and Test Exceptions were met with the exception that only a 60-mL batch contact sample was maintained, which was less than the 250-mL volume indicated in the Test Plan. All available feed, including most of the volume set aside for batch contact tests, was processed in an effort to obtain a discernable Cs breakthrough profile. A summary of test conditions is provided in Table S.4.

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(a) TRU = transuranics.

**Table S.4. R&T Test-Condition Summary**

| R&T Test Condition | Discussion |
|---|---|
| Prepare a composite of AN-102 actual waste from collected samples and pre-treat the waste according to the nominal plant flowsheet. PNWD was to proceed after the R&T lead evaluated stepwise both the composite material and the diluted material Na concentrations. | These conditions were followed. Seven archived samples of AN-102 were combined to give a 1.17-L composite sample. The Na concentration in the composite sample was measured to be 9.0 M. The composite was diluted with 0.001M NaOH to 5.8 M Na. The Na concentration was adjusted to 6.8 M with concentrated NaOH, and the material was heated to 50°C. Aliquots of 1.5 M strontium nitrate (99 mL) and 1 M sodium permanganate (104 mL) were slowly added. The solution was mixed at temperature for 4 hours. After cooling, the mixture was centrifuged and the centrifuged liquid was decanted and filtered. The Na concentration at this point was measured to be 7.2 M. The filtrate was diluted to 4.9 M Na with 0.001M NaOH, resulting in an ion exchange feed volume of 2.36 L. |
| PNWD will analyze the actual waste test feed, effluent, and eluate for selected analytes. | These conditions were followed. |
| A batch contact sample was to be reserved for testing if needed. | The sample set aside for batch contact testing was added to the ion exchange process feed in an effort to obtain a more complete or definitive breakthrough profile. |
| The resin was to be sampled in accordance with the protocol P1-RF. | This test condition was met. |
| The dry H-form RF resin bed mass was to be determined. | The test plan indicated three specific RF resins and corresponding dry hydrogen form masses for (expanded Na-form) 10-mL resin beds. These resins were not selected by R&T for actual waste testing. Instead, a different resin (Microbeads Lot # 5E-370/641) was used per the Bechtel National, Inc. (BNI) R&T lead direction. The dry H-form mass loaded into each the ion exchange column was determined according to the methodology described in the test plan.. |
| RF resin preconditioning steps were to be conducted in accordance with the protocol P1-RF[a]. Individual steps are not repeated here because of their extensive nature. | Preconditioning outside and inside the column was conducted on each resin bed as described in the protocol and test plan. |
| The ion exchange apparatus was to be built with 1.5-cm ID columns in a lead-lag format. | The ion exchange apparatus was built per the diagram in the test plan with 1.5 cm ID columns obtained from Spectrum Chromatography, Houston, TX). |
| AN-102 processing was to be conducted to exceed 50% Cs breakthrough on the lead column or until available feed was exhausted. Details of processing are not repeated here because of their extensive nature. | Processing was conducted in accordance with the test plan. The R&T lead provided specific direction where ranges were defined as follows:<br>• actual waste feed flowrate at 3 BV/h<br>• feed displacement volume of 2 apparatus volumes (AVs)<br>• water rinse volume of 2 AVs<br>• lead- and lag-column elution volumes of 30 BVs.<br>All available feed was used in the test; however, only 10% $^{137}$Cs breakthrough from the lead column was achieved. |
| Store the eluted resin in water as the H-form between ion exchange tests. | This condition was not applicable; no additional ion exchange test was conducted. Resins were stored in the water rinse solution after AN-102 processing and elution. |

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(a) WTP doc. no. 097893, CA Nash and CE Duffey, August 17, 2004.

## Simulant Use

Simulants were not used for this scope of testing; actual AN-102 pre-treated tank waste was tested.

## Discrepancies and Follow-on Tests

A clear 50% $C/C_o$ Cs breakthrough point was not established with the current test. An approximate 50% $C/C_o$ breakthrough could be estimated from batch contact testing using the effluent AN-102 matrix. Except for Cs content, the compositions of the effluent and feed were virtually identical. Development of the isotherm could be conducted at various (added) Cs concentrations.

# 1.0 Introduction

Forty years of plutonium production at the U.S. Department of Energy (DOE) Hanford Site has left a legacy of liquid waste generated as a byproduct of reprocessing operations. The wastes are a complex mixture composed mostly of sodium nitrate, nitrite, hydroxide, and sulfate, along with a broad spectrum of minor and trace metals, organics, and radionuclides stored in underground storage tanks. The DOE Office of River Protection (ORP) has contracted Bechtel National Incorporated (BNI) to build a processing plant, the River Protection Project-Waste Treatment Plant (RPP-WTP), that will chemically separate the highly radioactive components (specifically Cs/$^{137}$Cs and, in selected cases, Sr/$^{90}$Sr and transuranics [TRU]) of the tank waste from the bulk (non-radioactive) constituents and immobilize the wastes by vitrification. The plant will produce two waste streams: a high-volume low-activity waste (LAW) that is $^{137}$Cs (and $^{90}$Sr and TRU) depleted and a low-volume high-activity waste (HLW) (the $^{137}$Cs, $^{90}$Sr, and TRU-rich fraction). The separation of the wastes into LAW and HLW will occur in pretreatment modules of the plant.

The RPP-WTP contract statement of work specifies ion exchange for removing $^{137}$Cs from tank waste supernatant to ultimately achieve a $^{137}$Cs loading of 0.3 Ci/m$^3$ or less in the immobilized LAW product.[a] Further, the contract specifies that cesium ion exchange will use the elutable SuperLig® 644 (SL-644) resin (registered trademark of IBC Advanced Technologies, Inc.) or the DOE-approved equivalent. SL-644 is solely available through IBC Advanced Technologies. To provide an alternative to this sole-source resin supply, DOE-ORP directed BNI to initiate a three-stage process to select and potentially implement an alternative ion exchange resin for cesium removal in the RPP-WTP.[b]

BNI completed the first step of this process by recommending that resorcinol formaldehyde (RF) resin be pursued as a potential alternative to SL-644.[c] The RF resin is an organic-based resin developed at Westinghouse Savannah River Company in the late 1980s. It was selected as an alternative cesium ion exchange technology for the Initial Pretreatment Module project, and extensive testing was performed to support that project during the late 1980s to early 1990s (Bray et al. 1996; Brown et al. 1995 and 1996; Kurath et al. 1994). Both batch and column testing of the ground-gel RF resin was conducted at Battelle—Pacific Northwest Division (PNWD) and the Savannah River National Laboratory. The resin was found to have a high loading and selectivity for cesium from Hanford Site tank wastes. The cesium could be eluted from the resin under acidic conditions.

BNI completed the second step of this process by developing an implementation plan.[d] BNI completed the first stage of the implementation plan with a spherical RF resin providing the best combination of characteristics required for WTP operations. The WTP Pretreatment Alternative Resin

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(a) DOE Contract No. DE-AC27-01RV14136 (DOE 2000) Section C.7.d.1.iii.

(b) CCN 030290, Letter from CB Reid, Office of River Protection, to RF Naventi, Bechtel National, Inc., dated March 13, 2002.

(c) R Peterson, H Babad, L Bray, J Carlson, F Dunn, A Pajunen, I Papp, and J Watson. 2002. *WTP Pretreatment Alternative Resin Selection* 24590-PTF-RPT-RT-02-001, Rev. 0, Bechtel National, Inc., Richland, WA.

(d) M Thorson. 2002. *Alternative Ion Exchange Resin Supplemental Research and Technology Plan – Case 20,* 24590-PTF-PL-RT-02-002, Rev. 0, Bechtel National, Inc., Richland, WA.

Selection report[(a)] reviewed available literature on RF performance relative to the WTP plant design and identified areas to target additional testing such as:

- Assess column chemical performance with Hanford-typical feeds.
- Address the potential for resin packing induced by swelling in the plant column with resultant high pressure drops, potential resin structural breakdown, and potential channeling.
- Determine elution performance for resin to achieve desired decontamination factor, and obtain data on cesium bleed from subsequent loading cycles.
- Determine concentration of residual metals, cesium, and other contaminants on spent resin as a function of the last elution conditions that may affect disposal requirements.

As part of the first stage of RF testing, spherical RF resin performance for Cs-removal was assessed using batch contact and column testing under the A-222 scoping statement (Fiskum et al. 2004b). The spherical resin was found to have good capacity, good kinetics, better elution, and better hydraulic properties than the ground-gel RF resin.

Based on first-stage test results, DOE-ORP directed BNI to initiate testing designed to evaluate spherical RF resin for cold commissioning in the WTP.[(b)] Technical scoping statements A-204, A-212, and A-235 from these plans address testing of the spherical RF resin with actual waste samples from Hanford waste tanks AP-101 and AN-102, as well as analysis of the spent resin.

This report summarizes the Hanford Tank 241-AN-102 waste pretreated to remove Sr/TRU constituents and diluted to 4.9 M $Na^+$ (hereafter referred to as AN-102) and the ion exchange testing of AN-102. Cesium load and elution performance was evaluated under nominal baseline plant operation conditions. Approximately 2.25 L of AN-102 were processed through the two 11-mL ion exchange resin beds in a lead-lag-column format. Cesium breakthrough and elution curves were generated. The fates of U, Pu, K, and other metals during ion exchange processing were evaluated. The effect of residual Cs on the resin beds and associated bleed into the effluent was also evaluated from both the lead column (that contained residual Cs from AP-101 processing) and the lag column (eluted resin bed from AP-101 processing).

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(a) Ibid footnote (c) previous page.

(b) CCN 083069, Letter from RJ Schepens, Office of River Protection, to JP Henschel, Bechtel National, Inc., “Direction to Perform Required Cesium Ion Exchange Alternative Resin Testing,” 03-WEC-006, effective date February 25, 2004.

# 2.0 Experimental

This section describes the spherical RF resin, resin preparation, actual Hanford Tank 241-AN-102 waste sampling, pre-treatment, and ion exchange processing.

## 2.1 Spherical RF Resin

The spherical RF resin used in the actual waste testing came from Microbeads (Skedsmokorset, Norway), Lot Number 5E-370/641. This resin lot was produced on May 24, 2005, in a 50-gal production batch. General manufacturing conditions of this resin are protected by patent; specific lot preparation conditions were forwarded under separate letter to the BNI R&T lead by Microbeads. The resin lot was received on June 6, 2005. The resin (~1.75 L) was received in the hydrogen-form, stored under water in a glass 2-L bottle. The headspace was minimal.

### 2.1.1 Resin Sampling and Pretreatment

Sub-samples of the RF resin were taken using the coring technique consistent with the American Society for Testing and Materials (ASTM) Method 2687, *Standard Practice for Sampling Particulate Ion-Exchange Materials* (ASTM 2001). After each sub-sampling event, the headspace was purged with nitrogen gas. A 63-mL settled-resin volume aliquot was sampled on July 10, 2005 (the settled-resin volume was measured in a graduated cylinder with tapping/vibration to constant volume).

The resin was pretreated according to Protocol P1-RF, *Hanford RPP-WTP Alternate Resin Program - Protocol P1-RF: Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column*.(a) The resin sub-sample was transferred to a beaker and soaked in a 5× volume (five times the settled resin volume) of water for 30 minutes with agitation every 10 minutes. After settling, the water was decanted, and a 5× volume of 1 M NaOH was added. The resin was soaked for 30 minutes with agitation every 10 minutes, and the solution pH was confirmed to exceed 14. The resin soak was continued overnight, and the final solution pH was confirmed to exceed 14. The solution was decanted, and a 3× volume of DI water was added; the slurry was again agitated every 10 minutes for 30 minutes. The water was decanted after settling, and fresh rinse water was added two successive times with similar agitation. The final solution pH was measured at 12.5 with medium-range pH paper. The water was decanted, and a 10× volume of 0.5 M nitric acid was added with agitation every 10 minutes for 2 hours, then the resin was allowed to settle. The acid was decanted and the resin rinsed with 3× volume of DI water with agitation every 10 minutes for 30 minutes. The water rinse was repeated for a total of three successive times. The final solution pH was 5. The relaxed H-form resin volume was measured similarly to the starting material in a graduated cylinder at 79.5 mL, exhibiting a bulk expansion factor of 26% from the as-received form.

### 2.1.2 Physical Properties(b)

A portion of the pretreated H-form resin was dried under nitrogen to a free-flowing state for microscopy examination. Surface and cross-section micrographs are provided in Figure 2.1. The dark and light surface color variation was also evident macroscopically. Generally, the particles appeared

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(a) WTP doc. no. 097893, CA Nash and CE Duffey, August 17, 2004.

(b) Pre-treated resin physical properties were evaluated under a different technical scoping statement, A-225. Technical details supporting Technical Scoping Statement A-225 will be reported separately.

homogenously spherical. Particle cross sections were obtained using a scalpel to cleave several resin beads. The cross-section micrographs show a uniform density, indicating that RF formation occurred through the entire sphere.

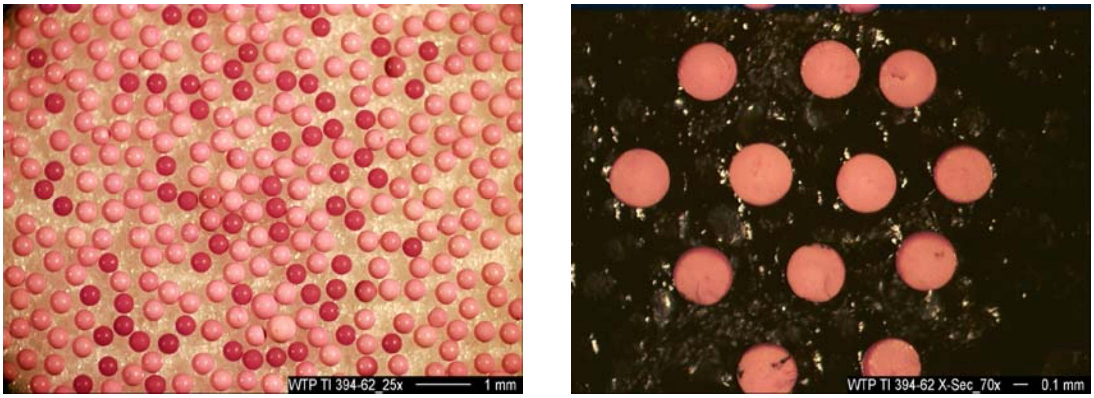

**Figure 2.1.** Micrographs of Pretreated H-Form Spherical RF Resin (Lot 5E-370/641) Surface (25×) and Cross-Section (70×)

The mean particle diameters were measured using a MicroTrac S-3000 with water and 0.001 M NaOH dispersion fluids for the H-form and Na-form resins, respectively. The average particle diameters on a volume basis were 421 microns in the H-form and 452 microns in the Na-form. These diameters correlated to a mean particle volume of 0.039 $mm^3$ and 0.048 $mm^3$, respectively, which reflects a 23% volume increase from the H-form to the Na-form. Additional particle size details are provided in Table 2.1.

**Table 2.1.** Pre-Treated Resin Particle-Size-Distribution Summary

| | **Volume Distribution (microns)** | | | | **Number Distribution (microns)** | | | | **Area Dist. (microns)** |
|---|---|---|---|---|---|---|---|---|---|
| **Resin ID** | **$m_v$** | **sd** | **Low 5%** | **High 90%** | **$m_n$** | **sd** | **Low 5%** | **High 90%** | **$m_a$** |
| 5E-370/641 H-form | 421 | 78 | 307 | 538 | 377 | 59 | 288 | 457 | 403 |
| 5E-370/641 Na-form | 452 | 83 | 333 | 575 | 406 | 70 | 308 | 493 | 434 |

$m_v$ = mean diameter volume distribution
$m_n$ = mean diameter number distribution
$m_a$ = mean diameter area distribution
sd = standard deviation
Low 5% = 5% of particles are below this value.
High 90% = 10% of particles are greater than this value.
Data generated from a Micro-Trac S-3000.

The particle size data reported in Table 2.1 are consistent with observations from the microscopy examination of the H-form resin. The micrographs presented in Figure 2.1 suggest the H-form resin particles are generally of uniform size and typically in the range 370 to 430 microns. The largest particle observed (near the bottom center of the 25× micrograph) has a diameter of approximately 560 microns.

The column diameter should be at least 20-times larger than the particle diameter to minimize wall effects (Korkisch 1989, p. 39). The inner diameter of the ion exchange columns was 1.5 cm. Therefore, the column diameter was nominally 33 times larger than the Na-form particle diameter, indicating that wall effects were minimized during ion exchange processing.

### 2.1.3 Dry Resin Mass

The pretreated H-form RF resin was subdivided into three 8-mL aliquots for ion exchange testing. The first two aliquots were used to fill the lead and lag columns. The third aliquot was used to determine the dry resin mass. The 8-mL volume fraction of H-form resin was transferred to a tared glass beaker. Excess water was removed, and the damp resin was dried under vacuum at $50^{o}$C to constant mass. Constant mass was defined as a mass change of <0.5% in a 7-h period. The dry H-form resin mass representing the 8-mL wet settled resin bed volume (BV) was 2.870 g. Thus the H-form resin packing density was 0.36 g/mL.

## 2.2 241-AN-102 Tank Waste

This section describes the Tank 241-AN-102 waste sampling, receipt, homogenization, and pre-treatment.

### 2.2.1 Sampling Event Description

Thirty grab samples were taken from Riser 022 in Hanford Tank 241-AN-102 between the dates of August 2, 2000, and August 14, 2000, to provide approximately 10 L of supernatant and 1 kg of wet sludge. The grab samples were taken by lowering a 500-mL bottle fitted with a rubber stopper in a weighted holder into the tank by a nylon-coated wire rope. When the bottle reached the desired depth, the stopper was removed from the opening remotely using the same rope, allowing waste material to flow into the bottle. The sampler was then raised to the top of the tank. The grab samples were taken from six different elevation levels in the tank.

### 2.2.2 Sample Receipt and Inspection

Twenty-seven tank 241-AN-102 samples were received at PNWD's Radiochemical Processing Laboratory (RPL) from the Hanford 222-S laboratory to support the Regulatory Data Quality Objectives (DQO) Step 1 work and for process testing. The received samples were inspected on December 4, 2000, according to Test Instruction TI-41500-009,(a) and this was previously reported (Urie et al. 2002). The objective of the inspection was to document the condition of the samples. Each jar was weighed and visually inspected for the appearance of a settled solids layer, a liquid layer, and a separable organic layer. The supernatant was very dark, making color determination difficult, and detecting an organic layer was impossible. Nineteen jars of the tank waste from supernatant layers in the tank were dedicated to the Regulatory DQO Task.

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(a) PR Bredt. 2001. *AN-102 Sample Inspection*. TI-41500-009, Rev. 0, Dec. 2000. Pacific Northwest National Laboratory, Richland, Washington.

Fifteen of these samples were selected for compositing. These samples represented three sample jars from each of five different levels in Tank 241-AN-102. The samples were selected so that the amount of material from each level was approximately the same. The tank retrieval location (measured in inches from the bottom of the tank), sample mass, and inspection results of these 15 samples are shown in Table 2.2. Each sample contained a nominal 500-mL supernatant volume with very little or no solids layer present.

**Table 2.2.** Tank 241-AN-102 Sample Inspection Results

| Jar ID | Sample Retrieval Level (in.) | Net Mass Retrieved (g) | Bottle and Lid Condition | Appearance of Solids | Appearance of Liquid |
|---|---|---|---|---|---|
| 2AN-00-01 | 346 | 664.51 | Good | None obs. (small layer likely) | ~500 mL, very dark brown/black soln. |
| 2AN-00-02 | 346 | 649.86 | Good | None obs. (small layer likely) | ~500 mL, very dark brown/black soln. |
| 2AN-00-03 | 346 | 659.85 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-05 | 280 | 662.00 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-06 | 280 | 652.81 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-07 | 280 | 643.01 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-10 | 214 | 652.78 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-11 | 214 | 658.01 | Good | Little layer light brown precipitate | ~500 mL, very dark solution |
| 2AN-00-12 | 214 | 648.29 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-13 | 148 | 654.54 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-15 | 148 | 658.84 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-16 | 148 | 660.62 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-17 | 82 | 666.00 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-18 | 82 | 664.14 | Good | None obs. (small layer likely) | ~500 mL, very dark solution |
| 2AN-00-19 | 82 | 670.11 | Good | Small layer light brown precipitate | ~500 mL, very dark solution |

Notes: obs = observed; soln = solution

### 2.2.3 241-AN-102 Sample Compositing Operation Description

The 15 AN-102 waste grab samples were mixed together to provide homogeneous composite slurry samples in accordance with Test Instruction TI-41500-010.[a] The material from the sample bottles was combined in a 7-L stainless steel compositing vessel equipped with a stainless steel propeller-bladed stir shaft for mixing as shown in Figure 2.2. Baffles in the mixer aided the mixing action. The samples were introduced into the vessel through a ⅛-in. mesh screen to catch any large particles. The stirrer was operated at low speed during the compositing process until all the solids were mobilized. The composited samples were then removed through a valve near the bottom of the vessel.

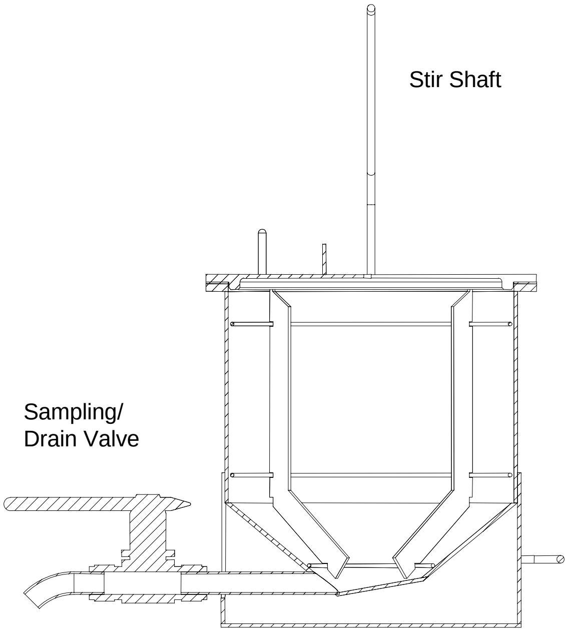

**Figure 2.2**. Schematic of Sample Compositing Vessel

Twenty-seven composite slurry sub-samples were taken from the homogenized Tank 241-AN-102 composite and placed into pre-weighed, volume-graduated, glass jars on February 5, 2001. Composite slurry sub-sample identities, masses, and volumes are provided in Table 2.3.

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(a) PR Bredt. 2001. *Regulatory Data Quality Objective: AN-102 Sample Compositing*, TI-41500-010, Rev. 0, Pacific Northwest National Laboratory, Richland, Washington.

**Table 2.3.** Tank 241-AN-102 Composited Waste Sub-Samples

| Composite Slurry Sample ID | Net Mass (g) | Volume (mL) | Composite Slurry Sample ID | Net Mass (g) | Volume (mL) |
|---|---|---|---|---|---|
| AN-102-DQO-MET-1 | 225.86 | 159 | AN-102-DQO-PCB 2 | 319.38 | ~230 |
| AN-102-DQO-MET-2 | 226.70 | 160 | AN-102-DQO-PCB 3 | 319.42 | 225 |
| AN-102-DQO-ANIONS | 204.47 | 147 | AN-102-DQO-PCB 4 | 313.70 | 221 |
| AN-102-DQO-CN | 269.11 | 190 | AN-102-DQO-PCB 5 | 350.95 | >250 |
| AN-102-DQO-VOA | 265.51 | 188 | AN-102-DQO-PCB 6 | 301.88 | 215 |
| AN-102-DQO-PEST 1 | 307.94 | 217 | AN-102-DQO-PCB 7 | 299.29 | 215 |
| AN-102-DQO-PEST 2 | 321.33 | 227 | AN-102-DQO-PCB 8 | 289.55 | 208 |
| AN-102-DQO-PEST 3 | 308.25 | 219 | AN-102-DQO-FEED A | 606.09 | ~425 |
| AN-102-DQO-PEST 4 | 331.10 | 235 | AN-102-DQO-FEED B | 607.07 | ~420 |
| AN-102-DQO-PEST 5 | 314.20 | 222 | AN-102-DQO-FEED C | 570.98 | 400 |
| AN-102-DQO-PEST 6 | 315.49 | 223 | AN-102-DQO-FEED D | 552.56 | ~390 |
| AN-102-DQO-PEST 7 | 311.09 | 220 | AN-102-DQO-FEED E | 580.55 | 400 |
| AN-102-DQO-PEST 8 | 329.29 | 234 | AN-102-DQO-FEED F | 305.37 | ~210 |
| AN-102-DQO-PCB 1 | 315.77 | 220 | **Total:** | **9462.87** | **~6670** |

The sum of the Tank AN-102 masses placed in the compositing vessel was 9865.37 g. The sum of the homogenized sub-sample masses was 9462.87 g. The net mass lost was 402.5 g or 4.08% of the total mass. The mass loss was attributed to the waste coating in the compositing vessel and/or to evaporation during preparation of the composite.

After collection, the Tank 241-AN-102 composite slurry samples were allowed to stand undisturbed for 4 days, allowing any solids to settle to the bottom of the jars to verify that homogenization was complete. The volume percent solids for each composite slurry sample was determined by visually examining the graduated markings on the jars and noting the total volume of material in the jar as well as the volume of the solids layer on the bottom. For all jars, the amount of solids was less than the lowest volume graduation (10 mL) on the jar, and therefore, a definitive volume percent measurement could not be obtained. All total volumes were greater than 100 mL, so the volume percent settled solids was considered to be less than 10%.

The composite slurry sub-samples were separated from the solids on April 6, 2001, according to Test Instruction TI-RPP-WTP-067.[a] The samples were centrifuged, and the resulting supernatants were decanted and centrifuged again until no additional solids were evident.

Seven of the clarified subsamples were selected for the Cs ion exchange process test. A summary of the sample identifications, initial gross masses (determined on April 6, 2001), final gross masses (determined on November 11, 2004), bottle tares, net masses, and volumes transferred are provided in Table 2.4. Each sample contained ≤10-mL gray solids that were compacted at the bottom of the bottle.

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(a) Jagoda LK. 2001. *Regulatory Data Quality Objective: AN-102 Composite Sample Solids Removal.* TI- RPT-WTP-067, Rev 1, Pacific Northwest National Laboratory, Richland, Washington.

The solids did not disperse easily upon shaking. The supernatant was a dark reddish color. The mass differences between the initial gross bottle masses and final gross bottle masses ranged from 3.6 to 22 g. Mass losses were attributed to water evaporation over the 3.6-y storage period.

**Table 2.4.** Actual Homogenized Tank 241-AN-102 Sub-Samples for Ion Exchange Feed Preparation

| Sample ID | Bottle Tare, g[(a)] | Initial Gross Mass, g[(b)] | Final Gross Mass, g[(c)] | Mass Diff., g | Empty Bottle Mass, g | Transferred Sample Mass, g | Volume, mL |
|---|---|---|---|---|---|---|---|
| AN-102-DQO-PCB 4 | 218.6 | 524.0 | 504.4 | 19.6 | 264.1 | 240.3 | 167.7 |
| AN-102-DQO-PCB 5 | 218.3 | 551.2 | 532.2 | 19.0 | 268.8 | 263.4 | 183.8 |
| AN-102-DQO-PCB 6 | 218.3 | 501.2 | 497.6 | 3.6 | 257.1 | 240.5 | 167.8 |
| AN-102-DQO-PCB 7 | 218.3 | 497.0 | 489.9 | 7.1 | 254.9 | 235.0 | 164.0 |
| AN-102-DQO-PCB 8 | 218.3 | 493.3 | 477.2 | 16.1 | 259.9 | 217.3 | 151.6 |
| AN-102-DQO-PEST 1 | 217.9 | 520.8 | 512.9 | 7.9 | 265.4 | 247.5 | 172.7 |
| AN-102-DQO-PEST 2 | 218.6 | 533.4 | 511.3 | 22.1 | 258.3 | 253.0 | 176.6 |
| Composite: | | | | | | 1697 | 1184 |

(a) From TI-41500-010, 2001.
(b) Gross mass measured on April 6, 2001, from TI-RPP-WTP-067, Rev. 1.
(c) Gross mass measured on November 11, 2004.

The bottle lids were stuck tight to the bottles and could not be freed with manipulators. Each lid had to have a hole drilled into it for access to the supernatant. Supernatant was decanted from each bottle through the access hole on November 11, 2004, to make a composite solution in a 4-L polyethylene jug. The empty bottle masses were measured after sample removal. An average of 56 g of residual solids and supernatant remained in the sample bottles.

### 2.2.4 241-AN-102 Pretreatment

The 241-AN-102 composite pretreatment was conducted according to Test Instruction TI-RPP-WTP-402.[(a)] A flowchart showing supernatant processing is provided in Figure 2.3. The collected supernatant was thoroughly mixed and then sub-sampled. The supernatant density was measured to be 1.43 g/mL (29°C), which was in good agreement with previously reported supernatant density of 1.42 g/mL (Urie et al. 2002). A 0.1-mL aliquot was placed in 10-mL 0.5 M nitric acid and submitted to the Analytical Support Operations (ASO) for direct Na analysis by inductively coupled plasma-atomic emission

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(a) B McNamara. 2004. *Preparation of Ion Exchange Process Feed: Compositing, Dilution, Sr/TRU Precipitation of AN-102 Hanford Tank Waste Samples*, TI-RPP-WTP-402, Battelle—Pacific Northwest Division, Richland WA.

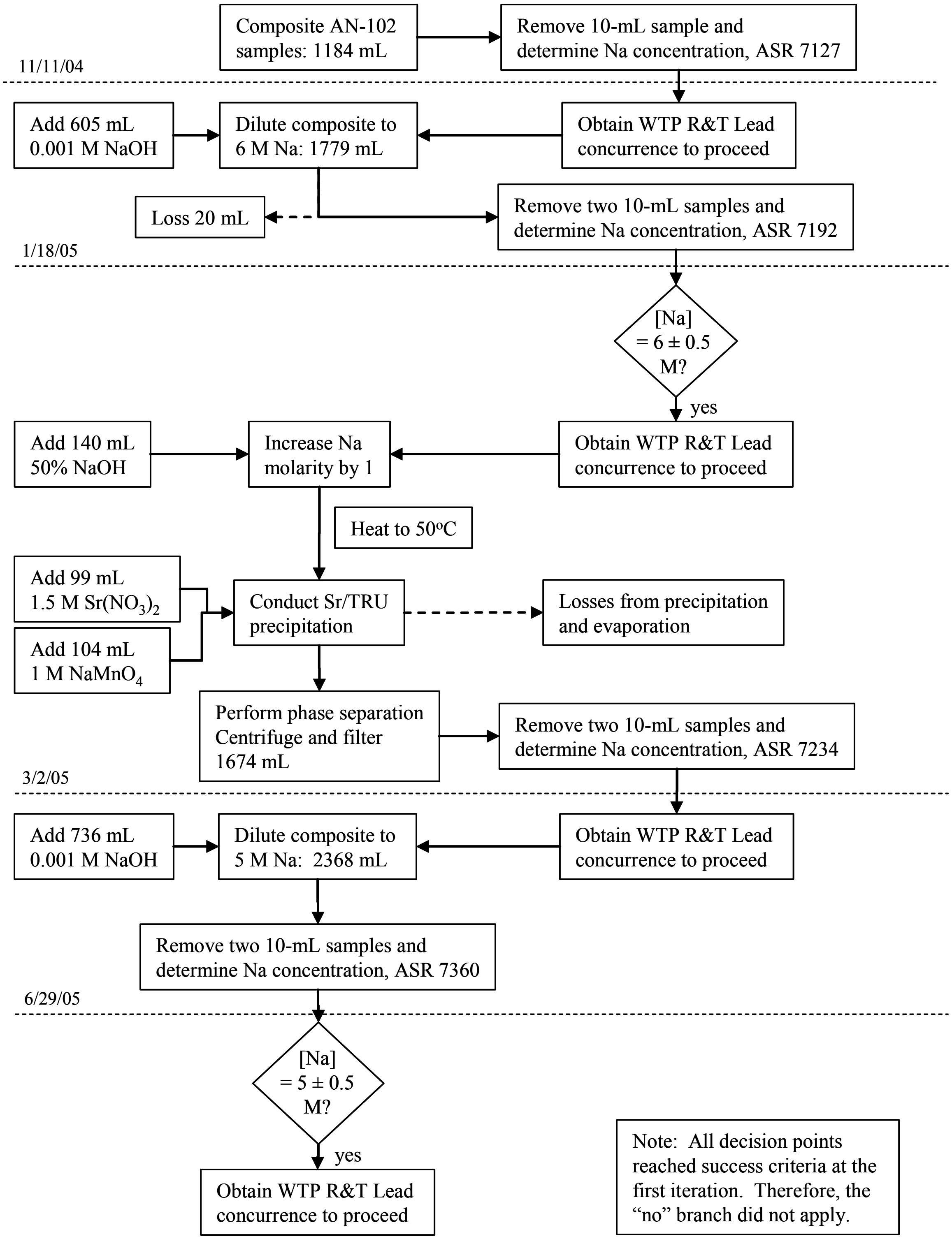

**Figure 2.3.** 241-AN-102 Processing and Pretreatment Flowchart

spectrometry (ICP-AES) according to Analytical Services Request (ASR) 7127. The average Na concentration was determined to be 8.97 M in a total volume of 1174 mL. A 605-mL volume of 0.001 M NaOH was added to the composite followed by thorough mixing on January 18, 2005. The Na concentration was measured by ICP-AES per ASR 7192. The solution density was 1.296 g/mL, and the average Na concentration was 5.76 M, meeting the 6 ± 0.5 M specification in the test plan.

Strontium-TRU separation began on March 2, 2005. The diluted supernatant was transferred to a stainless steel pot modified with welded handles for ease of manipulator use. The supernatant was heated to 50$^{o}$C using a hot plate for the heat source powered through a temperature controller. The temperature was monitored using a type K thermocouple. The solution was mixed using an overhead stirrer equipped with a paddle-wheel-type stainless steel stir blade mounted to a stainless steel shaft. The supernatant was continuously stirred during the following reagent additions and heating time. A 140-mL volume of 19 M NaOH (50.4 weight percent, VWR International, Lot # 4181) was added slowly. The temperature was allowed to equilibrate for 50 minutes. A 99-mL volume of 1.5 M strontium nitrate (Aldrich, Lot # 09319BF) was added over a 5-min period. The solution temperature dropped only to 49$^{o}$C. A 104-mL volume of 1 M sodium permanganate (Aldrich Lot # 04612PQ) was slowly added over a 5-min period. The solution was mixed at temperature for 4 h. Solution temperatures ranged from nominally 47$^{o}$C to 52$^{o}$C. After the 4-h digestion time, the hot plate was turned off; stirring continued until the solution cooled to ambient temperature. Stirring continued for another 24 h.

After the Sr/TRU precipitation, heating, and cooling, the mixture was removed in ~250-mL aliquots for phase separation by centrifuging. The mixture was centrifuged at 1000 rpm for 30 min. The supernatant fraction was decanted through a 0.45-µm pore size nylon filter on March 4, 2005. The solution filtered rapidly. Residual solids in the stainless steel pot, centrifuge bottles, and filter were disposed. The supernatant density was measured to be 1.319 g/mL (29$^{o}$C) and an aliquot removed for analysis of Na by ICP-AES according to ASR 7234. The Na concentration was 7.22 M. A 736-mL volume of 0.001 M NaOH was added to the pretreated supernatant with thorough mixing on June 29, 2005. The diluted supernatant density was determined to be 1.230 g/mL (26$^{o}$C). The Na concentration was determined by ICP-AES according to ASR 7360 to be 4.89 M, within the 5.0 ± 0.2 M range allowed per the test plan. The pretreatment processing was completed on June 29, 2005.

The ion exchange testing started 2 months following the feed pretreatment. Additional solids were evident in the feed, so the supernatant was again filtered on August 29, 2005, through a 0.45-µm pore size nylon filter.

## 2.3 Ion Exchange Column System

A schematic of the ion exchange column system is shown in Figure 2.4. The system consisted of two small columns containing the ion exchange material, a small metering pump, three valves, a pressure gauge, and a pressure relief valve. Valves 1, 2, and 3 were three-way valves that could be turned to the flow position, sample position, or no-flow position. Valve 1 was placed at the outlet of the pump and was used to eliminate air from the system, purge the initial volume of the system, or isolate the columns from the pump. Valves 2 and 3 were primarily used for obtaining samples and could also be used to isolate the columns from the rest of the system. Both the 10-psi trigger pressure relief valve (Swagelok, Solon, OH) and the 15-psi pressure gauge (Ashcroft, Stratford, CT) were plumbed in line and before the first column.

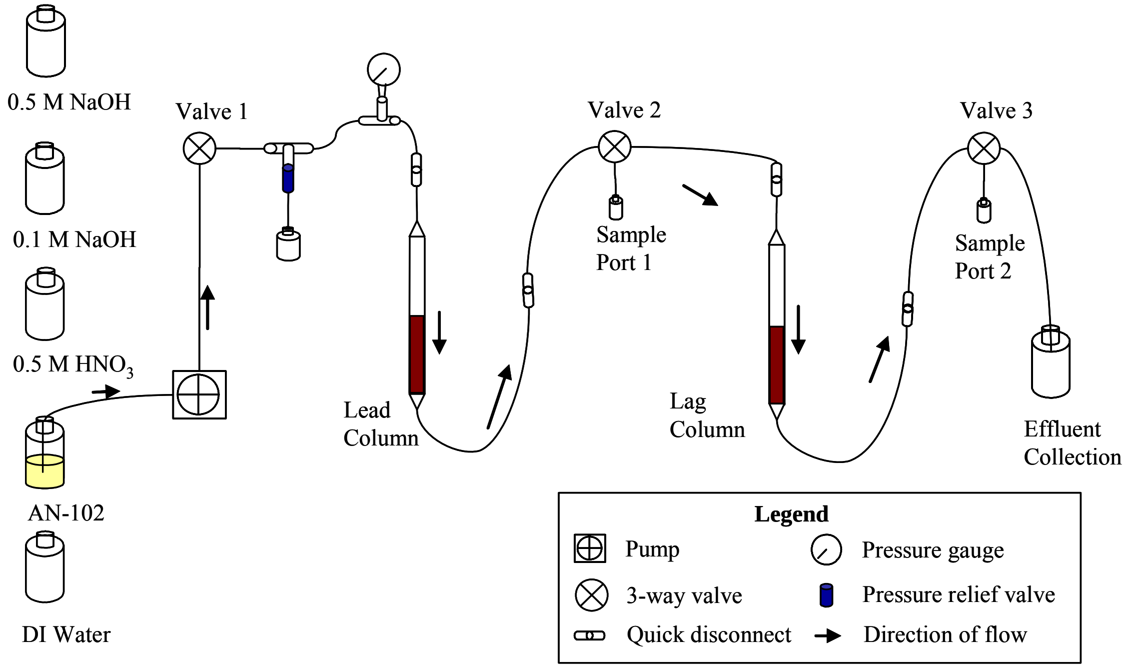

**Figure 2.4.** Schematic of Cesium Ion Exchange Column System

Two columns were connected in series with the first column referred to as the lead column and the second column referred to as the lag column. The columns were standard 1.5-cm diameter by 15-cm tall Spectra/Chrom Organic columns (Spectrum Chromatography, Houston, TX). The top and bottom fittings were constructed of Teflon. Stainless steel, 200-mesh screens stabilized with snug-fitting O-rings supported each resin bed. The cavity below the screen support was filled with 3-mm-diameter glass beads, reducing the fluid-filled volume from 2 mL to 1 mL. A decal millimeter scale (Oregon Rule Co., Oregon City, Oregon) affixed to the column allowed for measurement of resin bed height and thus shrinkage and swelling.

The connecting tubing was 1/8-in OD, 1/16-in ID polyethylene. Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column switching. An FMI QVG50 pump (Fluid Metering, Inc., Syosset, New York) equipped with a ceramic and Kynar® coated low-flow piston pump head was used for all fluid introduction. The flow rate was controlled with a remotely operated FMI stroke-rate controller. The pump was calibrated with the stroke-rate controller and could provide pumping rates from 0.08 to16 mL/min. The volume actually pumped was determined using the mass of the fluid and the fluid density. The pressure indicated on the pressure gauge remained below 5 psi during all runs.

The fluid level above the support screen was maintained at about 11-cm. Depending on whether the resin was expanded as the Na-form (6.3 cm tall or 11 mL) or contracted as the H-form (5 cm tall or 8.8 mL), fluid volume above the resin bed varied from 8.3 mL to 10.6 mL, respectively. The total holdup volume of the Cs ion exchange system was the summed volume of all fluid-filled parts from the inlet line to the effluent line and was estimated to be 48 mL. A photograph of the ion exchange apparatus is shown in Figure 2.5. The lead column was on the left, and the lag column was on the right. Both resins were in the H-form when this photo was taken. Before any processing, the fluid level was increased to 11-cm above the support screen.

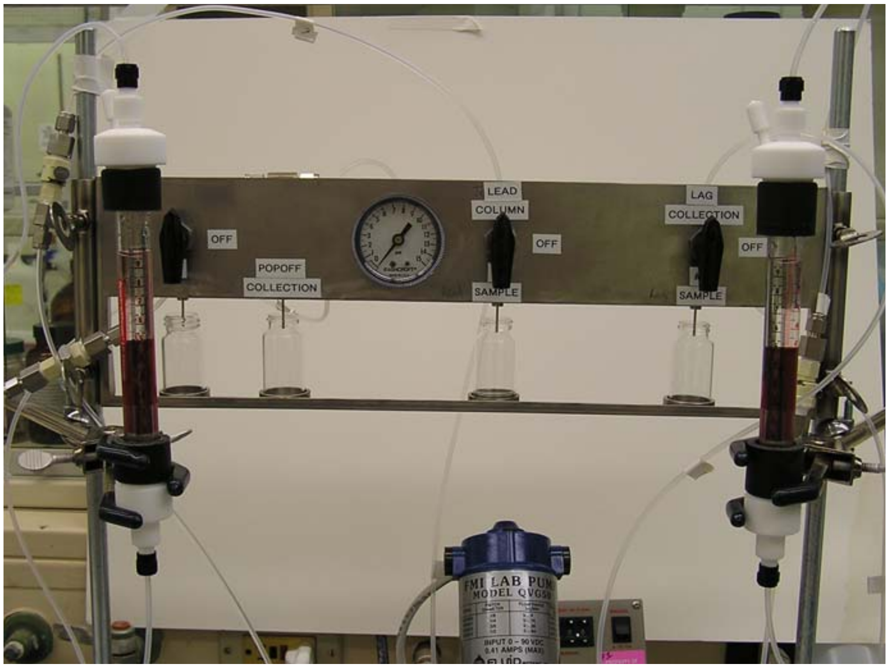

**Figure 2.5.** Photograph of the Ion Exchange Test Apparatus with H-Form Resin

## 2.4 Resin Bed Preparation and Previous Waste Processing

The following sections describe the actual process test conditions for in-column pretreatment, simulant processing, and actual waste processing. In all cases, fluids were processed downflow. The solution volume above each resin bed was not purposefully manipulated. Fluids added to the column would variously mix into the solution above the resin bed, depending on the different solution densities. Visual observations of the refractive index changes showed that the high-density tank waste tended to drop quickly through the 0.5 M NaOH to the resin bed surface (and perhaps beyond the surface). The low-density 0.1 M NaOH displacement fluid, in contrast, exhibited more of a plug flow pattern in displacing the tank waste.

### 2.4.1 In-Column Resin Pretreatment

Each pretreated 8-mL H-form resin fraction was transferred to separate beakers and contacted with 5× volumes (40 mL) of 1 M NaOH. The soak continued for 55-min with agitation every 10 min. The resin slurries were then quantitatively transferred into the columns using additional deionized (DI) water to aid the transfer. The lead-column resin was identified as "Resin A," and the lag-column resin was identified as "Resin B."[(a)] Each resin bed was individually pretreated in the column by processing sequentially

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(a) After column position switching during the subsequent AN-102 process test; the Resin B column was placed in the lead position.

water, 0.5 M $HNO_3$, water, and then 0.5 M NaOH. Specific in-column pretreatment parameters are provided in Table 2.5.

**Table 2.5.** Column Process Testing Parameters

| Process step | Solution | Total Volume | | | Flowrate | | Time |
|---|---|---|---|---|---|---|---|
| | | BV(a) | AV(b) | mL | BV/h | mL/min | h |
| *In-Column Pretreatment, Columns in Parallel, 7/14/05 (Resin A/Resin B)* | | | | | | | |
| Water rinse | DI water | 5.9/6.4 | 1.4/1.5 | 66/71 | 2.7/2.9 | 0.51/0.55 | 2.2/2.2 |
| Acid wash | 0.5 M $HNO_3$ | 7.6/7.7 | 1.8/1.8 | 85/85 | 2.8/2.8 | 0.52/0.53 | 2.7/2.7 |
| Water rinse | DI water | 2.6/2.6 | 0.61/0.61 | 29/29 | 1.7/1.7 | 0.31/0.31 | 1.6/1.6 |
| Regeneration | 0.5 M NaOH | 5.1/5.2 | 1.2/1.2 | 57/58 | 2.8/2.3 | 0.52/0.43 | 1.8/2.3 |
| *Simulant AP-101 Test, Columns in Series, 7/18/05* | | | | | | | |
| Regeneration (cont.) | 0.5 M NaOH | 0.846 | 0.196 | 9.42 | 2.99 | 0.554 | 0.28 |
| Loading (Resin A) | AP-101 Simulant | 131 | 30.3 | 1454 | 2.94 | 0.546 | 44.2 |
| Loading (Resin B)(c) | AP-101 Simulant | 120 | 27.9 | 1338 | 2.94 | 0.546 | 44.2 |
| Feed displacement | 0.1 M NaOH | 7.49 | 1.74 | 83.4 | 2.96 | 0.549 | 2.53 |
| Water rinse | DI water | 7.7 | 1.79 | 85.8 | 3.10 | 0.576 | 2.48 |
| *Simulant AP-101 Test, Columns in Parallel (Resin A/Resin B)* | | | | | | | |
| Elution | 0.5 M $HNO_3$ | 15.3/15.2 | 3.54/3.52 | 170/169 | 1.50/1.48 | 0.278/0.275 | 10.3/10.3 |
| Water rinse | DI water | 7.97/7.96 | 1.85/1.85 | 88.8/88.6 | 1.38/1.41 | 0.256/0.261 | 5.78/5.67 |
| *Dual Column Actual AP-101DF Waste Test, 8/15/05* | | | | | | | |
| Regeneration | 0.5 M NaOH | 10.8 | 2.49 | 120 | 2.57 | 0.477 | 4.18 |
| Loading (Resin A) | AP-101DF | 137 | 31.9 | 1531 | 2.89 | 0.536 | 47.3 |
| Loading (Resin B)(c) | AP-101DF | 135 | 31.2 | 1498 | 2.89 | 0.536 | 47.3 |
| Feed displacement | 0.1 M NaOH | 8.55 | 1.98 | 95.2 | 3.11 | 0.577 | 2.75 |
| Water rinse | DI water | 8.67 | 2.01 | 96.5 | 2.86 | 0.545 | 2.38 |
| *Resin A (Lead Column) Only* | | | | | | | |
| Elution | 0.5 M $HNO_3$ | 15.6 | 3.63 | 174 | 1.42 | 0.263 | 11.0 |
| Water rinse | DI water | 3.19 | 0.74 | 35.5 | 1.47 | 0.273 | 2.17 |
| *Regenerate Lead Column, 8/29/05* | | | | | | | |
| Regenerate | 0.5 M NaOH | 6.11 | 1.42 | 68.0 | 3.09 | 0.57 | 2.07 |
| *Switch Column Positions; Actual AN-102 Waste Test, Columns in Series, 8/29/05* | | | | | | | |
| Regenerate | 0.5 M NaOH | 2.93 | 0.68 | 32.6 | 2.82 | 0.53 | 1.03 |
| Loading (Resin B) | AN-102 | 202 | 46.8 | 2247 | 2.95 | 0.547 | 68.6 |
| Loading (Resin A) (c) | AN-102 | 197 | 45.7 | 2194 | 2.95 | 0.547 | 68.6 |
| Feed displacement | 0.1 M NaOH | 9.61 | 2.23 | 107 | 3.03 | 0.563 | 3.17 |
| Water rinse | DI water | 9.05 | 2.10 | 101 | 2.90 | 0.538 | 3.13 |
| *Resin B (Lead Column) Only* | | | | | | | |
| Elution | 0.5 M $HNO_3$ | 30.4 | 13.0 | 339 | 1.41 | 0.261 | 21.7 |
| Water rinse | DI water | 3.02 | 1.29 | 33.6 | 1.43 | 0.265 | 2.12 |
| *Resin A (Lag Column) Only* | | | | | | | |
| Elution | 0.5 M $HNO_3$ | 29.4 | 12.6 | 327 | 1.41 | 0.262 | 20.8 |
| Water rinse | DI water | 3.04 | 1.30 | 33.8 | 1.45 | 0.269 | 2.10 |

(a) BV = bed volume (nominally 11 mL in the Na-form volume as loaded in the column).
(b) AV = apparatus volume (nominally 48 mL).
(c) The feed volume through the lag column was reduced, relative to the lead column, because of sampling from the lead column.

### 2.4.2 Bed Volume Definition

The resin BV was defined as the volume of the resin bed after in-column pretreatment in the 0.5 M NaOH regeneration solution. Both Resin A and Resin B BVs were 11.1 mL.

### 2.4.3 Simulant and Actual AP-101 Waste Processing

The shakedown test with AP-101 simulant and process testing with actual AP-101 tank waste have been previously reported.[a] Column A was placed in the lead position, and 1.4 L simulated AP-101 tank waste was processed sequentially through the ion exchange resin beds for a full shakedown test. After simulant processing, 0.1 M NaOH feed displacement and water rinse were passed through the system. The columns were then separated and eluted, rinsed, and regenerated individually.

The beds had been stored approximately 24 days in the H-form in DI water since the end of the simulant AP-101 processing. Then the ion exchange system was loaded into the Shielded Analytical Laboratory (SAL) hot cell with the resin in the H-form in such a manner as to minimize disturbance of the resin beds. The resin beds were regenerated to the Na-form by processing 10.8 BVs of 0.5 M NaOH solution sequentially through the two columns. A 1.5-L aliquot of actual waste AP-101 was processed sequentially through the resin beds, followed by feed displacement solution and water rinse. The lead column was eluted with 15.6 BVs of 0.5 M $HNO_3$. The lag column (Resin B) was calculated to be loaded with 5.67E+4 μCi $^{137}$Cs and 2.9 mg total Cs.

## 2.5 AN-102 Ion Exchange Processing

The resin beds had been stored approximately 11 days since the end of the actual waste AP-101 processing. The AN-102 process testing was conducted according to Test Instruction TI-RPP-WTP-427.[b] All processing was performed in the hot cells at temperatures ranging from 26 to 27°C. The lead column (Resin A) was regenerated with 6 BVs of 0.5 M NaOH; the effluent was collected from the lead column. The regenerated lead column (Resin A) was placed in the lag position, and the partially-loaded lag column (Resin B) was placed in the lead position. Placing the partially-loaded column in the lead position and the eluted column in the lag (or polishing) position emulated planned WTP plant operations. The columns were connected in series, and an additional 3 BVs of 0.5 M NaOH were processed to flush water from the lines and the lead column (Resin B). The AN-102 loading, feed displacement, and DI water rinse steps were conducted by passing these solutions through both resin beds connected in series. The elution and elution rinse were conducted on each resin separately. The experimental conditions for each process step are shown in Table 2.5.

During the loading phase, nominal 2-mL samples were collected from both the lead and lag columns at the sample collection ports. The solution in the lag column remained static during the lead-column sampling time of about 4 minutes. Samples were collected after the first 5 BVs were processed and again at nominal 10-BV increments. The feed was processed for 68.6 h continuously. The AN-102 effluent was collected in three effluent bottles. The first bottle collected the first 60 BVs processed; the second

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(a) SK Fiskum, MJ Steele, and DL Blanchard. Draft. *Spherical Resorcinol-Formaldehyde Resin Testing for $^{137}$Cs Removal from Simulated and Actual Hanford Waste Tank 241-AP-101 Diluted Feed (Envelope A) Using Small Column Ion Exchange*. RPP-WTP-134, Battelle—Pacific Northwest Division, Richland WA.

(b) SK Fiskum. 2005. *Cesium Removal from AN-102 Actual Tank Waste Using Spherical Resorcinol-Formaldehyde Resin*, TI-RPP-WTP-427, Battelle—Pacific Northwest Division, Richland, WA effluent collection contained the 61- to 140-BV fraction; the third effluent collection contained the 141- to 202-BV fraction. Feed displacement and water rinse samples were collected in 1-BV increments. The eluate and follow-on water rinse were collected in nominal 1.4-BV increments.

Cesium load and elution performance was determined from effluent $^{137}$Cs concentrations. The collected samples were analyzed directly by gamma energy analysis (GEA) to determine the $^{137}$Cs concentration. Breakthrough and elution curves were generated based on the feed $^{137}$Cs concentration ($C_o$) and the effluent Cs concentration (C).

### 2.5.1 Sample Analysis

A summary of the sample and sub-sample collections and analyses from the various tests and process steps are provided in Table 2.6.

**Table 2.6.** AN-102 Process Sampling Interval and Analyses

| **Process Step** | **Lead Column** | **Lag Column** | **Sample Size (mL)** | **Process Sample Analyses** | **ASO Sample Analysis** |
|---|---|---|---|---|---|
| *Dual Column AN-102 Waste Test, 8/29/05* | | | | | |
| Regeneration | Every 1.5 BVs | – | 2 | GEA | – |
| Regeneration (cont.) | – | Every 1.5 BVs | 2 | GEA | – |
| Loading | Every 10 BVs | Every 10 BVs | 2 | GEA | – |
| Feed displacement | – | Every 1.7 BVs | 2 | GEA | – |
| Water rinse | – | Every 1.7 BVs | 2 | GEA | – |
| Elution | Every 1.4 BVs | – | 0.1 | GEA | GEA (selected samples) |
| Water rinse | Every 1 BV | – | 2 | GEA | – |
| *Composite Samples* | | | | | |
| Effluent composite 0-60 BV | – | 1 composite | 2 | GEA | – |
| Effluent composite 61-140 BV | – | 1 composite | 2 | GEA | – |
| Effluent composite 140-202 BV | – | 1 composite | 2 | GEA | – |
| AN-102 representative effluent composite from the three sub-composites | – | 1 composite | 15 | – | ICP-AES, GEA, IC,[(a)] free hydroxide, U, Pu, Am, Cm, total alpha, $^{133}$Cs, $^{85}$Rb |
| AN-102 lead column eluate composite | 1 composite | – | 15 | – | ICP-AES, GEA, IC, U, Pu, Am, Cm, total alpha, $^{133}$Cs, $^{85}$Rb |
| AN-102 lag-column eluate composite | 1 composite | – | 15 | – | ICP-AES, GEA, IC, U, Pu, Am, Cm, total alpha, $^{133}$Cs, $^{85}$Rb |

(a) IC = ion chromatography.

### 2.5.1.1 Process Sample Analysis

All density determinations were performed in the SAL in duplicate by measuring the net solution masses in 10-mL Class A volumetric flasks.

Feed, load, effluent, and elution sample $^{137}$Cs concentrations were determined using ASO-calibrated GEA spectrometers. To support this analysis, all samples and sub-samples were collected and packaged in 2-mL volumes to accommodate the calibrated detector geometry. Exact volumes were calculated from the measured net mass and solution densities. Because of the high dose rate from $^{137}$Cs, the feed and eluate samples required dilution before removal from the hot cell. These samples were diluted with water. The extent of dilution was determined by mass. The sample count time was adjusted to accommodate the specific sample $^{137}$Cs concentration.

A composite effluent sample was generated by combining pro-rated sub-sample volumes from each of the three effluent collection bottles into a single vial. The effluent composite represented the 0 to 202 BVs processed.

Selected elution sub-samples were forwarded to the ASO for independent confirmatory analysis. Once the process sample GEA results were confirmed with the independent ASO-generated results, eluate composite samples were prepared according to Test Instruction TI-RPP-WTP-431.(a) The lead-column eluate samples were combined in their entirety into a composite. Pro-rated sub-sample volumes from each lag-column elution sample were combined to prepare a representative lag-column eluate composite.

The composite eluates were submitted to the ASO for analysis by GEA for gamma emitters, ICP-AES for large suite of metals and non-metals, ion chromatography (IC) for anions, kinetic phosphorescence analysis (KPA) for U, inductively coupled plasma-mass spectrometry (ICP-MS) for $^{133}$Cs and $^{85}$Rb (lead column only), and radiochemistry for Pu, Am, Cm, and total alpha. The AN-102 feed and the effluent composite were similarly analyzed with the inclusion of free hydroxide by titration.

### 2.5.1.2 Analytical Services

A crosswalk of sample identification and ASO ASR numbers is provided in Table 2.7. The ASO was responsible for preparing and analyzing the appropriate analytical batch and instrument quality control (QC) samples as well as providing any additional processing to the sub-samples that might be required (e.g., acid digestion, radiochemical separations). The ASO was responsible for sample distribution from the SAL to the various analytical workstations.

*Sample Preparation*

Hydroxide, IC, and GEA analyses were conducted directly on dilutions of the feed and effluent. A diluent blank was also distributed with the analytical sub-samples for analysis.

Analysis by ICP-AES, ICP-MS, and radiochemical techniques required acid digestion of the feed and effluent. Feed and effluent sample aliquots (1.0 mL) were acid-digested in duplicate according to procedure PNL-ALO-128, *$HNO_3$-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block*

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(a) SK Fiskum. 2005. *Eluate and Effluent Compositing for Sample Submission Supporting A-204 AP-101 and A-212 AN-102 Cesium Ion Exchange Processing*. Battelle—Pacific Northwest Division, Richland, WA.

*Heater*. The acid-digested solutions were brought to a nominal 25-mL volume; absolute volumes were determined based on final solution weights and densities. Along with the sample and duplicate, the ASO processed a digestion preparation blank (PB), two blank spikes (BSs) (one for ICP-AES and one for ICP-MS), and two matrix spikes (MSs) (one for ICP-AES and one for ICP-MS). Aliquots of the BS, MS, and PB, along with aliquots of the duplicate samples, were delivered to the analytical workstations for analyses.

**Table 2.7**. Sample ASR and RPL Identifications

| Sample Identification | ASR | RPL ID | Sample Description | Purpose |
|---|---|---|---|---|
| ***AN-102 Pretreatment*** | | | | |
| TI402AN-102ARComp | 7127 | 05-00225 | AN-102 composite | Initial Na concentration determination |
| TI402AN102D | 7192 | 05-01060 | AN-102 diluted to 6 M Na | Confirmation that 6 M Na was achieved |
| | | 05-01061 | field duplicate | |
| TI402AN102SrTRU | 7234 | 05-01274 | AN-102 with Sr/TRU removed | Determine Na molarity after Sr/TRU removal |
| TI402AN102-PTDil | 7360 | 05-01933 | AN-102 after Sr/TRU removal and dilution to 5 M Na | Determine Na molarity after Sr/TRU removal and dilution in preparation for ion exchange processing |
| ***AN-102 Ion Exchange Processing*** | | | | |
| AN-102L-E2, -E3, -E4, -E5, -E6, -E12, -E16, -E21; AN-102P-E3, -E4, -E11, -E13 | 7467 | 05-02517 – 05-02528 | Selected elution samples | Verify eluate recovery and elution profile |
| AN102P-E3, -E4 | 7489 | 06-00033 | Selected elution samples | Recheck analytical results |
| TI427AN102-IXF | | 06-00029 | AN-102 Feed | Feed characterization (duplicate of 05-01933) |
| AN-102-EFF-Comp | | 06-00030 | Effluent composite, 0-202 BVs | Effluent characterization |
| AN102-LE-Comp | | 06-00031 | Lead-column elution composite | Elution characterization (lead) |
| AN102-PE-Comp | | 06-00032 | Lag-column elution composite | Elution characterization (lag) |

Eluate samples were simply diluted 20.9× in 0.5 M $HNO_3$ (or water for IC) for distribution to the various workstations for instrumental analysis. A diluent blank was distributed with the analytical samples for analysis.

*Analysis*

Hydroxide was determined using potentiometric titration with standardized HCl according to procedure RPG-CMC-228, *Determination of Hydroxyl ($OH^-$) and Alkalinity of Aqueous Solutions, Leachates, and Supernates and Operation of Brinkman 636 Auto-Titrator*. The free hydroxide was defined as the first inflection point on the titration curve.

Anions were determined using a Dionix ICS 2500 IC system equipped with a conductivity detector according to procedure PNL-ALO-212, *Determination of Inorganic Anions by Ion Chromatography*.

The ICP-AES analysis was conducted according to procedure RPG-CMC-211, *Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry (ICPAES)*.

The ICP-MS analysis was conducted according to procedure PNNL-AGG-415, Rev. 1, *ICP/MS Analysis Using Perkin Elmer Elan DRCII ICPMS*. The ICP-MS was used to determine $^{133}$Cs and $^{85}$Rb. The Cs isotopic ratio was calculated from data reported by Urie et al. (2002), incorporating the $^{137}$Cs decay correction and supernatant dilution as determined from the diluted K concentration (dilution factor of 1.90). The $^{133}$Cs concentration (5.04 μg/mL) determined using the dilution factor agreed with the measured $^{133}$Cs concentration (4.89 μg/mL) within 3%. Therefore, the derived isotopic composition was applied to the current AN-102 processing.

The Rb isotopic ratio in AN-102 was not known. The total Rb reported by Urie et al. (2002) at 8.74 μg/mL was dilution-corrected to 4.59 μg/mL. The $^{85}$Rb determined by ICP-MS was 1.57 μg/mL. If the natural isotopic abundance of 72% is applied to the $^{85}$Rb, the total Rb would be 2.18 μg/mL, 47% of the dilution-corrected value. The isotopic fraction of Rb was probably not natural; $^{87}$Rb has ~3% fission yield. Therefore, only the $^{85}$Rb concentrations, not the total Rb, are provided. However, the fractionation of Rb during ion exchange processing can be assessed by evaluating $^{85}$Rb. A summary of the derived and measured Cs and Rb concentrations is provided in Table 2.8. The K concentration is also provided as a point of reference because it should react similarly to Cs and Rb and was the basis for the applied dilution factor. (The Na concentration was altered by both dilution and NaOH addition so could not be used as the basis of the dilution factor.)

**Table 2.8.** Total Cs and Rb Concentrations in AN-102 Feed

| **Isotope/ Element** | **Concentration [(a)]** | **Isotopic Ratio[(a)]** | **AN-102 Feed Concentration (Dil Fac 1.90[(b)])** | **Calculated Isotopic Ratio[(b)]** | **Measured Concentration (ASR 7489)[(b)]** |
|---|---|---|---|---|---|
| | **μg/mL** | **%** | **μg/mL** | **%** | **μg/mL** |
| $^{133}$Cs | 9.60 | 60.1 | 5.04 | 61.6 | 4.89 |
| $^{135}$Cs | 2.65 | 16.6 | 1.39 | 17.0 | 1.35[(c)] |
| $^{137}$Cs | 3.70 | 23.2 | 1.76 | 21.4 | 1.70 |
| Total Cs | 15.95 | 99.9 | 8.19 | 100 | 7.93[(d)] |
| Rb | 8.74 | not reported | 4.59 | -- | 1.57 (as $^{85}$Rb) |
| K | 1,980 | -- | 1,040 | -- | 1,040 |

(a) Urie et al. (2002), reference date April 1, 2001.
(b) Overall dilution factor from as-received condition to feed condition = 1.90; reference date August 15, 2005.
(c) The derived $^{135}$Cs concentration.
(d) The derived total Cs concentration.

Uranium was determined using a Chemchek Instruments KPA according to procedure RPG-CMC-4014, Rev. 1, *Uranium by Kinetic Phosphorescence Analysis.*

Gamma emitters were determined on direct or diluted sample aliquots according to RPG-CMC-450, Rev. 0, *GEA and Low Energy Photon Spectrometry*. Long (14-h) count times were used in an attempt to discern low-activity and low-energy gamma emitter concentrations.

Isotopes of $^{238}$Pu, $^{239+240}$Pu, $^{241}$Am, $^{242}$Cm, and $^{243+244}$Cm were determined, after radiochemical separations, by alpha spectrometry. Total alpha was determined by directly plating sample aliquots onto counting planchets and counting with ZnS scintillation detectors. High solids content in the feed and effluent samples tended to bias results low. The sum of alpha was simply the summation of measured specific alpha emitters and in these cases was a better estimation of the total alpha activity. A summary of the radiochemical alpha analytical procedures is provided in Table 2.9.

**Table 2.9.** Radiochemical Alpha Analysis Procedure Identification

| Analyte | Procedure | Title |
|---|---|---|
| Pu, Am, and Cm | RPG-CMC-417, Rev. 1 | Separation of U, Am/Cm, and Pu and Actinide Screen by Extraction Chromatography |
| | RPG-CMC-496, Rev. 0 | Precipitation Plating of Actinides for High-Resolution Alpha Spectrometry |
| | RPG-CMC-422, Rev. 1 | Solutions Analysis: Alpha Spectrometry |
| Total alpha | RPG-CMC-4001, Rev. 1 | Source Preparation for Gross Alpha and Gross Beta Analysis |
| | RPG-CMC-408, Rev. 1 | Total Alpha and Beta Analysis |

# 3.0 Results and Discussion

This section describes results from the AN-102 pretreatment processing and the dual column ion exchange processing.

## 3.1 Pretreatment and Feed Composition

The ICP-AES analysis of AN-102 supernatant obtained for the various process steps is summarized in Table 3.1. The analyte uncertainties were ± 15% where concentrations were >EQL.(a) The bolded values in Table 3.1 indicate analytes that were listed as analytes of interest on the ASR. As such, the QC results for these analytes were evaluated. The non-bolded analyte values are provided for information only; their results may not have been evaluated for QC purposes. Only Na was listed as an analyte of interest for ASRs 7127 and 7192. The ASR 7489 ICP-AES analysis essentially duplicated the ASR 7360 analysis.

The Na concentration target for the first dilution was 6.0 ± 0.5 M. The resulting Na concentration of 5.74 M (ASR 7192) was within this criterion. An average dilution factor of 1.58 (± 4%) was observed for other (opportunistic) ICP-AES analytes greater than the EQL, which agrees very well with the dilution factor of 1.56 indicated by the Na analysis.

The supernatant input to the Sr/TRU precipitation was diluted with 50% NaOH, 1.5 M $Sr(NO_3)_2$, and 1 M $KMnO_4$ and heated, resulting in evaporative losses. The supernatant volume following Sr/TRU removal (1674 mL) was only 5% different from the ingoing volume (1759 mL). Thus, spectator ion concentrations should be nearly unchanged before and after precipitation. A results comparison between ASRs 7192 and 7234 showed ~50% of the Ca was removed from solution; Cr and Pb concentration decreased nearly 30%; Sr (stable) concentration increased nearly 100-fold. The fates of TRUs were not assessed.

The Na concentration in the diluted Sr/TRU-separated supernatant (ASR 7360) was 4.89 M, meeting the acceptance criterion of 5 ± 0.2 M. The dilution factor calculated from ICP-AES analytes >EQL averaged 1.49 (± 2.6%). In contrast, the Mn concentration was reduced by a factor of 4, indicating that it continued to precipitate following the Sr/TRU solids separation. This was consistent with the observation of black solids (presumably $MnO_2$) on the bottle wall that accumulated during the 117-day storage period.

The follow-on AN-102 feed analysis results conducted according to ASR 7489 agreed well with those obtained from ASR 7360 for analytes >EQL. The re-analysis was deemed necessary on the feed because additional precipitate formed during the 61-day storage time between pretreated feed filtration and ion exchange analysis. Strontium concentration remained virtually unchanged. The Mn concentration (<EQL) appeared to drop by greater than a factor of 3 during the additional 61-day storage period between ASR 7360 analysis and final filtration just before ion exchange processing. The complete feed characterization results (inclusive of IC, hydroxide, ICP-AES, and radionculides) supporting ASR 7489 are summarized in Table 3.2.

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(a) EQL = estimated quantitation limit.

**Table 3.1.** ICP-AES Analysis of AN-102 During and After Pretreatment Steps

|  | **As-Received** | **Diluted** | **Sr/TRU Removed** | **Sr/TRU Removed and Diluted(a)** | **Sr/TRU Removed and Diluted(b)** |  |
|---|---|---|---|---|---|---|
| ***ASR ID>*** | ***ASR 7127*** | ***ASR 7192*** | ***ASR 7234*** | ***ASR 7360*** | ***ASR 7489*** |  |
| ***RPL ID>*** | ***05-00225*** | ***05-01060*** | ***05-01274*** | ***05-01933*** | ***06-00029*** |  |
| **Analyte** | **Average, µg/mL(c)** | **Average, µg/mL(c)** | **Average, µg/mL(c)** | **Average, µg/mL(c)** | **Average, µg/mL(c)** | **Average, M(c)** |
| Ag | <1.2 | <1.2 | **<0.30** | **<0.31** | **<0.43** | **<4.0E-6** |
| Al | 6080 | 3850 | **3740** | **2480** | 2,520 | 9.32E-2 |
| As | [8.5] | <4.2 | <1.0 | [2.2](d) | <1.7 | <2.3E-5 |
| Ba | [1.8] | <0.78 | **<0.19** | **[0.48](d)** | **[0.35]** | **[2.5E-6]** |
| Ca | 507 | 352 | **164** | 108 | 116 | 2.89E-3 |
| Cd | 63.4 | 39.7 | **37.8** | **25.0** | **25.3** | **2.25E-4** |
| Cr | 132 | 82.6 | **52.6** | **34.0** | **33.1** | **6.37E-4** |
| K | 2470 | [1250] | **1450** | **969** | **1,040** | **2.66E-2** |
| Mn | 2.05 | [1.2] | 7.12 | [1.7] | **[0.47]** | **[8.6E-6]** |
| Na | **206,000** | **132,000** | **166,000** | **112,500** | **113,500** | **4.94E+0** |
| (Na, M) | **(8.96 M)** | **(5.74 M)** | **(7.22 M)** | **(4.89 M)** | **(4.94 M)** | **--** |
| Ni | 439 | 276 | **260** | **175** | **175** | **2.98E-3** |
| P | 1910 | 1210 | **1140** | **762** | 762 | 2.46E-2 |
| Pb | 168 | 102 | **79.0** | **54.6** | **56.6** | **2.73E-4** |
| Se | <6.5 | <6.6 | <1.6 | [2.4] (d) | [2.4] | [3.1E-5] |
| Sr | 2.82 | 1.71 | **165** | **113** | **114** | **1.30E-3** |
| Th | <4.0 | <4.1 | **<0.98** | **<1.0** | **<1.0** | **<4.4E-6** |
| U | <150 | <156 | **<37** | **<38** | **[60]** | **[2.5E-4]** |
| W | 134 | 85.5 | 83.4 | **59.2** | 61.6 | 3.35E-4 |
| **Density** | **g/mL** | **g/mL** | **g/mL** | **g/mL** |  |  |
| Density | 1.43(e) | 1.30(e) | 1.319 (29°C) | 1.230 (26°C) | -- | -- |

(a) Sample removed on 6/29/05.

(b) Sample removed on 8/29/05.

(c) The overall analytical uncertainty for the analytes of interest was ±15%. Bracketed results were <EQL and >MDL, and error was likely to exceed ± 15%.

(d) High analytical blank values were obtained.

(e) Temperature not recorded.

Notes:

"--" = not measured

Bolded analyte values indicate analytes of interest where data were evaluated relative to QC. Non-bolded analyte values were obtained opportunistically and are provided for information-only as their data may not have been evaluated with respect to QC.

**Table 3.2.** Composition of AN-102 Pretreated Feed

| **Analyte** | **Method** | **Average, µg/mL(a)** | **Average, M(a)** | **Analyte** | **Method** | **Average, µg/mL(a)** | **Average, M(a)** |
|---|---|---|---|---|---|---|---|
| Ag | ICP-AES | <0.43 | <4.0E-6 | $NO_3^-$ | IC | 102,000 | 1.65E+0 |
| Al(b) | ICP-AES | 2,520 | 9.32E-2 | $OH^-$ | Titration | 18,100 | 1.07E+0 |
| As | ICP-AES | <1.7 | <2.3E-5 | P(b) | ICP-AES | 762 | 2.46E-2 |
| Ba | ICP-AES | [0.35] | [2.5E-6] | Pb | ICP-AES | 56.6 | 2.73E-4 |
| Ca(b) | ICP-AES | 116 | 2.89E-3 | $PO_4^{3-}$ | IC | 2050 | 2.16E-2 |
| Cd | ICP-AES | 25.3 | 2.25E-4 | $^{85}$Rb | ICP-MS | 1.57 | 1.85E-5 |
| $Cl^-$ | IC | 1750 | 4.94E-2 | Se | ICP-AES | [2.4] | [3.1E-5] |
| Cr | ICP-AES | 33.1 | 6.37E-4 | Sr | ICP-AES | 114 | 1.30E-3 |
| Cs(c) | ICP-MS | 7.93 | 5.92E-5 | $SO_4^{2-}$ | IC | 6,350 | 6.61E-2 |
| K | ICP-AES | 1,040 | 2.66E-2 | Th | ICP-AES | <1.0 | <4.4E-6 |
| Mn | ICP-AES | [0.47] | [8.6E-6] | U | ICP-AES | [60] | [2.5E-4] |
| Na | ICP-AES | 113,500 | 4.94E+0 | U | KPA | 8.06 | 3.39E-5 |
| Ni | ICP-AES | 175 | 2.98E-3 | W(b) | ICP-AES | 61.6 | 3.35E-4 |
| $NO_2^-$ | IC | 40,600 | 8.83E-1 | -- | -- | -- | -- |
| **Radionuclide** | | **Method** | **µCi/mL(a)** | **Radionuclide** | | **Method** | **µCi/mL(a)** |
| $^{137}$Cs | | GEA | 1.48E+2(d) | $^{242}$Cm | | AEA(e) | [4.0E-5] |
| $^{60}$Co | | GEA | 1.74E-2 | $^{243+244}$Cm | | AEA(e) | 5.09E-4 |
| $^{238}$Pu | | AEA(e) | 1.99E-4(f) | Gross alpha | | Radchem | 8.36E-3 |
| $^{239+240}$Pu | | AEA(e) | 5.61E-4 | Sum of alpha | | AEA(e) | 1.22E-2 |
| $^{241}$Am | | AEA(e) | 1.08E-2 | -- | | -- | -- |

(a) The overall analytical uncertainty was typically ±15%. Results in brackets were <EQL and >MDL (MDL = method detection limit), and errors likely exceeded ± 15%.

(b) Opportunistic analytes measured by ICP-AES. Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

(c) Total Cs was determined from $^{137}$Cs (GEA), $^{133}$Cs (ICP-MS), and the calculated $^{135}$Cs isotopic ratio of 17%.

(d) The ASO-determined $^{137}$Cs concentration was in good agreement with the $^{137}$Cs concentration of 143 µCi/mL determined during process testing from analytical duplicates.

(e) AEA = alpha energy analysis.

(f) Based on one result; the duplicate sample result appeared to be biased high by a factor of 2.

Notes:

ASR 7489 reference date = August 15, 2005.

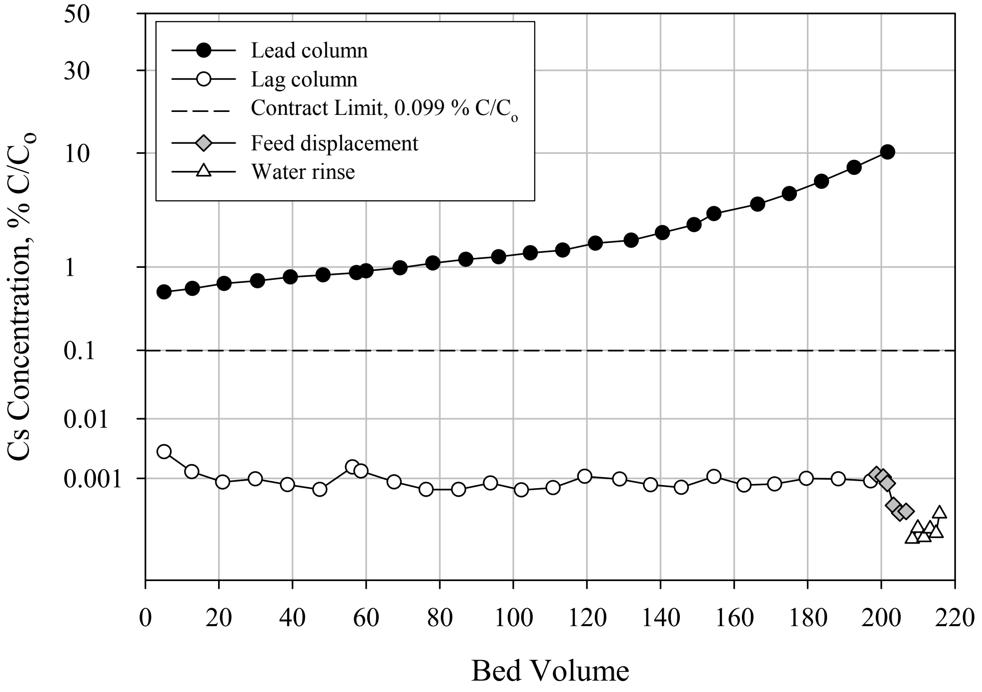
"--" = not applicable

## 3.2 AN-102 Ion Exchange Processing

The Cs load and elution characteristics of AN-102 pretreated tank waste are described in the following sections.

### 3.2.1 Cs Load

The Cs effluent concentrations from the lead and lag columns are shown in Figure 3.1 as %C/C$_o$[a] vs. the BVs of feed processed through each column. The abscissa reflects BVs as a function of the resin in the expanded regeneration condition of 11 mL. During process testing, the C$_o$ value for $^{137}$Cs was determined to be 143 μCi/mL (7.9 μg/mL total Cs). All sample $^{137}$Cs results were greater than the detection limit. The plotted data and measured $^{137}$Cs concentrations are provided in Appendix B.



| Conditions: | | |
|---|---|---|
| | • Spherical RF Lot number 5E-370/641 | • Flow Rate = 2.95 BV/h |
| | • Process temperature = 26 to 27°C | • $^{137}$Cs C$_o$ = 143 μCi/mL |
| | • BV in 0.5 M NaOH feed condition = 11 mL | • Na concentration = 4.94 M |

**Figure 3.1.** $^{137}$Cs Breakthrough Curves for AN-102 Tank Waste, Probability Plot

The contract $^{137}$Cs removal limit is also shown in Figure 3.1. The C/C$_o$ value of 0.099% corresponds to the contract limit of 0.3 Ci/m$^3$ for $^{137}$Cs in the LAW glass. The C/C$_o$ value corresponding to this limit was determined using the Na concentration of 4.94 M and a $^{137}$Cs concentration of 143 μCi/mL in the AN-102 feed, a 12 wt% waste $Na_2O$ loading in the glass, and a glass product density of 2.71 g/mL

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(a) C/C$_o$ = analyte concentration in column effluent (C) divided by analyte concentration in feed (C$_o$).

(Muller and Pegg, 2003, see Appendix A). Projecting to a 2015 WTP start date, $^{137}$Cs decay would result in a concentration of 114 µCi/mL. The associated contract limit for Cs removal would be 0.124% $C/C_o$ and the decontamination factor would be 809 $C_o/C$.

The Cs breakthrough on the lead column was virtually immediate at 0.75 µCi/mL. Slow bleed of $^{137}$Cs from the lead column was a vestige of the partial Cs loading associated with AP-101 processing where 0.057 Ci $^{137}$Cs (or 2.9 mg Cs) remained on the resin bed. The 50% breakthrough was not clearly extrapolated from this load profile. Application-specific sigmoidal curve-fitting programs may be useful in projecting the 50% Cs breakthrough.

The $^{137}$Cs did not break through the lag column over the entire 202-BVs processed. The decontamination factor (DF) for the composite AN-102 effluent was 1.11E+5, virtually two orders of magnitude greater than the contract limit DF of 1.01E+3. This demonstrated that the spherical RF efficiently removed Cs from the low-K tank waste.

The effect of feed displacement (0.1 M NaOH) and water rinse are also shown in Figure 3.1 as a continuation from the lag column effluent collection. The first 5 BVs of feed displacement processed essentially flushed the remaining AN-102 tank waste from the system. The $C/C_o$ remained constant until 1 AV was flushed through the system, at which point the % $C/C_o$ value dropped about a factor of 3. The Cs concentration in the water rinse samples appeared to be generally constant.

The fractionation of the waste constituents is evaluated in Table 3.3, which compares the constituent concentrations in the feed and the effluent. The slight dilution resulting from mixing the initial AV of 0.5 M NaOH with the effluent was taken into account for the "dilution corrected" values. The ratios of the feed analyte to effluent analyte concentrations are also provided. If the resin had no affinity for the analyte, the ratio would equal 1; analyte was potentially lost to the resin where the ratio exceeded 1. Major constituents (Na, K, Al, $NO_3^-$, and $NO_2^-$) in the effluent were similar in concentration to the feed within experimental error. Virtually all elements and ions greater than the EQL (except Cs) were quantitatively recovered in the effluent. Barium concentration (<EQL) was higher in the feed indicating it may have partially exchanged onto the resin. The Mn concentration (<EQL) did not change indicating no additional precipitation (and filtration by the resin bed) occurred. The Si concentration changed appreciably, but this effect was not considered to be associated with an ion exchange process. It is possible silicates precipitated and were effectively filtered by the resin bed.

Table 3.3 also shows the Cs fractionation in the effluent for both $^{133}$Cs and $^{137}$Cs. The effluent $^{133}$Cs was <EQL (thus subject to high uncertainty) and resulted in a DF of 2700, a factor of 2.7× higher than the contract limit. The $^{137}$Cs evaluation resulted in a DF of 111,000. The discrepancy in DFs may be associated with ICP-MS analysis limitations at low concentrations in the tank waste matrix or residual $^{133}$Cs bleed associated with the AP-101 simulant shakedown test.

The total Cs loaded on the ion exchange resin beds after the load and rinses was calculated from integrating the $^{137}$Cs load and breakthrough curves. The lead column (Resin B) was calculated to hold 0.371 Ci $^{137}$Cs, equivalent to 19.7 mg Cs and a loading density of 6.9 mg Cs/g dry H-form resin. The lag column was loaded with 7.84E-3 Ci $^{137}$Cs, equivalent to 0.42 mg Cs and a loading density of 0.15 mg Cs/g dry H-form resin.

**Table 3.3.** AN-102 Cs-Decontaminated Product Effluent

| Analyte | Feed Concentration, M | Effluent Concentration, M | Ratio Feed to Effluent | Dilution-Corrected(a) | |
|---|---|---|---|---|---|
| | | | | Effluent Conc., M | Ratio Feed to Effluent |
| *Target Analytes* | | | | | |
| Ag | <4.0E-6 | <3.9E-6 | na | <4.0E-6 | na |
| As | <2.3E-5 | <2.3E-5 | na | <2.3E-5 | na |
| Ba | [2.55E-6] | [1.09E-6] | [2.33] | [1.12E-6] | **[2.28]** |
| Cd | 2.25E-4 | 2.23E-4 | 1.01 | 2.28E-4 | 0.98 |
| Cr | 6.37E-4 | 6.37E-4 | 1.00 | 6.50E-4 | 0.98 |
| $^{133}$Cs | 3.67E-5 | [1.35E-8] | [2715] | [1.38E-8] | [2657] |
| K | 2.66E-2 | 2.76E-2 | 0.96 | 2.82E-2 | 0.94 |
| Mn | [8.56E-6] | [8.56E-6] | [1.00] | [8.74E-6] | [0.98] |
| Na | 4.94E+0 | 4.83E+0 | 1.02 | 4.93E+0 | 1.00 |
| Ni | 2.98E-3 | 2.93E-3 | 1.02 | 2.99E-3 | 1.00 |
| Pb | 2.73E-4 | 2.33E-4 | 1.17 | 2.38E-4 | 1.15 |
| $^{85}$Rb | 1.85E-5 | 1.56E-5 | 1.18 | 1.60E-5 | 1.16 |
| Se | [3.10E-5] | [3.55E-5] | [0.88] | [3.62E-5] | [0.86] |
| Sr | 1.30E-3 | 1.30E-3 | 1.00 | 1.33E-3 | 0.98 |
| Th | <4.4E-6 | <4.3E-6 | na | <4.4E-6 | na |
| U(b) | [2.54E-4] | [2.35E-4] | [1.08] | [2.40E-4] | [1.06] |
| U (KPA)(c) | 3.39E-5 | 3.30E-5 | 1.03 | 3.37E-5 | 1.00 |
| Chloride | 4.94E-2 | 4.80E-2 | 1.03 | 4.90E-2 | 1.01 |
| Nitrite | 8.83E-1 | 8.48E-1 | 1.04 | 8.66E-1 | 1.02 |
| Nitrate | 1.64E+0 | 1.57E+0 | 1.04 | 1.61E+0 | 1.02 |
| Phosphate | 2.16E-2 | 2.11E-2 | 1.03 | 2.15E-2 | 1.00 |
| Sulfate | 6.61E-2 | 6.45E-2 | 1.02 | 6.59E-2 | 1.00 |
| Free hydroxide | 1.07E+0 | 1.06E+0 | 1.01 | 1.08E+0 | 0.99 |
| *Opportunistic ICP-AES Analytes(d)* | | | | | |
| Al | 9.32E-2 | 9.27E-2 | 1.01 | 9.47E-2 | 0.98 |
| B | 1.95E-3 | 1.50E-3 | 1.30 | 1.53E-3 | 1.27 |
| Be | <1.2E-6 | [1.22E-6] | na | [1.25E-6] | na |
| Bi | [1.10E-5] | [1.00E-5] | [1.10] | [1.03E-5] | [1.07] |
| Ca | 2.89E-3 | 2.79E-3 | 1.04 | 2.86E-3 | 1.01 |
| Co | [3.31E-5] | [3.22E-5] | [1.03] | [3.29E-5] | [1.00] |
| Cu | 1.68E-4 | 1.55E-4 | 1.08 | 1.58E-4 | 1.06 |
| Dy | <3.4E-6 | <3.3E-6 | na | <3.4E-6 | na |
| Eu | [1.74E-6] | [1.65E-6] | [1.06] | [1.68E-6] | [1.04] |
| Fe | [6.00E-5] | [5.19E-5] | [1.16] | [5.31E-5] | [1.13] |
| La | [1.15E-5] | [1.08E-5] | [1.07] | [1.10E-5] | [1.04] |
| Li | [1.49E-4] | [1.40E-4] | [1.07] | [1.43E-4] | [1.04] |
| Mg | <5.9E-5 | <5.8E-5 | na | <5.9E-5 | na |

**Table 3.3** (contd)

| Analyte | Feed Concentration, M | Effluent Concentration, M | Ratio Feed to Effluent | Dilution-Corrected(a) | |
|---|---|---|---|---|---|
| | | | | Effluent Conc., M | Ratio Feed to Effluent |
| Mo | 2.36E-4 | 2.36E-4 | 1.00 | 2.41E-4 | 0.98 |
| Nd | [5.06E-5] | [4.92E-5] | [1.03] | [5.03E-5] | [1.01] |
| P | 2.46E-2 | 2.45E-2 | 1.00 | 2.50E-2 | 0.98 |
| Pd | [1.27E-4] | [1.22E-4] | [1.04] | [1.25E-4] | [1.02] |
| Rh | [7.43E-5] | [7.09E-5] | [1.05] | [7.25E-5] | [1.03] |
| Ru | 1.25E-4 | 1.25E-4 | 1.00 | 1.27E-4 | 0.98 |
| Sb | 1.72E-5 | [1.64E-5] | [1.05] | [1.68E-5] | [1.03] |
| Si | 2.84E-3 | [6.05E-4] | [4.69] | [6.18E-4] | **[4.59]** |
| Sn | [1.22E-4] | [1.35E-4] | [0.91] | [1.38E-4] | [0.89] |
| Te | [3.92E-5] | [4.00E-5] | [0.98] | [4.08E-5] | [0.96] |
| Ti | [4.18E-6] | [3.55E-6] | [1.18] | [3.63E-6] | [1.15] |
| Tl | <7.6E-6 | <7.5E-6 | na | <7.6E-6 | na |
| V | [1.04E-5] | [1.00E-5] | [1.04] | [1.02E-5] | [1.02] |
| W | 3.35E-4 | 3.33E-4 | 1.01 | 3.40E-4 | 0.98 |
| Y | [2.81E-6] | [2.70E-6] | [1.04] | [2.76E-6] | [1.02] |
| Zn(e) | [3.42E-5] | [2.46E-5] | na | [2.52E-5] | na |
| Zr | [1.32E-5] | [1.21E-5] | [1.09] | [1.23E-5] | [1.07] |
| **Radionuclide** | **Feed Concentration, µCi/mL** | **Effluent Concentration, µCi/mL** | **Ratio Feed to Effluent** | **Dilution-Corrected(a)** | |
| | | | | **Effluent Conc., µCi/mL** | **Ratio Feed to Effluent** |
| $^{60}$Co | 1.74E-2 | 2.06E-2 | 0.90 | 2.10E-2 | 0.88 |
| $^{137}$Cs | 1.48E+2 | 1.33E-3 | 111,000 | 1.36E-3 | 109,000 |
| $^{238}$Pu | 1.99E-4(f) | 1.74E-4 | 1.14 | 1.77E-4 | 1.12 |
| $^{239+240}$Pu | 5.61E-4 | 4.96E-4 | 1.13 | 5.06E-4 | 1.11 |
| $^{241}$Am | 1.08E-2 | 1.02E-2 | 1.05 | 1.04E-2 | 1.03 |
| $^{242}$Cm | 4.00E-5 | 3.82E-5 | 1.05 | 3.90E-5 | 1.02 |
| $^{243+244}$Cm | 5.09E-4 | 4.38E-4 | 1.16 | 4.48E-4 | 1.14 |
| Total alpha | 8.36E-3 | 8.53E-3 | 0.98 | 8.71E-3 | 0.96 |
| Sum of alpha | 1.21E-2 | 1.13E-2 | 1.07 | 1.16E-2 | 1.05 |

(a) The dilution correction accounts for the AV of 0.5 M NaOH in the ion exchange system mixed with the composite effluent. For AN-102 processing, this was a 2% correction factor.

(b) The U (ICP-AES) values were barely >MDL; the U (KPA) values were considered more accurate.

(c) Estimated values for U (KPA); see discussion Section 4.2.4.

(d) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

(e) The process blank result for Zn was high, nearly equaling the analyte concentration; recovery calculation was not applicable.

(f) Based on one result; the duplicate sample result appeared to be biased high by a factor of 2.

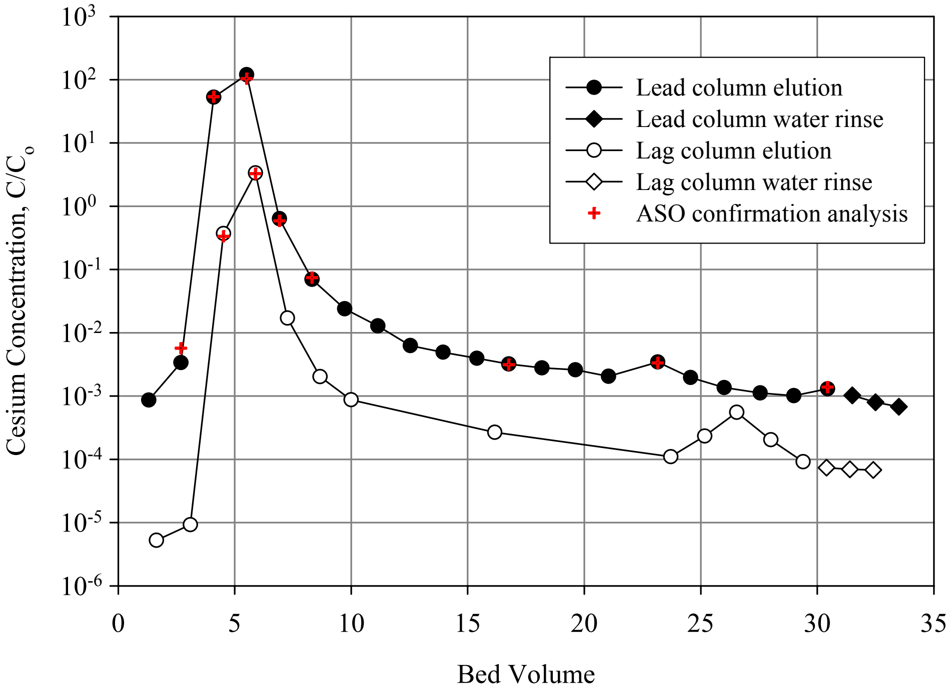
"na" = not applicable, not detected in feed or effluent

Bolded ratios highlight significant change from the feed concentration. Ratios >1 indicate analyte may have exchanged onto the resin or may be lost from the effluent through some other mechanism.

Results from ASR 7489. Reference date August 15, 2005.

### 3.2.2 Elution and Eluant Rinse

The Cs elution and water rinse profiles for the lead and lag columns are provided graphically as $C/C_o$ versus BVs processed in Figure 3.2. The ordinate is a logarithmic scale to clearly show the large range of $C/C_o$ values obtained. The abscissa is given in BVs relative to the regeneration condition on a linear scale. The results from selected samples independently prepared and analyzed by the ASO are also shown, and they agreed well with the process data.



Conditions:
- Spherical RF Microbeads 5E-370/641
- BV in 0.5 M NaOH feed condition = 11 mL
- $^{137}$Cs $C_o$ = 143 µCi/mL; Cs = 7.9 µg/mL
- Process temperature = 25-27°C
- Eluant = 0.5 M $HNO_3$
- Flow Rate = 1.41 BV/h

**Figure 3.2.** Lead- and Lag-Column Elution Profiles from Actual AN-102 Waste Processing

The majority of the $^{137}$Cs was contained in elution BVs 4 to 6. The lead-column $C/C_o$ peak value was found to be 120; the lag-column peak $C/C_o$ value was 3.3. Elution tailed to a level of 1E-3 $C/C_o$ for the lead column and 1E-4 $C/C_o$ for the lag column. Integrating the elution curves resulted in Cs recoveries of 99.6% and 103% for the lead and lag resins, respectively, relative to the calculated Cs loading on each column.

Another perspective for evaluating the elution is to plot the BV on a logarithmic scale as shown in Figure 3.3. The tailing section of the elution profile (BVs 11 to 30) was virtually linear, and an estimate can be obtained for reaching a specific elution condition by extrapolation. The cause of the slight jump in eluate Cs concentrations at ~23 BVs (lead column) and 27 BVs (lag column) was not clear.

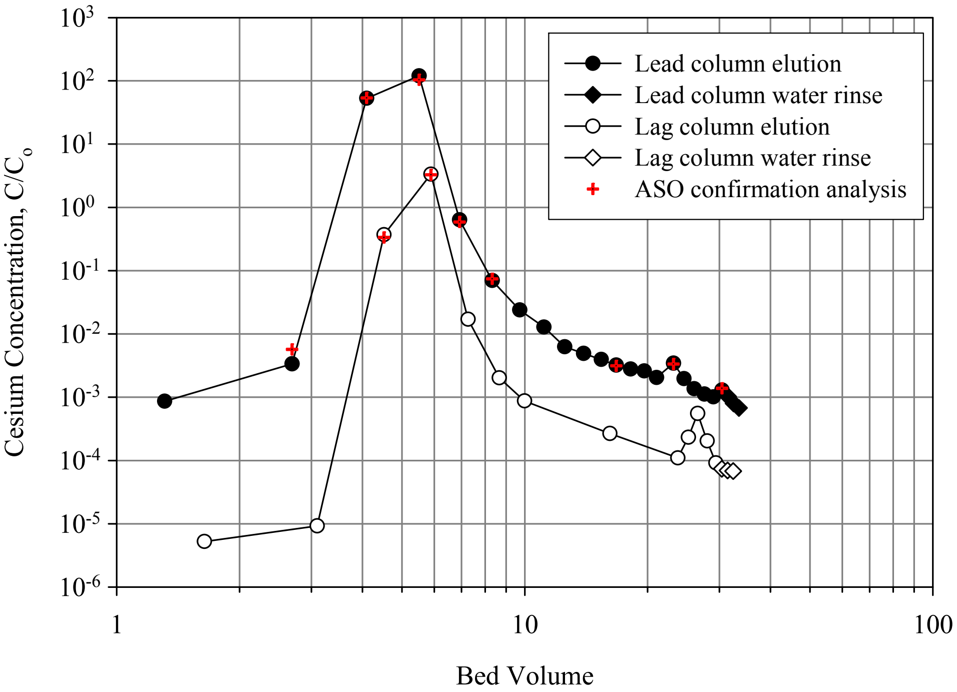


**Figure 3.3.** Lead- and Lag-Column Elution Profiles from Actual AN-102 Waste Processing—Log-Log Scale

The composite eluate analytical results are shown in Table 3.4 and Table 3.5. Sodium was the dominant component detected using ICP-AES. About 10% of the feed Pb was recovered in the eluate. The Cu and Zn also appear to some extent in the eluates. The Ba, Sb, and Zn recoveries in the eluate were confounded by the relatively high analyte values in the diluent blank. As expected, $^{137}$Cs was the dominant radionuclide detected. Nitrate was detected consistent with the eluant composition, along with a trace level of chloride and sulfate.

The SL-644 ion exchanger appeared to have a significantly more active exchange, reaction, or adsorption chemistry for Cr than did spherical RF resin. Processing through spherical RF resulted in Cr recoveries of ~0.24 mg (~0.3% of feed) in the lead column eluate and ~0.17 mg (~0.2% of feed) in the lag column eluate (results were <EQL and confounded with Cr in the process blank); the feed Cr concentration was 6.37E-4 M. A significantly larger fraction of Cr (5.6% or 4.5 mg) was recovered in the eluate from processing the AN-102/C-104 tank waste through SL-644 (Fiskum et al., 2004a). The feed Cr concentration (2.08E-3 M) processed through the SL-644 resin was about a factor of 3 higher than that processed through the RF resin, however, the eluate Cr mass obtained from spherical RF processing was nearly a factor of 20 less than that obtained with SL-644. Although the feed Cr concentration may be pertinent to the eluate Cr recovery, the overall Cr recoveries in SL-644 process eluates (AP-101 2.4 mg or 1.4% [Fiskum et al., 2002a], AZ-102 11.3 mg or 0.70% [Fiskum et al. 2002b], AZ-101 5.6 mg or 0.72% [Fiskum et al. 2003a], AP-104/SY-101 4.5 mg or 0.59% [Fiskum et al 2003b]) were consistently higher than those obtained with RF.

**Table 3.4.** Lead- and Lag-Column Composite Eluate Analysis

| | | Diluent Blank | Lead-Column Eluate | | Lag-Column Eluate | |
|---|---|---|---|---|---|---|
| **Analyte** | **Method** | µg/mL | µg/mL | **% recovered** | µg/mL | **% recovered** |
| *Target Analytes* | | | | | | |
| Ag | ICP-AES | <0.44 | <0.35 | <1.2E+1 | <0.35 | <1.2E+1 |
| As | ICP-AES | <1.8 | <1.4 | <1.2E+1 | <1.4 | <1.2E+1 |
| Ba(a) | ICP-AES | [0.78] | [0.55] | [2.4E+1] | [0.40] | [1.6E+1] |
| Cd | ICP-AES | <0.12 | [0.30] | [1.8E-1] | [0.17] | [9.9E-2] |
| $Cl^-$ | IC | [0.069] | [3.7] | [3.2E-2] | [2.3] | [1.9E-2] |
| Cr(a) | ICP-AES | [0.23] | [0.70] | [3.2E-1] | [0.53] | [2.3E-1] |
| $^{133}Cs$ | ICP-MS | [1.0E-4] | 34.6 | 107 | na | na |
| K | ICP-AES | <95 | <75 | <1.1E+0 | [82] | [1.1E+0] |
| Mn | ICP-AES | <0.050 | [0.09] | [2.7E+0] | [0.079] | [2.4E+0] |
| Na | ICP-AES | <12 | 928 | 1.2E-1 | 1,085 | 1.4E-1 |
| Ni | ICP-AES | <0.31 | 3.48 | 3.0E-1 | 6.53 | 5.4E-1 |
| $NO_2^-$ | IC | <0.05 | <2.4 | <8.9E-4 | <1.9 | <6.8E-4 |
| $NO_3^-$ | IC | <0.02 | 29,250(b) | na | 28,800(c) | na |
| Pb | ICP-AES | <1.9 | 38.6 | 1.0E+1 | 33.7 | 8.6E+0 |
| Se(a) | ICP-AES | [2.07] | <1.5 | <9.4E+0 | <1.5 | <9.0E+0 |
| $SO_4^{2-}$ | IC | <0.02 | [26] | [6.2E-2] | [24] | [5.5E-2] |
| Sr(a) | ICP-AES | [0.08] | 0.74 | 9.7E-2 | 0.587 | 7.5E-2 |
| Th | ICP-AES | <1.0 | <0.81 | <1.2E+1 | <0.81 | <1.2E+1 |
| U | ICP-AES | <57 | <45 | <1.1E+1 | <45 | <1.1E+1 |
| U | KPA | 1.52E-3 | 2.31 | 4.3E+0 | 1.11 | 2.0E+0 |
| *Opportunistic Analytes(d)* | | | | | | |
| Al | ICP-AES | <2.9 | [7.05] | [4.2E-2] | [3.75] | [2.2E-2] |
| B | ICP-AES | <0.38 | 6.55 | 4.7E+0 | [2.10] | [1.4E+0] |
| Be | ICP-AES | <0.011 | [0.02] | [2.2E+1] | <0.008 | <1.2E+1 |
| Bi | ICP-AES | <1.7 | <1.4 | <8.9E+0 | <1.4 | <8.6E+0 |
| Ca | ICP-AES | <1.1 | [1.80] | [2.3E-1] | [2.35] | [2.9E-1] |
| Co | ICP-AES | <0.31 | <0.24 | <1.9E+0 | <0.24 | <1.8E+0 |
| Cu | ICP-AES | <0.81 | 8.68 | 1.23E+1 | 9.05 | 1.24E+1 |
| Dy | ICP-AES | <0.55 | <0.44 | <1.2E+1 | <0.44 | <1.2E+1 |
| Eu | ICP-AES | <0.17 | <0.14 | <7.9E+0 | <0.14 | <7.6E+0 |
| Fe | ICP-AES | <0.51 | [0.95] | [4.3E+0] | [0.61] | [2.6E+0] |
| La | ICP-AES | <0.77 | <0.61 | <5.7E+0 | <0.61 | <5.5E+0 |
| Li | ICP-AES | <0.57 | <0.45 | <6.5E+0 | <0.45 | <6.3E+0 |

**Table 3.4** (contd)

| | | Diluent Blank | Lead-Column Eluate | | Lag-Column Eluate | |
|---|---|---|---|---|---|---|
| **Analyte** | **Method** | **μg/mL** | **μg/mL** | **% recovered** | **μg/mL** | **% recovered** |
| Mg | ICP-AES | <1.5 | <1.1 | <1.2E+1 | <1.2 | <1.2E+1 |
| Mo(a) | ICP-AES | [0.41] | [0.29] | [1.9E-1] | [0.31] | [2.0E-1] |
| Nd | ICP-AES | <3.6 | <2.8 | <5.8E+0 | <2.8 | <5.6E+0 |
| P | ICP-AES | <2.0 | [2.70] | [5.3E-2] | [1.80] | [3.4E-2] |
| Pd | ICP-AES | <9.6 | <7.6 | <8.5E+0 | <7.6 | <8.2E+0 |
| $PO_4^{3-}$ | IC | [0.51] | <10 | <7.3E-2 | <8.0 | <5.7E-2 |
| $^{85}$Rb | ICP-MS | [3.7E-4] | 0.587 | 5.63E+0 | na | na |
| Rh | ICP-AES | <4.4 | <3.5 | <6.9E+0 | <3.5 | <6.6E+0 |
| Ru | ICP-AES | <1.1 | <0.90 | <1.1E+0 | <0.90 | <1.0E+0 |
| Sb(a) | ICP-AES | [2.59] | [1.75] | [1.3E+1] | [1.85] | [1.3E+1] |
| Si | ICP-AES | <2.9 | [14] | [2.6E+0] | [9.10] | [1.7E+0] |
| Sn | ICP-AES | <12 | <9.6 | <1.0E+1 | <9.6 | <9.6E+0 |
| Te | ICP-AES | <2.5 | <2.0 | <5.9E+0 | <2.0 | <5.7E+0 |
| Ti | ICP-AES | <0.17 | <0.13 | <1.0E+1 | <0.13 | <9.7E+0 |
| Tl | ICP-AES | <1.6 | <1.2 | <1.2E+1 | <1.2 | <1.2E+1 |
| V | ICP-AES | <0.33 | <0.26 | <7.3E+0 | <0.26 | <7.1E+0 |
| W | ICP-AES | <1.1 | <0.84 | <2.0E-1 | <0.84 | <2.0E-1 |
| Y | ICP-AES | <0.15 | <0.12 | <7.1E+0 | <0.12 | <6.8E+0 |
| Zn(a) | ICP-AES | 1.67 | 5.72 | 3.85E+1 | 5.45 | 3.55E+1 |
| Zr | ICP-AES | <0.42 | <0.33 | <4.2E+0 | <0.33 | <4.0E+0 |

(a) Analyte diluent blank values were high relative to the sample results.

(b) The measured nitrate concentration of 0.472 M was consistent with the calculated molarity incorporating dilution from the lead-column AV of 26 mL water:
$[(339 - 26)\ \text{mL} * 0.5\ \text{M}\ HNO_3]/339\ \text{mL} = 0.462\ \text{M}\ HNO_3$.

(c) The measured nitrate concentration of 0.464 M was consistent with the calculated molarity incorporating dilution from the lag-column AV of 26 mL water:
$[(327 - 26)\ \text{mL} * 0.5\ \text{M}\ HNO_3]/327\ \text{mL} = 0.460\ \text{M}\ HNO_3$.

(d) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

Results from ASR 7489.

Notes: The overall error was estimated to be within ± 15%. Values in brackets were within 10-times the detection limit, and errors were likely to exceed ± 15%.

na = not applicable

**Table 3.5.** Lead- and Lag-Column Eluate Composite Analysis, Radiochemistry

| | | | Lead-Column Eluate | | | Lag-Column Eluate | | |
|---|---|---|---|---|---|---|---|---|
| Analyte | Method | Blank µCi/mL | Cs eluate µCi/mL | Error % | Recov. % | Cs eluate µCi/mL | Error % | Recov. % |
| $^{137}$Cs | GEA | <9E-5 | 1,110 | 5 | 1.13E+2[b] | 24.7 | 3 | 2.43E+0 |
| $^{238}$Pu | Radchem | <4E-8 | 1.61E-5 | 8 | 7.31E-1 | 1.18E-5 | 8 | 5.19E-1 |
| $^{239+240}$Pu | Radchem | 6.51E-8 | 3.20E-5 | 5 | 8.58E-1 | 1.94E-5 | 7 | 5.02E-1 |
| $^{241}$Am | Radchem | <4E-8 | 6.77E-5 | 4 | 9.49E-2 | 4.25E-5 | 4 | 5.74E-2 |
| $^{242}$Cm | Radchem | <2E-8 | <5.E-7 | na | <2.E-1 | <4.E-7 | na | <1.E-1 |
| $^{243+244}$Cm | Radchem | <2E-8 | 3.96E-6 | 15 | 1.17E-1 | 1.00E-5 | 8 | 2.86E-1 |
| Total alpha | Radchem | <2E-5 | <4.E-4 | na | <7.2E-1 | <4.E-4 | na | <7.E-1 |
| Alpha sum[a] | Radchem | 1.61E-7 | 1.20E-4 | 3 | 1.49E-1 | 8.40E-5 | 3 | 1.00E-1 |

(a) The alpha sum (total of individually-measured Pu, Am, and Cm alpha emitters) provided the best estimate of the total alpha activity in the samples.

(b) The $^{137}$Cs recovery was calculated relative to AN-102 feed $^{137}$Cs concentration; it did not correct for the $^{137}$Cs remaining from AP-101DF processing. See Section 3.2.3.

na = not applicable

ASR 7489, reference date August 15, 2005.

The WTP will need to understand the sodium usage for RF resin regeneration. The Na usage can be estimated from the total cation exchange equivalent. The resin cation exchange equivalent was estimated from the eluate metal recoveries. Table 3.6 summarizes relevant recovered analyte concentrations and milliquivalents (meq) from the lead and lag resin beds. As expected, Na was the dominant meq source. The average 1.34 meq/mL (expanded Na-form resin) indicated that per 1-L RF resin, 1.34 moles Na will be consumed during the conversion process to the Na-form. In comparison, SL-644 test data from AN-102/C-104 processing showed a sum of 8.1 meq were recovered in the eluate, which corresponded to 0.78 meq/mL (based on the expanded Na-form resin of 10.4 mL) (Fiskum et al., 2004a). Thus on a per unit volume expanded resin bed basis, the RF resin requires 1.7× the Na mass for resin regeneration compared to SL-644.

**Table 3.6.** Milliequivalents Recovered in Eluates

| **Analyte (Charge)**[c] | **Lead Column**[a] | | | **Lag Column**[b] | | |
|---|---|---|---|---|---|---|
| | **μg/mL** | **M** | **meq** | **μg/mL** | **M** | **meq** |
| Cs (1) | 59.3 | 4.40E-4 | 1.49E-1 | 1.32 | 9.80E-6 | 3.21E-3 |
| Na (1) | 928 | 4.03E-2 | 1.37E+1 | 1085 | 4.72E-2 | 1.54E+1 |
| Ni (2) | 3.48 | 5.92E-5 | 4.01E-2 | 6.53 | 1.11E-4 | 7.28E-2 |
| Pb (2) | 38.6 | 1.86E-4 | 1.26E-1 | 33.7 | 1.62E-4 | 1.06E-1 |
| Sr (2) | 0.736 | 8.39E-6 | 5.69E-3 | 0.587 | 6.70E-6 | 4.38E-3 |
| Cu (2) | 8.68 | 1.37E-4 | 9.25E-2 | 9.05 | 1.42E-4 | 9.32E-2 |
| U (2)[d] | 2.31 | 9.71E-6 | 6.58E-3 | 1.11 | 4.64E-6 | 3.04E-3 |
| $^{85}$Rb (1) | 0.587 | 6.90E-6 | 2.34E-3 | na | na | na |
| Sum meq | | | 1.41E+1 | | | 1.57E+1 |
| meq/mL[e] | | | 1.27 | | | 1.42 |

(a) Lead column eluate volume was 339 mL.
(b) Lag column eluate volume was 327 mL.
(c) Several cations exist in multiple oxidation states, those shown were applied to the calculation.
(d) U was assumed to exist as the uranyl ($UO_2^{2+}$)
(e) Based on 11.1 mL total resin bed volume.
na = not analyzed; meq = milliequivalents

### 3.2.3 Activity Balance for $^{137}$Cs

An activity balance for $^{137}$Cs was completed to compare the $^{137}$Cs recovered in various process streams to the $^{137}$Cs present in the combined feed sample and residual $^{137}$Cs from AP-101DF processing. The activity balance verified that all $^{137}$Cs was accounted for, thus supporting overall experimental integrity. These results are summarized in Table 3.7. The lag-column Cs loading was calculated by integrating the lead-column Cs breakthrough and subtracting the lag-column effluent Cs breakthrough. Good agreement between the eluted Cs and the calculated loading was obtained. The activity sum for the various process streams resulted in 99.6% Cs recovery. The overall analysis uncertainty was estimated to be ±8%, and the calculated bias was within the experimental uncertainty.

**Table 3.7.** Activity Balance for $^{137}$Cs

| **Solution Matrix** | **$^{137}$Cs, μCi** | **$^{137}$Cs, %** |
|---|---|---|
| **Input** | | |
| Carryover from AP-101 | 5.67E+4 | 1.50E+1 |
| Feed Sample | 3.22E+5 | 8.50E+1 |
| **Sum of inputs** | 3.79E+5 | 1.00E+2 |
| **Output** | | |
| Composite Effluent | 2.84E+0 | 7.50E-4 |
| Load samples | 1.75E+2 | 4.62E-2 |
| Feed displacement | 9.55E-2 | 2.52E-5 |
| Water rinse | 1.54E-2 | 4.07E-6 |
| Eluate lead column | 3.69E+5 | 9.74E+1 |
| Water rinse, lead column | 3.99E+0 | 1.05E-3 |
| Eluate lag column | 8.17E+3 | 2.16E+0 |
| Water rinse, lag column | 3.41E-1 | 9.00E-5 |
| **Total $^{137}$Cs Recovery** | 3.77E+5 | 9.96E+1 |

### 3.2.4 Resin Volume Changes

Like SL-644, the RF resin is known to change in volume as a function of the solution pH and ionic strength. The resin BV change history is shown in Table 3.8. After loading into the hot cell, the H-form resin volume appeared slightly smaller, attributed to the tighter packing associated with small jostling and vibrations. Over the limited number of cycles tested, the resin appeared to trend toward higher volumes in the Na-form. From the first cycle to third cycle, the Na-form volume increased from 11.1 mL to 11.5 mL. The H-form resin volume change was less pronounced.

The variation in BV as a function of the process step (given in Table 3.8) for both resin beds is shown in Figure 3.4. The absolute BV (a) and relative BV (b) are shown. The relative BVs were normalized to the volume in the 0.5 M NaOH regeneration condition just before AP-101 simulant loading. All processing from the initial in-column bed conditioning steps and the three process cycles (simulant AP-101, actual waste AP-101, and actual waste AN-102) are shown. The spherical RF demonstrated a nominal 22% volume change from the Na-form to the H-form. In contrast, SL-644 was found to expand and contract between 25% and 30% depending on lot production or formulation parameters (Fiskum et al. 2004b). The volume calculation error was determined to be 0.35 mL based on the height measurement uncertainty of 0.2 mm. The error bars are added for the AP-101 simulant lead column only. All data points had similar errors.

**Table 3.8.** RF Resin Bed Volume Changes

| Solution Matrix | Process Step Number | Symbol | Resin Volume, mL(1) | |
|---|---|---|---|---|
| | | | **Resin A (Lead)** | **Resin B (Lag)** |
| Initial packing | 1 | P | 10.8 | 11.1 |
| DI water | 2 | W | 10.4 | 10.6 |
| 0.5 M $HNO_3$ | 3 | E | 8.8 | 8.8 |
| DI water | 4 | W | 8.7 | 8.8 |
| 0.5 M NaOH | 5 | R | 11.1(2) | 11.1(2) |
| AP-101 simulant | 6 | F | 11.0 | 11.1 |
| 0.1 M NaOH | 7 | FD | 10.9 | 11.1 |
| DI water | 8 | W | 10.4 | 11.0 |
| 0.5 M $HNO_3$ | 9 | E | 8.7 | 8.8 |
| DI water | 10 | W | 8.7 | 8.9 |
| Transfer to Hot Cell | 11 | W | 8.7 | 8.5 |
| 0.5 M NaOH | 12 | R | 11.3 | 11.3 |
| AP-101DF | 13 | F | 11.1 | 11.1 |
| 0.1 M NaOH | 14 | FD | 11.3 | 11.5 |
| DI water | 15 | W | 10.8 | 11.0 |
| 0.5 M $HNO_3$ | 16 | E | 9.0 | — |
| DI water | 17 | W | 9.0 | — |
| | | | **Resin A (Lag)** | **Resin B (Lead)** |
| 0.5M NaOH | 18 | R | 11.3 | 11.3 |
| AN-102 | 19 | F | 11.5 | 11.5 |
| 0.1 M NaOH | 20 | FD | 11.5 | 11.3 |
| DI water | 21 | W | 11.3 | 11.1 |
| 0.5 M $HNO_3$ | 22 | E | 9.0 | 9.2 |
| DI water | 23 | W | 9.0 | 8.8 |

(1) Calculated pretreated resin mass dry H-form: 2.870 g.

(2) Reference resin bed volume.

Note: The inside diameter of each column was 1.5 cm.

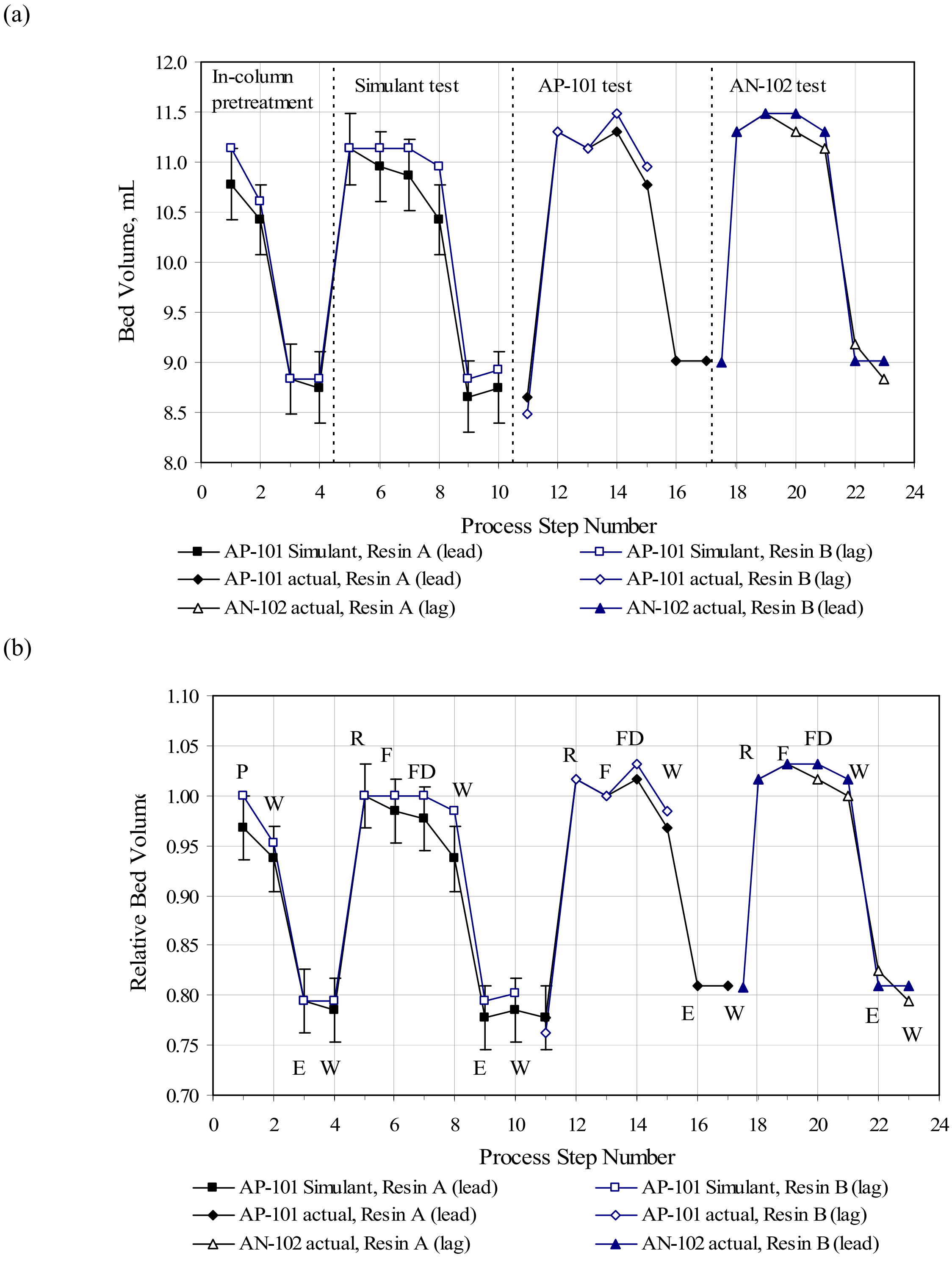


**Figure 3.4.** Bed Volume Comparison of the Lead and Lag Columns During Various Process Stages (a) Absolute BV; (b) Relative BV

# 4.0 Quality Control

The following sections describe the quality assurance (QA) and QC requirements, implementation, and results.

## 4.1 Quality-Assurance Requirements

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP QA organization. This work was performed to the quality requirements of NQA-1-1989, Part I, “Basic and Supplementary Requirements,” NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev. 13, *Quality Assurance Requirements and Descriptions* (QARD). These quality requirements were implemented through PNWD’s *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual* and to the approved Test Plan, TP-RPP-WTP-378, Rev. 0, and Test Exception 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. The analytical requirements were implemented through WTPSP’s Statement of Work (WTPSP-SOW-005) with the RPL ASO.

Experiments that were not method-specific were performed in accordance with PNWD’s procedures QA-RPP-WTP-1101 “Scientific Investigations” and QA-RPP-WTP-1201 “Calibration Control System,” verifying that sufficient data were taken with properly calibrated measuring and test equipment (M&TE) to obtain quality results.

BNI’s QAPjP, 24590-QA-0001, was not applicable because the work was not performed in support of environmental/regulatory testing, and the data should not be used as such.

PNWD addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNWD’s procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the test-plan objectives. This review procedure is part of PNWD’s *WTPSP Quality Assurance Requirements and Description Manual*.

## 4.2 Analytical Results

Data quality and QC are discussed for each analytical method. Analytical results and batch QC results are summarized in Table 4.1 through Table 4.5 along with QC and duplicate precision (relative percent difference [RPD]), and MS and BS recoveries. The RPD was evaluated only for analytes >EQL. All raw and reduced data are maintained and/or cross-referenced in data files under Project 42365 at PNWD.

The AN-102 feed and effluent sample preparation and analyses were conducted simultaneously with the AP-101DF feed and effluent preparation and analysis. Similarly, the AN-102 eluate composite analysis was conducted with the AP-101 eluate composite analysis and a simulant process test eluate analysis (supporting technical scoping statement A-225). Therefore, the analytical batch and instrument QC applied to all sample sets. In some cases, the batch MS may be associated with the AP-101DF matrix and the post spike and serial dilution may be associated with the simulant eluate matrix.

### 4.2.1 Sample Preparation

Because of the high sample activity, all initial sample preparations (sub-sampling, dilution, and/or digestion) were conducted in the SAL.

The stepwise pretreatment of AN-102 supernatant required only Na analysis by ICP-AES. Because Na was a major component (5 to 9 M), only sample dilution was necessary for analysis. In these cases, only a diluent blank, sample duplicate, and instrument QC were required.

Acid digestion of the tank waste was required to analyze U (KPA), total alpha, Pu, Am, Cm, ICP-AES for minor and trace metal constituents, and ICP-MS. In these cases, a MS, BS, PB, sample duplicate, and instrument QC were required. For the radiochemical analytes (Pu, Am, Cm, and total alpha), the BS and MS were prepared at the analytical workstation, not during the digestion process. These were termed "post-digestion spikes." Because of the large dilutions required of highly radioactive samples for radioisotopic analysis, spiking at the digestion stage would have required too much consumption of rare and expensive spike standards ($^{239}$Pu, $^{241}$Am, and associated tracers). Routine practice dictated the use of these spikes at the workstation on the smaller aliquots actually submitted through the radiochemical separations and analysis process.

The feed and effluent were diluted as necessary and analyzed directly by GEA, IC, and titration (hydroxide). The eluate samples (0.5 M $HNO_3$ matrix) were diluted as needed and analyzed directly by all methods. In these cases, a sample duplicate and a diluent blank were also prepared.

### 4.2.2 Inductively-Coupled Plasma-Atomic Emission Spectrometry

The U concentration in the feed and effluent measured by ICP-AES under ASR 7489 was not credible. It was barely detected above the MDL and it did not agree well with previous measures of the analyte (ASRs 7234 and 7360) that resulted in <38 μg/mL values or the U (KPA) measurement, which was much more consistent with previous analytical results . The Zn (opportunistic analyte) analysis can only be interpreted as "less-than" values because the process blank nearly equaled the measured sample concentrations.

The tank waste matrix analytical batch MS was prepared with a companion sample AP-101DF tank waste. The BS and MS recoveries were within the acceptance criteria. The sample Na concentration was too high to evaluate the MS and post-spike recoveries. The samples required further dilution (serial dilution) at the workstation to bring Na in the calibration range. Elements As and Se were not present in the spiking solution, so their BS and MS recoveries could not be evaluated. These elements were post-spiked into the prepared samples at the workstation with acceptable QC recovery.

The eluate sample suite included the companion sample from A-225 testing that was post-spiked at the workstation as part of the batch QC. The post-spiked Na and K were <25% of the sample concentrations, so recoveries of these analytes could not be evaluated. Analytes at >EQL met the precision requirements.

Overall analyte concentration uncertainties were within ± 15% for analytes >EQL. All instrument QC met acceptance criteria.

### 4.2.3 Inductively Coupled Plasma-Mass Spectrometry

The ICP-MS was used to determine the $^{133}$Cs and $^{85}$Rb concentrations. The tank waste MS was prepared from an aliquot of the AN-102 effluent. The eluate MS was prepared from the lead column eluate sub-sample. Overall analyte concentration uncertainties were within ± 15%. All batch and instrument QC requirements were met.

### 4.2.4 Uranium by Kinetic Phosphorescence Analysis

The U (KPA) results, though significantly less than the U (ICP-AES) results, were considered more accurate than the ICP-AES results. The U (KPA) preparation batch did not include a low-level U BS or MS. The U MS and BS prepared as part of the digestion batch included a plethora of interfering analytes that precluded direct analysis by KPA. Therefore, U (KPA) BS and MS samples were not measured and recoveries were not reported; the analysis results were caveated as estimates. The companion sample, AP-101DF, U result (42.6 mg/L) agreed well with the previously reported result (Fiskum et al. 2000, after dilution correction). Because the eluate was directly analyzed (no digestion preparation), a BS and MS were not required. The RPDs of duplicate samples were found to be within the acceptance criterion of <15%. All instrument QC requirements were met.

### 4.2.5 Gamma Energy Analysis

Gamma energy analysis was conducted directly and non-destructively on direct or diluted samples. Therefore, laboratory control samples as well as BS, and MS QC samples were not required. All batch and instrument QC requirements were met. Analyte concentration uncertainty represented the total propagated uncertainty from all sources, including sub-sampling, calibration, and counting uncertainty.

### 4.2.6 Americium, Curium, and Plutonium

The alpha spectral analyses suffered several problems. The AN-102 feed matrix $^{238}$Pu RPD was high at 80%. The duplicate sample value was probably biased high by $^{241}$Am cross contamination. Data calculations were therefore based on one sample data point (1.99E-4 µCi/mL). The feed sample RPDs for $^{239+240}$Pu and $^{243+244}$Cm exceeded the acceptance criterion. The result averages were used for isotope recovery calculations.

One lead eluate sample preparation for the Am/Cm analysis resulted in poor alpha spectral resolution, and the data were rejected; therefore, the RPDs could not be evaluated. Both the lead- and lag-column eluate $^{238}$Pu results exceeded the RPD acceptance criterion. The lead-column duplicate eluate $^{238}$Pu result (7.39E-6 µCi/mL) appeared unlikely in that the relative $^{239+240}$Pu isotopic ratio was 3× higher than found in the other samples; therefore, the reported duplicate value was assumed to be biased low and was rejected. The lag-column eluate $^{243+244}$Cm RPD results exceeded the acceptance criterion. The averages of the lag-column isotopic results were used for isotope recovery evaluations.

All analyte concentrations were well below the minimum reportable quantity (MRQ). The BS and MS recoveries met the acceptance criteria. All instrument QC requirements were met. Reported analyte concentration uncertainty represented the total propagated uncertainty from all sources, including sub-sampling, calibration, and counting uncertainty.

### 4.2.7 Total Alpha

The BS and MS recoveries met the acceptance criteria. The RPD exceeded the acceptance criteria for the feed matrix. The feed and effluent contained high dissolved solids that tend to interfere with the total alpha measurement by absorption of the alpha particles. The sum of alpha isotopes (Pu, Am, and Cm) measured by alpha energy analysis (AEA) was a better measure of the total alpha composition. The RPD of the calculated sum met the acceptance criterion for the feed and effluent; the lag-column eluate RPD was a factor of 2× higher than the acceptance criterion. All instrument QC requirements were met.

### 4.2.8 Ion Chromatography

All batch and instrument QC requirements were met.

### 4.2.9 Hydroxide

All batch QC requirements were met. The diluent blank pH was 4.45 and therefore was not subject to analysis by titration.

**Table 4.1.** QC Results of Sodium Analysis Supporting Pretreatment

| | | | **Diluent Blank** | **Sample Na** | **Duplicate Na** | **RPD** | **Serial Dilution** |
|---|---|---|---|---|---|---|---|
| **ASR** | **Description** | **ASO ID** | **μg/mL$^{(a)}$** | **μg/mL$^{(a)}$** | **μg/mL$^{(a)}$** | **%** | **%** |
| | | *Criteria>* | *<EQL* | *none* | *none* | *≤10* | *<10* |
| 7127 | As-received composite | 05-00225 | <15 | 224,000 | -- | -- | 0.8 |
| 7127.01 | As-received composite | 05-00225 | <24 | 200,000 | 195,000 | 2.5 | 5.2 |
| 7192 | Diluted composite | 05-01060 | [31] | 137,000 | na | 6.9 | 3.1 |
| | Diluted composite, duplicate | 05-01061 | | 128,000 | na | | |
| 7234 | Sr/TRU removed supernatant | 05-01274 | <29 | 166,000 | 166,000 | 0.2 | 3.2 |
| 7360 | Diluted supernatant after Sr/TRU removal | 05-01933 | <30 | 112,500 | 113,500 | 0.6 | not reported |

(a) The overall error for values without brackets was estimated to be within ±15% (analytes greater than the EQL). Bracketed values identify sample concentrations that were <EQL but >MDL, and errors likely exceeded 15%. The MDL was typically a factor of 10 lower than the EQL.

"--" indicates measurement was not made.

### **Table 4.2.** Analysis QC Results of AN-102 Ion Exchange Feed and Effluent

| Analyte | Method | MRQ µg/mL | EQL µg/mL | Prep Blank µg/mL[a] | Feed 06-00029 Sample µg/mL[a] | Feed 06-00029 Duplicate µg/mL[a] | Effluent 06-00030 µg/mL[a] | RPD (06-00029) % | Serial Dilution (06-00029) % | BS Recovery % | 06-00026 MS[c] Recovery % | 06-00029 Post Spike A % | 06-00029 Post Spike B % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ***Criteria>*** | | | | | | | | ***≤15[b]*** | ***≤10*** | ***80 - 120*** | ***75 - 125*** | ***75 - 125*** | ***75 - 125*** |
| *Target Analytes* | | | | | | | | | | | | | |
| Ag | ICP-AES | 14 | 4.4 | <0.44 | <0.44 | <0.43 | <0.43 | -- | -- | 90 | 93 | 102 | na |
| As | ICP-AES | 72 | 17 | <1.7 | <1.75 | <1.71 | <1.7 | -- | -- | **nm** | **nm** | 111 | na |
| Ba | ICP-AES | 78 | 1.2 | <0.12 | [0.31] | [0.39] | [0.15] | -- | -- | 101 | 101 | 101 | na |
| Cd | ICP-AES | 7.5 | 1.2 | [0.14] | 25.0 | 25.5 | 25.1 | 2.1 | 7.3 | 100 | 100 | 99 | na |
| Cr | ICP-AES | 15 | 1.8 | [0.23] | 32.6 | 33.6 | 33.1 | 3.3 | 6.3 | 102 | 104 | 103 | na |
| $^{133}$Cs | ICP-MS | 1.0 | 1E-4 | 2.9E-4 | 4.87 | 4.90 | [1.8E-3] | 0.64 | -- | 101 | 100 | 102 | na |
| K | ICP-AES | nmrq | 950 | <95 | 1,030 | 1,050 | 1,080 | 1.2 | -- | 100 | 113 | na | na |
| Mn | ICP-AES | nmrq | 0.5 | <0.050 | [0.45] | [0.49] | [0.47] | -- | -- | 99 | 99 | 99 | na |
| Na | ICP-AES | 180 | 120 | <12 | 113,000 | 114,000 | 111,000 | 1.3 | 3.7[d] | 103 | nr[e] | nr[e] | na |
| Ni | ICP-AES | 30 | 3.1 | [0.73] | 173 | 177 | 172 | 2.6 | 5.6 | 101 | 101 | 103 | na |
| Pb | ICP-AES | 300 | 19 | <1.9 | 56.0 | 57.1 | 48.3 | 1.9 | -- | 84 | 101 | 103 | na |
| $^{85}$Rb | ICP-MS | nmrq | 1E-3 | <0.026 | 1.57 | 1.57 | 1.33 | 0.32 | -- | 100 | 98 | 102 | na |
| Se | ICP-AES | 50 | 19 | <1.9 | [2.4] | [2.5] | [2.8] | -- | -- | **nm** | **nm** | 106 | na |
| Sr | ICP-AES | nmrq | 0.48 | [0.046] | 112 | 116 | 114 | 3.3 | 3.3 | 102 | 104 | 102 | na |
| Th | ICP-AES | 500 | 10 | <1.0 | <1.03 | <1.01 | <1.00 | -- | -- | 102 | 105 | na | 104 |
| U | ICP-AES | 1,000 | 570 | <57 | [64] | [57] | [56] | -- | -- | 103 | 104 | na | 105 |
| U | KPA | 50 | | 0.189 | 7.87 | 8.25 | 7.85 | 5 | -- | **nm** | **nm** | na | na |
| Chloride | IC | 10 | 125 | [0.069] | 1,800 | 1,700 | 1,700 | 5 | -- | 97 | 97 | na | na |
| Nitrite | IC | 3,000 | 239 | <0.048 | 40,300 | 40,900 | 39,000 | 1 | -- | 100 | 104 | na | na |
| Nitrate | IC | 3,000 | 996 | <0.20 | 101,000 | 102,000 | 97,600 | 1 | -- | 97 | 98 | na | na |
| Phosphate | IC | nmrq | 100 | [0.51] | 2,100 | 2,000 | 2,000 | 3 | -- | 84 | 86 | na | na |
| Sulfate | IC | 2,300 | 100 | <0.20 | 6,400 | 6,300 | 6,200 | 2 | -- | 94 | 94 | na | na |
| Hydroxide | Titration | 17 | na | na | 17,950 | 18,300 | 18,000 | 2.8/1.3[f] | -- | 95 | 91 | na | na |
| *Opportunistic Analytes[g]* | | | | | | | | | | | | | |
| Al | ICP-AES | nmrq | 29 | <2.9 | 2,490 | 2,540 | 2,500 | 2.3 | 4.1 | 98 | 97 | 98 | na |
| B | ICP-AES | nmrq | 3.8 | <0.38 | 20.9 | 21.3 | 16.2 | 1.9 | 9.5 | 87 | 94 | 98 | na |
| Be | ICP-AES | nmrq | 0.11 | <0.011 | <0.011 | <0.010 | [0.011] | -- | -- | 102 | 106 | 105 | na |

**Table 4.2** (contd)

|  |  | MRQ | EQL | Prep Blank | Feed 06-00029 Sample | Feed 06-00029 Duplicate | Effluent 06-00030 | RPD (06-00029) | Serial Dilution (06-00029) | BS Recovery | 06-00026 MS(f) Recovery | 06-00029 Post Spike A | 06-00029 Post Spike B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Analyte** | **Method** | **µg/mL** | **µg/mL** | **µg/mL(a)** | **µg/mL(a)** | **µg/mL(a)** | **µg/mL(a)** | **%** | **%** | **%** | **%** | **%** | **%** |
| ***Criteria>*** |  |  |  |  |  |  |  | ***≤15(b)*** | ***≤10*** | ***80 - 120*** | ***75 - 125*** | ***75 - 125*** | ***75 - 125*** |
| Bi | ICP-AES | nmrq | 17 | <1.7 | [2.6] | [2.0] | [2.1] | -- | -- | 102 | 104 | 100 | na |
| Ca | ICP-AES | nmrq | 11 | <1.1 | 115 | 117 | 112 | 1.8 | 30 | 98 | 101 | 99 | na |
| Co | ICP-AES | nmrq | 3.1 | <0.31 | [1.9] | [2.0] | [1.9] | -- | -- | -- | -- | 102 | na |
| Cu | ICP-AES | nmrq | 8.1 | <0.81 | 10.6 | 10.7 | 9.85 | 0.8 | -- | 104 | 105 | 101 | na |
| Dy | ICP-AES | nmrq | 5.5 | <0.55 | <0.55 | <0.54 | <0.54 | -- | -- | -- | -- | na | 104 |
| Eu | ICP-AES | nmrq | 1.7 | <0.17 | [0.28] | [0.25] | [0.25] | -- | -- | -- | -- | na | 104 |
| Fe | ICP-AES | nmrq | 5.1 | [0.85] | [3.0] | [3.7] | [2.9] | -- | -- | 103 | 102 | 102 | na |
| La | ICP-AES | nmrq | 7.7 | <0.77 | [1.6] | [1.6] | [1.5] | -- | -- | 103 | 107 | na | 105 |
| Li | ICP-AES | nmrq | 5.7 | <0.57 | [0.97] | [1.1] | [0.97] | -- | -- | 101 | 101 | 101 | na |
| Mg | ICP-AES | nmrq | 14 | <1.4 | <1.4 | <1.4 | <1.4 | -- | -- | 100 | 100 | 103 | na |
| Mo | ICP-AES | nmrq | 3.3 | <0.33 | 22.3 | 22.9 | 22.6 | 2.8 | 11 | 90 | 98 | 104 | na |
| Nd | ICP-AES | nmrq | 36 | <3.6 | [7.6] | [7.0] | [7.1] | -- | -- | 104 | 108 | na | na |
| P | ICP-AES | nmrq | 20 | <2.0 | 752 | 771 | 758 | 2.5 | 3.0 | 88 | 91 | 99 | na |
| Pd | ICP-AES | nmrq | 96 | <9.6 | [14] | [13] | [13] | -- | -- | -- | -- | na | 96 |
| Rh | ICP-AES | nmrq | 44 | <4.4 | [7.9] | [7.4] | [7.3] | -- | -- | -- | -- | na | 105 |
| Ru | ICP-AES | nmrq | 11 | <1.1 | 12.5 | 12.8 | 12.6 | 1.9 | -- | -- | -- | na | 103 |
| Sb | ICP-AES | nmrq | 20 | <2.0 | [2.1] | <2.0 | [2.0] | -- | -- | -- | -- | 112 | na |
| Si | ICP-AES | nmrq | 29 | <2.9 | 79.2 | 80.4 | [17] | 1.5 | -- | 74 | 91 | 105 | na |
| Sn | ICP-AES | nmrq | 120 | <12 | [15] | [14] | [16] | -- | -- | -- | -- | 105 | na |
| Te | ICP-AES | nmrq | 25 | <2.5 | [5.0] | [5.0] | [5.1] | -- | -- | -- | -- | na | 104 |
| Ti | ICP-AES | nmrq | 1.7 | <0.17 | [0.20] | [0.20] | [0.17] | -- | -- | 88 | 97 | 100 | na |
| Tl | ICP-AES | nmrq | 16 | <1.6 | <1.6 | <1.53 | <1.52 | -- | -- | -- | -- | 99 | na |
| V | ICP-AES | nmrq | 3.3 | <0.33 | [0.55] | [0.51] | [0.51] | -- | -- | 103 | 104 | 102 | na |
| W | ICP-AES | nmrq | 11 | <1.1 | 60.9 | 62.2 | 61.2 | 2.2 | 10 | 96 | 101 | 105 | na |
| Y | ICP-AES | nmrq | 1.5 | <0.15 | [0.26] | [0.24] | [0.24] | -- | -- | -- | -- | 102 | na |
| Zn | ICP-AES | nmrq | 1.4 | 1.89 | 1.91 | 2.56 | 1.61 | 29.1 | -- | 102 | 103 | 104 | na |
| Zr | ICP-AES | nmrq | 4.2 | <0.42 | [1.2] | [1.2] | [1.1] | -- | -- | 96 | 110 | 101 | na |

**Table 4.2** (footnotes)

(a) The overall error for values without brackets was estimated to be within ±15% (analytes greater than the EQL). Bracketed values identify sample concentrations that were <EQL but >MDL, and errors likely exceeded 15%. The MDL was typically a factor of 10 lower than the EQL.
(b) The required RPD for Na was <10%.
(c) The MS sample was prepared from batch companion sample AP-101DF. Data are provided for information only relative to batch QC performance.
(d) A second serial dilution was required for Na analysis which was conducted companion sample AP-101DF.
(e) The Na concentration in the samples swamped the MS and post-spike concentrations.
(f) The second RPD was determined from the duplicate effluent composite analysis.
(g) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

BS = blank spike
EQL = estimated quantitation limit
MRQ = minimum reportable quantity
MS = matrix spike
na = not applicable
nm = not measured
nmrq = no minimum reportable quantity
nr = not reported
RPD = relative percent difference
"--" indicates calculation was not required
The bolded and shaded results (nm) indicate non-compliance with BNI acceptance criteria; see discussion.
Data are from ASR 7489.

## **Table 4.3.** Analysis QC Results for Eluate Composite Samples

| | | | | | **Lead-Column Eluate** | | | **Lag-Column Eluate** | | | **Serial** | **Post Spike** | **Post Spike** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Analyte** | **Method** | **MRQ** | **EQL** | **Diluent Blank** | **06-00031 Sample** | **06-00031 Duplicate** | **RPD** | **06-00032 Sample** | **06-00032 Duplicate** | **RPD** | **Dilution (06-00035)(a)** | **A (06- 00035)** | **B (06-00035)** |
| | | **μg/mL** | **μg/mL** | **μg/mL(b)** | **μg/mL(b)** | **μg/mL(b)** | **%** | **μg/mL(b)** | **μg/mL(b)** | **%** | **%** | **%** | **%** |
| ***Criteria>*** | | | | | | | ***≤15(c)*** | | | ***≤15(c)*** | ***≤10*** | ***75 - 125*** | ***75 - 125*** |
| *Target Analytes* | | | | | | | | | | | | | |
| Ag | ICPAES | 14 | 3.5 | <0.35 | <0.35 | <0.35 | -- | <0.35 | <0.35 | -- | -- | 98 | na |
| As | ICPAES | 72 | 14 | <1.4 | <1.4 | <1.4 | -- | <1.4 | <1.4 | -- | -- | 107 | na |
| Ba | ICPAES | 78 | 1.0 | [0.78] | [0.45] | [0.65] | -- | [0.34] | [0.45] | -- | 0.6 | 101 | na |
| Cd | ICPAES | 7.5 | 1.0 | <0.10 | [0.28] | [0.31] | -- | [0.19] | [0.15] | -- | -- | 102 | na |
| Cr | ICPAES | 15 | 1.4 | [0.23] | [0.71] | [0.69] | -- | [0.52] | [0.54] | -- | 2.2 | 104 | na |
| $^{133}$Cs | ICP-MS | 10 | 21 | [1.0E-4] | 34.6 | 34.6 | 0 | na | na | -- | -- | 97(d) | na |
| K | ICPAES | nmrq | 750 | <75 | <75 | <75 | -- | [82] | [82] | -- | 2.0 | **nm** | **nm** |
| Mn | ICPAES | nmrq | 0.4 | <0.040 | [0.086] | [0.085] | -- | [0.084] | [0.074] | -- | -- | 102 | na |
| Na | ICPAES | 180 | 93 | <9.3 | 932 | 923 | 0.9 | 1,090 | 1,080 | 1.0 | **nm** | **nm** | **nm** |
| Ni | ICPAES | 30 | 2.5 | <0.25 | 3.52 | 3.43 | 2.4 | 6.55 | 6.51 | 0.7 | -- | 104 | na |
| Pb | ICPAES | 300 | 15 | <1.5 | 38.8 | 38.4 | 1.1 | 33.8 | 33.5 | 0.8 | -- | 105 | na |
| $^{85}$Rb | ICP-MS | nmrq | 4.2 | [3.7E-4] | 0.586 | 0.587 | 0.1 | na | na | -- | -- | 104(d) | na |
| Se | ICPAES | 50 | 15 | [2.1] | <1.5 | <1.5 | -- | <1.5 | <1.5 | -- | -- | 103 | na |
| Sr | ICPAES | nmrq | 0.38 | [0.082] | 0.737 | 0.734 | 0.5 | 0.590 | 0.584 | 0.9 | -- | 101 | na |
| Th | ICPAES | 500 | 8.1 | <0.81 | <0.81 | <0.81 | -- | <0.81 | <0.81 | -- | -- | na | 103 |
| U | ICPAES | 1,000 | 450 | <45 | <45 | <45 | -- | <45 | <45 | -- | -- | na | 97 |
| U | KPA | 50 | | 1.52E-3 | 2.31 | 2.31 | 0 | 1.10 | 1.11 | 1 | na | na | na |
| Chloride | IC | 10 | 1.3 | [0.069] | [3.8] | [3.6] | -- | [2.6] | [2.0] | -- | na | na | na |
| Nitrite | IC | 3,000 | 1.9 | <0.048 | <2.4 | <2.4 | -- | <1.9 | <1.9 | -- | na | na | na |
| Nitrate | IC | 3,000 | 400 | <0.20 | 29,100 | 29,400 | 1 | 28,800 | 28,800 | 0 | na | na | na |
| Phosphate | IC | na | 10 | [0.51] | <10 | <10 | -- | <8.0 | <8.0 | -- | na | na | na |
| Sulfate | IC | 2,300 | 10 | <0.20 | [26] | [26] | -- | [24] | [24] | -- | na | na | na |
| *Opportunistic Analytes(e)* | | | | | | | | | | | | | |
| Al | ICP-AES | nmrq | 23 | <2.27 | [7.1] | [7.0] | -- | [3.9] | [3.6] | -- | 0.8 | 100 | na |
| B | ICP-AES | nmrq | 3.0 | <0.30 | 6.58 | 6.51 | 0.9 | [2.1] | [2.1] | -- | 1.5 | 103 | na |
| Be | ICP-AES | nmrq | 0.084 | <0.0084 | [0.015] | [0.015] | -- | <0.0084 | <0.0084 | -- | -- | 102 | na |
| Bi | ICP-AES | nmrq | 14 | <1.36 | <1.4 | <1.4 | -- | <1.4 | <1.4 | -- | -- | 101 | na |
| Ca | ICP-AES | nmrq | 8.4 | <0.84 | [1.9] | [1.7] | -- | [2.9] | [1.8] | -- | 69 | 100 | na |

## **Table 4.3** (contd)

| Analyte | Method | MRQ | EQL | Diluent Blank | Lead-Column Eluate | | | Lag-Column Eluate | | | Serial Dilution (06-00035)(a) | Post Spike A (06- 00035) | Post Spike B (06-00035) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | 06-00031 Sample | 06-00031 Duplicate | RPD | 06-00032 Sample | 06-00032 Duplicate | RPD | | | |
| | | µg/mL | µg/mL | µg/mL(b) | µg/mL(b) | µg/mL(b) | % | µg/mL(b) | µg/mL(b) | % | % | % | % |
| ***Criteria>*** | | | | | | | ***≤15(c)*** | | | ***≤15(c)*** | ***≤10*** | ***75 - 125*** | ***75 - 125*** |
| Co | ICP-AES | nmrq | 2.4 | <0.24 | <0.24 | <0.24 | -- | <0.24 | <0.24 | -- | -- | 103 | na |
| Cu | ICP-AES | nmrq | 6.4 | <0.64 | 8.72 | 8.63 | 1.0 | 9.12 | 8.98 | 1.5 | -- | 105 | na |
| Dy | ICP-AES | nmrq | 4.4 | <0.44 | <0.44 | <0.44 | -- | <0.44 | <0.44 | -- | -- | na | 101 |
| Eu | ICP-AES | nmrq | 1.4 | <0.14 | <0.14 | <0.14 | -- | <0.14 | <0.14 | -- | -- | na | 102 |
| Fe | ICP-AES | nmrq | 4.0 | <0.40 | [0.98] | [0.92] | -- | [0.57] | [0.65] | -- | -- | na | 103 |
| La | ICP-AES | nmrq | 6.1 | <0.61 | <0.61 | <0.61 | -- | <0.61 | <0.61 | -- | -- | na | 100 |
| Li | ICP-AES | nmrq | 4.5 | <0.45 | <0.45 | <0.45 | -- | <0.45 | <0.45 | -- | -- | 102 | na |
| Mg | ICP-AES | nmrq | 11 | <1.15 | <1.1 | <1.1 | -- | <1.1 | <1.1 | -- | -- | 105 | na |
| Mo | ICP-AES | nmrq | 2.6 | [.41] | [0.29] | [0.28] | -- | [0.31] | [0.30] | -- | -- | 106 | na |
| Nd | ICP-AES | nmrq | 28 | <2.82 | <2.8 | <2.8 | -- | <2.8 | <2.8 | -- | -- | na | na |
| P | ICP-AES | nmrq | 16 | <1.61 | [2.8] | [2.6] | -- | [1.8] | <1.6 | -- | -- | 103 | na |
| Pd | ICP-AES | nmrq | 76 | <7.61 | <7.6 | <7.6 | -- | <7.6 | <7.6 | -- | -- | na | 110 |
| Rh | ICP-AES | nmrq | 35 | <3.49 | <3.5 | <3.5 | -- | <3.5 | <3.5 | -- | -- | na | 100 |
| Ru | ICP-AES | nmrq | 9.0 | <0.90 | <0.90 | <0.90 | -- | <0.90 | <0.90 | -- | -- | na | 101 |
| Sb | ICP-AES | nmrq | 16 | [2.59] | [1.7] | [1.8] | -- | [1.9] | [1.8] | -- | -- | 106 | na |
| Si | ICP-AES | nmrq | 23 | <2.31 | [14] | [14] | -- | [9.2] | [9.0] | -- | 1.8 | 106 | na |
| Sn | ICP-AES | nmrq | 96 | <9.59 | <9.6 | <9.6 | -- | <9.6 | <9.6 | -- | -- | 100 | na |
| Te | ICP-AES | nmrq | 19 | <1.95 | <1.9 | <1.9 | -- | <1.9 | <1.9 | -- | -- | na | 105 |
| Ti | ICP-AES | nmrq | 1.3 | <0.13 | <0.13 | <0.13 | -- | <0.13 | <0.13 | -- | -- | 100 | na |
| Tl | ICP-AES | nmrq | 12 | <1.24 | <1.2 | <1.2 | -- | <1.2 | <1.2 | -- | -- | 100 | na |
| V | ICP-AES | nmrq | 2.6 | <0.26 | <0.26 | <0.26 | -- | <0.26 | <0.26 | -- | -- | 99 | na |
| W | ICP-AES | nmrq | 8.4 | <0.84 | <0.84 | <0.84 | -- | <0.84 | <0.84 | -- | -- | 102 | na |
| Y | ICP-AES | nmrq | 1.2 | <0.12 | <0.12 | <0.12 | -- | <0.12 | <0.12 | -- | -- | 100 | na |
| Zn | ICP-AES | nmrq | 1.1 | 1.67 | 5.61 | 5.82 | 3.7 | 5.49 | 5.41 | 1.4 | -- | 106 | na |
| Zr | ICP-AES | nmrq | 3.3 | <0.33 | <0.33 | <0.33 | | <0.33 | <0.33 | -- | -- | 102 | na |

**Table 4.3** (footnotes)

(a) The simulant eluate generated from A-225 testing was included in the analytical batch. It was used as the source for batch QC: serial dilution and post spiking. Data are provided for information only relative to batch QC performance for the serial dilution and post spike recoveries.
(b) The overall error for values without brackets was estimated to be within ±15% (analytes greater than the EQL). Bracketed values identify sample concentrations that were <EQL but >MDL, and errors likely exceeded 15%. The MDL was typically a factor of 10 lower than the EQL.
(c) The Na RPD was to be ≤ 10%.
(d) The ICP-MS post-spike was conducted on sample 06-00031.
(e) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.
BS = blank spike
EQL = estimated quantitation limit
MRQ = minimum reportable quantity
MS = matrix spike
na = not applicable
nm = not measured
RPD = relative percent difference
"--" indicates calculation was not required
The bolded and shaded results (nm) indicate non-compliance with BNI acceptance criteria; see discussion.
Data are from ASR 7489.

## **Table 4.4.** Radiochemical Analysis QC Results for Feed and Effluent Samples

| | | | | AN-102 Feed, 06-00029 | | | AN-102 Effluent, 06-00030 | | | | 06-00026 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Sample>* | **MRQ** | **Prep Blank** | **Sample** | **Duplicate** | **RPD** | **Sample** | **Duplicate** | **RPD** | **BS Recovery** | **MS Recovery** |
| | *Units>*<br>*Uncertainty>* | **µCi/mL** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **%** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **%** | **%** | **%** |
| | *Criteria>* | | | | | ***≤15*** | | | ***≤15*** | ***80 - 120*** | ***75 - 125*** |
| **Analyte** | **Method** | | | | | | | | | | |
| $^{137}$Cs | GEA | 5.0E-2 | <9E-5<br>-- | 1.48E+2<br>4% | 1.48E+2<br>4% | 0 | 1.33E-3<br>7% | na | na | na | na |
| $^{238}$Pu | Radchem | 1.0E-2 | 1.42E-5<br>10% | 1.99E-4<br>9% | 4.64E-4$^{(a)}$<br>5% | **80** | 1.68E-4<br>8% | 1.79E-4<br>9% | 6 | na | na |
| $^{239+240}$Pu | Radchem | 1.0E-2 | 5.12E-6<br>17% | 5.14E-4<br>6% | 6.08E-4<br>4% | **17** | 4.61E-4<br>5% | 5.30E-4<br>5% | 14 | 99 | 81 |
| $^{241}$Am | Radchem | 1.0E-2 | 1.55E-5<br>6% | 1.04E-2<br>2% | 1.11E-2<br>2% | 7 | 1.03E-2<br>2% | 1.01E-2<br>2% | 2 | 107 | 103 |
| $^{242}$Cm | Radchem | 1.0E-2 | <3E-7<br>-- | 4.23E-05<br>16% | 3.76E-5<br>18% | 12 | 4.00E-5<br>16% | 3.64E-5<br>17% | 9 | na | na |
| $^{243+244}$Cm | Radchem | 1.0E-2 | 5.36E-6<br>10% | 4.45E-04<br>5% | 5.73E-4<br>5% | **25** | 4.56E-4<br>5% | 4.20E-4<br>5% | 8 | na | na |
| Total alpha | Radchem | 1.0E-2 | <5E-4<br>-- | 7.05E-3<br>6% | 9.67E-3<br>5% | **31** | 9.10E-3<br>5% | 7.95E-3<br>5% | 13 | 97 | 81 |
| Sum of alpha | Radchem | nmrq | 3.91E-5<br>5% | 1.16E-2<br>2% | 1.28E-2<br>2% | 10 | 1.14E-2<br>2% | 1.13E-2<br>2% | 1 | na | na |
| *Opportunistic Analyte*$^{(b)}$ | | | | | | | | | | | |
| $^{60}$Co | GEA | nmrq | <8E-5<br>-- | 1.85E-2<br>10% | 1.63E-2<br>10% | 13 | 2.06E-2<br>2% | na | na | na | na |

(a) The duplicate sample may have been contaminated from incomplete $^{241}$Am separation.
(b) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.
BS = blank spike
MRQ = minimum reportable quantity
MS = matrix spike
na = not applicable or not required
nmrq = no minimum reportable quantity
RPD = relative percent difference
The bolded and shaded results indicate non-compliance with BNI acceptance criteria; see discussion.
ASR 7489, reference date is August 15, 2005.

**Table 4.5.** Radiochemical Analysis QC Results for Eluate Samples

| | | | **Diluent** | **Lead-Column Eluate, 06-00031** | | | **Lag-Column Eluate, 06-00032** | | | **BS** | **MS** |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Sample>* | **MRQ** | **Blank** | **Sample** | **Duplicate** | **RPD** | **Sample** | **Duplicate** | **RPD** | **Recovery** | **Recovery** |
| | *Units>*<br>*Uncertainty>* | **µCi/mL** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **%** | **µCi/mL**<br>**± 1σ** | **µCi/mL**<br>**± 1σ** | **%** | **%** | **%** |
| | *Criteria>* | | | | | ***≤15*** | | | ***≤15*** | ***80 - 120*** | ***75 - 125*** |
| **Analyte** | **Method** | | | | | | | | | | |
| $^{137}$Cs | GEA | 5.0E-2 | <9E-5 | 1.11E+3<br>4% | 1.10E+3<br>4% | 1 | 2.48E+1<br>3% | 2.46E+1<br>3% | 1 | na | na |
| $^{238}$Pu | Radchem | 1.0E-2 | <4E-8 | 2.48E-5<br>6% | 7.39E-6[(a)]<br>12% | **108** | 9.68E-6<br>8% | 1.40E-5<br>9% | **36** | na | na |
| $^{239+240}$Pu | Radchem | 1.0E-2 | 6.51E-8<br>31% | 3.14E-5<br>5% | 3.25E-5<br>6% | 3 | 1.88E-5<br>6% | 1.99E-5<br>8% | 6 | 99 | 81 |
| $^{241}$Am | Radchem | 1.0E-2 | <4E-8 | rejected data(b) | 6.77E-5<br>4% | nm | 4.06E-5<br>5% | 4.43E-5<br>4% | 9 | 107 | 103 |
| $^{242}$Cm | Radchem | 1.0E-2 | <2E-8 | rejected data(b) | <5E-7<br>-- | nm | <4E-7<br>-- | <3E-7<br>-- | na | na | na |
| $^{243+244}$Cm | Radchem | 1.0E-2 | <2E-8 | rejected data(b) | 3.96E-6<br>15% | nm | 4.59E-6<br>13% | 1.54E-5<br>6% | **108** | na | na |
| Total alpha | Radchem | 1.0E-2 | <2E-5 | <4E-4 | <4E-4 | na | <4E-4 | <4E-4 | na | 97 | 81 |
| Sum of alpha | Radchem | nmrq | 1.61E-7<br>13% | rejected data(b) | 1.12E-4<br>3% | nm | 7.37E-5<br>3% | 9.36E-5<br>3% | **24** | na | na |

(a) The Pu isotopic ratio was unlikely; the reported $^{238}$Pu concentration was suspected to be biased low. Refer to the discussion in Section 4.2.6.

(b) The Am/Cm alpha energy resolution was too poor to quantitate the alpha peaks; the sample data were rejected. Process evaluations were conducted relative to results from the duplicate sample. Refer to the discussion in Section 4.2.6.

BS = blank spike
MRQ = minimum reportable quantity
MS = matrix spike
na = not applicable
nm = not measured
nmrq = no minimum reportable quantity
RPD = relative percent difference
The bolded and shaded results indicate non-compliance with BNI acceptance criteria; see discussion.
ASR 7489, reference date is August 15, 2005.

# 5.0 Conclusions

The AN-102 Hanford tank waste was successfully processed using spherical RF resin in a lead-lag column format under nominal baseline plant processing conditions following Sr/TRU removal and appropriate dilution. The Cs breakthrough occurred slowly on the lead column; a distinct breakthrough profile was not obtained because not enough feed was available.

The following Cs ion exchange results for the actual waste AN-102 were obtained:

- The total process volume before 50% breakthrough was >200 BVs.
- An overall DF of 1.11E+5 was demonstrated for the 202-BVs processed.
- The lead column was partially loaded with AP-101DF Cs from previous testing; the lead-column effluent was immediately greater than the contract limit %$C/C_o$.
- The lead-column 50% Cs breakthrough could not be determined; column modeling programs may be useful in discerning the extrapolated 50% breakthrough.
- The Cs was largely (>99.9%) eluted from the lead and lag columns in the first 9 BVs processed. Tailing was observed in the Cs elution profile after this point.
- Virtually 100% of the calculated Cs loaded on the lead-column ion exchanger was accounted for in the composite eluate.
- An activity balance for $^{137}$Cs indicated that 99.6% of the $^{137}$Cs present in the feed sample was accounted for in the samples and process streams (mostly in the eluate), which is indicative of good experimental integrity.
- The eluted column following AP-101DF processing worked well in the lag position to provide an AN-102 process effluent DF two orders of magnitude below the contract limit DF.

The fates of other metals were evaluated based on feed, effluent, and eluate analysis.

- Neither U nor actinides appeared to exchange onto the RF resin.
- Insignificant quantities of Cr and other metals, aside from Na and Cs, were observed in the eluate. The Ba and Zn exchange behaviors were inconclusive.
- About 10% of the Pb fed through the system was recovered in each of the lead- and lag-column eluates.(a)
- Small amounts of Pu were recovered in the eluates (lead column ~0.9%; lag column ~0.5%).

---

(a) Lead (and several other RCRA metals) will be analyzed in the spent resin and reported in a later report.

# Appendix A

# Calculations

# Appendix A: Calculations

## Cesium-137 Contractual Limit in AN-102 Envelope C Vitrification Feed

Assumptions

1) Concentration of $Na_2O$ in Envelope A glass = 12% (=12 g $Na_2O$/100 g glass)

2) For maximum $^{137}$Cs concentration in glass, assume that all Na comes from the feed. If some Na is added to the vitrification feed, multiply the maximum $^{137}$Cs value determined below by the ratio of the total Na:feed Na.

3) Glass density = 2.71 MT/$m^3$ (=2.71 g/mL)

4) Maximum $^{137}$Cs in glass = 0.3 Ci/$m^3$ (= 0.3 Ci/1E+6 mL = 3E-7 Ci/mL)

5) AN-102 ion exchange feed Na concentration = 4.94 M

6) AN-102 ion exchange feed $^{137}$Cs concentration = 143 μCi/mL (8/15/05)

Na Loading in Glass

12 g $Na_2O$/100g glass * 1 mole $Na_2O$/62 g $Na_2O$) * (2 mole Na/mole $Na_2O$)*
(23 g Na/mole Na) * (2.71 g glass/mL glass) = 0.241 g Na/mL glass

Maximum $^{137}$Cs:Na in glass

(3.0E-7 Ci $^{137}$Cs/mL glass)/(0.241 g Na/mL glass) = 1.24 E-6 Ci $^{137}$Cs/g Na

(1.24E-6 Ci $^{137}$Cs/g Na) * (23 g Na/mole) = 2.86E-5 Ci $^{137}$Cs/mole Na

Maximum $^{137}$Cs:Na in LAW vitrification feed

(2.86E-5 Ci $^{137}$Cs/mole Na) * (4.94 mole Na/L feed) = 1.41 E-4 Ci $^{137}$Cs/L
= 141 μCi $^{137}$Cs/L
= 0.141 μCi $^{137}$Cs/mL

AN-102 actual waste Cs fraction remaining ($C/C_o$) Contractual Limit

(0.141 μCi $^{137}$Cs/ mL)/(143 μCi $^{137}$Cs/mL) = 9.88E-4 $C/C_o$
= 0.0988 % $C/C_o$

Decontamination Factor Contract Limit

1/(9.88E-4 $C/C_o$) = 1012 $C_o/C$

## Cesium-137 Contractual Limit in AN-102 Envelope C Vitrification Feed (2015 Start Date)

Assumptions

1) Concentration of $Na_2O$ in Envelope A glass = 12% (=12 g $Na_2O$/100 g glass)

2) For maximum $^{137}$Cs concentration in glass, assume that all Na comes from the feed. If some Na is added to the vitrification feed, multiply the maximum $^{137}$Cs value determined below by the ratio of the total Na:feed Na.

3) Glass density = 2.71 MT/m$^3$ (=2.71 g/mL)

4) Maximum $^{137}$Cs in glass = 0.3 Ci/m$^3$ (= 0.3 Ci/1E+6 mL = 3E-7 Ci/mL)

5) AN-102 ion exchange feed Na concentration = 4.94 M

6) AN-102 ion exchange feed $^{137}$Cs concentration = 114 μCi/mL (8/15/15)

Na Loading in Glass

12 g $Na_2O$/100g glass * 1 mole $Na_2O$/62 g $Na_2O$) * (2 mole Na/mole $Na_2O$)*
(23 g Na/mole Na) * (2.71 g glass/mL glass) = 0.241 g Na/mL glass

Maximum $^{137}$Cs:Na in glass

(3.0E-7 Ci $^{137}$Cs/mL glass)/(0.241 g Na/mL glass) = 1.24 E-6 Ci $^{137}$Cs/g Na

(1.24E-6 Ci $^{137}$Cs/g Na) * (23 g Na/mole) = 2.86E-5 Ci $^{137}$Cs/mole Na

Maximum $^{137}$Cs:Na in LAW vitrification feed

(2.86E-5 Ci $^{137}$Cs/mole Na) * (4.94 mole Na/L feed) = 1.41 E-4 Ci $^{137}$Cs/L
= 141 μCi $^{137}$Cs/L
= 0.141 μCi $^{137}$Cs/mL

AN-102 actual waste Cs fraction remaining ($C/C_o$) Contractual Limit

(0.141 μCi $^{137}$Cs/ mL)/(114 μCi $^{137}$Cs/mL) = 1.24E-3 $C/C_o$
= 0.124 % $C/C_o$

Decontamination Factor Contract Limit

1/(9.88E-4 $C/C_o$) = 809 $C_o/C$

# Appendix B

# Ion Exchange Processing

# Appendix B: Ion Exchange Processing

**Table B.1.** Lead- and Lag-Column Effluent Cs Concentrations During Feed Processing, Feed Displacement, and Water Rinse

| **Lead Column** | | | **Lag-Column** | | |
|---|---|---|---|---|---|
| **BV processed** | **Effluent (C), μCi/mL** | **% $C/C_o$** | **BV processed** | **Effluent (C), μCi/mL** | **% $C/C_o$** |
| *Feed Processing* | | | | | |
| 5.1 | 7.51E-1 | 0.53 | 5.1 | 4.13E-3 | 2.89E-3 |
| 12.8 | 8.35E-1 | 0.58 | 12.6 | 1.87E-3 | 1.31E-3 |
| 21.4 | 9.45E-1 | 0.66 | 21.0 | 1.23E-3 | 8.58E-4 |
| 30.5 | 1.01E+0 | 0.71 | 29.9 | 1.39E-3 | 9.72E-4 |
| 39.4 | 1.11E+0 | 0.78 | 38.6 | 1.10E-3 | 7.67E-4 |
| 48.3 | 1.17E+0 | 0.82 | 47.4 | 8.94E-4 | 6.26E-4 |
| 57.4 | 1.25E+0 | 0.87 | 56.3 | 2.26E-3 | 1.58E-3 |
| 60.0 | 1.29E+0 | 0.91 | 58.6 | 1.91E-3 | 1.34E-3 |
| 69.2 | 1.40E+0 | 0.98 | 67.6 | 1.23E-3 | 8.62E-4 |
| 78.1 | 1.58E+0 | 1.10 | 76.3 | 8.95E-4 | 6.27E-4 |
| 87.1 | 1.71E+0 | 1.20 | 85.1 | 8.94E-4 | 6.26E-4 |
| 96.0 | 1.83E+0 | 1.28 | 93.8 | 1.17E-3 | 8.21E-4 |
| 104.6 | 2.00E+0 | 1.40 | 102.2 | 8.81E-4 | 6.16E-4 |
| 113.4 | 2.13E+0 | 1.49 | 110.8 | 9.65E-4 | 6.75E-4 |
| 122.3 | 2.49E+0 | 1.75 | 119.4 | 1.55E-3 | 1.08E-3 |
| 132.0 | 2.68E+0 | 1.87 | 128.9 | 1.38E-3 | 9.63E-4 |
| 140.5 | 3.16E+0 | 2.21 | 137.3 | 1.09E-3 | 7.64E-4 |
| 149.1 | 3.76E+0 | 2.63 | 145.6 | 9.78E-4 | 6.84E-4 |
| 158.2 | 4.72E+0 | 3.30 | 154.5 | 1.53E-3 | 1.07E-3 |
| 166.6 | 5.71E+0 | 4.00 | 162.7 | 1.08E-3 | 7.53E-4 |
| 175.1 | 7.02E+0 | 4.91 | 171.0 | 1.13E-3 | 7.88E-4 |
| 184.0 | 8.80E+0 | 6.16 | 179.6 | 1.41E-3 | 9.88E-4 |
| 192.8 | 1.13E+1 | 7.88 | 188.3 | 1.38E-3 | 9.69E-4 |
| 201.9 | 1.45E+1 | 10.17 | 197.1 | 1.28E-3 | 8.95E-4 |
| – | – | – | Comp. 0 - 60 | 2.13E-3 | 1.49E-3 |
| – | – | – | Comp. 61 - 141 | 8.82E-4 | 6.17E-4 |
| – | – | – | Comp. 142 - 202 | 1.44E-3 | 1.01E-3 |
| *Feed Displacement* | | | | | |
| – | – | – | 198.7(a) | 1.68E-3 | 1.17E-3 |
| – | – | – | 200.5 | 1.50E-3 | 1.05E-3 |
| – | – | – | 201.6 | 1.16E-3 | 8.09E-4 |
| – | – | – | 203.3 | 4.54E-4 | 3.18E-4 |
| – | – | – | 205.0 | 3.28E-4 | 2.29E-4 |
| – | – | – | 206.7 | 3.52E-4 | 2.46E-4 |
| *Water Rinse* | | | | | |
| – | – | – | 208.4(a) | 1.02E-4 | 7.16E-5 |
| – | – | – | 209.9 | 1.67E-4 | 1.17E-4 |
| – | – | – | 211.6 | 1.10E-4 | 7.69E-5 |
| – | – | – | 213.2 | 1.64E-4 | 1.14E-4 |
| – | – | – | 214.9 | 1.33E-4 | 9.34E-5 |
| – | – | – | 215.8 | 3.17E-4 | 2.22E-4 |
| (a) BV is a continuation of BV recorded from previous steps. | | | | | |

**Table B.2.** Lead- and Lag-Column Elution and Water Rinse Cs Concentrations

| Lead Column Eluate and Rinse | | | | Lag Column Eluate and Rinse | | | |
|---|---|---|---|---|---|---|---|
| **BV Processed** | **$^{137}$Cs (C), µCi/mL** | **$C/C_o$** | **ASO Confirmation $^{137}$Cs, µCi/mL** | **BV Processed** | **$^{137}$Cs (C), µCi/mL** | **$C/C_o$** | **ASO Confirmation $^{137}$Cs, µCi/mL** |
| *Elution* | | | | | | | |
| 1.31 | 1.23E-1 | 8.61E-4 | – | 1.64 | 7.46E-4 | 5.22E-6 | – |
| 2.69 | 4.81E-1 | 3.37E-3 | 8.44E-1 | 3.10 | 1.32E-3 | 9.24E-6 | – |
| 4.10 | 7.56E+3 | 5.29E+1 | 7.97E+3 | 4.52 | 5.31E+1 | 3.71E-1 | 4.96E+1 |
| 5.51 | 1.71E+4 | 1.20E+2 | 1.54E+4 | 5.89 | 4.77E+2 | 3.34E+0 | 4.86E+2 |
| 6.92 | 9.07E+1 | 6.35E-1 | 8.72E+1 | 7.26 | 2.44E+0 | 1.70E-2 | – |
| 8.32 | 9.94E+0 | 6.96E-2 | 1.10E+1 | 8.66 | 2.88E-1 | 2.02E-3 | – |
| 9.72 | 3.42E+0 | 2.39E-2 | – | 9.99 | 1.25E-1 | 8.74E-4 | – |
| 11.14 | 1.83E+0 | 1.28E-2 | – | 22.33 | 3.80E-2 | 2.66E-4 | – |
| 12.53 | 8.96E-1 | 6.27E-3 | – | 23.71 | 1.57E-2 | 1.10E-4 | – |
| 13.94 | 7.00E-1 | 4.90E-3 | – | 25.17 | 3.33E-2 | 2.33E-4 | – |
| 15.39 | 5.64E-1 | 3.95E-3 | – | 26.54 | 7.88E-2 | 5.52E-4 | – |
| 16.76 | 4.58E-1 | 3.20E-3 | 4.64E-1 | 28.00 | 2.90E-2 | 2.03E-4 | – |
| 18.18 | 3.97E-1 | 2.78E-3 | – | 29.39 | 1.31E-2 | 9.14E-5 | – |
| 19.62 | 3.71E-1 | 2.59E-3 | – | No data. | | | |
| 21.04 | 2.93E-1 | 2.05E-3 | – | | | | |
| 23.15 | 4.94E-1 | 3.45E-3 | 4.95E-1 | | | | |
| 24.56 | 2.80E-1 | 1.96E-3 | – | | | | |
| 26.00 | 1.94E-1 | 1.36E-3 | – | | | | |
| 27.55 | 1.60E-1 | 1.12E-3 | – | | | | |
| 28.99 | 1.45E-1 | 1.01E-3 | – | | | | |
| 30.44 | 1.86E-1 | 1.30E-3 | 2.04E-1 | | | | |
| *Water Rinse* | | | | | | | |
| 31.5(a) | 1.46E-1 | 1.02E-3 | – | 30.4(a) | 1.05E-2 | 7.36E-5 | – |
| 32.5 | 1.14E-1 | 7.97E-4 | – | 31.4 | 9.95E-3 | 6.96E-5 | – |
| 33.5 | 9.63E-2 | 6.74E-4 | – | 32.4 | 9.73E-3 | 6.81E-5 | – |
| (a) BV is a continuation from elution BV sum. | | | | | | | |

# Appendix C

# Cesium Ion Exchange Testing Personnel

# Appendix C: Cesium Ion Exchange Testing Personnel

**Cognizant Scientists**
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D. L. Blanchard

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T. L. Trang-Le

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