PNWD-3195
WTP-RPT-015

Aging Study and Small Column Ion Exchange Testing of SuperLig® 644 for Removal of ^{137}Cs from Simulated AW-101 Hanford Tank Waste

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September 2002

Prepared for Bechtel National, Inc.
under Contract No. 24590-101-TSA-W000-0004

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Battelle - Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specifications TSP-W375-00-00028 and TSP-W375-00-00034 and Test Plans CHG-TP-41500-013, Rev. 0 and TP-PNNL-WTP-044, 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
WTP R&T Support Project

Date

G. Todd Wright, Manager
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Date

Summary

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 flowsheet includes the use of Cs-selective, 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. The RPP-WTP Development Requirements Document (DRD)[1] task 8.2.6 and the RPP-WTP Research and Technology schedule identify Cs and Tc ion exchange process verification tests [proposal reference Standard 2 item (a)(3)(ii)]. The DRD task 8.2.3 identifies tests for evaluating the effects of storage conditions on the performance of SL-644 resin [proposal reference Standard 2 item (a)(3)(ii)].

Battelle Pacific Northwest Division (PNWD) was contracted to perform Cs ion exchange studies under contract 24590-101-TSA-W000-0004, and work breakdown structures BN.02.08.01.01 and BN.02.08.01.03. The Cs ion exchange activities are further defined in Technical Scoping Statements B-42 and B-53, which are included in Appendix C of the Research and Technology Plan[2]. 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.

Success criteria for Cs removal are defined by the Cs load/breakthrough curve and the final effluent ^{137}Cs concentration. The test specification[3] defined successful Cs removal as 50% breakthrough from the lead column occurring at ≥150 bed volumes (BVs) of AW-101 simulant (Envelope A) waste loading. The final effluent was to contain ≤0.087 µCi ^{137}Cs/mL. These criteria were met with the newly-received SL-644 resin, 010319SMC-IV-73, 212- to 425 µm particle size. The success criterion for the aging study[4] was to ensure the Cs distribution coefficients (K_d) resulted in a feed condition K_d value of ≥450 mL/g for a two-year aged batch of SL-644. The aged SL-644, 981112YK-N3-16/18 (16/18), resin met this success criterion.

This report summarizes testing of three batches of SuperLig®644 ion exchange materials and two different particle-size distributions (PSDs). The first batch came from a two-year aged production batch 981112YK-N3-16/18; only the as-received particle size distribution was tested.

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1 PL-W375-TE00002, Rev. 1, River Protection Project Waste Treatment Plant Development Requirements Document, October 31, 2000, M. E., Johnson and T. W. Crawford, CH2MHill Hanford Group, Inc., Richland, WA. DRAFT.
2 Barnes, S., R. Roosa and R. Peterson. 2002. 'Research and Technology Plan', 24590-WTP-PL-RT-01-002, Rev. 1, RPP-WTP project.
3 Test Specification: "Tank 241-AN-102 and 241-AP-101 Ion Exchange," TSP-W375-00-00028, Rev. 1, M. E. Johnson, CH2M HILL Hanford Group, Dec. 11, 2000 and Test Plan "Actual Waste Ion Exchange Testing for the RPP-WTP Project," CHG-TP-41500-013, Rev. 0, D. L. Blanchard, Jan. 24, 2000.
4 Test Specification: "Evaluating SuperLig 644 Storage on Exchange Capacity and Effectiveness of Up Flow Elution," TSP-W375-00-00034, M. E. Johnson, CH2M HILL Hanford Group, Dec. 6, 2000 and PNNL Test Plan "Evaluation of SuperLig 644 Storage on Exchange Capacity and Effectiveness of Up Flow Elution," TP-PNNL-WTP-044, Rev. 0, S. K. Fiskum Feb. 5, 2001.

The second batch was from previously tested aged (approximately 2 y) 644BZ.[5] The third batch was from a recent production batch 010319SMC-IV-73 (–73); the as-received and the dry-sieved 212- to 425-μm PSDs were tested. Batch contacts were conducted with the materials in the as-received condition and in the hydrogen form at three different Cs concentrations to determine batch-distribution coefficients. Three column experiments were conducted with two columns in series, each containing a nominal 10-mL resin bed. Two tests used the –73 and 16/18 SL-644 batch materials in the as-received resin PSD. The third test was conducted with the 212- to 425-μm size fraction of the –73 material. The test matrix was AW-101 simulant (5 M Na^+). Load and elution profiles were determined. After demonstrating successful Cs removal, the system was to be used on hot-cell testing of actual tank waste.

A summary of performance measures for each resin batch is shown in Table S1. The estimated 50% breakthrough refers to the volume of waste processed through an ion exchange column, measured in BVs, to the point where the Cs effluent concentration is equivalent to 50% of the influent Cs concentration. It represents a measure of the effective capacity of the SL-644 resin. The 16/18 batch 50% Cs breakthrough occurred at 100 BVs (BV determined in contact with regeneration solution, 0.25 M NaOH, equivalent to 128 BVs in contact with the AW-101 feed). The –73 batch of resin (as-received PSD) reached a 12% Cs breakthrough at 148 BVs with the 50% breakthrough extrapolated to 270 BVs (regeneration condition). The 212- to 425-μm sieved fraction of the –73 resin batch resulted in 1% breakthrough at 143 BVs (regeneration condition); the extrapolated 50% breakthrough occurred at approximately 270 BVs. The overall decontamination factors (DFs) at the tested BVs (regeneration condition) of effluent for the different resins tested are shown in Table S1.

Table S1. Summary of Performance Measures

SuperLig® 644 Batch ID	**Estimated 50% Breakthrough, BV Regeneration condition (Feed condition)(1)**	**DF(2) (BVs processed)**	**K_d, mL/g (Na-form)(3)**
Small PSD, nominally 212- to 425-μm			
644BZ	—	—	700
010319SMC-IV-73	270 (300)	6.6 E+5 (143)	900
Large PSD, nominally 212- to 1000-μm			
981112YK-N3-16/18	100 (128)	94 (116)	630
010319SMC-IV-73	270 (300)	581 (137)	720

(1) Regeneration condition refers to resin in contact with 0.25 M NaOH; feed condition refers to resin in contact with the AW-101 simulant feed at about 5 M Na. The resin expansion is greater in the 0.25 M NaOH than in the AW-101 feed.
(2) The decontamination factor is calculated by dividing the feed Cs concentration by the composite effluent Cs concentration.
(3) The K_d value is the interpolated value at the feed condition Na/Cs mol ratio of nominally 6E+4.

[5] IBC could not identify the production date for SL-644 batch 644BZ.

The calculated feed condition K_d values obtained from batch-contact equilibrium data are also shown in Table S1. The equilibrium feed condition K_d value for the 16/18 batch was somewhat lower than the similarly sized –73 resin. However, the 16/18 batch material was shown to be significantly less effective for Cs removal based on actual column testing than the analogous –73 resin. The large PSD –73 resin still did not meet the required DF of 2200 (1.75E-5 Ci ^{137}Cs/mole Na) for Cs removal after processing 150 BVs[6]. The 212- to 412-µm PSD of –73 did work well for Cs removal, and this material was transferred to the hot cells for use on actual waste testing.

The batch-contact testing for the aging-effect study of the SL-644 was inconclusive. When comparing similar PSDs, the new material exhibited slightly higher K_d values than the aged materials. Column testing however indicated Cs exchange kinetics may have been greatly affected. Comparing the load profiles shows much more rapid breakthrough on the aged material. However, previous testing with the 644BZ also showed rapid Cs breakthrough.[7] This indicates production batch variability may be a source of variable performance, i.e., the older production batches had poorer kinetics than the newer production batch. A more detailed study may be required to determine if variability in performance is due to an aging phenomenon, a resin variability problem, or a combination of both. These items are addressed in Appendix C of the RPP-WTP Research and Technology Plan[(2)], test scoping statements S-105 and S-106.

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[6] The Test Specification for this work required a product effluent at 1.75E-5 Ci ^{137}Cs/mole Na based on a maximum 20 wt% waste Na_2O loading to meet LAW vitrification criteria, with a corresponding DF of 2200. The minimum waste Na_2O loading is 14 wt% and corresponds to 2.5E-5 Ci ^{137}Cs/mole Na and a DF of 1552. The DF calculation makes the following assumptions: all Na comes from the tank waste, the glass density is 2.66 g/mL, the waste Na concentration is 5 M, and the waste contains 194 µCi/mL ^{137}Cs.

[7] Kurath, D. E., D. L. Blanchard, and J. R., Bontha, 2000. *Small Column Ion Exchange Testing or Superlig 644 for Removal of ^{137}Cs from Hanford Tank waste Envelope A (Tank 241-AW-101)*, BNFL-RPT-014, Rev. 0, PNWD-3001.

Terms and Abbreviations

16/18	SL-644 resin batch 981112YK-N3-16/18
–73	SL-644 resin batch 010319SMC-IV-73
ASR	Analytical Services Request
AV	apparatus volume
BV	bed volume
C/C_0	analyte concentration in column effluent divided by analyte concentration in feed
CMC	Chemical Measurements Center
DF	decontamination factor
DI	deionized
EDTA	ethylenediaminetetraacetic acid
GEA	gamma energy analysis
IC	ion chromatography
ICP-AES	inductively coupled plasma/atomic emission spectrometry
ICP-MS	inductively coupled plasma/mass spectrometry
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
NPT	National Pipe Thread
NTA	nitrilotriacetic acid
PNWD	Battelle Pacific Northwest Division
PSD	particle size distribution
TIC	total inorganic carbon
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-activity fractions through specific pretreatment processes. The current flowsheet for pretreating the Hanford high-level tank wastes includes the use of Cs-selective SuperLig® 644 (SL-644) resin for ^{137}Cs removal from the aqueous waste fraction. IBC Advanced Technologies, Inc., American Fork, UT, developed and supplied this material.

This report describes the results of batch distribution contacts of aged SL-644 (batches 644BZ and 981112YK-N3-16/18) and freshly produced SL-644 (010319SMC-IV-73). It also presents results from small column testing of two production batches, 981112YK-N3-16/18 and 010319SMC-IV-73, of the SL-644 ion exchange material. The effect of particle size on the latter batch was also evaluated using the dry-sieved portion 212- to 425-μm particle size. In all cases, the sample processed was simulated AW-101 Hanford tank waste (Golcar, et al. 2000) diluted to nominally 5 M Na.

The objectives of this work were to:

- determine if the Cs exchange capacity of SL-644 resin has decreased after storage in contact with air for approximately two years.[8]
- demonstrate the ion exchange system and resin performance for ^{137}Cs decontamination from tank waste simulant for subsequent use on actual tank waste.[9]
- develop loading and elution breakthrough profiles with the AW-101 simulant.

[8] Test Specification: "Evaluating SuperLig 644 Storage on Exchange Capacity and Effectiveness of Up Flow Elution," TSP-W375-00-00034, M. E. Johnson, CH2M HILL Hanford Group, Dec. 6, 2000 and Test Plan "Evaluation of SuperLig 644 Storage on Exchange Capacity and Effectiveness of Up Flow Elution," TP-PNNL-WTP-044, Rev. 0, S. K. Fiskum Feb. 5, 2001.

[9] Test Specification: "Tank 241-AN-102 and 241-AP-101 Ion Exchange," TSP-W375-00-00028, Rev. 1, M. E. Johnson, CH2M HILL Hanford Group, Dec. 11, 2000 and Test Plan "Actual Waste Ion Exchange Testing for the RPP-WTP Project," CHG-TP-41500-013, Rev. 0, D. L. Blanchard, Jr., Jan. 24, 2000.

2.0 Experimental

2.1 SL-644 Resin Properties

The SL-644 batch, 981112YK-N3-16/18 (hereafter called 16/18), was prepared by IBC Advanced Technologies Inc. (IBC) in November 1998. It had been stored in a plastic bottle and periodically opened to allow air to enter/exit and tumbled to mix the resin in an effort to mimic non-airtight storage conditions. Another batch, 644BZ, also prepared by IBC, was reported to be over 2 years old[10] and had been used previously in waste testing (Kurath et al. 1999, Kurath et al. 2000). The 16/18 batch and 644BZ batch were provided to Battelle via CH2MHILL Hanford Group and BNFL, Inc., respectively. Production batch 010319SMC-IV-73 (hereafter called –73), prepared by IBC in March 2001, was obtained directly from the manufacturer. The 16/18 batch material exhibited a distinct salt and pepper appearance in the as-received form. The –73 batch material exhibited a reddish-black appearance, peppered lightly with light-brown specks. The 644BZ batch material appeared completely soot black. Aliquots of the 16/18 and –73 resin batches were sieved through 18, 30, 40, 50, 70, 100, and 140 sieve screens to characterize the particle-size distributions (PSDs). The PSD of the –73 batch material expanded in a solution of 3 M NaOH-2 M $NaNO_3$-0.1 M KNO_3 was determined using a Coulter® LS particle-size analyzer and reported by personnel at IBC.

The as-received bulk density of SL-644 was determined by weighing approximately 4 g into a 10-mL graduated cylinder and measuring the volume. The H-form (acid form) of the resin was obtained by contacting the as-received resin with 3 sequential contacts of 0.5 M HNO_3 in a 1:10 volume ratio of resin to acid. This was followed with successive contacts with deionized (DI) water to neutral pH and air-drying to constant weight. The Na-form of the resin was obtained by contacting about 0.2-g of H-form resin with three sequential contacts of 5-mL of 1 M NaOH followed by DI water rinses until excess base was removed. In all cases, solutions were decanted through tared filter paper to determine and correct for lost fines. The Na-form resin was then allowed to dry to constant mass at room temperature under vacuum in an effort to minimize oxidation. The F-factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger. This was obtained by drying approximately 0.5 g resin under vacuum at 50^{o}C to constant mass. The F-factor was determined on the H-form and the as-received form 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).[11] Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.[12]

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[10] IBC reported in a telephone conversation that they could not identify the date SL-644 batch 644BZ was produced, but did confirm it was produced prior to 1998.

[11] After initial drying at ambient temperature under vacuum to constant mass, the resin was heated to 50^{o}C. The heated product appeared (visual inspection) to have degraded thus potentially nullifying subsequent mass measurements.

[12] Test Instruction TI-PNNL-WTP-020, Rev. 0, "Batch Contact Test Instructions for Various SL-644 Production Batches with Envelope A Simulant," S. K. Fiskum, 12/14/00; TI-RPP-WTP-065, Rev. 0, "Batch Contact Test Instructions for Two SL-644 Production Batches with AW-101 Simulant," S. K. Fiskum, 3/19/01; and TI-RPP-WTP-065, Rev. 1, "Batch Contact Test Instructions for Two SL-644 Production Batches with AW-101 Simulant," S. K. Fiskum, 4/3/01.

2.2 AW-101 Simulant Feed Preparation

Several batches of AW-101 Hanford tank waste simulant solutions were prepared as previously described (Golcar et al. 2000). After preparation of the first batch of simulant, the preparation recipe was altered by reducing the manganese addition, better reflecting the final required concentration of 6.6E-5 M. The nominal feed composition is shown in Table 2.1. The different tests identifying specific AW-101 simulant preparations are shown in Table 2.2. Appendix A shows the component materials and masses for each feed preparation batch. All preparations were filtered before use. Aliquots of Feeds 1, 4, and 5 were submitted for analysis by inductively-coupled plasma-atomic emission spectrometry (ICP-AES), ion chromatography (IC), inductively-coupled plasma-mass spectrometry (ICP-MS), total organic carbon (TOC) and total inorganic carbon (TIC).

Table 2.1. Targeted AW-101 Simulant Feed Composition

Cations, M		**Anions, M**	
Na	5.0 E+0	Cl	6.9 E-2
Al	5.1 E-1	F	1.1 E-2
K	4.3 E-1	CO_3	1.0 E-1
Ba	1.3 E-4	NO_2	7.9 E-1
Ca	4.1 E-4	NO_3	1.5 E+0
Cs	6.4 E-5	PO_4	1.7 E-3
Fe	5.0 E-5	SO_4	2.4 E-3
Li	5.5 E-4	OH	1.9 E+0
Mg	1.5 E-3	**Organics, M**	
Mn	6.6 E-5	EDTA	3.7 E-3
Mo	2.9 E-4	Citrate	3.7 E-3
Ni	1.3 E-4	Gluconate	3.7 E-3
Rb	1.0 E-5	Nitrilotriacetic acid	3.7 E-3
Si	2.9 E-3	Iminodiactic acid	3.7 E-3
Sr	1.3 E-5	TOC(1)	1.86 g C/L
Mole Ratios		**Density, g/mL**	
Na/Cs	7.81 E+4	Density	1.23
K/Cs	6.72 E+3		
(1) TOC = total organic carbon			

Table 2.2. AW-101 Preparation and Associated Test

Test Identification	AW-101 Feed ID	Test Start Date
Batch Contact Testing, Resin Batch		
981112YK-N3-16/18 and 644BZ	Feed 1	12/28/00
981112YK-N3-16/18 and 010319SMC-IV-73 as-received	Feed 5	4/10/01
981112YK-N3-16/18 and 010319SMC-IV-73 212- to 425-µm	Feed 4	3/28/01
Column Ion Exchange Testing, Resin Batch		
981112YK-N3-16/18	Feed 2	2/19/01
010319SMC-IV-73 as-received	Feed 5	4/9/01
010319SMC-IV-73 212- to 425-µm	Feed 4	3/27/01

2.3 Batch Contacts

Duplicate aliquots of the AW-101 simulant subsamples were prepared at three Cs concentrations. Aliquots were spiked with additional 0.1 M $CsNO_3$ stock solution, increasing the Cs concentrations as shown in Table 2.3. Batch contacts were performed with resin in the as-received form and the H-form. The Cs distribution was monitored using ^{137}Cs tracer.

Table 2.3. Targeted Cs Concentrations in the AW-101 Simulant Used for the Batch Distribution Tests

Solution	Targeted Cs Molarity	Targeted Na/Cs Mole Ratio
AW-101	6.4 E-5	7.8 E+4
AW-101 Cs Spike 1	1.0 E-3	5.0 E+3
AW-101 Cs Spike 2	5.0 E-3	1.0 E+3

The batch distribution tests were performed at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.1 g of SL-644 exchanger was contacted with 10 mL of AW-101 simulant. The exchanger mass was determined to an accuracy of 0.0002 g. The simulant waste volume was transferred by pipet; the actual volume was determined by mass difference and solution density. Agitation was provided by a reciprocal shaker for approximately 24 h. The temperature was not controlled, but was generally constant at 24 to 28 ^{o}C during the contact period. After contact, the samples were filtered through 0.2-µm nylon membrane syringe filters. Sample-specific volumes and resin masses are given in Appendix B.

All batch distribution contacts were performed in duplicate. Simulant-only samples (uncontacted aliquots) were used to determine the initial Cs (ICP-MS), Na, and K (ICP-AES) concentrations. All solutions were analyzed by gamma energy analysis (GEA) to determine the ^{137}Cs concentration. Final Cs concentrations were calculated relative to the recovered ^{137}Cs tracer. The final Na and K concentrations were determined with ICP-AES on the first batch contact test (as-received resin form), only.

2.4 Ion Exchange Column System

A schematic of the ion exchange column system is shown in Figure 2.1. The system consisted of two small columns containing the ion exchange material, a small metering pump, three valves, a pressure gauge, and a pressure relief valve. Valves 1, 2, and 3 are three-way valves that could be turned to the flow position, sample position, or no-flow position. Valve 1 was placed at the outlet of the pump and was used to eliminate air from the system, purge the initial volume of the system, or isolate the columns from the pump. Valves 2 and 3 were primarily used for obtaining samples and could also be used to isolate the columns from the rest of the system. 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 Savannah River Technology Center 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 fittings to accommodate ¼-in stainless steel National Pipe Thread (NPT) fittings. 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. A decal millimeter scale affixed to the column allowed for measurement of resin bed height and thus shrinkage and swelling. 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 1/8-in OD, 1/16-in ID polyethylene. Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column switching. A FMI QVG50 pump (Fluid Metering, Inc., Syosset, New York) equipped with a ceramic and Kynar® coated low-flow piston pump head was used for all fluid introduction. The flow rate was controlled with a remotely operated FMI stroke-rate controller. The pump was calibrated with the stroke-rate controller and could provide pumping rates from 0.08 to16 mL/min. The volume actually pumped was determined using the mass of the fluid and the fluid density. The pressure indicated on the pressure gauge remained below 5 psi during all runs. The 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.

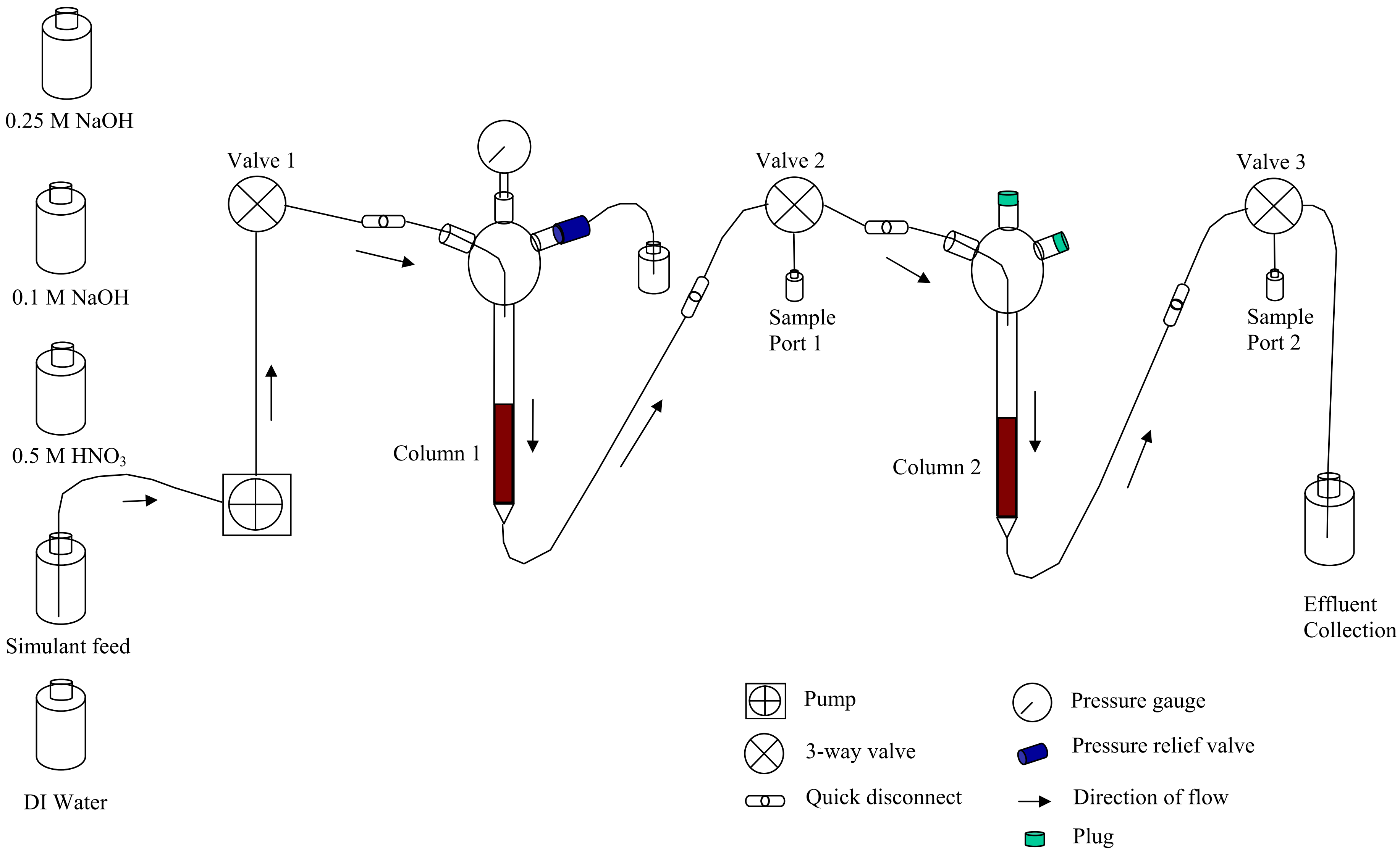


Figure 2.1. Cesium Ion Exchange Column System

2.5 Column Run Experimental Procedure and Conditions

Three complete Cs ion exchange tests were performed. The initial SL-644 resin and bed preparations were performed using a procedure similar to that recommended by Hassan et al. (1999). Before packing the resin beds, aliquots of SL-644 resin, given in Table 2.4, were placed in separate beakers. The two resin aliquots were soaked in about 40 mL of 1 M NaOH (~10:1 solution to resin volume ratio) with light agitation for 2 h. The NaOH was decanted, and the SL-644 was slurried with about 40 mL of DI water. The resin was then quantitatively transferred into the columns using additional DI water to aid the transfer.

Table 2.4. SL-644 Resin Masses

	Batch ID		
	010319SMC-IV-73	**010319SMC-IV-73**	**981112YK-N3-16/18**
Particle size distribution	212- to 425-μm	As-received	As-received
Lead column resin mass(1)	4.17 g	3.43 g	2.50 g
Lag column resin mass(1)	4.20 g	3.42 g	2.50 g
Run date	3/27/01	4/9/01	2/19/01
(1) Resin was weighed in the as-received form.			

All solutions were transferred downflow through the ion exchange column. The experimental conditions for each process step of the three separate runs are shown in Table 2.5 through Table 2.7. The bed volumes (BV) and flow rates (BV/h) relate to the bed size in the regeneration condition (0.25 M NaOH). All processing was performed at ambient temperature, nominally 24°C. The ion exchange system was separated at the third quick disconnect, and a pump was connected to the second column such that each column could be conditioned individually. The resin was initially washed with 0.5 M HNO_3 followed by DI water and regenerated with 0.25 M NaOH.

Four different tests (labeled A-D, Table 2.5) were conducted with the 16/18 resin ion exchange setup. Various conditioning processes were explored in an attempt to improve Cs ion exchange performance. The 16/18 resin batch was pre-conditioned with 6 BVs of 0.5 M HNO_3, 2 apparatus volumes (AVs) of DI water, then 7 BVs of 0.25 M NaOH. A simple simulant was processed (Test A) resulting in early Cs breakthrough. The 0.25 M NaOH conditioning step was increased from 6 BVs to 12 BVs prior to processing the AW-101 simulant (Test B). This change did not improve performance and the standard method of conditioning with nominally 6 BVs 0.25 M NaOH was resumed. A long (~80 BVs) 0.5 M HNO_3 wash was applied, following Test B elution, in an attempt to improve subsequent Cs load performance. The simple simulant was then processed (Test C) showing significant improvement in Cs load behavior. The long acid wash was repeated following Test C, then the AW-101 simulant (Test D) was processed. The best performance obtained with the 16/18 resin batch material (Test D), defined in Table 2.5 under the sub-heading *Detail of Reported Run*, is used for comparison to the –73 resin performance in this report.

The loading, feed displacement, and DI water rinse steps were conducted by passing the solutions through both resin beds connected in series. Nominally 150 BVs of AW-101 simulant were prepared for loading, however, only 116 BVs were processed for the 16/18 resin batch experiment (Test D). Small samples (about 2 mL) were collected from the lead and lag columns starting at 5 BV, then at nominal 10 BV increments thereafter. The effluent was collected as a single composite. The effluent from the as-received particle size –73 resin was first collected in a bottle until close to 150 BVs were collected, and then a second effluent collection bottle was used to collect the remainder. The 0.1 M NaOH was used as feed displacement, and deionized (DI) water was used to rinse the NaOH solution through the system. The feed displacement and DI water rinses were collected individually as composite fractions.

The elution and elution-rinse steps were conducted on both columns separately. The Cs was eluted with 0.5 M HNO_3 and samples were collected in 1-BV increments. Elution continued until C/C_o (effluent Cs concentration divided by the feed Cs concentration) was below 1%. The nitric acid solution was displaced with nominally three AVs of DI water.

Typically a nominal 3-cm solution height was maintained above the resin beds. The solution above the lead resin bed was drained to within about 1-cm of the resin surface when the solution feed was changed. Flow through the column was then temporarily stopped, and a nominal 3-cm head of feed was allowed to build up above the resin bed. This helped minimize reagents mixing above the lead resin bed, thus speeding the reagent transition in the column system.

Table 2.5. Experimental Conditions for SL-644 Batch 981112YK-N3-16/18[(1)]

Process step	Solution	Average Total Volume[(2)]			Average Flow rate[(2)]		Time
		BV[(3)]	AV[(4)]	mL	BV/h	mL/min	h
Resin Pre-conditioning; Columns in Parallel							
Bed conditioning	0.5 M HNO_3	3.5	-	33	2.7	0.42	1.3
Bed conditioning	DI water	-	3.7	81	2.8	0.44	3.1
Bed conditioning	0.25 M NaOH	7.7	-	72	3.6	0.56	2.1
Test A Load, Feed Displacement, Rinse; Columns in Series							
Loading	Simple simulant[(5)]	13.5	-	127	2.6	0.41	5.2
Feed displacement	0.1 M NaOH	-	2.9	134	2.8	0.49	4.6
DI water rinse	DI water	-	1.7	79	3.1	0.45	2.9
Resin Elution; Columns in Parallel							
Elution	0.5 M HNO_3	18.1	-	170	1.8	0.28	10.1
Eluent rinse	DI water	-	3.1	71	2.9	0.46	2.6
Resin Conditioning; Columns in Parallel							
Bed conditioning	0.25 M NaOH	12	-	110	3.1	0.49	3.7
Test B Load, Feed Displacement, Rinse; Columns in Series							
Loading	AW-101 Feed	44	-	418	3.0	0.46	15
Feed displacement	0.1 M NaOH	-	3.6	149	3.3	0.51	4.9
DI water rinse	DI water	-	1.7	80	3.0	0.47	2.8

Table 2.5 (contd)

Process step	Solution	Average Total Volume(2)			Average Flow rate(2)		Time
		BV(3)	AV(4)	mL	BV/h	mL/min	h
Resin Elution; Columns in Parallel							
Elution	0.5 M HNO_3	7	-	64	0.84	0.13	8.2
Wash	0.5 M HNO_3	73	-	688	3.0	0.47	24
DI water rinse	DI water	-	3.3	77	3.0	0.47	2.6
Resin Conditioning; Columns in Parallel							
Bed conditioning	0.25 M NaOH	6.6	-	61	3.2	0.50	2.0
Test C Load, Feed Displacement, Rinse; Columns in Series							
Bed conditioning	0.25 M NaOH	2.8	-	26	4.0	0.63	0.7
Loading	Simple simulant(5)	25	-	234	2.9	0.46	8.6
Feed displacement	0.1 M NaOH	-	2.9	132	3.1	0.49	4.5
DI water rinse	DI water	-	1.8	82	2.8	0.44	3.1
Resin Elution; Columns in Parallel							
Elution	0.5 M HNO_3	26	-	245	4.0	0.62	6.9
Wash	0.5 M HNO_3	51	-	480	4.1	0.65	12.5
DI water rinse	DI water	-	3.0	68	3.0	0.46	2.5
Detail of Reported Run							
Process step	**Solution**	**Total Volume**			**Flow rate**		**Time**
		BV(3)	**AV(4)**	**mL**	**BV/h**	**mL/min**	**h**
Lead Column Resin Conditioning							
Bed conditioning	0.25 M NaOH	5.6	-	53	3.0	0.47	1.9
Lag Column Resin Conditioning							
Bed conditioning	0.25 M NaOH	5.4	-	51	2.9	0.45	1.9
Test D Load, Feed Displacement, Rinse; Columns in Series (6)							
Bed conditioning	0.25 M NaOH	5.6	-	53	2.8	0.44	2.0
Loading lead column	AW-101 Feed	116	-	1093	2.9	0.46	39
Loading lag column(7)	AW-101 Feed	114	-	1067	2.9	0.46	39
Feed displacement	0.1 M NaOH	-	3.1	132	3.1	0.48	4.5
DI water rinse	DI water	-	1.8	85	3.0	0.46	3.0
Lead Column Resin Elution							
Elution	0.5 M HNO_3	19	-	175	1.2	0.19	16
Wash	0.5 M HNO_3	64	-	600	3.0	0.46	21.5
Eluant rinse	DI water	-	3.2	75	2.9	0.46	2.7
Lag Column Resin Elution							
Elution	0.5 M HNO_3	18	-	167	1.1	0.18	15
Wash	0.5 M HNO_3	62	-	579	2.8	0.44	22
Eluant rinse	DI water	-	3.4	72	2.8	0.44	2.7

(1) Test Instruction TI-PNNL-WTP-006, Rev. 1 (2/15/01) and Rev. 2 (2/23/01), "Performance Test of Dual Column SuperLig 644 Cesium Ion Exchange System Using AW-101 Waste Simulant (Envelope A)," S. K. Fiskum.
(2) The average volumes and flow rates through the lead and lag columns are given.
(3) Bed volumes: lead 9.5 mL and lag 9.2 mL (0.25 M NaOH condition); resin masses: lead and lag columns 2.50 g (as-received form).
(4) Apparatus volumes: 42 mL 2 columns in series; 23 mL for the lead and 21 mL for the lag columns, separately.
(5) Simple simulant composition is 2 M NaOH-1.5 M $NaNO_3$-1.5 M $NaNO_2$-7.5E-5 M Cs.

(6) Process run began 2/28/01.
(7) The feed volume through the lag column is reduced because of sampling from the lead column.

Table 2.6. Experimental Conditions for SL-644 010319SMC-IV-73, As-Received Particle Size[1]

Process step	Solution	Total Volume			Flow rate		Time
		BV[2]	AV[3]	mL	BV/h	mL/min	H
Lead Column Resin Pre-conditioning							
Bed conditioning	0.5 M HNO_3	7.2	-	60	2.6	0.36	2.7
Bed conditioning	DI water	-	3.2	70	2.6	0.36	3.2
Bed conditioning	0.25 M NaOH	5.4	-	45	2.6	0.35	2.1
Lag Column Resin Pre-conditioning							
Bed conditioning	0.5 M HNO_3	8.4	-	69	3.1	0.42	2.7
Bed conditioning	DI water	-	3.2	67	2.4	0.34	3.3
Bed conditioning	0.25 M NaOH	5.1	-	42	2.4	0.33	2.1
Load, Feed Displacement, Rinse; Columns in Series [4]							
Bed conditioning	0.25 M NaOH	0.7	-	6	2.6	0.35	0.3
Loading Lead column	AW-101 Feed	232	-	1929	2.8	0.38	81.6
Loading Lag column[5]	AW-101 Feed	226	-	1874	2.8	0.38	81.6
Feed displacement	0.1 M NaOH	-	3.0	125	2.8	0.38	5.4
DI water rinse	DI water	-	2.0	84	3.3	0.46	3.6
Lead Column Resin Elution							
Elution	0.5 M HNO_3	19	-	159	1.2	0.17	16.0
Eluant rinse	DI water	-	2.9	67	2.7	0.37	3.0
Lag Column Resin Elution							
Elution	0.5 M HNO_3	17	-	142	1.1	0.15	16.0
Eluant rinse	DI water	-	3.1	66	2.7	0.37	3.0

(1) Test Instruction TI-PNNL-WTP-006, Rev. 5, "Performance Test of Dual Column SuperLig 644 Cesium Ion Exchange System Using AW-101 Waste Simulant (Envelope A)," S. K. Fiskum, 4/3/01.
(2) Bed volumes: lead 8.4 mL and lag 8.2 mL (0.25 M NaOH condition); resin masses: lead 3.43 g and lag 3.42 g (as-received form).
(3) Apparatus volume: 42 mL for two columns in series, 23 mL for lead and 21 mL for the lag columns individually.
(4) Process run began 4/9/01.
(5) The feed volume through the lag column is reduced because of sampling from the lead column.

Table 2.7. Experimental Conditions for SL-644 Batch 010319SMC-IV-73, 212- to 425-µm Particle Size[1]

Process step	Solution	Total Volume			Flow rate		Time
		BV[2]	AV[3]	mL	BV/h	mL/min	H
Lead Column Resin Pre-conditioning							
Bed conditioning	0.5 M HNO_3	13.8	-	152	0.88	0.16	15.7
Bed conditioning	DI water	-	3.0	69	2.14	0.39	2.9
Bed conditioning	0.25 M NaOH	5.2	-	57	2.1	0.38	2.5
Lag Column Resin Pre-conditioning							
Bed conditioning	0.5 M HNO_3	13.3	-	146	0.85	0.16	15.7
Bed conditioning	DI water	-	3.1	66	2.1	0.38	2.9
Bed conditioning	0.25 M NaOH	5.0	-	55	2.0	0.36	2.5
Load, Feed Displacement, Rinse; Columns in Series [4]							
Bed conditioning	0.25 M NaOH	3.7	-	41	1.9	0.35	1.9
Loading Lead column	AW-101 Feed	143	-	1576	2.6	0.47	53
Loading Lag column[5]	AW-101 Feed	140	-	1538	2.6	0.47	53
Feed displacement	0.1 M NaOH	-	2.6	121	2.5	0.46	4.4
DI water rinse	DI water	-	1.8	84	2.5	0.46	2.9
Lead Column Resin Elution							
Elution	0.5 M HNO_3	12.5	-	133	0.92	0.17	13.3
Eluant rinse	DI water	-	2.6	60	2.6	0.48	2.1
Lag Column Resin Elution							
Elution	0.5 M HNO_3	12.3	-	131	0.91	0.17	13.3
Eluant rinse	DI water	-	2.7	57	2.5	0.46	2.1

(1) Test Instruction TI-PNNL-WTP-006, Rev. 4, "Performance Test of Dual Column SuperLig 644 Cesium Ion Exchange System Using AW-101 Waste Simulant (Envelope A)," S. K. Fiskum, 3/23/01.
(2) Bed volumes: lead 11.2 mL and lag 10.8 mL (0.25 M NaOH condition); resin masses: lead 4.17 g and lag 4.20 g (as-received form).
(3) Apparatus volume: 42 mL for columns in series; 23 mL for lead and 21 mL for the lag columns individually.
(4) Process run began 3/27/01.
(5) The feed volume through the lag column is reduced because of sampling from the lead column.

The sampling and analysis protocol is summarized in Table 2.8. The ^{137}Cs content was determined in most of the samples using a benchtop GEA spectrometer. This allowed for near real-time monitoring of Cs load and elution. The eluate samples from the 212- to 425-µm particle size resin were composited, and the composite was subsampled for GEA, ICP-AES, TOC, and IC analyses.

The sodium and other metal concentrations were determined with ICP-AES. The OH^- concentration was determined by potentiometric titration with hydrochloric acid. Anions were determined using IC. Total organic carbon was determined using hot persulfate wet oxidation in conjunction with a CO_2 trap.

Table 2.8. Sampling Interval and Analyses

Process Step	Lead Column BV	Lag Column BV	Approximate Sample Size, mL	Analysis
0.5 M HNO_3 conditioning	(1)	(1)	(1)	(1)
DI water conditioning	(2)	(2)	(2)	(2)
0.25 M NaOH conditioning	(3)	(3)	(3)	(3)
Loading	Every 10 BV	Every 10 BV	2	GEA(4)
Feed displacement	-	1 composite		GEA
DI water rinse	-	1 composite		GEA
Elution	Every 1 BV	Every 1 BV	8-10 (1 BV)	GEA
Elution composite				ICP-AES, IC, TOC, GEA
Eluant rinse	1 composite	1 composite		GEA

(1) Preconditioning 0.5 M HNO_3 effluent samples were taken in 1-BV increments from the lead and lag columns for the 16/18 resin test only. These were analyzed by ICP-AES.
(2) The preconditioning water rinse effluent sample was taken as a composite for the 16/18 resin test only, and analyzed by ICP-AES
(3) The preconditioning 0.25 M NaOH effluent samples were taken as composites for the 16/18 resin test only, and analyzed by ICP-AES.
(4) Selected samples were also analyzed by ICP-AES.

3.0 Results and Discussion

3.1 SL-644 Resin Properties

The dry-sieved PSDs for the as-received SL-644 resins are provided in Table 3.1. The 644BZ (Kurath et al. 2000) and 981020mb48-563 (Hassan et al. 2000) batch PSDs are also shown for comparison. A large mass fraction of both the 16/18 and –73 resins consisted of particles between 425 and 1000 µm. Most of the mass fraction of the as-received 644BZ batch contained particles below 425 µm in diameter. The 981020mb48-563 material is consistent with the 16/18 resin in particle size mass fractions. The expanded resin particle size will be higher yet.

Table 3.1. Dry Particle-Size Weight-Percent Distribution of Various Batches of As-Received SL-644

Sieve Size(1)	Particle Size (µm)	981112YK-N3-16/18 wt %	010319SMC-IV-73 wt %	Sieve Size(1)	Particle Size (µm)	644BZ wt %	Sieve Size(1)	Particle Size (µm)	981020mb48-563 wt %
18	>1000	1.78	0.06	18		NM	10	>2000	0
30	600-1000	55.25	37.27	30		NM	30	600-2000	57.3
40	425-600	20.12	38.23	40	>425	9.9	40	425-600	23.7
50	300-425	13.76	18.01	70	212-425	88	50	300-425	13.7
70	212-300	7.93	6.08				70	212-300	5.1
100	150-212	1.11	0.26	100	150-212	2.1	80	180-212	0.11
140	106 -150	0.02	0.06	200	74-150	0		<180	NM
	<106	0.03	0.03		<74	0			

(1) U. S. standard sieve size corresponds to ASTM E-11 specification.
(2) NM = not measured.

The expanded volume percent PSD of batch -1-319SMC-IV-73, as reported by IBC, is shown in Figure 3.1. Fifteen percent by volume of this material exceeds a particle size of 948 µm in the test solution (3 M NaOH + 2 M $NaNO_3$ + 0.1 M KNO_3). Over 50% of the expanded resin volume is represented by particles exceeding 700 µm. The expanded resin particle size is expected to be similar to the expanded resin particle size in tank waste and tank waste simulant matrices when diluted to 5 M Na.

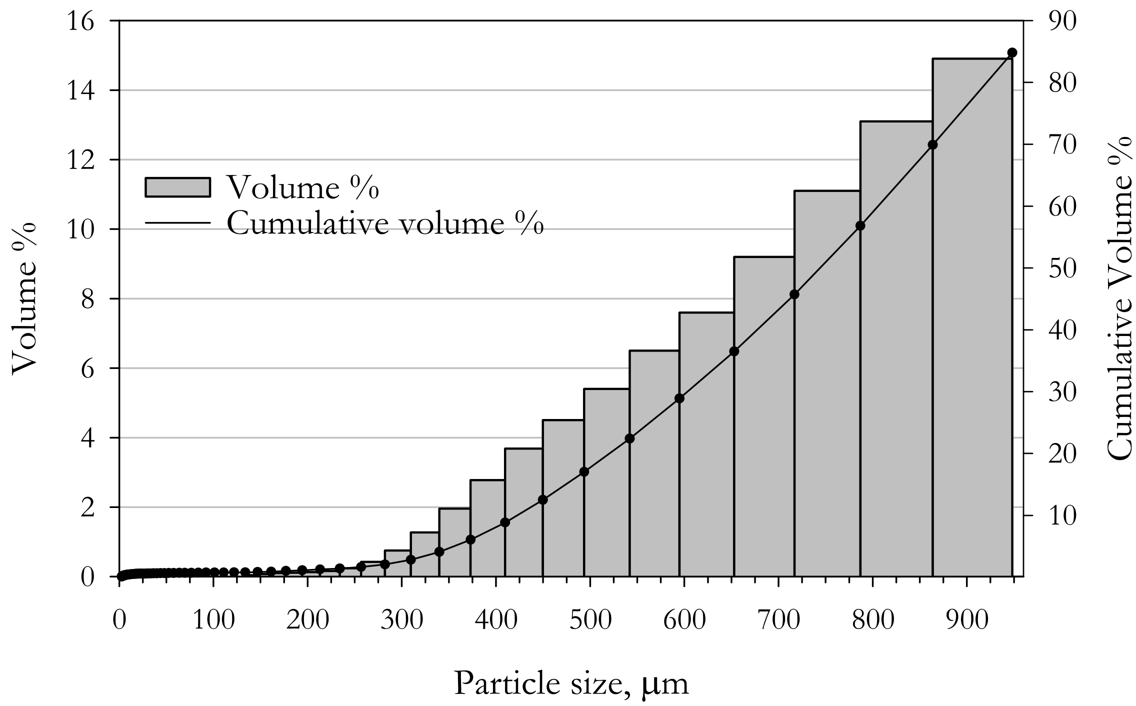


Figure 3.1. SL-644 Resin 010319SMC-IV-73, As-received Particle Size Expanded in 3 M NaOH + 2 M $NaNO_3$ + 0.1 M KNO_3 (15 volume percent is greater than 948 μm, the largest diameter quantified), IBC 3/19/01 (Appendix B)

Various properties of the SL-644 resins are shown in Table 3.2. The L-factor indicates the loss in mass as a result of acid washing (corrected for water loss).[13] It is determined according to the following equation:

$$L = \frac{(m_H * F_H)}{(m_i * F)} \quad \textbf{(1)}$$

where L = mass of dry H-form resin/g dry as-received resin, where drying is conducted at 50°C under vacuum

m_H = final H-form resin mass

F_H = mass of dry H-form resin/mass of H-form resin

m_i = initial mass of the as-received form of resin

F = mass of dry as-received resin/mass of as-received resin.

[13] The mass loss is largely attributed to K removal from the SL-644 exchange sites as well as to inactive materials and K salt(s). A carbonate salt is implicated because of the effervescence noted during acid washing.

The mass increase factor, I_{Na}, defines the mass increase upon conversion from the dry hydrogen form to the sodium form and is calculated according to the following equation:

$$I_{Na} = \frac{m_{Na}}{m_H * F_H} \qquad \textbf{(2)}$$

where I_{Na} = dry Na-form resin mass (dried under vacuum, ambient temperature)/dry H-form resin mass

m_{Na} = vacuum-dried mass of the Na-form resin.

The Na-form dry-bed resin densities, ρ, were obtained according to the following equation:

$$\rho = \frac{m * F * L * I_{Na}}{BV} \qquad \textbf{(3)}$$

where ρ = dry Na-form resin mass/BV in column

m = as-received resin mass, loaded in the ion exchange column

BV = resin bed volume, determined from column tests discussed in Section 3.4.5

F, L, and I_{Na} have already been defined.

The dry bed density for the H-form resin (0.5 M HNO_3) was calculated according to Equation 3 with the omission of the I_{Na} factor.

Table 3.2. Physical Properties of Various Batches of SL-644

Batch ID	**010319SMC-IV-73**	**010319SMC-IV-73**	**981112YK-N3-16/18**
Particle size distribution	212- to 425-μm	As-received	As-received
Bulk density, as-received form	0.74 g/mL	0.84 g/mL	0.80 g/mL
F-factor (for water loss)	0.877	0.871	0.891
L-factor (solids fraction remaining after conversion to H-form)	0.538	0.556	0.493
I_{Na} factor (mass increase from H-form to Na-form)	1.25	1.22	1.29
Dry bed density in feed	**g/mL**	**g/mL**	**g/mL**
0.25 M NaOH	0.22	0.24	0.15
AW-101 Simulant	0.24	0.26	0.19
0.5 M HNO_3	0.22	0.24	0.20

The mass increase from the dry H-form to the dry Na-form was 22 to 29%. If the Na-form conversion caused all available exchange sites to be converted from H to Na, then the total ionic capacity can be calculated according to the following derivation:

$$Na\ (g) + H\ form\ (g) = Na\ form\ (g)$$

$$Na\ (g) = Na\,form\ (g) - H\,form\ (g)$$

$$Na\ (g) = Na\,form\ (g) - \frac{Na\,form\ (g)}{I_{Na}}$$

$$\frac{Na\ (g)}{Na\,form\ (g)} = 1 - \frac{1}{I_{Na}}$$

Thus for the –73 resin, 212- to 425 µm particle size, the ionic capacity for Na can be estimated according to the following equation as 8.7 mmoles Na per gram of Na-form resin.

$$\frac{Na\ (g)}{Na\ \ form\ (g)} = 1-\left(\frac{1}{1.25}\right) = \frac{0.2\,g\ \ Na}{g\ \ Na\,form} = \frac{0.2\,g\,Na / 23\,g/mole}{g\ \ Na\,form} = \frac{8.7\ mmoles\ Na}{g\ \ Na\ \ form}$$

The I_{Na} factor may be biased high if drying of the Na-form resin was incomplete (ambient temperature under vacuum). If the Na-form was incompletely dried, then I_{Na} will be lower and the calculated ionic capacity will be lower.

The dry bed densities of the smaller particle-size and as-received particle size resin are approximately equivalent. The magnitude differences in the densities of the 16/18 and –73 as-received batches are varied, reflective of the much greater volume changes as a function of feed matrix associated with the 16/18 batch. The density change for the 16/18 batch from the regeneration to feed conditions is similar to the volume changes (and by inference, density changes) found with batch 644BZ (Kurath et al. 2000). The 0.25 M NaOH dry bed densities are equivalent to the 0.5 M HNO_3 form dry bed densities for the –73 resin. In these cases, the decrease in mass associated with the change from the Na-form to the H-form was equivalent to the decrease in volume upon contracting from the Na-form to the H-form.

The average expanded-particle size of the dry-sieved 212- to 425-µm fraction was estimated based on the resin expansion factor and volume percent PSD reported by IBC. The calculated volume of the dry particle range was multiplied by the expansion factor [dry bulk density (0.84 g/mL *0.871) divided by the dry bed density in 0.25 M NaOH (0.24 g/mL)]. The expanded particle size diameter range was then backcalculated. The cumulative volume percent results were applied to the calculated expanded particle size range. The average particle size of the 212- to 425-µm dry-sieved fraction corresponds to 540-µm diameter expanded in the 0.25 M NaOH. As a general rule, the column diameter should be 20 times greater than the average resin particle diameter to prevent wall-effects (Korkisch, 1989). Given the diameter of the column at 1.46 cm, the column diameter is 27 times the average diameter of the 212- to 425-µm diameter dry-sieved resin.

3.2 AW-101 Simulant Composition

Several batches of AW-101 simulant were prepared to support the batch contact and column ion exchange work. Compositions of the different AW-101 preparations are provided in Table 3.3. The relevant Chemical Measurements Center (CMC) Analytical Services Request (ASR) identifications are provided and the minimum reportable quantity (MRQ) is also shown. The data for Feeds 1, 4 and 5 are from analytical results; the composition for Feed 2 is calculated based on the simulant recipe.

The TOC and TIC results are reported for two different methods, furnace oxidation (F) and hot persulfate oxidation (P). The results between these two methods do not agree with each other but are consistent between batches. The variation in results between these two methods is a result of the ease or difficulty in which they oxidize carbonate and various organic constituents (Baldwin, Stromatt, and Winters 1994).

Table 3.3. Feed AW-101 Simulant Composition

Preparation ID	**Feed 1**	**Feed 2[1]**	**Feed 4**	**Feed 5**	
ASR ID	**6014**	**NA**	**6104**	**6106**	
CMC ID	**01-0414**	**NA**	**01-0973**	**01-1002**	**MRQ[5]**
Analyte	**μg/mL**	**μg/mL**	**μg/mL**	**μg/mL**	**μg/mL**
Al	12,700	13,700[3]	13,000	13,300	75
B	78.5	NA	158	155	NMRQ
Ba	16.6	18.3	18.1	17.1	2.3
Ca	<25	16.5	<31	<31	150
Cs	9.3	8.5	11.4	11.8	1.5
Fe	[8.1]	2.7	[8.9]	[4.8]	150
K	12,700	16,800[3]	16,300	19,400	75
Li	<3	3.8	[4.8]	<4	NMRQ
Mg	[4]	363	[19]	<13	150
Mn	80.6	3.6	<7	<7	150
Mo	[27]	27.5	[25]	24	90
Na	98,800	115,000[3]	118,000	104,000	75
Ni	[7.6]	7.7	[7.0]	[5.1]	30
P	[71]	53.4	[60]	[51]	600
Si	<50	82	[175]	[159]	170
Sr	[1.5]	1.1	<2	<2	NMRQ
F^-	600	210	310	470	150
Cl^-	2320	2460	2660	2590	3
NO_3^-	88,200	95,200	107,000	114,000	3000
NO_2^-	35,300	36,300	38,300	39,000	3000
PO_4^{-3}	<500	160	<250	<250	2500
SO_4^{-2}	<500	230	<250	<250	2300
TIC-P[2]	1410	6000	1390	1370	150
TIC-F[2]	2520	6000	2960	2560	150
TOC-P[2]	1720	5860	1695	1630	1500
TOC-F[2]	510	5860	320	610	1500
OH^-, M	2.02 M	2.3 M[3]	NM[4]	NM[4]	NMRQ
Density	1.238 g/mL	1.237 g/mL	1.234 g/mL	1.237 g/mL	
Na/Cs Mole ratio	6.1 E+4	7.8 E+4	6.0 E+4	5.1 E+4	

(1) Calculated concentration based on preparation records, not analyzed.
(2) TIC-P and TOC-P designate analysis using the hot persulfate method and TOC-F and TIC-F designate analysis using the furnace method.
(3) Actual values for Na, Al, K, and OH are probably lower because adsorbed water in the stock materials, KOH, $Al(NO_3)_3$-$9H_2O$, and NaOH, were not accounted for when weighed.
(4) NM = not measured
(5) MRQ = minimum reportable quantity; NMRQ = no minimum reportable quantity was specified
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 analyte is less than the instrument detection limit or less than the lowest calibration standard. The dilution-corrected instrument detection limit is reported.

3.3 Batch Distribution Coefficients

The as-received form of SL-644 resin batches 16/18 and 644BZ were contacted with the AW-101 Feed 1 preparation. The equilibrium Cs, Na, and K concentrations were determined and the results are summarized in Table 3.4. The nominal errors of the analytical methods are ± 15% (2-σ).

Table 3.4. Equilibrium Na, K, and Cs Concentration after Batch Contact(1) with SL-644 Batches 981112YK-N3-16/18 and 644BZ (as-received)

Batch Contact Test	CMC ID	Na, μg/mL	K, μg/mL	Cs, μg/mL	Cs K_d, mL/g	Cs K_d', mL/g(2)
Control/No resin						
Unspiked	01-0396	101,000	12,600	9.59	–	–
Unspiked duplicate	01-0397	95,400	12,000	9.68	–	–
Spike 1	01-0402	97,700	12,400	131	–	–
Spike 1 duplicate	01-0403	96,900	12,300	138	–	–
Spike 2	01-0408	102,000	12,900	658	–	–
Spike 2 duplicate	01-0409	97,500	12,200	642	–	–
Average		98,400	12,400	–	–	–
644BZ						
Unspiked/Feed condition	01-0398	98,200	13,800	1.06	862	1385
Unspiked/feed condition duplicate	01-0399	96,700	13,700	1.09	777	1248
Spike 1	01-0404	95,300	13,500	43.3	232	372
Spike 1 duplicate	01-0405	102,000	14,600	43.3	236	378
Spike 2	01-0410	97,500	13,800	418	60	125
Spike 2 duplicate	01-0411	96,000	13,500	417	62	129
Average		97,600	13,800	–	–	–
981112YK-N3-16/18						
Unspike/Feed condition	01-0400	97,700	13,800	1.30	620	979
Unspiked/feed condition duplicate	01-0401	98,700	14,000	1.43	585	922
Spike 1	01-0406	95,700	12,300	47.9	206	324
Spike 1 duplicate	01-0407	101,000	14,300	38.5	274	432
Spike 2	01-0412	103,000	14,700	452	49	100
Spike 2 duplicate	01-0413	94,400	13,300	442	51	104
Average		98,400	13,700	–	–	–

(1) AW-101 Simulant Feed 1
(2) K_d' is calculated with as-received exchanger mass mathematically corrected for the mass loss on washing and mass gain on conversion to the Na-form according to Equation 5.

The spread between the high (103,000 μg/mL) and low (94,400 μg/mL) Na concentrations is 9.1%. Within the error of the method, the Na concentration remains unchanged for the batch contacts. The spread from the high (14,700 μg/mL) and low (12,200 μg/mL) K concentration is 20%. The samples contacted with exchanger have, on average, 11% higher K concentrations (13,800 μg/mL) than the controls (12,400 μg/mL). In the nominal 10-mL volume of AW-101 simulant, the K concentration increase corresponds to 14 mg or 14% by weight of the resin mass. The increase in K concentration is attributable to K leaching out of the SL-644 material. The manufacturer supplied SL-644 in the K form; thus the mass loss from acid washing (see Table 3.2) is in part attributable to K loss.

The Cs batch distribution coefficient (K_d) values given in Table 3.4 were determined from the as-received form resin according to the standard formula shown in Equation 4.

$$K_d = \frac{(C_0 - C_1)}{C_1} * \frac{V}{M * F} \quad \textbf{(4)}$$

where C_0 = initial ^{137}Cs concentration
C_1 = final ^{137}Cs concentration
V = volume of the liquid sample (mL)
M = mass of the ion exchanger (g), as-received form
F = mass of the dried resin divided by the mass of the as-received resin.

This K_d value determination is biased low because the exchanger mass (M) is contaminated with other materials left over from the manufacturing process. Also, because SL-644 resin was contacted with the AW-101 simulant containing 5 M Na, the resin is presumed to convert to the Na-form. A more appropriate basis of exchanger mass is provided when starting from the H-form resin mass (impurities removed) and correcting for Na-form mass increase. The resin mass was mathematically converted to the Na-form mass for the K_d value calculations according to Equation 5.

$$K_d^1 = \frac{(C_0 - C_1)}{C_1} * \frac{V}{M * L * F * I_{Na}} \quad \textbf{(5)}$$

The K_d value based on the corrected mass is shown in Table 3.4 as K_d^1, demonstrating the significant mass correction effect on the K_d value determination.

Subsequent batch contacts were conducted using the H-form of the resin. The Cs distribution coefficients and best-fit curves for multiple batch contact tests and multiple resin batches are plotted in Figure 3.2 a-b and Figure 3.3 a-b. The K_d values were determined according to Equation 6.

$$K_d = \frac{(C_0 - C_1)}{C_1} * \frac{V}{m * F * I_{Na}} \quad \textbf{(6)}$$

where m = mass of the H-form of resin
F = mass of dried H-form resin/mass of H-form resin.

The data associated with each data point are found in Appendix B. The initial Na/Cs mole ratios were calculated based on the measured Na and Cs concentrations in the uncontacted simulant. The final Cs concentrations were calculated based on ^{137}Cs tracer recoveries. The Na^+ concentrations are assumed to remain constant during the batch contacts. The H^+ form of the resin contains 2.2 meq H^+ per gram (Rapko et al. 2002). Thus, the quantity of H^+ added with the resins is small relative to the moles of Na^+ in the contact solution (phase ratio of 100 mL of solution: gram of exchanger). In these experiments, the simulant solutions are estimated to have nominally 50 meq of Na^+, while the resin aliquot contains 0.22 meq of H^+**.**

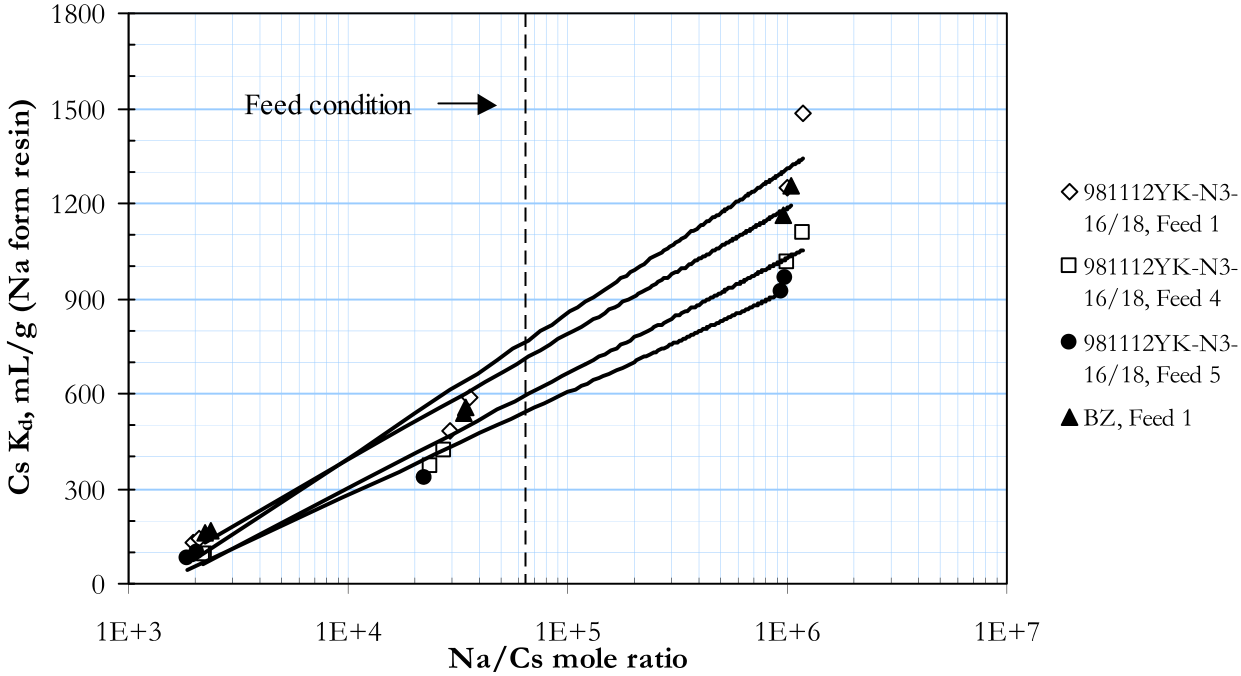
Three K_d curves for the 16/18 batch were produced in conjunction with the other SL-644 batch contacts representing Feeds 1, 4, and 5. All points resulted in good precision except for one duplicate set 16/18 batch Feed 1 at Na/Cs mole ratio of 1E+6. The data pair at the low Cs concentration are at K_d = 1480 mL/g and 1252 mL/g (Figure 3.2a); relative to the other two batch contact series with the 16/18 resin batch, it appears one or both of these points may be outliers. Contacts with the 16/18 as-received resin, mass-corrected to the Na-form, resulted in 979 and 922 mL/g (Na/Cs mole ratio = 4.5E+5) (see Table 3.4).

The effect of PSD on the K_d values was evaluated with the aged resin and the new resin. The aged resin batch K_d values are shown in Figure 3.2a. If the data points at 1480 and 1252 mL/g are outliers and removed, then the curve tracks well with the other two 16/18 curves (Feed 4 and Feed 5). The 644BZ Feed 1 curve then becomes steeper than all 16/18 curves. Because the 644BZ resin has a much smaller PSD than the 16/18 batch, it is expected to outperform the 16/18 batch. However, comparing the two curves generated specifically from Feed 1, the 644BZ and 16/18/curves are virtually equivalent. The PSD effect was also evaluated with the –73 resin batch (Figure 3.2b). The small PSD resulted in better performance evidenced by its steeper and higher curve relative to that of the large particle size material.

Resin aging effects were evaluated by comparing the K_d values from equivalent PSDs of different resin production batches. The K_d values generated from the small PSD of resins 644BZ and –73 are shown in Figure 3.3a. The new resin resulted in a notably higher and steeper K_d curve. The Kd values generated from the large PSD of resins 16/18 and –73 are compared in Figure 3.3b. The curves are much more closely packed, with the –73 resin generally higher than the 16/18 resin. Comparing the two curves generated specifically from Feed 5, the –73 resin demonstrated superior performance relative to the aged resin.

Slight differences in feed composition may also contribute to the observed K_d value variability. The results of 16/18 resin batch can be directly compared to the other resins because it was contacted in each feed tested, thus eliminating the issue of feed variability.

a)



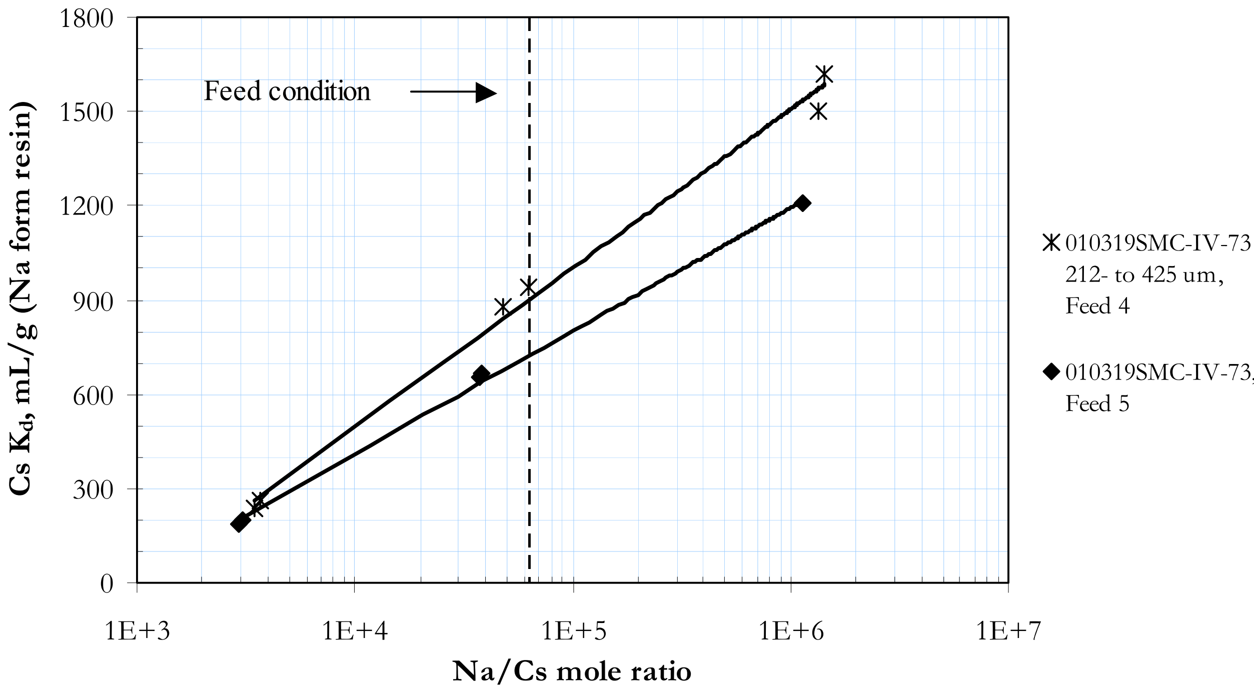


Figure 3.2. K_d Values as a Function of Equilibrium Na/Cs Mole Ratio in AW-101 Simulant Relative to the Na Resin Form

a) Aged resins comparing different PSDs, large PSD 981112YK-N3-16/18 and small PSD 644BZ.
b) New resin (010319SMC-IV-73) comparing different PSDs.

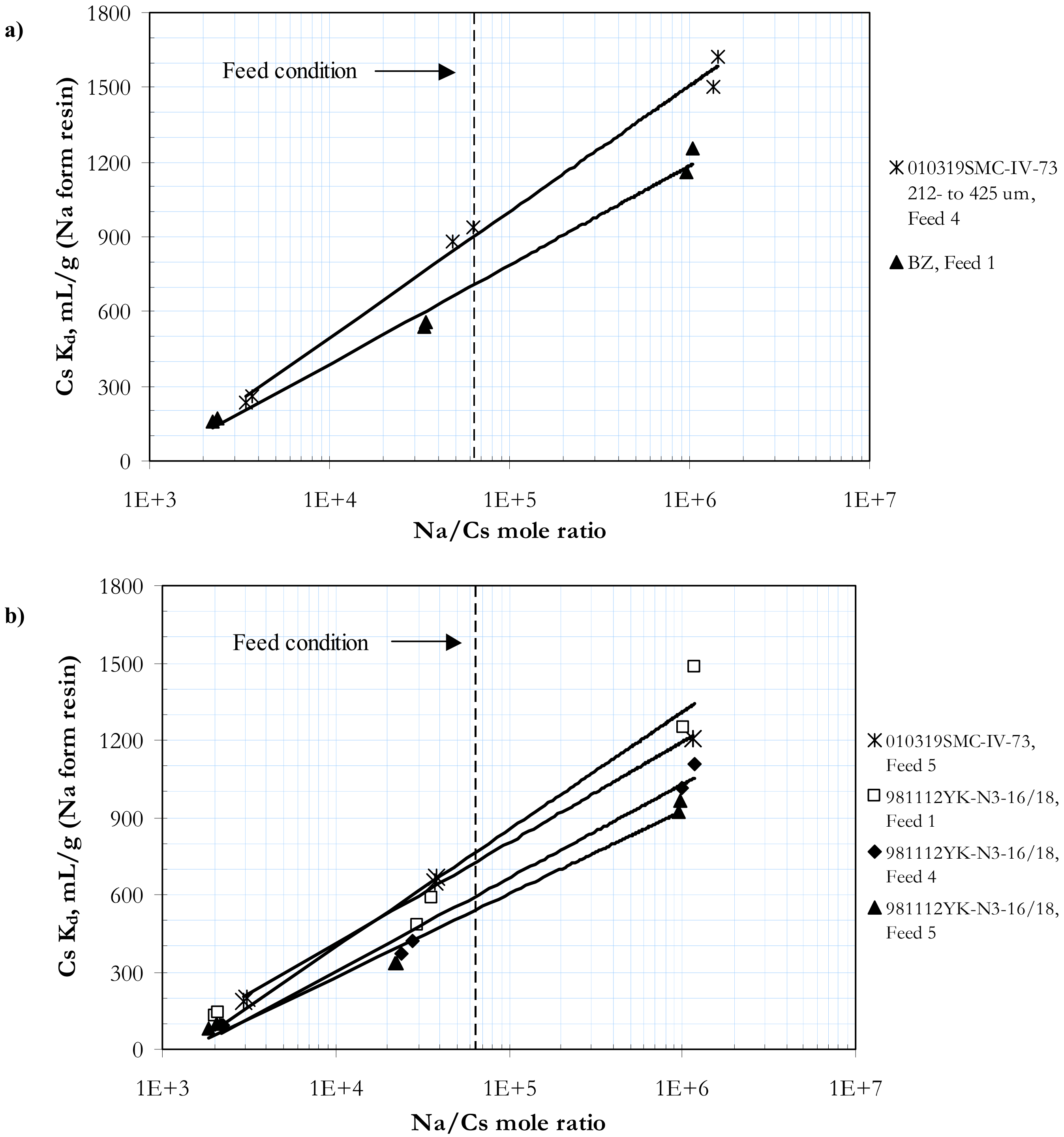


Figure 3.3. K_d Values as a Function of Equilibrium Na/Cs Mole Ratio in AW-101 Simulant Relative to the Na Resin Form

a) Small PSD resins, aged resin (644BZ) and new resin (010319SMC-IV-73).
b) Large PSD resins, aged (981112YK-N3-16/18) and new resin (010319SMC-IV-73).

Other parameters, such as manufacturing conditions, may also affect the Cs ion exchange performance of SL-644. Thus a better experimental approach to determine aging effects would have been to use one resin batch and evaluate its performance when new and after aging. In all cases, however, the equilibrium feed condition K_d values for AW-101 simulant at nominally 6.3E+4 Na/Cs mole ratio exceeded the threshold value of 450 mL/g.[14]

The feed condition K_d values are estimated from the point at which the K_d curve crosses the feed condition Na/Cs mole ratio of 6.3E+4. The column distribution ratio, Cs λ, can be estimated from the K_d value and the appropriate bed density, ρ_b, using the following relationship:

$$Cs\,\lambda = K_d * \rho_b \tag{7}$$

A summary of the dry-bed densities in contact with the AW-101 waste simulant, the K_d values, and the predicted Cs λ values are provided in Table 3.5. Under ideal conditions, the Cs λ value is the point at which the Cs breakthrough curve passes through C/C_o[15] = 50%. The predictions indicate the 16/18 resin batch material will not quite meet the specification of reaching 100 BVs before 50% breakthrough. The small particle size –73 resin is expected to reach 50% breakthrough at 200 BVs, which is slightly better than the large particle size material.

Table 3.5. Predicted Cs λ Values

SL-644 Resin	Na/Cs mole ratio	K_d, mL/g (Na-form)	Bed density in AW-101 Simulant, g/mL	Predicted Cs λ (BV AW-101 Simulant)	Bed density in 0.25 M NaOH, g/mL	Predicted Cs λ (BV 0.25 M NaOH)
981112YK-N3-16/18	5.7E+4	630	0.19	120	0.15	95
010319SMC-IV-73 (as received)	5.1E+4	720	0.26	190	0.24	170
010319SMC-IV-73 (212- to 425-μm)	6.0E+4	900	0.24	220	0.22	200
644BZ	6.1E+4	700	NA	–	NA	–
NA = not analyzed						

[14] Test Specification: Evaluating SuperLig 644 Storage on Exchange Capacity and Effectiveness of Up Flow Elution TSP-W375-00-00034, M. E. Johnson, 12/6/00.
[15] The C_o refers to the ^{137}Cs concentration in the feed sample. C refers to the Cs concentration at the column effluent point.

3.4 Column Testing

3.4.1 Initial Conditioning for Cesium Ion Exchange

The 16/18 resin bed conversion from Na-form to H-form was monitored by the resin-bed height. In all matrices, this resin appeared black. For the –73 resin batch, the resin bed conversion progress from Na-form to H-form and vice versa could be monitored visually according to the color of the resin bed as well as resin-bed height. In this case, the Na-form appeared black in the columns whereas the H-form appeared brown.

Once loaded in the columns, the 16/18 resin preconditioning was evaluated by measuring the effluent composition by ICP-AES. Sodium and K were the only cations found above the reagent blank concentration; the results are summarized in Table 3.6. The Na concentration is expected to be high initially because the resin was soaked in 1 M NaOH before loading in the columns. The K concentration is expected to be removed with washing. Concentrations of Na and K remained high (lead column) or continued to climb (lag column) throughout the 0.5 M HNO_3 preconditioning process. A significant mass fraction of the as-received resin contains residual materials from the production process that can be removed by acid washing. The continued elution of Na and K during the preconditioning phase indicates that these residual materials could still be present to some degree after acid washing. It is not known what effect the continued presence of some residual fraction of production material will have on ion exchange processing. Elution of Na and K abruptly ceased with the DI water rinse. The low Na concentration found in the 0.25 M NaOH regeneration effluent shows evidence of dilution with the DI water in the apparatus volume as well as Na exchange onto the resin.

Table 3.6. Effluent Preconditioning Reagents, SL-644 Batch 16/18

Preconditioning Reagent	Volume, mL	Na, μg/mL	K, μg/mL	Volume, mL	Na, μg/mL	K, μg/mL
	Lead column(1)			Lag column(2)		
0.5 M HNO_3	11.6	2590	1190	9.9	790	550
0.5 M HNO_3	13.9	2740	2470	9.7	1910	1680
0.5 M HNO_3	11.5	2010	2270	9.1	2740	3130
DI water(3)	53.1	140	< 40	40.4	150	120
0.25 M NaOH(4)	50.0	1390	< 40	43.5	1400	< 50

(1) Resin mass in column was 2.5006 g, as-received form.
(2) Resin mass in column was 2.5001 g, as-received form.
(3) First composite fraction of DI water collected was measured by ICP-AES.
(4) First composite fraction of 0.25 M NaOH collected was measured by ICP-AES.
Note: Less-than values indicate analyte concentration was less than the instrument detection limit. The dilution-corrected instrument detection limit is reported.

3.4.2 Initial Testing with AW-101 Simulant on SL-644 16/18

The lead column Cs load profiles for the simple simulant and AW-101 simulant tests are shown in Figure 3.4. After the standardized preconditioning procedure was performed, a simple simulant composed of 2 M NaOH + 1.5 M $NaNO_3$ + 1.5 M $NaNO_2$ + 7.5E-5 M Cs (10 μg/mL) was loaded first through the columns to verify acceptable performance (Test A). Very early breakthrough from the lead column was found for this simulant test, 6% C/C_o at 13 BVs. The load was stopped and the column rinsed, eluted and regenerated, using nearly twice the 0.25 M NaOH volume as was used initially. Then AW-101 simulant was loaded onto the column (Test B). Again, early breakthrough from the lead column was noted, 24% C/C_o at 44 BVs. Cycling the resin from the Na-form to the H-form through both the pre-conditioning steps and the simple simulant run was not sufficient to produce an acceptable Cs load behavior.

The columns were washed extensively at a nominal flow rate of 3 BV/h with 0.5 M HNO_3 with a total of 83.7 BVs through the lead column and 76.3 BVs through the lag column following the AW-101 simulant load and rinse cycle. The follow-on simple simulant Test C showed a marked improvement in Cs loading behavior where 0.2% C/C_o lead column breakthrough was obtained at 25 BVs. The Cs breakthrough for this matrix was nearly two orders of magnitude lower than the first simple simulant test. The loading was stopped and the resin beds rinsed, eluted, and then washed again with the large eluant volume. The follow-on AW-101 simulant Test D resulted in a Cs breakthrough from the lead column of 11% C/C_o at 44 BVs. Both the AW-101 simulant load profiles resulted in linear load behaviors on the resin. The slopes appear to be equivalent, however, the long 0.5 M HNO_3 conditioning wash resulted in delayed Cs breakthrough by approximately 20 BVs. Test D with AW-101 simulant is compared to the –73 resin testing in subsequent sections of this report.

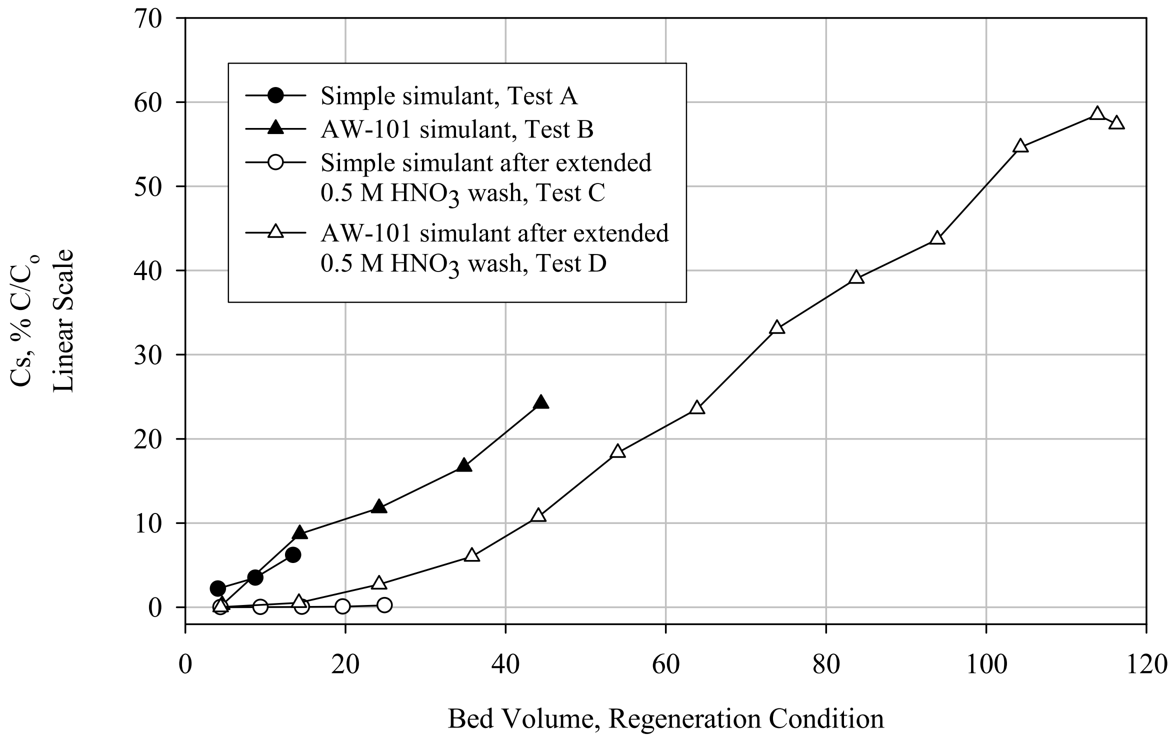


Figure 3.4. ^{137}Cs Breakthrough Curves for a Simple Simulant and AW-101 Simulant Feed Sample with SL-644Batches 16/18 With and Without an Extended 0.5 M HNO_3 Resin Conditioning Wash

3.4.3 AW-101 Simulant Loading (^{137}Cs Breakthrough Curves)

After the resin beds were conditioned, the columns were connected in series, and the loading phase was initiated with the AW-101 simulant spiked with ^{137}Cs tracer. The resin shrinkage from the 0.25 M NaOH feed to the AW-101 simulant feed varied with the –73 resin shrinking 6% (large particle size distribution) and 8% (average of small particle size distribution) and the 16/18 resin shrinking 20%. In all cases the resin color remained black in the AW-101 feed condition.

The Cs loading curves, defined by ^{137}Cs measurement, are shown in Figure 3.5 as % C/C_o vs. the BVs of feed processed through each column. The lead and lag columns are shown for two runs; the lag-column samples for the small particle-size run were at or below the detection level (1.4E-2%) and are not shown. The BV on the abscissa scale reflects BV as a function of the resin-bed size in the expanded 0.25 M NaOH regeneration condition. To determine the volume in the simulant feed condition, the BV will need to be multiplied by the appropriate expansion factor (e.g., 1.2 for the –16/18 batch resin and 1.06 for the –73 large particle size resin batch). The % C/C_o is plotted on a probability scale. Under ideal conditions, the load profile will show a linear shape at onset of Cs breakthrough on a probability scale.

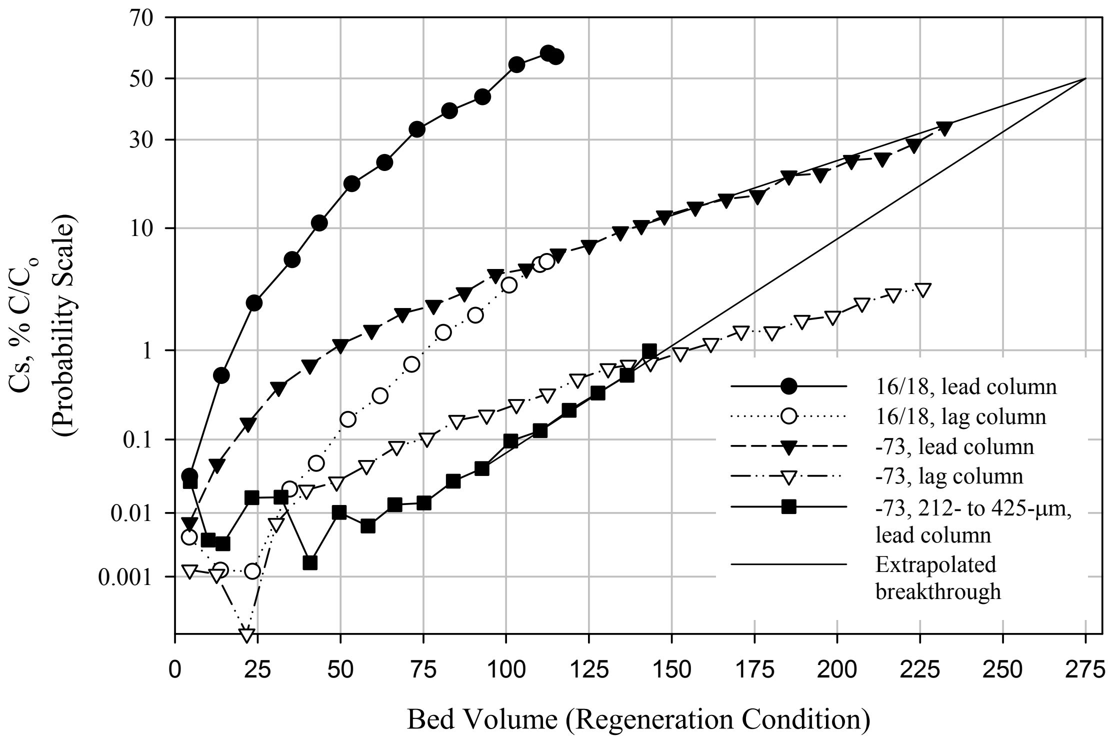


Figure 3.5. [137]Cs Breakthrough Curves and Extrapolations for AW-101 Simulant Feed Sample with Various Batches of SL-644

The 16/18 resin batch resulted in a linear loading curve on a linear scale (Figure 3.4), reaching a 50% breakthrough at ~100 BVs. This loading behavior is an indication of poor Cs transit into the ion exchange material. The –73 as-received PSD resin displays a linear load profile on the probability scale after 50 BVs were loaded. Initial load samples indicated early breakthrough to nearly 1% C/C_o at 50 BVs. The small particle-size –73 resin resulted in nearly complete Cs removal through the first 70 BVs. After 90 BVs, a linear (probability scale) breakthrough is manifested with a steeper breakthrough loading than the as-received PSD resin. The delayed and steeper Cs breakthrough on the –73 small particle size resin relative to the larger particle size is consistent with improved mass transfer of Cs into the solids phase exchanger. The smaller particle size has a shorter diffusion path and less resistance to mass transfer.

The experimental 50% Cs breakthrough, the point at which the C/C_o value is 50% (0.5), is normally a direct indicator of the effective capacity of the resin. Based on interpolation and extrapolation of these load curves, the experimental 50% Cs breakthrough values can be estimated. The experimental values for the lead columns are shown in Table 3.7 along with the predicted Cs λ values. The experimental 16/18 batch resin 50% breakthrough value was virtually identical to the predicted Cs λ value. The extrapolated

50% breakthrough values for the –73 resin were higher than the predicted Cs λ values. Extrapolation from C/C_o of 34% (large particle size) may be fairly accurate; extrapolation from C/C_o of 1% (small particle size) is subject to much uncertainty. The extrapolations resulted in equal 50% breakthrough values between the different PSDs. The experimental values were plausibly consistent with the predicted Cs λ values.

Table 3.7. Experimental 50% Cs Breakthrough Values Compared with Predicted Cs λ Values

BV Condition	981112YK-N3-16/18 Interpolated (Predicted)	010319SMC-IV-73 (as received) Extrapolated (Predicted)	010319SMC-IV-73 (212- to 425-μm) Extrapolated (Predicted)
0.25M NaOH	100 (95)	270 (170)	270 (200)
Feed	128 (120)	300 (190)	300 (220)

The decontamination factors (DFs) were calculated on composite effluents from each run and are summarized in Table 3.8. The DFs may be compared to the contractual limit for Cs removal of C/C_o = 0.045% (DF = 2,200), for actual AW-101 waste based on the maximum 20 wt% waste Na_2O loading[16] in the glass (Kurath 2000). In only one case, small particle size –73 resin, was sufficient Cs removed to meet the targeted DF of 2,200 after processing nearly 150 BVs AW-101 simulant. On this basis, the –73 small particle size resin was forwarded to the hot cells for subsequent actual waste testing.

Table 3.8. Decontamination Factors for ^{137}Cs from AW-101 Simulant

Resin Identification	Load volume, BV (mL)	% C/C_o	Overall DF
981112YK-N3-16/18	114 (1067)	1.06	94
010319SMC-IV-73 first collection	137 (1136)	0.172	581
010319SMC-IV-73 second collection	89 (738)	1.87	53
010319SMC-IV-73 (212- to 425-μm)	140 (1538)	0.0015	66,000

Direction to PNWD was revised to perform calculations based on the minimum 14 wt% waste Na_2O loading in glass. The contractual limit for Cs removal based on 14 wt% waste Na_2O loading in the glass is C/C_o = 0.064% (DF = 1550). Instead of targeting the 150-BV loading, direction was provided to PNWD to evaluate the lead column Cs breakthrough at 100 BVs and 72 BVs. The measured lead column breakthroughs at 100 BVs and 72 BVs are shown in Table 3.9. Also provided are the estimated Cs DFs for the composite effluent. The DFs were calculated by integrating the lag column breakthrough profile to the indicated BV. The 16/18 resin batch did not meet the required DF after even as little as a 72-BV loading; a 68-BV loading would have met the DF target. The 100-BV loading of the –73 as-received PSD resin did not meet the required DF for minimum waste loading or the maximum waste loading; however, the DF was met for the maximum waste loading at 72 BVs.

[16] The DF calculation assumes all Na comes from the tank waste, the glass density is 2.66 g/mL, the waste Na concentration is 5 M, and the waste contains 194 μCi/mL ^{137}Cs.

Table 3.9. Lead Column Cs Breakthroughs and Effluent DFs from AW-101 Simulant at 100-BV and 72-BV Loadings

Resin Identification	Lead column % C/C_o at 100 BVs(1)	DF at 100 BVs(2)	Lead column % C/C_o at 72 BVs	DF at 72 BVs(2)
981112YK-N3-16/18	50	160	31	810
010319SMC-IV-73	4.9	1,500	2.4	4,100
010319SMC-IV-73 (212-to 425-µm)	0.095	8.6 E+5	0.014	1.2 E+5
(1) Interpolated from loading curve. (2) Estimate based on integrated results.				

Selected samples from the AW-101 simulant-loading phase on 212- to 425-µm –73 resin and the effluent were analyzed by ICP-AES for Na and K concentrations. The results are summarized in Table 3.10, along with the feed concentrations.

Table 3.10. Na and K Analysis of Selected AW-101 Simulant Feed Samples and Effluent

Sample ID	BV	Na, M(1)	K, M(1)
AW-101 Feed	—	5.15	0.417
Lead column			
SL101-F3	22.1	5.04	0.394
SL101-F10	87.4	5.26	0.425
SL101-F17	157	5.52	0.442
Lag column			
SP101-F3	21.7	5.07	0.408
SP101-F10	85.1	4.91	0.404
SP101-F17	153	4.91	0.399
AW-101 Effluent	—	5.30	0.437
(1) The overall estimated error is ± 15% 2-σ.			

No significant changes in the Na and K concentrations were evident in the initial through the last load samples within the error of the analytical method (±15%). The average Na concentration was 5.2 M with a standard deviation of 4%. The average K concentration was 0.416 M with a standard deviation of 2%.

3.4.4 Elution

At the completion of the DI water wash, elution of the lead and lag columns was initiated by pumping 0.5 M HNO_3 into each column. Due to holdup in the system (nominally 22-mL AV), the first couple of BVs probably contained a substantial amount of the DI water rinse that preceded the elution. In all cases, the resin bed shrank upon conversion to the acid form to a similar volume observed in the acid conditioning step.

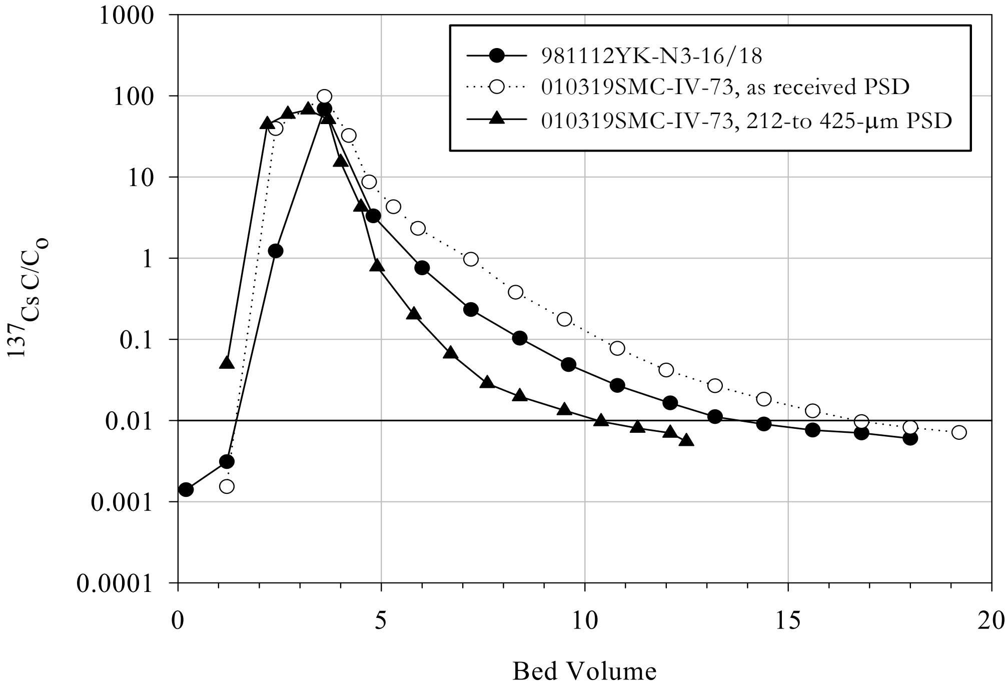
The lead column elution C/C_o values for ^{137}Cs are shown in Figure 3.6a. The ordinate is a logarithmic scale to clearly show the large range of C/C_o values. The abscissa defines BVs as a function of the expanded resin bed in 0.25 M NaOH. Over 99% of the ^{137}Cs eluting from the lead column was contained in the second through sixth BV samples. The peak C/C_o values were found to be nearly 100. The elution cutoff of C/C_o = 0.01 was reached at 10 BVs for the small particle-size resin and at 14 and 17 BVs for the larger particle-size distributions. Clearly the smaller particle size resin releases the Cs more efficiently. Current plant design operation requires 1 % C/C_o to be reached by 15 BVs eluant loading. The –73 larger particle-size material elution profile resulted in greater tailing and the total volume required to meet the 1 % C/C_o slightly exceeded the design-basis eluant volume.

The lag column elution C/C_o values for ^{137}Cs are shown in Figure 3.6b. As with the lead column, most of the ^{137}Cs was contained in the second through sixth BVs. The elution profiles of the larger particle-size resin beds were virtually identical with peak C/C_o values of 20 and 22. The elution C/C_o cutoff of 0.01 was reached at 10 and 11 BVs. The smaller particle-size fraction contained significantly less Cs and reached a maximum C/C_o of 12 and the elution cutoff of 0.01 at about 5 BVs.

The eluate samples from the lead and lag columns of the –73 212- to 425-µm particle-size resin test were composited separately. Samples of the lead and lag column composites were submitted to the analytical laboratory for analysis with ICP-AES, IC, GEA, and TOC. The analytical results are shown in Table 3.11. Sodium was the dominant component detected with ICP-AES, although a number of other metals were also detected. The Na/K mole ratio (10.8) in the eluate is identical to that of the AW-101 simulant feed (10.7); the Na/Al mole ratio (93) in the eluate is a factor of 10 higher than the feed (9.2). The Mg appears to have been concentrated in the eluate one-hundred fold. The Na/Mg mole ratio in the eluate is 28 whereas the feed Na/Mg mole ratio is 3000. The only anion detected was nitrate. A small amount of total organic carbon was detected, but it is not known if this is from residual waste in the column system or from organic materials leaching from the resin.

Most of the specified minimum reportable quantity (MRQ) levels were met with some exceptions. The large amount of nitrate prevented the detection limit for Cl from meeting the MRQ level of 3 µg/mL. The large concentration of nitrate required large sample dilutions that, in turn, increased the method detection limit for the other components. In any case, the anion concentrations other than nitrate are expected to be small. This is somewhat confirmed by the fact that no P was detected with the ICP-AES analysis. The TIC analysis was not performed because carbonate is known to evolve as CO_2 in acidic solutions.

a)



b)

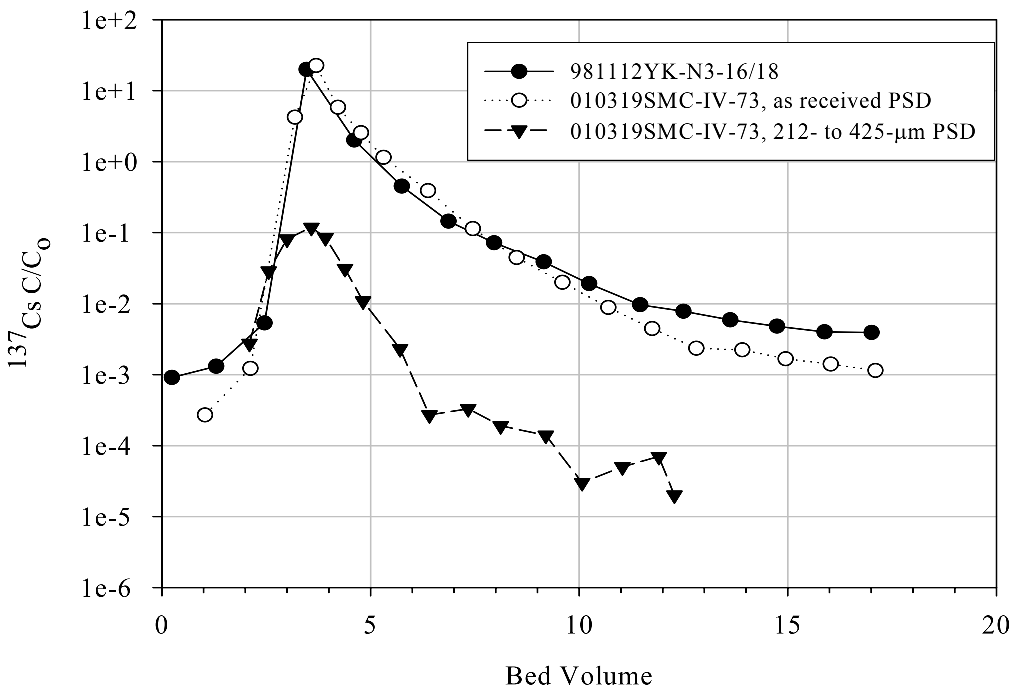


Figure 3.6. Cesium Elutions (a) Lead Columns (b) Lag Columns

Table 3.11. Analysis of the Lead and Lag Column Eluate Composite
From the 010319SMC-IV-73, 212- to 425-μm, Test

Cations (ICP-AES)(1)				Cations (ICP-AES), continued			
Analyte	μg/mL			Analyte	μg/mL		
	Lead	Lag	MRQ(2)		Lead	Lag	MRQ
Al	14.9	8.63	7.5E+1	U	< 10	<2	6.0E+2
Ba	0.66	0.57	7.8E+1	Zn	[0.27]	[0.05]	1.65E+1
Ca	< 2	[0.45]	1.5E+2	B	23	13	NMRQ
Cd	< 0.1	< 0.02	7.5E+0	P	< 0.5	< 0.1	NMRQ
Co	< 0.3	< 0.05	3.0E+1	Sr	< 0.1	[0.02]	NMRQ
Cr	< 0.1	[0.06]	1.5E+1	TOC and Anions (IC)(1)			
Cs(3)	107	0.14	NMRQ	Analyte	μg/mL		
Cu	< 0.2	< 0.03	1.7E+1		Lead	Lag	MRQ(2)
Fe	3.2	2.87	1.5E+2	TOC	68	<33	1.5E+3
K	186	491	7.5E+1	F^-	<50	<50	1.5E+2
La	< 3	< 0.05	3.5E+1	Br^-	<50	< 50	NMRQ
Mg	44.3	20.6	1.5E+2	Cl^-	<50	< 50	3.0E+0
Mn	[1.2]	1.1	1.5E+2	NO_3^-	26,500	25,600	3.0E+3
Mo	< 3	< 0.05	9.0E+1	NO_2^-	<100	< 100	3.0E+3
Na	1,200	1,220	7.5E+1	PO_4^{-3}	<100	< 100	2.5E+3
Ni	[0.23]	< 0.03	3.0E+1	SO_4^{-2}	<100	< 100	2.3E+3
Pb	< 0.5	< 0.1	3.0E+2	$C_2O_4^{-2}$	<100	< 100	NMRQ
Si	27.8	17.4	1.7E+2	Density, g/mL			
Sn	< 8	<2	1.5E+3	Density	1.008	1.007	NMRQ
Ti	< 2	< 0.03	1.7E+1				

(1) The overall error is estimated to be within ±15% (2-σ). Values in brackets are within 10 times the instrument detection limit, and errors are likely to exceed ±15%.
(2) MRQ is minimum reportable quantity. NMRQ is no minimum reportable quantity identified by the client.
(3) The Cs concentration is calculated based on the ^{137}Cs recovery.

3.4.5 Activity Balance for ^{137}Cs

An activity balance for ^{137}Cs was completed to compare the ^{137}Cs recovered in various process streams relative to the ^{137}Cs present in the feed sample (Table 3.12). As expected, most of the ^{137}Cs was found in the eluate streams. The lead column with the 16/18 batch material lost nominally 22% Cs to the lag column; the lead column with the –73 resin (as-received PSD) lost nearly 10% of the Cs to the lag column. The lead column with the 212- to 425-μm particle-size –73 resin lost less than 1% Cs to the lag column. The low overall Cs recovery from the 16/18 resin batch run is not understood at this time, but is probably due to analytical error.

Table 3.12. Activity Balance for ^{137}Cs

Matrix	981112YK-N3-16/18		010319SMC-IV-73 as-received particle size		010319SMC-IV-73 212- to 425-μm particle size	
	Net Counts	% ^{137}Cs in Feed Sample	Net Counts	% ^{137}Cs in Feed Sample	Net Counts	% ^{137}Cs in Feed Sample
Feed Sample	1.31 E+6	100	2.11 E+6	100	1.67 E+6	100
Effluent	1.32 E+4	1.06	1.61 E+4	0.76	<186	<0.011
Load samples	8.94 E+3	0.68	6.74 E+3	0.32	55	3.3 E-3
Feed displacement	1827	0.14	1656	0.078	6	3.5 E-4
DI Water Rinse	238	0.018	60	2.8 E-3	6	3.5 E-4
Column #1 Eluate	8.29 E+5	63.5	1.77 E+6	83.8	1.72 E+6	103
Column #1 DI water rinse	16	1.2 E-3	143	6.7 E-3	130	7.8 E-3
Column #2 Eluate	2.86 E+5	21.9	1.97 E+5	9.31	2.23 E+3	0.13
Column #1 DI water rinse	3	2 E-4	0.08	4 E-6	0.6	4 E-5
Total ^{137}Cs Recovery	1.14 E+6	87.3 %	1.99 E+6	93.8 %	1.72 E+6	103 %

3.4.6 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 et. al. 1999). The actual BV as a function of process step for the lead columns are tabulated in Table 3.13. These values are normalized to the BV in the initial regeneration condition and graphed in Figure 3.7, to better show the relative volume changes. Two process cycles are graphed with the first cycle consisting of the bed conditioning steps (process steps 1 to 4) and the second cycle consisting of the actual process test (process steps 5 to 9). Each process step is denoted with a letter defined as follows: P (initial column packing), W (DI water), E (elution with 0.5 M HNO_3), ER (elution rinse with DI water), R (regeneration with 0.25 M NaOH), F (feed), and FD (feed displacement with 0.1 M NaOH).

The associated shrinking and swelling behaviors of lag column resins mimicked those of the lead column. The resin beds expand to a maximum size in dilute caustic and shrink to a minimal size in the 0.5 M HNO_3. The 16/18 batch material resulted in >50% volume increase from the 0.5 M HNO_3 matrix to the 0.25 M NaOH matrix. The –73 resin resulted in a corresponding nominal 21% volume increase, regardless of particle-size distribution.

The 16/18 batch resin shrinking and swelling is similar to previously reported results (Kurath 2000). It results in wide resin-volume variations that are reproducible in magnitude even with multiple resin cycling. The drop in volume between the regeneration condition and the feed condition is calculated at 22%. The –73 batch mirrors the 16/18 batch volume changes, but at a much smaller magnitude.

Table 3.13. SL-644 Lead Column Bed Volumes

Feed	Step Number	Symbol	981112YK-N3-16/18, mL	010319SMC-IV-73 as-received particle size, mL	010319SMC-IV-73 212- to 425-μm particle size, mL
Initial packing	1	P	9.9	7.7	10.9
0.5 M HNO_3	2	E	6.2	7.0	9.2
DI water	3	ER	6.2	6.7	9.2
0.25 M NaOH	4	R	9.5	8.4	11.2
AW-101 simulant	5	F	7.4	7.9	10.2
0.1 M NaOH	6	FD	9.4	8.7	10.9
DI water	7	W	10.2	8.4	10.9
0.5 M HNO_3	8	E	5.5	6.9	8.9
DI water	9	ER	5.5	6.9	8.9

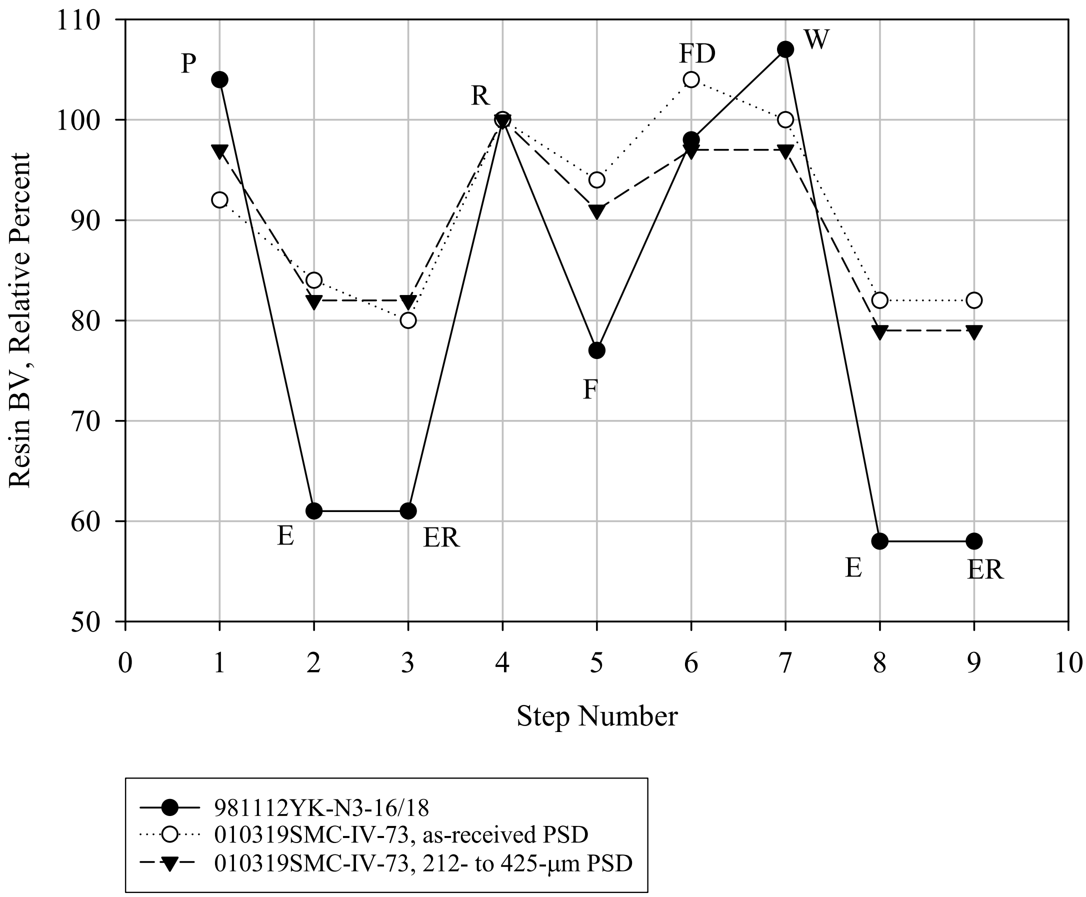


Figure 3.7. Bed Volume Comparison of the Lead Columns in Various Feed Conditions Normalized to the Regeneration Condition

4.0 Conclusions and Recommendations

Initial simulant testing with the SL-644 proved invaluable in ensuring a working system before placing the Cs ion exchange system into the hot cells to conduct testing on actual Hanford tank waste.

The batch contact study resulted in acceptable K_d values for the AW-101 simulant feed condition for the three resin batches tested. The experimental K_d values at the feed condition (Na/Cs mole ratio of nominally 6 E+4) were 630, 700, 720, and 900 for the SL-644 batches 16/18, 644BZ, –73 as-received, and –73 dry-sieved 212- to 425-µm PSD batches, respectively. The equilibrium data predicts Cs λ values of 95 BVs for batch 16/18, 170 BVs for the –73 as-received particle size, and 200 BVs for batch –73 212- to 425-µm dry-sieved PSD (as BVs in 0.25 M NaOH).

For Envelope A waste, the Cs removal by ion exchange must achieve 1.75E-5 Ci ^{137}Cs/mole Na, based on the maximum 20 wt% waste Na_2O loading in glass. The 16/18 resin batch, resulting in early breakthrough and a virtually linear loading profile, failed to meet the simultaneous target processing conditions and Cs-removal contract limits. The –73 batch, that was produced shortly before use, worked much better for Cs removal, however, the smaller particle size resin was necessary to meet the test specification Cs removal for 20 wt% Na_2O glass loading.

An aging effect may be evident in comparing the performance of the two-year aged SL-644 16/18 and 644BZ batches with freshly-prepared –73 material; the new material had higher K_d values than the aged materials. The feed condition K_d value from the aged 16/18 resin surpassed the acceptance criteria (450 mL/g) and appeared adequate to meet the needed Cs λ value, yet the Cs loading behavior during the column test was poor. It is not known if this is due to a production problem, an aging phenomenon, or a combination of both. It would have been useful to conduct a load and elute cycle on this material before aging so a comparison before and after aging could be made. A particle size effect was evident in batch contact testing, the smaller size distribution had higher K_d values than the large particle size distribution.

In summary:

- Equilibrium data from the 16/18 resin batch contact was not a good indication of resin performance during column tests. Although the predicted Cs λ value for the 16/18 batch contact data and actual column loading data agreed, Cs loading was slow, thus leading to poor DFs.

- The aging study was inconclusive relative to SL-644 performance. A comparison of freshly-prepared material and aged material from the same SL-644 production batch would have been more helpful.[17]

[17] Test activities scheduled in fiscal year 2002 at PNWD are anticipated to resolve this issue.

- Particle size is an important factor affecting Cs removal. The smaller particle-size fraction of the –73 resin resulted in higher K_d values and an ion exchange composite effluent DF 100 times greater than the larger particle size material.[18]

- The following DFs and associated processed BVs (in the regeneration condition) were obtained with ion exchange column runs with AW-101 simulant:

Resin Identification	DF	BV processed	DF for 72-BV processing[(1)]
981112YK-N3-16/18	**94**	116	**813**
010319SMC-IV-73	**584**	137	9,500
010319SMC-IV-73 (212- to 425-µm)	66,000	140	1.51E+5

(1) Estimate based on integration of load profile.
Bolded and highlighted values indicate the contract-required Cs removal was not met.

  Only the –73 212- to 425-µm SL-644 resin batch met the test DF requirement (2200, based on maximum Na_2O waste loading) at 150 BVs. Both –73 particle size distributions met the minimum 14 wt% waste Na_2O loading criteria for Cs removal (DF = 1550) with processing of 72 BVs.

- The small particle-size fraction of the recently produced resin best met the performance criteria delineated in the test plan. Therefore, this material was forwarded to the hot cell for use in actual waste testing.

[18] Test activities are being conducted at SRTC to evaluate PSD on Cs ion exchange performance (TSP-W375-01-00023, Rev. 0, task specification for evaluating effects of resin particle size and solution temperature on SuperLig® 644 and SuperLig® 639 resins performance with LAW Envelope A simulant)

5.0 References

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APPENDIX A

AW-101 Simulant Preparation

Table A.1. Simulant AW-101 (Envelope A) Preparations

Compound	Targeted M	FW	Feed 1		Feed 2		Feed 4		Feed 5	
			Mass used g	Calculated M	Mass used g	Calculated M	Mass used g	Calculated M	Mass used g	Calculated M
EDTA	3.70E-3	292.24	2.1634	3.70E-3	2.1606	3.70E-3	2.1652	3.70E-3	4.3255	3.70E-3
Citric acid	3.70E-3	210.14	1.5553	3.70E-3	1.5557	3.70E-3	1.5577	3.71E-3	3.1104	3.70E-3
$Na_3HEDTA\text{-}2H_2O$	3.70E-3	344.00	2.5458	3.70E-3	2.5447	3.70E-3	2.5459	3.70E-3	5.0905	3.70E-3
Na_3NTA	3.70E-3	257.10	1.9028	3.70E-3	1.9058	3.71E-3	1.9027	3.70E-3	3.8045	3.70E-3
NaGluconate	3.70E-3	218.00	1.6132	3.70E-3	1.6148	3.70E-3	1.6130	3.70E-3	3.2273	3.70E-3
Na_2Iminodiacetate	3.70E-3	177.07	1.3102	3.70E-3	1.3144	3.71E-3	1.3104	3.70E-3	2.6211	3.70E-3
$Fe(NO_3)_3\text{-}9H_2O$	5.00E-5	404.02	0.0400	4.95E-5	0.0395	4.89E-5	0.0409	5.06E-5	0.0814	5.04E-5
$Mg(NO_3)_2\text{-}6H_2O$	1.50E-3	256.40	0.7696	1.50E-3	0.7663	1.49E-3	0.7691	1.50E-3	1.5381	1.50E-3
$Mn(NO_3)_2$, 50%	6.63E-5	4.30 M	1.122 mL	2.41E-3	0.0309 mL	6.64E-5	0.0308 mL	6.62E-5	0.0616 mL	6.62E-5
MoO_3	2.86E-4	143.95	0.0827	2.87E-4	0.0826	2.87E-4	0.0828	2.88E-4	0.1647	2.86E-4
$Ni(NO_3)_2\text{-}6H_2O$	1.33E-4	290.80	0.0777	1.34E-4	0.0766	1.32E-4	0.0774	1.33E-4	0.1544	1.33E-4
SiO_2	2.93E-3	60.08	0.3520	2.93E-3	0.3520	2.93E-3	0.3523	2.93E-3	0.7044	2.93E-3
$BaNO_3$	1.33E-4	261.38	0.0696	1.33E-4	0.0698	1.34E-4	0.0693	1.33E-4	0.1385	1.32E-4
$Ca(NO_3)_2$	4.13E-4	236.16	0.1949	4.13E-4	0.1947	4.12E-4	0.1952	4.13E-4	0.3907	4.14E-4
$Sr(NO_3)_2$	1.30E-5	211.65	0.0054	1.28E-5	0.0054	1.28E-5	0.0056	1.32E-5	0.0107	1.26E-5
$RbNO_3$	1.00E-5	147.47	0.0030	1.02E-5	0.0027	9.15E-6	0.0030	1.02E-5	0.0056	9.49E-6
$CsNO_3$	6.40E-5	194.92	0.0251[(a)]	7.01E-5	0.0250	6.41E-5	0.0243	6.23E-5	0.0502	6.44E-5
$LiNO_3$	5.51E-4	69.00	0.0764	5.54E-4	0.0765	5.54E-4	0.0761	5.51E-4	0.1516	5.49E-4
KOH	4.30E-1	56.11	48.2596	4.30E-1	48.226	4.30E-1	56.22[(b)]	5.01E-1	96.5	4.30E-1
NaOH	3.89E+0	40.00	311.2	3.89E+0	311.4	3.89E+0	356.53[(b)]	4.46E+0	622.3	3.89E+0
$Al(NO_3)_3\text{-}9H_2O$	5.06E-1	375.15	379.7	5.06E-1	379.73	5.06E-1	403.2[(b)]	5.37E-1	759.3	5.06E-1
Na_2CO_3	1.00E-1	105.99	21.1992	1.00E-1	21.199	1.00E-1	21.199	1.00E-1	42.3959	1.00E-1
Na_2SO_4	2.36E-3	142.05	0.6707	2.36E-3	0.6714	2.36E-3	0.6705	2.36E-3	1.3413	2.36E-3
$NaHPO_4\text{-}7H_2O$	1.73E-3	268.07	0.9277	1.73E-3	0.9243	1.72E-3	0.9280	1.73E-3	1.8548	1.73E-3
NaCl	6.93E-2	58.45	8.1019	6.93E-2	8.1021	6.93E-2	8.0850	6.92E-2	16.2025	6.93E-2
NaF	1.10E-2	41.99	0.9237	1.10E-2	0.9231	1.10E-2	0.9234	1.10E-2	1.8477	1.10E-2
$NaNO_2$	7.90E-1	69.00	109.02	7.90E-1	109.03	7.90E-1	109.00	7.90E-1	218.04	7.90E-1
Final Volume			2-L		2-L		2-L		4-L	

(a) $CsNO_2$ was used instead of $CsNO_3$.

(b)Additional reagent was added to increase molarity, hygroscopic nature biased the mass low.

APPENDIX B

Resin Properties

Table B.1. F-factor for SL-644

Test Identification	Resin Batch	F-factor					
		as-received form			H-form		
Batch Contact Testing		Sample	Duplicate	Average	Sample	Duplicate	Average
981112YK-N3-16/18 and 644BZ	16/18	0.890	0.893	0.891(1)	0.930	0.930	0.930
	644BZ	0.907	0.908	0.908	0.942	0.941	0.941
981112YK-N3-16/18 and 010319SMC-IV-73 as-received particle size	16/18	0.886	0.883	0.884	0.937	0.939	0.938
	-73	0.870	0.871	0.871(2)	0.938	0.938	0.938
981112YK-N3-16/18 and 010319SMC-IV-73 212- to 425-μm particle size	16/18	na	na	na	0.920	0.922	0.921
	-73, 212-425	0.879	0.876	0.877(3)	0.861	0.855	0.858

(1) F-factor for the 16/18 column ion exchange test
(2) F-factor for the –73 as-received column ion exchange test
(3) F-factor for the –73 212- to 425-μm column ion exchange test

Table B.2 Mass Loss Factor, L

Batch Contacts with SL-644 as-received and converted to H+ form

SL-644 Resin Conversion to H+ form

TI-41500-020, Rev. 0

Batch ID	Beaker tare, g	Beaker + resin, g	Resin, g	F factor	Lost fines, g	Corrected Resin mass, g	Beaker + H+ resin, g	H+ resin, g	F factor	Corrected resin mass, H-form g	Loss on conversion, g	% mass loss	L factor, mass loss
644BZ	69.8225	72.8353	3.0128	0.9078	0.036	2.7023	71.2131	1.3906	0.9413	1.3090	1.3934	51.6%	0.4844
981112YK-N3-16/18	68.9750	71.9881	3.0131	0.8911	0.032	2.6565	70.3667	1.3917	0.9296	1.2937	1.3627	51.3%	0.4870

Second run for conversion to H+ form

TI-41500-020, Rev. 0, Addendum A

Batch ID	Beaker tare, g	Beaker + resin, g	Resin, g	F factor	Lost fines, g	Corrected Resin mass, g	Beaker + H+ resin, g	H+ resin, g	F factor	Corrected resin mass, H-form g	Loss on conversion, g	% mass loss	L factor, mass loss
644BZ	69.8104	71.2255	1.4151	0.9078	0.0397	1.2486	70.4481	0.6377	0.9413	0.6003	0.6483	51.9%	0.4808
981112YK-N3-16/18	68.9679	72.0426	3.0747	0.8911	0.0772	2.6711	70.3922	1.4243	0.9296	1.3240	1.3470	50.4%	0.4957

Third run for conversion to H+ form

TI-RPP-WTP-065, Rev. 0

Batch ID	Beaker tare, g	Beaker + resin, g	Resin, g	F factor	Lost fines, g	Corrected Resin mass, g	Beaker + H+ resin, g	H+ resin, g	F factor	Corrected resin mass, H-form g	Loss on conversion, g	% mass loss	L factor, mass loss
010319SMC-IV-73	65.9989	70.6845	4.6856	0.8773	0.0772	4.0429	68.646	2.6471	0.8576	2.2702	1.7728	43.8%	0.5615

212- to 425-µm particle size

SL-644 Resin Conversion to H+ form

TI-RPP-WTP-065, Rev. 1

Batch ID	Beaker tare, g	Beaker + resin, g	Resin, g	Lost fines, g	F factor	Corrected Resin mass, g	Beaker + H+ resin, g	H+ resin, g	F factor	Corrected resin mass, H-form g	Loss on conversion, g	% mass loss	L factor, mass loss
010219SMC-IV-73**	66.0006	71.0002	4.9996	0.0471	0.8708	4.3126	68.5569	2.5563	0.938	2.3978	1.9148	44.4%	0.5560
981112YK-N3-16/18	68.9678	73.968	5.0002	0.0328	0.8842	4.3922	71.2971	2.3293	0.9376	2.1840	2.2082	50.3%	0.4972

**particle size was as-received.

Table B. 2, continued, Mass Loss Factor, L

TI-RPP-WTP-065, Rev. 1, Addendum 2

Batch ID	Beaker tare, g	Beaker + resin, g	Resin, g	Lost fines, g	F factor	Corrected Resin mass, g	Beaker + H+ resin, g	H+ resin, g	F factor	Corrected resin mass, H-form g	Loss on conversion, g	% mass loss	L factor, mass loss
010219SMC-IV-73*	41.5989	43.1796	1.5807	0	0.870	1.3752	42.3817	0.7828	0.925	0.7241	0.6511	47.3%	0.5265
010219SMC-IV-73*	43.6911	45.5331	1.842	0	0.873	1.6081	44.6077	0.9166	0.925	0.8479	0.7602	47.3%	0.5273

*212- to 425-µm particle size

L-Factor is calculated by dividing the H-form resin mass-corrected for water content by the processed resin mass-corrected for fines loss and water content.

Grand Average	**644BZ**	**981112YK-N3-16/18**	**010219SMC-IV-73 as-received**	**010219SMC-IV-73 sieved 212- to 425- mm**
	0.483	**0.493**	**0.5560**	**0.538**

Table B.3 Mass Increase Factor, I_{Na}

Mass Increase Factors, I_{Na}; Conversion from H-form to Na-form

TI-RPP-WTP-065, Rev. 1, Addendum

Batch ID	ID	Vial tare, g	Vial + resin, g	Resin, g	F-factor	Lost fines, g	Resin minus fines, g	Vial plus Na+ resin, g	Na+ resin, g	Gain on conversion, g	% mass gain	I factor, mass gain	Average
010219SMC-IV-73*	Na1S	17.2044	17.3925	0.1881	0.938	0	0.1764	17.417	0.2126	0.0362	20.5%	1.205	1.20
010219SMC-IV-73**	Na1AR	17.1599	17.3628	0.2029	0.938	0	0.1903	17.3912	0.2313	0.0410	21.5%	1.215	1.22
010219SMC-IV-73**	Na2AR	16.9784	17.1771	0.1987	0.938	0	0.1864	17.2052	0.2268	0.0404	21.7%	1.217	

*212- to 425-micron particle size

**particle size was as-received.

Lost fines mass could not be measured; they were observed by the analyst as being insignificant.

TI-RPP-WTP-065, Rev. 1, Addendum 2

Batch ID	ID	Vial tare, g	Vial + resin, g	Resin, g	F-factor	Lost fines, g	Resin minus fines, H-form g	Vial plus Na+ resin, g	Na+ resin, g	Gain on conversion, g	% mass gain	I factor, mass gain	Average
010219SMC-IV-73*	Na3S	17.1211	17.4768	0.3557	0.925	0	0.3290	17.5328	0.4117	0.0827	25.1%	1.251	1.25
010219SMC-IV-73*	Na4S	17.0706	17.3821	0.3115	0.925	0	0.2881	17.4303	0.3597	0.0716	24.8%	1.248	

*212- to 425-micron particle size

No test instruction--LRB 57692 pages 15 and 16, 2/12/01

981112YK-N3-16/18

Sample ID	ID	Beaker tare, g	Beaker + resin, g	Resin, g	Estimated F-factor*	Lost fines, g	Resin minus fines, H-form g	Beaker + Na^+ resin, g	Na^+ resin, g	Gain on conversion, g	% mass gain	I factor, mass gain	Average
16/18 from above (air dried)	A	52.9230	53.6598	0.7368	0.93	0.1132	0.579948	53.6653	0.7423	0.1624	28.0%	1.280	1.29
16/18 previously dried	B	51.0339	51.7925	0.7586	1.00	0.1314	0.6272	51.8477	0.8138	0.1866	29.8%	1.298	

*Note, F-factors estimated, not actually determined with this weighing.

APPENDIX C

Batch Contacts Results

Batch Contact Calculated Results

Kd determination TI-PNNL-WTP-020, Rev. 0

Sample ID	IX Material	Resin mass, g (m)	F factor, water loss	L factor, washing loss	I_{Na} mass gain factor	Corrected resin mass, g	Simulant volume(a), mL (V)	cpm/mL (Aeq)	comparitor cpm/mL, (Ao)	Fraction Cs remaining	Equilibrium Cs conc., M	Na/Cs mole ratio	Kd	Kd'
SimA-Cs-C	--	none	--	--	--	--	10.0040	121.0						
SimA-Cs-CD	--	none	--	--	--	--	10.0138	123.1	122.0	1.000	7.00E-5	6.29E+4	--	--
SimA-BZ	BZ	0.1000	0.9078	0.4826	1.29*	0.0565	10.0207	13.9		0.114	7.94E-6	5.54E+5	862	1385
SimA-BZD	BZ	0.1066	0.9078	0.4826	1.29*	0.0602	10.0199	14.4		0.118	8.23E-6	5.35E+5	777	1248
SimA-BZH	BZH+	0.1027	0.9413	1.0000	1.29*	0.1247	10.0398	7.4		0.060	4.22E-6	1.04E+6	1619	1255
SimA-BZHD	BZH+	0.1016	0.9413	1.0000	1.29*	0.1234	10.0152	8.0		0.065	4.57E-6	9.63E+5	1499	1162
SimA-16/18AR	16/18AR	0.1116	0.8911	0.4914	1.29	0.0630	10.0295	17.1		0.140	9.79E-6	4.50E+5	620	979
SimA-16/18ARD	16/18AR	0.1057	0.8911	0.4914	1.29	0.0597	10.0121	18.8		0.154	1.08E-5	4.09E+5	585	922
SimA-16/18H	16/18H+	0.1002	0.9296	1.0000	1.29	0.1202	10.0218	6.5		0.053	3.73E-6	1.18E+6	1912	1482
SimA-16/18HD	16/18H+	0.1003	0.9296	1.0000	1.29	0.1203	10.0147	7.6		0.062	4.36E-6	1.01E+6	1615	1252
SimA-Cs-C100	--	none	--	--	--	--	10.0200	154.8						
SimA-Cs-C100D	--	none	--	--	--	--	10.0198	154.8	154.8	1.000	1.00E-3	4.40E+3	--	--
SimA-Cs-BZ100	BZAR	0.0986	0.9078	0.4826	1.29*	0.0557	10.0247	50.4		0.326	3.26E-4	1.35E+4	232	372
SimA-Cs-BZ100D	BZAR	0.0969	0.9078	0.4826	1.29*	0.0548	10.0096	50.4		0.326	3.26E-4	1.35E+4	236	378
SimA-BZH100	BZH+	0.1012	0.9413	1.0000	1.29*	0.1229	10.0040	20.4		0.132	1.32E-4	3.34E+4	692	537
SimA-BZH100D	BZH+	0.1009	0.9413	1.0000	1.29*	0.1225	10.0192	19.8		0.128	1.28E-4	3.44E+4	720	558
SimA-16/18AR100	16/18AR	0.0970	0.8911	0.4914	1.29	0.0548	10.0073	55.8		0.360	3.61E-4	1.22E+4	206	324
SimA-16/18AR100D	16/18AR	0.1007	0.8911	0.4914	1.29	0.0569	10.0095	44.8		0.289	2.90E-4	1.52E+4	274	432
SimA-16/18H100	16/18H+	0.0987	0.9296	1.0000	1.29	0.1184	10.0073	23.2		0.150	1.50E-4	2.94E+4	619	480
SimA-16/18H100D	16/18H+	0.1020	0.9296	1.0000	1.29	0.1223	10.0031	19.0		0.123	1.23E-4	3.58E+4	755	585
SimA-Cs-C600	--	none	--	--	--	--	10.0708	124.6						
SimA-Cs-C600D	--	none	--	--	--	--	10.0337	125.2	124.9	1.000	4.94E-3	8.91E+2	--	--
SimA-BZ600	BZAR	0.1048	0.9078	0.4826	1.29*	0.0459	10.0464	79.6		0.637	3.14E-3	1.40E+3	60	125
SimA-BZ600D	BZAR	0.1018	0.9078	0.4826	1.29*	0.0446	10.0316	79.3		0.635	3.13E-3	1.40E+3	62	129
SimA-BZ600H	BZH+	0.1012	0.9413	1.0000	1.29*	0.0953	10.0189	49.5		0.396	1.96E-3	2.25E+3	160	160
SimA-BZ600HD	BZH+	0.1032	0.9413	1.0000	1.29*	0.0971	10.0053	47.1		0.377	1.86E-3	2.37E+3	170	170
SimA-16/18AR600	16/18AR	0.1039	0.8911	0.4914	1.29	0.0455	10.0072	86.0		0.688	3.40E-3	1.29E+3	49	100
SimA-16/18AR600D	16/18AR	0.1065	0.8911	0.4914	1.29	0.0466	10.0006	84.2		0.674	3.33E-3	1.32E+3	51	104
SimA-16/18H600	16/18H+	0.1004	0.9296	1.0000	1.29	0.0933	9.9861	56.0		0.448	2.21E-3	1.99E+3	132	132
SimA-16/18H600D	16/18H+	0.1038	0.9296	1.0000	1.29	0.0965	9.9923	52.9		0.423	2.09E-3	2.11E+3	141	141

(a)AW101 Simulant volume is determined based on the density of 1.2381 g/mL. The slight dilution attributed to spiking is considered inconsequential.

Na molarity = 4.4

*Estimated I_{Na} factor

AR indicates resin as-received; H indicates resin is in the H form.

Kd = (Ao-Aeq)/Aeq x V/(m*F)

Kd' = (Ao-Aeq)/Aeq x V/(m*F*L*I_{Na}) corrects for mass losses from washing and gain from Na conversion

where F = F factor for water loss, Ao is the initial Cs concentration and Aeq is the final Cs concentration, V is contact solution volume and m is the resin mass, and L = mass loss factor after washing and I_{Na} is the mass increase factor in Na conversion.

Sample ID	IX Material	Resin mass, g (m)	F factor, water loss	L factor, washing loss	I_{Na} mass gain factor	Corrected resin mass, g	Simulant volume*, mL (V)	cpm/mL (Aeq)	Comparitor, cpm/mL (Ao)	Fraction Cs remaining	Equilibrium Cs conc., M	Na/Cs mole ratio	Kd	Kd'
SimA-Cs-C	--	none	--	--	--	--	9.4132	128.8						
SimA-Cs-CD	--	none	--	--	--	--	10.0883	129.8	129.3	1.000	6.22E-5	8.04E+4	--	--
SimA-NAR	N (AR)	0.1049	0.8773	0.5380	1.25	0.0619	10.1045	12.6		0.097	6.05E-6	8.26E+5	1020	1516
SimA-NARD	N (AR)	0.1077	0.8773	0.5380	1.25	0.0635	10.1130	12.4		0.096	5.97E-6	8.38E+5	1010	1501
SimA-NH	NH+	0.0998	0.8576	1.0000	1.25	0.1070	10.1132	7.7		0.059	3.69E-6	1.36E+6	1877	1501
SimA-NHD	NH+	0.0979	0.8576	1.0000	1.25	0.1050	10.1171	7.3		0.056	3.49E-6	1.43E+6	2025	1620
SimA-16/18H	16/18H+	0.1058	0.9211	1.0000	1.29	0.1257	10.1473	8.8		0.068	4.23E-6	1.18E+6	1426	1106
SimA-16/18HD	16/18H+	0.0962	0.9211	1.0000	1.29	0.1143	10.1304	10.4		0.080	4.99E-6	1.00E+6	1311	1016
SimA-Cs-C100	--	none	--	--	--	--	10.0998	134.4						
SimA-Cs-C100D	--	none	--	--	--	--	10.0223	137.8	136.1	1.000	1.08E-3	4.61E+3	--	--
SimA-Cs-NAR100	N (AR)	0.0973	0.8773	0.5380	1.25	0.0574	10.1708	29.5		0.217	2.35E-4	2.13E+4	430	639
SimA-Cs-NAR100D	N (AR)	0.1067	0.8773	0.5380	1.25	0.0629	10.1611	26.9		0.198	2.14E-4	2.33E+4	440	654
SimA-NH100	NH+	0.1013	0.8576	1.0000	1.25	0.1086	10.1054	13.0		0.096	1.04E-4	4.83E+4	1101	881
SimA-NH100D	NH+	0.0972	0.8576	1.0000	1.25	0.1042	7.8112	10.0		0.074	7.99E-5	6.26E+4	1177	941
SimA-16/18H100	16/18H+	0.0944	0.9211	1.0000	1.29	0.1122	10.1018	26.5		0.194	2.11E-4	2.37E+4	481	373
SimA-16/18H100D	16/18H+	0.1002	0.9211	1.0000	1.29	0.1191	10.1026	22.7		0.167	1.81E-4	2.76E+4	546	423
SimA-Cs-C600	--	none	--	--	--	--	10.1329	128.2						
SimA-Cs-C600D	--	none	--	--	--	--	10.1179	133.0	130.6	1.000	5.12E-3	9.77E+2	--	--
SimA-NAR600	N (AR)	0.1005	0.8773	0.5380	1.25	0.0593	10.1269	64.9		0.497	2.54E-3	1.97E+3	116	173
SimA-NAR600D	N (AR)	0.0999	0.8773	0.5380	1.25	0.0589	10.1323	64.5		0.494	2.53E-3	1.98E+3	119	176
SimA-N600H	NH+	0.101	0.8576	1.0000	1.25	0.1083	10.0280	37.1		0.284	1.45E-3	3.44E+3	292	234
SimA-N600HD	NH+	0.1004	0.8576	1.0000	1.25	0.1076	10.1508	34.5		0.264	1.35E-3	3.70E+3	328	263
SimA-16/18H600	16/18H+	0.1012	0.9211	1.0000	1.29	0.1202	8.9600	57.6		0.441	2.26E-3	2.22E+3	122	95
SimA-16/18H600D	16/18H+	0.1024	0.9211	1.0000	1.29	0.1217	9.0117	58.3		0.446	2.28E-3	2.19E+3	119	92

*AW101 Simulant volume is determined based on the density of 1.2534 g/mL. The slight dilution attributed to spiking is considered inconsequential.

"N" refers to the new resin, ID 010319SMC-IV-73; the small particle size distribution 212-425 um was tested.

Na molarity = 5

*Estimated I_{Na} factor

AR indicates resin as-received; H indicates resin is in the H form.

Kd = (Ao-Aeq)/Aeq x V/(m*F)

Kd' = (Ao-Aeq)/Aeq x V/(m*F*L*I_{Na}) corrects for mass losses from washing and gain from Na conversion

where F = F factor for water loss, Ao is the initial Cs concentration and Aeq is the final Cs concentration, V is contact solution volume and m is the resin mass, and L = mass loss factor after washing and I_{Na} is the mass increase factor in Na conversion.

Kd determination TI-RPP-WTP-065 Rev. 1

The 010319SMC-IV-73 Resin is in the as-received particle-size distribution

Sample ID	IX Material	Resin mass, g (m)	F factor, water loss	L factor, washing loss	I_{Na} mass gain factor	Corrected resin mass, g	Simulant volume*, mL (V)	cpm/mL (Aeq)	Comparitor, cpm/mL (Ao)	Fraction Cs remaining	Equilibrium Cs Conc., M	Na/Cs mole ratio	Kd, mL/g	Kd', mL/g
S101-C	--	none	--	--	--	--	10.0610	139.5						
S101-CD	--	none	--	--	--	--	10.0723	133.3	136.4	1.0	6.43E-5	7.53E+4	--	--
S101-73AR	73AR	0.0966	0.8708	0.5560	1.22	0.0571	10.0795	18.3		0.134	8.36E-6	5.79E+5	772	1138
S101-73ARD	73AR	0.1025	0.8708	0.5560	1.22	0.0605	10.0835	14.6		0.107	6.68E-6	7.25E+5	940	1386
S101-73H	73H+	0.1001	0.9380	1.0000	1.22	0.1146	10.0702	9.3		0.068	4.23E-6	1.14E+6	1471	1206
S101-73HD	73H+	0.1002	0.9380	1.0000	1.22	0.1147	10.0647	9.2		0.068	4.21E-6	1.15E+6	1474	1208
S101-16/18H	16/18H+	0.1005	0.9376	1.0000	1.29	0.1216	10.0771	10.8		0.079	4.93E-6	9.81E+5	1242	963
S101-16/18HD	16/18H+	0.1009	0.9376	1.0000	1.29	0.1220	10.0610	11.2		0.082	5.10E-6	9.49E+5	1191	924
S101-C100	--	none	--	--	--	--	10.0866	138.5						
S101-C100D	--	none	--	--	--	--	10.0702	135.0	136.7	1.0	1.07E-3	4.52E+3	--	--
S101-73AR100	73AR	0.0997	0.8708	0.5560	1.22	0.0589	10.0825	29.8		0.218	2.36E-4	2.05E+4	418	616
S101-73AR100D	73AR	0.1052	0.8708	0.5560	1.22	0.0621	10.0906	28.3		0.207	2.24E-4	2.16E+4	423	623
S101-73H100	73H+	0.1010	0.9380	1.0000	1.22	0.1156	10.0999	15.9		0.116	1.26E-4	3.85E+4	812	666
S101-73H100D	73H+	0.1004	0.9380	1.0000	1.22	0.1149	10.0861	16.2		0.119	1.29E-4	3.77E+4	796	652
S101-16/18H100	16/18H+	0.0991	0.9376	1.0000	1.29	0.1199	10.0729	27.5		0.201	2.18E-4	2.22E+4	430	334
S101-16/18H100D	16/18H+	0.1000	0.9376	1.0000	1.29	0.1210	10.0764	27.3		0.200	2.16E-4	2.24E+4	431	334
S101-C600	--	none	--	--	--	--	10.1202	136.2						
S101-C600D	--	none	--	--	--	--	10.0725	134.0	135.1	1.0	5.12E-3	9.45E+2	--	--
S101-73AR600	73AR	0.0997	0.8708	0.5560	1.22	0.0589	10.1062	72.8		0.539	2.76E-3	1.76E+3	100	147
S101-73AR600D	73AR	0.0987	0.8708	0.5560	1.22	0.0583	10.0707	71.0		0.525	2.69E-3	1.80E+3	106	156
S101-73H600	73H+	0.0998	0.9380	1.0000	1.22	0.1142	10.0807	43.2		0.320	1.64E-3	2.96E+3	229	188
S101-73H600D	73H+	0.1007	0.9380	1.0000	1.22	0.1152	10.0805	41.6		0.308	1.58E-3	3.07E+3	240	197
S101-16/18H600	16/18H+	0.0996	0.9376	1.0000	1.29	0.1205	10.0857	69.3		0.513	2.62E-3	1.84E+3	103	79
S101-16/18H600D	16/18H+	0.0999	0.9376	1.0000	1.29	0.1208	10.0832	62.9		0.465	2.38E-3	2.03E+3	124	96

*AW101 Simulant volume is determined based on the density of 1.2334 g/mL. The slight dilution attributed to spiking is considered inconsequential.

Na molarity = 4.84

*Estimated I_{Na} factor

AR indicates resin as-received; H indicates resin is in the H form.

Kd = (Ao-Aeq)/Aeq x V/(m*F)

Kd' = (Ao-Aeq)/Aeq x V/(m*F*L*I_{Na}) corrects for mass losses from washing and gain from Na c where Fw = F factor for water loss

where F = F factor for water loss, Ao is the initial Cs concentration and Aeq is the final Cs concentration, V is contact solution volume and m is the resin mass, and L = mass loss factor after washing and I_{Na} is the mass increase factor in Na conversion.

Batch contacts **Four SL-644 Resin Batches**

Feed 1		Feed 4		Feed 5	
981112YK-N3-16/18		**981112YK-N3-16/18**		**981112YK-N3-16/18**	
TI-PNNL_WTP-020, Rev. 0		TI-RPP-WTP-065, Rev. 0		TI-RPP-WTP-065, Rev. 1	
Na/Cs mole ratio	Kd	Na/Cs mole ratio	Kd	Na/Cs mole ratio	Kd
1.18E+06	1482	1.18E+06	1106	9.81E+05	963
1.01E+06	1252	1.00E+06	1016	9.49E+05	924
2.93E+04	480	2.37E+04	373	2.22E+04	334
3.58E+04	585	2.76E+04	423	2.24E+04	334
1.99E+03	132	2.22E+03	95	1.84E+03	79
2.11E+03	141	2.19E+03	92	2.03E+03	96
Curve fit equation		**Curve fit equation**		**Curve fit equation**	
198.4 * Ln(x)-1431.9		157.93 * Ln(x)-1153.8		139.86 * Ln(x)-1007	
R^2 value		**R^2 value**		**R^2 value**	
0.969		0.989		0.986	
feed condition Na/Cs mole ratio		**feed condition Na/Cs mole ratio**		**feed condition Na/Cs mole ratio**	
6.1E+4		6.0E+4		5.1E+4	
feed condition Kd		**feed condition Kd**		**feed condition Kd**	
7.59E+02		5.90E+02		5.38E+02	

Average Kd of 981112YK-N3-16/18 6.29E+02

Feed 1		Feed 4		Feed 5	
644BZ		**010319SMC-IV-73, 212-425 μm**		**010319SMC-IV-73, full psd**	
TI-PNNL-WTP-020, Rev. 0		TI-RPP-WTP-065, Rev. 0		TI-RPP-WTP-065, Rev. 1	
Na/Cs mole ratio	Kd	Na/Cs mole ratio	Kd	Na/Cs mole ratio	Kd
1.04E+06	1255	1.36E+06	1501	1.14E+06	1206
9.63E+05	1162	1.43E+06	1620	1.15E+06	1208
3.33E+04	537	4.83E+04	881	3.85E+04	666
3.44E+04	558	6.26E+04	941	3.77E+04	652
2.24E+03	160	3.44E+03	234	2.96E+03	188
2.37E+03	170	3.70E+03	263	3.07E+03	197
Curve fit equation		**Curve fit equation**		**Curve fit equation**	
172.89 * Ln(x)-1203.1		219.38 * Ln(x)-1524.2		170.27 * Ln(x)-1159.2	
R^2 value		**R^2 value**		**R^2 value**	
0.990		0.994		0.999	
feed condition Na/Cs mole ratio		**feed condition Na/Cs mole ratio**		**feed condition Na/Cs mole ratio**	
6.1E+4		6.0E+4		5.1E+4	
feed condition Kd		**feed condition Kd**		**feed condition Kd**	
7.06E+02		8.99E+02		7.21E+02	

Feed 2 Na/Cs mole ratio 7.80E+04
(not used for batch contacts)

Overall average Na/Cs mole ratio **6.26E+4**

Targeted AW101 Na/Cs mole ratio 7.80E+04

APPENDIX D

AW-101 Simulant Column Run Conditions
And Count Results
Spreadsheet Calculations

Column Run Calculated Results

Cs-137 Contractual Limit in AW-101 Envelope A Vitrification Feed

Assumptions, Maximum Waste Na_2O Loading in Glass

1) Concentration of Na_2O in Env. A glass = 20% (= 20 g Na_2O / 100 g glass)

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

3) Glass density = 2.66 MT/m^3 (=2.66 g/mL)

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

5) AW-101 actual waste ^{137}Cs concentration = 178 µCi / mL / 4.59 M Na
(= 194 µCi / mL / 5 M Na)

Na Loading in Glass

20 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) = 0.395 g Na / mL glass

Maximum ^{137}Cs:Na in glass
(3.0E-7 Ci ^{137}Cs / mL glass) / (0.395 g Na / mL glass) = 7.60 E-7 Ci ^{137}Cs / g Na

(7.60 E-7 Ci ^{137}Cs / g Na) * (23 g Na / mole) = 1.75E-5 Ci ^{137}Cs / mole Na

Maximum ^{137}Cs:Na in feed
(1.75E-5 Ci ^{137}Cs / mole Na) * (5 mole Na / L feed) = 8.74 E-5 Ci ^{137}Cs / L
= 87.4 µCi ^{137}Cs / L
= 0.0874 µCi ^{137}Cs / mL

AW-101 actual waste Cs fraction remaining (C/C_o) Contractual Limit

(0.0874 µCi ^{137}Cs/ mL) / (194 µCi ^{137}Cs / mL) = 4.51 E-4 C/C_o
= 0.0451 % C/C_o

DF for AW-101 Contractual Limit

(194 µCi ^{137}Cs / mL) / ((0.0874 µCi ^{137}Cs/ mL)) = 2220

Cs-137 Contractual Limit in AW-101 Envelope A Vitrification Feed

Assumptions, Minimum Waste Na_2O Loading in Glass

6) Concentration of Na_2O in Env. A glass = 14% (=14 g Na_2O / 100 g glass)

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

8) Glass density = 2.66 MT/m^3 (=2.66 g/mL)

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

10) AW-101 actual waste ^{137}Cs concentration = 178 µCi / mL / 4.59 M Na
(= 194 µCi / mL / 5 M Na)

Na Loading in Glass

14 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) = 0.276 g Na / mL glass

Maximum ^{137}Cs:Na in glass
(3.0E-7 Ci ^{137}Cs / mL glass) / (0.276 g Na / mL glass) = 1.09 E-6 Ci ^{137}Cs / g Na

(1.09 E-6 Ci ^{137}Cs / g Na) * (23 g Na / mole) = 2.50E-5 Ci ^{137}Cs / mole Na

Maximum ^{137}Cs:Na in feed
(2.5E-5 Ci ^{137}Cs / mole Na) * (5 mole Na / L feed) = 1.25 E-4 Ci ^{137}Cs / L
= 125 µCi ^{137}Cs / L
= 0.125 µCi ^{137}Cs / mL

AW-101 actual waste Cs fraction remaining (C/Co) Contractual Limit

(0.125 µCi ^{137}Cs/ mL) / (194 µCi ^{137}Cs / mL) = 6.44 E-4 C/Co
= 0.0644 % C/Co

DF for AW-101 Contractual Limit

(194 µCi ^{137}Cs / mL) / ((0.125 µCi ^{137}Cs/ mL)) = 1552

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