PNWD-3267
WTP-RPT-041, Rev 0

Small Column Ion Exchange Testing of SuperLig® 644 for Removal of ^{137}Cs from Hanford Waste Tank 241-AZ-102 Concentrate (Envelope B)

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April 2003

WTP
Project
Report

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Test specification: 24590-PTF-TSP-RT-01-002, Rev. 1
Test plan: TP-RPP-WTP-111, Rev. 0
Test exceptions: None
R&T focus area: Pretreatment
Test Scoping Statement(s): B-44

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-01-002, Rev. 1 and Test Plan TP-RPP-WTP-111, Rev. 0. 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:

_____ _____

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

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G. Todd Wright, Manager Date
Research and Technology

Summary

The U. S. Department of Energy is tasked with the disposition of high-level radioactive waste stored at the Hanford site. The waste is to be vitrified following specific pretreatment processing, separating the waste into a small-volume high-level waste fraction and a large-volume low-activity waste fraction. The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for ^{137}Cs removal from Hanford high-level tank waste is ion exchange. The current pretreatment flowsheet includes the use of Cs-selective, elutable, organic ion exchanger SuperLig® 644 (SL-644) material for Cs removal from the aqueous waste fraction. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. SL-644 has been shown to be effective in removing Cs from a variety of Hanford tank wastes.

Battelle—Pacific Northwest Division (PNWD) was contracted to perform Cs ion exchange studies under Contract 24590-101-TSA-W000-0004. The Cs ion exchange activities are further defined in Appendix C of the *Research and Technology Plan*[(a)] under Technical Scoping Statement B-44. These studies are to verify design and operating parameters for plant-scale ion exchange systems. Test results will also be used to validate ion exchange models.

Objectives

Previous testing conducted at the Savannah River Technology Center with Hanford Tank 241-AZ-102 (AZ-102) waste resulted in poor ion exchange performance. The poor load performance was attributed to the possibility of *in situ* precipitation of metal hydroxides on the ion exchanger and/or the low ionic strength of the supernatant combined with the high Cs concentration. Because the low ionic strength (Na molarity equal to 2.77) of the AZ-102 supernatant was suspected to contribute to the poor Cs ion exchange performance, the AZ-102 was concentrated to nominally 5 M Na for performance testing.

The Cs ion exchange test objectives were to develop load and elution breakthrough profiles using 241-AZ-102 concentrated to 4.6 M Na (AZ-102C); produce and characterize the Cs eluate; remove ^{137}Cs from the AZ-102C to meet low-activity waste (LAW) vitrification criteria; and develop batch-distribution coefficients for AZ-102C. The final effluent was to contain <0.322 μCi ^{137}Cs/mL, based on a 5-wt% waste Na_2O loading in the waste glass.[(b)] All testing objectives were met.

Conduct of Test

This report summarizes testing of the SL-644 in batch-contact studies and in a dual small-column system. The test matrix was Hanford tank waste 241-AZ-102 (Envelope B) concentrated by evaporation to 4.6 M Na, 1.246 g/mL (26°C), and 2.00E+3 μCi ^{137}Cs/mL. Batch contacts were performed with the waste at three Cs concentrations at a phase ratio of 100 (liquid volume to exchanger mass) with SL-644. The ion exchange processing system was composed of a lead and lag column with resin bed volumes

(a) S. Barnes, R. Roosa, and R. Peterson. 2002. *Research and Technology Plan*, 24590-WTP-PL-RT-01-002, Rev. 1.

(b) The minimum waste Na_2O loading is 5 wt% for Envelope B tank waste; the maximum waste Na_2O loading is 5.5 wt%, corresponding to 0.292 μCi/mL ^{137}Cs.

(BV) of 10.2 mL (L/D[a] = 4.2) during the conditioning phase with 0.25 M NaOH, and 9.5 mL (L/D = 3.9) during the AZ-102C loading phase. Proper functioning of the ion exchange apparatus and resin beds had initially been tested with an AW-101 simulant. The resin beds had then been used to process 1.2 L of AP-101 diluted feed (an Envelope A waste feed) and 0.75 L of AN-102 (an Envelope C waste feed) combined with wash and leachate solutions of C-104 solids. The AZ-102C waste volume processed was 1.07 L, corresponding to 105 BVs. All ion exchange process steps were tested, including resin-bed preparation, loading, feed displacement, water rinse, elution, eluant rinse, and resin regeneration.

Results and Performance Against Objectives

The batch-contact performance data are summarized in Table S.1. The Cs λ value (column distribution ratio) represents a measure of the effective capacity of the SL-644 resin—the higher the λ value, the higher loading capacity. The batch-contact tests resulted in a predicted Cs distribution coefficient (K_d) of 160 mL/g in the feed condition (Na/Cs mole ratio of 8.8 E+3). Using a bed density of 0.241 g/mL in the 0.25 M NaOH regeneration condition, the Cs λ value is predicted to be 38 BVs.

Table S.1. Summary of Performance Measures

Flow rate, BV/h	Interpolated Cs 50% Breakthrough, BV		Composite DF[2]	Maximum DF[3]	K_d, mL/g (feed condition)	Predicted Cs λ, BV
	Lead Column	Lag Column				
1.37	93	NM[1]	1.04 E+5	3.7 E+5	160	38

(1) NM = not measured; the AZ-102C feed did not break through the lag column.
(2) The DF was calculated by dividing the feed Cs concentration by the composite effluent Cs concentration, based on the total of 105 BVs of feed.
(3) The maximum DF was obtained by dividing the feed Cs concentration by the lowest sample Cs concentration (in this case, this was from the lead column; the lag column samples were slightly higher in Cs concentration than lead column samples).

The ion exchange column performance of SL-644 with AZ-102 waste feed was much better than previously observed in a similar test of SL-644 with AZ-102 at the Savannah River Technology Center.[b] The improved performance may be a result of the concentration of AZ-102 (during which solids were observed to precipitate), differences in the resin batches, storage/process histories, or a combination of these factors. The 50% Cs breakthrough from column testing was measured for the lead column at 93 BVs. No breakthrough was observed from the lag column. The decontamination factor (DF) for ^{137}Cs was based on the ^{137}Cs concentration in the feed relative to the ^{137}Cs concentration in the composite effluent sample. The composite DF for ^{137}Cs was 1.04 E+5. The maximum DF, 3.7 E+5, measured the best performance that could be expected from this column system. It was calculated relative to the sample containing the lowest ^{137}Cs concentration, i.e., the third sample from the lead column taken after loading 14.9 BVs. The effluent ^{137}Cs concentration was 1.92 E-2 μCi/mL. The LAW vitrified waste

(a) L/D, equal to length over diameter, is the resin-bed aspect ratio.
(b) Hassan NM, WD King, DJ McCabe, and ML Crowder. 2001. *Small-Scale Ion Exchange Removal of Cesium and Technetium from Envelope B Hanford Tank 241-AZ-102*, WSRC-TR-2000-00419, SRT-RPP-2000-00036, Savannah River Technology Center, Westinghouse Savannah River Co. Aiken, SC.

form must be no greater than 0.3 Ci/m^3; this limit can be converted to a ^{137}Cs maximum concentration of 0.32 μCi ^{137}Cs/mL in the ion exchange effluent.[(a)] The composite effluent ^{137}Cs concentration was below the contract limit.

The lead column was eluted with 0.5 M HNO_3 to C/C_o of 1% in 11 BVs with >94% of the ^{137}Cs contained in 4 BVs of eluant. The peak ^{137}Cs C/C_o value was 71 (based on 1-BV collection increments of nominally 10-mL). The ^{137}Cs concentration in the composite eluate was 1.45 E+4 μCi/mL, corresponding to a C/C_o of 7.23.

QA Requirements

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. This work was conducted to the quality requirements of NQA-1-1989 and NQA-2a-1990, Part 2.7 as instituted through PNWD's *Waste Treatment Plant Support Project Quality Assurance Requirements and Description* (WTPSP) Manual, and to the approved Test Plan, TP-RPP-WTP-111.

PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with 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.

Issues

The composite regeneration effluent solution was slightly acidic. As such, it would be inadequate for recycle as a feed-displacement solution.

(a) The conversion requires the following assumptions: Envelope B LAW will contain 5 wt% Na_2O, all Na comes from the tank waste, the glass density is 2.66 g/mL, and the waste Na concentration is 4.6 M. For maximum waste loading, 5.5 wt% Na_2O, the maximum ^{137}Cs concentration is 0.29 μCi/mL.

Terms and Abbreviations

AP-101DF	AP-101 tank waste diluted to 5 M Na
AN-102/C-104	AN-102 tank waste mixed with wash and leachate solutions from C-104 solids
AZ-102C	AZ-102 tank waste supernate concentrated to 4.6 M Na
ASR	analytical service request
AV	apparatus volume
BV	bed volume
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed
CMC	chemical measurement center
DF	decontamination factor
DI	deionized
F-factor	mass of dry ion exchanger divided by mass of wet exchanger
FMI	Fluid Metering, Inc., Syosset, NY
GEA	gamma energy analysis
HP	hot persulfate
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
IDL	instrument detection limit
λ	column distribution ratio
LAW	low-activity waste
L/D	length over diameter ratio
M	molarity, moles/liter
meq	milli-equivalents
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
NPT	national pipe thread
PNWD	Battelle—Pacific Northwest Division

PSD	particle size distribution
ρ	dry bed density
RPL	Radiochemical Processing Laboratory
RPP-WTP	River Protection Project-Waste Treatment Plant
SRTC	Savannah River Technology Center
TC	total carbon
TIC	total inorganic carbon
TIMS	thermal ionization mass spectrometry
TOC	total organic carbon

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1.0 Introduction

The U. S. Department of Energy plans to vitrify tank wastes at the Hanford Site in preparation for permanent disposal. Before vitrification, tank wastes will be divided into low-activity and high-level fractions through specific pretreatment processes. The pretreatment flowsheet for the Hanford tank wastes includes the use of SuperLig® 644 (SL-644) material for ^{137}Cs removal from the aqueous waste fraction. The SL-644 is a Cs-selective, organic ion exchanger and has been shown to be effective in removing Cs from a variety of Hanford tank wastes (Hassan, McCabe, and King 2000; Hassan et al. 2000; Hassan et al. 2001; King, Hassan, and McCabe 2001; Kurath, Blanchard, and Bontha 2000a; Kurath, Blanchard, and Bontha 2000b; Fiskum, Blanchard, and Arm 2002a and b; Fiskum et al. 2002a). The SL-644 has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

Previous testing conducted at Savannah River Technology Center (SRTC) with Hanford tank 241-AZ-102 (AZ-102) waste resulted in poor ion exchange performance. The poor load performance was attributed to the possibility of *in situ* precipitation of metal hydroxides on the ion exchanger and/or the low ionic strength of the supernatant combined with the high Cs concentration. Because the low ionic strength (Na molarity equal to 2.77) of the AZ-102 supernatant was suspected to contribute to the poor Cs ion exchange performance, the AZ-102 was concentrated to nominally 5 M Na for performance testing.

This report summarizes batch-contact studies of SL-644 and dual small-column testing of the SL-644 ion exchange material. The test matrix for the small-column ion exchange and SL-644 batch contact was AZ-102 Hanford tank waste, concentrated to 4.6 M Na by evaporation (hereafter referred to as AZ-102C). Approximately 1.07 L of AZ-102C was processed through the ion exchange column system. The ion exchange process steps tested include resin-bed preparation, loading, feed displacement, water rinse, elution, and resin regeneration.

The objectives of this work were to:

- develop distribution coefficient (K_d) values as a function of Na/Cs molar concentration for SL-644 in the AZ-102C matrix
- demonstrate the ^{137}Cs decontamination of Envelope B tank waste sample AZ-102C and provide a Cs-decontaminated sample for downstream process testing (i.e., batch-contact studies for ^{99}Tc removal and ^{99}Tc measurements using a prototype Tc monitor)
- develop Cs load and elution profiles
- demonstrate the effectiveness of all SL-644 ion exchange process steps, including loading, feed displacement, deionized water (DI) washing, elution, and resin regeneration.[(a)]

(a) Test Specification: "Tank 241-AZ-101 and 241-AZ-102 Ion Exchange Test Specification," 24590-PTF-TSP-RT-01-002, Rev. 1, James Toth, Bechtel National Inc., October, 2001 and Test Plan "Tank 241-AZ-101 and 241-AZ-102 Waste Sample Ion Exchange Testing," TP-RPP-WTP-111, Rev. 0, D. L. Blanchard, November 5, 2001.

2.0 Experimental

This section describes materials, experimental approach to batch-contact tests and column tests, sample analyses, and calculations. Experimental data were recorded in specific test instructions (as identified) and analytical reports. All raw data are maintained in the Project File 42365.

2.1 SL-644 Resin

The SL-644 was obtained from IBC production batch number 010319SMC-IV-73 prepared at IBC on 3/19/01. This material batch exhibited a black-red appearance peppered with light-brown specks. It was received from the vendor as a dry, granular, free-flowing material in a 1-L polyethylene bottle with an approximately 32% gaseous headspace. There was no indication that this headspace was filled with nitrogen or other inert gas, and no attempt was made to exclude air during storage. The as-received resin form was not identified by the vendor; it was found to contain potassium salts (Fiskum, Blanchard, and Arm 2002a). Before sampling SL-644 from the IBC-supplied plastic bottle, the bulk resin bottle was turned several times to produce a well-mixed material. Sub-samples were removed immediately after mixing. The sieve results of the resin batch were previously reported (Fiskum, Blanchard, and Arm 2002a) and are reproduced in Table 2.1. The dry-sieved fraction incorporating the 212- to 425-µm particle sizes was used in the ion exchange columns.[(a)] This fraction represents 22 wt% of the as-received material. The average particle size corresponded to 540-µm in diameter, expanded in 3 M NaOH-2 M $NaNO_3$-0.1 M KNO_3 (Fiskum, Blanchard, and Arm 2002a). As a general rule, the column diameter should be 20 times greater than the resin particle diameter to minimize wall effects (Korkisch 1989, p. 39). Given the diameter of the column at 1.46 cm, the column diameter was 27 times the average diameter of the 212- to 425-µm dry-sieved resin particles expanded in caustic solution. For comparison with previous reports, the SL-644 dry-sieved particle-size distribution used for AZ-102 testing at SRTC (Hassan et al. 2001) is also shown in Table 2.1.

Table 2.1. Dry Particle-Size Weight-Percent Distribution of Various Batches of As-Received SL-644

Sieve Size[(1)]	Particle Size (µm)	010319SMC-IV-73 Wt% (PNWD)	981020mb48-563 Wt% (SRTC)
18	>1000	0.06	not analyzed
30	600–1000	37.27	57.33
40	425–600	38.23	23.73
50	300–425	18.01	13.71
70	212–300	6.08	5.12
100	150–212	0.26	0.11
140	106–150	0.06	not analyzed
>140	<106	0.03	not analyzed

(1) U. S. standard sieve size corresponds to ASTM E-11 specification.

(a) This particle size distribution (PSD) was used successfully in AW-101 simulant testing. It is not representative of the PSD that will be used in the plant.

Properties of the 212- to 425-µm 010319SMC-IV-73 SL-644 resin have been previously reported (Fiskum, Blanchard, Arm 2002a), and selected properties are reproduced in Table 2.2. The F-factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger and was determined at the same time the batch-contact samples and column resin fractions were weighed. The F-factor was obtained by drying approximately 0.5 g resin, under vacuum, at 50°C to constant mass. The F-factor was determined on the H-form (for batch-contact studies) and the as-received form (for column testing) of the resin. The F-factor for the Na-form of the resin was performed differently because of stability problems observed in prior tests on the Na-form of resin (Steimke et al. 2001). Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.[(a)] The L-factor represents the fractional mass remaining after washing the as-received resin form with 0.5 M HNO_3 and DI water and correcting for residual water content as described above. The I_{Na}-factor represents the fractional mass gain upon conversion from the H-form to the Na-form, correcting for water content as described above.

Table 2.2. SL-644 Properties

Property	010319SMC-IV-73
Bulk density of as-received form resin, g/mL	0.74
F-factor, as-received	0.877
L, conversion to H-form, fractional mass remaining	0.538
F-factor, H-form[(a)]	0.762
I_{Na}, fractional mass gain from H-form to Na-form	1.25
(a) SL-644 was stored for 1 year in the H-form before sampling for batch contacts. The H-form F-factor was used in batch-contact calculations.	

2.2 AZ-102 Feed

The AZ-102 sample receipt, phase separation, mixing, subsampling, evaporative concentration, and analysis were reported separately (Fiskum et al. 2002b). Solids were observed to precipitate during the evaporative concentration. The major components and estimated weight percentages in the solids (based on anion, metals, and XRD analysis) were: sodium fluorosulfate (Na_3FSO_4), 57%; sodium oxalate ($Na_2C_2O_4$), 28%; and sodium nitrite ($NaNO_2$), 7%. Sodium nitrate ($NaNO_3$) was also believed to be present in the solids (4wt%), and the remaining material (4wt%) was not identified. The total volume of concentrated AZ-102 (AZ-102C) available for Cs ion exchange and batch-contact processing was about 1.8 L. The AZ-102C feed composition is summarized in Table 2.3. The AZ-102C composition generally agreed with the AZ-102 composition reported by Hassan et al. (2001), allowing for evaporative concentration, with the exception of oxalate and OH^-. The OH^- concentration in the AZ-102C feed was 5 times higher than previously reported, after correcting for the concentration factor. The oxalate concentration was generally unchanged at 3.6E-2 M.[(b)]

(a) After initial drying at ambient temperature under vacuum to constant mass, the resin was heated to 50°C. The heated product appeared (visual inspection) to have degraded, thus potentially nullifying subsequent mass measurements.

(b) Solids formed upon evaporative concentration of the AZ-102 contained significant oxalate.

Table 2.3. Composition of AZ-102C (Envelope B)

Cations, M		Anions, M	
Na^+	4.61 E+0	AlO_2^- [(b)]	3.44 E-02
K^+	1.70 E-1	Cl^-	< 3.95 E-3
Cs^+	5.21 E-4[(a)]	CO_3^{2-} (HP)	1.21 E+0
Ca^{++}	<9 E-4	CO_3^{2-} (F)	<1E-1[(c)]
Cd^{++}	<2 E-5	CrO_4^{-2} [(b)]	2.90 E-2
U (Uranyl) [(b)]	6.37 E-5	F^-	9.29 E-2[(d)]
Mo	1.14E-3	NO_2^-	1.69 E+0
Ni^{++}	<6.8E-5	NO_3^-	6.08 E-1
Pb^{++}	<6.3E-5	OH^-	1.11 E+0
Mole ratios		PO_4^{-3} [(b)] (ICP)	9.25 E-3
Na/Cs mole ratio	8.85 E+3	PO_4^{-3} (IC)	1.42 E-2
K/Cs mole ratio	3.26 E+2	SO_4^{-2}	3.68 E-1
Radionuclides, µCi/mL		Oxalate	1.90 E-2[(e)]
^{60}Co	<3 E-2	TOC (HP)	8.33 E-2
^{134}Cs	1.26 E+0	TOC (F)	1.16 E+0[(c)]
^{137}Cs	2.00 E+3	TC (HP)	1.29
^{154}Eu	<1 E-1	TC (F)	1.24
		Solution Density, g/mL	1.246 (T = 26°C)

(a) The Cs isotopic distribution ratio determined by thermal ionization mass spectrometry (TIMS) is 52.3 wt% ^{133}Cs, 14.9 wt% ^{135}Cs, and 32.8 wt% ^{137}Cs.
(b) Al, Cr, and P determined by ICP-AES; U determined by KPA. The ionic form is assumed on the basis of waste chemistry.
(c) The furnace method determined total carbon (TC) and TOC; the TIC was calculated by difference TIC = TC-TOC. The hot persulfate method (HP) was considered more accurate for TIC and the furnace method more accurate for TC.
(d) The F results should be considered the upper-bound concentration since the F peak shape and retention time suggests the presence of co-eluting anion(s), possibly formate or acetate.
(e) Duplicate oxalate analysis resulted in 1900 and 1450 µg/mL RPD = 27%.

HP = hot-persulfate method; F = furnace method
Bracketed results indicate that the analyte concentration uncertainty exceeds 15%. Less-than results indicate that the analyte concentration was below the instrument detection limit (IDL); the dilution-corrected IDL is given.
Analytical details are provided with Analytical Service Request (ASR) 6280, sample ID 02-0751.

Total carbon (TC), total inorganic carbon (TIC or carbonate), and total organic carbon (TOC) are reported here and elsewhere for two different analytical methods: hot-persulfate (HP) oxidation and furnace (F) oxidation. The differences in the two methods were reflective of the ease with which various organic constituents oxidize in the given method. For these analyses, the furnace oxidation method historically provided more accurate results for TC on tank waste matrices and the hot-persulfate method provided more accurate TIC results on tank waste matrices. The most accurate TOC was therefore the difference TC/F – TIC/HP. The TIC concentration was further evaluated relative to the third equivalency

point (considered to be the HCO_3^- neutralization) on the OH^- titration curve. The reported HCO_3^- concentration of 1.12 M was in good agreement with the TIC determined by the hot-persulfate (1.21 M). Phosphate was reported based on P determination by inductively coupled plasma atomic emission spectrometry (ICP-AES) and on PO_4 determination by ion chromatography (IC).

The anionic charge sum (6.85 M) was not in balance with the cationic charge sum (4.78 M). Therefore one or more anions were positively biased. In comparison with the characterization data reported by Hay and Bronikowski (2000), the carbonate concentration was most likely biased high.

2.3 Batch Contacts

The batch contacts were performed with the H-form of SL-644 batch 010319SMC-IV-73, 212- to 425-µm dry-particle-size resin. The sieved resin fraction was washed on 3/24/01 by contacting three times with 0.5 M HNO_3 followed by four contacts with DI water. The resin was then air-dried and stored in a polyethylene bottle for nearly 1 year. Resin for all subsequent batch contacts was used directly from the storage bottle with no additional conditioning. The F-factor, 0.762, was determined on the stored H-form of SL-644 at the same time aliquots were taken for the batch-contact test. The F-factor represents the ratio of resin mass dried under vacuum at 50°C to the initial resin mass.

The efficacy of the 1-year aged resin was tested before use on the actual tank waste sample. An AW-101 simulant was prepared as described by Golcar et al. (2000). Batch contacts were performed in duplicate at three Cs concentrations, similarly to previously reported AW-101 simulant batch-contact work (Fiskum, Blanchard, and Arm 2002a). The liquid volume to solids mass ratio was 100, the batch contact time was 24 h, and the contact temperature was nominally 24°C. The Cs equilibrium concentration was determined using a ^{137}Cs tracer and counting by gamma energy analysis (GEA).

After determining that the SL-644 was adequate for the purpose, the resin was used for AZ-102C batch-contact testing.[(a)] Batch contacts were performed using feed at three different Cs concentrations. Aliquots of the AZ-102C tank waste samples were tested without spiking, and additional aliquots were spiked with 0.5 M $CsNO_3$ to obtain stock solutions of nominally 4 E-3 M and 7 E-3 M Cs. The initial Cs concentrations in the stock contact solutions and the corresponding Na/Cs and K/Cs mole ratios are given in Table 2.4.

Table 2.4. Initial Cs Concentrations in the AZ-102C Solutions Used for the Batch K_d Tests

Solution	Target initial Cs conc. [M]	Target nominal Na/Cs[(a)] mole ratio	Target nominal K/Cs[(a)] mole ratio
Un-spiked	5.2 E-4	8.9 E+3	3.3 E+2
Cs Spike 1	4.0 E-3	1.2 E+3	4.2 E+1
Cs Spike 2	7.0 E-3	6.6 E+2	2.4 E+1
(a) Na^+ and K^+ are the primary cations that compete with Cs^+ for ion exchange with SL-644.			

(a) Batch contact tests for the AW-101 simulant and AZ-102C actual waste were conducted according to Test Instruction TI-RPP-WTP-164, Rev. 0 *Batch Contact of AZ-101 and AZ-102 Concentrate Tank Waste with SuperLig 644 (Batch ID 010319SMC-IV-73)*, S. K. Fiskum, February 2002.

The batch K_d tests were performed in duplicate at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.07 g of exchanger were contacted with 7 mL of feed. The exchanger mass was determined to an accuracy of 0.0002 g. The waste volume was transferred by pipet, and the actual volume was determined by mass difference with an accuracy of 0.0002 g and the solution density. Samples were agitated with an orbital shaker for approximately 48 h at ambient hot cell temperatures ranging from 23 to 27°C during the two days of contact.

The SL-644 resin generally appeared to float when initially contacted with AZ-102C. Attempts to swirl the vial to pull resin into better solution contact were not successful, and in some cases appeared to be counter-productive. At the end of the 48-h contact period, a small amount of resin was still evident on the vial walls and floating on the surface. At worst case, approximately 5% of the resin volume was visually estimated to be floating and on vial walls.[(a)] At the end of the contact time, the samples were passed through a 0.45 mm nylon syringe filter. Filtrate aliquots were counted for ^{137}Cs.

All Cs K_d measurements were determined by measuring ^{137}Cs on both the stock solution (initial concentration), and the contacted solution (final concentration). Initial ^{133}Cs concentrations were confirmed by inductively coupled plasma-mass spectrometry (ICP-MS).

The batch-distribution coefficients, K_d (with units of mL/g), were determined using the following relationship:

$$K_d = \frac{(C_o - C_{eq})}{C_{eq}} * \frac{V}{m * F * I_{Na}} \tag{2.1}$$

where C_o = initial ^{137}Cs concentration
C_{eq} = equilibrium ^{137}Cs concentration
V = volume of the liquid sample (mL)
m = SL-644 ion exchanger H-form mass (g)
F = water loss factor, H-form resin (0.762)
I_{Na} = mass correction factor for conversion of SL-644 from H-form to Na-form mass (1.25).

The Cs λ value (column distribution ratio) is a function of the dry-bed density (ρ) and feed-condition equilibrium-distribution coefficient, and was obtained as shown in Equation 2.2.

$$\lambda = K_d * \rho \tag{2.2}$$

(a) AZ-101 batch contacts were conducted in parallel to the AZ-102C batch contacts. The SL-644 contacted with the AZ-101 did not float and behaved well. The AZ-101 density was 1.224 g/mL.

The SL-644 dry-bed resin density was determined from the ion exchange processing. It was calculated from the column dry-bed resin mass (M_c) and the resin bed volume (BV) according to Equation 2.3.

$$\rho = \frac{M_c}{BV} \tag{2.3}$$

where M_c = corrected dry bed resin mass (discussed in Section 2.4)
BV = resin-bed volume in AZ-102C or 0.25 M NaOH (discussed in Section 3.3.5).

2.4 Column Run Experimental Conditions

Figure 2.1 shows a schematic of the ion exchange-column system. The system consisted of two small serial columns containing the SL-644 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 to obtain samples and could also be used to isolate the columns from the rest of the system. The 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 prepared at the SRTC Glassblowing Laboratory. Each column consisted of a 15-cm glass column with a 24/40 taper ground-glass fitting on top and a threaded fitting on the bottom. A polyethylene bushing was installed in the glass-threaded fitting to accommodate ¼-in. stainless steel national pipe thread (NPT) fitting. The inside diameter of each column was 1.46 cm, which corresponded to a volume of 1.67 mL/cm. A stainless steel, 200-mesh screen supported the resin bed. The height of the resin bed (and thus shrinkage and swelling) was measured with a decal millimeter scale affixed to the column. The upper section contained four entry ports and a taper joint with screw cap that securely fitted the column. The lead column assembly used a pressure relief valve (10 psi trigger), pressure gauge, and sample inlet; the remaining port was plugged. The lag column assembly used one port for sample entry, and the other three ports were plugged. In both columns, the inlet sample lines extended through the port opening to the top of the column. The connecting tubing was ⅛-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, NY) equipped with a ceramic and Kynar® coated low-flow piston pump head was used to introduce all fluids. The flow rate was controlled with a remotely operated FMI stroke-rate controller. The pump was set up to deliver flowrates from 0.08- to 16-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 total holdup volume of the Cs ion exchange system was the summed volume of all fluid-filled parts, and was estimated to be 42 mL.

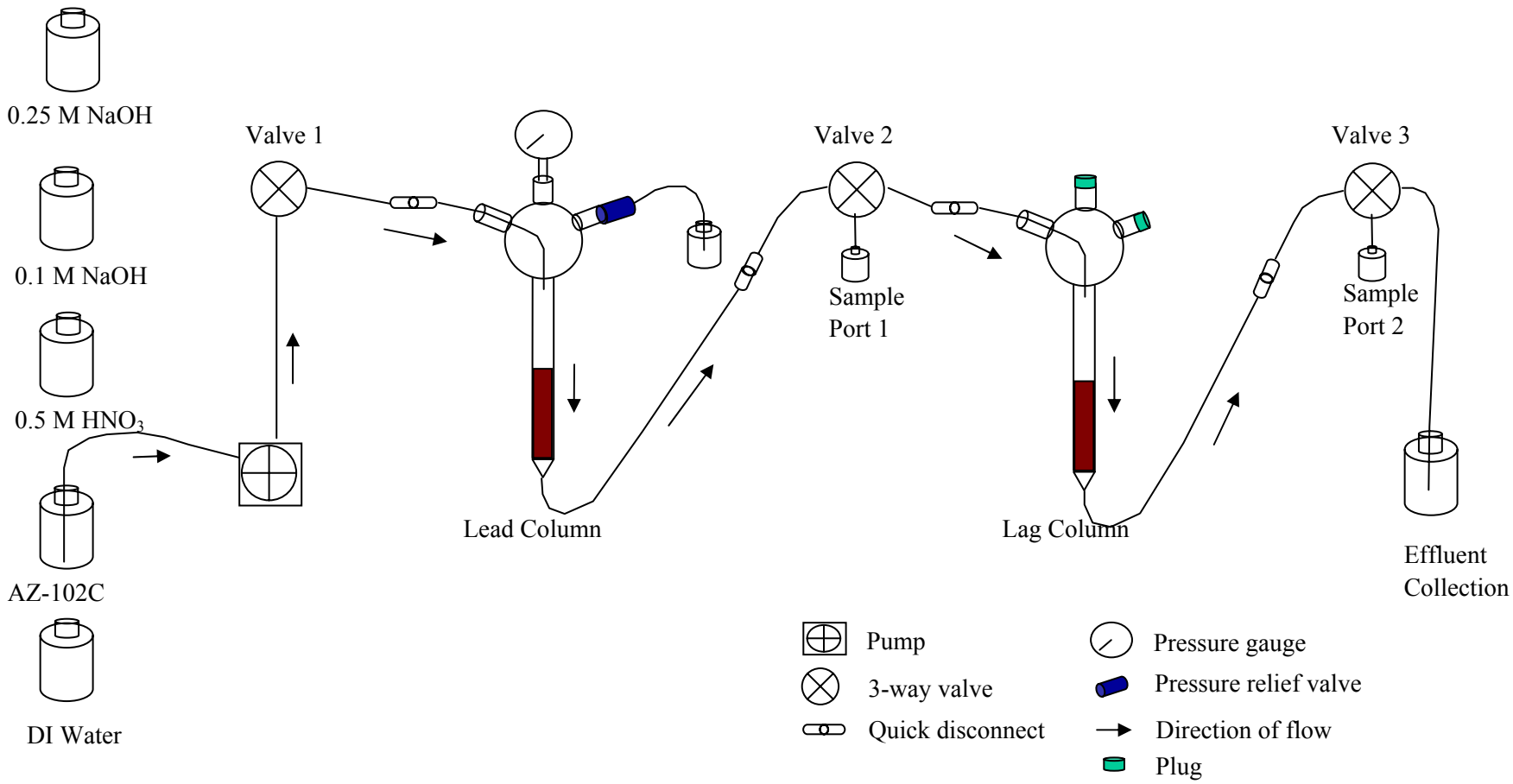


Figure 2.1. Cesium Ion Exchange Column System

Before installing the system into the hot cell, both of the resin beds were individually cycled through the acid form. After the resin cycling, the mass of the washed Na-form SL-644 (M_c) was calculated to be 2.4 g, on a dry-weight basis, in each column according to Equation 2.4.

$$M_c = M * L * F * I_{Na} \tag{2.4}$$

where M_c = Na-form SL-644 mass in the resin bed
M = SL-644 as-received resin mass
L = fractional mass remaining after washing (0.538)
F = water-loss factor, as-received form (0.877)
I_{Na} = fractional mass gain on conversion from H-form to Na-form (1.25) (this factor is set to 1 when calculating the dry-bed density in the H-form or 0.5 M HNO_3).

The entire ion exchange system was then used for a full shakedown experiment with AW-101 simulant (Fiskum, Blanchard, Arm 2002a). Both columns were individually eluted, rinsed, and regenerated. The ion exchange system was then transferred to the hot cell and used to process AP-101DF tank waste (Fiskum et al. 2002a). Only the lead column was eluted to a ^{137}Cs concentration C/C_o of 4 E-3, which was equivalent to a ^{137}Cs concentration of 0.5 μCi/mL. The lead column was then rinsed with DI water, regenerated with 0.25 M NaOH, and again rinsed with DI water. The lag column contained an estimated 35-μCi ^{137}Cs from the lead column Cs breakthrough. After an 8-week storage period (resin as Na form in DI water), the lead and lag column positions were switched, and 0.75 L of AN-102/C-104 was processed (Fiskum, Blanchard, and Arm 2002b). Again, only the lead column was eluted to a ^{137}Cs concentration C/C_o of 6 E-3, which was equivalent to a ^{137}Cs concentration of 1 μCi/mL. The lead column was then rinsed with DI water, regenerated with 0.25 M NaOH, and again rinsed with DI water. The lag column contained an estimated 0.22-μCi ^{137}Cs based on the integration of the lead column Cs breakthrough.

The system was stored for 118 days since the end of the AN-102/C-104 column run. The lead and lag columns were switched, and the apparatus volume (AV) of DI water was displaced with 0.25 M NaOH before introducing AZ-102C feed. All subsequent processing was performed in the hot cells at temperatures ranging from 25 to 27°C. Table 2.5 shows the experimental conditions for each process step, where one bed volume (BV) is the volume in 0.25 M NaOH (10.2 mL). The bed conditioning, AZ-102C loading, feed displacement, and DI water-rinse steps were conducted by passing these solutions through both resin beds connected in series.[(a)] The AZ-102C effluent was collected in twelve effluent bottles. The first bottle collected 41 mL, nominally one AV, and consisted primarily of the displaced regeneration solution. The remaining effluent was collected in nominally 10-BV fractions. After ascertaining that the ^{137}Cs concentration met product specifications, the individual effluent fractions were combined (excluding the initial effluent bottle).[(b)] Sampling of the feed displacement solution began

(a) The ion exchange processing of the AZ-102C actual waste was conducted according to Test Instruction TI-PNNL-WTP-132, Rev. 0, *Separation of Cesium from Hanford Tank Waste 241-AZ-102 Using the Dual Small-Column SuperLig® 644 Cesium Ion Exchange System*, S. K. Fiskum, November 2001.

(b) Compositing the Cs-decontaminated effluent was conducted according to Test Instruction TI-RPP-WTP-145, Rev. 0, *Compositing AZ-102C Cesium Ion Exchange Effluent and Subsequent Sub-Sampling for Analysis*, S. K. Fiskum, November, 2001.

immediately after switching the feed line into the 0.1 M NaOH solution. The elution was conducted on the lead column only, continuing until 14.8 BV had been processed through the column. The resin bed was then rinsed, regenerated, and rinsed again as shown in Table 2.5. Because initial test results indicated low Cs recovery in the eluate, the lead column was re-eluted 16 days later.[a] The re-elution process was halted when it was apparent that little additional Cs was removed. Only the initial 14.8 BVs of Cs eluate samples were composited[b] and sampled for analysis.

Table 2.5. Experimental Conditions for AZ-102C Ion Exchange

Process Step	Solution	Total Volume			Flow rate		Time	T
		BV[a]	AV[b]	mL	BV/h	mL/min	h	°C
Two Columns in Series[c]								
DI water displacement	0.25 M NaOH	8.9	2.2	91	2.4	0.42	3.6	25
Loading Lead column	AZ-102C Feed	105	25.5	1071	1.4	0.23	80.2	26
Loading Lag column[d]	AZ-102C Feed	100	24.3	1022	1.4	0.23	80.2	26
Feed displacement	0.1 M NaOH	10.5	2.6	107	2.6	0.44	4.0	25
DI water rinse	DI water	9.2	2.2	94	2.6	0.44	3.2	25
Lead Column Only								
Elution	0.5 M HNO_3	14.8	6.6	151	0.74	0.13	20.2	25
Eluant rinse	DI water	4.4	2.0	45	2.6	0.45	1.75	25
Regeneration [e]	0.25 M NaOH	4.4	2.0	45	0.95	0.16	4.75	26
Rinse	DI water	4.6	2.0	47	2.7	0.45	1.75	26-27
Second Elution Cycle								
Elution [f]	0.5 M HNO_3	6.5	2.9	66	2.8	0.48	2.5	25
Eluant rinse	DI water	4.8	2.1	49	2.6	0.44	1.9	25
Regeneration[g]	0.25 M NaOH	4.9	2.2	50	0.83	0.14	5.9	25
Rinse	DI water	4.1	1.8	41	2.5	0.42	1.7	25

(a) BV = bed volume (10.2 mL in 0.25 M NaOH regeneration condition).
(b) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column, and 21 mL for lag column).
(c) Run date began on 11/5/01.
(d) The feed volume through the lag column was reduced because of sampling from the lead column.
(e) The eluant rinse ended on 11/10/01; regeneration began 3 days later on 11/13/01.
(f) The second elution began 16 days after the regeneration rinse on 11/29/01.
(g) Regeneration was initiated 11/30/01, ½ day following the eluant rinse.

(a) The continued elution was conducted according to Test Instruction TI-PNNL-WTP-147, Rev. 0, *Continued Cesium Elution Following Cs Removal from Hanford Tank Waste 241-AZ-102 Using the Dual Small-Column SuperLig® 644 Cesium Ion Exchange System*, S. K. Fiskum, November, 2001.

(b) The Cs eluate samples were composited according to Test Instruction TI-RPP-WTP-151, Rev. 0, *Preparing a Composite Solution of the Acid Eluant Samples from AZ-102C Cs Ion Exchange Lead Column,* S. K. Fiskum, January 2001.

2.5 Column Sampling

The sampling and analysis protocol is shown in Table 2.6. During the loading phase, small samples (about 2 mL) were collected from the lead and lag columns at nominal 5-BV increments. The flow rate averaged 1.4 BV/h. The flow rate increased slightly during sample collection from the lead column because of the siphoning effect at the sample port. The feed displacement, DI water rinse, elution, and elution rinse samples were taken at 1-BV increments at flow rates shown in Table 2.5.

Table 2.6. Sampling Interval and Analyses

Process Step	Frequency		Approximate Sample Size (mL)	Analyses
	Lead Column	Lag Column		
Loading	Every 5 BV	Every 5 BV	2	GEA
Feed displacement	none	Every 1 BV	10	GEA
DI water rinse	none	Every 1 BV	10	GEA
Elution	Every 1 BV	NA	10	GEA
Eluant rinse	Every 1 BV	NA	10	GEA
Composite Samples				
Effluent – 1	NA	NA	2	GEA
Effluent composite (bottles 2-12)	NA	NA	10	GEA, ICP-AES, IC, U, OH, TOC/TIC
Regeneration	1 composite	NA	10	ICP-AES, GEA, OH-
Eluate	1 composite	NA	10	ICP-AES, GEA, TIMS, TOC, IC, U,

GEA = gamma energy analysis
ICP-AES = inductively-coupled plasma atomic energy spectrometry
IC = ion chromatography
ICP-MS = inductively-coupled plasma mass spectrometry
OH = hydroxide
TOC = total organic carbon
TIC = total inorganic carbon
TIMS = thermal ionization mass spectrometry
NA = not applicable

2.6 Sample Analysis

The ^{137}Cs concentration was determined using a bench-top GEA spectrometer. This allowed for rapid sample analysis. Selected sample results were later confirmed by GEA analysis in the Chemical Measurements Center (CMC) analytical laboratory. The effluent composite sample was submitted for various analyses: GEA, ICP-AES, TOC/TIC, IC, U, and OH^-. The eluate samples required dilution before removal from the hot cell to reduce the dose rate from ^{137}Cs. The extent of dilution was determined by mass difference. Once the GEA results were confirmed for the eluate samples, the eluate samples were composited, and a sample of the composite was submitted for various analyses: GEA, ICP-AES, TOC, IC, Cs isotopic distribution, and U. A sample of the regeneration solution was taken for ICP-AES analysis and OH^- determination.

The Na and other metal concentrations were determined with ICP-AES. The OH^- concentration was determined by potentiometric titration with standardized HCl. Uranium was determined using kinetic phosphorescence. Anions were determined using IC. TOC and TIC were determined by silver-catalyzed

hot-persulfate oxidation and furnace oxidation methods. The Cs isotopic distribution was determined by thermal ionization mass spectrometry (TIMS) directly on the eluate.

3.0 Results and Discussion

This section describes the results from batch-contact and column testing of AZ-102C with SL-644.

3.1 Confirmation of SL-644 Efficacy for Batch Contacts Using AW-101 Simulant

The aging of SL-644 resin has been a concern. Even short storage times of the resin in the Na or K form have shown signs of degradation in terms of color and Cs-removal effectiveness. The resin used for the batch contacts was stored for about 1 year in a polyethylene bottle in the dry H-form. No effort was made to diminish its contact with air. The efficacy of using this 1-year aged resin for batch contacts was first tested with AW-101 simulant, and its performance was compared to the performance obtained about 1 year before.

The AW-101 simulant K_d values from the 1-year aged H-form SL-644 were calculated using Equation 2.1 and were based on the ^{137}Cs tracer concentrations as measured by GEA. The Na/Cs mole ratios were calculated based on the as-prepared simulant composition. The equilibrium Cs concentrations were based on the ^{137}Cs concentrations and the ratio of ^{137}Cs: total Cs as-prepared for the unspiked and spiked solutions. The Na concentration was assumed to be constant for the batch contacts.

The calculated ^{137}Cs K_d values for AW-101 simulant are plotted as a function of the equilibrium Na:total Cs mole ratio in Figure 3.1. The current test results are plotted with the previous test results (Fiskum, Blanchard, and Arm 2002a) for comparison. The K_d of the aged H-form SL-644 resin appeared degraded about 20% relative to the initial testing one year ago, as evidenced by the decreased slope and intercept. The resin degradation suggested by the drop in the resin batch-contact performance is expected to be very different than the degradation of the resin in the columns due to the very different storage and processing histories.

3.2 AZ-102C Batch-Contact Results

The K_d values were calculated using Equation 2.1 and were based on ^{137}Cs concentrations as measured by GEA. The Na/Cs mole ratios were calculated based on the measured Na and total Cs concentrations in the uncontacted AZ-102C. The equilibrium Cs concentrations were based on the ^{137}Cs concentrations and the ratio of ^{137}Cs: total Cs determined for the unspiked and spiked solutions. The Na concentration was assumed to be constant for the batch contacts. Since the quantity of H^+ added with the resin was small relative to the moles of Na^+ and OH^- in the contact solution (phase ratio of 100 mL of solution: gram of exchanger), this was a reasonable assumption. In these batch-contact experiments, the 7-mL waste-solution volumes were estimated to contain 7.8 meq of OH^- and 32 meq of Na^+, while the 0.07 g resin mass contained 0.12 meq of H^+.[(a)]

(a) The SL-644 contains 2.2 meq H^+ per gram of H-form resin (Rapko et al. 2002).

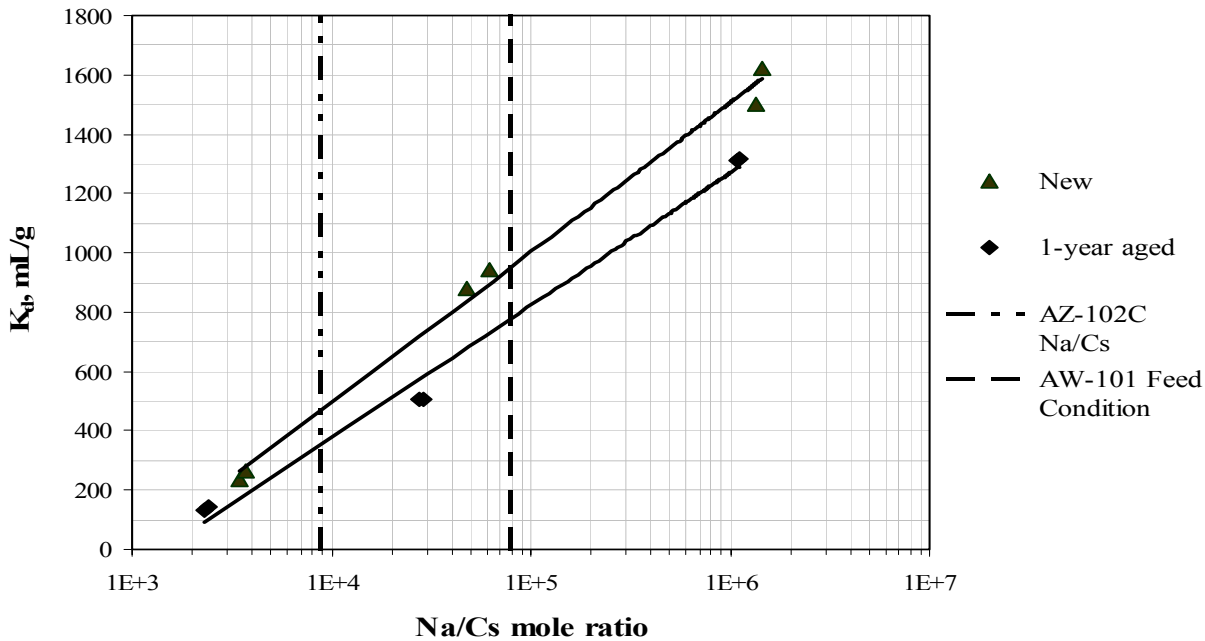


Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for SL-644 (010319SMC-IV-73 212- to 425-µm Particle-Size Distribution [PSD]) New Resin and One-Year Aged Resin in AW-101 Simulant (T = 23°C)

The calculated ^{137}Cs K_ds for AZ-102C are plotted as a function of the equilibrium Na:total Cs mole ratios in Figure 3.2. The best-fit logarithmic regression is shown where

$$K_d = 52.6 * Ln\left(\frac{[Na]}{[Cs]}\right) - 318 \text{, and } r^2 = 0.98. \quad (3.1)$$

At the nominal feed condition of 8.84 E+3 Na:Cs mole ratio, the SL-644 K_d value was 160 mL/g.

For comparison, the K_d values obtained at the lower Na:Cs mole ratios for AP-101DF (Fiskum et al. 2002a) and AN-102/C-104 (Fiskum, Blanchard, and Arm 2002b) are also shown. The AZ-102C feed condition K_d values clearly resulted in a lower slope than similar values generated with AP-101DF and AN-102/C-104. The decreased slope may be attributed, in part, to insufficient contact with the exchanger. Part of the exchanger tended to float in the AZ-102C matrix.

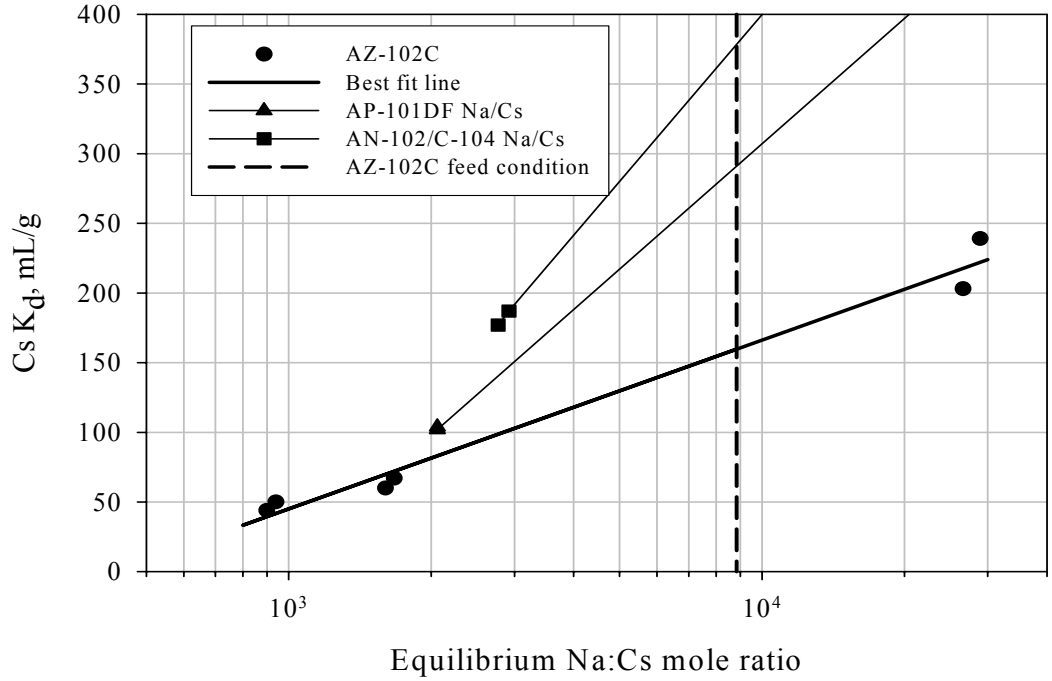


Figure 3.2. ^{137}Cs Distribution Coefficients (K_d) for SL-644 (AZ-102C) (T = 23 to 27°C)

The calculated dry-bed densities calculated according to Equation 2.3 using the resin masses and volumes in the ion exchange column system (discussed in Section 3.3.5) are given in Table 3.1. Good agreements of the dry-bed densities were obtained between the lead and lag columns.

Table 3.1. Dry Bed Density

Property	**010319SMC-IV-73, Lead Column**	**010319SMC-IV-73, Lag Column**
Column resin mass, g, as-received[(a)]	4.17	4.20
Corrected resin mass, M_c, in column, g	2.46	2.48
BV, 0.25 M NaOH, mL	10.2	10.0
BV, AZ-102C, mL	9.5[(b)]	9.4
BV, 0.5 M HNO_3, mL	8.1	not performed
Dry bed density, ρ_b, in feed		
Na-form		
0.25 M NaOH, g/mL	0.241	0.248
AZ-102C, g/mL	0.259[(c)]	0.264
H-form		
0.5 M HNO_3, g/mL	0.243	not performed

(a) The 212- to 425-µm particles size resin mass was measured in the as-received form.
(b) The resin BV in the lead column increased to 10.4 mL after processing 45 to 50 BVs.
(c) The dry-bed density decreased to 0.236 g/mL when the resin bed expanded to 10.4 mL after processing 45 to 50 BVs.

The predicted Cs λ value was calculated according to Equation 2.4 to be 41 BVs in the AZ-102C feed condition, and 38 BVs in the 0.25 M NaOH regeneration condition. These values are the approximate points at which the Cs breakthrough curve is predicted to pass through C/C_o = 50%. Figure 3.3 shows the Cs λ values as a function of the Na:Cs mole ratio for the SL-644 (calculated bed density in 0.25 M NaOH of 0.24 g/mL).

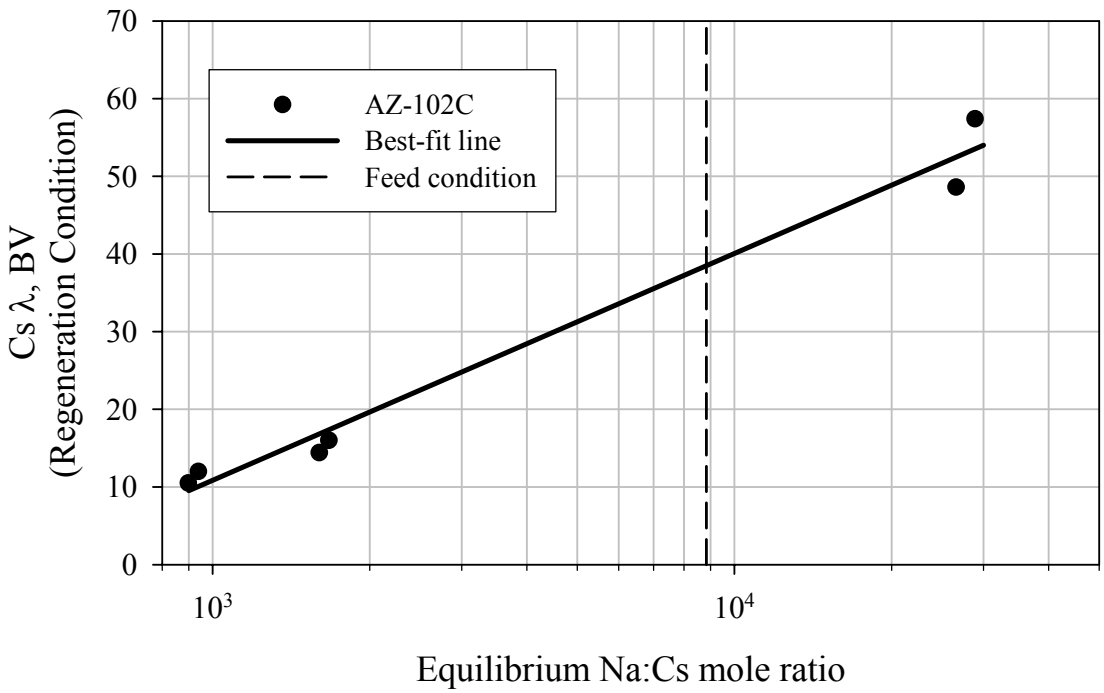


Figure 3.3. Estimated ^{137}Cs λ Values for SL-644 (AZ-102C) (T = 23 to 27°C)

3.3 Column Test

The column system used for AZ-102C processing was the same system used for processing the AW-101 simulant, AP-101DF actual waste, and AN-102/C-104 actual waste. No resin fouling was observed through all ion exchange processing steps.

3.3.1 Loading, Feed Displacement, and Rinse

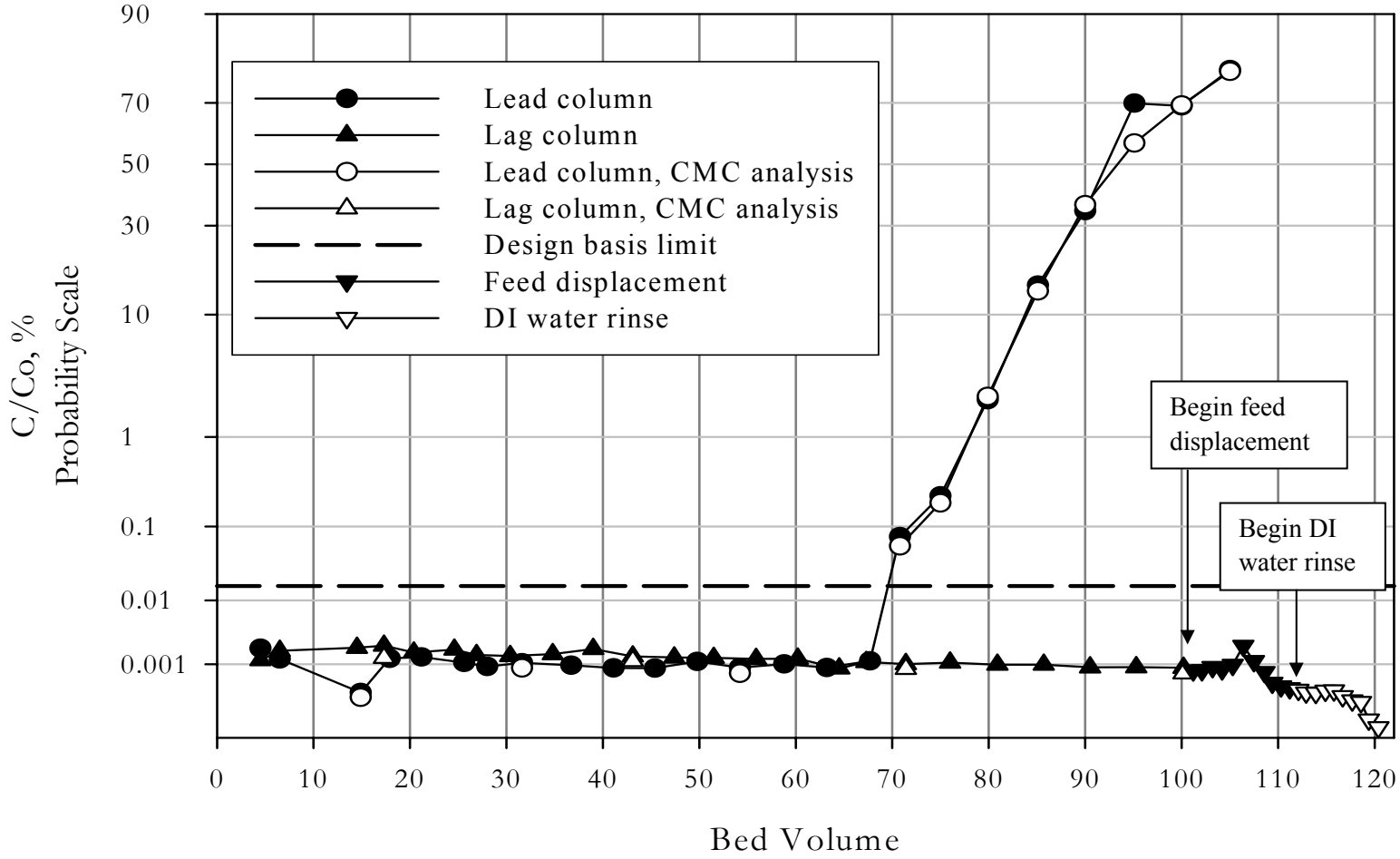
The loading phase was initiated with AZ-102C waste. Approximately 1.0 AV (41 mL) of effluent was initially collected in a separate collection bottle. Most of this effluent was 0.25 M NaOH from the regeneration step mixed with some AZ-102C in the ion exchange apparatus. This solution was maintained separately and not mixed with the final Cs-decontaminated AZ-102C effluent composite; thus, most of the apparatus 0.25 M NaOH solution was prevented from mixing with the AZ-102C effluent. The resin beds shrank an average 6.6% to 9.5 mL as they converted from the regeneration solution to the AZ-102C feed. The lead column resin-BV inexplicably increased in volume to 10.4 mL after processing 45 to 50 BVs of feed.

Figure 3.4 shows the ^{137}Cs effluent concentrations from the columns as % C/C_o 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 10.2 mL. The C_o value for ^{137}Cs was determined to be 2005 μCi/mL. The C/C_o is plotted on a probability scale as this scale results in a straight line if the resin performs according to ideal ion exchange theory. The C/C_o values, determined using the bench-top GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the CMC analytical laboratory. Analytical results and calculations are located in the Appendices C and D.

The load profile shows excellent removal of Cs from the feed where breakthrough begins at 68 BVs. The breakthrough increased rapidly, culminating with a 50% Cs breakthrough at approximately 93 BVs. This value is over twice the Cs λ value predicted from batch-contact studies (38 BVs). The load characteristics of this waste agreed with modeling predictions (Hamm, Smith, and McCabe 2000) where higher Cs concentrations result in steep breakthrough profiles. Cs capacity for this resin was high. A total of 66 mg (0.49 mmoles) Cs was loaded on the 2.46 g Na-form resin (1.97 g resin H-form resin) lead column at a 50% breakthrough of 93 BVs.

The ^{137}Cs concentrations in the lag-column effluent samples were relatively constant over the course of the loading phase and were only slightly (factor of 1.5) higher in ^{137}Cs concentration than those obtained from the first 68-BVs from the lead column. This observation was different than that of AN-102/C-104 processing where ^{137}Cs concentration in the lag-column samples were found to be 50 times higher than the lead-column samples, a result of ^{137}Cs "bleed-off" from previous waste processing (Fiskum, Blanchard and Arm, 2002b). Two major factors masked this effect for the AZ-102C processing. The previous feed (AN-102/C-104) loading was minimal in that only 0.75 L was processed loading a total of 6 mg Cs. Furthermore, the ^{137}Cs concentration in AZ-102C (2005 μCi/mL) was over an order of magnitude greater than the previous AN-102/C-104 feed (161 μCi/mL). Thus, the relative proportion of the ^{137}Cs bleed-off from the AN-102/C-104 processing would appear to be an order of magnitude lower (measured as C/C_o).

The total quantity of ^{137}Cs processed through the ion exchange system was 2220 mCi (26 mg ^{137}Cs, 78 mg total Cs). The breakthrough to the lag column was not certain. Integrating the ^{137}Cs activity in the lead-column samples resulted in a calculated 10% Cs breakthrough. Integration of the individual lead-column eluate samples resulted in 94.6% ^{137}Cs recovery, indicating 5.4% Cs breakthrough. The composite eluate analysis resulted in 98.9% ^{137}Cs recovery, indicating 1.1% Cs breakthrough. The estimated Cs breakthrough was assigned the median value of 5.4%, which was equivalent to 120 mCi ^{137}Cs and 4.2 mg Cs. Thus the total ^{137}Cs loaded onto the lead column was calculated to be 2100 mCi (24 mg ^{137}Cs or 74 mg total Cs). The total lag-column effluent contained 1.92E-2 μCi ^{137}Cs/mL corresponding to 0.00078% (i.e., 1.92E-2 μCi/mL x 897 mL / 2.22E+6 μCi x 100) of the total ^{137}Cs processed through the columns.



Conditions: SL-644 batch number 010319SMC-IV073 212- to 425-µm dry particle size
BV in 0.25 M NaOH feed condition = 10.2 mL
^{137}Cs C_o = 2.00 E+3 µCi/mL

Process temperature = 26°C
Flow rate = 1.4 BV/h
Na concentration = 4.61 M.

Figure 3.4. ^{137}Cs Breakthrough Curves for AZ-102C Tank Waste, Probability Plot

The contract ^{137}Cs removal limit, 0.0162% or 0.322 μCi/mL ^{137}Cs, is also shown in Figure 3.4. The contract limit was derived from a maximum of 0.3 Ci/m^3 for ^{137}Cs in the low-activity waste (LAW) glass, a Na concentration of 4.61 M in the AZ-102C, a ^{137}Cs feed concentration of 2.00 E+3 μCi/mL, a minimum 5 wt% total Na_2O loading in the glass (where all Na is assumed to come from the waste itself), and a glass product density of 2.66 g/mL. The maximum loading for AZ-102C in glass is 5.5 wt% waste Na_2O and corresponds to 0.292 μCi/mL, C/C_o of 0.0146%, and a decontamination factor (DF) of 6870. The lag-column effluent remained an order of magnitude below the maximum waste loading contract limit. The DFs were calculated on selected effluent samples and the composite effluent, and are summarized in Table 3.2.

Table 3.2. Selected Decontamination Factors for ^{137}Cs from AZ-102C

Sample	Volume Processed, BV (mL)	^{137}Cs Concentration μCi/mL[(a)]	C/C_o, %	DF[(b)]
Third lead column sample	14.9 (152)	5.43E-3	2.71E-4	3.69E+5
Final lead column sample	105 (1071)	1.57E+3	78.1	1.28
Fourth lag column sample	17.3 (176)	2.55E-2	1.27E-3	7.86E+4
Final lag column sample	100 (1022)	1.40E-2	6.98E-4	1.43E+5
Composite effluent	87.9 (897)[(c)]	1.92E-2	9.55E-4	1.05E+5

(a) The ^{137}Cs uncertainty ranged from 3% to 4% relative error, 1-σ.
(b) The maximum waste Na_2O loading DF requirement was 6870.
(c) The composite volume represents the effluent volume collected, as opposed to the volume processed. This volume is lower than the process volume because samples were taken during the processing run.

This performance is significantly better than observed in the previous column test of SL-644 with AZ-102 (Hassen et al 2001). The authors of that study indicated that the "evidence is strong, but not conclusive" that Al and Ca species precipitated in the resin beds during their test, which used unconcentrated AZ-102 feed. As noted in Sec. 2.2, solids precipitated during concentration of the AZ-102 feed prior to the ion exchange test represented in Figure 3.4. However, almost all (at least 96wt%) of the solids that formed during the evaporation did not contain Al or Ca. The reason for the different results is unclear, but the results presented here clearly show that SL-644 can meet the baseline requirements for removing Cs from AZ-102 supernate concentrated to approximately 5 M Na.

The Cs-decontaminated effluent was characterized, and the results are summarized in Table 3.3. Most analyte concentrations were equivalent to the feed concentration within the experimental error of the method (typically ±15%). The anionic charge is higher than the cationic charge, as was found in the feed. Again, one or more of the anions is biased high. Previous testing showed U was removed by SL-644 processing. The effluent analysis results for AZ-102C were different indicating only 27% U was removed.

The ^{137}Cs bleed-off continued through the first 5 BVs of the feed displacement (Figure 3.4). The lag-column flow rate seemed to slow down after collecting the third feed-displacement sample. This was evidenced by fluid filling the space above the resin bed. The flow corrected itself during collection of the seventh feed-displacement sample. It was also apparent that the sixth feed-displacement sample (6.2 BVs

or 1.5 AVs of feed displacement) had significant coloration clearly evident through the cell window. This was accompanied by a slight rise in C/C_o.

The introduction of water caused a sharp drop in $\%C/C_o$ after 4 BVs (1 AV) were passed through the system. The slight rise in $\%C/C_o$ during feed displacement may be caused by the decrease in ionic strength and/or decrease in hydroxide concentration (Rapko et al. 2002) of the rinse solution. The subsequent drop in Cs bleed-off was related to the drop in feed Na concentration, which acted as a competitor to Cs on the ion exchanger.

Table 3.3. AZ-102C Cs-Decontaminated Product

Cations	M	% Change from Feed	Anions	M	% Change from Feed
Na^+	4.57 E+0	-0.9	AlO_2^- [(a)]	3.43 E-2	-0.3
K^+	1.67 E-1	-1.8	Cl^-	<3.6 E-3	NA
Cs^+	4.99 E-9	>>-99	CO_3^{2-} (HP)	1.15 E+0	-5.0
Ca^{++}	<9 E-4	NA	CO_3^{2-} (F)	6.9 E-1[(c)]	>590[(c)]
Cd^{++}	<2 E-5	NA	CrO_4^{-2} [(a)]	2.83 E-2	-2.4
Mo	1.12E-3	-1.8	F^-	8.84 E-2	-4.8
Ni	<6.8E-5	NA	NO_2^-	1.63 E+0	-3.6
Pb	<6.5E-5	NA	NO_3^-	5.83 E-1	-4.1
U (Uranyl) [(a)]	4.64 E-4	-27	OH^-	1.14 E+0	3.2
Radionuclides	**µCi/mL**		PO_4^{-3} [(a)] (ICP)	9.14 E-3	-1.2
^{60}Co	<2 E-5	NA	PO_4^{-3} (IC)	1.34 E-2	-5.6
^{134}Cs	<2 E-5	NA	SO_4^{-2}	3.52 E-1	-4.3
^{137}Cs	1.92 E-2	>>-99	Oxalate	1.53 E-2	-19
^{154}Eu	<5E-5	NA	TOC (HP)	4.37 E-2[(b)]	-48[(b)]
Solution Density	**g/mL**		TOC (F)	4.9 E-1[(c)]	-58[(c)]
Density	1.241 T = 27°C	-0.4	TC (HP)	1.19 E+0	-7.8
			TC (F)	1.18 E+0	-4.8

(a) Al, Cr, and P determined by ICP-AES. Uranyl determined by KPA. The ionic form is assumed on the basis of waste chemistry.
(b) The TOC determination for the feed had high uncertainty; one sample resulted in <4.2E-2 M, and the duplicate resulted in 8.3E-2 M. The comparison was made with the single measured value 8.3E-2 M.
(c) The furnace method determined total carbon (TC) and TOC; the TIC was calculated by difference TIC = TC-TOC. The hot persulfate method (HP) is considered more accurate for TIC and the furnace method more accurate for TC.
NA = not applicable; concentration was less than the instrument detection limit; HP = hot-persulfate method; F = furnace method.
Less-than results indicate that the analyte concentration was below the method detection limit; the method detection limit is given.
Analytical details are provided with Analytical Service Request (ASR) 6280, sample ID 02-0752.

3.3.2 Elution and Eluant Rinse

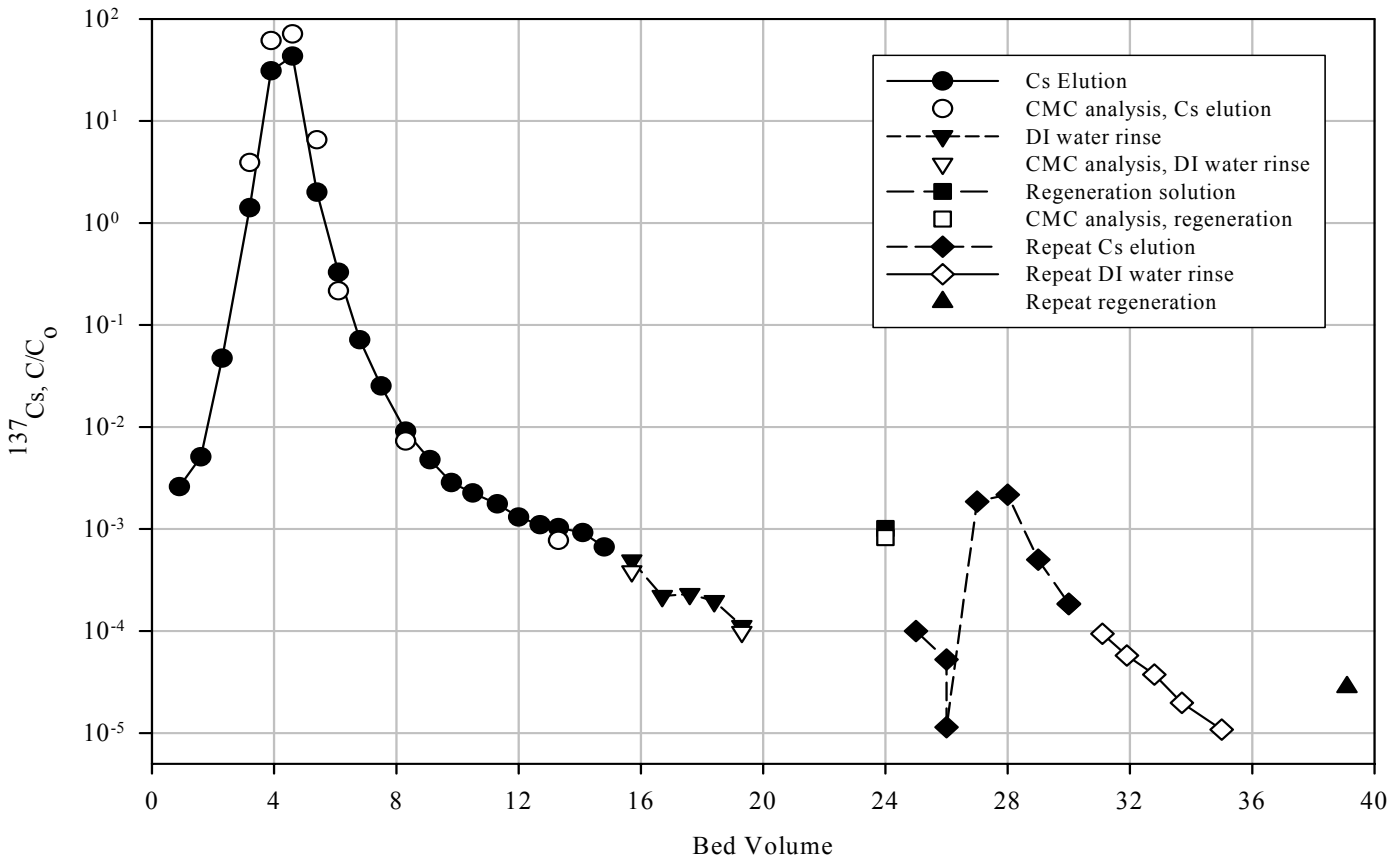
Figure 3.5 shows the lead column elution and eluant rinse profiles. The ordinate shows the ^{137}Cs C/C_o[(a)] values on a logarithmic scale to clearly show the large range of C/C_o values obtained. The abscissa is given in BVs relative to the regeneration condition. Most of the ^{137}Cs was contained in elution BVs 3 through 6. The peak value of C/C_o was found to be 71. Integrating the elution peak resulted in 95% ^{137}Cs recovery in the eluate; analyzing the composited eluate resulted in 98.9% ^{137}Cs recovery. This indicated that only 1 to 5% Cs breakthrough from the lead column onto the lag column was obtained. The elution cutoff of $C/C_o = 0.01$ was reached at 8.3 BV, but elution was continued beyond this because of the lag between sample collection and the determination of the Cs concentration. A C/C_0 of 0.001 was reached within an additional 6 BVs.

The lead column was rinsed, regenerated, rinsed, and briefly eluted again.[(b)] The second elution resulted in little additional Cs removal from the resin bed. The second elution peak C/C_o was found to be 0.002, higher than the last eluate sample from the first elution process. The integrated peak represented 4.6E-3% of the loaded Cs. The C/C_o values for the eluant rinse with DI water dropped rapidly in ^{137}Cs concentration, indicating that DI water did not continue Cs elution.

Also shown in Figure 3.5 are the composite regeneration solution C/C_o values. The regeneration effluent volumes were 4.4 and 4.9 BVs; the point at 24 BVs represents the regeneration effluent volume collected from 19 to 24 BVs, and the point at 39 BVs represents the composite regeneration effluent volume collected from 35 to 39 BVs. The regeneration solution was expected to result in higher C/C_o values than obtained with the last DI water sample because the solution contains the competing Na ion. It is interesting to note that the initial regeneration effluent resulted in nominally 0.1% C/C_o, nearly an order of magnitude higher than the final DI water rinse sample and nearly equivalent to the last elution sample. The second re-generation solution resulted in a *significantly* lower C/C_o of 0.003%, about half an order of magnitude higher than the final rinse sample.

(a) The C_o refers to the ^{137}Cs concentration in the AZ-102C sample fed to the lead column. For elution, the C/C_o value is an indication of the extent to which ^{137}Cs is concentrated relative to the feed. It is an indirect measure of the extent to which the resin is actually eluted.

(b) Initial results indicated significant Cs remained on the column.



Conditions: SL-644 batch # 010319SMC-IV-073 212- to 425-µm dry particle size

BV in the 0.25M NaOH regeneration condition = 10.4 mL

^{137}Cs C_o = 2005 µCi/mL

Process temperature = 28°C

Flow rate = 0.95 BV/h

Eluant = 0.5 M HNO_3.

Figure 3.5. AZ-102C ^{137}Cs Elution and Eluant Rinse of the Lead Column

The eluate samples from the lead column first elution were composited, and a sub-sample was taken for analysis. Table 3.4 shows the analytical results. Sodium was the dominant component detected using ICP-AES with some Cd, Cr, Cu, Fe, Ni, Pb, Zn, and U eluting as well. Table 3.5 summarizes these analyte recoveries on a mass and molar basis. The U recovered in the eluate (5.5 mg) represented 34% of the U loaded in the AZ-102C feed (16.2 mg). The remaining U was recovered in the LAW effluent (the calculated LAW U recovery was 73%, Table 3.3). Although a smaller fractional recovery of Cr was obtained (0.70 %) in the eluate, the mole fraction in the eluate was higher than that found for U. The fractional recoveries for Cu, Fe, Ni, Pb, and Zn were calculated to one significant figure because the elements were not detected in the feed. The high Ni recovery (>300 %) was probably a result of Ni loading on the lag column during the previous AN-102/C-104 processing (the lag column for this test was not eluted). The total mmoles of Na (8.1) dominated the eluate composition followed by Ni and Cr at 0.24 and 0.22 mmoles, respectively. As expected, ^{137}Cs was the only radionuclide detected by GEA.

Table 3.4. Inorganic and Organic Analytes in the Lead Column Eluate Composite

Analyte	Method	MRQ[a] µg/mL	Cs eluate[b] µg/mL	% Recovered	Analyte	Method	MRQ µg/mL	Cs eluate[b] µg/mL	% Recovered
Al	ICP-AES	NMRQ[c]	[12]	[0.17]	Ni	ICP-AES	NMRQ	95.1	>300
B	ICP-AES	NMRQ	[15]	[2.7]	Pb	ICP-AES	NMRQ	[36]	[>40]
Ba	ICP-AES	NMRQ	[4.0]	[>27]	Si	ICP-AES	NMRQ	<52	<3
Ca	ICP-AES	NMRQ	<27	NM	Sn	ICP-AES	NMRQ	<160	NM
Cd	ICP-AES	NMRQ	[8.3]	[>60]	Sr	ICP-AES	NMRQ	<2	NM
Co	ICP-AES	NMRQ	<5	NM	Ti	ICP-AES	NMRQ	<3	NM
Cr	ICP-AES	NMRQ	74.8	0.68	U	KPA	NMRQ	36.5	34
Cs[d]	GEA/TIMS	NMRQ	508	98.9	Zn	ICP-AES	NMRQ	[13]	[>20]
^{134}Cs µCi/mL	GEA	NMRQ	8.30E+0	90.4	TOC	Hot Pers.	1500	<2500	<1.4
^{137}Cs µCi/mL	GEA	1.00E-02	1.45E+4	98.9	TOC	Furnace	1500	6,300[e]	6.2
Cu	ICP-AES	NMRQ	[13]	[>40]	Cl^-	IC	10	75	>7
Fe	ICP-AES	NMRQ	[12]	[>40]	F^-	IC	NMRQ	42	0.33
K	ICP-AES	75	<210	<0.4	NO_3^-	IC	3000	29,200	[f]
La	ICP-AES	NMRQ	<5	NM	PO_4^{-3}	IC	NMRQ	<26	<0.3
Mg	ICP-AES	NMRQ	<10	NM	SO_4^{-2}	IC	NMRQ	250	0.10
Mn	ICP-AES	NMRQ	<5	NM	$C_2O_4^{-2}$	IC	NMRQ	200	1.63
Mo	ICP-AES	NMRQ	<5	<2	**Solution Density**		**MRQ**	**g/mL**	
Na	ICP-AES	75	1220	0.16	Density		NMRQ	1.010 g/mL, T= 25°C	

Table 3.4 (cont'd)

Analyte	Method	MRQ[(a)] µg/mL	Cs eluate[(b)] µg/mL	% Recovered	Analyte	Method	MRQ µg/mL	Cs eluate[(b)] µg/mL	% Recovered

(a) MRQ is minimum reportable quantity.
(b) The overall error is estimated to be within ±15%. Values in brackets are within 10 times the detection limit, and errors are likely to exceed ±15%. Less-than values indicate that the analyte was not detected by the instrument, and the reported value represents the IDL multiplied by the sample dilution factor.
(c) NMRQ is no minimum reportable quantity requested. Analytes are reported opportunistically.
(d) The total Cs concentration was calculated based on the ^{137}Cs concentration and the isotopic distribution ratio.
(e) The TOC result was within 4X the MDL and therefore had high uncertainty.
(f) The eluate matrix is 0.5 M HNO_3.
NM = analyte not detected in the feed and/or eluate.
Analytical details are provided with Analytical Service Request (ASR) 6374, sample ID 02-1698.

The major anion detected was NO_3^-, which was not surprising since the eluant was 0.5 M HNO_3. However, in addition to NO_3^-, anions of SO_4^{2-}, $C_2O_4^{2-}$, Cl^-, and F^- were also detected. The molar concentrations of F^- and SO_4^{2-} are equivalent in the eluate, but the molar concentration of F^- is almost a factor of four lower than SO_4^{2-} in both the feed (Table 2.3) and the Cs-decontaminated effluent (Table 3.3). Therefore, it seems likely that the F^- and SO_4^{2-} were from continued precipitation of Na_3FSO_4, a major solid phase formed during the AZ-102 concentration process (Fiskum et al. 2002b). Similarly, the $C_2O_4^{2-}$ may be from continued precipitation of $Na_2C_2O_4$, the second most abundant solid phase from AZ-102 concentration. Alternatively, some residual feed ion pairs may have remained on the resin bed then eluted with the 0.5 M HNO_3. The presence of Cl cannot be explained.

The minimum reportable quantity (MRQ) is provided in Table 3.4 for information along with the actual analytical result. In cases where a result was below the instrument detection limit (IDL), then the dilution-corrected IDL is provided as a "less-than" value. In most cases, the analyte concentrations were higher than the requested MRQs. The MRQ was not met in only two cases (K and TOC). Because of the high radiation sample dose, large dilutions were required to remove subsamples from the hot cell to the analytical workstation. The TIC analysis was not completed because carbonate is known to evolve as CO_2 in acidic solutions. The total and free hydroxide analysis was not completed because the eluate was acidic.

Table 3.5. Select Analyte Recoveries in Eluate

Analyte	AZ-102C feed, total mg	Amount recovered in eluate, mg	Recovery in Eluate, %	Recovery in eluate, total mmoles
U	16.2	5.53	34.0	0.0232
Cd	<2	[1.3]	[>60]	[0.011]
Cr	1,620	11.3	0.70	0.22
Cu	<4.3	[2.0]	[>40]	[0.031]
Fe	<4.3	[1.8]	[>40]	[0.032]
Na	114,000	185	0.16	8.07[(a)]
Ni	<4.3	14.4	>300	0.24
Pb	<14	[5.4]	[>40]	[0.026]
Zn	<7.5	[2.0]	[>20]	[0.030]

Table 3.5 (cont'd)

Analyte	AZ-102C feed, total mg	Amount recovered in eluate, mg	Recovery in Eluate, %	Recovery in eluate, total mmoles
(a) An estimated 1.8 mmoles of Na were anticipated to be associated with the measured F^-, SO_4^{2-}, and $C_2O_4^{2-}$ as Na_3FSO_4 and $Na_2C_2O_4$. The mmoles Na exchanged onto the column can be estimated by difference (8.1 - 1.8 = 6.3). Values in brackets are based on results reported within 10 times the detection limit, and errors are likely to exceed ±15%.				

3.3.3 Regeneration

Table 3.6 shows the composition of the regeneration solution. Sodium was the only cation found above the blank concentration; however, its concentration was far smaller than expected. The regeneration solution was found to still be slightly acidic; therefore, the OH^- concentration could not be determined. The volume of regeneration solution processed was 2.0 AVs, which represented 4.4 BVs. A volume larger than 2.0 AVs (4.4 BVs) 0.25 M NaOH will be required to neutralize the resin bed and system apparatus.

Table 3.6. Composition of Regeneration Solution

Analyte	Concentration, µg/mL	Concentration, M
Na^+	[10]	[4.3 E-4]
K^+	< 20	< 5 E-4
B	[0.72]	[6.7E-5]
Cr	[0.22]	[4.2E-6]
OH^-	NM	NM
^{137}Cs	1.64E+0 µCi/mL	NA
Total cesium	5.74E-2	4.3E-7
NM: the composite solution was slightly acidic, pH = 4. NA = not applicable Results in brackets indicate that the analyte concentration was less than the estimated quantitation limit but greater than the detection limit. Less-than values are less than the instrument detection limit corrected for sample dilution.		

The total mmoles of Na exchanged onto the resin was calculated to be 5.5 mmoles per 2.46 g dry Na-form resin according to the Equation 3.2. This result is lower than the calculated 6.3 mmoles Na (8.1 mmoles total Na minus 1.8 mmoles Na associated with the precipitates) obtained during elution of the ion exchange column (see Table 3.5). This indicated the resin was ~90% (5.5/6.3) converted to the Na form.

$$(V - V_H) * C - (C_E * V_E) \tag{3.2}$$

where: V = volume of 0.25 M NaOH processed, 45 mL
V_H = holdup volume (volume of 0.25 M NaOH remaining in system), 23 mL

C = NaOH concentration, 0.25 M
C_E = Na concentration in regeneration effluent, [4.3E-4]M
V_E = regeneration effluent volume, 45 mL

The current design basis utilizes spent regeneration solution as feed displacement for follow-on processing. For this to be effective, the spent regeneration solution must be caustic. The acidic nature of the composite regeneration solution precludes this follow-on use. The acidity may be a result of mixing of rinse solutions in the system apparatus, reducing the efficiency of the rinse process. In this column system, the largest mixing areas were both above and below the resin beds. Regeneration effluent acidity may be reduced by minimizing all mixing areas in the column apparatus, utilizing a larger DI water wash volume after elution, and using a larger regeneration solution volume.

3.3.4 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 feed sample (Table 3.7). A 1 to 5% fraction of the Cs broke through the lead column to the lag column. As expected, virtually all ^{137}Cs loaded on the lead column was found in the eluate stream, recovering 98.9% of the ^{137}Cs present in the initial AZ-102C feed. The total Cs recovery, calculated from summation of all process streams, was 104%. The high bias is largely attributed to analytical errors associated with the large dilutions performed remotely in the hot cells required for analysis.

Table 3.7. Activity Balance for ^{137}Cs

Solution	^{137}Cs, µCi	^{137}Cs Relative to Feed Sample, %
Initial loading		
Feed Sample (AZ-102C)	2.22 E6	100
Initial AN-102/C-104 loading	2.22 E-4	1E-8
Process streams		
AZ-102C Effluent	1.7E+1	8.1E-4
Load samples (lead and lag columns)	1.0E+2	4.5E-3
Feed displacement	2.0E+0	9.0E-5
DI Water Rinse	5.0E-1	2.3E-5
Column #1 Eluate	2.19E+6	9.89E+1[(a)]
Column #1 DI water rinse	2.3E+1	1.0E-3
Column #1 regeneration	7.4E+1	3.3E-3
Column #1 Eluate (repeat)	9.2E+1	4.1E-3
Column #1 DI water rinse	4.0E+0	1.8E-4
Column #1 regeneration	2.8E+0	1.3E-4
Lag column Cs loading	1.2E+5	5.4E+0[(b)]
Total ^{137}Cs Recovery	**2.43E+6**	**1.04E+2**

(a) Integration of the lead column eluate sample results indicated the eluate recovery was 94.6%.
(b) Integration of the lead column load samples indicated breakthrough was at 10%; integration of the lead column eluate samples resulted in 94.6 % recovery indicating the breakthrough was 5.4%, the composite eluate recovery of 98.9% indicated the breakthrough was 1.1%. The median value is reported.

3.3.5 SL-644 Resin Volume Changes

The SL-644 resin is known to change in volume as a function of the solution pH and ionic strength (Hassan, King, and McCabe 1999). The resin BV change history is shown in Table 3.8. The columns are labeled 1 and 2. Column 1 was the lead column for the AW-101 simulant test and the AP-101DF test; Column 2 was the lag column for these tests. Results from both tests have been previously reported (Fiskum, Blanchard, Arm 2002a; Fiskum et al. 2002a). These columns were switched for the AN-102/ C-104 ion exchange test. Thus Column 1 was placed in the lag position, and Column 2 was placed in the lead position (Fiskum, Blanchard, and Arm 2002b). The column positions were switched again for the AZ-102C ion exchange test, where Column 1 was placed back into the lead position and Column 2 back to the lag position.

The variation in BV as a function of the process steps for both columns is shown in Figure 3.6 and Figure 3.7. In Figure 3.6, the BVs are normalized to the volume in the 0.25 M NaOH regeneration condition just before AZ-102C loading. Each process step is denoted with a number corresponding to the number in Table 3.8. An anomalous behavior was observed during the load phase of AZ-102C (Step 32). The lead column resin bed expanded after loading 508 mL AZ-102C in a 3-h time frame from 9.5 mL to 10.4 mL.

Table 3.8. SL-644 BVs

Feed	Symbol	Process Step	010319SMC-IV-73 212- to 425-μm particle size	
			Column 1	Column 2
Initial column positions			**Lead column, mL**	**Lag column, mL**
Initial packing	P	1	10.9	10.9
0.5 M HNO_3	E	2	9.2	8.9
DI water	W	3	9.2	8.9
0.25 M NaOH	R	4	11.2	10.8
AW-101 simulant	F	5	10.2	10.0
0.1 M NaOH	FD	6	10.9	10.7
DI water	W	7	10.9	10.9
0.5 M HNO_3	E	8	8.9	8.9
DI water	W	9	8.9	8.7
Re-fluidize bed	RP	10	7.5	7.5
0.25 M NaOH	R	11	11.0	10.9
DI water	W	12	10.5	11.0
0.5 M HNO_3	E	13	7.9	7.7
DI water	W	14	7.9	7.7
0.25 M NaOH	R	15	10.7	10.5
AP-101DF	F	16	9.7	9.7
0.1 M NaOH	FD	17	10.5	10.4
DI water	W	18	10.4	10.5
0.5 M HNO_3	E	19	7.7	—
DI water	W	20	7.7	—
0.25 M NaOH	R	21	10.5	—
DI water	W	22	10.2	—
Switch column positions			**Lag column, mL**	**Lead column, mL**
0.25 M NaOH	R	23	10.5	10.4

Table 3.8 (cont'd)

Feed	Symbol	Process Step	010319SMC-IV-73 212- to 425-µm particle size	
			Column 1	Column 2
AN-102/C-104	F	24	9.9	9.9
0.1 M NaOH	FD	25	10.7	10.4
DI water	W	26	10.7	10.4
0.5 M HNO_3	E	27	—	7.7
DI water	W	28	—	7.5
0.25 M NaOH	R	29	—	10.2
DI water	W	30	—	10.4
Switch column positions			**Lead column, mL**	**Lag column, mL**
0.25 M NaOH	R	31	10.2	10.0
AZ-102C	F	32	9.5 (jumped to 10.4)	9.4
0.1 M NaOH	FD	33	10.7	10.0
DI water	W	34	10.9	10.3
0.5 M HNO_3	E	35	8.1	—
DI water	W	36	8.2	—
0.25 M NaOH	R	37	10.4	—
DI water	W	38	10.7	—
0.5 M HNO_3	E	39	7.9	—
DI water	W	40	7.8	—
0.25 M NaOH	R	41	10.5	—
DI water	W	42	10.7	—

In Figure 3.7, the observed volume changes show clearly the resin bed history with respect to expansion and contraction observed with repeated cycling. The volume contraction after the initial 0.5 M HNO_3 conditioning and AW-101 processing became more pronounced with cycling. The first volume contraction stabilized at 9.2 mL; subsequent volume contractions stabilized at around 7.8 mL. After initial cycling, the resin beds appeared to remain fairly consistent with respect to swelling and shrinking. The AZ-102C processing distinctly showed continued resin bed expansion from the feed displacement to the DI water rinse (Steps 33 and 34), whereas these volumes in other cycles remained essentially constant.

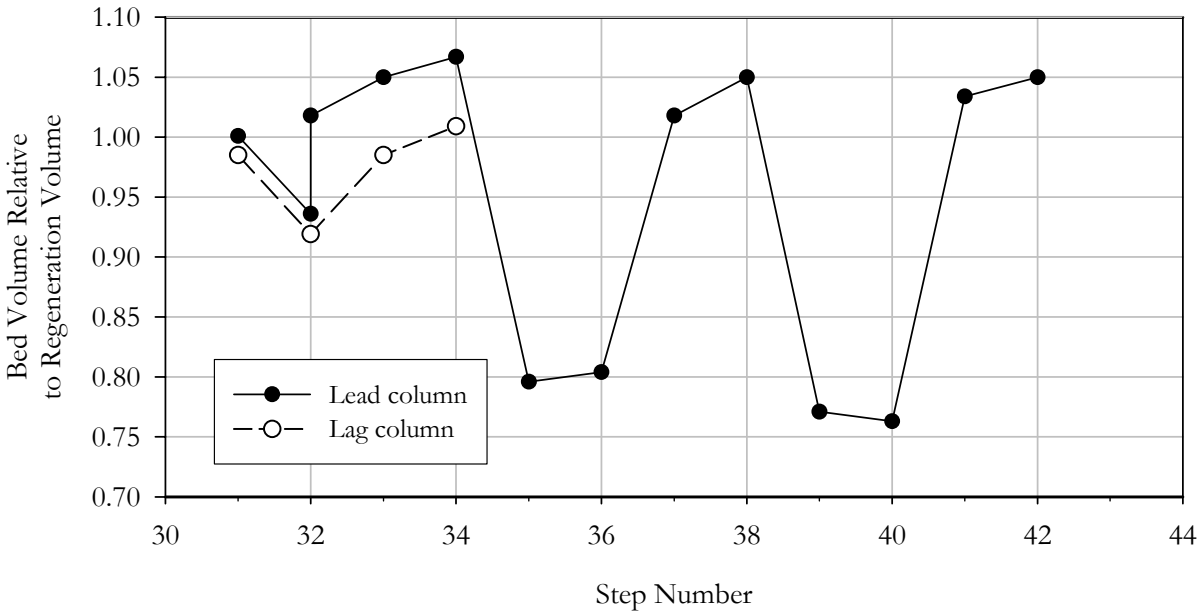


Figure 3.6. Relative BVs For AZ-102C Processing
(relative to regeneration condition, 10.2 mL)

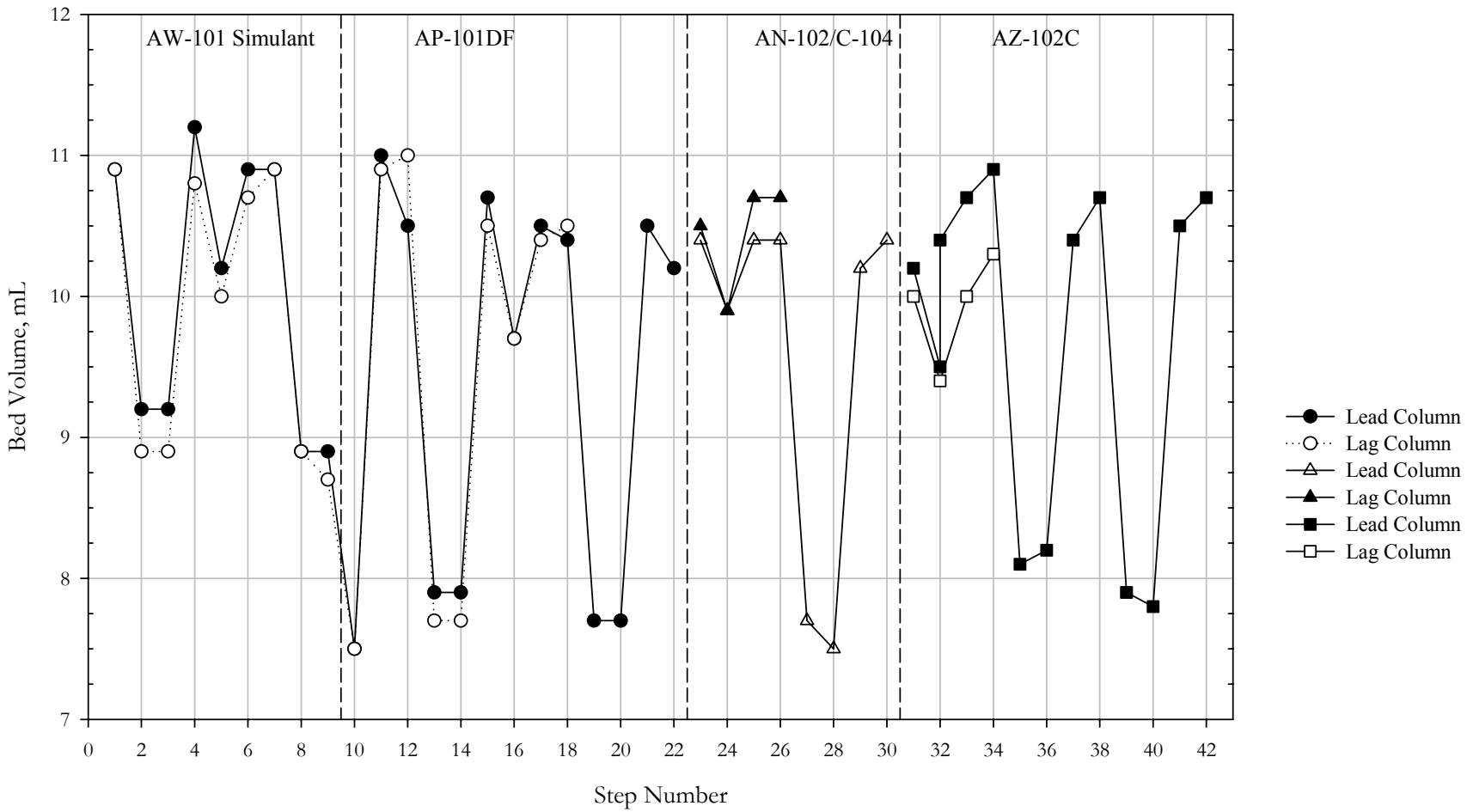


Figure 3.7. Comparison of BVs of the Lead and Lag Columns for all Test Cycles

(SL-644 Batch 010319SMC-IV-73, 212- to 425-µm; solid points indicate Column 1, hollow points indicate Column 2)

4.0 Conclusions

Good column performance of Envelope B tank waste sample AZ-102C was demonstrated. Decontamination of ^{137}Cs from AZ-102C was demonstrated and a Cs-decontaminated sample was provided for downstream process testing.

- An overall DF of 1.05E+5 was obtained providing a Cs-decontaminated effluent with a ^{137}Cs concentration of 1.92E-2 μCi/mL. This is 5.9% of the contract limit of 3.22E-1 μCi/mL in the treated effluent (based on minimum 5.0 wt% waste Na_2O loading) and 6.6% of the contract limit 2.92E-1 μCi/mL (based on maximum 5.5 wt% waste Na_2O loading).
- The AZ-102C effluent was forwarded for down-stream process testing (i.e., batch-contact studies for ^{99}Tc removal and ^{99}Tc measurements using a prototype Tc monitor).

Cs load and elution profiles were developed.

- The AZ-102C feed loading continued through 79% Cs breakthrough on the lead column. The 50% Cs breakthrough point was interpolated to be 93 BVs from the lead column breakthrough profile. No Cs breakthrough was observed from the lag column.
- The Cs-loaded lead column was efficiently eluted with 0.5 M HNO_3. The majority of the ^{137}Cs was eluted from the column in 2.5 BVs of eluate (2.5 to 5.0 BV fraction), although 8 BVs of eluant were required to reach the elution end point of a $C/C_o = 0.01$. The peak ^{137}Cs C/C_o value was 71 (lead column). Nearly 99% of the ^{137}Cs present in the feed sample was recovered in the composite eluate fraction.

The effectiveness of SL-644 ion exchange process steps was demonstrated, including loading, feed displacement, DI water washing, and elution; the resin regeneration may not have been sufficient.

- An activity balance for ^{137}Cs indicated that 104% 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 regeneration solution effluent was slightly acidic; however, the calculated Na uptake appeared to be adequate.

Distribution coefficient (K_d) values were determined as a function of Na/Cs mole ratio for SL-644 in the AZ-102C matrix.

- The batch-contact equilibrium data resulted a feed condition K_d of 160 mL/g, corresponding to a predicted Cs λ of 38 BVs (0.25 M NaOH condition) at a Na/Cs mole ratio of 8.84 E+3, 25 to 26°C.
- Performance degradation was demonstrated on the dry H-form resin, stored for nominally 1 year, where a 20% drop was observed in the AW-101 simulant feed condition K_d.

5.0 References

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Appendix A

General Calculations

Appendix A: General Calculations

^{137}Cs Concentration Contractual Limit in AZ-102 Concentrate Envelope B Vitrification Feed

Assumptions, Minimum Waste Loading

1) Concentration of Na_2O in Env. B glass = 5% (= 5 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 Vit feed, multiply the maximum ^{137}Cs value determined below by the ratio of total Na:feed Na.
3) Glass density = 2.66 MT/m^3 (=2.66 g/mL)
4) Maximum ^{137}Cs in glass = 0.3 Ci/m^3 (=0.3 Ci/1E+6 mL = 3E-7 Ci/mL)
5) AZ-102C (C = concentrate) actual waste Na concentration = 4.61 M
6) AZ-102 actual waste ^{137}Cs concentration = 2.005E3 μCi/mL/4.61 M Na

Na Loading in Glass

5 g Na_2O/100g glass * 1 mole Na_2O/62 g Na_2O) * (2 mole Na/ mole Na_2O)*
(23 g Na/mole Na) * (2.66 g glass/mL glass) = 0.0987 g Na/mL glass

Maximum ^{137}Cs:Na in glass

(3.0E-7 Ci ^{137}Cs/mL glass)/(0.0987 g Na/mL glass) = 3.04 E-6 Ci ^{137}Cs/g Na

(3.04 E-6 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 6.99E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in feed

(6.99E-5 Ci ^{137}Cs/mole Na) * (4.61 mole Na/L feed) = 3.22 E-4 Ci ^{137}Cs/L
= 3.22 E+2 μCi ^{137}Cs/L
= 0.322 μCi ^{137}Cs/mL

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

(0.322 μCi ^{137}Cs/ mL)/(2.005E3 μCi ^{137}Cs/mL) = 1.61 E-4 C/C_o
= 0.0161 % C/C_o

DF = C_o/C

(2.005E3 μCi ^{137}Cs/mL)/(0.321 μCi ^{137}Cs/mL) = 6250

^{137}Cs Concentration Contractual Limit in AZ-102 Concentrate Envelope B Vitrification Feed

Assumptions, Maximum Waste Loading

1) Concentration of Na_2O in Env. B glass = 5.5% (= 5.5 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 Vit feed, multiply the maximum ^{137}Cs value determined below by the ratio of total Na:feed Na.
3) Glass density = 2.66 MT/m^3 (=2.66 g/mL)
4) Maximum ^{137}Cs in glass = 0.3 Ci/m^3 (=0.3 Ci/1E+6 mL = 3E-7 Ci/mL)
5) AZ-102C (C = concentrate) actual waste Na concentration = 4.61 M
6) AZ-102 actual waste ^{137}Cs concentration = 2.005E3 μCi/mL/4.61 M Na

Na Loading in Glass

5.5 g Na_2O/100g glass * 1 mole Na_2O/62 g Na_2O) * (2 mole Na/ mole Na_2O)*
(23 g Na/mole Na) * (2.66 g glass/mL glass) = 0.109 g Na/mL glass

Maximum ^{137}Cs:Na in glass

(3.0E-7 Ci ^{137}Cs/mL glass)/(0.109 g Na/mL glass) = 2.75 E-6 Ci ^{137}Cs/g Na

(2.75 E-6 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 6.33E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in feed

(6.33E-5 Ci ^{137}Cs/mole Na) * (4.61 mole Na/L feed) = 2.92 E-4 Ci ^{137}Cs/L
= 2.92 E+2 μCi ^{137}Cs/L
= 0.292 μCi ^{137}Cs/mL

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

(0.292 μCi ^{137}Cs/ mL)/(2.005E3 μCi ^{137}Cs/mL) = 1.46 E-4 C/C_o
= 0.0146% C/C_o

DF = C_o/C

(2.005E3 μCi ^{137}Cs/mL)/(0.292 μCi ^{137}Cs/mL) = 6866

Appendix B

Batch-Contact Calculations

Appendix B: Batch-Contact Calculations

TI-PNNL-WTP-164, Rev. 0

Batch Contact of AW-101Simulant with SL-644

	m	**F**	**I_{Na}**			**V**			**C_o & C_{eq}**	**Average C_o**						
Sample ID	H-form resin mass, g	F-factor (water loss)	Na form mass gain factor	Corrected resin mass, g	Net simulant mass, g	Simulant volume*, mL	Analytical sample, g	Analytical sample, mL	cpm/mL	average comp., cpm/mL	**K_d**	**K_d'**	Fraction Cs remaining	Eq. Cs conc., µg/mL**	Eq. Cs conc., M**	Na/Cs mole ratio
S-TI164-S0	none	1	1	0	12.3703	9.9760	6.2345	5.0278	140.5	140.4	NA	NA	1.0	4.26	3.20E-5	1.56E+5
S-TI164-S0-D	none	1	1	0	12.367	9.9734	6.2298	5.0240	140.3							
S-TI164-S0-73	0.104	0.7619	1.25	0.0991	12.3604	9.9681	6.2025	5.0020	9.9	NA	1650	1320	0.071	0.30	2.27E-6	2.20E+6
S-TI164-S0-73-D	0.1023	0.7619	1.25	0.0974	12.3714	9.9769	6.1891	4.9912	10.2	NA	1638	1310	0.072	0.31	2.32E-6	2.15E+6
S-TI164-S1	none	1	1	0	12.3753	9.9801	6.237	5.0298	144.0	144.1	NA	NA	1.0	137	1.03E-3	4.86E+3
S-TI164-S1-D	none	1	1	0	12.3778	9.9821	6.2497	5.0401	144.3							
S-TI164-S1-73	0.1048	0.7619	1.29	0.1030	12.346	9.9565	6.2554	5.0447	23.7	NA	634	491	0.164	22.5	1.69E-4	2.95E+4
S-TI164-S1-73-D	0.1044	0.7619	1.29	0.1026	12.3734	9.9785	6.2516	5.0416	23.8	NA	634	491	0.170	23.2	1.75E-4	2.86E+4
S-TI164-S2	none	1	1	0	12.3976	9.9981	6.2408	5.0329	142.0	142.5	NA	NA	1.0	661	4.97E-3	1.01E+3
S-TI164-S2-D	none	1	1	0	12.3561	9.9646	6.2348	5.0281	142.9							
S-TI164-S2-73	0.1043	0.7619	1.25	0.0993	12.3659	9.9725	6.2193	5.0156	61.3	NA	166	133	0.431	284	2.14E-3	2.34E+3
S-TI164-S2-73-D	0.1039	0.7619	1.25	0.0990	12.3586	9.9666	6.2294	5.0237	59.1	NA	178	142	0.415	274	2.06E-3	2.42E+3

*AW101 Simulant volume was determined based on mass delivered and the solution density of 1.24 g/mL. The slight dilution attributed to spiking was considered inconsequential.

**Equilibrium Cs concentration is based on the ^{137}Cs tracer recovery.

Na concentration was prepared to 5 M.

NA = not applicable

$K_d = (C_o - C_{eq})/C_{eq} \times V/(m*F)$

$K_d' = (C_o - C_{eq})/C_{eq} \times V/(m*F*I_{Na})$

where m is mass of SL-644 in H-form

F = F-factor for water loss

and I_{Na} = Na form mass gain factor from H-form to Na form.

Batch Contact of AZ-102 Concentrate Tank Waste with SuperLig 644

AZ-102C feed density 1.246 g/mL

	m	**F**	**I_{Na}**			**V**	**C_o**					**C_{eq}**				
Sample ID	H-form SL-644 Resin, g	F-factor	I_{Na} factor	corrected resin mass, g	AZ-102C, g	AZ-102C, mL	Starting [Cs], M	[Na], M	CMC ID	Net Cs-137 µCi/mL	Fraction of original Cs-137	Equilibrium [Cs], M	Equilibrium Na:Cs mole ratio	K_d, mL/g	Na-form dry bed density*	λ, BVs
AZ102-TI-164-S0	none	1	1	0	3.6982	2.9681	5.21E-4	4.61	02--2300	2.18E+3	1.0	5.21E-4	8.84E+3	NA	NA	NA
AZ102-TI-164-S0-D	none	1	1	0	3.6908	2.9621	5.21E-4	4.61	02--2301	2.25E+3	1.0	5.21E-4	8.84E+3	NA	NA	NA
AZ102-TI-164-S0-73	0.0735	0.7619	1.25	0.0700	8.7749	7.0425	5.21E-4	4.61	02--2302	7.35E+2	3.32E-1	1.73E-4	2.66E+4	203	0.24	49
AZ102-TI-164-S0-73D	0.0703	0.7619	1.25	0.0670	8.7887	7.0535	5.21E-4	4.61	02--2303	6.77E+2	3.06E-1	1.59E-4	2.89E+4	239	0.24	57
AZ102-TI-164-S1	none	1	1	0	3.6967	2.9669	4.58E-3	4.61	02--2304	2.36E+3	1.0	4.58E-3	1.01E+3	NA	NA	NA
AZ102-TI-164-S1-D	none	1	1	0	3.3406	2.6811	4.58E-3	4.61	02--2305	2.34E+3	1.0	4.58E-3	1.01E+3	NA	NA	NA
AZ102-TI-164-S1-73	0.0723	0.7619	1.25	0.0689	8.764	7.0337	4.58E-3	4.61	02--2306	1.48E+3	6.30E-1	2.88E-3	1.60E+3	60	0.24	14
AZ102-TI-164-S1-73D	0.0727	0.7619	1.25	0.0692	8.7684	7.0372	4.58E-3	4.61	02--2307	1.42E+3	6.04E-1	2.77E-3	1.67E+3	67	0.24	16
AZ102-TI-164-S2	none	1	1	0	3.7031	2.9720	7.38E-3	4.61	02--2308	2.32E+3	1.0	7.38E-3	6.25E+2	NA	NA	NA
AZ102-TI-164-S2-D	none	1	1	0	3.0783	2.4705	7.38E-3	4.61	02--2309	2.28E+3	1.0	7.38E-3	6.25E+2	NA	NA	NA
AZ102-TI-164-S2-73	0.0745	0.7619	1.25	0.0710	8.7856	7.0510	7.38E-3	4.61	02--2310	1.53E+3	6.65E-1	4.91E-3	9.39E+2	50	0.24	12
AZ102-TI-164-S2-73D	0.0735	0.7619	1.25	0.0700	8.749	7.0217	7.38E-3	4.61	02--2311	1.60E+3	6.96E-1	5.14E-3	8.98E+2	44	0.24	10

*dry-bed density in the 0.25 M NaOH matrix

NA = not analyzed

$$K_d = (C_o - C_{eq})/C_{eq} \times V/(m*F*I_{Na})$$

Average Cs-137 2.22E+3Cs-137 µCi/mL S0

Average Cs-137 2.35E+3Cs-137 µCi/mL S1

Average Cs-137 2.30E+3Cs-137 µCi/mL S2

Appendix C

Column-Testing Calculations

Appendix C: Column-Testing Calculations

AZ-102C Test Run

SL-644 batch 010310SMC-IV-73 212- to 425-µm particle size

Bed volume in 0.25M NaOH 10.2 mL

	Densities	
HNO3	1.012	g/mL
0.1M NaOH	0.999	
0.25M NaOH	1.006	
AZ-102C	1.2462	

	apparatus volume	
total system	42	mL
column 1	23	
column 2	21	

AN-102/C-014 finish date

7/10/2001 118.62 days system was idled

Conditioning 11/5/01

Lead column + Lag column

start	end	Δt	g mass	mL volume	mL/min flow rate	BV/hr	BV	AV
0.25M NaOH wash								
11/5/2001 14:57	11/5/2001 18:36	3:39	91.7	91.2	0.42	2.45	8.9	2.17

AZ-102C actual waste loading starting on 11/5/01

Initial effluent collection

1 apparatus volume of feed

start	end	Δt	mass	cumul. volume	flow rate	BV/hr	apparatus volume
11/5/2001 18:46	11/5/2001 21:40	2:54	41.2	41.0	0.235	1.38	1.0

Feed flow

start	end	Δt	mass	cumul. volume	flow rate	BV/hr	cumulat. BV	Sample ID	Lead column sampling Vial tare	Vial + smpl	mass	volume	total vol.	**BV**	Running total sample vol
11/5/2001 21:40	11/5/2001 22:00	0:20	3.9	44.1	0.156	0.92	4.3	AZ102L-F1	17.1337	19.3829	2.2492	1.805	45.889	**4.5**	1.8048
11/5/2001 22:17	11/5/2001 23:42	1:25	20.7	60.7	0.195	1.15	6.0	AZ102L-F2	17.1230	19.8664	2.7434	2.201	66.688	**6.5**	4.0063
11/6/2001 1:03	11/6/2001 6:17	5:14	99.3	140.4	0.254	1.49	13.8	AZ102L-F3	17.1545	19.6606	2.5061	2.011	151.753	**14.9**	6.0173
6:32	8:40	2:08	33.7	167.4	0.211	1.24	16.4	AZ102L-F4	17.1589	19.7290	2.5701	2.062	182.889	**17.9**	8.0796
8:53	11:00	2:07	37.1	197.2	0.234	1.38	19.3	AZ102L-F5	17.2274	19.6733	2.4459	1.963	216.341	**21.2**	10.0423
11:18	14:20	3:02	51.0	238.1	0.225	1.32	23.3	AZ102L-F6	17.1636	19.6237	2.4601	1.974	260.653	**25.6**	12.0164
15:43	17:01	1:18	25.1	258.3	0.258	1.52	25.3	AZ102L-F7	17.2084	20.0611	2.8527	2.289	285.745	**28.0**	14.3055
17:22	20:27	3:05	38.2	288.9	0.166	0.97	28.3	AZ102L-F8	17.0871	20.8494	3.7623	3.019	322.016	**31.6**	17.3245
11/6/2001 21:46	11/7/2001 1:07	3:21	52.6	331.1	0.210	1.24	32.5	AZ102L-F9	17.1750	23.4078	6.2328	5.001	374.291	**36.7**	22.3260
11/7/2001 1:28	11/7/2001 4:20	2:52	49.8	371.1	0.232	1.37	36.4	AZ102L-F10	17.1872	19.1315	1.9443	1.560	419.186	**41.1**	23.8861
4:43	7:40	2:57	48.3	409.8	0.219	1.29	40.2	AZ102L-F11	17.3173	19.9686	2.6513	2.128	463.072	**45.4**	26.0136
8:02	11:00	2:58	50.3	450.2	0.227	1.33	44.1	AZ102L-F12	17.2127	19.8050	2.5923	2.080	507.971	**49.8**	28.0938
11:26	14:20	2:54	49.9	490.2	0.230	1.35	48.1	AZ102L-F13	17.2647	19.7327	2.4680	1.980	553.200	**54.2**	30.0742
14:42	17:40	2:58	52.8	532.6	0.238	1.40	52.2	AZ102L-F14	17.1680	19.1680	2.0000	1.605	599.608	**58.8**	31.6791
18:01	21:00	2:59	50.4	573.1	0.226	1.33	56.2	AZ102L-F15	17.1600	19.3483	2.1883	1.756	644.193	**63.2**	33.4351
11/7/2001 21:20	11/8/2001 0:21	3:01	50.8	613.8	0.225	1.32	60.2	AZ102L-F16	17.1604	20.0594	2.8990	2.326	690.506	**67.7**	35.7614
11/8/2001 1:51	11/8/2001 3:41	1:50	32.0	639.5	0.233	1.37	62.7	AZ102L-F17	17.1384	19.5853	2.4469	1.963	721.783	**70.8**	37.7248
4:15	6:58	2:43	48.3	678.3	0.238	1.40	66.5	AZ102L-F18	17.2588	19.9166	2.6578	2.133	765.463	**75.0**	39.8576
7:22	10:20	2:58	55.4	722.7	0.250	1.47	70.9	AZ102L-F19	17.3043	20.1190	2.8147	2.259	814.697	**79.9**	42.1162
10:43	13:45	3:02	59.6	770.5	0.263	1.55	75.5	AZ102L-F20	17.2551	20.3027	3.0476	2.446	867.724	**85.1**	44.5617
14:05	17:00	2:55	57.4	816.6	0.263	1.55	80.1	AZ102L-F21	17.1775	19.9230	2.7455	2.203	918.495	**90.0**	46.7648
17:20	20:20	3:00	58.7	863.7	0.262	1.54	84.7	AZ102L-F22	17.0484	19.3844	2.3360	1.874	969.550	**95.1**	48.6393
11/8/2001 20:38	11/8/2001 23:40	3:02	57.4	909.8	0.253	1.49	89.2	AZ102L-F23	17.0902	20.1563	3.0661	2.460	1019.931	**100.0**	51.0997
11/9/2001 0:03	11/9/2001 3:00	2:57	58.0	956.3	0.263	1.55	93.8	AZ102L-F24	17.1432	19.3116	2.1684	1.740	1071.192	**105.0**	52.8397
ΔT, total	80:14:00			**average**	**0.234**	**1.37**									

Lag column sampling

Date	Sample ID	start	end	Vial tare	Vial + sample	sample mass	volume	total vol.	**BV**
11/5/2001	AZ-102P-F1	22:10	22:15	17.0901	19.5669	2.477	1.987	46.07	**4.52**
	AZ-102P-F2	23:53	0:01	17.2804	20.6728	3.392	3.372	66.05	**6.48**
11/6/2001	AZ-102P-F3	6:27	6:32	17.2270	19.7576	2.531	2.031	147.77	**14.49**
	AZ-102P-F4	8:48	8:53	17.1340	19.2764	2.142	1.719	176.53	**17.31**
	AZ-102P-F5	11:08	11:18	17.1821	18.9437	1.762	1.414	207.71	**20.36**
	AZ-102P-F6	14:29	15:41	17.2051	20.5224	3.317	2.662	251.30	**24.64**
	AZ-102P-F7	17:12	17:21	17.1880	20.4258	3.238	2.598	274.04	**26.87**
	AZ-102P-F8	21:36	21:46	17.2298	23.5426	6.313	5.066	309.76	**30.37**
11/7/2001	AZ-102P-F9	1:20	1:28	17.2697	21.4740	4.204	3.374	355.34	**34.84**
	AZ-102P-F10	4:26	4:43	17.2258	20.9653	3.740	3.001	398.30	**39.05**
	AZ-102P-F11	7:50	8:00	17.3229	20.3830	3.060	2.456	439.51	**43.09**
	AZ-102P-F12	11:13	11:26	17.2472	21.2433	3.996	3.207	483.08	**47.36**
	AZ-102P-F13	14:29	14:38	17.3132	20.3468	3.034	2.434	525.56	**51.53**
	AZ-102P-F14	17:50	18:00	17.3260	20.3006	2.975	2.387	570.32	**55.91**
	AZ-102P-F15	21:08	21:19	17.1173	21.1335	4.016	3.223	613.98	**60.19**
11/8/2001	AZ-102P-F16	1:41	1:51	17.2322	21.7617	4.530	3.635	658.38	**64.55**
	AZ-102P-F17	4:05	4:15	17.1648	20.6418	3.477	2.790	686.85	**67.34**
	AZ-102P-F18	7:13	7:21	17.1201	20.2604	3.140	2.520	728.13	**71.38**
	AZ-102P-F19	10:31	10:42	17.2389	20.6738	3.435	2.756	775.34	**76.01**
	AZ-102P-F20	13:57	14:05	17.3165	20.4420	3.126	2.508	825.67	**80.95**
	AZ-102P-F21	17:11	17:19	17.0888	19.6770	2.588	2.077	873.81	**85.67**
	AZ-102P-F22	20:28	20:36	17.0358	19.3541	2.318	1.860	922.77	**90.47**
	AZ-102P-F23	23:51	0:01	17.0639	20.7771	3.713	2.980	971.81	**95.28**
11/9/2001	AZ-102P-F24	3:15	3:26	17.1059	21.8949	4.789	3.843	1022.19	**100.22**

0.1M NaOH feed displacement

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV	C/Co	Sample density
AZ102-FD-1	3:36	3:59	0:23	17.1770	29.5637	12.39	9.94	0.43	2.54	0.97	0.97	0.24	0.24	7.91E-6	1.246
AZ102-FD-2	3:59	4:21	0:22	17.0785	28.5703	11.49	9.22	0.42	2.47	0.90	1.88	0.22	0.46	7.93E-6	1.246
AZ102-FD-3	4:21	4:42	0:21	17.2852	28.1550	10.87	8.72	0.42	2.44	0.86	2.73	0.21	0.66	8.96E-6	1.246
AZ102-FD-4	4:42	5:04	0:22	17.2425	28.0201	10.78	8.65	0.39	2.31	0.85	3.58	0.21	0.87	8.24E-6	1.246
AZ102-FD-5	5:04	5:30	0:26	17.1590	28.4221	11.26	9.54	0.37	2.16	0.94	4.52	0.23	1.10	9.82E-6	1.18
AZ102-FD-6	5:30	5:51	0:21	17.2066	28.3515	11.14	10.60	0.50	2.97	1.04	5.56	0.25	1.35	2.00E-5	1.05
AZ102-FD-7	5:51	6:13	0:22	17.0996	28.5422	11.44	11.68	0.53	3.12	1.15	6.70	0.28	1.63	1.13E-5	0.98
AZ102-FD-8	6:13	6:36	0:23	17.1557	27.4960	10.34	10.68	0.46	2.73	1.05	7.75	0.25	1.88	7.38E-6	0.97
AZ102-FD-9	6:36	6:57	0:21	17.1717	26.1073	8.94	9.30	0.44	2.60	0.91	8.66	0.22	2.10	4.85E-6	0.96
AZ102-FD-10	6:57	7:18	0:21	17.1484	26.1150	8.97	9.31	0.44	2.61	0.91	9.57	0.22	2.32	4.24E-6	0.96
AZ102-FD-11	7:18	7:39	0:21	17.0842	26.2388	9.2	9.76	0.46	2.73	0.96	10.53	0.23	2.56	3.94E-6	0.94
	ΔT, total	4:03					**average**	**0.44**	**2.61**						
						total vol	**107.40**								

DI water rinse

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV
AZ102-Fdi-1	7:43	8:03	0:20	17.1278	25.7211	8.593	8.593	0.43	2.53	0.84	0.84	0.20	0.20
AZ102-Fdi-2	8:03	8:24	0:21	17.2036	26.3510	9.147	9.147	0.44	2.56	0.90	1.74	0.22	0.42
AZ102-Fdi-3	8:24	8:48	0:24	17.2011	27.3354	10.134	10.134	0.42	2.48	0.99	2.73	0.24	0.66
AZ102-Fdi-4	8:48	9:10	0:22	17.0834	26.5651	9.482	9.482	0.43	2.54	0.93	3.66	0.23	0.89
AZ102-Fdi-5	9:10	9:31	0:21	17.2487	26.7820	9.533	9.533	0.45	2.67	0.93	4.60	0.23	1.12
AZ102-Fdi-6	9:31	9:53	0:22	17.1846	26.4675	9.283	9.283	0.42	2.48	0.91	5.51	0.22	1.34
AZ102-Fdi-7	9:53	10:15	0:22	17.1827	26.7614	9.579	9.579	0.44	2.56	0.94	6.45	0.23	1.57
AZ102-Fdi-8	10:15	10:35	0:20	17.1587	26.3988	9.240	9.240	0.46	2.72	0.91	7.35	0.22	1.79
AZ102-Fdi-9	10:35	10:55	0:20	17.1869	25.6666	8.480	8.480	0.42	2.49	0.83	8.18	0.20	1.99
AZ102-Fdi-10	10:56	11:19	0:23	17.2026	27.6091	10.407	10.407	0.45	2.66	1.02	9.20	0.25	2.24
ΔT, total		3:12					**average**	**0.44**	**2.57**				
						total vol	**93.9 mL**						

Cs elution from lead column,
Separate columns

Sample ID	start	end	Δt	Vial tare	Vial + sample	mass collected	volume	flow rate	BV/hr	cumul. BV
	HNO3 wash--lead column									
AZ102L-E1	11:47	13:00	1:13	17.1585	26.2693	9.1108	9.00	0.12	0.73	0.88
AZ102L-E2	13:01	14:00	0:59	17.1099	24.2582	7.1483	7.06	0.12	0.70	1.58
AZ102L-E3	14:01	15:00	0:59	17.0984	24.1358	7.0374	6.95	0.12	0.69	2.26
AZ102L-E4	15:01	16:00	0:59	17.1169	26.6698	9.5529	9.44	0.16	0.94	3.18
AZ102L-E5	16:01	17:00	0:59	17.1069	24.6126	7.5057	7.42	0.13	0.74	3.91
AZ102L-E6	17:01	18:00	0:59	17.1140	24.3592	7.2452	7.16	0.12	0.71	4.61
AZ102L-E7	18:01	19:00	0:59	17.2610	25.0439	7.7829	7.69	0.13	0.77	5.37
AZ102L-E8	19:01	20:00	0:59	17.1653	24.5191	7.3538	7.27	0.12	0.72	6.08
AZ102L-E9	20:01	21:00	0:59	17.1112	24.7442	7.6330	7.54	0.13	0.75	6.82
AZ102L-E10	21:02	22:00	0:58	17.1685	24.4314	7.2629	7.18	0.12	0.73	7.52
AZ102L-E11	22:00	23:00	1:00	17.1470	24.7331	7.5861	7.50	0.12	0.73	8.26
AZ102L-E12	23:01	0:00	0:59	17.2168	25.9274	8.7106	8.61	0.15	0.86	9.10
AZ102L-E13	0:00	1:00	1:00	17.2652	24.7709	7.5057	7.42	0.12	0.73	9.83
AZ102L-E14	1:00	2:00	1:00	17.1810	24.6294	7.4484	7.36	0.12	0.72	10.55
AZ102L-E15	2:00	3:00	1:00	17.1751	24.7030	7.5279	7.44	0.12	0.73	11.28
AZ102L-E16	3:01	4:00	0:59	17.1953	24.7538	7.5585	7.47	0.13	0.74	12.01
AZ102L-E17	4:01	5:00	0:59	17.1458	24.3407	7.1949	7.11	0.12	0.71	12.71
AZ102L-E18	5:00	6:00	1:00	17.2034	23.7136	6.5102	6.43	0.11	0.63	13.34
AZ102L-E19	6:00	7:06	1:06	17.1556	25.1342	7.9786	7.88	0.12	0.70	14.11
AZ102L-E20	7:08	8:00	0:52	17.1630	24.6927	7.5297	7.44	0.14	0.84	14.84
ΔT, total		20:13:00					**average**	**0.13**	**0.74**	
					sum	**153.18**	**151.37 mL**			

DI water

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102-Edi-1	8:03	8:23	0:20	17.2749	26.3946	9.120	9.120	0.46	2.68	0.9	0.9	0.40	0.40
AZ102-Edi-2	8:24	8:44	0:20	17.2678	26.7359	9.468	9.468	0.47	2.78	0.9	1.8	0.41	0.81
AZ102-Edi-3	8:45	9:06	0:21	17.2284	26.4806	9.252	9.252	0.44	2.59	0.9	2.7	0.40	1.21
AZ102-Edi-4	9:07	9:27	0:20	17.1799	25.8171	8.637	8.637	0.43	2.54	0.8	3.6	0.38	1.59
AZ102-Edi-5	9:28	9:48	0:20	17.2415	26.0399	8.798	8.798	0.44	2.59	0.9	4.4	0.38	1.97
	ΔT, total		1:45				**average**	**0.45**	**2.64**				
						total vol	**45.28 mL**						

Regeneration with 0.25M NaOH

Sample ID	Start	End	Δt	Bottle tare	Bottle + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102L-RGN	8:26	10:07	1:41	25.4	39.3	13.9	13.9	0.14	0.81	1.4	1.4	0.60	0.60
	10:08	11:38	1:30		54.5	15.2	15.2	0.17	0.99	1.5	2.9	0.66	1.27
	11:39	13:10	1:31		70.7	16.2	16.2	0.18	1.05	1.6	4.4	0.70	1.97
ΔT, total		4:44					**average**	**0.16**	**0.95**				
						sum	**45.3 mL**						

Rinse with DI water

Sample ID	Start	End	Δt	Bottle tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102-DIRinse-Final	13:23	13:50	0:27	14.2	26.0	11.8	11.8	0.44	2.57	1.2	1.2	0.51	0.51
	13:51	14:32	0:41		44.1	18.1	18.1	0.44	2.60	1.8	2.9	0.79	1.30
	14:33	15:08	0:35		60.8	16.7	16.7	0.48	2.81	1.6	4.6	0.73	2.03
ΔT, total		1:45					**average**	**0.45**	**2.66**				
						sum	**46.6 mL**						

Cs elution from lead column, repeat elution

Sample ID	start	end	Δt	Vial tare	Vial + sample	mass collected	volume	flow rate	BV/hr	cumul. BV	C/Co
HNO3 wash--lead column											
AZ102L-E1-R	8:43	9:04	0:21	17.2068	27.9832	10.7764	10.65	0.51	2.98	1.04	9.99E-5
AZ102L-E2-R	9:04	9:25	0:21	17.2606	26.1134	8.8528	8.75	0.42	2.45	1.90	5.25E-5
AZ102L-E3-R	9:25	9:46	0:21	17.247	26.2706	9.0236	8.92	0.42	2.50	2.78	1.14E-5
AZ102L-E4-R	9:47	10:09	0:22	17.2516	27.2294	9.9778	9.86	0.45	2.64	3.74	1.86E-3
AZ102L-E5-R	10:10	10:30	0:20	17.2444	26.3900	9.1456	9.04	0.45	2.66	4.63	2.17E-3
AZ102L-E6-R	10:31	10:52	0:21	17.2281	26.4951	9.2670	9.16	0.44	2.57	5.53	5.00E-4
AZ102L-E7-R	10:53	11:14	0:21	17.2856	26.6228	9.3372	9.23	0.44	2.58	6.43	1.85E-4
AZ102L-E8-R	11:15	11:16	0:01	17.24	17.9503	0.7103	0.70	0.70	4.13	6.50	1.83E-4
ΔT, total			2:33				**average**	**0.48**	**2.81**		
						sum	**66.30 mL**				

DI water rinse

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102-Edi-1-R	11:20	11:40	0:20	17.1879	25.8448	8.657	8.657	0.43	2.55	0.8	0.8	0.38	0.38
AZ102-Edi-2-R	11:41	12:01	0:20	17.2041	25.9557	8.752	8.752	0.44	2.57	0.9	1.7	0.38	0.76
AZ102-Edi-3-R	12:02	12:22	0:20	17.2151	26.4315	9.216	9.216	0.46	2.71	0.9	2.6	0.40	1.16
AZ102-Edi-4-R	12:22	12:43	0:21	17.1110	26.2132	9.102	9.102	0.43	2.55	0.9	3.5	0.40	1.55
AZ102-Edi-5-R	12:43	13:12	0:29	17.2364	30.1875	12.951	12.951	0.45	2.63	1.3	4.8	0.56	2.12
	ΔT, total		1:52				**average**	**0.44**	**2.60**				
						total vol	**48.678 mL**						

Regeneration with 0.25M NaOH

Sample ID	Start	End	Δt	Bottle tare	Bottle + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102L-RGN	8:13	9:34	1:21	14.1	24.9	10.8	10.8	0.13	0.78	1.1	1.1	0.47	0.47
	9:35	11:04	1:29		37.3	12.4	12.4	0.14	0.82	1.2	2.3	0.54	1.01
	11:05	12:48	1:43		52.2	14.9	14.9	0.14	0.85	1.5	3.7	0.65	1.66
	12:49	14:08	1:19		63.7	11.5	11.5	0.15	0.86	1.1	4.9	0.50	2.16
ΔT, total		5:55					**average**	**0.14**	**0.83**				
						sum	**49.6 mL**						

Rinse with DI water

Sample ID	Start	End	Δt	Bottle tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AZ102-DIRinse-Final	14:13	14:47	0:34	14.2	28.2	14.0	14.0	0.41	2.42	1.4	1.4	0.61	0.61
	14:48	15:20	0:32		42.0	13.8	13.8	0.43	2.54	1.4	2.7	0.60	1.21
	15:22	15:54	0:32		55.7	13.7	13.7	0.43	2.52	1.3	4.1	0.60	1.80
ΔT, total		1:41					**average**	**0.42**	**2.49**				
						sum	**41.5 mL**						

Counting Data for TI-RPP-WTP-132, Rev. 0

Average Background Count Rate **82.57**

300 sec count time MDL 19.11 cpm
3.72E-07 C/Co
7.645 cpm/g

AZ102 Comparitor Standard density **1.2462** g/mL

Position 1 on counter		net cpm/g	cpm/mL
2.78E-4 g AZ-102C	**Average**	**2.06E+7**	**2.56E+7**
	standard deviation	**1.12E+6**	**1.40E+6**
	% standard deviation	**5.5**	**5.5**

Position 6 on counter		net cpm/g	cpm/mL
4.889E-3 g AZ-102C	**Average**	**1.07E+6**	**1.24E+6**
	standard deviation	**1.04E+5**	**2.68E+4**
	% standard deviation	**9.8**	**2.2**

AZ-102 run starting 11/5/01
Lead column, loading phase

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	BV	% C/Co	DF	µCi Cs-137
AZ102L-F1	P1	381	475	1.86E-5	**4.5**	1.86E-3	5.4E+4	0.060
AZ102L-F2	P1	291	363	1.42E-5	**6.5**	1.42E-3	7.1E+4	0.056
AZ102L-F3	P1	70	87	3.40E-6	**14.9**	3.40E-4	2.9E+5	0.012
AZ102L-F4 D	P1	265	330	1.29E-5	**17.9**	1.29E-3	7.8E+4	0.013
AZ102L-F5 D	P1	279	348	1.36E-5	**21.2**	1.36E-3	7.4E+4	0.013
AZ102L-F6	P1	224	280	1.09E-5	**25.6**	1.09E-3	9.2E+4	0.037
AZ102L-F7	P1	193	240	9.37E-6	**28.0**	9.37E-4	1.1E+5	0.039
AZ102L-F8	P1	220	275	1.07E-5	**31.6**	1.07E-3	9.3E+4	0.059
AZ102L-F9	P1	200	250	9.75E-6	**36.7**	9.75E-4	1.0E+5	0.093
AZ102L-F10	P1	182	227	8.84E-6	**41.1**	8.84E-4	1.1E+5	0.025
AZ102L-F11	P1	181	226	8.81E-6	**45.4**	8.81E-4	1.1E+5	0.034
AZ102L-F12	P1	232	289	1.13E-5	**49.8**	1.13E-3	8.9E+4	0.032
AZ102L-F13	P1	186	231	9.03E-6	**54.2**	9.03E-4	1.1E+5	0.032
AZ102L-F14	P1	211	263	1.03E-5	**58.8**	1.03E-3	9.7E+4	0.029
AZ102L-F15	P1	184	230	8.97E-6	**63.2**	8.97E-4	1.1E+5	0.028
AZ102L-F16	P1	249	310	1.21E-5	**67.7**	1.21E-3	8.3E+4	0.005
AZ102L-F17	P1	15538	19363	7.56E-4	**70.8**	7.56E-2	1323	0.293
AZ102L-F18	P1	48700	60690	2.37E-3	**75.0**	2.37E-1	422	0.938
AZ102L-F19	P6	23804	29664	2.23E-2	**79.9**	2.23E+0	45	1.55
AZ102L-F20	P6	161013	200654	1.51E-1	**85.1**	1.51E+1	6.6	8.33
AZ102L-F21dR	P6	368947	459782	3.46E-1	**90.0**	3.46E+1	2.9	13.02
AZ102L-F22dR	P6	744943	928348	6.99E-1	**95.1**	6.99E+1	1.4	24.58
AZ102L-F23	P6	736682	918053	6.91E-1	**100.0**	6.91E+1	1.4	25.53
AZ102L-F24	P6	841966	1049258	7.90E-1	**105.0**	7.90E+1	1.3	23.51
							sum (µCi)	**98.315**

AZ-102 run starting 11/5/01
Lag column, loading phase

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	µCi Cs-137
AZ102P-F1	P1	244.19	304.31	1.19E-5	**4.5**	1.19E-3	8.4E+4	0.043
AZ102P-F2	P1	347.87	433.52	1.69E-5	**6.5**	1.69E-3	5.9E+4	0.083
AZ102P-F3	P1	389.34	485.20	1.89E-5	**14.5**	1.89E-3	5.3E+4	0.069
AZ102P-F4	P1	422.84	526.95	2.06E-5	**17.3**	2.06E-3	4.9E+4	0.019
AZ102P-F5	P1	332.03	413.78	1.62E-5	**20.4**	1.62E-3	6.2E+4	0.015
AZ102P-F6	P1	362.57	451.83	1.76E-5	**24.6**	1.76E-3	5.7E+4	0.085
AZ102P-F7	P1	296.67	369.70	1.44E-5	**26.9**	1.44E-3	6.9E+4	0.067
AZ102P-F8	P1	284.4	354.41	1.38E-5	**30.4**	1.38E-3	7.2E+4	0.132
AZ102P-F9	P1	307.10	382.71	1.49E-5	**34.8**	1.49E-3	6.7E+4	0.094
AZ102P-F10	P1	373.59	465.56	1.82E-5	**39.0**	1.82E-3	5.5E+4	0.100
AZ102P-F11	P1	281.07	350.27	1.37E-5	**43.1**	1.37E-3	7.3E+4	0.062
AZ102P-F12	P1	272.04	339.01	1.32E-5	**47.4**	1.32E-3	7.6E+4	0.078
AZ102P-F13	P1	268.03	334.02	1.30E-5	**51.5**	1.30E-3	7.7E+4	0.057
AZ102P-F14	P1	256.79	320.01	1.25E-5	**55.9**	1.25E-3	8.0E+4	0.054
AZ102P-F15	P1	257.72	321.17	1.25E-5	**60.2**	1.25E-3	8.0E+4	0.076
AZ102P-F16	P1	177.96	221.77	8.66E-6	**64.5**	8.66E-4	1.2E+5	0.057
AZ102P-F17	P1	227.54	283.57	1.11E-5	**67.3**	1.11E-3	9.0E+4	0.055
AZ102P-F18	P1	210.34	262.13	1.02E-5	**71.4**	1.02E-3	9.8E+4	0.047
AZ102P-F19	P1	224.12	279.30	1.09E-5	**76.0**	1.09E-3	9.2E+4	0.042
AZ102P-F20	P1	206.17	256.93	1.00E-5	**80.9**	1.00E-3	1.0E+5	0.038
AZ102P-F21	P1	206.19	256.95	1.00E-5	**85.7**	1.00E-3	1.0E+5	0.038
AZ102P-F22	P1	186.28	232.14	9.06E-6	**90.5**	9.06E-4	1.1E+5	0.033
AZ102P-F23	P1	187.65	233.85	9.13E-6	**95.3**	9.13E-4	1.1E+5	0.035
AZ102P-F24	P1	183.42	228.58	8.92E-6	**100.2**	8.92E-4	1.1E+5	0.055
							sum (µCi)	**1.433**

	Count position	net cpm/g	net cpm/mL	C/Co	C/Co, %	DF		µCi Cs-137
AZ102FEcomp1	P1	40.12	38.71	1.95E-6	1.95E-4	5.12E+5		0.008
AZ102FEcomp2	P1	325.66	405.84	1.58E-5	1.58E-3	6.31E+4		0.057
AZ102FEcomp3	P1	409.40	510.20	1.99E-5	1.99E-3	5.02E+4		0.075
AZ102FEcomp4	P1	339.95	423.64	1.65E-5	1.65E-3	6.05E+4		0.063
AZ102FEcomp5	P1	307.07	382.67	1.49E-5	1.49E-3	6.69E+4		0.057
AZ102FEcomp6	P1	259.64	323.57	1.26E-5	1.26E-3	7.92E+4		0.048
AZ102FEcomp7	P1	259.08	322.86	1.26E-5	1.26E-3	7.93E+4		0.048
AZ102FEcomp8	P1	263.28	328.11	1.28E-5	1.28E-3	7.81E+4		0.049
AZ102FEcomp9	P1	220.11	274.30	1.07E-5	1.07E-3	9.34E+4		0.041
AZ102FEcomp10	P1	214.68	267.53	1.04E-5	1.04E-3	9.57E+4		0.040
AZ102FEcomp11	P1	186.07	231.89	9.05E-6	9.05E-4	1.10E+5		0.035
AZ102FEcomp12	P1	199.73	248.90	9.72E-6	9.72E-4	1.03E+5		0.038
							sum (µCi)	**0.56**

Feed Displacement all on position 1

Sample ID	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	BV+	µCi Cs-137
AZ102-FD-1	162.62	162.62	7.91E-6	1.0	7.91E-4	101.2	0.158
AZ102-FD-2	162.89	162.89	7.93E-6	1.9	7.93E-4	102.1	0.147
AZ102-FD-3	184.17	184.17	8.96E-6	2.9	8.96E-4	103.2	0.195
AZ102-FD-4	169.40	169.40	8.24E-6	4.0	8.24E-4	104.2	0.178
AZ102-FD-5	201.90	201.90	9.82E-6	5.1	9.82E-4	105.3	0.222
AZ102-FD-6	410.89	410.89	2.00E-5	6.2	2.00E-3	106.4	0.447
AZ102-FD-7	231.91	231.91	1.13E-5	7.3	1.13E-3	107.5	0.259
AZ102-FD-8	151.69	151.69	7.38E-6	8.3	7.38E-4	108.6	0.153
AZ102-FD-9	99.61	99.61	4.85E-6	9.2	4.85E-4	109.4	0.087
AZ102-FD-10	87.07	87.07	4.24E-6	10.1	4.24E-4	110.3	0.076
AZ102-FD-11	81.00	81.00	3.94E-6	11.0	3.94E-4	111.2	0.072
						sum (µCi)	**2.00**

DI Rinse all on position 1

Sample ID	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	BV+	µCi Cs-137
AZ102-Fdi-1	75.88	75.88	3.69E-6	0.8	3.69E-4	112.1	0.064
AZ102-Fdi-2	67.79	67.79	3.30E-6	1.7	3.30E-4	112.9	0.060
AZ102-Fdi-3	66.82	66.82	3.25E-6	2.7	3.25E-4	113.9	0.066
AZ102-Fdi-4	73.38	73.38	3.57E-6	3.7	3.57E-4	114.9	0.068
AZ102-Fdi-5	73.86	73.86	3.59E-6	4.6	3.59E-4	115.8	0.069
AZ102-Fdi-6	58.46	58.46	2.84E-6	5.5	2.84E-4	116.7	0.053
AZ102-Fdi-7	49.63	49.63	2.41E-6	6.4	2.41E-4	117.7	0.046
AZ102-Fdi-8	46.09	46.09	2.24E-6	7.4	2.24E-4	118.6	0.042
AZ102-Fdi-9	22.26	22.26	1.08E-6	8.2	1.08E-4	119.4	0.018
AZ102-Fdi-10	16.16	16.16	7.86E-7	9.2	7.86E-5	120.4	0.016
						sum (µCi)	**0.50**

AZ-102 run starting 11/5/01.
Lead column, eluting phase

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	BV	µCi Cs-137
AZ102L-E1-d	P1	6.59E+4	6.67E+4	2.60E-3	0.9	49
AZ102L-E2-d	P1	1.29E+5	1.31E+5	5.10E-3	1.6	97
AZ102L-E3-d	P1	1.19E+6	1.21E+6	4.72E-2	2.3	897
AZ102L-E4-dd	P1	3.57E+7	3.61E+7	1.41E+0	3.2	26808
AZ102L-E5-dd	P6	3.77E+7	3.82E+7	3.09E+1	3.9	587253
AZ102L-E6-dd	P6	5.26E+7	5.32E+7	4.31E+1	4.6	818034
AZ102L-E7-dd	P1	5.07E+7	5.13E+7	2.00E+0	5.4	38076
AZ102L-E8-d	P6	4.00E+5	4.05E+5	3.28E-1	6.1	6228
AZ102L-E9-d	P1	1.81E+6	1.83E+6	7.14E-2	6.8	1357
AZ102L-E10-d	P1	6.39E+5	6.46E+5	2.52E-2	7.5	480
AZ102L-E11-d	P1	2.30E+5	2.33E+5	9.08E-3	8.3	173
AZ102L-E12-d	P1	1.21E+5	1.23E+5	4.78E-3	9.1	91
AZ102L-E13-d	P1	7.21E+4	7.30E+4	2.85E-3	9.8	54
AZ102L-E14-d	P1	5.70E+4	5.77E+4	2.25E-3	10.5	43
AZ102L-E15-d	P1	4.46E+4	4.51E+4	1.76E-3	11.3	33
AZ102L-E16-d	P1	3.30E+4	3.34E+4	1.31E-3	12.0	25
AZ102L-E17-d	P1	2.79E+4	2.82E+4	1.10E-3	12.7	21
AZ102L-E18-d	P1	2.61E+4	2.64E+4	1.03E-3	13.3	20
AZ102L-E19-d	P1	2.33E+4	2.36E+4	9.23E-4	14.1	18
AZ102L-E20-d	P1	1.68E+4	1.70E+4	6.65E-4	14.8	13

Deionized water rinse following elution

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	BV	µCi Cs-137	BV + elution BV
AZ102-Edi-1	P1	12596	12596	4.92E-4	0.9	8.99	15.7
AZ102-Edi-2	P1	5623	5623	2.20E-4	1.8	4.17	16.7
AZ102-Edi-3	P1	5889	5889	2.30E-4	2.7	4.27	17.6
AZ102-Edi-4	P1	5013	5013	1.96E-4	3.6	3.39	18.4
AZ102-Edi-5	P1	2876	2876	1.12E-4	4.4	1.98	19.3
					sum (µCi)	**22.79**	

Regeneration with 0.25 M NaOH Position 1

Sample ID	net cpm/g	net cpm/mL	C/Co	µCi Cs-137
AZ102-RGN	25720	25875	1.01E-3	91.8

AZ-102 run starting 11/5/01.
Lead column, Repeat eluting phase

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	µCi Cs-137	BV	BV+
AZ102L-E1-Rd	P1	2.53E+3	2.56E+3	9.99E-5	2.13	1.0	25
AZ102L-E2-Rd	P1	1.33E+3	1.34E+3	5.25E-5	0.92	1.9	26
AZ102L-E3-Rd	P1	2.87E+2	2.91E+2	1.14E-5	0.20	2.8	26
AZ102L-E4-Rd	P1	4.70E+4	4.75E+4	1.86E-3	36.67	3.7	27
AZ102L-E5-Rd	P1	5.49E+4	5.56E+4	2.17E-3	39.32	4.6	28
AZ102L-E6-Rd	P1	1.27E+4	1.28E+4	5.00E-4	9.19	5.5	29
AZ102L-E7-Rd	P1	4.69E+3	4.75E+3	1.85E-4	3.43	6.4	30
AZ102L-E8-Rd	P1	4.62E+3	4.68E+3	1.83E-4	0.26	6.5	30
				sum (µCi)	**92.12**		

Deionized water rinse following elution

Sample ID	Count position	net cpm/g	net cpm/mL	C/Co	µCi Cs-137	BV	BV +
AZ102-Edi-1R	P1	2404	2404	9.38E-5	1.63	0.8	31.1
AZ102-Edi-2R	P1	1473	1473	5.75E-5	1.01	1.7	31.9
AZ102-Edi-3R	P1	957	957	3.74E-5	0.69	2.6	32.8
AZ102-Edi-4R	P1	506	506	1.98E-5	0.36	3.5	33.7
AZ102-Edi-5R	P1	276	276	1.08E-5	0.28	4.8	35.0
				sum (µCi)	**3.97**		

Regeneration with 0.25 M NaOH Position 1

Sample ID	net cpm/g	net cpm/mL	C/Co	BV+	µCi Cs-137	
AZ102-RGN-R	719	723	2.82E-5	39.1	**2.81**	**total**

AZ-102 run starting 11/5/01

Lead column, loading phase, CMC analysis, ASR 6280

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	BV	DF	
AZ-102C (Feed)	02-0751	2005	3	1.00E+0				**2219.9** mCi Cs-137 loaded
AZ102L-F3	02-0757	5.43E-03	4	2.71E-6	2.71E-4	14.9	3.69E+05	
AZ102L-F8	02-0758	1.73E-02	3	8.63E-6	8.63E-4	31.6	1.16E+05	
AZ102L-F13	02-0759	1.40E-02	3	6.98E-6	6.98E-4	54.2	1.43E+05	
AZ102L-F17	02-0760	1.13E+00	3	5.64E-4	5.64E-2	70.8	1.77E+03	
AZ102L-F18	02-0902	3.86	3	1.93E-3	1.93E-1	75.0	5.19E+02	
AZ102L-F19	02-0903	47.7	3	2.38E-2	2.38E+0	79.9	4.20E+01	
AZ102L-F20	02-0761	2.78E+02	4	1.39E-1	1.39E+1	85.1	7.21E+00	
AZ102L-F21	02-0904	7.27E+02	3	3.63E-1	3.63E+1	90.0	2.76E+00	
AZ102L-F22	02-0762	1.14E+03	3	5.69E-1	5.69E+1	95.1	1.76E+00	
AZ102L-F23	02-0763	1.38E+03	3	6.88E-1	6.88E+1	100.0	1.45E+00	
AZ102L-F24	02-0905	1660	3	8.28E-1	8.28E+1	105.0	1.21E+00	
AZ102L-F24D	02-0905	1470	3	7.33E-1	7.33E+1	105.0	1.36E+00	
AZ102L-F24 average	02-0905	1565	1.9	7.81E-1	7.81E+1	105.0	1.28E+00	
AZ102P-F4	02-0764	2.55E-02	3	1.27E-5	1.27E-03	17.3	7.86E+04	
AZ102P-F11	02-0765	2.28E-02	3	1.14E-5	1.14E-03	43.1	8.79E+04	
AZ102P-F18	02-0766	1.68E-02	3	8.38E-6	8.38E-04	71.4	1.19E+05	
AZ102P-F24	02-0767	1.40E-02	4	6.98E-6	6.98E-04	100.2	1.43E+05	
AZ102C-EC	02-0752	1.92E-02	3	9.55E-6	9.55E-04		1.05E+05	
composite effluent		19.5713	uCi total Cs-137 recovered in efluent					

Feed Displacement

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	BV+ load
AZ102-FD-2	02-0753	1.32E-02	4	6.58E-6	6.58E-04	102.1
AZ102-FD-6	02-0754	2.85E-02	3	1.42E-5	1.42E-03	106.4

DI water rinse

						BV+load+FD
AZ102-Fdi-4	02-0755	4.06E-03	3	2.02E-6	2.02E-04	114.9
AZ102-Fdi-9	02-0756	1.45E-03	4	7.23E-7	7.23E-05	119.4

Lead column, eluting phase

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	BV	total mCi Cs-137	
AZ102L-E4	02-0768	7.78E+03	3	3.88E+0	3.2	7.34E+01	
AZ102L-E5	02-0793	1.22E+05	3	6.08E+1	3.9	9.05E+02	
AZ102L-E6	02-0769	1.42E+05	3	7.08E+1	4.6	1.02E+03	
AZ102L-E7	02-0794	1.30E+04	3	6.48E+0	5.4	1.00E+02	
AZ102L-E8	02-0770	4.31E+02	3	2.15E-1	6.1	3.13E+00	
AZ102L-E11	02-0771	1.45E+01	3	7.23E-3	8.3	1.09E-01	
AZ102L-E18	02-0772	1.54E+00	3	7.68E-4	13.3	9.91E-03	
					sum mCi Cs-137	**2.10E+03**	**95%** recovery in eluate

DI water rinse

					BV+ eluate
AZ102L-Edi-1	02-0773	7.65E-01	3	3.82E-4	15.7
AZ102L-Edi-5	02-0774	1.95E-01	3	9.73E-5	19.3

Regeneration with 0.25 M NaOH Analytical Lab Data

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	Cs-137, µg/mL	total Cs, µg/mL	µCi Cs-137
AZ102-RGN	02-0775	1.64E+00	3	8.18E-04	0.0188506	0.0574713	**74.3**

AZ-102C Cs IX Lead Column Breakthrough Curve Integration

Activity of Cs-137 in Feed = 2005 uCi/mL

Sample	Processed Vol (mL)	Cs-137 C/Co	Cs-137 Conc (µCi/mL)	Δ Vol (mL)	C/Co Midpoint	Midpoint Conc (µCi/mL)	Area (uCi)	Cs-137 (µCi/mL) CMC Analysis	C/Co Midpoint	Area (µCi Cs-137)
AZ102L-F1	45.9	1.86E-05	3.72E-02	45.9	1.86E-05	3.72E-02	1.71E+00	---	---	---
AZ102L-F2	66.7	1.24E-05	2.48E-02	20.8	1.55E-05	3.10E-02	6.45E-01	---	---	---
AZ102L-F3	151.8	3.40E-06	6.82E-03	85.1	7.89E-06	1.58E-02	1.35E+00	5.43E-3	---	---
AZ102L-F4	182.9	1.29E-05	2.58E-02	31.1	8.14E-06	1.63E-02	5.08E-01	---	---	---
AZ102L-F5	216.3	1.36E-05	2.73E-02	33.5	1.32E-05	2.65E-02	8.88E-01	---	---	---
AZ102L-F6	260.7	1.09E-05	2.19E-02	44.3	1.23E-05	2.46E-02	1.09E+00	---	---	---
AZ102L-F7	285.7	9.37E-06	1.88E-02	25.1	1.01E-05	2.03E-02	5.10E-01	---	---	---
AZ102L-F8	322.0	1.07E-05	2.15E-02	36.3	1.00E-05	2.01E-02	7.31E-01	1.73E-2	---	---
AZ102L-F9	374.3	9.75E-06	1.95E-02	52.3	1.02E-05	2.05E-02	1.07E+00	---	---	---
AZ102L-F10	419.2	8.84E-06	1.77E-02	44.9	9.29E-06	1.86E-02	8.37E-01	---	---	---
AZ102L-F11	463.1	8.81E-06	1.77E-02	43.9	8.82E-06	1.77E-02	7.77E-01	---	---	---
AZ102L-F12	508.0	1.13E-05	2.26E-02	44.9	1.00E-05	2.01E-02	9.04E-01	---	---	---
AZ102L-F13	553.2	9.03E-06	1.81E-02	45.2	1.02E-05	2.04E-02	9.21E-01	1.40E-2	---	---
AZ102L-F14	599.6	1.03E-05	2.06E-02	46.4	9.64E-06	1.93E-02	8.97E-01	---	---	---
AZ102L-F15	644.2	8.97E-06	1.80E-02	44.6	9.61E-06	1.93E-02	8.60E-01	---	---	---
AZ102L-F16	690.5	1.21E-05	2.43E-02	46.3	1.05E-05	2.11E-02	9.79E-01	---	---	---
AZ102L-F17	721.8	7.56E-04	1.52E+00	31.3	3.84E-04	7.70E-01	2.41E+01	1.13E+0	5.72E-1	1.79E+1
AZ102L-F18	765.5	2.37E-03	4.75E+00	43.7	1.56E-03	3.13E+00	1.37E+02	3.86E+0	2.50E+0	1.09E+2
AZ102L-F19	814.7	2.23E-02	4.48E+01	49.2	1.23E-02	2.48E+01	1.22E+03	4.77E+1	2.58E+1	1.27E+3
AZ102L-F20	867.7	1.51E-01	3.03E+02	53.0	8.67E-02	1.74E+02	9.21E+03	2.78E+2	1.63E+2	8.64E+3
AZ102L-F21	918.5	3.46E-01	6.94E+02	50.8	2.49E-01	4.98E+02	2.53E+04	7.27E+2	5.03E+2	2.55E+4
AZ102L-F22	969.5	6.99E-01	1.40E+03	51.1	5.22E-01	1.05E+03	5.35E+04	1.14E+3	9.34E+2	4.77E+4
AZ102L-F23	1019.9	6.91E-01	1.39E+03	50.4	6.95E-01	1.39E+03	7.02E+04	1.38E+3	1.26E+3	6.35E+4
AZ102L-F24	1071.2	7.90E-01	1.58E+03	51.3	7.40E-01	1.48E+03	7.61E+04	1.57E+3	1.47E+3	7.55E+4
Full load:	**1107.2 mL (obtained by mass difference of feed bottle)**						2.36E+05	uCi		2.22E+5

(CMC analyses resulted in good agreement)

Amount of Cs that Broke through Lead Column				
= Amount of Cs on Lag Column	=	235638	uCi	2.22E+5
	=	236	mCi	222.16518
Amount of Cs Fed to Lead Column	=	2.22E+06	uCi	
	=	2220	mCi	
Percentage of Cs-137 on Lag Column	=	10.6%		10.0%
Amount of Cs-137 Loaded on Lead Column =		1984	mCi	1998
Amount of Cs-137 Loaded on Lead Column =		22.8	mg Cs-137	23.0
Amount of total Cs loaded on lead column =		69.5	mg Cs	70.0

sum through 67.7 BV **1.47E+01 uCi Cs-137**

"---" indicates no data taken

Appendix D

Analytical Data

Appendix D: Analytical Data

Table D.1. Sample Identification

Sample Description	ASR	RPL ID	Sample Identification	Extended Sample Description
Batch Contact Testing				
Batch contact with	6413	02-2300	AZ-102-TI164-S0-FA	Batch contact control, unspiked,
SL-644 010319SMC-IV-73		02-2301	AZ-102-TI164-S0-D-FA	Batch contact control, unspiked, duplicate
212- to 425-µm particle size		02-2302	AZ-102-TI164-S0-73-FA	Batch contact with SL-644, unspiked
H form resin		02-2303	AZ-102-TI164-S0-73D-FA	Batch contact with SL-644, unspiked, duplicate
		02-2304	AZ-102-TI164-S1-FA	Batch contact control, 4E-3M Cs
		02-2305	AZ-102-TI164-S1-D-FA	Batch contact control, 4E-3 M Cs, duplicate
		02-2306	AZ-102-TI164-S1-73-FA	Batch contact with SL-644, 4E-3 M Cs
		02-2307	AZ-102-TI164-S1-73D-FA	Batch contact with SL-644, 4E-3 M Cs, duplicate
		02-2308	AZ-102-TI164-S2-FA	Batch contact control, 7E-3 M Cs
		02-2309	AZ-102-TI164-S2-D-FA	Batch contact control, 7E-3 M Cs, duplicate
		02-2310	AZ-102-TI164-S2-73-FA	Batch contact with SL-644, 7E-3
		02-2311	AZ-102-TI164-S2-73D-FA	Batch contact with SL-644, 7E-3 M Cs, duplicate
Column Run				
Initial Feed Sample, AZ-102C and	6280	02-0751	AZ102C	AZ-102C filtrate composite/Cs IX feed
Column processing samples		02-0757—02-0763	AZ102L-F3, -F8, -F13, -F17, -F20, -F22, -F23	Lead column load samples
		02-0764—02-0767	AZ102P-F5, -F11, -F18, -F24	Lag column load samples
		02-0753—02-0754	AZ-102-FD-2 and -6	Feed displacement samples
		02-0755—02-0756	AZ-102-Fdi-4 and -9	Water rinse samples
		02-0768—02-0772	AZ102L-E4, -E6, -E8, -E11, -E18	Lead column elution samples
		02-0773—02-0774	AZ102-Edi-1, -5	Elution rinse samples
		02-0775	AZ102-RGN	Regeneration solution analytical sample
		02-0752	AZ102C-EC-D	Effluent composite
Additional feed and elution samples	6305	02-0902—02-0905	AZ102L-F18, -F19, -F21, -F24	Lead column load samples
		02-0906—02-0908	AZ102L-E3, -E9, -E10	Lead column eluate samples
Additional feed and elution samples	6310	02-0951—02-0954	AZ102-F0DDD3, -F0DDD2, -F24D, -F14	Lead column load and comparator samples
		02-0955—02-0956	AZ102L-E5DD, AZ102L-E10D	Diluted eluate samples
Composite eluate	6374	02-1698	AZ102C-CsE-comp	Composited eluate samples

Notes: 1) The suffix letter "D" indicates that the sample was diluted prior to submission for analysis. Unidentified samples are associated with other tests unrelated to this work.
2) RPL = Radiochemical Processing Laboratory.

Appendix E

Cesium Ion Exchange and Batch Contacts Testing Personnel

Appendix E: Cesium Ion Exchange and Batch Contacts Testing Personnel

Cognizant Scientists
S. K. Fiskum
D. L. Blanchard
S. A. Arm

Hot Cell Technicians
F. V. Hoopes
M. A. Mann
F. H. Steen
J. W. Chenault
D. E. Rinehart

Analytical Support
S. J. Bos
J. P. Bramson
L. P. Darnell
T. O. Farmer
S. K. Fiskum
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