Cesium Recovery Using Savannah River Laboratory Resorcinol-Formaldehyde Ion Exchange Resin

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MAR 1990
Received
R.E. Lerch
EDMC

March 1990

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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CESIUM RECOVERY USING SAVANNAH RIVER LABORATORY RESORCINOL-FORMALDEHYDE ION EXCHANGE RESIN

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Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY

A new ion exchange resin has been developed by the Savannah River Laboratory (SRL) for the recovery of radioactive ^{137}Cs from alkaline wastes produced by reprocessing fuels from nuclear reactors. The SRL resin is a condensation polymer made from resorcinol and formaldehyde. It has been shown to be stable to chemical and radiation attack in the highly concentrated and caustic supernatant waste. Small-scale testing was completed with excellent results (Bibler and Wallace 1987). The Pacific Northwest Laboratory (PNL) was requested to initiate a test program designed to provide additional experimental data needed to support further consideration of the resorcinol-formaldehyde ion exchange treatment for the Savannah River Site (SRS) alkaline waste.

Based on the results of the PNL study, the resorcinol-formaldehyde ion exchange resin was outstanding and performed better than any other know exchanger. The average ion exchange column loading was 220 column volumes (cv) of synthetic SRS waste at 50% cesium breakthrough, or 0.05 moles Cs/L exchanger. A cesium decontamination factor (DF) of $>10^4$ was found for the treated SRS alkaline waste. A loss in cesium capacity of approximately 5 to 20% was observed after completion of seven loading/elution cycles. Approximately 99% of the cesium was eluted from the ion exchange resin in less than 10 cv of 1 M formic acid. Cesium elution product decontamination factors for sodium and potassium were ~1400 and ~2400, respectively. A detailed analysis of the results will be performed by SRL and only a general review will be included in this document.

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1.0 INTRODUCTION AND OBJECTIVES

A new ion exchange resin has been developed by the Savannah River Laboratory (SRL) for the recovery of radioactive ^{137}Cs from alkaline wastes that were produced by reprocessing fuels from nuclear reactors. The SRL resin is a condensation polymer made from resorcinol and formaldehyde. It has been shown to be stable to chemical and radiation attack in the highly concentrated and caustic supernatant waste. Small-scale testing (2 mL) at SRL has been completed with excellent results (Bibler and Wallace 1987). Boulder Scientific Co. (BSC), Boulder, Colorado then prepared kilogram quantities of the resin (BSC-187) for SRL. To establish the chemical longevity of the resin, the Pacific Northwest Laboratory (PNL) was requested to complete multiple feed/elution cycles using ^{137}Cs-spiked synthetic alkaline waste and multiple ion exchange columns.

This study was confined to the experimental measurements associated with the loading and formic acid elution of multiple 200-mL ion exchange columns. The apparatus for these tests had previously been used to develop a multiple zeolite (IONSIV IE-96) ion exchange column system for the West Valley Nuclear Services Co., Inc. In that case, the performance observed with the laboratory columns was demonstrated to be equivalent to that observed in 4-ft-diameter by 10-ft-tall columns (Bray et al. 1989).

2.0 CONCLUSIONS

At the request of the Savannah River Operations Office of the U.S. Department of Energy (DOE) and the SRL, PNL has evaluated a new cesium-selective resorcinol-formaldehyde (CSRF) ion exchange resin. This resin was developed by SRL for the recovery of cesium from alkaline wastes now being stored at the Savannah River Site (SRS). Based on the results of this study, the CSRF ion exchange resin is expected to perform better than any other known ion exchange resin. This major conclusion is based on the following observations:

- The CSRF ion exchange resin performed with excellent results during six loading/elution cycles completed in the "carousel configuration." Each of the three ion exchange columns was loaded and eluted twice, and no loss of capacity of the resin was observed. The average cesium loading was 220 cv at 50% cesium breakthrough, or 0.05 moles Cs/L exchanger.

- No detectable cesium was observed exiting the second and third columns in series when the first column had been loaded to >0.5 C/C_0 (C/C_0 is the ratio of effluent cesium concentration to that in the original feed). When the first column reached a 0.5 C/C_0 breakthrough, the effluent from the second and third columns had a cesium decontamination factor (DF) of $>10^4$. In addition, no loss of cesium DF was found when the first loaded/eluted column was placed in the third position during the subsequent loading cycle.

- A loss in cesium capacity of approximately 5 to 20% was observed after completion of seven CSRF loading/elution cycles.

- Approximately 99% of the cesium was eluted from the CSRF ion exchange resin in less than 10 cv of 1 M formic acid, at flow rates that were varied from 0.5 to 2 cv/h.

- Analyses of the feed and the formic acid elution product after CSRF ion exchange show a DF for cesium from sodium of 1400, and for cesium from potassium of 2400.

- The 14.25-in.-tall test column containing 200 mL of CSRF ion exchange resin expanded by 1.5 in. during loading and shrank by 5 in. during elution. In addition, the resin pulled away from the column wall during formic acid elution.

3.0 EQUIPMENT AND EXPERIMENTAL DESIGN

The laboratory work was divided into two phases. Both Phase 1 and Phase 2 consisted of the operation of a system for removing cesium, containing ^{137}Cs tracer, from simulated SRS alkaline salt solution.

3.1 PHASE 1--CAROUSEL CONFIGURATION

The system consisted of three ion exchange columns (A, B, and C) arranged in carousel configuration and designed so that they could be sequenced in the following manner during the completion of six cesium loading/elution tests:

Test #	Column Sequence
1	A, B, C
2	B, C, A
3	C, A, B
4	A, B, C
5	B, C, A
6	C, A, B

The tests were conducted as follows:

1) Three columns (200 mL of BSC-187, 30 to 60 mesh) were arranged in series and loaded downflow at a flow rate of 2 cv/h, which is comparable to that expected at the SRS.

2) Each of the three columns was filled with 72 g of as-received BSC-187 pretreated with 2 M sodium hydroxide (NaOH). The columns were connected in series and the excess 2 M NaOH above each exchange bed was removed through the bottom sampling valve. Feed was started through the first column and the liquid heads above each column were re-established.

3) When the first column was loaded to ~50% cesium breakthrough [^{137}Cs effluent count rate (C) ÷ ^{137}Cs feed count rate (Co) = 0.5] at approximately 250 cv, feed flow was interrupted and the system was washed downflow first with 3 cv of 2 M NaOH solution and then with 6 cv of water (2 cv/h). All three columns were then in an interstitial water condition. The NaOH wash was used to remove residual sodium aluminate from the system prior to water washing, so that aluminum precipitation would be avoided.

4) The first loaded and washed column was then detached and eluted downflow with 5 to 15 cv of 1 M formic acid at flow rates varying from 0.5 to 2 cv/h and rinsed with 3 cv of water at 2 cv/h. The first column was then reattached in the third position. The previous second column in the series then became the first, and the previous third column became the second.

5) Before loading again for the next cycle, 3 cv of 2 M NaOH wash (2 cv/h) was fed to the new first column to prevent the feed from contacting water (to prevent aluminum precipitation).

6) Samples of the feed, midfluent between the first and second columns, midfluent between the second and third columns, and the effluent were collected at regular intervals (~4 to 16 h during loading) for ^{137}Cs radiochemical analysis to establish breakthrough values (C/C_0) and DFs.

7) The temperature of the water-jacketed ion exchange columns was maintained at 35°C using a temperature-controlled water bath.

3.2 PHASE 2--SINGLE-COLUMN CONFIGURATION

The objective of Phase 1 was to repeat the feed/elution cycle six times to establish the longevity of the resin. At the end of these cycles, each of the three columns had been loaded and eluted twice. No decrease in capacity had been observed. To obtain additional data, Phase 2 was initiated using only Column C, which was loaded and eluted an additional five times using the same conditions outlined for Phase 1.

3.3 DESCRIPTION OF EQUIPMENT

The experimental apparatus consisted primarily of three columns, a feed bottle, and a weighing effluent bottle. Each column had a capacity of 200 mL of ion exchange material and was 1 in. in diameter and 16.5 in. tall (including head space for solution above the resin bed). They were equipped with water jackets connected to a temperature bath so that a constant temperature was maintained. The columns were connected so that they were fed downflow from a 15-L feed tank. A feed pump located just upstream of the first column was used to control the flow rate. A valve was located at the bottom of each column from which each column's effluent was sampled. The effluent from the last column in series was routed to the weighing bottle. The column volumes

of the effluent processed were determined by the effluent weight and specific gravity of the feed. A 24,600 ± 0.1-g balance was used to determine the weight of the effluent.

3.4 SIMULATED SRS ALKALINE WASTE

Simulated SRS alkaline supernatant was prepared according to Table 3.1 and traced with ^{137}Cs.

3.5 SAMPLE ANALYSIS

A standard sample (4 mL) of the feed (Co) was compared with the effluent (C) obtained during ion exchange processing of the SRS feed. Gamma counting was used for the determination of ^{137}Cs and was performed in the laboratory using a multichannel analyzer and NaI well detector. Samples of effluent from the bottom of each column were taken every 4 to 16 h. This was accomplished by opening the valve at the bottom of each column and collecting ~5 mL of column effluent in a plastic beaker. Exactly 4 mL of the effluent was then transferred to a test tube for counting. The test tubes and corks were numbered and the counting results logged onto data sheets, along with the results of the sample counting.

TABLE 3.1. SRS Simulated Supernatant

Component	Concentration, mole/L	Concentration, g/L
NaOH	2.90	116.0
$Al(NO_3)_3 \cdot 9\ H_2O$	0.38	142.6
$NaNO_3$	1.19	101.2
$NaNO_2$	0.71	49.0
$Na_2CO_3 \cdot H_2O$	0.20	24.8
Na_2SO_4	0.17	24.2
KNO_3	0.015	1.52
$CsNO_3$	0.00025	0.049

4.0 RESULTS AND DISCUSSION

A test program was designed to provide the experimental data needed to support further consideration of CSRF ion exchange treatment of SRS alkaline wastes. A detailed analysis of the results will be performed by SRL, and only a general review will be included in this document. This program was completed in two phases: 1) carousel configuration, and 2) single-column configuration. Results of these tests are given below.

4.1 CAROUSEL CONFIGURATION

The CSRF ion exchange resin performed with excellent results during six loading/elution cycles completed in the "carousel configuration." Each of the three ion exchange columns was loaded and eluted twice. No loss of cesium capacity of the resin was observed after the completion of this work. The average cesium loading was 220 cv at 0.5 C/C_0, or a cesium loading capacity of 0.05 moles Cs/L exchanger.

The results of the first and second loading/elution cycles (SRL #1 and #2) were not typical of those of the remaining tests. The reason for the initial poor loading performance (SRL #1) has not been identified. The resin had been presoaked in 2 M NaOH and was loaded into Column A. One possible cause for the poor loading performance was inadequate packing of the initial resin in the loading column resulting in solution channeling. However, tests SRL #3 through #6 showed very sharp cesium breakthrough performance. No detectable cesium was observed exiting the second and third columns in series (SRL #2 through #6) when the first column had been loaded to >0.5 C/C_0. When the first column reached a 0.5 C/C_0 breakthrough, the effluent from the second and third columns had a cesium decontamination factor of $>10^4$. In addition, no loss of Cs DF was found when the first loaded/eluted column was placed in the third position during the subsequent loading cycle. The cesium breakthrough values are shown in Table 4.1 and in Figure 4.1 as a function of the cumulative number of column volumes of feed passed through the column during the six loading/elution cycles. The column volumes of feed that passed through the loading column before a cesium value of 0.5 C/C_0 was obtained during each cycle varied from 195 to 239, with an

TABLE 4.1. Cesium Breakthrough Values as a Function of Feed Volume, Phase 1--Carousel Configuration

Run #1 Column A(1)[a]		Run #2 Column B(1)		Run #3 Column C(1)		Run #4 Column A(2)		Run #5 Column B(2)		Run #6 Column C(2)	
cv	C/C_o	cv	C/C_o	cv	C/C_o	cv	C/C_o	cv	C/C_o	cv	C/C_o
4	0.0016	3	0.02	52	<0.0005	177	0.0002	175	0.0002	135	0.0001
41	0.018	63	0.02	106	<0.0005	195	0.004	218	0.05	207	0.006
48	0.056	96	0.04	158	0.0005	203	0.03	226	0.15	216	0.065
70	0.10	104	0.05	187	0.31	207	0.07	233	0.30	220	0.11
101	0.19	125	0.10	195	0.50	210	0.10	237	0.47	228	0.26
129	0.29	141	0.16			220	0.23	238	0.50	232	0.39
161	0.36	160	0.27			225	0.34			239	0.50
170	0.40	172	0.34			231	0.50				
217	0.41	188	0.43								
		218	0.50								
		237	0.67								

(a) ( ) = number of times column was used as the loading column.

average value of 220 cv. Therefore, the capacity of the resin can be estimated as 220 times the cesium feed concentration (0.00025 M Cs) or 0.05 moles Cs/L exchanger.

4.2 CESIUM ELUTION USING FORMIC ACID

After cesium had been loaded onto the first column to ~0.50 C/C_o breakthrough, the system was prepared for cesium elution from the first of three columns by flushing the three-column system with NaOH to remove the aluminum-containing feed and then flushing with water to remove the NaOH. The loading column was then isolated and a solution of 1 M formic acid was used to elute the cesium downflow through the column. The elution results (SRL #1 through #6) are shown in Figure 4.2. Approximately 99% of the cesium was eluted in less than 10 cv of 1 M formic acid, at flow rates that were varied from 0.5 to 2 cv/h.

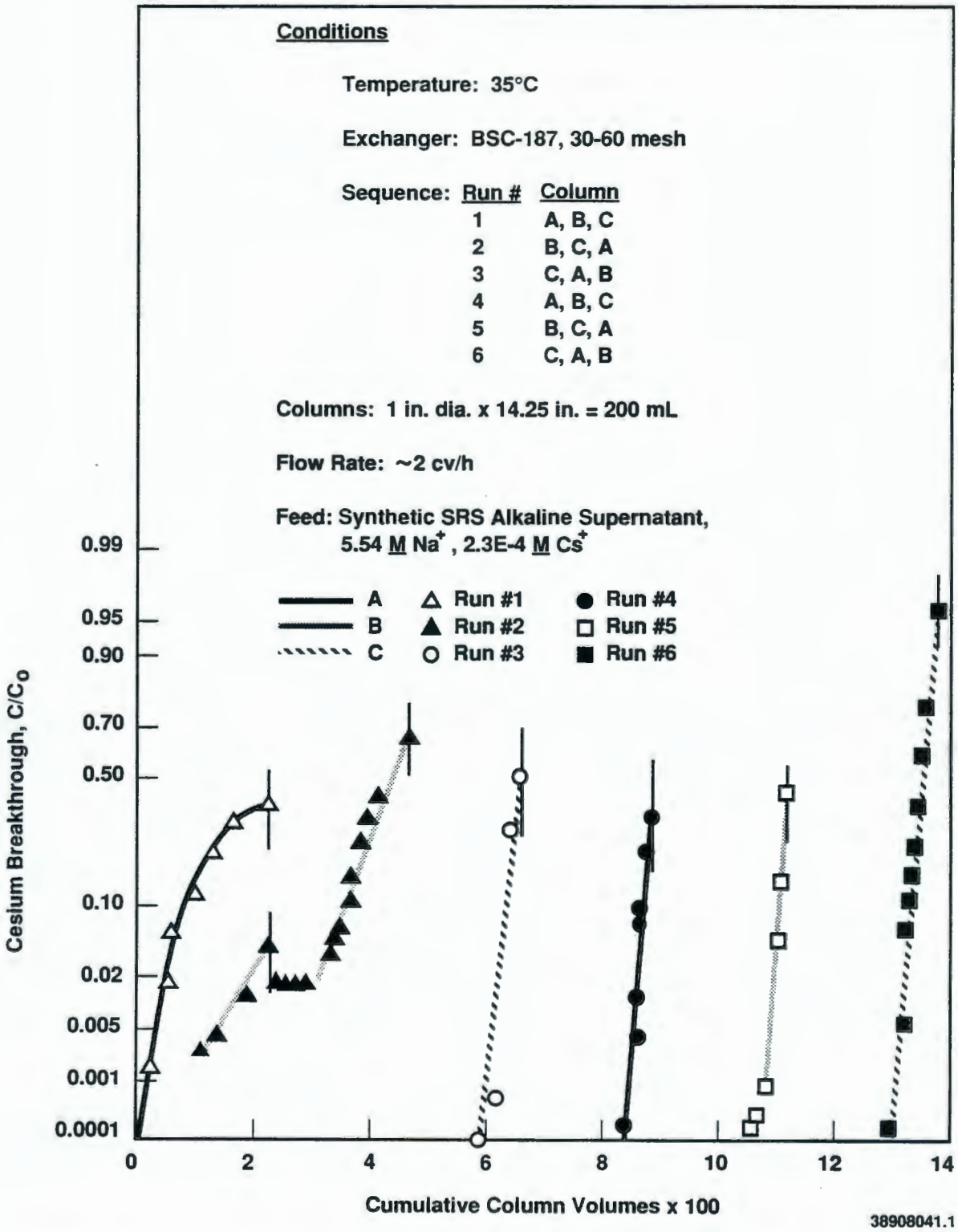

FIGURE 4.1. Cesium Breakthrough Profile for SRS Alkaline Waste Using BSC-187 Ion Exchange Resin

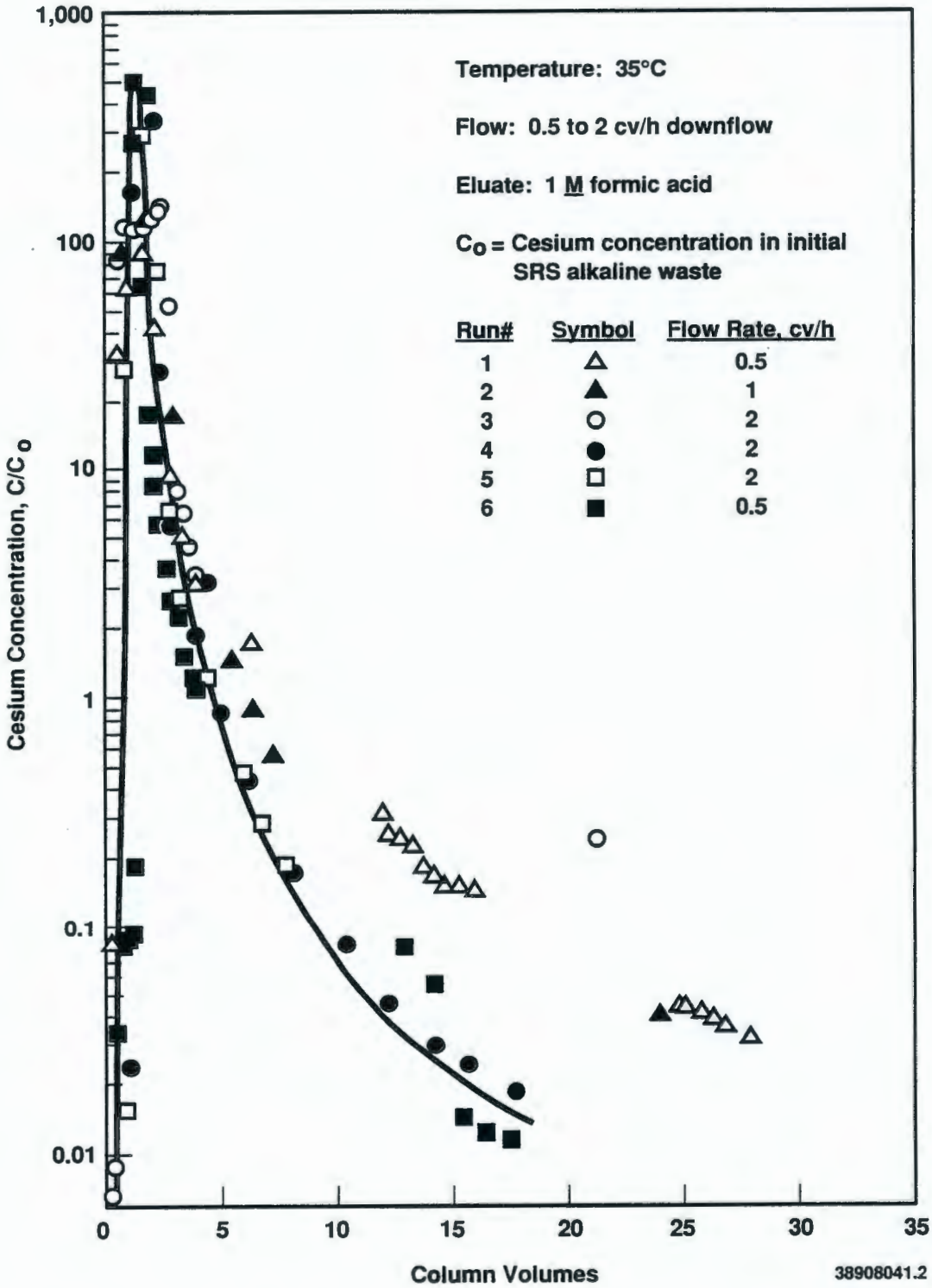

FIGURE 4.2. Cesium Elution Profile of BSC-187 Ion Exchange Resin Using Formic Acid

An analysis of a sample of the feed and the formic acid product following test SRL #11 is shown in Table 4.2. The results show that the DF for cesium from sodium was 1400, and that the DF for cesium from potassium was 2400.

4.3 SINGLE-COLUMN CONFIGURATION

The previously described carousel ion exchange tests did not shown a loss in cesium capacity after two loading/elution cycles for each of three columns. Therefore, five additional simplified tests were performed using only Column C with no trailing columns. The ion exchange resin continued to show excellent performance during the next five loading/elution cycles (SRL #7 through #11). The cesium loading capacity, based on column volumes of feed passed through the column until a cesium C/C_0 of 0.5 was obtained, varied, as follows: 195 cv (SRL #3), 239 cv (SRL #6), 233 cv (SRL #7), 203 cv (SRL #8), 188 cv (SRL #9), 190 cv (SRL #10), and 186 cv (SRL #11). Compared to the average value of 220 cv for the carousel loadings, a loss in capacity of approximately 20% appears to be a reasonable estimate after completion of a total of seven loading/elution cycles. However, the loading on the last cycle with this bed was only 5% lower than the loading on the first cycle. The results are shown in Table 4.3.

TABLE 4.2. Decontamination Factors During Loading/Elution

SRL #11 Feed:	121 g Na/L 0.583 g K/L 0.048 g Cs/L
Formic Acid Product:	0.61 g Na/L 0.0017 g K/L 0.342 g Cs/L
DF:	Na, 1,423 K, 2,443

TABLE 4.3. Cesium Breakthrough Values as a Function of Feed Volume, Phase 2--Single Column Configuration

Run #7 Column C(3)[a]		Run #8 Column C(4)		Run #9 Column C(5)		Run #10 Column C(6)		Run #11 Column C(7)	
cv	C/C_o	cv	C/C_o	cv	C/C_o	cv	C/C_o	cv	C/C_o
203	0.0001	107	0.07	175	0.165	170	0.07	171	0.20
208	0.0014	143	0.04	188	0.50	190	0.50	174	0.25
213	0.012	160	0.04	197	0.76	201	0.69	179	0.35
223	0.19	197	0.32	199	0.81	208	0.78	183	0.42
233	0.50	201	0.43	201	0.84	212	0.83	185	0.46
244	0.78	203	0.50	203	0.90	216	0.84	186	0.50
		205	0.58					199	0.71
		213	0.77						

(a) ( ) = number of times column has been used as the loading column.

4.4 PHYSICAL PROPERTIES

The CSRF ion exchange resin was pretreated with 2 M NaOH before being placed in a column (1 in. in diameter by 16.5 in. in length) having a 200-mL capacity. The 72 g of exchanger gave a bed that was 14.25 in. in height. During subsequent loading, NaOH and H_2O washing, formic acid elution, and H_2O and NaOH pretreatment of the ion exchange resin, the organic resin expanded and contracted in bed height. The maximum expansion (14.25 + 1.5 in.) occurred after the completion of loading followed by NaOH and H_2O washing, and prior to formic acid elution. The maximum contraction (14.25 - 5 in.) of the bed occurred during formic acid elution. The expansion and contraction observations during the first nine tests are summarized in Table 4.4. In addition, it was observed that during formic acid elution, the resin contracts not only in length but away from the side walls of the ion exchange column. Channeling during formic acid elution was predicted based on the

TABLE 4.4. Expansion and Contraction of Columns During Cycles

Run #	Sequence	Change in Length of Ion Exchange Column, in.[a] Before[b] Loading	After Loading	After NaOH Wash	After H_2O Wash	After[c] Elution
1	A, B, C	--[d]	--	--	--	--
2	B	+1.25	--	--	--	--
	C	+2	--	--	--	--
	A	+1	--	--	--	+1
3	C	+1.75	+0.75	+0.75	+1.38	--
	A	+1.5	-1	-0.5	+1	+0.5
	B	-1.25	-1.25	-1	+1.5	--
4	A	+0.5	-1	-0.75	+0.13	-5
	B	+1	-0.25	0	+1.5	--
	C	-2	-1.25	-1	+1.38	--
5	B	--	-0.25	0	+1	-4.5
	C	--	0	0	+1.5	--
	A	--	-0.5	-0.25	+1	--
6	C	+1	-0.5	-0.5	+0.5	-5.25
	A	+0.25	-1	-0.5	+0.75	--
	B	-1.75	--	--	--	--
7	C	+1.5	-0.38	-0.5	+0.5	-5.5
8	C	+0.75	-1.25	-0.5	+0.75	-4
9	C	+1	-0.25	0	+0.5	-5

(a) Expansion or contraction of resin assuming that 14.25 in. = 200 mL.
(b) After H_2O and 2 M NaOH wash.
(c) Only first column in series was eluted with 1 M formic acid.
(d) -- signifies no data available.

physical contraction of the organic ion exchange resin but, as shown in Figure 4.2, was not observed in the cesium elution profile except for the first elution #1.

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