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ABSTRACT

A laboratory investigation was made of an ion exchange process for removing radioactive cesium from acidic purex waste. Twenty-seven laboratory ion exchanger runs are reported. Parameters investigated include comparison of Zeolon-900, AW-500 and Duolite ARC-359 ion exchangers, pH of feed solutions, composition of sodium scrub solutions, composition of eluting solutions and upflow versus downflow elution. The cause and rate of zeolite degradation was investigated.

CONTENTS

INTRODUCTION

When Purex starts in 1983, it will be necessary to have flowsheets in place for the removal of cesium from Purex current acid waste (CAW). Previously, cesium was removed from CAW by precipitating it with phosphotungstic acid (PTA) prior to the strontium solvent extraction step.[1,2] PTA precipitation was not entirely satisfactory because of poor material balances across the precipitation step, excessive cesium losses, and the chemicals required were expensive and could not be recovered for reuse. Research was started to find an alternate to the PTA process.[3]

Preliminary laboratory work showed that zeolitic ion exchangers could remove cesium from CAW. Results of this work indicated that the ion exchange process worked better when the strontium and rare earths in the CAW were removed by solvent extraction first. The resulting raffinate was then passed through an ion exchanger. Before a flowsheet could be designed, additional laboratory work was needed to investigate parameters of the ion exchange process. The ion exchangers of interest were:

1. Zeolon-900 - a self-bonded synthetic mordenite manufactured by Norton Company, Akron, Ohio.

2. AW-500 - a naturally occurring chabazite marketed by the Linde Division of Union Carbide Corporation, Tarrytown, New York. A binder is added to the chabazite to hold the crystals together. Union Carbide changed the name of AW-500 to IONSIV IE-95. In this document, however, the designation AW-500 is used.

3. Duolite ARC-359 - a phenolic cation exchange resin manufactured by Diamond Shamrock Company, Redwood City, California. Duolite ARC-359 is a more highly refined version of Duolite C-3.

The Zeolon-900, AW-500 and Duolite ARC-359 used were aggregates which passed a 20-mesh screen but were retained on a 50-mesh screen. The particle size of the aggregates was between 279 and 864 μm. A Zeolon-900 extrudate was also tested. The extrudate was 1/16 inch in diameter and between 1/4 and 1/2 inch long.

The laboratory experiments reported in this document were generated over an 18-month period. During that time, pilot plant tests of an ion exchange process were also made.(4) Severe deterioration of the AW-500 ion exchanger was experienced in the pilot plant. Additional laboratory work was initiated to determine the cause of the deterioration.

When the work reported in this document was started, the plans were to make only a few ion exchange runs to confirm earlier work(3) and no attempt was made to design a statistical set of experiments. The data collected are not amenable to rigorous statistical analysis. However, some statistical correlations can be made.

Data from the individual laboratory ion exchange runs are included in Appendix A. The data include cesium breakthrough curves, elution curves and decontamination factors. These data are extensive and may be used for interpretation beyond what is included in this document.

SUMMARY AND CONCLUSION

Cesium can be removed from acidic waste streams with a zeolitic material such as Zeolon-900 or AW-500. The pH of the feed solution has a slight effect on the capacity of the zeolite, i.e., the lower the pH the higher the capacity. The capacity of the zeolite ranges between 60 and 100 μmol of cesium per mℓ of ion exchanger (0.23 to 0.38 mol/gal). The capacity of the phenolic Duolite ARC-359 is too low for serious consideration. Loading rates between one and three column volumes per hour had little effect on the capacity of the zeolitic ion exchangers. High flow rates resulted in higher waste losses, however.

Nitric acid is not as effective as ammonium carbonate-ammonium hydroxide [$(NH_4)_2CO_3$-NH_4OH] for eluting cesium from zeolites. Twenty to 22 column volumes were needed to remove 99% of the cesium from AW-500 with downflow elution. Zeolon-900 required 47 column volumes to elute 99% of the cesium. Upflow elution of Zeolon-900 required 23 column volumes. Upflow elution was not tried with AW-500. Duolite ARC-359 required between 12 and 14 column volumes to elute 99% of the cesium downflow. However, it should be pointed out that the initial amount of cesium on the Duolite ARC-359 column at the start of elution was an order of magnitude lower than on the zeolite columns.

Sodium decontamination factors were six times higher with AW-500 than with Zeolon-900 and almost two times higher than with Duolite ARC-359. Iron decontamination factors with AW-500 were more than three times higher than with Zeolon-900 and 70 times higher than with Duolite ARC-359. Aluminum decontamination factors were about the same for AW-500 and Zeolon-900.

Degradation of zeolitic ion exchangers is caused by chelating agents in the feed solutions. The tests did not identify any one chelating agent being worse than another for causing degradation. The amount of degradation was found to be exponential with the numbers of cycles or volume of feed. The percent of zeolite degradation followed the formula $y = 0.7e^{1.24x}$, where y = percent by volume of AW-500 deteriorated and x = the number of cycles. A parametric study of zeolite stability showed AW-500 degraded at a higher rate than Zeolon-900.

The data indicate a flowsheet can be designed to recover cesium from the acidic raffinate from B Plant 1A column (IAW). Factors which must be considered in designing the flowsheet are: (1) design a column large enough to handle all the cesium from one campaign or large enough to hold one-half the cesium from a campaign and provide enough lag storage to accumulate feed stock while the column is eluted; and (2) replace the zeolite ion exchanger after two cycles.

ION EXCHANGER EXPERIMENTS

PURPOSE

The laboratory experiments were planned to evaluate: (1) the loading characteristics of Zeolon-900, AW-500 and Duolite ARC-359 as a function of feed pH and flow rates; (2) the eluting characteristics of the ion exchangers as a function of eluant composition and direction of flow; (3) the ability of the ion exchangers to decontaminate the cesium from sodium, iron and aluminum; and (4) the stability of the zeolitic ion exchangers. A listing of the individual ion exchange runs is presented in Table 1.

EQUIPMENT

The laboratory ion exchange column was 1.9 cm in diameter. It was filled to a height of 17 cm with 20 mℓ of wet settled ion exchanger. The ion exchanger was supported by a 50-mesh screen. Solutions were pumped to the exchange column by a Fluid Metering Incorporated, Oyster Bay, New York, laboratory pump. Solutions were pumped through the column, either downflow or upflow, by changing connections to the ion exchange column. Effluents from the column were collected in 20-mℓ increments by a Scientific Manufacturing Instruments, Emeryville, California, fraction collector. A sketch of the equipment is shown in Figure 1.

Feed to the ion exchange column was synthetic IAW, composed of the following:

Iron	0.097<u>M</u>
Aluminum	0.106<u>M</u>
Magnesium	1.06×10^{-3}<u>M</u>
Calcium	9.97×10^{-4}<u>M</u>
Sodium	1.92<u>M</u>
Cesium	3.85×10^{-3}<u>M</u>
Rare earths	2.5×10^{-3}<u>M</u>
NO_3^-	1.22
$SO_4^=$	0.174<u>M</u>
Citrate	0.23<u>M</u>
Hydroxyacetate	0.15<u>M</u>

TABLE 1. Experiments for Removing Cesium From CAW.

Run	Ion Exchanger	Feed pH	Feed rate, CV/hr[a]	Scrub Composition	Eluent	
					Composition	Flow
1	Zeolon-900A[b]	4.5	1	$0.15\underline{M}\ (NH_4)_2CO_3$	$3\underline{M}\ (NH_4)_2CO_3$-$2\underline{M}\ NH_4OH$	Down
2	Zeolon-900A	6.5	1	$0.3\underline{M}\ HNO_3$	$10\underline{M}\ HNO_3$	Up
3	Zeolon-900A	1.8	1	$0.15\underline{M}\ (NH_4)_2CO_3$	$10\underline{M}\ HNO_3$	Up
4	Zeolon-900A	4.5	2	$0.15\underline{M}\ (NH_4)_2CO_3$	$10\underline{M}\ HNO_3$	Up
5	Zeolon-900A	4.5	3	$0.3\underline{M}\ HNO_3$	$3\underline{M}\ (NH_4)_2CO_3$-$2\underline{M}\ NH_4OH$	Up
6	Zeolon-900A	4.5[c]	1	$0.3\underline{M}\ HNO_3$	$3\underline{M}\ (NH_4)_2CO_3$-$2\underline{M}\ NH_4OH$	Up
7	AW-500	4.5	1	$0.15\underline{M}\ (NH_4)_2CO_3$	"	Down
8	Zeolon-900E	4.5	1	"	"	Down
9	AW-500	4.5[c]	1	"	"	Down
10	AW-500	4.5	1	"	"	Down
11	ARC-359	10.0[c]	1	"	"	Down
12	ARC-359	11.0[c]	1	"	"	Down
13	AW-500	4.5[d]	1	"	"	Down
13A	AW-500	4.5[d]	1	"	"	Down
14	AW-500	4.5[e]	1	"	"	Down
15	ARC-359	4.5[c]	1	"	"	Down
16	AW-500[f,1]	4.5[c]	1	"	"	Down
17	AW-500[f,2]	4.5[c]	1	"	"	Down
18	AW-500[f,3]	4.5[c]	1	"	"	Down
19	AW-500[f,4]	4.5[c]	1	"	"	Down
20	AW-400[f,5]	4.5[c]	1	"	"	Down
21	AW-500[f,1]	4.5[c,e]	1	"	"	Down
22	AW-500[f,2]	4.5[c,e]	1	"	"	Down
23	AW-500[f,3]	4.5[c,e]	1	"	"	Down
24	AW-500[f,4]	4.5[c,e]	1	"	"	Down
25	AW-500[f,1]	3.9[c,e,g]	1	"	"	Down
26	AW-500[f,2]	3.6[c,e,g]	1	"	"	Down
27	AW-500[f,3]	3.8[c,e,g]	1	"	"	Down

[a]CV/hr = column volumes per hour (20 mℓ/hr).
[b]A = 20- to 50-mesh aggregate; E = 1/16-inch extrudate.
[c]*Feed contained fission products other than* ^{137}Cs.
[d]Feed contained ^{95}tracer.
[e]Feed saturated with B Plant 1AX ($0.5\underline{M}$ HDEHP, $0.3\underline{M}$ TBP, NPH).
[f]Stability test; number represents number of cycles.
[g]Additional aluminum added to feed to act as blocking ion.

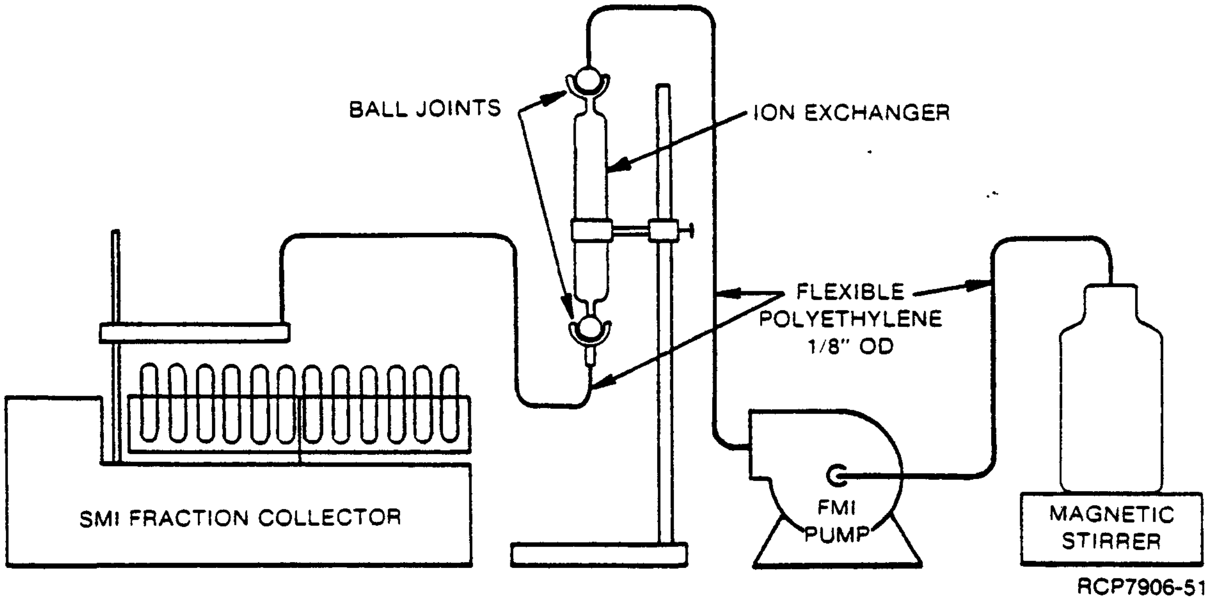

FIGURE 1. Laboratory Ion Exchange Column.

The synthetic IAW feed solution was spiked with ^{137}Cs so cesium in column effluents could be followed by gamma energy analysis (GEA). For some of the runs, an actual waste solution from Tank 101-AY was used as a spike. The 101-AY material contained ^{106}Ru - ^{106}Rh, ^{144}Ce - ^{144}Pr as well as ^{137}Cs.

LOADING CHARACTERISTICS

In all of the runs, feed solution was processed downflow through the ion exchange column. In most of the runs, 100 column volumes of feed was pumped through the column. Effluents from the column were collected in 20-mℓ (one column volume) increments. Cesium and other gamma-emitting isotopes were determined by GEA. Following the loading cycle, interstitial feed solution was displaced with two column volumes of water. Co-absorbed sodium was eluted from the column with approximately 10 column volumes of sodium scrub solution. Sodium elution was also downflow. Cesium was eluted from the column with between 30 and 60 column volumes of eluant. Direction of eluant flow was either up or down as noted in Table 1. Following elution, the column was reconditioned by pumping four column volumes of <u>6M</u> $NaNO_3$ and two column volumes of water downflow through the column. Reconditioning was necessary to convert the ion exchanger back to the sodium form following elution.

The ion exchanger loading characteristics are summarized in Table 2. The table shows the number of column volumes to 5% instantaneous breakthrough for each run. Table 2 also shows the slope of the breakthrough curve, the cumulative waste loss, the amount of cesium on the column at 5% breakthrough, and the capacity of the ion exchanger at 5% breakthrough.

TABLE 2. Ion Exchanger Loading Characteristics.

Run	Ion Exchanger	CV to 5% Breakthrough	Slope A of Breakthrough	Cumulative Waste Loss	Cesium on Column, μmol	Capacity of Ion Exchanger, μmol/mℓ
1	Zeolon-900	80	2.94	0.57	6.16	306
2	"	90	2.01	0.78	6.88	344
3	"	30	2.00	- - - Early Bleedthrough - - -		
4	"	70	2.00	0.78	1.23	61.4
5	"	53	1.6	0.93	0.93	46.4
6	"	70	2.9	0.58	1.23	61.5
7	AW-500	70	3.98	0.45	1.23	61.6
8	Zeolon-900E*	24	0.86	1.7	0.42	20.9
9	AW-500	73	4.59	0.50	5.52	276
10		- - - Not Loaded to 5% Breakthrough - - -				
11	ARC-359	11	3.79	0.49	0.09	4.4
12	"	3	0.88	1.07	0.02	1.2
13	AW-500	38	1.56	0.9	0.40	19.7
14	"	80	5.88	0.3	0.64	31.9
15	ARC-359	1.6	1.58	0.6	0.01	0.6
16	AW-500	94	5.32	0.35	1.76	87.8
17	"	98	7.09	0.22	1.83	91.6
18	"	102	4.60	0.35	1.90	95.2
19	"	98	5.40	0.35	2.09	104
20	"	102	15.6	0.15	2.18	109
21	"	>110			2.35	118
22	"	87	3.19	0.6	1.85	92.5
23	"	91	4.1	0.45	1.94	96.9
24	"	68	3.67	0.65	1.45	90.4
25	"	82	4.3	0.4	1.59	79.4
26	"	80	3.8	0.5	1.55	77.3
27	"	67	4.4	0.38	1.30	64.8

*Extrudate.

The performance of an ion exchange column is usually defined by a breakthrough curve where the ratio of the effluent concentration (C) to the feed concentration (Co) of the ion being removed (C/Co) is plotted versus the feed throughput. When equilibrium is favorable, the ion initially on the bed is displaced and moves faster through the bed than the incoming ions. This effect produces a self-sharpening boundary between the loaded and unloaded portions of the bed. The shape of this boundary reaches steady-state within a short distance and remains unchanged as it moves down the column. The breakthrough curve is determined by the steady-state shape of this boundary.

Logarithmic probability coordinates are frequently used for plotting breakthrough curves. Plotting C/Co on the probability scale and feed throughput on the logarithmic scale largely eliminates the S-shaped curve obtained with linear scale.

The equation for a straight line breakthrough curve on log probability paper is given by:

$$C/Co = 1/2\ [1 + Pi(A \ln n/n_o)]$$

where:

$Pi(x)$ = normal probability integral = $\frac{1}{\sqrt{2\pi}} \int_{-x}^{x} e \frac{1}{\sqrt{2\pi}} \int_{-x}^{x} e^{-t^2/2}\, dt$

A = slope of line

n = column volumes of feed solution

n_o = column volumes of feed at 50% breakthrough (C/Co = 0.5).

The slope A of the breakthrough curve is a measure of process kinetics and is a complex function of the ion exchange properties and operating conditions. A more complete description of the function of slope A may be found in References 5 and 6. In general, the steeper the slope of the breakthrough curve, the better the absorption of the desired ion on the ion exchanger.

Cesium concentrations in the feed solutions were determined by atomic absorption. The concentration of cesium in column effluents can be calculated by multiplying the concentration of ^{137}Cs in the effluent by the total feed solution divided by the concentration of ^{137}Cs in the feed solution. As our feed makeup was used for a number of runs, an error in cesium concentration in the feed solution would be reflected in a number of runs.

CAPACITY

The capacity of the ion exchanger at 5% instantaneous breakthrough was calculated assuming an ideal breakthrough curve.(5) The difference between the amount of cesium pumped to the ion exchange column to 5% instantaneous breakthrough and the cumulative waste loss to 5% breakthrough divided by the volume of the ion exchanger in the column gives the capacity of the column.

The high capacity of the Zeolon-900 is due to the two high capacities obtained in runs 1 and 2. These numbers are undoubtedly high. A better number probably would be 61.5 µmol/mℓ (from runs 4 and 6). Column volumes to 5% breakthrough is a good indicator of ion exchanger capacity. The data in Table 3 indicate AW-500 has a slightly higher capacity than Zeolon-900; however, the difference appears to be within experimental variation. Therefore, the conclusion is the capacity of AW-500 and Zeolon-900 is the same.

TABLE 3. Average Loading Characteristics.

Characteristic*	AW-500	Zeolon-900
Column volume to 5% breakthrough		
$\bar{x}$	83.8	72.6
s	18.05	13.74
n	16	5
Slope A		
$\bar{x}$	5.17	2.24
s	3.15	0.55
n	16	5
Capacity, µmol/mℓ		
$\bar{x}$	81.3	164
s	27.2	148
n	15	5

*$\bar{x}$ = average decontamination factor; s = standard deviation; n = number of runs used for calculation.

Feed Flow Rate

The effect of flow rate on cesium loading characteristics can be seen in Table 4 which is a summary of runs 1, 4, 5 and 6.

TABLE 4. Effect of Flow Rate on Zeolon-900 Loading Characteristics.

Run	Flow Rate, CV/hr	CV to 5% Breakthrough	Waste Loss	Slope A	Capacity, μmol/mℓ
1	1	80	0.57	2.94	306
4	2	70	0.78	2.00	61.4
5	3	53	0.93	1.6	46.4
6	1	70	0.58	2.90	61.5

From the data shown in Table 4, it is difficult to say whether flow rate has an effect on the capacity of the ion exchanger. The 306 μmol/mℓ from run 1 is apparently an error. Time did not permit rerunning virgin Zeolon-900 to test whether or not the material has large irreversible capacity. Certainly, there does not appear to be any difference in capacity between one and two column volumes per hour. Possibly the capacity is reduced somewhat at the higher flow rates. The slope A of the breakthrough curve is affected by flow rate as might be expected because slope A is a function of the ion exchange properties and operating conditions of the system. The plot of slope A as a function of flow rate is shown in Figure 2.

Cumulative cesium waste loss to 5% instantaneous breakthrough as a function of flow rate is plotted in Figure 3. As expected, waste losses increased as flows increased because of flatter breakthrough curve. No attempts were made in these studies to see if waste losses and shape of breakthrough curve was dependent on linear velocity or column flow rate.

The effect of flow rate on loading characteristics of AW-500 was not tested; however, it is expected to be similar to Zeolon-900.

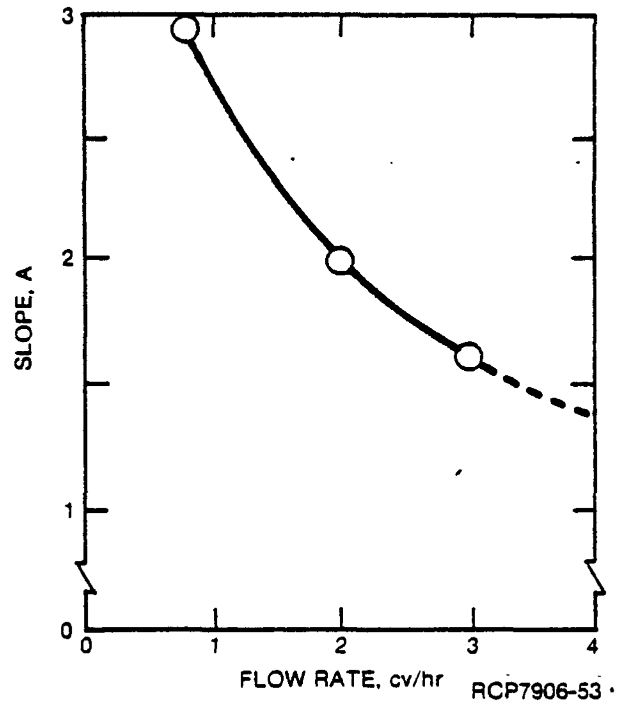

FIGURE 2. Slope of Cesium Break-Through Curve, A, as a Function of Flow Rate for Zeolon-900.

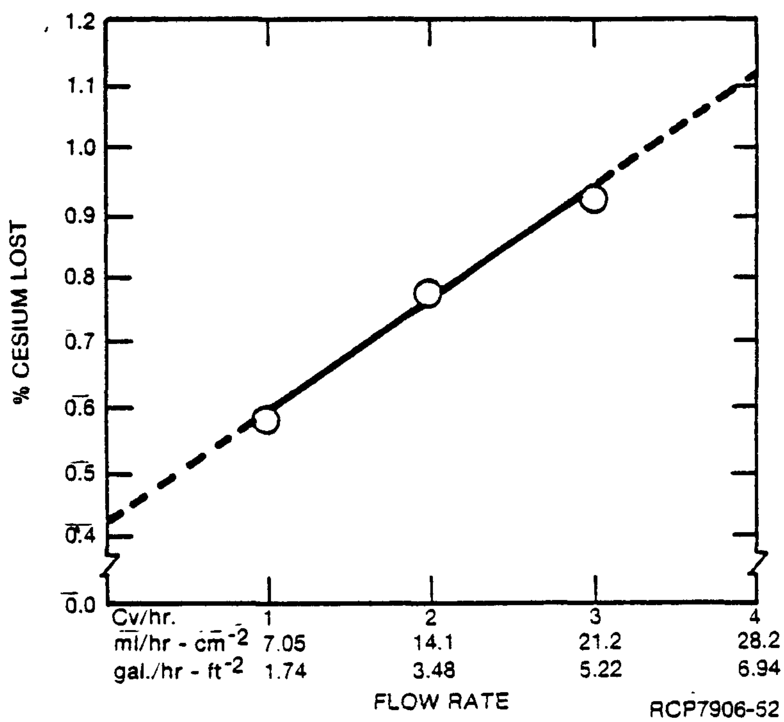

FIGURE 3. Cumulative Cesium Waste Loss to 5% Breakthrough as a Function of Flow Rate.

Feed pH

The effect of pH on Zeolon-900 loading characteristics is shown in Table 5.

TABLE 5. Effect of Feed pH on Zeolon-900 Loading Characteristics.

Run	Feed pH	CV to 5% Breakthrough	Slope A	Capacity, μmol/mℓ
3	1.8	30	2.0	---
1	4.5	80	2.9	306
6	4.5	70	2.9	61.5
2	6.5	90	2.0	344

The data in Table 5 are very scattered which makes it difficult to draw any firm conclusions. Early bleedthrough was observed in run 3 at pH 1.8, which may be due to incomplete elution of the column in run 2. Because of the similarity between Zeolon-900 and AW-500, the effect of feed pH on the loading characteristics of AW-500 was not tested.

Duolite ARC-359 has been successfully used to remove cesium from highly alkaline wastes.[7] Because of the high success of Duolite ARC-359 with a wide variety of alkaline wastes, the effect of feed pH on the loading characteristics of a Duolite ARC-359 column was tested. The results are shown in Table 6.

TABLE 6. Effect of Feed pH on Duolite ARC-359 Loading Characteristics.

Run	Feed pH	CV to 5% Breakthrough	Slope A	Capacity, μmol/mℓ
15	4.5	1.6	1.58	0.6
11	10	11	3.79	4.4
12	11	3	0.88	1.2

The data in Table 6 show that Duolite ARC-359 is not satisfactory for removing cesium from an acidic solution. It absorbs cesium from alkaline solutions, but does not have the capacity of AW-500 or Zeolon-900. Attempts to raise the feed pH higher than 11 resulted in precipitation of iron and aluminum hydroxides.

Figure 4 shows comparison of cesium absorption on Zeolon-900, AW-500, and Duolite ARC-359 as a function of pH. These data[7], developed from equilibrium batch contacts rather than column runs, indicate that the pH of the feed solution has to be 12 or higher before the Duolite ARC-359 can effectively absorb cesium. The data confirm earlier conclusions that zeolitic material is needed to absorb cesium from acid waste.[3]

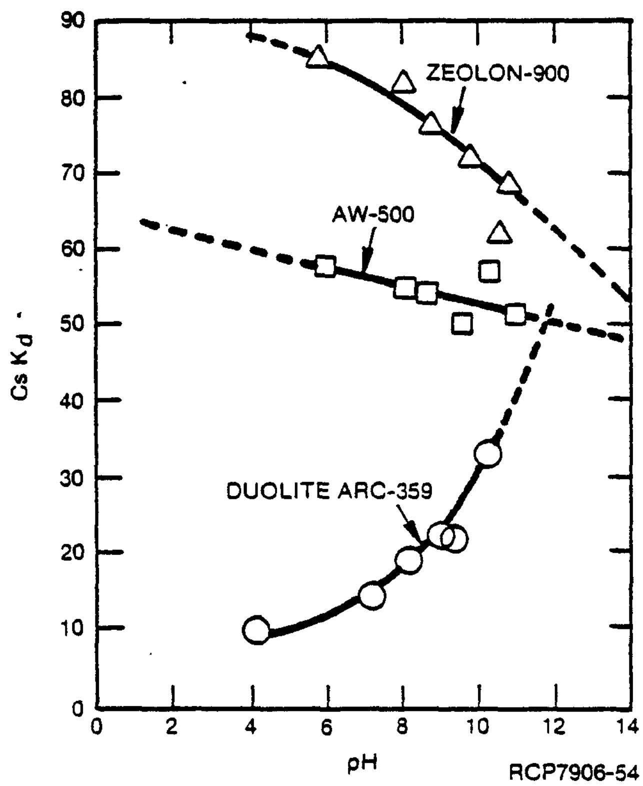

FIGURE 4. Cesium Absorption on Ion Exchangers as a Function of pH.

ELUTING CHARACTERISTICS

The eluting characteristics of AW-500, Zeolon-900, and Duolite ARC-359 were determined as a function of eluent composition and direction of flow. The number of column volumes of eluent needed to elute 50% and 90% of the cesium on the column at the start of the elution cycle are shown in Table 7. The data in Table 7 were obtained by plotting the percent of cesium remaining on the column against column volumes of eluent on semilogarithmic graph paper. The plots, which are straight lines, are provided in Appendix A.

TABLE 7. Cesium Elution Data.

Run	Ion Exchanger	Flow	Eluent	CV to Elute	
				50%	99%
1	Zeolon-900	Down	3M $(NH_4)_2CO_3$-2M NH_4OH	13.5	47
2	"	Up	10M HNO_3	6.5	56
3	"	"	"	14	39
4	"	"	10M HNO_3	11	35
5	"	"	3M $(NH_4)_2CO_3$-2M NH_4OH	8	23
6	Zeolon-900	Up	"	8	23
7	AW-500	Down	"	7	20
9	"	"	"	7	20
10	AW-500	"	"	9.5	22
11	Duolite ARC-359	"	"	3.8	11.5
12	Duolite ARC-359	Down	3M $(NH_4)_2CO_3$-2M NH_4OH	4.2	13.5

The data in Table 7 indicate Duolite ARC-359 requires at least volume of eluent to remove cesium. However, it should be pointed out that the amount of cesium on the Duolite ARC-359 column prior to elution was very small. AW-500 required about half the volume of eluting agent to remove cesium as Zeolon-900. Upflow elution of Zeolon-900 required about half the volume of eluting agent as downflow elution. Zeolon-900 could be eluted upflow with about the same volume of 3M $(NH_4)_2CO_3$-2M NH_4OH required for the downflow elution of AW-500. Strong nitric acid (10M) was not as effective for eluting cesium from Zeolon-900 as $(NH_4)_2CO_3$-NH_4OH.

DECONTAMINATION FACTORS

Decontamination factors from sodium, iron and aluminum are shown in Table 8. Decontamination factors were determined by dividing contaminate (sodium, iron, aluminum) per microcurie ^{137}Cs in the feed by contaminant per microcurie ^{137}Cs in the product solution. The data are scattered and it is difficult to make comparisons (Table 9) because much of the Zeolon-900 was eluted with nitric acid.

The data indicate sodium decontamination with Zeolon-900 is an order of magnitude higher than with Duolite ARC-359. Sodium decontamination with AW-500 is two orders of magnitude higher than with Duolite ARC-359. A similar pattern can be seen with iron and aluminum. Based on these data, AW-500 would produce the purest product material.

STABILITY

In pilot plant runs, the ion exchanger bed of AW-500 deteriorated at the end of the fifty cycle.[4] Deterioration was confirmed in laboratory runs 16 through 20 in which an AW-500 bed was repetitively loaded, water-washed, scrubbed and eluted. After the fifty cycle, the AW-500 was removed from the column and wet screened with gentle rubbing on a 50-mesh screen. Rubbing was done with a soft rubber spatula. The ion exchanger had softened and degraded so badly that it all passed through the 50-mesh screen.

A second series of load, wash, scrub, and elute cycles were made (runs 21 through 24). The ion exchanger was removed from the column after each cycle and wet screened with gentle rubbing. The volume percent of ion exchanger lost after each cycle is shown in Table 10 and plotted in Figure 5. The data show that ion exchange deterioration occurs exponentially with the number of cycles and that the AW-500 ion exchanger is good for only two cycles before it is so badly degraded it would not be prudent to continue using it.

TABLE 8. Decontamination Factors.

Run	Ion Exchanger	Elements		
		Sodium	Iron	Aluminum
1	Zeolon-900	12.9	2,759	>602
2		357	519	35.5
3		1,625	>7,759	>7,794
4		711	>7,870	>712
5		407	>8,427	>496
6	Zeolon-900	881	7,027	>339
7	AW-500	1,872	...	...
8	Zeolon-900*	3,739	...	...
9	AW-500	9,391	3.6×10^4	>2,424
10	AW-500	4,412	>245	>712
11	Duolite ARC-359	105	28	>256
12	Duolite ARC-359	28	164	1,100
13	AW-500	3,535	1.76×10^4	1,313
14	AW-500	6,619	1.23×10^4	>4,262
15	Duolite ARC-359	17	647	119
16	AW-500	3,760	6,573	1,233
17		6,624	3.30×10^4	>1,284
18		1.07×10^4	$>2.73 \times 10^4$	>1,590
19		8,054	7.93×10^4	>2,303
20		9,031	1.24×10^4	>1,836
21		509	1.61×10^4	>1,224
22		3,379	1.20×10^4	1,401
23		6,596	3.26×10^4	>1,371
24		1,030	2.08×10^4	>341
25		4,232	1,051	>1,618
26		6,453	3,055	2,261
27	AW-500	1,226	745	>1,712

*Extrudate

TABLE 9. Decontamination Comparison Data.*

Contaminate	$\bar{x}$	n	s
Zeolon-900			
Sodium	796	5	511
Iron	5,730	6	3,280
Aluminum	>1,660	6	3,010
AW-500			
Sodium	4,790	17	3,262
Iron	>19,500	16	19,900
Aluminum	>1,683	16	879
Duolite ARC-359			
Sodium	50	3	48
Iron	280	3	325
Aluminum	>490	3	530

*$\bar{x}$ = average decontamination factor; s = standard deviation; n = number of runs used for calculation.

TABLE 10. Ion Exchanger Lost.

Run	Loss	Cumulative Loss
21	2.5	2.5
22	5.1	7.5
23	13.5	32.5
24	97	98

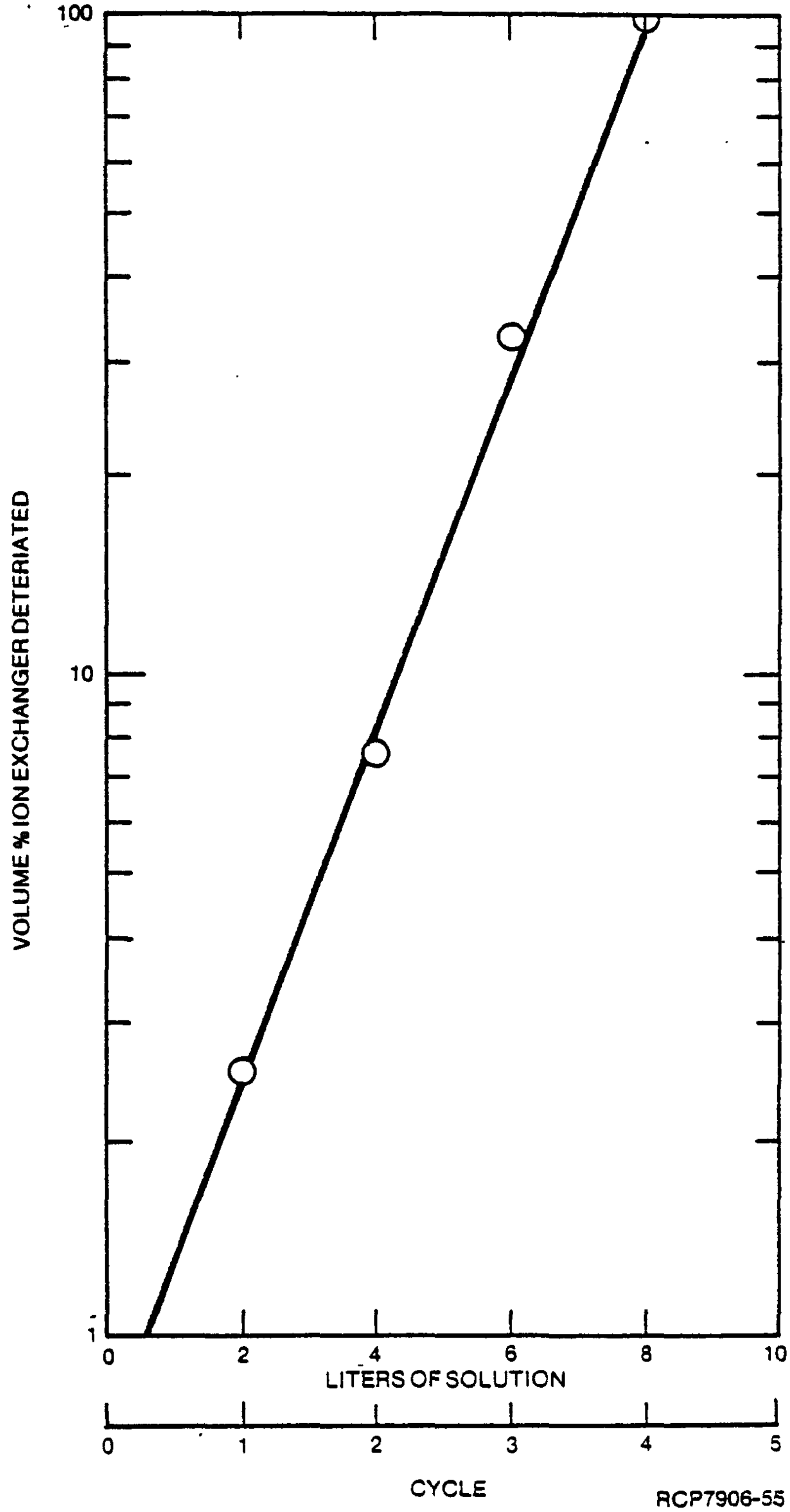

FIGURE 5. Deterioration of AW-500 in Ion Exchanger.

The cause of the deterioration was thought to be caused by the chelates used in the strontium solvent extraction process attacking the amorphous binder holding the zeolite crystals together. A parametric study was designed in an effort to identify which chelate or chelates were causing the deterioration.

The zeolitic materials used for the tests were AW-500 and Zeolon-900. The zeolites were first wet screened using a 50-mesh screen. Zeolites with particle sizes greater than 50 mesh were air dried. Ten-cubic-centimeter, tapped volume portions of the screened zeolites were placed in 100 mℓ of solution in capped, 4-oz bottles. The solutions consisted of distilled water (used as a control), and synthetic 1AW (the solvent extraction effluents generated after strontium is removed from CAW).

The composition of the 1AW used in this study is the same as the synthetic 1AW described previously except the complexant level was raised to 0.4M. Complexants used in the study included hydroxyacetic acid, citric acid, N-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), and ethylenediaminetetraacetic acid (EDTA). The first set of solutions in the study contained 0.1M each of the four complexants. Subsequent solutions contained combinations of three complexants, two complexants, and one complexant. The total complexant concentration remained at 0.4M. The pH of the solutions was initially 4.5 in each case. For each solution used, an identical second solution was saturated with B Plant 1AX consisting of 0.5M di(2-ethylhexyl)phosphoric acid (HDEHP), 0.3M tributyl phosphate (TBP), and n-paraffin hydrocarbon (NPH).

The bottles containing the zeolites were gently swirled once or twice a week. After standing a number of days, the zeolite was again wet screened on a 50-mesh screen. While screening, the zeolites were gently rubbed with a rubber spatula. When deterioration or softening of the zeolite particle occurred, the particle broke apart and passed through the 50-mesh screen. The volume of zeolite retained on the screen was measured. Volume percent lost was determined by difference. At no time was there any noticeable swelling of the particles.

Table 11 shows the volume percent AW-500 and Zeolon-900 lost in distilled water as a function of time. Table 12 shows the volume percent AW-500 lost in synthetic 1AW as a function of time. The 1AW contains combinations of the chelates as described previously. The X indicates which chelates are present. An X under the column marked 1AX indicates the solution was saturated with B Plant 1AX. In runs 33, 34, 35 and 36, the zeolites were contacted with solutions simulating the cesium eluting agent with various free hydroxide concentrations. Table 13 shows the percent Zeolon-900 lost in identical solutions to those used on the AW-500.

TABLE 11. Percent AW-500 and Zeolon-900 Lost in Water as a Function of Time.

Time, days	AW-500		Zeolon-900	
	Water	Water + 1AX	Water	Water + 1AX
28	--	--	4	6
29	16	14	--	--
63	29	24	--	--
64	--	--	10	18
70	32	36	--	--
71	--	--	14	10
74	30	36	--	--
77	--	--	18	16

TABLE 12. Ion Exchanger (AW-500) Loss as a Function of Time, Complexant and Solvent.

Days	Run	pH	Complexant in 1AW				1AX	Percent Lost	Chelates
			HEDTA	EDTA	Citric Acid	Hydroxyacetic Acid			
29	3a	4.5	X	X	X	X		20	4
29	4a	4.5	X	X	X	X	X	16	
63	3b	4.5	X	X	X	X		60	
63	4b	4.5	X	X	X	X	X	50	
70	3c	4.5	X	X	X	X		76	
70	4c	4.5	X	X	X	X	X	70	
74	3d	4.5	X	X	X	X		70	
74	4d	4.5	X	X	X	X	X	60	
75	3c	4.5	X	X	X	X		52	
75	4e	4.5	X	X	X	X	X	62	
76	4	4.5	X	X	X		X	74	3
77	6	4.5	X	X	X			76	
77	7	4.5		X	X	X		71	
77	8	4.5		X	X	X	X	70	
77	9	4.5	X		X	X		82	
83	10	4.5	X		X	X	X	80	
83	11	4.5	X	X		X		84	
83	12	4.5	X	X		X	X	86	
76	13	4.5	X			X		70	2
84	14	4.5	X			X	X	86	
84	15	4.5		X		X		76	
84	16	4.5		X		X	X	80	
84	17	4.5			X	X		50	
85	18	4.5			X	X	X	74	
85	19	4.5	X	X				78	
85	20	4.5	X	X			X	78	
85	21	4.5	X		X			83	
85	22	4.5	X		X		X	87	
85	23	4.5		X	X			84	
85	24	4.5		X	X		X	82	
85	25	4.5				X		68	1
88	26	4.5				X	X	63	
88	27	4.5	X					92	
88	28	4.5	X				X	93	
88	29	4.5		X				93	
88	30	4.5		X			X	92	
88	31	4.5			X			94	
88	32	4.5			X		X	90	
88	33		$3\underline{M}$ $(NH_4)_2CO_3$					87	
89	34		$3\underline{M}$ $(NH_4)_2CO_3$ -- $1.5\underline{M}$ NH_4OH					85	
89	35		$3\underline{M}$ $(NH_4)_2CO_3$ -- $2\underline{M}$ NH_4OH					80	
89	36		$3\underline{M}$ $(NH_4)_2CO_3$ -- $3\underline{M}$ NH_4OH					82	

TABLE 13. Ion Exchanger (Zeolon-900) Loss as a Function of Time, Complexant and Solvent.

Days	Run	pH	Complexant in 1AW				1AX	Percent Lost	Chelates
			HEDTA	EDTA	Citric Acid	Hydroxyacetic Acid			
28	3a	4.5	X	X	X	X		8	
28	4a	4.5	X	X	X	X	X	6	
64	3b	4.5	X	X	X	X		8	
64	4b	4.5	X	X	X	X	X	8	
71	3c	4.5	X	X	X	X		10	
71	4c	4.5	X	X	X	X	X	8	4
77	3d	4.5	X	X	X	X		16	
77	4d	4.5	X	X	X	X	X	16	
84	3e	4.5	X	X	X	X		16	
84	4e	4.5	X	X	X	X	X	21	
91	5	4.5	X	X	X			36	
91	6	4.5	X	X	X		X	26	
91	7	4.5		X	X	X		32	
92	8	4.5		X	X	X	X	28	3
92	9	4.5	X		X	X		34	
92	10	4.5	X		X	X	X	32	
95	11	4.5	X	X		X		43	
95	12	4.5	X	X		X	X	30	
95	13	4.5	X			X		44	
95	14	4.5	X			X	X	20	
95	15	4.5		X		X		40	
96	16	4.5		X		X	X	48	
96	17	4.5			X	X		40	
96	18	4.5			X	X	X	26	
96	19	4.5	X	X				20	2
97	20	4.5	X	X			X	26	
97	21	4.5	X		X			20	
97	22	4.5	X		X		X	24	
98	23	4.5		X	X			28	
98	24	4.5		X	X		X	24	
98	25	4.5				X		30	
98	26	4.5				X	X	26	
98	27	4.5	X					22	
98	28	4.5	X				X	24	1
99	29	4.5		X				26	
99	30	4.5		X			X	18	
99	31	4.5			X			26	
99	32	4.5			X		X	28	
99	33		3$\underline{M}$ $(NH_4)_2CO_3$					26	
99	34		3$\underline{M}$ $(NH_4)_2CO_3$ -- 1.5$\underline{M}$ NH_4OH					26	
99	35		3$\underline{M}$ $(NH_4)_2CO_3$ -- 2.0$\underline{M}$ NH_4OH					28	
99	36		3$\underline{M}$ $(NH_4)_2CO_3$ -- 3.0$\underline{M}$ NH_4OH					30	

Data from the ion exchanger parametric stability study were analyzed by D. F. Shepard of the Statistical Sciences Group. The data show:

1. The saturation of water with 1AX did not significantly increase the rate of degradation of either AW-500 or Zeolon-900.
2. During the test period, AW-500 degraded at a higher rate than Zeolon-900.
3. The presence of complexants resulted in a doubling of the rate of degradation of AW-500.
4. The presence of chelates did not change the rate of degradation of Zeolon-900.

Mr. Shepard's report is attached as Appendix B.

As mentioned earlier, degradation of the ion exchanger was thought to be due to the complexants attacking the amorphous material used to bind the zeolite crystals together. This amorphous material is made up of sodium aluminates and sodium silicates. It was theorized that possibly adding excess aluminum to the ion exchange feed solution would tie up the excess complexant in the feed thereby inhibiting attack on the amorphous binders. In runs 25, 26 and 27, excess aluminum was added to the feed solutions in an attempt to tie up the excess complexants. At the end of three cycles, the AW-500 had softened and 78% of the ion exchanger was lost.

Instead of inhibiting degradation of the ion exchanger, excess aluminum increased the degradation by a factor of 2.7. No explanation of this increase in ion exchanger degradation can be given. Additional work is needed to fully evaluate the use of blocking ions in a Purex CAW ion exchange flowsheet.

ACKNOWLEDGEMENTS

I would like to thank S. K. Fritz and S. B. Wilson-Wright, para-chemists par excellent, for making all of the ion exchange runs and collecting the analytical data.

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APPENDIX A

ION EXCHANGE RUNS FOR RECOVERING CESIUM FROM B PLANT 1AW WHILE PROCESSING PUREX ACID WASTE

Analysis of the column effluents for each run is shown in the appendix where effluents (designated by the letter L) were those collected during the loading cycle. Effluents collected furing water wash are designated by W. Sodium scrub effluents are designated by the letter S, and cesium eluent solutions by the letter E. The number following the letter designates the column volume analyzed. For example, L-20 would be the twentieth column volume during the loading cycle, S-1 would be the first scrub column volume, and E-5 would be the fifth eluent column volume. The tables show the concentrations of isotopes present in the effluents and also the ratio of the concentration of the isotope in the effluents divided by the concentration of the isotope in the feed solution to the column. This ratio is called C/Co. Eluent effluents with ^{137}Cs C/Co greater than 0.05 were composited and called product solution. The analyses of the products are also shown in the tables.

Cesium breakthrough curves during the loading cycle and elution curves are shown in the appendix. Also shown are plots of the percent cesium not eluted from the ion exchange column.

Run 1

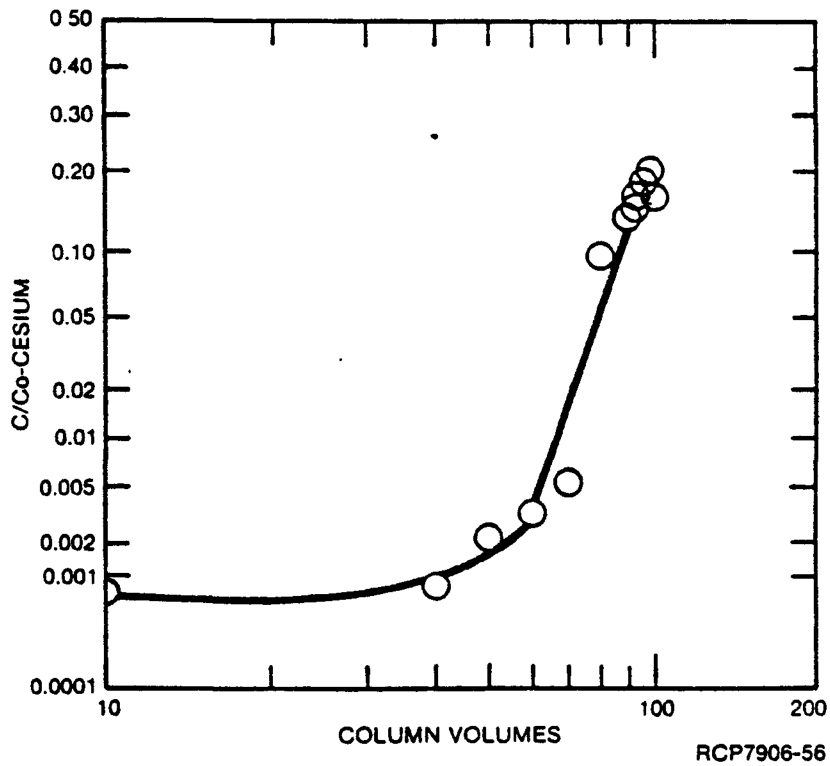
Determine cesium loading, scrubbing, and eluting characteristics of Zeolon 900.

	Feed	Product
^{137}Cs	1.62×10^{3} μCi/ℓ	2184 μCi/ℓ
Cs^{+}	3.85×10^{-3}M	---
Na^{+}	1.92M	0.202M
Fe^{3+}	0.097M	4.74×10^{-5}M
Al^{3+}	0.106M	$<2.36 \times 10^{-4}$M
pH	4.5	---

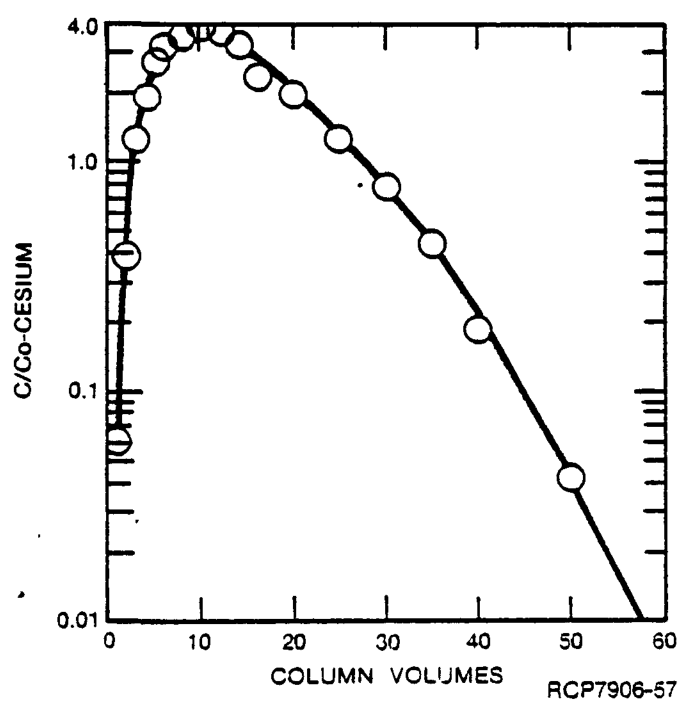
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

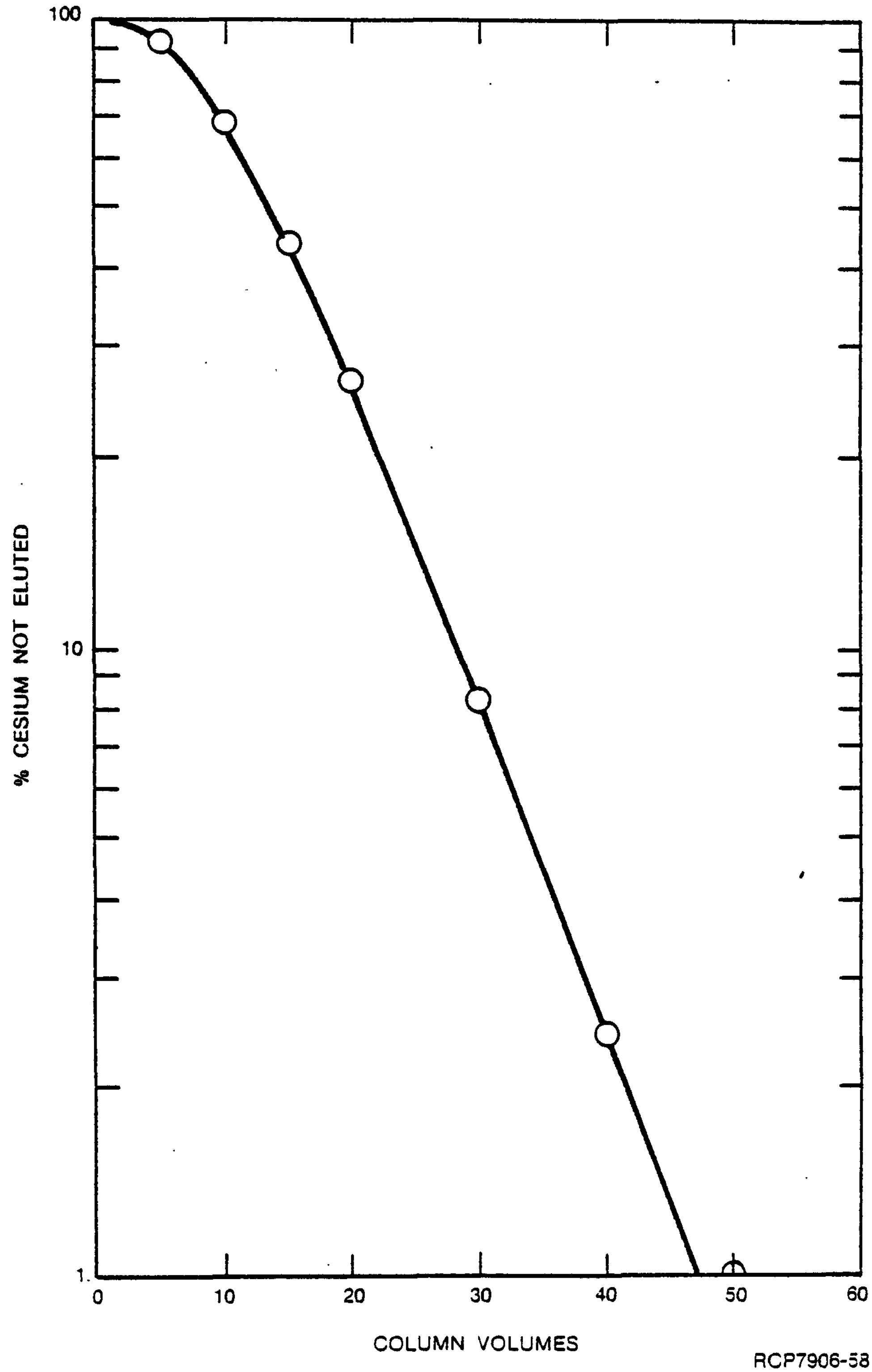
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	1	0.0007		E-1	96	0.059
L-20	1	0.0007		E-2	619	0.382
L-30	5	0.0031		E-3	1964	1.21
L-40	1	0.0007		E-4	3018	1.86
L-50	3	0.002		E-5	4323	2.67
L-60	5	0.003		E-6	5060	3.12
L-70	9	0.005		E-8	5508	3.40
L-80	142	0.093		E-10	6026	3.72
L-90	205	0.135		E-12	5899	3.64
L-96	294	0.181		E-14	5151	3.18
L-98	320	0.198		E-16	3816	2.36
L-100	265	0.163		E-20	3150	1.94
W-1,2	266	0.164		E-25	2025	1.25
S-1	294	0.181	2.13	E-30	1273	0.785
S-2	114	0.070	1.16	E-35	706	0.435
S-3	12	0.007	0.32	E-40	292	0.180
S-4	15	0.010	0.19	E-50	67	0.041
S-5	19	0.012	0.41	E-60	11	0.007
S-6	21	0.013	0.33	E-65	5	0.003
S-7	21	0.013	0.34			
S-8	22	0.014	0.25			
S-9	27	0.017	0.27			
S-10	45	0.028	0.19			
S-11	77	0.047	0.08			



Cesium Breakthrough Curve - Run 1



Cesium Elution Curve - 1



Percent Cesium Not Eluted - Run 1

Run 2

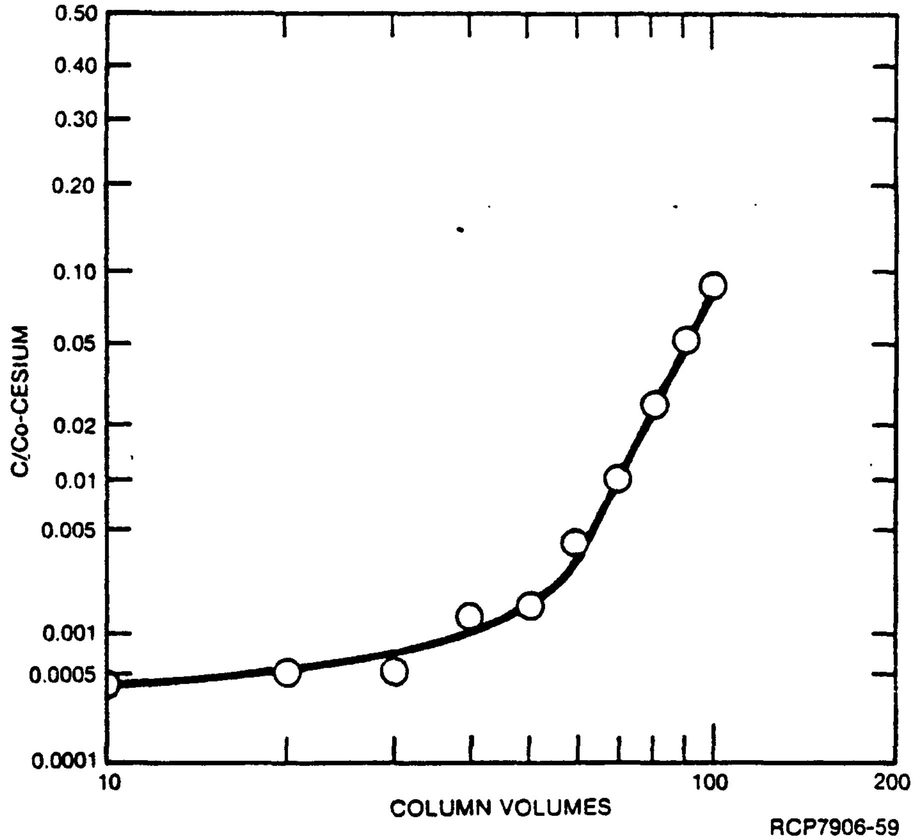
Determine the effectiveness of scrubbing coabsorbed sodium from Zeolon 900 with dilute nitric acid and eluting cesium with strong nitric acid upflow.

	Feed	Product
^{137}Cs	1.29×10^3 μCi/ℓ	1.49×10^3 μCi/ℓ
Cs^+	3.85×10^{-3}M	---
Na^+	1.92M	6.22×10^{-3}M
Fe^{3+}	0.097M	2.16×10^{-4}M
Al^{3+}	0.106M	3.45×10^{-3}M
pH	6.5	---

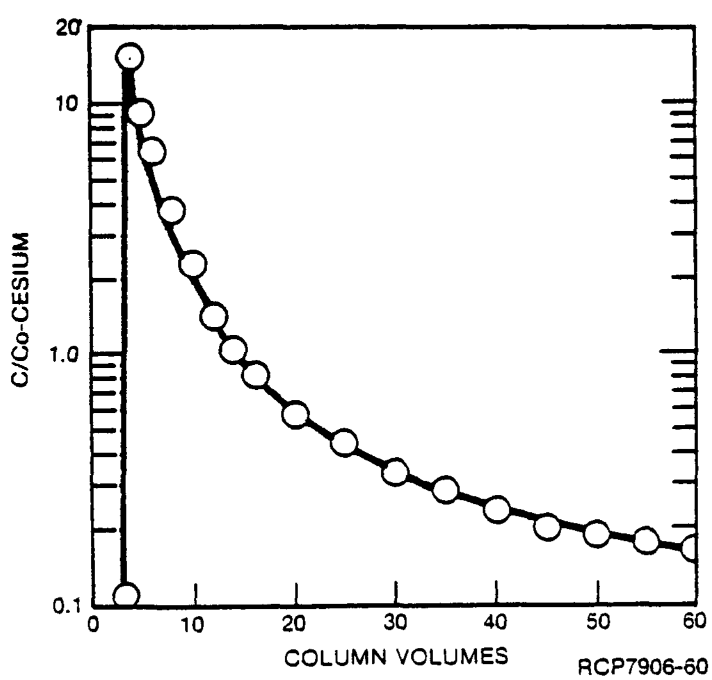
Scrub: 0.3M HNO_3

Eluant: 10M HNO_3, upflow

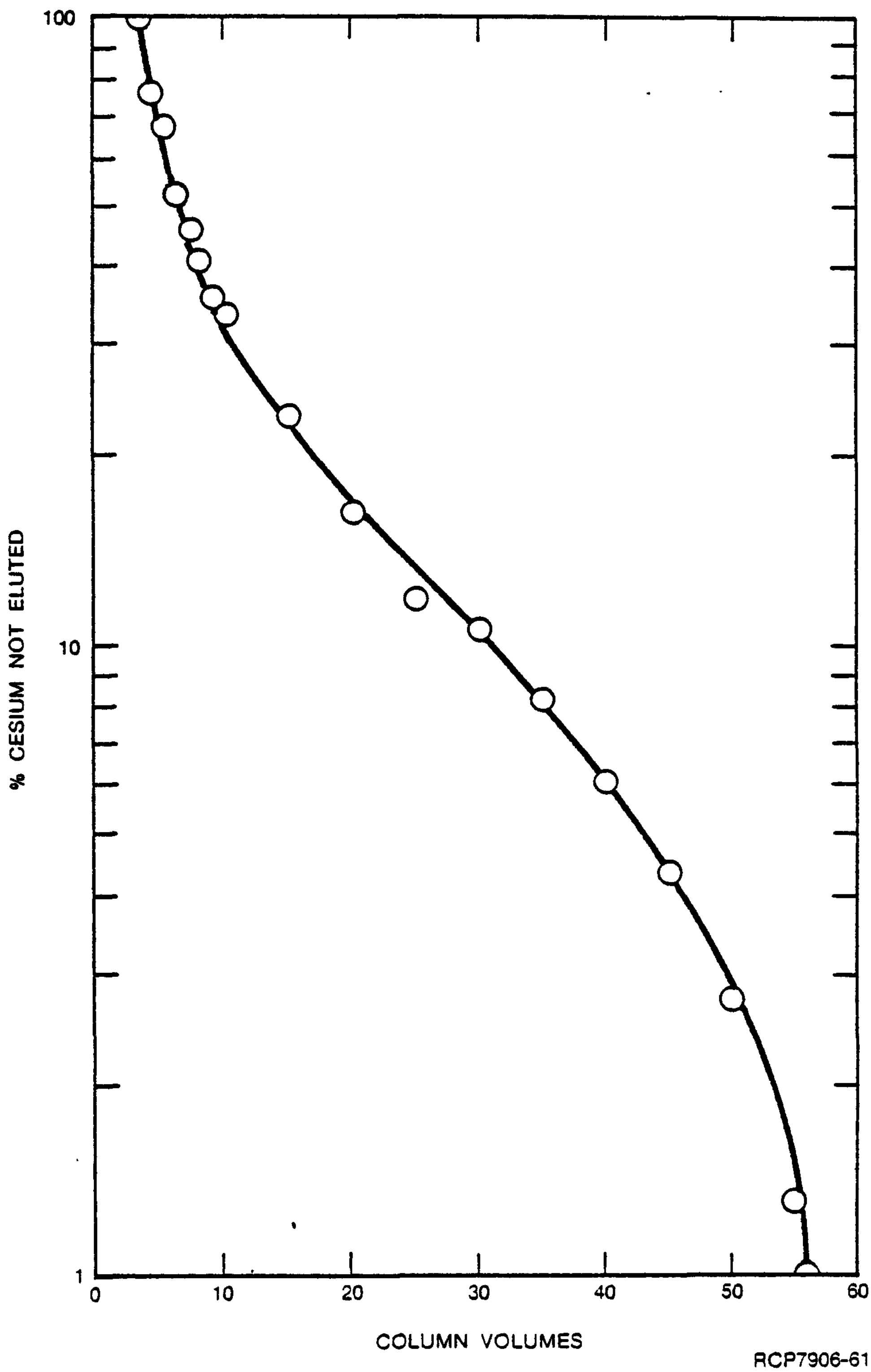
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	0.5	0.0004		E-1	5	0.004
L-20	0.6	0.0005		E-2	6	0.005
L-30	0.6	0.0005		E-3	14	0.011
L-40	2	0.0015		E-4	19800	15.3
L-50	2	0.0015		E-5	11700	9.07
L-60	5	0.0039		E-6	8210	6.36
L-70	13	0.010		E-8	4930	3.80
L-80	33	0.026		E-10	2970	2.30
L-90	65	0.050		E-12	1800	1.40
L-100	111	0.086		E-14	1320	1.02
				E-16	1040	0.81
S-1	823	0.638	1.24	E-20	735	0.57
S-2	256	0.199	0.93	E-25	561	0.43
S-3	51	0.040	0.97	E-30	429	0.33
S-4	56	0.044	1.04	E-35	366	0.28
S-5	36	0.028	0.66	E-40	297	0.23
S-6	11	0.008	0.21	E-45	254	0.20
S-7	10	0.007	0.19	E-50	211	0.16
S-8	10	0.007	0.20	E-55	220	0.17
S-9	9	0.007	0.22	E-60	204	0.16
S-10	10	0.007	0.23			
S-11	9	0.007	0.17			



Cesium Breakthrough Curve - Run 2



Cesium Elution Curve - Run 2



Percent Cesium Not Eluted - Run 2

Run 3

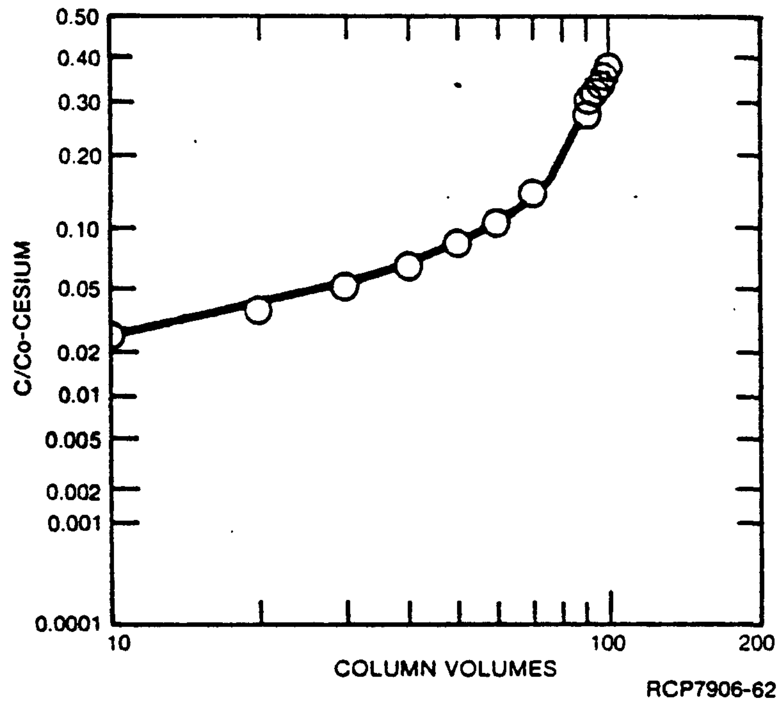
Determine effectiveness of scrubbing coabsorbed sodium from Zeolon 900 with dilute ammonium carbonate and eluting cesium upflow with strong nitric acid.

	Feed	Product
^{137}Cs	1.40×10^3 μCi/ℓ	4.468×10^3 μCi/ℓ
Cs^+	8.84×10^{-4}M	---
Na^+	1.92M	3.77×10^{-3}M
Fe^{3+}	0.097M	$<3.99 \times 10^{-5}$M
Al^{3+}	0.106M	$<4.34 \times 10^{-5}$M
pH	1.8	---

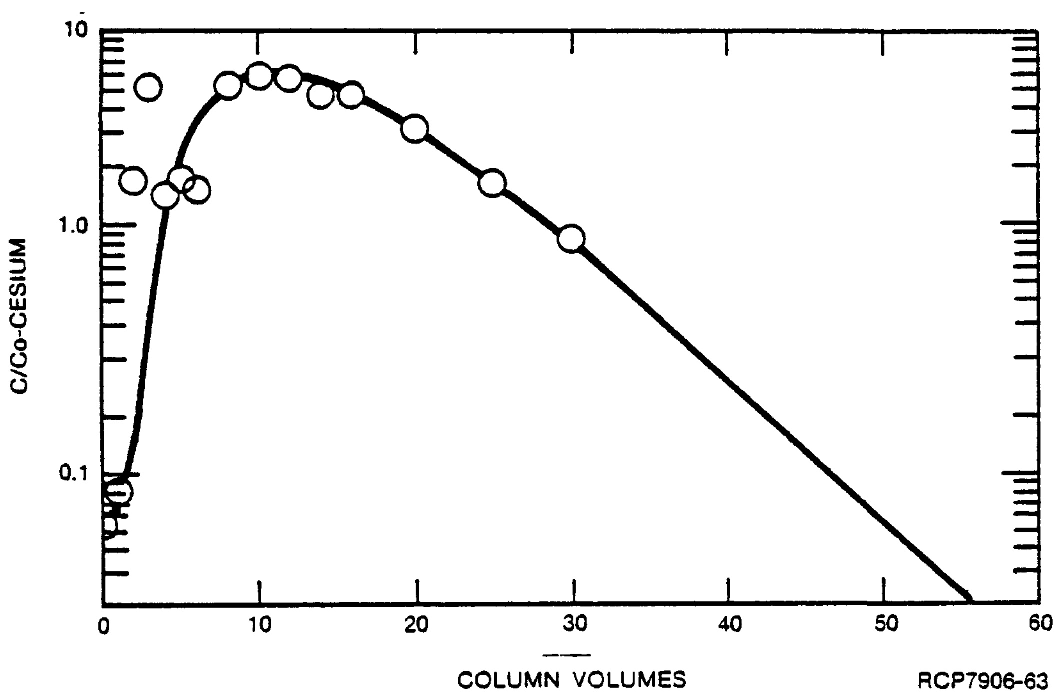
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 10M HNO_3, upflow

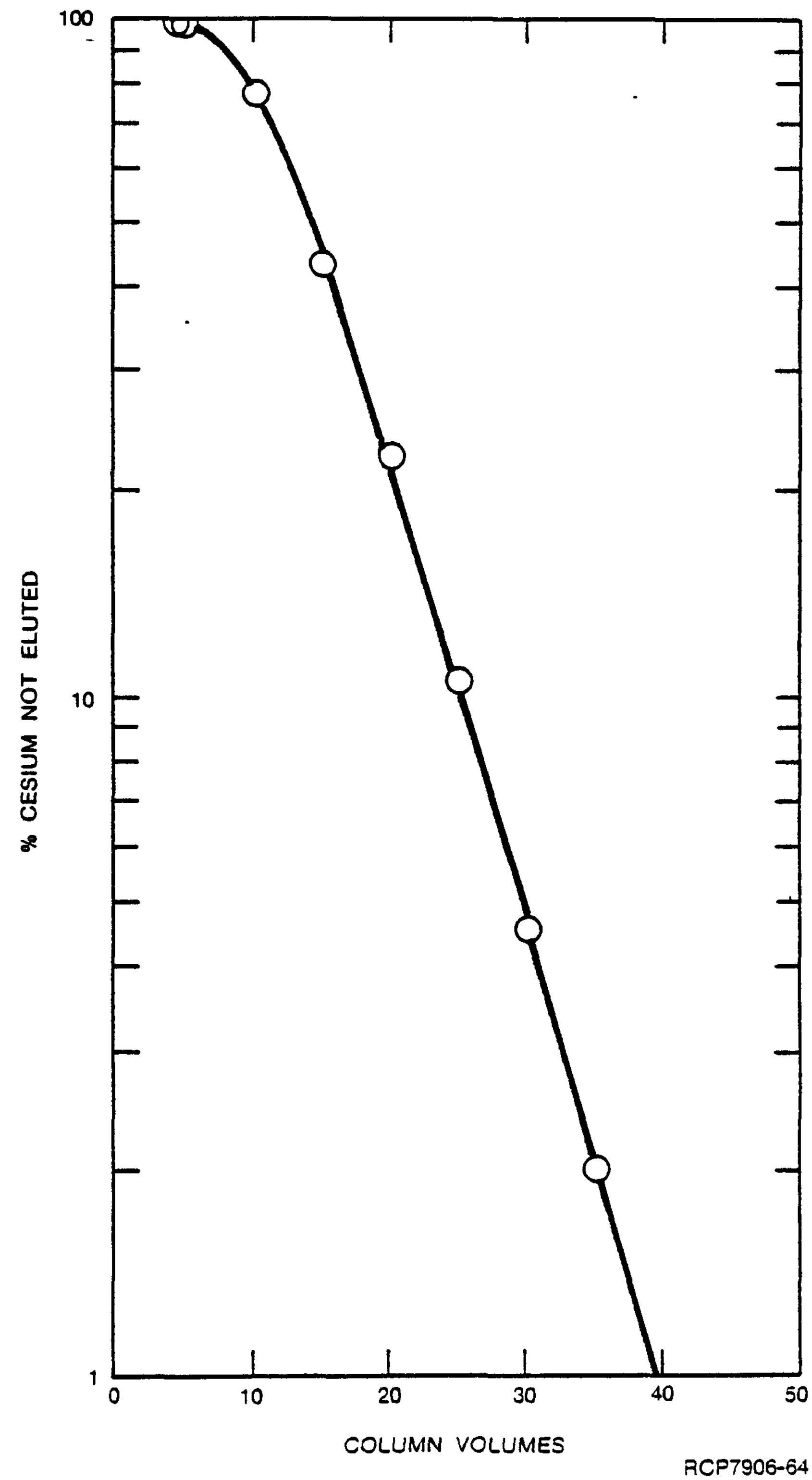
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	33	0.024		E-1	55	0.039
L-20	51	0.036		E-2	2287	1.63
L-30	69	0.049		E-3	6831	4.87
L-40	87	0.062		E-4	1920	1.37
L-50	114	0.081		E-5	2306	1.64
L-60	142	0.101		E-6	2024	1.44
L-70	193	0.138		E-8	7012	5.00
L-80	277	0.197		E-10	7921	5.65
L-90	377	0.269		E-12	7635	5.44
L-94	433	0.309		E-14	6273	4.47
L-98	512	0.365		E-16	6467	4.61
L-100	536	0.382		E-20	4200	2.99
W-1,2	53	0.038		E-25	2237	1.59
S-1	576	0.41	1.89	E-30	1138	0.81
S-2	376	0.27	1.26			
S-3	4	0.003	0.022			
S-4	3	0.002	0.016			
S-5	19	0.014	0.272			
S-6	22	0.016	0.276			
S-7	23	0.016	0.269			
S-8	23	0.016	0.266			
S-9	25	0.018	0.276			
S-10	36	0.026	0.266			



Cesium Breakthrough Curve - Run 3



Cesium Elution Curve - Run 3



Percent Cesium Not Eluted - Run 3

Run 4

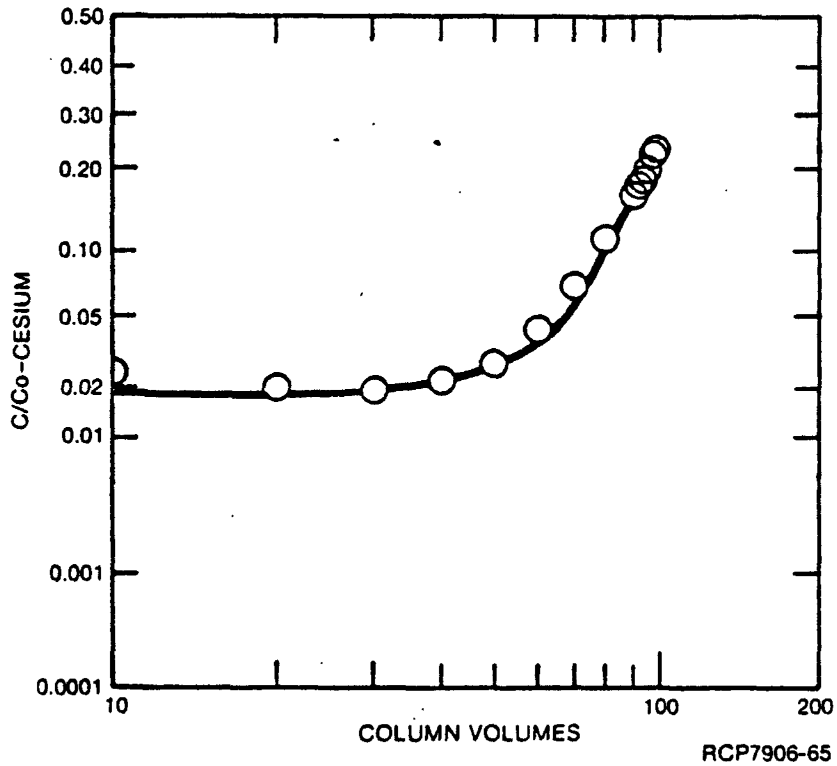
Determine cesium breakthrough curve loading Zeolon 900 at a rate of 2 column volumes per hour. Cesium eluted upflow with strong nitric acid.

	Feed	Product
^{137}Cs	1.44×14^3 µCi/ℓ	4.64×10^3 µCi/ℓ
Cs^+	8.84×10^{-4}M	---
Na^+	2.08M	9.42×10^{-3}M
Fe^{3+}	0.127M	$<5.2 \times 10^{-5}$M
Al^{3+}	0.084M	$<3.8 \times 10^{-4}$M
pH	4.5	---

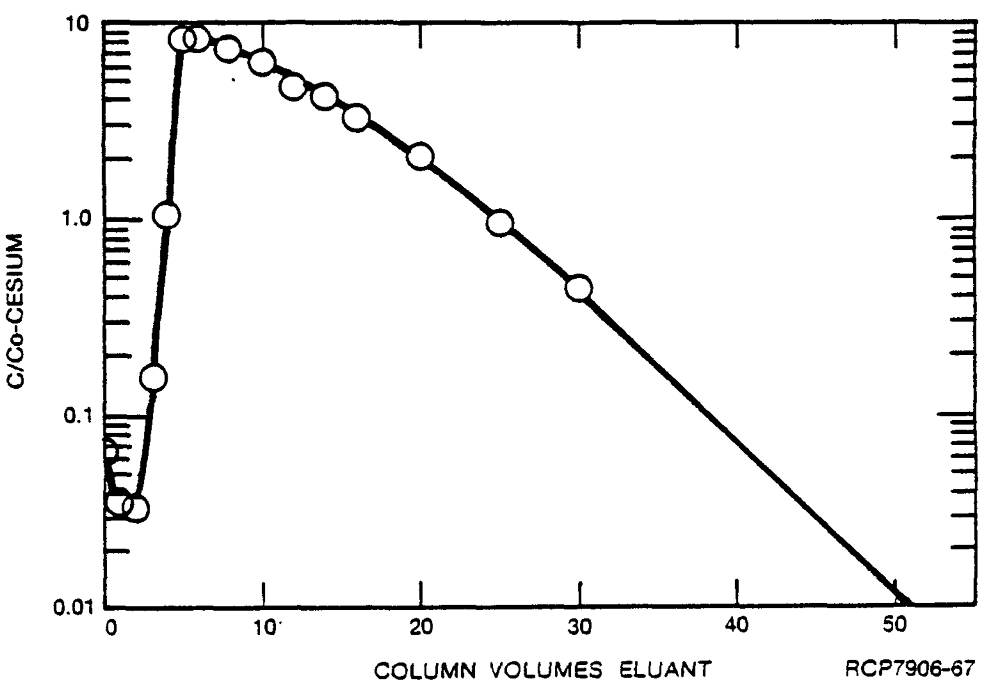
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 10M HNO_3, upflow

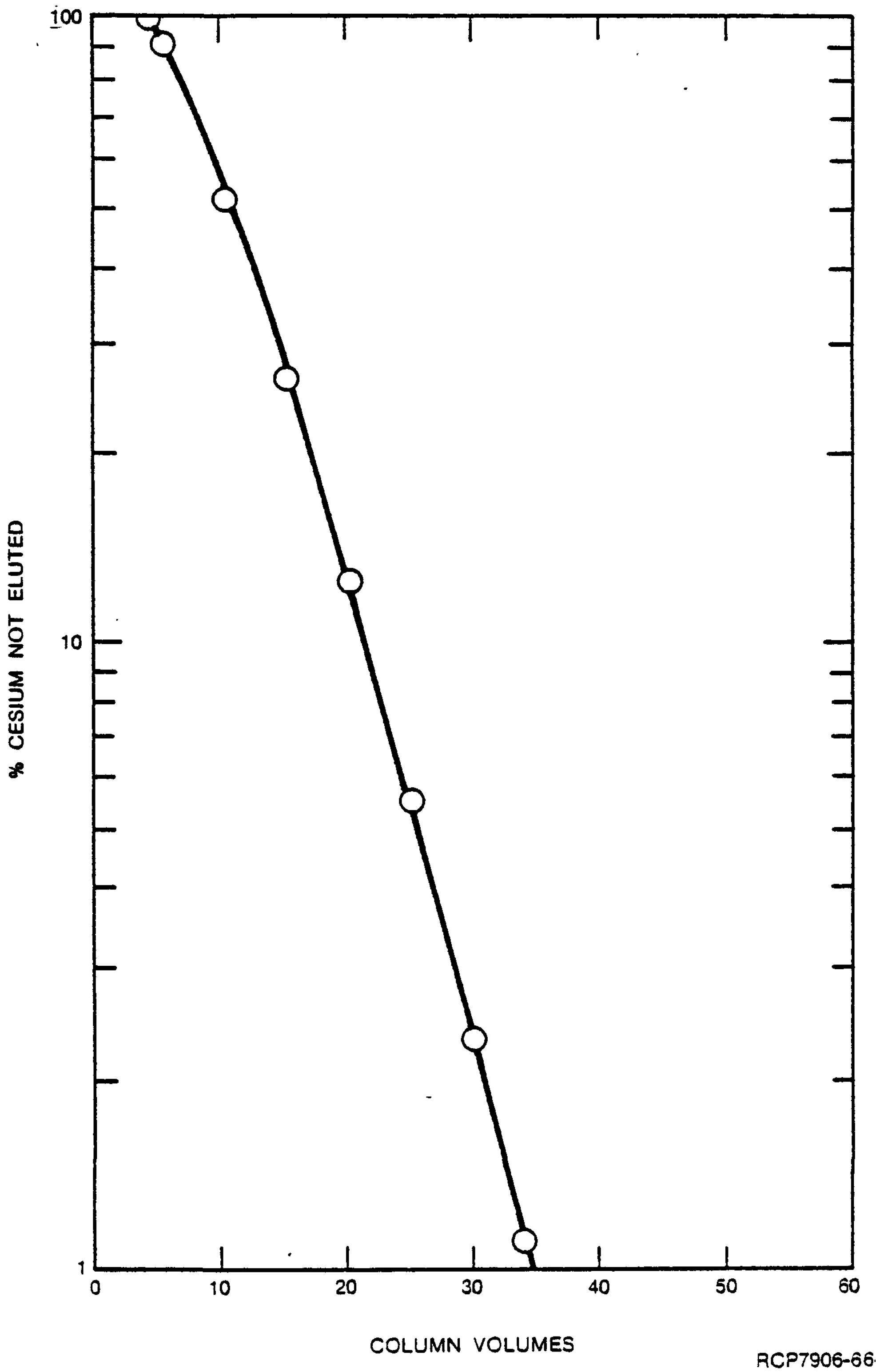
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	µCi/ℓ	C/Co			µCi/ℓ	C/Co
L-10	33	0.023		E-1	50	0.035
L-20	27	0.019		E-2	48	0.033
L-30	30	0.019		E-3	221	0.154
L-40	30	0.021		E-4	2950	2.05
L-50	38	0.026		E-5	11700	8.13
L-60	58	0.040		E-6	11700	8.13
L-70	97	0.067		E-8	10300	7.16
L-80	156	0.108		E-10	9060	6.30
L-90	232	0.161		E-12	6780	4.71
L-94	264	0.183		E-14	6000	4.17
L-98	322	0.224		E-16	4670	3.25
L-100	338	0.235		E-20	2920	2.03
W-1,2	343	0.238		E-25	1340	0.93
S-1	361	0.251	1.98	E-30	621	0.43
S-2	173	0.120	1.28			
S-3	9	0.006	0.087			
S-4	13	0.009	0.094			
S-5	19	0.013	0.146			
S-6	20	0.014	0.142			
S-7	23	0.016	0.205			
S-8	26	0.018	0.211			
S-9	39	0.027	0.195			
S-10	91	0.063	0.129			



Cesium Breakthrough Curve - Run 4



Cesium Elution Curve - Run 4



Percent Cesium Not Eluted - Run 4

Run 5

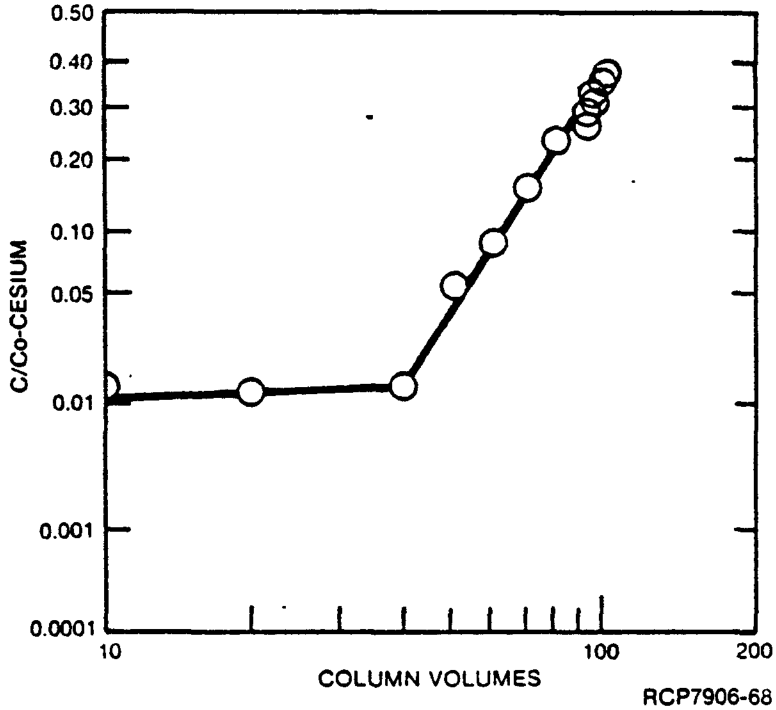
Determine cesium breakthrough curve loading Zeolon 900 at a rate of 3 column volumes per hour. Cesium was eluted upflow with ammonium carbonate.

	Feed	Product
^{137}Cs	1.38×10^3 μCi/ℓ	4.56×10^3 μCi/ℓ
Cs^+	8.84×10^{-4}M	---
Na^+	2.08M	0.0169M
Fe^{3+}	0.127M	<4.98 x 10^{-5}M
Al^{3+}	0.106M	<7.06 x 10^{-4}M
pH	4.5	---

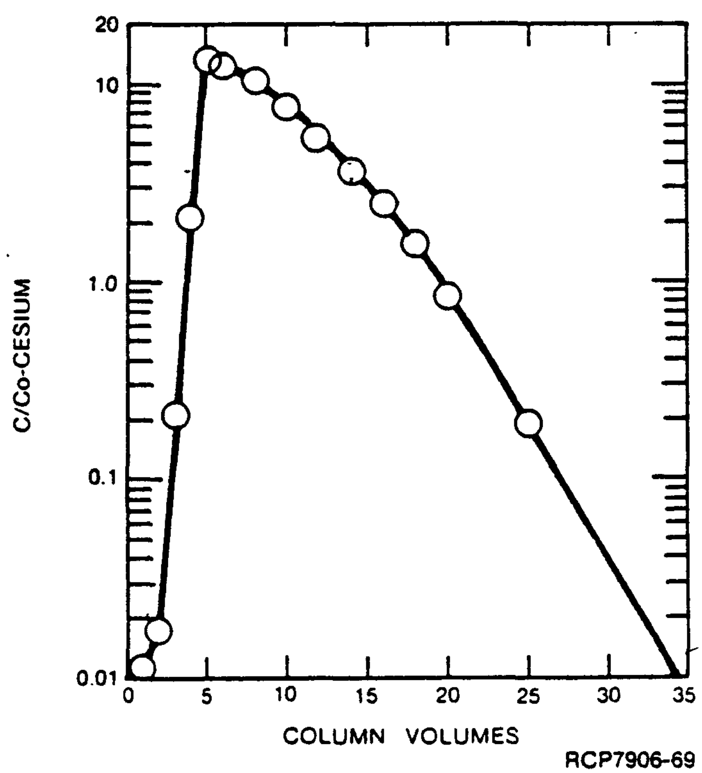
Scrub: 0.3M HNO_3

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, upflow

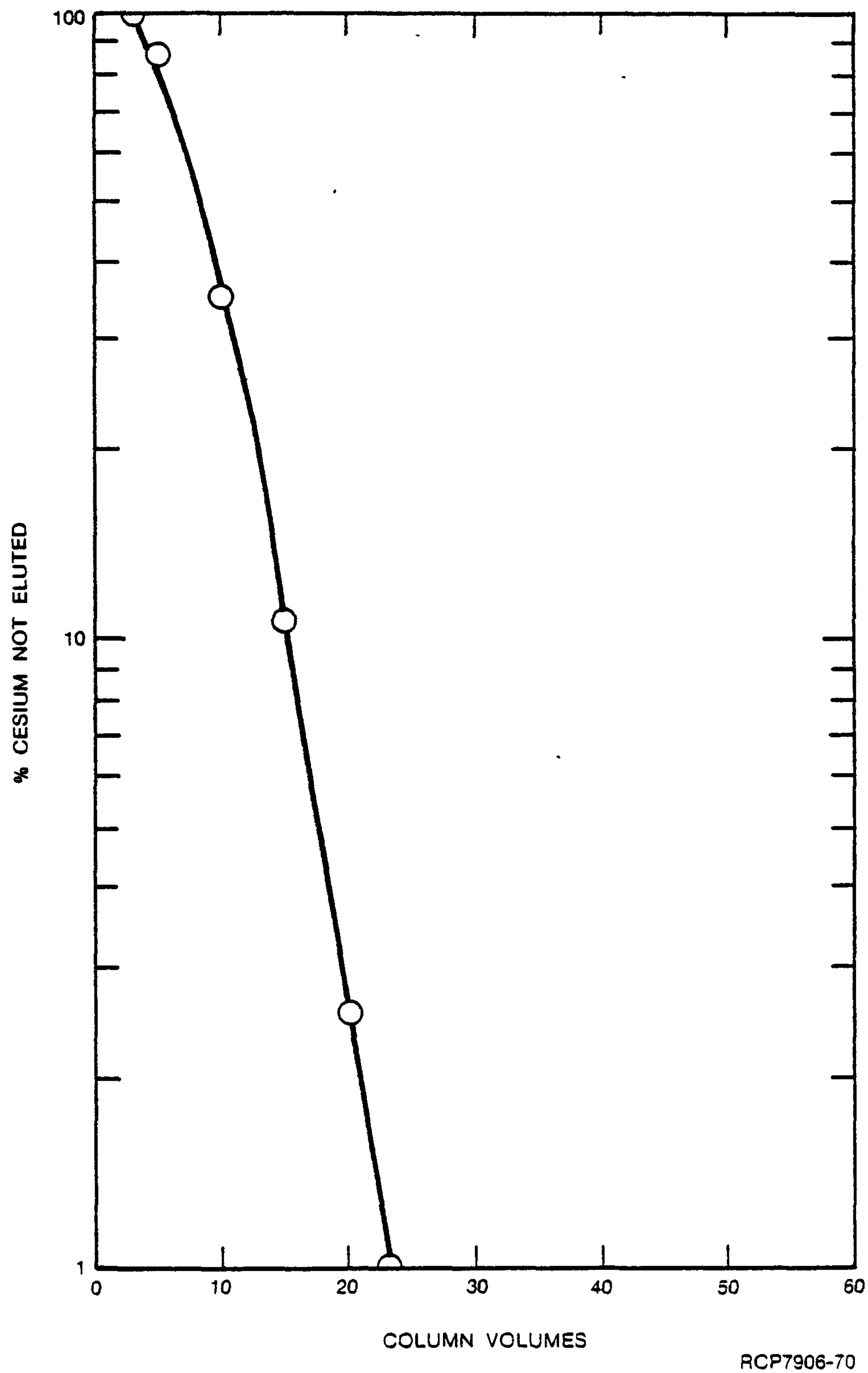
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	17	0.012		E-1	15	0.011
L-20	15	0.011		E-2	98	0.071
L-30	13	0.009		E-3	300	0.217
L-40	31	0.022		E-4	2810	2.04
L-50	71	0.051		E-5	18100	13.1
L-60	118	0.086		E-6	16800	12.2
L-70	206	0.149		E-8	14200	10.3
L-80	317	0.230		E-10	10300	7.43
L-90	356	0.259		E-12	7210	5.22
L-94	425	0.308		E-14	4870	3.53
L-98	475	0.344		E-16	3420	2.48
L-100	504	0.365		E-18	2110	1.53
S-1	516	0.374	2.56	E-20	1420	0.83
S-2	457	0.331	2.56	E-25	259	0.19
S-3	407	0.295	2.41			
S-4	82	0.059	0.883			
S-5	31	0.022	0.474			
S-6	30	0.022	0.463			
S-7	29	0.021	0.450			
S-8	30	0.022	0.335			
S-9	29	0.021	0.400			
S-10	18	0.013	0.271			
W-1,2	12	0.009	0.252			



Cesium Breakthrough Curve - Run 5



Cesium Elution Curve - Run 5



Percent Cesium Not Eluted - Run 5

Run 6

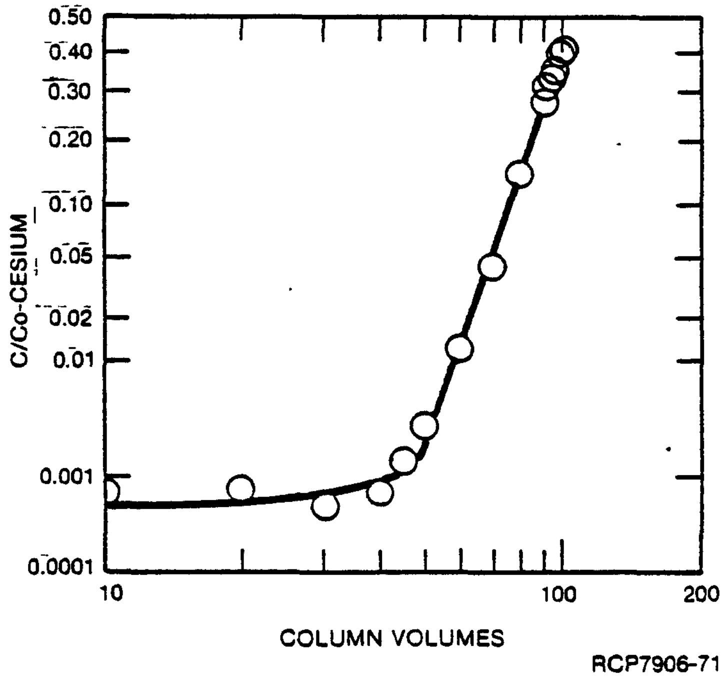
Determine path of rare earths while loading, scrubbing and eluting cesium from Zeolon 900.

	Feed	Product
^{137}Cs	4.15 x 10 μCi/ℓ	1.38 x 10^4 μCi/ℓ
^{144}Ce	71.7 μCi/ℓ	ND
Cs^+	8.84 x 10^{-3}M	---
Na^+	2.08M	7.85 x 10^{-3}M
Fe^{3+}	0.127M	6.01 x 10^{-5}M
Al^{3+}	0.084M	<8.24 x 10^{-4}M
pH	4.5	---

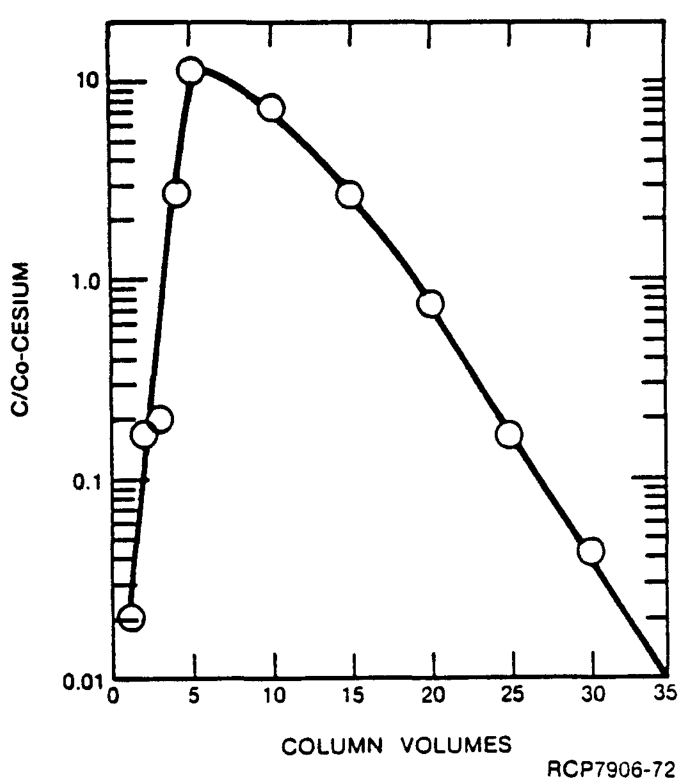
Scrub: 0.3M HNO_3

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, upflow

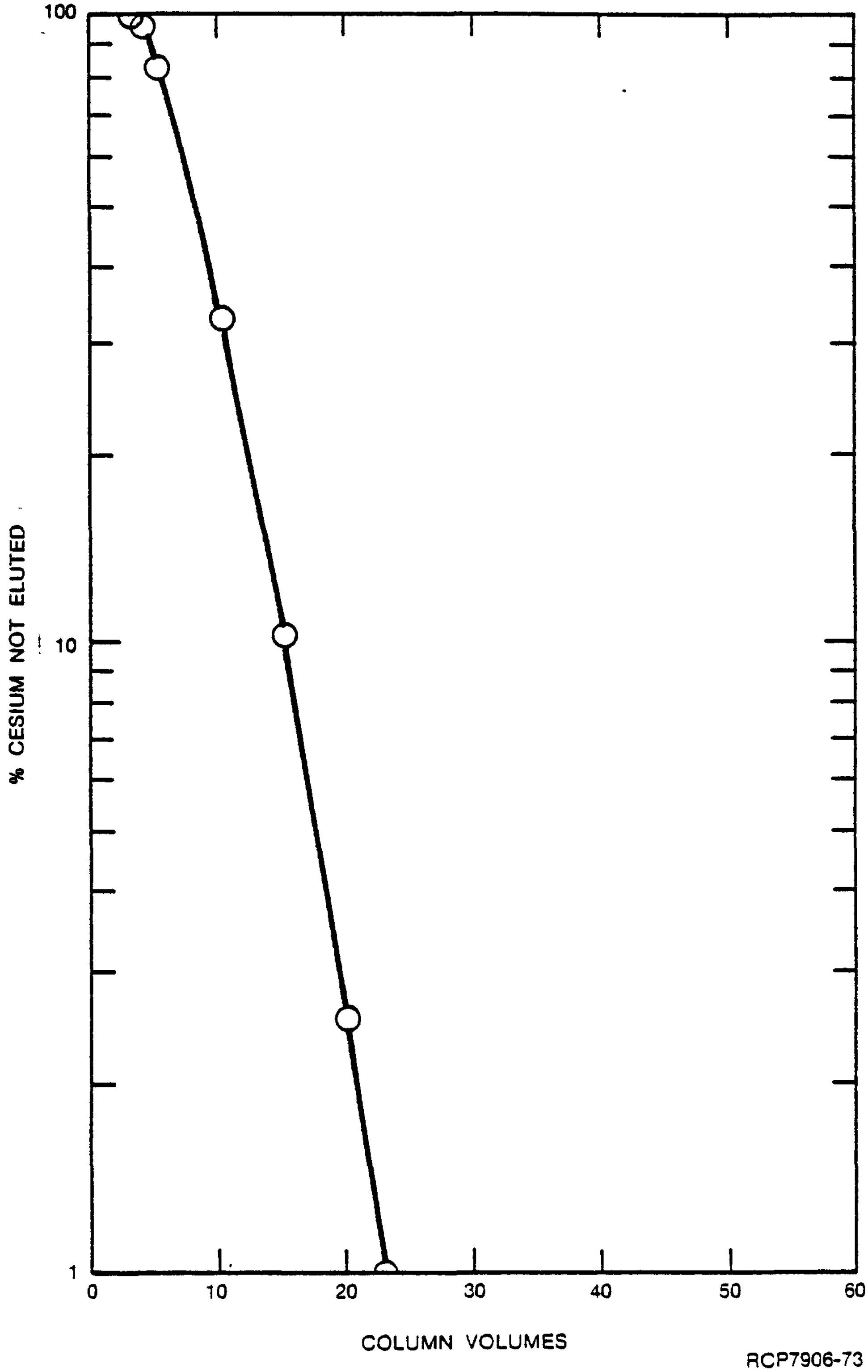
Effluent	^{137}Cs		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.0007	68	0.95		E-1	81	0.011
L-20	3	0.0007	69	0.96		E-2	696	0.168
L-30	2	0.0005	70	0.98		E-3	798	0.192
L-40	3	0.0007	69	0.96		E-4	11000	2.64
L-50	12	0.0029	66	0.92		E-5	46300	11.2
L-60	56	0.013	65	0.90		E-10	29400	7.08
L-70	191	0.046	64	0.89		E-15	11000	2.64
L-80	586	0.141	72	1.00		E-20	3010	0.73
L-90	1192	0.287	75	1.04		E-25	671	0.16
L-94	1406	0.34	72	1.00		E-30	174	0.042
L-100	1664	0.41	68	0.95				
S-1	1683	0.41	68	0.94	2.61			
S-2	1899	0.46	72	1.00	2.96			
S-3	1913	0.46	74	1.03	2.80			
S-4	67	0.016	25	0.35	1.16			
S-5	108	0.026			0.298			
S-6	116	0.028			0.346			
S-7	113	0.027			0.272			
S-8	105	0.025			0.071			
S-9	103	0.025			0.158			
S-10	68	0.016						



Cesium Breakthrough Curve - Run 6



Cesium Elution Curve - Run 6



Percent Cesium Not Eluted - Run 6

Run 7

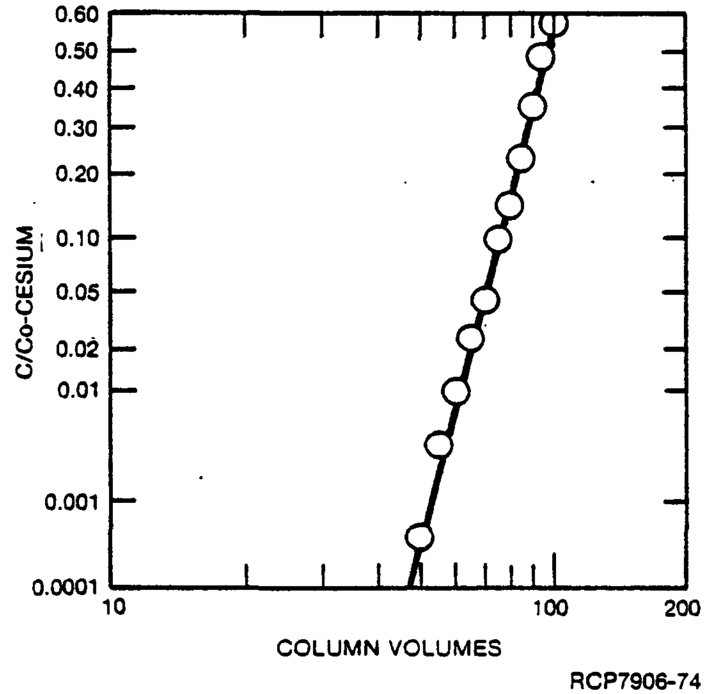
Determine cesium loading, scrubbing and eluting characteristics on AW-500.

	Feed	Product
^{137}Cs	1.17×10^{3} μCi/ℓ	3.18×10^{3} μCi/ℓ
Cs^{+}	8.84×10^{-4} M	---
Na^{+}	2.08M	3.02×10^{-3} M
Fe^{3+}	0.127M	---
Al^{3+}	0.084M	---
pH	4.5	---

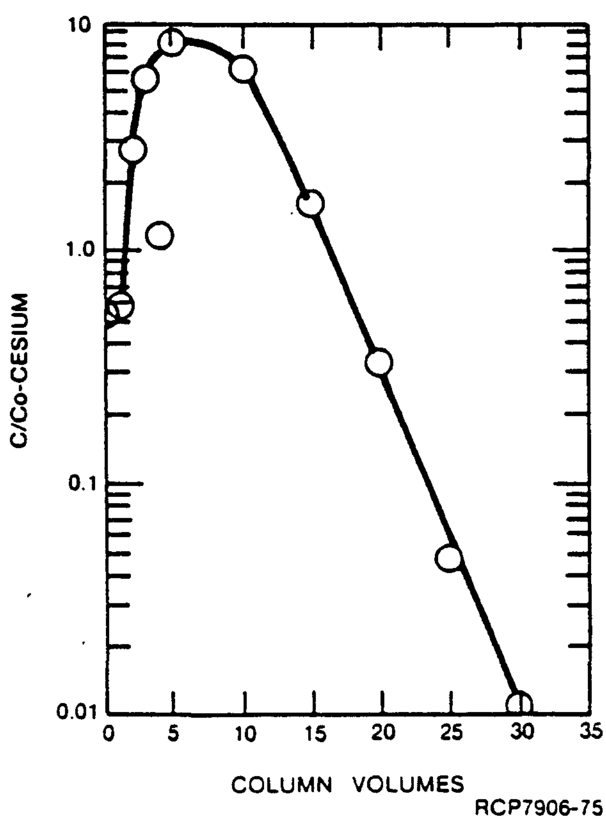
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

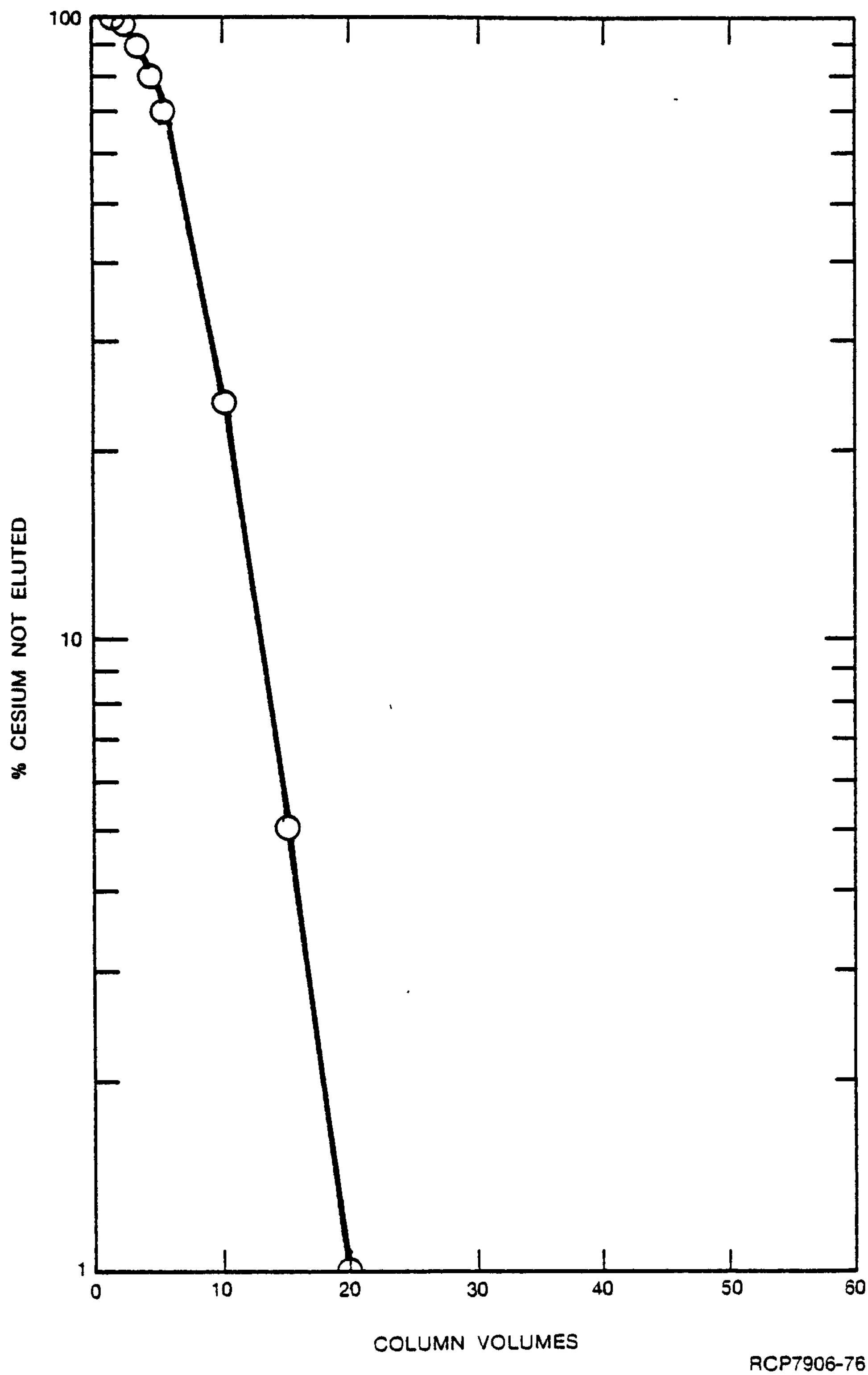
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	0.9	0.0007		E-1	663	0.568
L-20	1.2	0.0009		E-2	3160	2.71
L-30	ND	--		E-3	6520	5.59
L-40	ND	--		E-4	8400	7.20
L-50	1.6	0.0014		E-5	9460	8.11
L-60	11	0.009		E-10	7240	6.21
L-70	50	0.043		E-15	1860	1.60
L-80	168	0.144		E-20	384	0.33
L-90	414	0.355		E-25	55	0.047
L-95	559	0.479		E-30	13	0.011
L-100	666	0.571				
W-1,2	669	0.573				
S-1	620	0.531	1.65			
S-2	41	0.035	0.068			
S-3	22	0.019	0.097			
S-4	33	0.028	0.067			
S-5	41	0.035	0.065			
S-6	43	0.037	0.067			
S-7	51	0.044	0.161			
S-8	226	0.194	0.119			
S-9	528	0.453	0.020			
S-10	536	0.460	0.004			
S-11	603	0.517	0.015			



Cesium Breakthrough Curve - Run 7



Cesium Elution Curve - Run 7



Percent Cesium Not Eluted - Run 7

Run 8

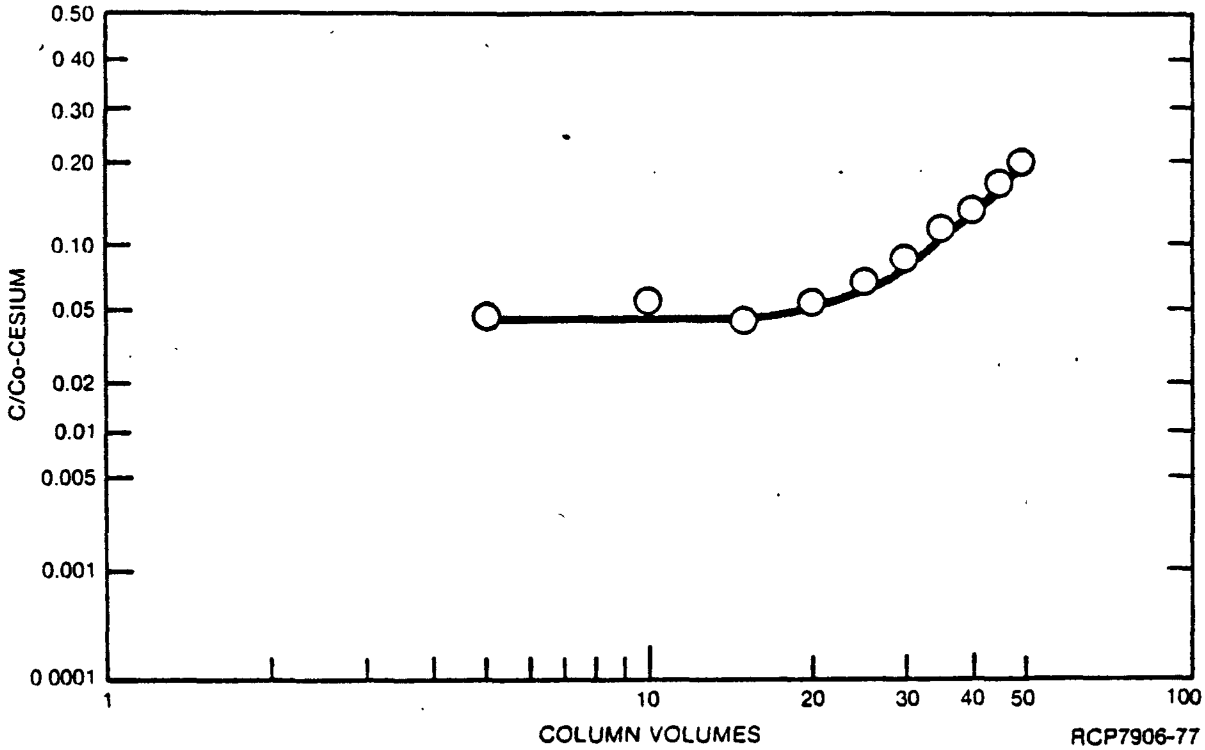
Determine cesium loading, scrubbing and eluting characteristics with Zeolon 900 extrudate.

	Feed	Product
^{137}Cs	1.86 x 16^{3} μCi/ℓ	2.15 x 10^{3} μCi/ℓ
Cs^{+}	8.84 x 10^{-4}M	---
Na^{+}	2.08M	6.43 x 10^{-4}M
Fe^{3+}	0.127M	---
Al^{3+}	0.084M	---
pH	4.5	---

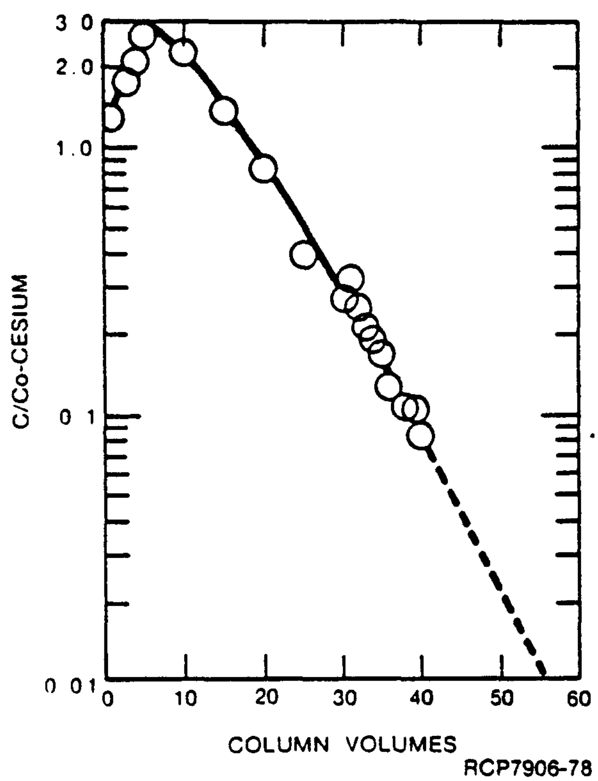
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-5	86	0.046		E-1	2390	1.29
L-10	98	0.053		E-3	3210	1.73
L-15	82	0.044		E-4	3820	2.06
L-20	100	0.054		E-5	4770	2.56
L-30	153	0.082		E-10	4160	2.24
L-40	256	0.138		E-15	2530	1.36
L-50	378	0.203		E-20	1550	0.83
W-1,2	367	0.197		E-25	731	0.39
S-1	65	0.035	0.430	E-30	497	0.27
S-2	12	0.004	0.090	E-35	313	0.17
S-3	21	0.011	0.123	E-40	152	0.082
S-4	32	0.017	0.145			
S-5	36	0.019	0.171			
S-6	50	0.027	0.166			
S-7	93	0.050	0.142			
S-8	135	0.073	0.101			
S-9	168	0.090	0.062			
S-10	196	0.105	0.030			



Cesium Breakthrough Curve - Run 8



Cesium Elution Curve - Run 8

Run 9

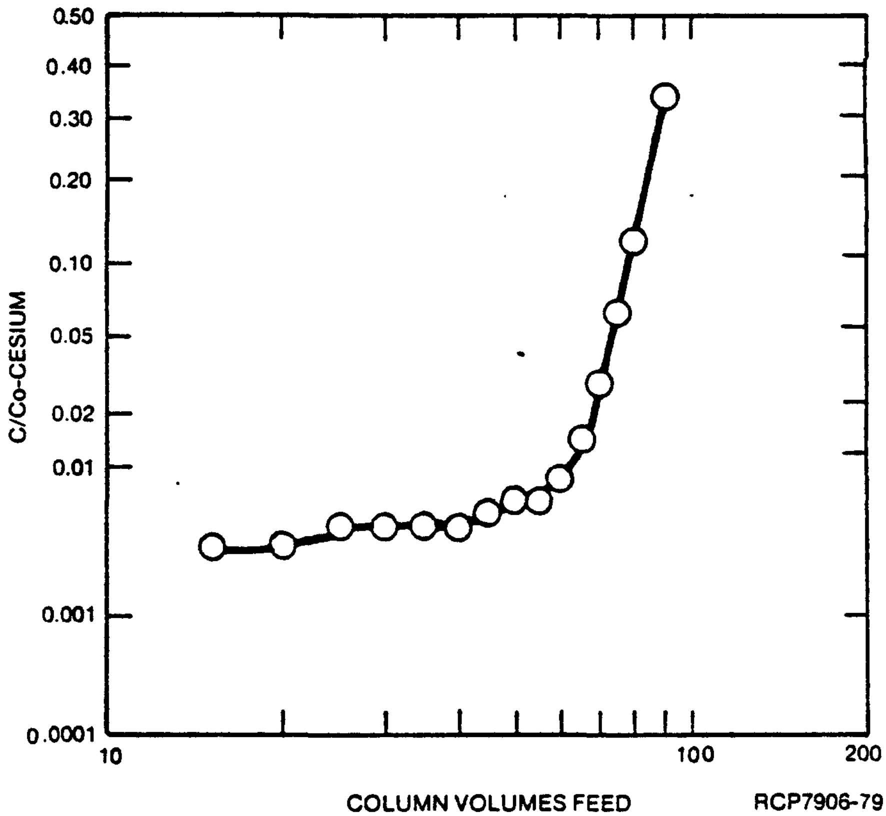
Determine cesium loading, scrubbing, and eluting characteristics with AW-500.

	Feed	Product
^{137}Cs	1.34×10^{3} μCi/ℓ	5.99×10^{3} μCi/ℓ
^{106}Rh	121 μCi/ℓ	ND
^{144}Ce	48 μCi/ℓ	<10 μCi/ℓ
Cs^{+}	3.8×10^{-3} M	---
Na^{+}	1.96M	9.33×10^{-4} M
Fe^{3+}	0.105M	1.3×10^{-5} M
Al^{3+}	0.122M	$<2.25 \times 10^{-4}$ M
pH	4.5	---

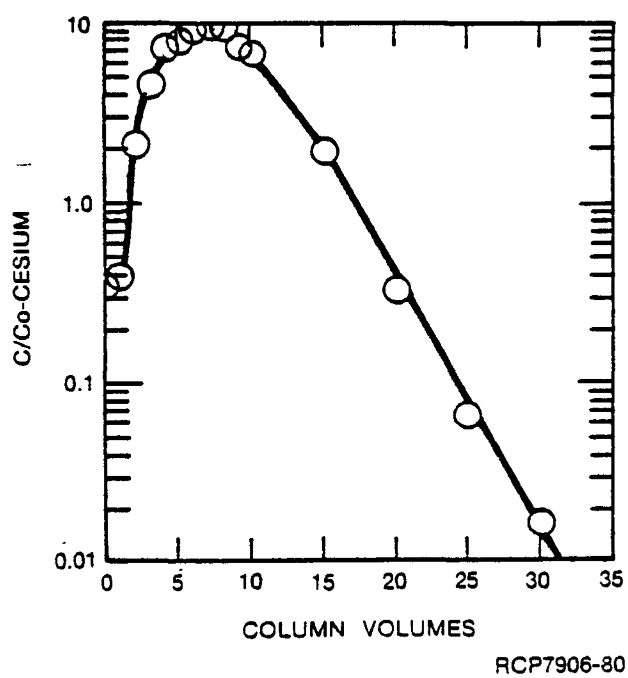
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

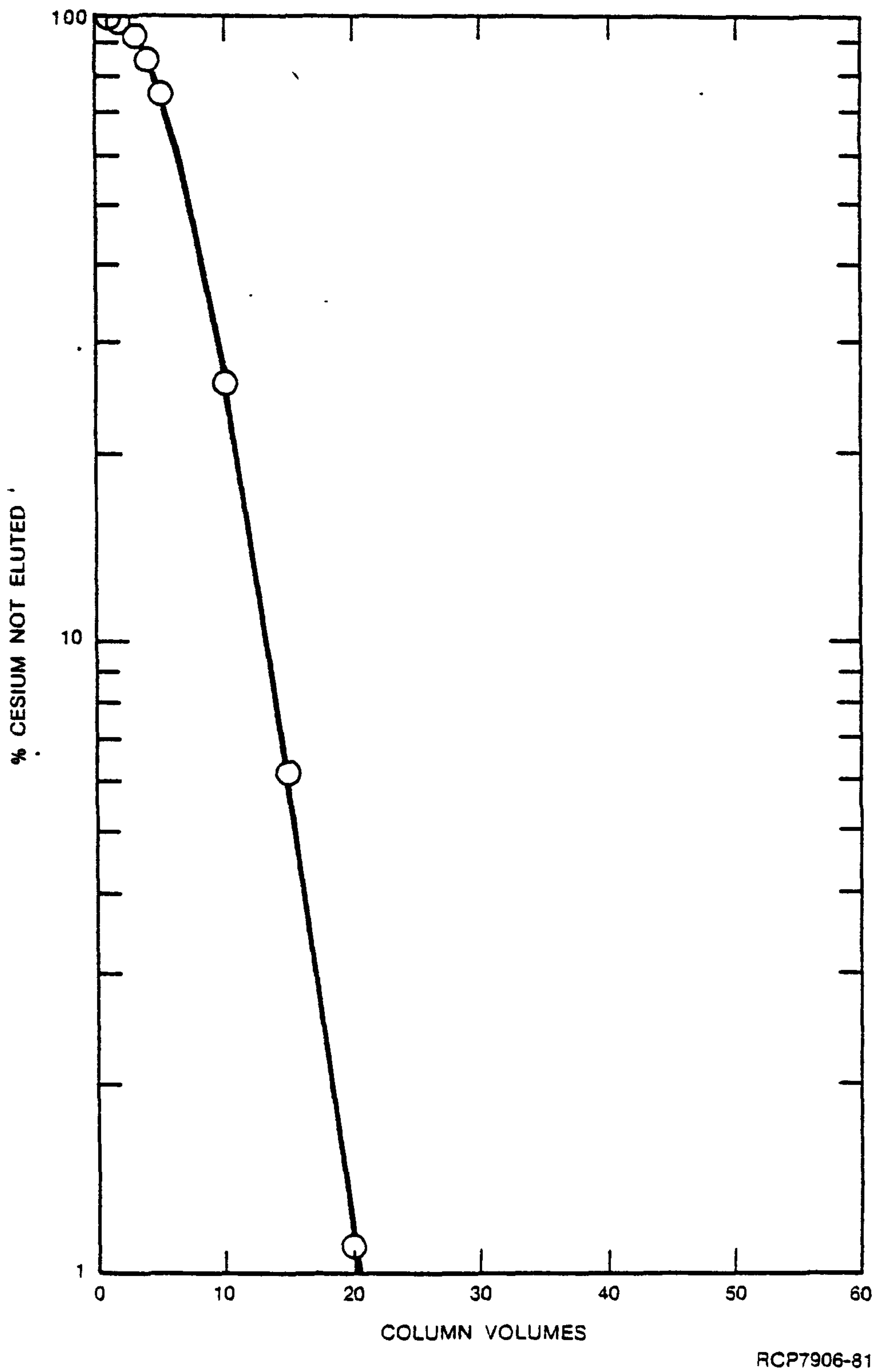
Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.002		E-1	521	0.388
L-20	4	0.003		E-2	2970	2.22
L-30	6	0.004		E-3	6050	4.51
L-40	5	0.004		E-4	9730	7.25
L-50	8	0.006		E-5	10600	7.93
L-60	11	0.008		E-6	12200	9.09
L-70	39	0.029		E-7	12600	9.41
L-80	162	0.120		E-8	12200	9.09
L-90	429	0.320		E-9	9590	7.15
W-1,2	87	0.065		E-10	8820	6.58
S-1	7	0.005	0.045	E-15	2550	1.90
S-2	9	0.007	0.053	E-20	433	0.33
S-3	13	0.009	0.105	E-25	90	0.067
S-4	17	0.013	0.134	E-30	23	0.017
S-5	21	0.015	0.271			
S-6	23	0.017	0.174			
S-7	24	0.018	0.175			
S-8	27	0.020	0.252			
S-9	139	0.104	0.120			
S-10	375	0.279	0.021			
S-11	458	0.341	0.004			



Cesium Breakthrough Curve - Run 9



Cesium Elution Curve - Run 9



Percent Cesium Not Eluted - Run 9

Run 10

Determine sodium, iron, and aluminum decontamination factors and cesium eluting characteristics with AW-500. Column was not loaded to breakthrough.

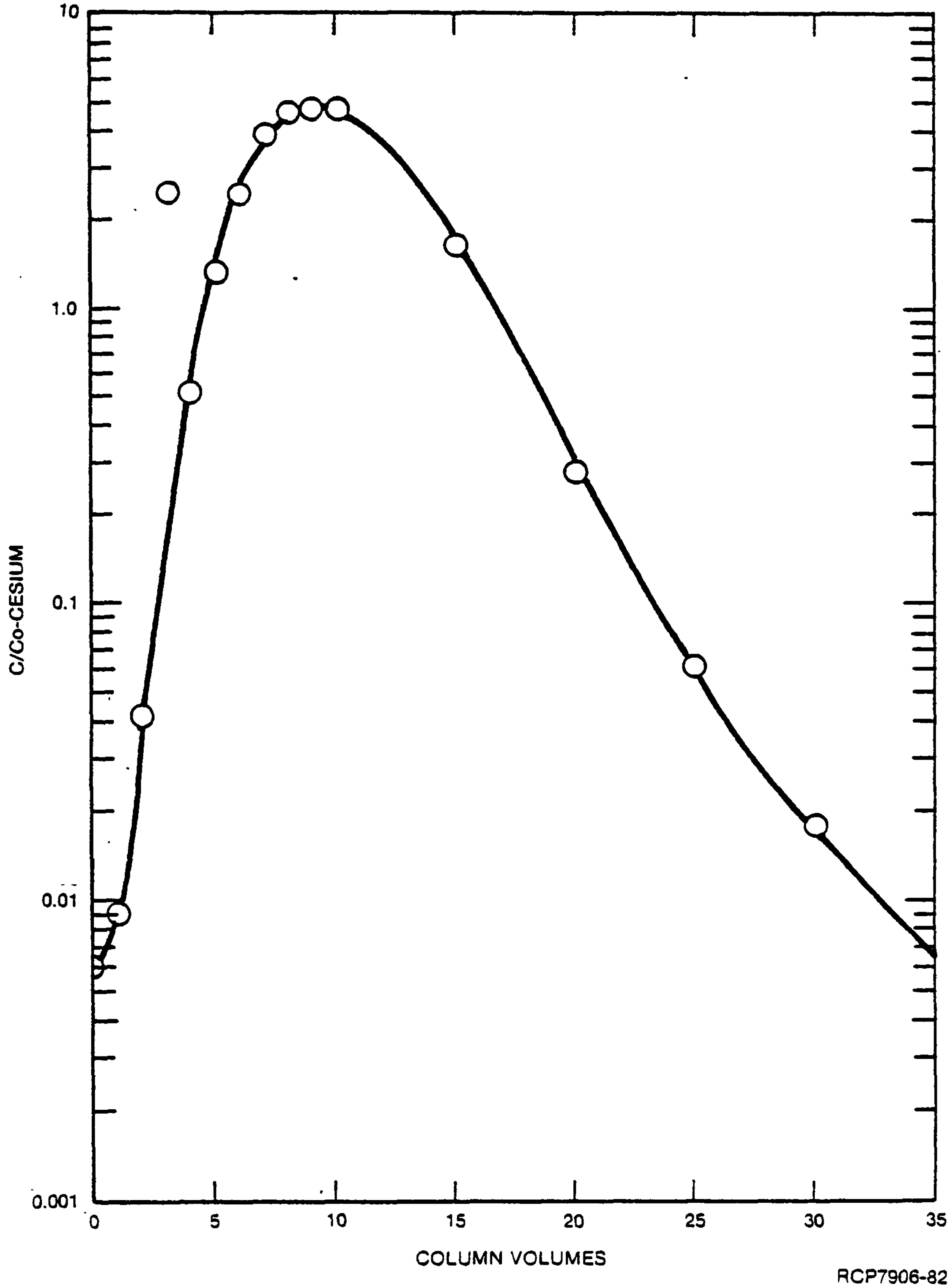
	Feed*	Product
^{137}Cs	1.38×10^3 μCi/ℓ	2.47×10^3 μCi/ℓ
Cs^+	3.8×10^{-3} M	---
Na^+	1.94M	7.87×10^{-4} M
Fe^{3+}	0.115M	<8.40×10^{-4} M
Al^{3+}	0.107M	<2.69×10^{-4} M
pH	4.5	---

Scrub: 0.15M $(NH_4)_2CO_3$

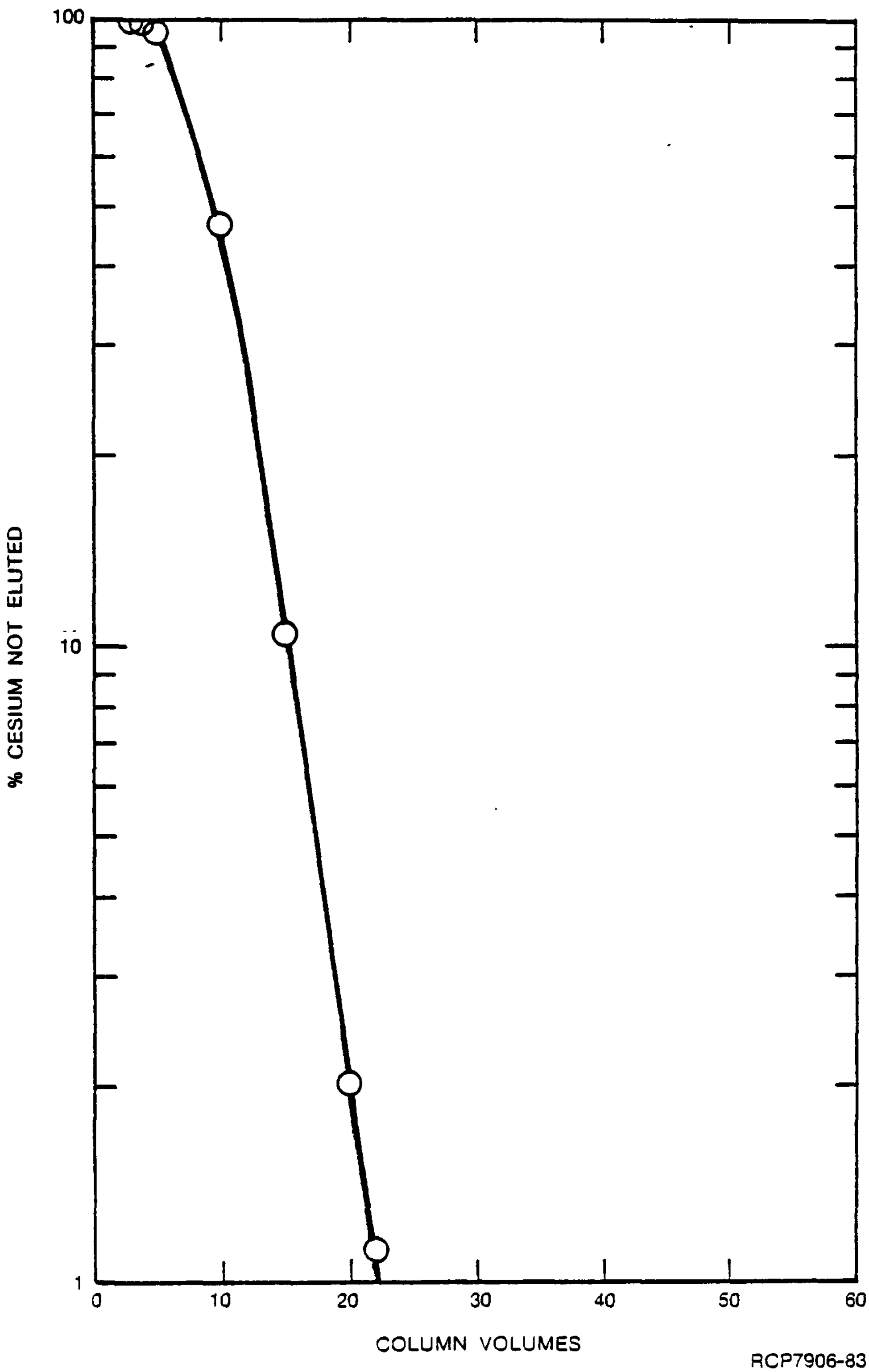
Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

Effluent	^{137}Cs		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	5	0.004		E-1	12	0.009
L-20	5	0.003		E-2	56	0.041
L-30	8	0.005		E-4	707	0.511
L-40	6	0.005		E-5	1830	1.32
W-1,2	9	0.007		E-6	3400	2.45
S-1	9	0.007	1.96	E-8	6380	4.61
S-2	2	0.001	0.089	E-10	6620	4.78
S-3	2	0.001	0.108	E-15	2260	1.63
S-4	5	0.003	0.096	E-20	390	0.28
S-5	4	0.003	0.150	E-25	85	0.061
S-6	ND		0.131	E-30	25	0.018
S-7	1	0.001	0.184			
S-8	1	0.001	0.175			
S-9	4	0.003				

*Only 40 column volumes of feed pumped through column.



Cesium Elution Curve - Run 10



Percent Cesium Not Eluted - Run 10

Run 11

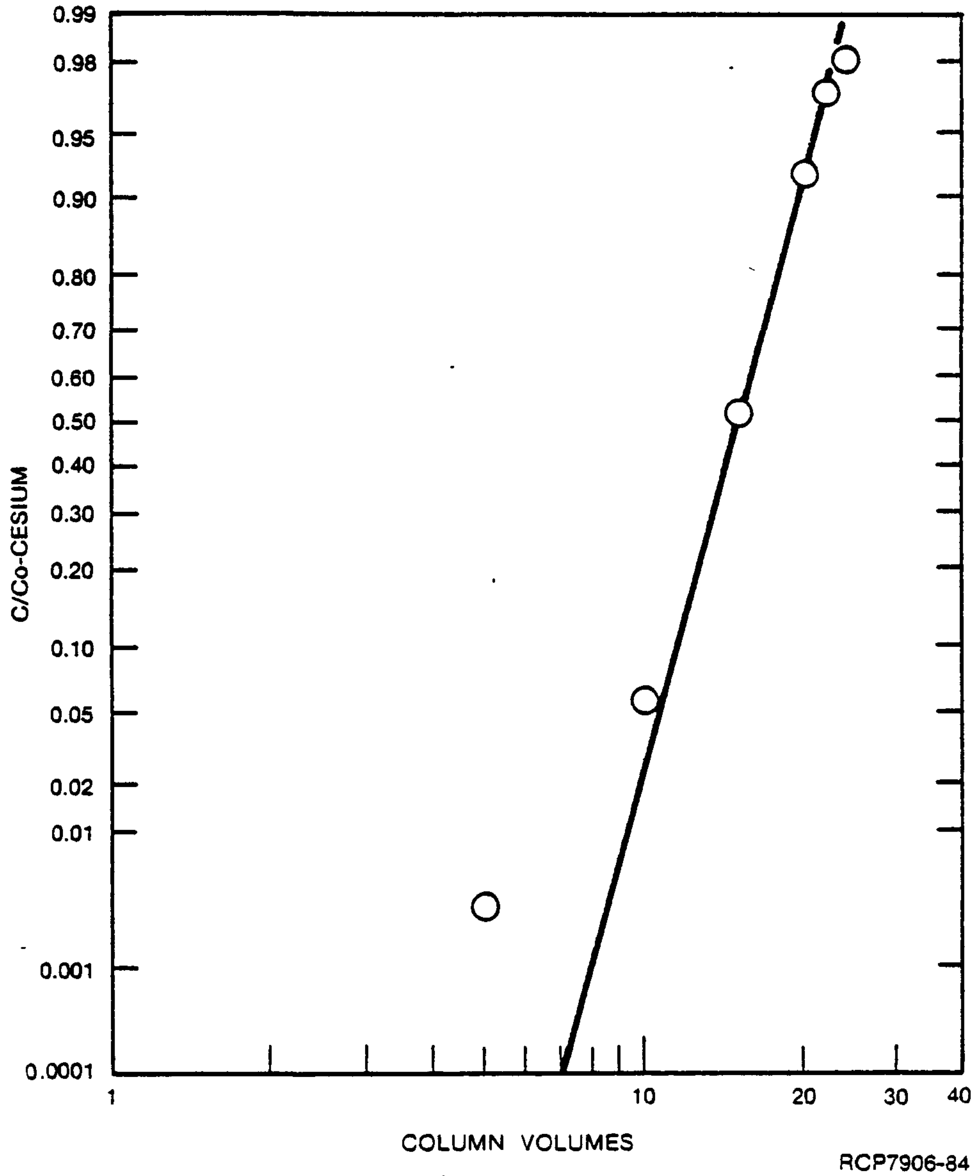
Determine cesium loading, scrubbing, and eluting characteristics with Duolite ARC-359 and feed solution at pH 10.

	Feed	Product
^{137}Cs	907 µCi/ℓ	1.10×10^3 µCi/ℓ
^{106}Rh	197 µCi/ℓ	---
^{144}Ce	72 µCi/ℓ	---
Cs^+	4×10^{-4}M	---
Na^+	2.59M	0.03M
Fe^{3+}	0.103M	4.44×10^{-3}M
Al^{3+}	0.100M	$<4.73 \times 10^{-4}$M
pH	10	---

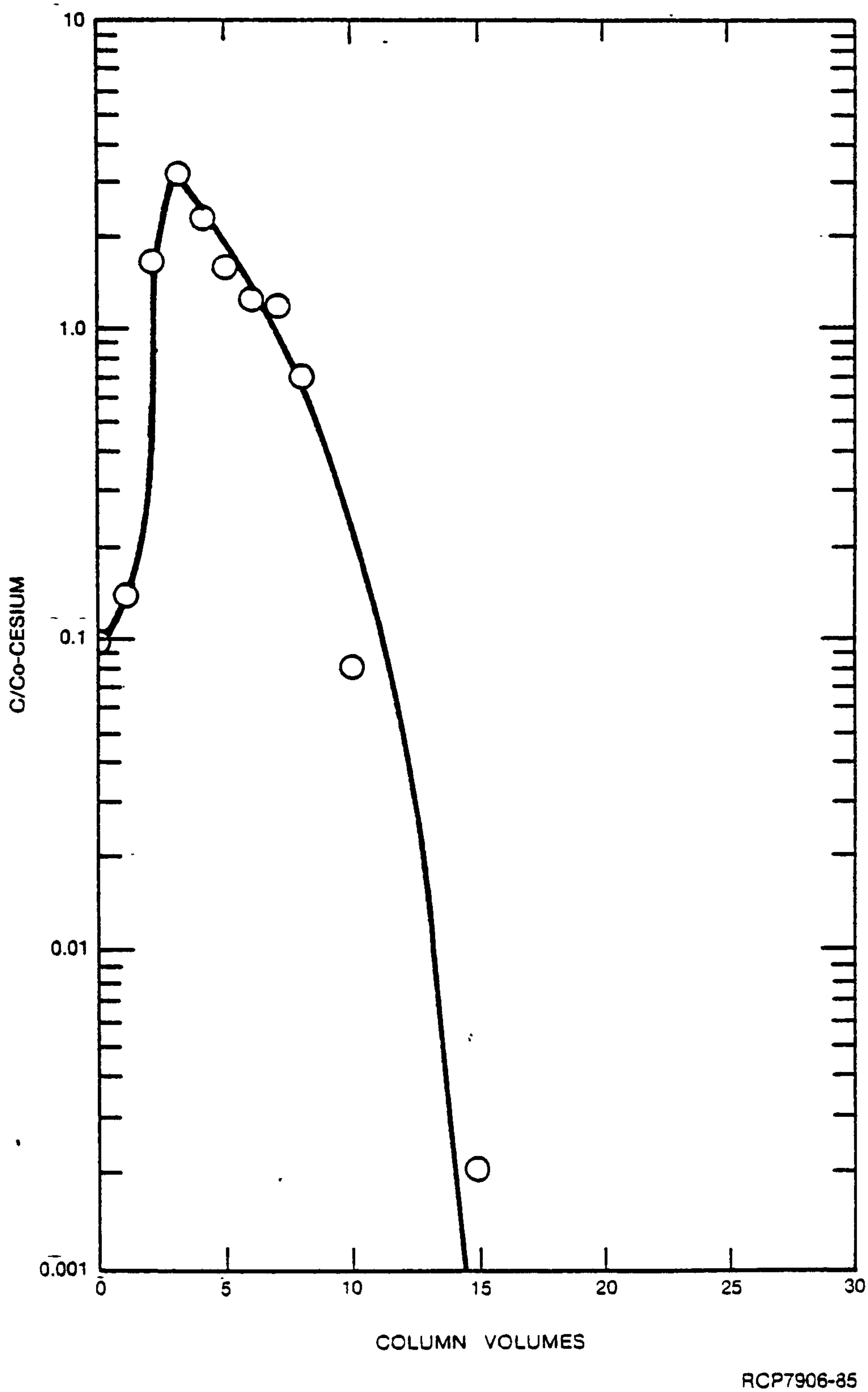
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

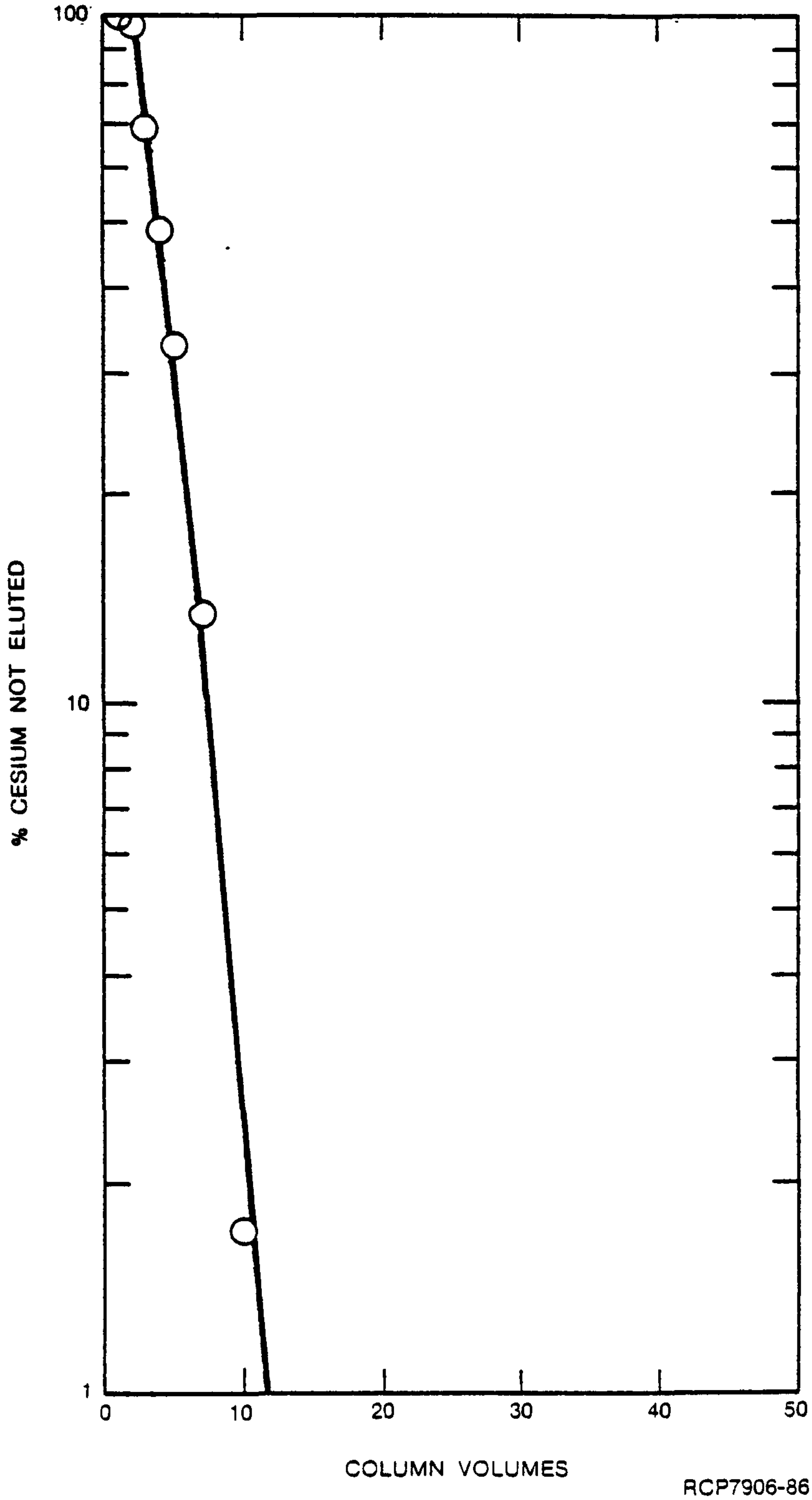
Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	µCi/ℓ	C/Co	µCi/ℓ	C/Co	µCi/ℓ	C/Co			µCi/ℓ	C/Co
L-5	3	0.003	181	0.919	69	0.96		E-1	123	0.136
L-10	50	0.055	200	1.02	77	1.07		E-2	1480	1.63
L-15	467	0.52	197	1.00	74	1.02		E-3	2820	3.11
L-20	836	0.92	209	1.06	79	1.09		E-4	2060	2.27
L-22	880	0.97	194	0.98	76	1.05		E-5	1380	1.53
L-24	885	0.98	199	1.01	75	1.04		E-6	1110	1.22
L-25	943	1.04	205	1.04	78	1.08		E-7	1050	1.16
W-1,2	962	1.06	207	1.05	79	1.09		E-8	614	0.68
S-1	714	0.79						E-10	72	0.08
S-2	36	0.040					0.120	E-15	2	0.002
S-3	41	0.045					0.120			
S-4	48	0.053					0.146			
S-5	53	0.058					0.151			
S-6	57	0.063					0.156			
S-7	58	0.064					0.168			
S-8	61	0.067					0.182			
S-9	69	0.076					0.172			
S-10	88	0.097					0.168			



Cesium Breakthrough Curve - Run 11



Cesium Elution Curve - Run 11



Percent Cesium Not Eluted - Run 11

Run 12

Determine cesium loading, scrubbing, and eluting characteristics with Duolite ARC-359 and feed solution at pH 11.

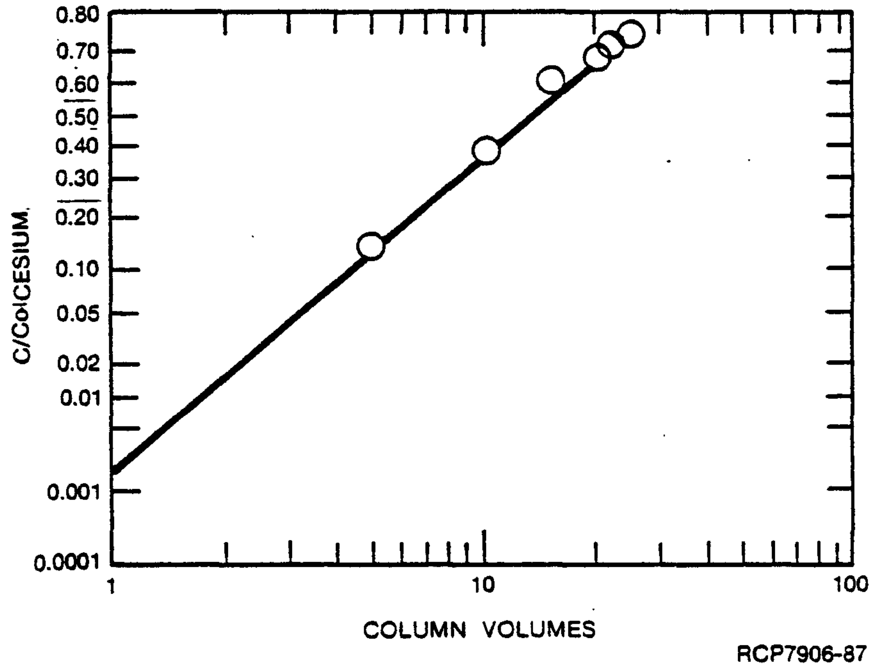
	Feed	Product
^{137}Cs	1290 μCi/ℓ	1270 μCi/ℓ
^{106}Rh	261 μCi/ℓ	---
^{144}Ce	99 μCi/ℓ	---
Cs^{+}	4×10^{-4} M	---
Na^{+}	2.01M	0.07M
Fe^{3+}	0.108M	6.47×10^{-4} M
Al^{3+}	0.078M	6.98×10^{-5} M
pH	11	

Scrub: 0.15M $(NH_4)_2CO_3$

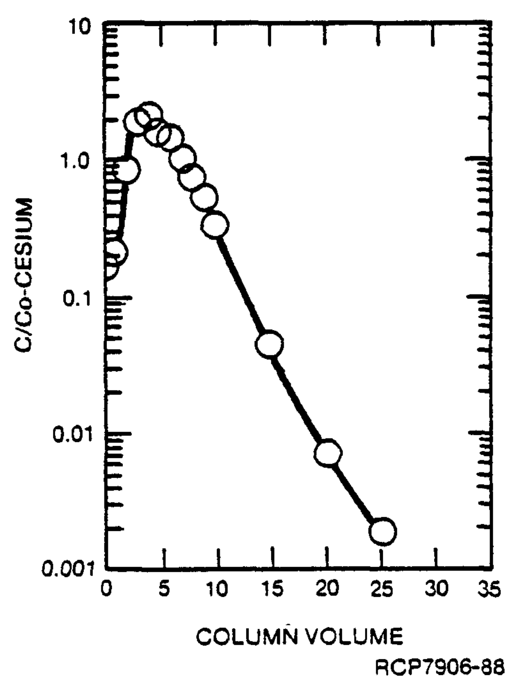
Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-5	170	0.132	251	0.96	84	0.85		E-1	267	0.21
L-10	481	0.374	266	1.02	100	1.01		E-2	1110	0.87
L-15	776	0.604	283	1.08	101	1.02		E-3	2490	1.90
L-20	871	0.677	265	1.02	97	0.98		E-4	2800	2.18
L-22	907	0.706	262	1.00	96	0.97		E-5	2100	1.64
L-25	948	0.737	281	1.08	103	1.04		E-6	1930	1.50
W-1,2	411	0.320	238	0.91	79	0.80		E-7	1380	1.08
S-1	64	0.049	52	0.20			0.53	E-8	973	0.76
S-2	43	0.033	29	0.11			0.40	E-10	436	0.34
S-3	38	0.029	16	0.06			0.36	E-15	58	0.05
S-4	45	0.035					0.43	E-20	9	0.007
S-5	65	0.051					0.32			
S-6	92	0.072					0.31			
S-7	117	0.091					0.27			
S-8	139	0.108					0.19			
S-9	168	0.131					0.21			
S-10	185	0.144					0.21			
S-11	216	0.168					0.15			

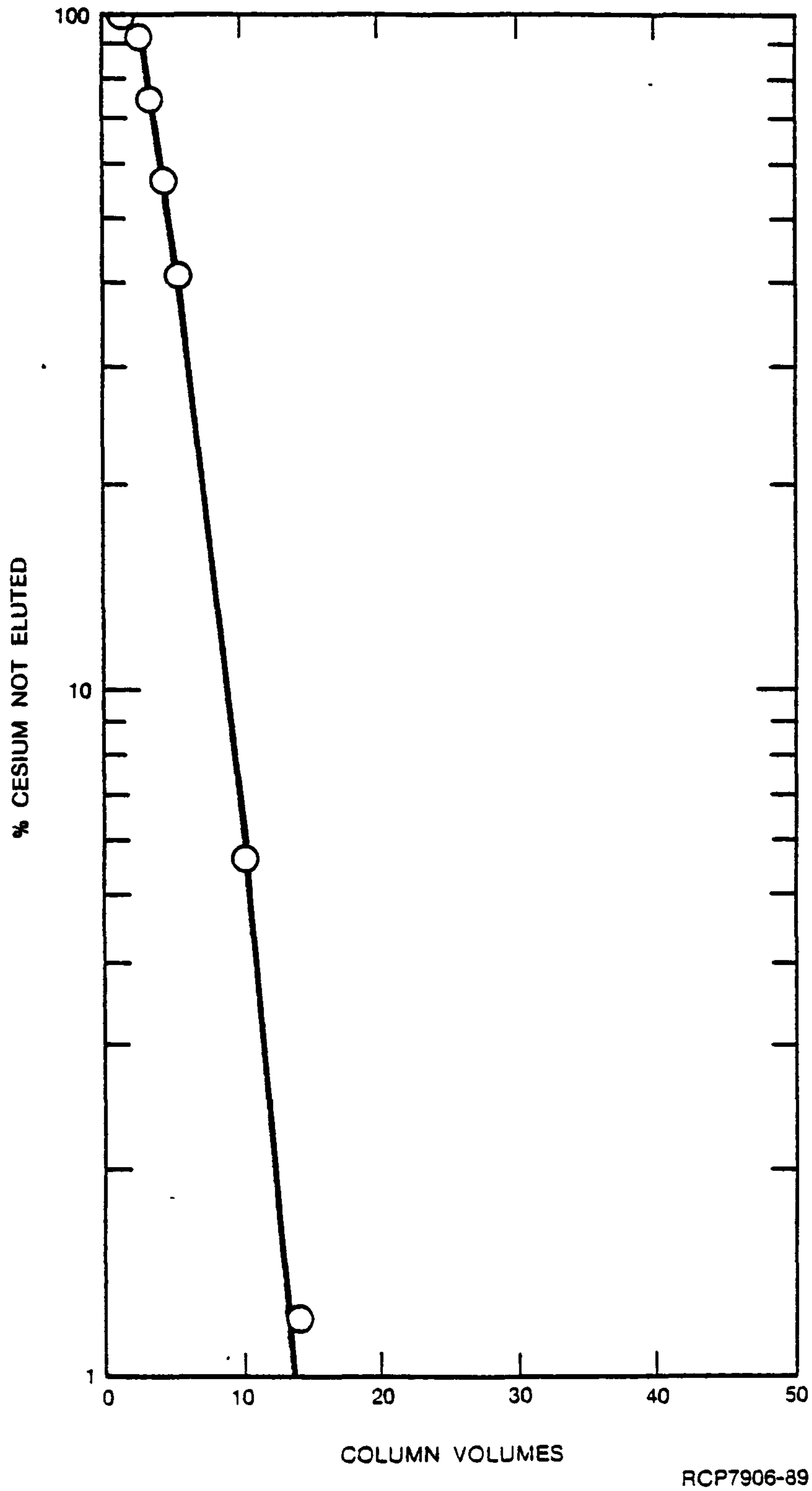
*Brown precipitate formed at H^{+}11.



Cesium Breakthrough Curve - Run 12



Cesium Elution Curve - Run 12



Percent Cesium Not Eluted - Run 12

Run 13

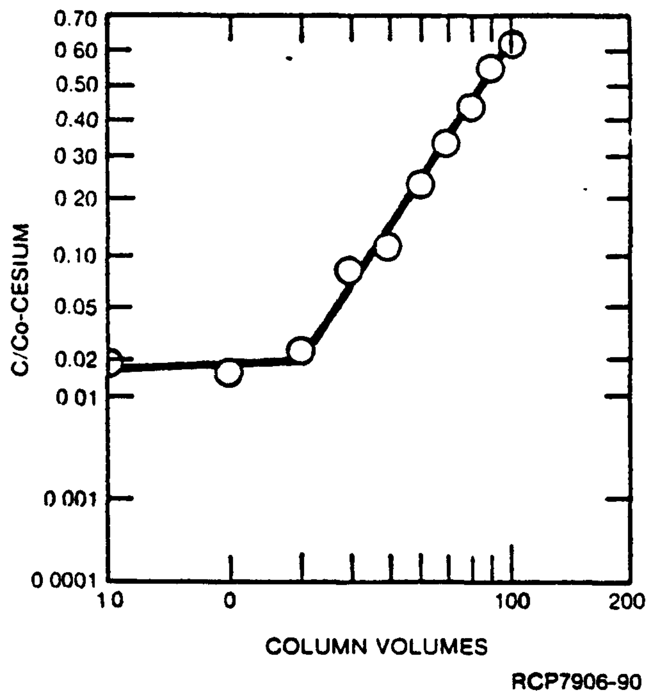
Determine strontium decontamination while recovering cesium on an AW-500 column.

	Feed	Product
^{137}Cs	1190 µCi/ℓ	3260 µCi/ℓ
^{85}Sr	4420 µCi/ℓ	21 µCi/ℓ
^{106}Rh	425 µCi/ℓ	---
Cs^+	5.24×10^{-4} M	---
Na^+	1.91M	1.48×10^{-3} M
Fe^{3+}	0.088M	1.37×10^{-5} M
Al^{3+}	0.104M	2.17×10^{-4} M
$Total_{Sr}$	9.20×10^{-5} M	---
pH	4.5	---

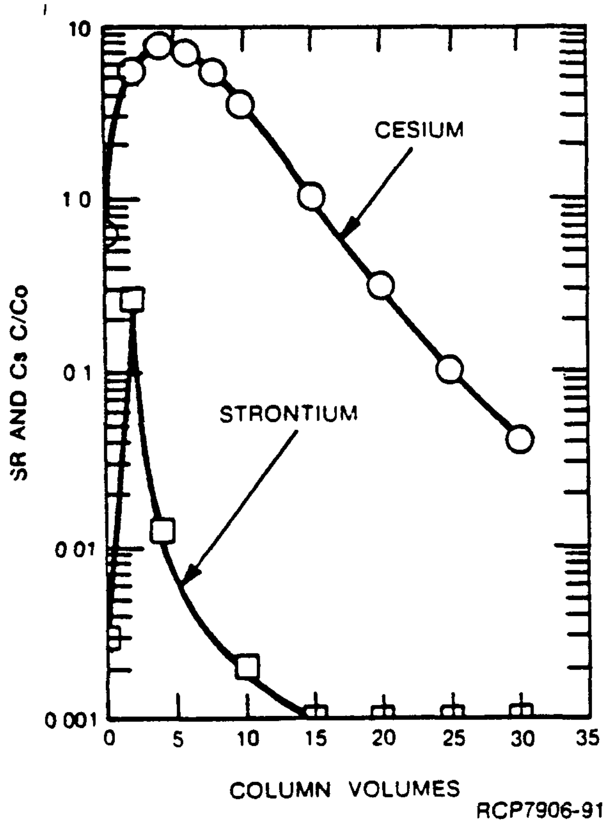
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{85}Sr		^{106}Rh		Na, M	Effluent	^{137}Cs		^{85}Sr	
	µCi/ℓ	C/Co	µCi/ℓ	C/Co	µCi/ℓ	C/Co			µCi/ℓ	C/Co	µCi/ℓ	C/Co
L-10	22	0.019	4350	0.98	413	0.97		E-2	6280	5.29	1110	0.25
L-20	19	0.016	4380	0.99	409	0.96		E-4	8710	7.33	54	0.012
L-30	28	0.023	4300	0.97	400	0.94		E-6	8130	6.84		
L-40	97	0.082	4380	0.99	408	0.96		E-8	6330	5.33		
L-50	136	0.11	4410	1.00	418	0.98		E-10	4040	3.40	11	0.002
L-60	258	0.22	4380	0.99	412	0.97		E-15	1190	1.01	6	0.001
L-70	396	0.33	4460	1.01	424	1.00		E-20	373	0.31	4	0.001
L-80	518	0.44	4360	0.99	410	0.97		E-25	121	0.10	3	0.001
L-90	646	0.54	4310	0.98	405	0.95		E-30	48	0.04	4	0.001
L-100	733	0.62	4330	0.98	412	0.97						
W-1,2	701	0.59	4050	0.92	388	0.91						
S-1	59	0.049	87	0.020	34	0.08	0.141					
S-2	109	0.092	10	0.002			0.076					
S-3	95	0.080	12	0.003			0.159					
S-4	58	0.049	15	0.003			0.214					
S-5	68	0.057	17	0.004			0.223					
S-6	201	0.17	22	0.005			0.197					
S-7	536	0.45	25	0.006			0.077					
S-8	694	0.58	19	0.004			0.018					
S-9	761	0.64	16	0.004			0.007					
S-10	740	0.62	14	0.003			0.004					



Cesium Breakthrough Curve - Run 13



Cesium Elution Curve - Run 13

Run 14

Determine cesium, ruthenium, and cerium loading, scrubbing, and eluting characteristics with AW-500 and feed saturated with 1AX.

	Feed*	Product
^{137}Cs	1220 µCi/ℓ	4160 µCi/ℓ
^{106}Rh	241 µCi/ℓ	---
^{144}Ce	80 µCi/ℓ	---
Cs^+	4×10^{-4}M	---
Na^+	1.98M	1.02×10^{-3}M
Fe^{3+}	0.098M	2.72×10^{-5}M
Al^{3+}	0.110M	<8.80×10^{-5}M
pH	4.5	---
TOC**	---	1.33M

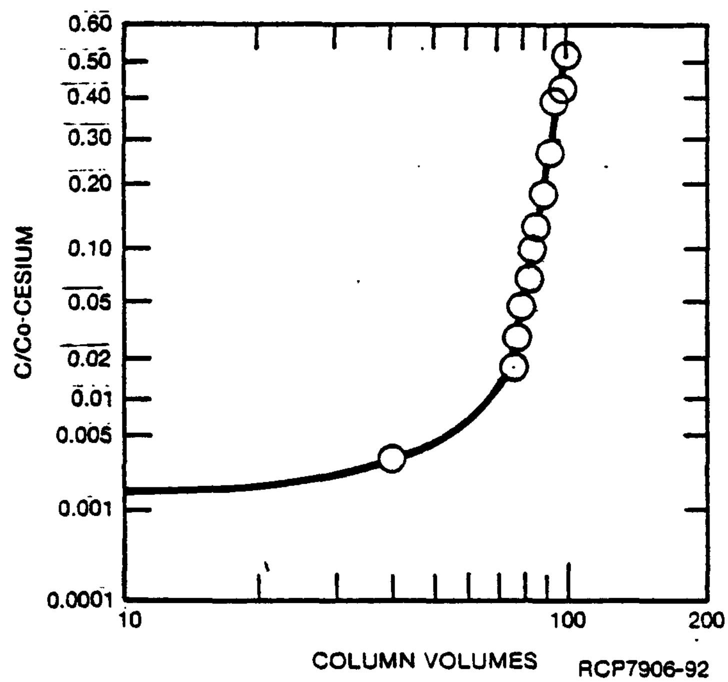
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

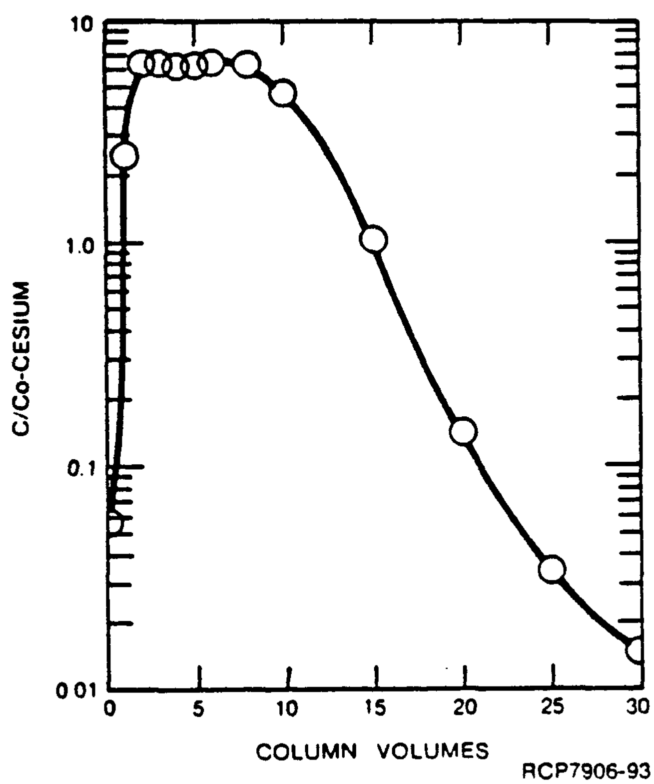
Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	µCi/ℓ	C/Co	µCi/ℓ	C/Co	µCi/ℓ	C/Co			µCi/ℓ	C/Co
L-10			268	1.11	91	1.14		E-1	2930	2.41
L-20			252	1.05	86	1.08		E-2	7770	6.37
L-30	11	0.009	266	1.10	85	1.06		E-3	7740	6.35
L-40	3	0.003	264	1.10	83	1.04		E-4	7550	6.14
L-75	35	0.029	275	1.14	86	1.08		E-5	7480	6.13
L-81	84	0.069	274	1.14	90	1.13		E-6	7770	6.38
L-85	152	0.128	255	1.06	79	0.99		E-8	7590	6.22
L-91	324	0.266	248	1.03	78	0.98		E-10	5610	4.60
L-96	519	0.426	257	1.07	75	0.94		E-15	1230	1.01
L-99	631	0.518	248	1.03	76	0.95		E-20	171	0.14
W-1,2	75	0.062	33	0.14	59	0.74		E-25	41	0.034
S-1	41	0.033					0.023	E-30	19	0.015
S-2	793	0.651					0.085			
S-3	98	0.081					0.062			
S-4	109	0.090					0.120			
S-5	59	0.049					0.216			
S-6	63	0.051					0.226			
S-7	394	0.323					0.097			
S-8	705	0.578					0.008			
S-9	739	0.606					<0.009			
S-10	690	0.566					<0.009			

*Saturated with B Plant Organic

**Total Organic Carbon



Cesium Breakthrough Curve - Run 14



Cesium Elution Curve - Run 14

Run 15

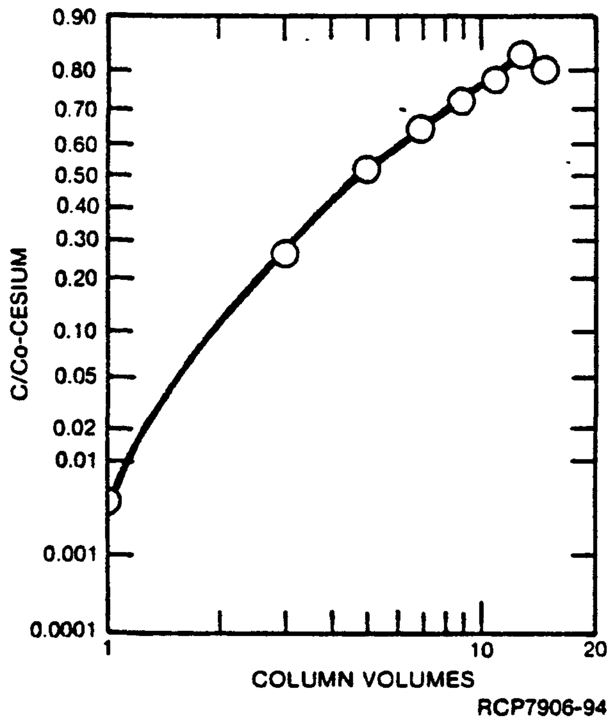
Determine cesium, ruthenium, and cerium loading, scrubbing, and eluting characteristic of Duolite ARC-359 with feed at pH 4.5.

	Feed	Product
^{137}Cs	1055 µCi/ℓ	340 µCi/ℓ
^{106}Rh	186 µCi/ℓ	---
^{144}Ce	71 µCi/ℓ	---
Cs^{+}	4×10^{-4} M	---
Na^{+}	1.98M	0.037M
Fe^{3+}	0.098M	4.88×10^{-5} M
Al^{3+}	0.110M	$<2.71 \times 10^{-4}$ M
pH	4.5	

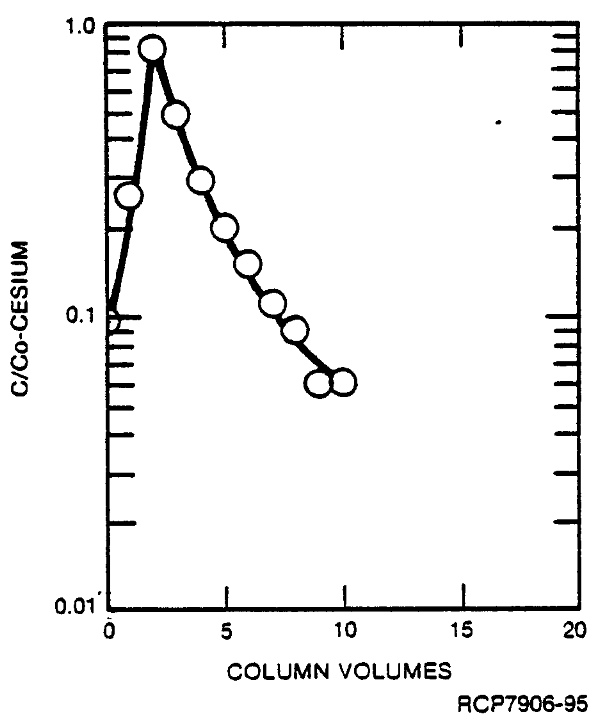
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	µCi/ℓ	C/Co	µCi/ℓ	C/Co	µCi/ℓ	C/Co			µCi/ℓ	C/Co
L-1	4	0.004	19	0.10	10	0.14		E-1	269	0.26
L-3	261	0.247	149	0.80	61	0.86		E-2	854	0.81
L-5	539	0.511	174	0.94	68	0.95		E-3	502	0.48
L-7	675	0.640	180	0.97	68	0.95		E-4	302	0.29
L-9	760	0.720	189	1.02	68	0.95		E-5	212	0.20
L-11	809	0.767	189	1.02	71	1.00		E-6	160	0.15
L-13	876	0.830	200	1.08	74	1.04		E-7	119	0.11
L-15	842	0.798	190	1.02	71	1.00		E-8	96	0.09
W-1,2	192	0.182						E-9	68	0.06
S-1	24	0.023					0.070	E-10	60	0.06
S-2	30	0.028					0.094			
S-3	36	0.034					0.117			
S-4	40	0.038					0.129			
S-5	46	0.043					0.167			
S-6	63	0.060					0.128			
S-7	78	0.074					0.094			
S-8	93	0.088					0.070			
S-9	100	0.095					0.046			
S-10	102	0.097					0.040			



Cesium Breakthrough Curve - Run 15



Cesium Elution Curve - Run 15

Run 16

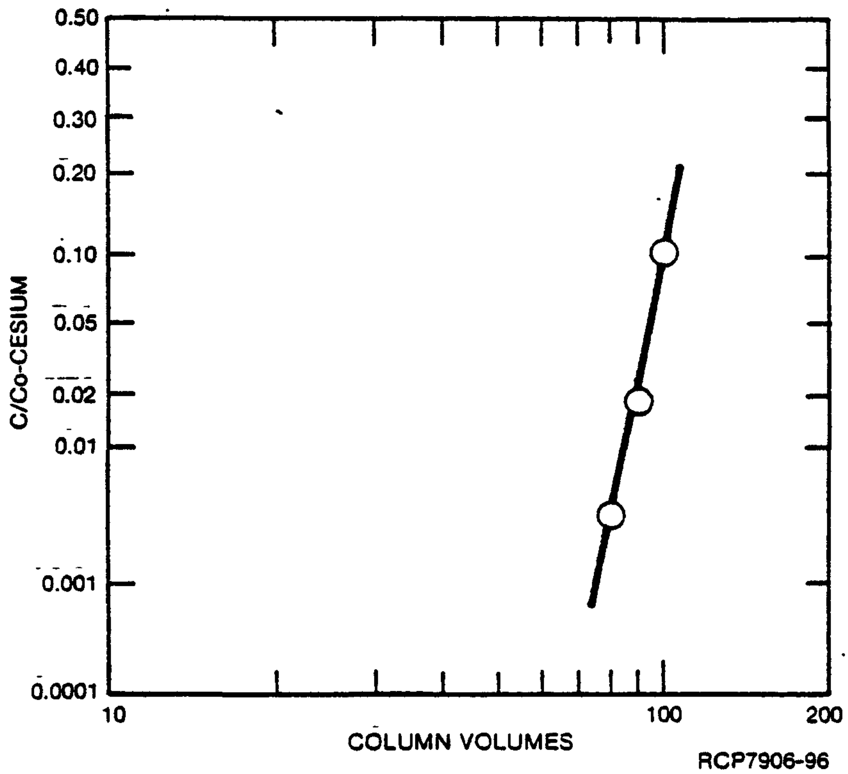
Measure the stability of AW-500 when repetitively loaded, scrubbed, and eluted; first cycle.

	Feed	Product
^{137}Cs	1.14×10^3 μCi/ℓ	4.07×10^3 μCi/ℓ
^{106}Rh	201 μCi/ℓ	---
^{144}Ce	72 μCi/ℓ	---
Cs^+	9.37×10^{-4}M	---
Na^+	2.18M	2.07×10^{-3}M
Fe^{3+}	0.102M	5.54×10^{-5}M
Al^{3+}	0.096M	$<2.78 \times 10^{-4}$M
pH	4.5	---

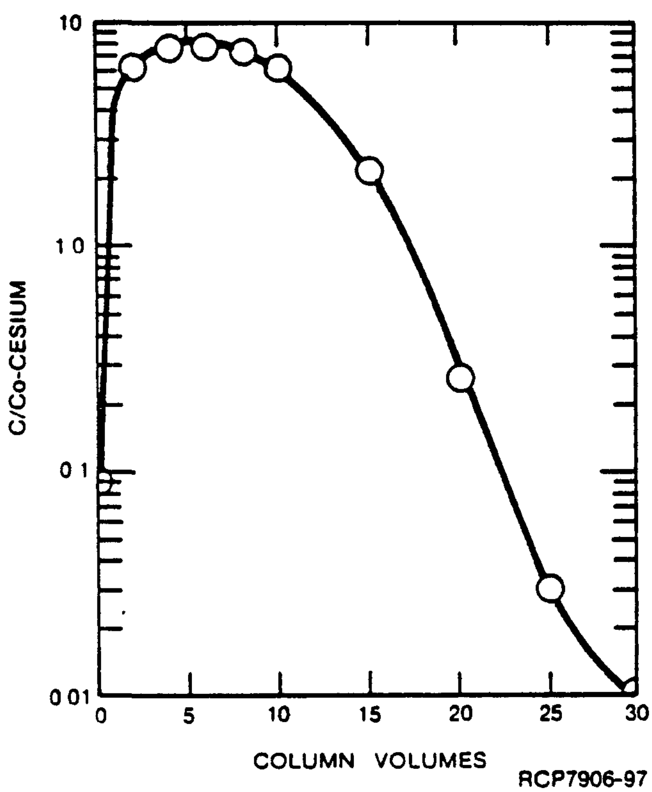
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10			205	1.02	74	1.02		E-2	7030	6.16
L-20			206	1.02	74	1.02		E-4	8620	7.56
L-30			199	0.99	71	0.99		E-6	8680	7.61
L-40			201	1.00	69	0.96		E-8	8160	7.15
L-50			200	1.00	72	1.00		E-10	7030	6.17
L-60			200	1.00	72	1.00		E-15	2450	2.15
L-70			203	1.01	71	0.98		E-20	293	0.26
L-80	4	0.003	200	1.00	69	0.95		E-25	36	0.03
L-90	21	0.018	195	0.97	71	0.98		E-30	13	0.01
L-100	119	0.104	208	1.03	75	1.04				
W-1,2	101	0.089	159	0.79	70	0.97				
S-1-10	106	0.093					0.128			



Cesium Breakthrough Curve - Run 16



Cesium Elution Curve - Run 16

Run 17

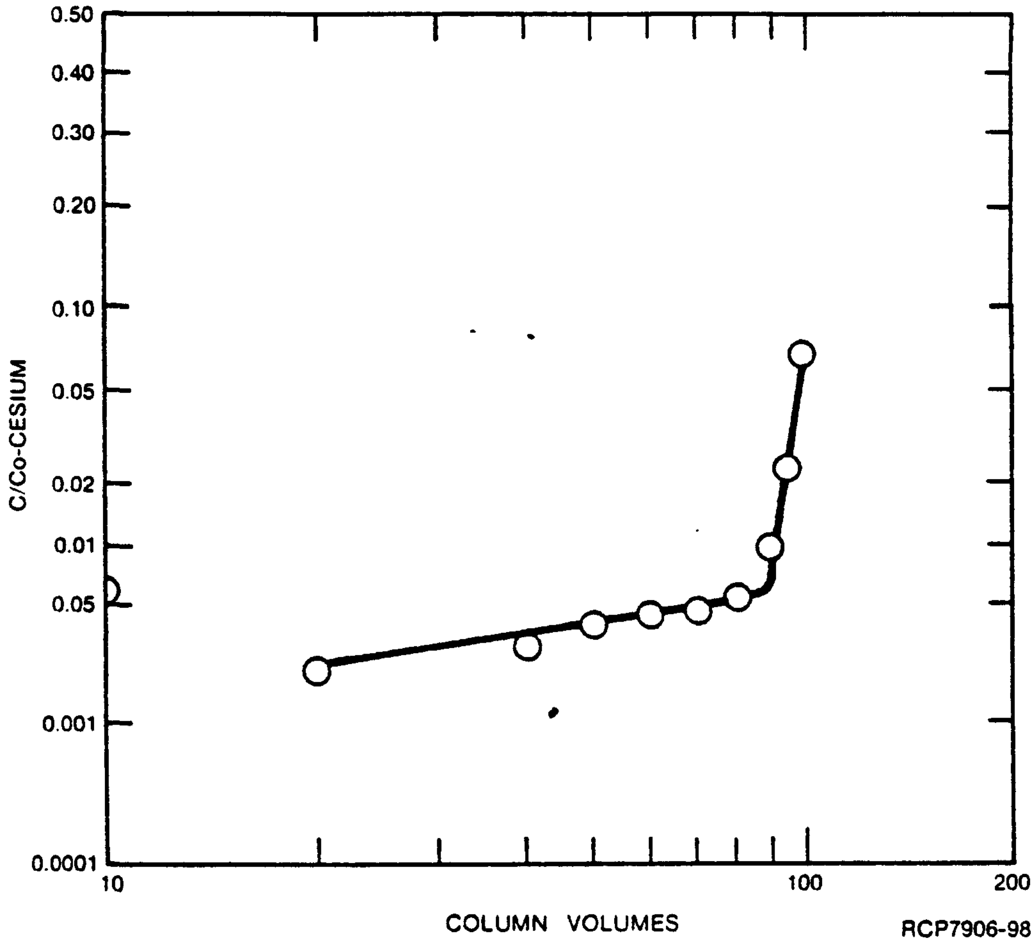
Determine stability of AW-500 when repetitively loaded, scrubbed, and eluted; second cycle.

	Feed	Product
^{137}Cs	1150 μCi/ℓ	4510 μCi/ℓ
^{106}Rh	202 μCi/ℓ	---
^{144}Ce	72 μCi/ℓ	---
Cs^{+}	9.37×10^{-4} M	---
Na^{+}	2.01M	1.19×10^{-3} M
Fe^{3+}	0.096M	1.14×10^{-5} M
Al^{3+}	0.093M	$<2.84 \times 10^{-4}$ M
pH	4.5	---

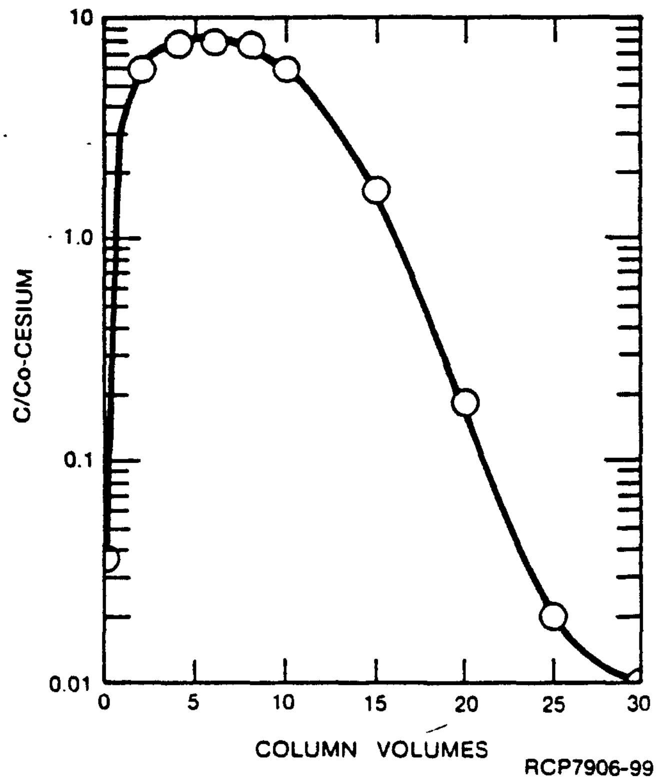
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH, downflow

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	6	0.006	204	1.01	75	1.04		E-2	6740	5.87
L-20	2	0.002	203	1.00	70	0.97		E-4	8750	7.62
L-30			228	1.13	77	1.07		E-6	8920	7.77
L-40	3	0.003	198	0.98	72	1.00		E-8	8610	7.50
L-50	4	0.004	222	1.10	76	1.06		E-10	6860	5.98
L-60	5	0.004	262	1.30	71	0.99		E-15	1880	1.64
L-70	5	0.004	203	1.00	72	1.00		E-20	204	0.18
L-80	6	0.005	205	1.01	72	1.00		E-25	27	0.02
L-90	11	0.010	203	1.00	71	0.99		E-30	15	0.01
L-100	79	0.069	209	1.03	75	1.04				
W-1,2	46	0.040	118	0.58	39	0.54				
S-1-10	40	0.035					0.162			



Cesium Breakthrough Curve - Run 17



Cesium Elution Curve - Run 17

Run 18

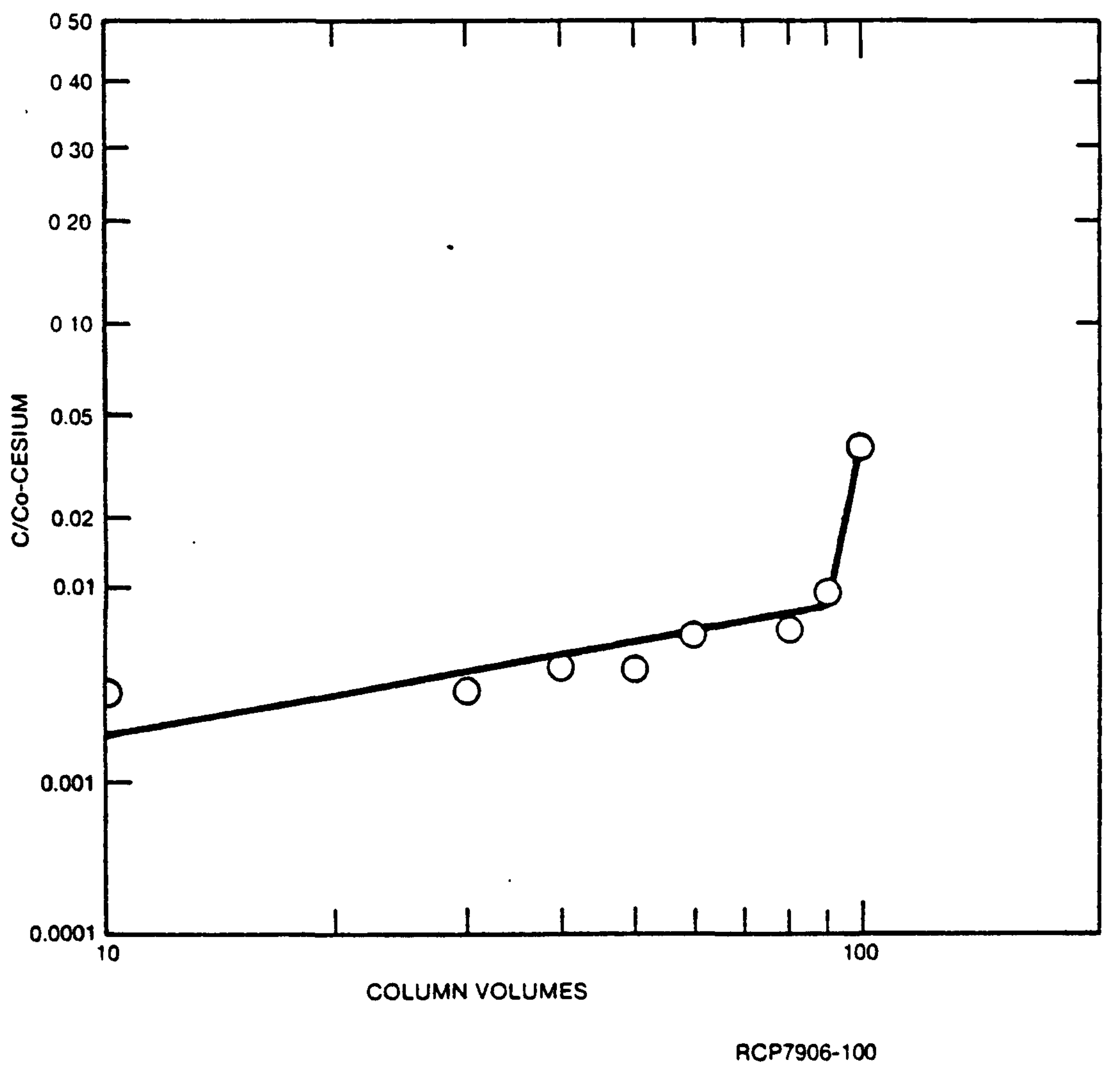
Measure the stability of AW-500 when repetitively loaded, scrubbed, and eluted; third cycle.

	Feed	Product
^{137}Cs	913 μCi/ℓ	3770 μCi/ℓ
^{106}Rh	156 μCi/ℓ	---
^{144}Ce	54 μCi/ℓ	---
Cs^{+}	9.37×10^{-4} M	---
Na^{+}	2.27M	8.73×10^{-4} M
Fe^{3+}	0.099M	<1.50×10^{-5} M
Al^{3+}	0.094M	<2.44×10^{-4} M
pH	4.5	---

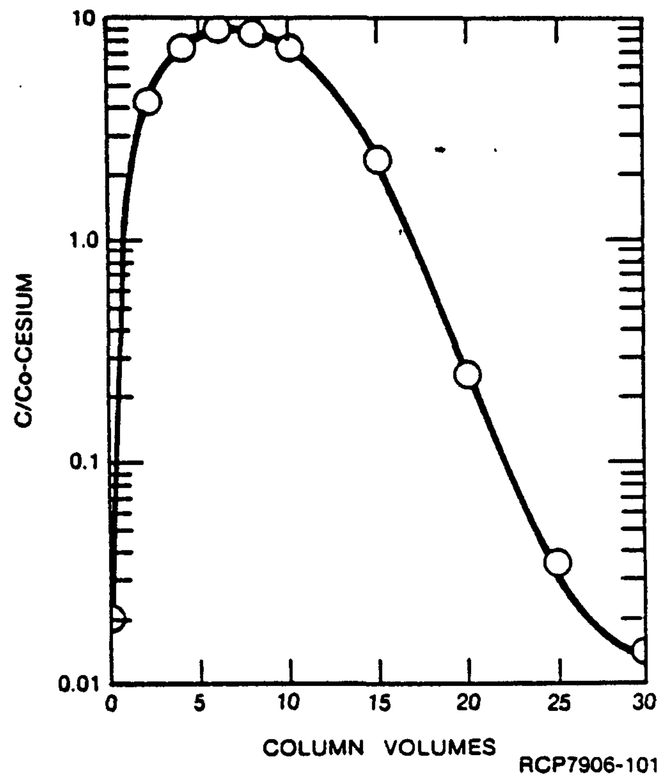
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.003	155	0.99	56	1.04		E-2	3840	4.20
L-20			151	0.97	55	1.02		E-4	6780	7.42
L-30	3	0.003	168	1.08	60	1.11		E-6	8020	8.78
L-40	4	0.004	158	1.01	58	1.07		E-8	7720	8.45
L-50	4	0.004	158	1.01	56	1.04		E-10	6590	7.21
L-60	5	0.006	156	1.00	56	1.04		E-15	2090	2.28
L-70	5	0.006	161	1.03	56	1.04		E-20	256	0.25
L-80	6	0.006	160	1.03	58	1.07		E-25	32	0.04
L-90	9	0.009	165	1.06	56	1.04		E-30	12	0.01
L-100	36	0.039	163	1.04	59	1.09				
W-1,2	28	0.031	121	0.78	43	0.80				
S-1-10	18	0.020					0.240			



Cesium Breakthrough Curve - Run 18



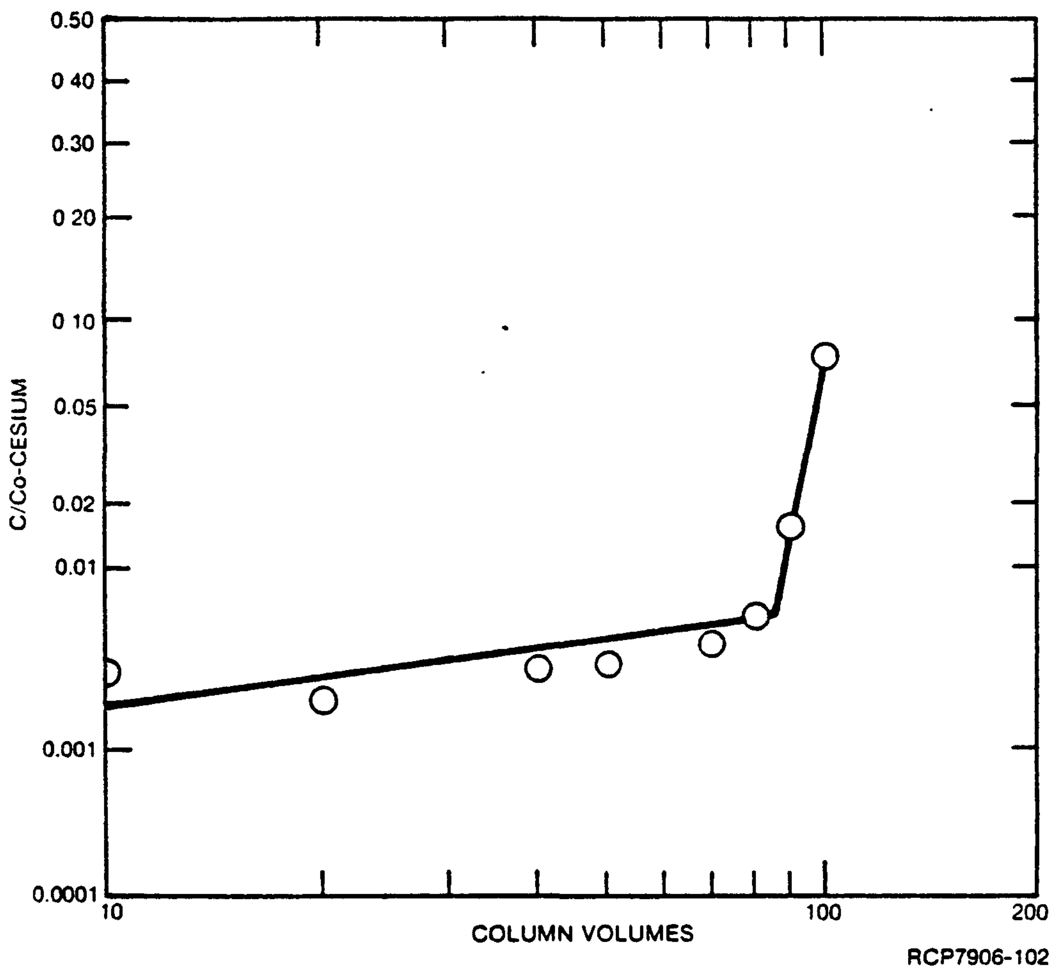
Cesium Elution Curve - Run 18

Run 19

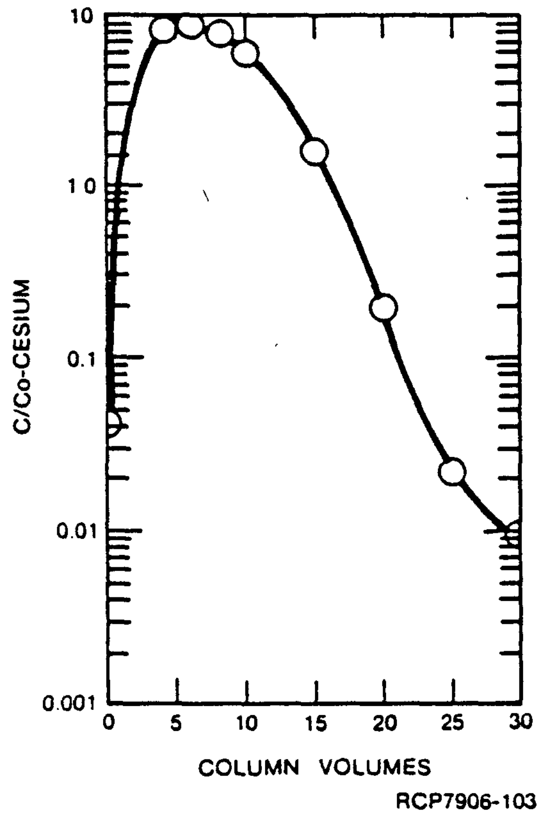
Measure the stability of AW-500 when repetitively loaded, scrubbed, and eluted; fourth cycle.

	Feed	Product
^{137}Cs	1170 μCi/ℓ	5120 μCi/ℓ
^{106}Rh	198 μCi/ℓ	---
^{144}Ce	71 μCi/ℓ	---
Cs^+	1.07×10^{-3}M	---
Na^+	1.80M	9.78×10^{-4}M
Fe^{3+}	0.120M	6.62×10^{-6}M
Al^{3+}	0.113M	<2.47×10^{-4}M
pH	4.5	---

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.002	199	1.01	71	1.00		E-2	47	0.04
L-20	2	0.002	195	0.98	69	0.97		E-4	9560	8.15
L-30			197	0.99	69	0.97		E-6	10100	8.63
L-40	3	0.002	193	0.97	67	0.94		E-8	9130	7.79
L-50	4	0.003	193	0.97	67	0.94		E-10	6940	5.92
L-60			191	0.96	68	0.96		E-15	1810	1.54
L-70	5	0.004	192	0.97	68	0.96		E-20	221	0.19
L-80	6	0.006	196	0.99	70	0.99		E-25	26	0.02
L-90	18	0.015	194	0.98	68	0.96		E-30	11	0.01
L-100	87	0.074	206	1.04	71	1.00				
W-1,2	77	0.066	164	0.83	58	0.82				
S-1-10	48	0.041					0.134			



Cesium Breakthrough Curve - Run 19



Cesium Elution Curve - Run 19

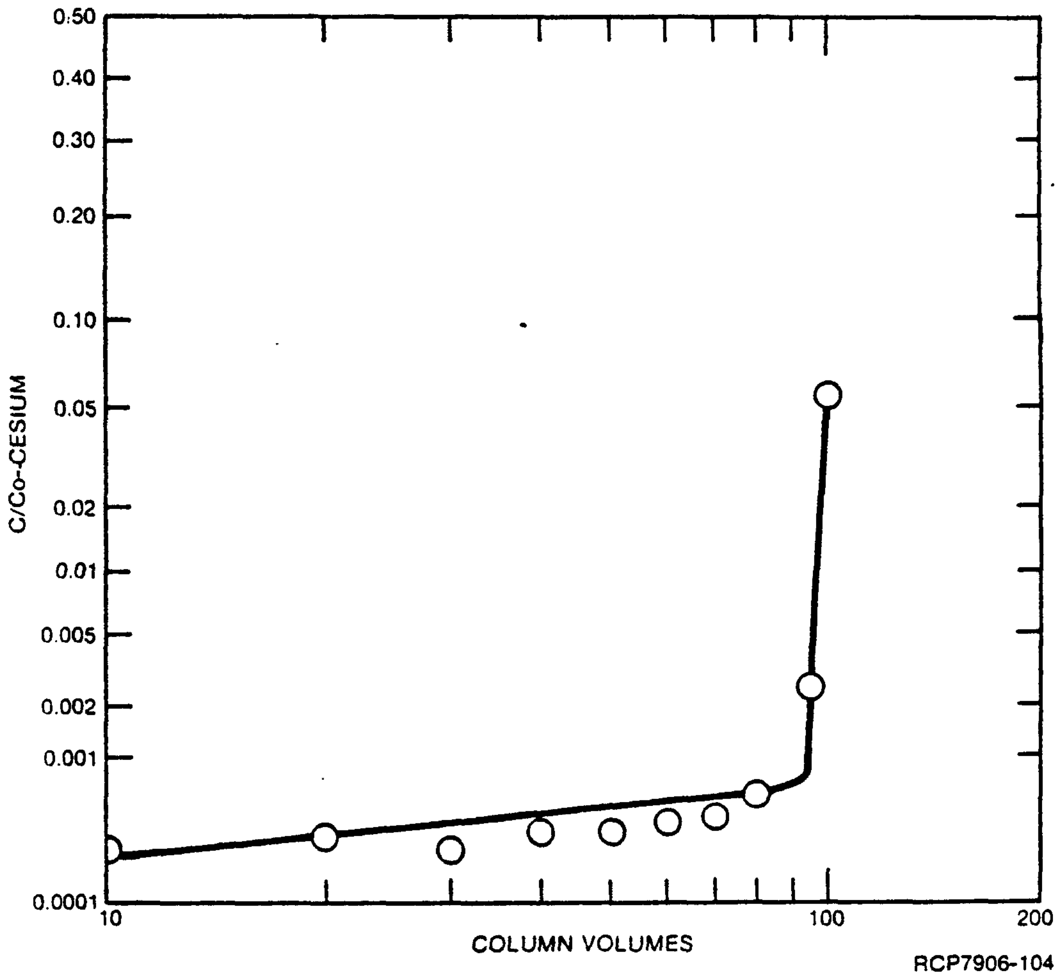
Run 20

Measure the stability of AW-500 when repetitively loaded, scrubbed, and eluted; fifth cycle.

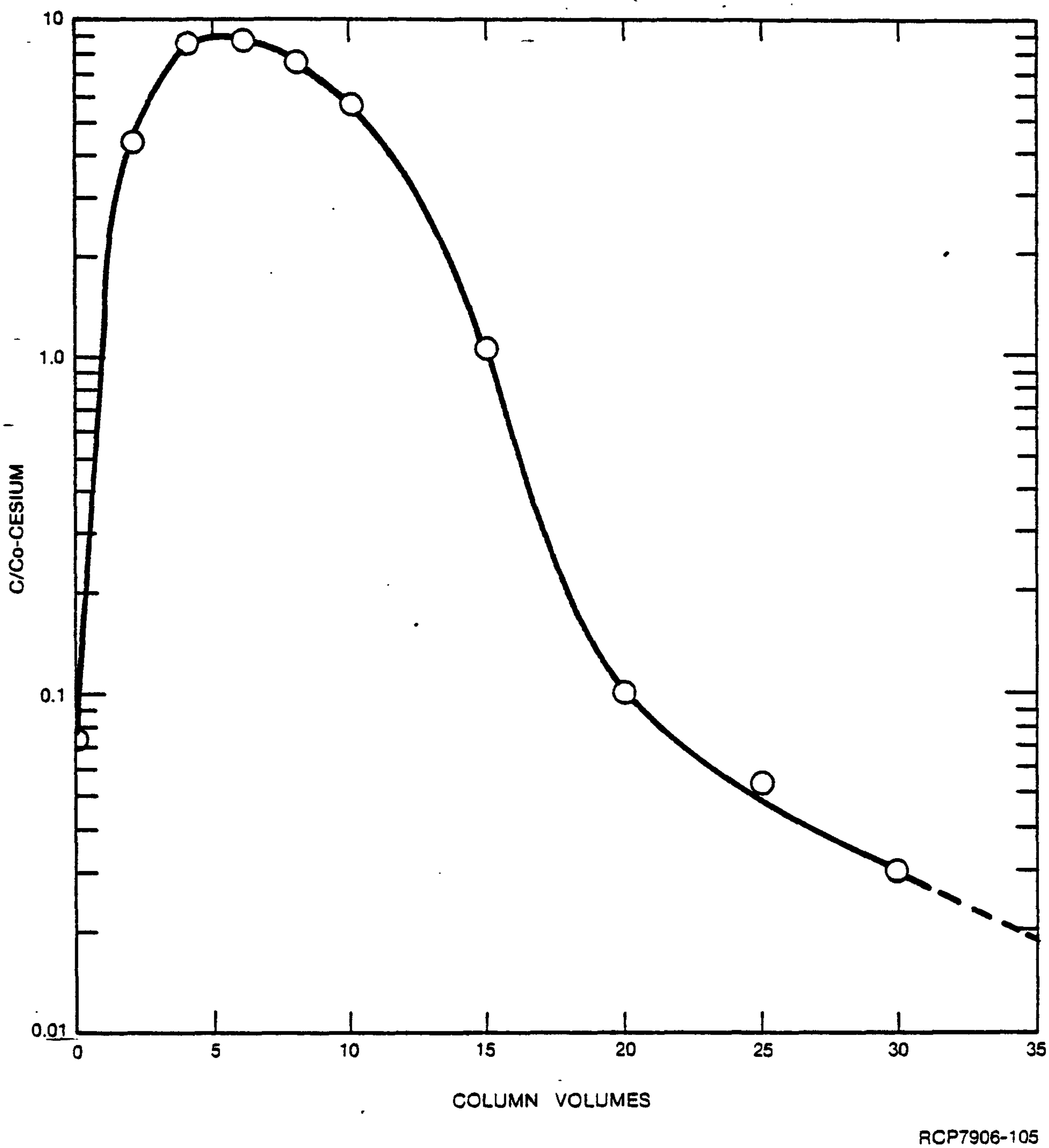
	Feed	Product
^{137}Cs	1180 μCi/ℓ	5170 μCi/ℓ
^{106}Rh	198 μCi/ℓ	---
^{144}Ce	68 μCi/ℓ	---
Cs^{+}	1.07×10^{-3} M	---
Na^{+}	1.75M	8.49×10^{-4} M
Fe^{3+}	0.111M	3.91×10^{-5} M
Al^{3+}	0.101M	<2.41×10^{-4} M
pH	4.5	---

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.003	180	0.91	65	0.96		E-2	5090	4.31
L-20	3	0.003	186	0.94	67	0.99		E-4	9950	8.42
L-30	3	0.003	194	0.98	67	0.99		E-6	10200	8.65
L-40	4	0.003	183	0.92	66	0.97		E-8	8900	7.53
L-50	4	0.003	193	0.98	67	0.99		E-10	6630	5.61
L-60	4	0.003	185	0.93	66	0.97		E-15	1240	1.05
L-70	5	0.004	191	0.96	66	0.97		E-20	121	0.10
L-80	7	0.006	130	0.66	67	0.99		E-25	62	0.05
L-95	31	0.026	194	0.98	66	0.97		E-30	35	0.03
L-100	65	0.055	192	0.97	68	1.00				
W-1,2	61	0.052	147	0.74	52	0.77				
S-1-10	85	0.072					0.402			

Note: Following the fifth cycle, the AW-500 was tested for deterioration by "rub sieving". The AW-500 had softened and deteriorated to the point all of the material passed through a 50 mesh (297um) screen.



Cesium Breakthrough Curve - Run 20



Cesium Elution Curve - Run 20

Run 21

Repetitive runs to test the stability of the zeolite ion exchanger (AW-500) with feed saturated with B Plant 1AX; first cycle.

	Feed*	Product
^{137}Cs	1130 μCi/ℓ	4320 μCi/ℓ
^{106}Rh	184 μCi/ℓ	---
^{144}Ce	64 μCi/ℓ	---
Cs^{+}	1.07×10^{-3} M	---
Na^{+}	1.89M	0.015M
Fe^{3+}	0.106M	2.65×10^{-5} M
Al^{3+}	0.094M	$<3.1 \times 10^{-4}$ M
pH	4.5	---

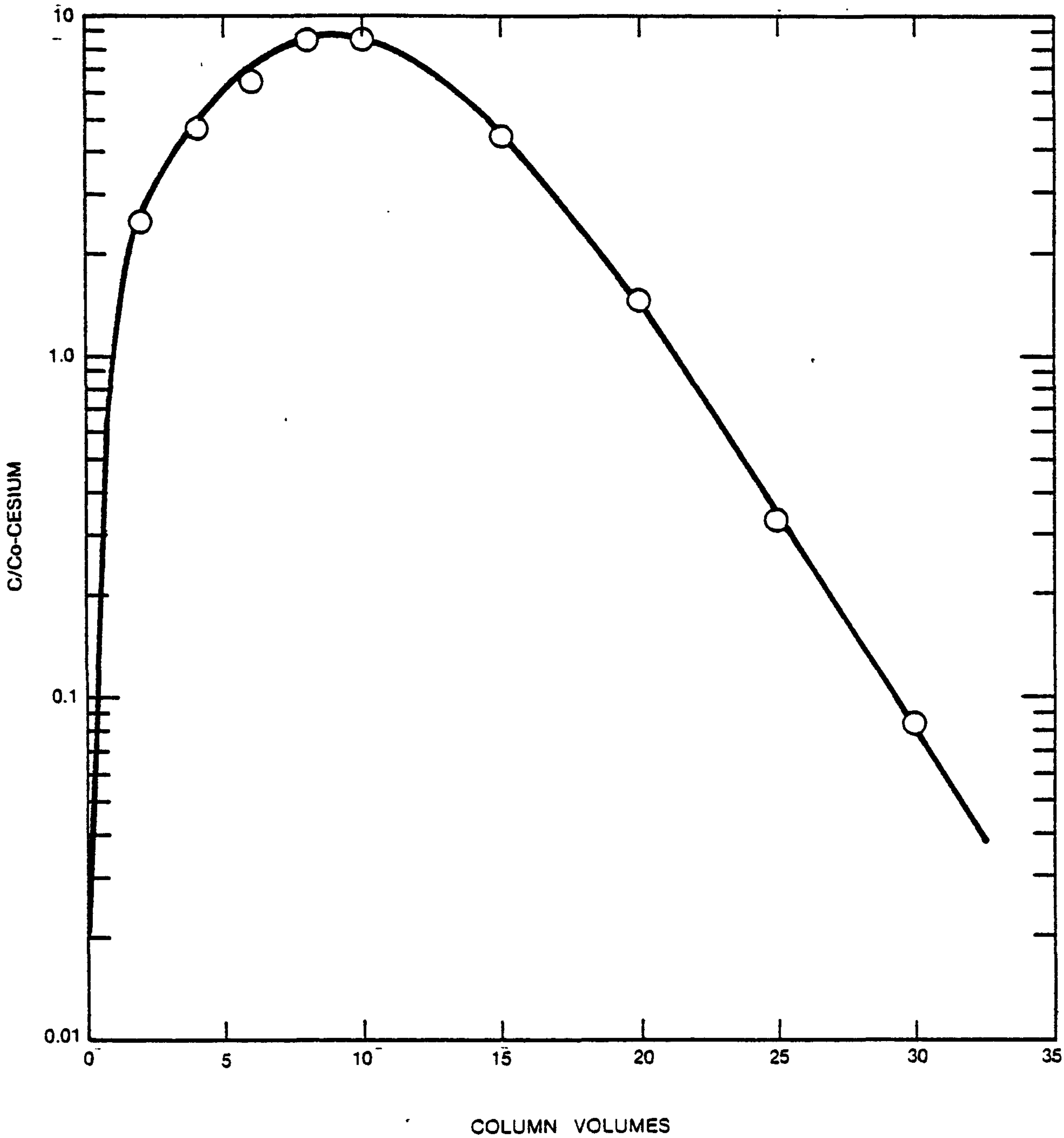
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	3	0.003	182	0.99	47	0.73		E-2	2770	2.44
L-20	2	0.002	185	1.01	45	0.70		E-4	5240	4.63
L-30	0	0	185	1.01	42	0.66		E-6	7260	6.41
L-40	0	0	191	1.04	43	0.67		E-8	9540	8.42
L-50	0	0	198	1.08	43	0.67		E-10	9660	8.53
L-60	0	0	190	1.03	38	0.59		E-15	4940	4.36
L-70	0	0	189	1.03	38	0.59		E-20	1660	1.46
L-80	0	0	193	1.05	37	0.58		E-25	369	0.33
L-90	0	0	189	1.03	36	0.56		E-30	94	0.083
L-100	0	0	186	1.01	33	0.52				
L-110	2	0.002	190	1.03	35	0.55				
W-1,2	0	0	70	0.38	10	0.16				
S-1-10	0	0	0	0	0	0	0.285			

*Saturated with B Plant 1AX (0.5M HDEHP, 0.3M TBP, NPH)

Volume at start of cycle - 20.0 cc
Volume at end of cycle - 19.5 cc
Percent lost first cycle - 2.5



Cesium Elution Curve - Run 21

Run 22

Repetitive runs to test the stability of the zeolite ion exchanger (AW-500) with feed saturated with B Plant 1AX; second cycle.

	Feed*	Product
^{137}Cs	1170 µCi/ℓ	4140 µCi/ℓ
^{106}Rh	187 µCi/ℓ	---
^{144}Ce	65 µCi/ℓ	---
Cs^{+}	1.07×10^{-3}M	---
Na^{+}	1.91M	0.002M
Fe^{3+}	0.098M	2.76×10^{-5}M
Al^{3+}	0.099M	2.50×10^{-4}M
pH	4.5	---

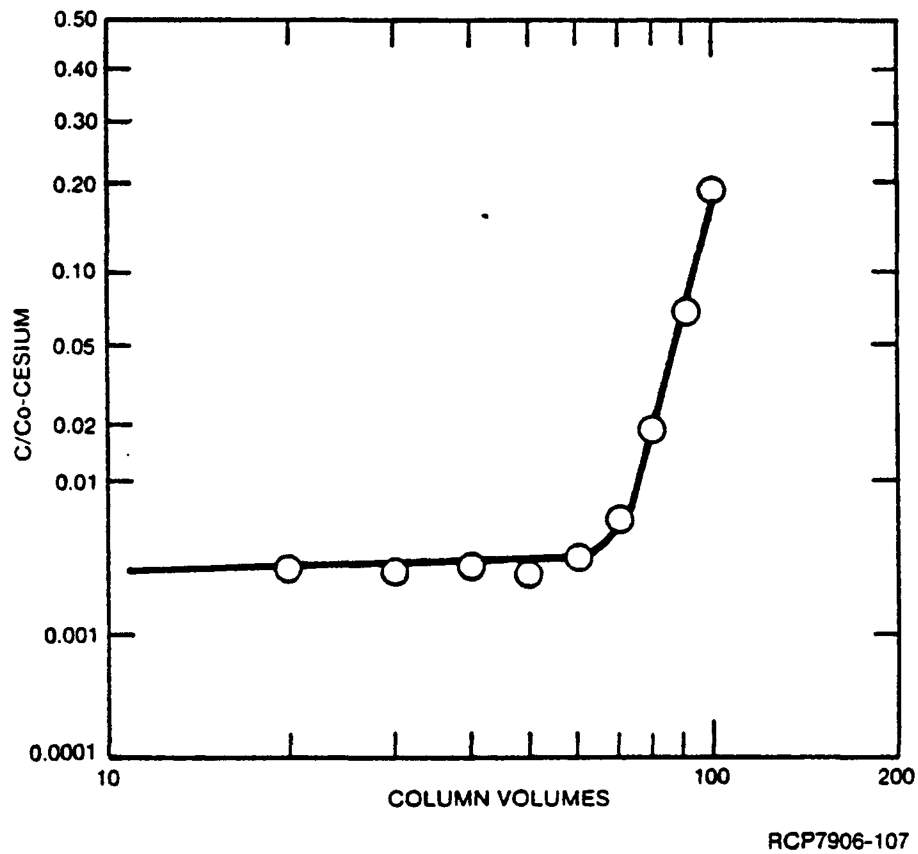
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

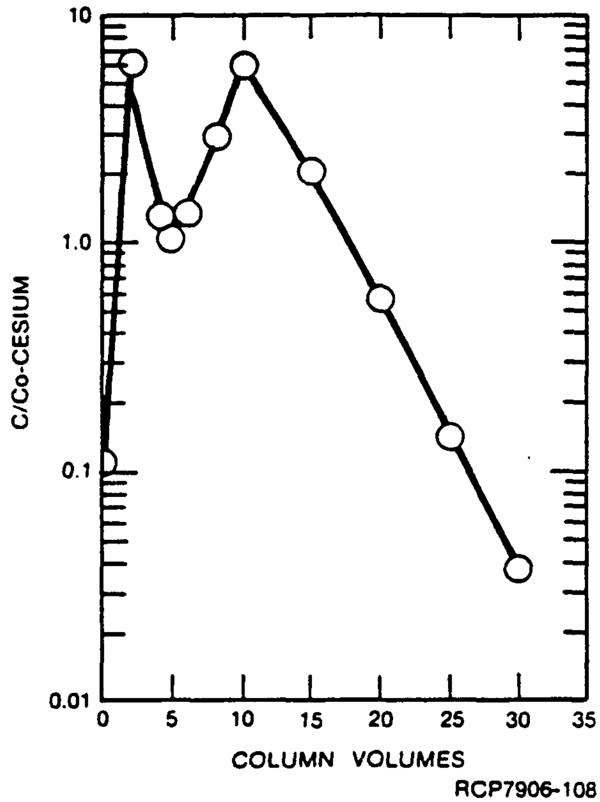
Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	µCi/ℓ	C/Co	µCi/ℓ	C/Co	µCi/ℓ	C/Co			µCi/ℓ	C/Co
L-10	-	-	186	0.99	65	1.00		E-2	7140	6.10
L-20	3	0.003	190	1.02	63	0.97		E-4	1520	1.30
L-30	3	0.003	186	0.99	63	0.97		E-6	1550	1.32
L-40	3	0.003	190	1.02	63	0.97		E-8	3390	2.90
L-50	3	0.003	203	1.09	63	0.97		E-10	6860	5.86
L-60	4	0.003	195	1.04	63	0.97		E-15	2430	2.08
L-70	7	0.006	192	1.03	62	0.95		E-20	657	0.56
L-80	23	0.020	189	1.01	60	0.92		E-25	159	0.14
L-90	79	0.067	194	1.04	62	0.95		E-30	44	0.038
L-100	219	0.19	192	1.03	58	0.89				
W-1,2	237	0.20	149	0.80	40	0.62				
S-1-10	125	0.11					0.126			

*Saturated with B Plant 1AX (0.5M HDEHP, 0.3M TBP, NPH)

Volume at start - 20 cc
Volume at start of cycle - 19.5 cc
Volume at end of cycle - 18.5 cc
Percent lost during cycle - 5.1
Percent lost from two cycles - 7.5



Cesium Breakthrough Curve - Run 22



Cesium Elution Curve - Run 22

Run 23

Repetitive runs to test the stability of the zeolite ion exchanger (AW-500) with feed saturated with B Plant 1AX; third cycle.

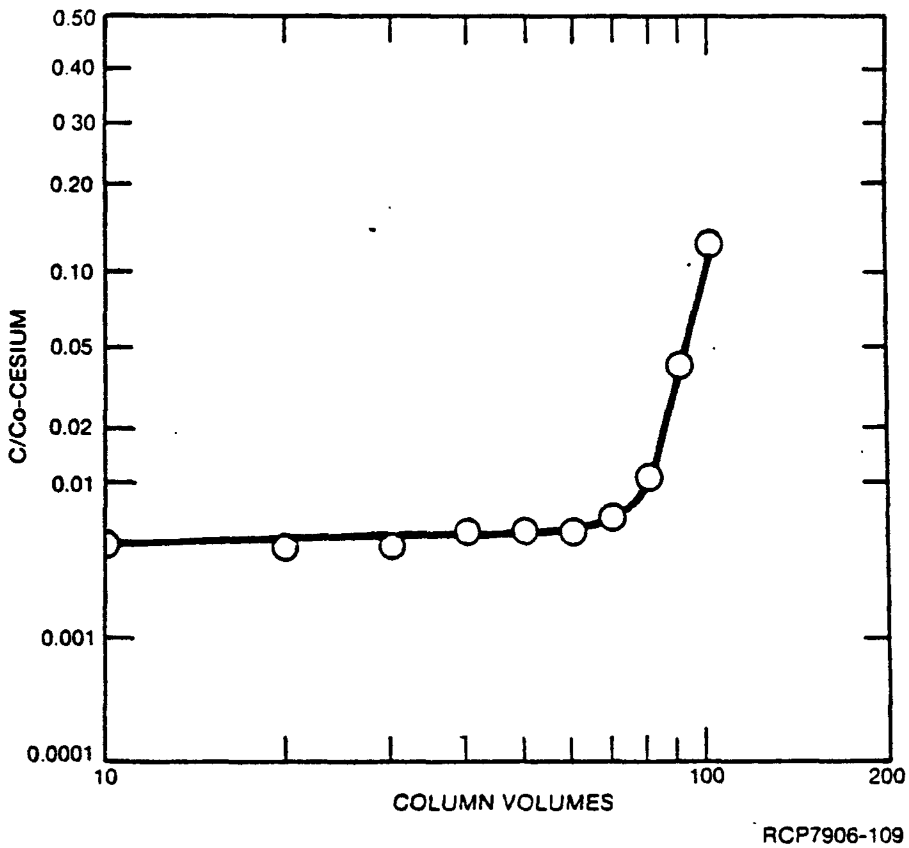
	Feed	Product
^{137}Cs	1090 μCi/ℓ	4011 μCi/ℓ
^{106}Rh	172 μCi/ℓ	---
^{144}Ce	56 μCi/ℓ	---
Cs^+	1.07×10^{-3}M	---
Na^+	1.9M	1.06×10^{-3}M
Fe^{3+}	0.102M	$<1.15 \times 10^{-5}$M
Al^{3+}	0.082M	$<2.20 \times 10^{-4}$M
pH	4.5	---

Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	4	0.004	170	0.99	50	0.90		E-2	7450	6.82
L-20	4	0.004	166	0.97	46	0.83		E-4	9150	8.38
L-30	5	0.004	173	1.01	48	0.86		E-6	9290	8.51
L-40	5	0.004	179	1.04	50	0.90		E-8	7810	7.15
L-50	6	0.005	179	1.04	49	0.88		E-10	6740	6.17
L-60	7	0.005	173	1.01	48	0.86		E-15	2850	2.61
L-70	7	0.006	169	0.98	47	0.85		E-20	667	0.61
L-80	14	0.013	177	1.03	49	0.88		E-25	151	0.14
L-90	44	0.041	178	1.03	48	0.86		E-30	41	0.038
L-100	141	0.129	170	0.99	44	0.79				
W-1,2	36	0.033	33	0.19	6	0.11				
S-1-10	84	0.077					0.140			

Volume at start - 20 cc
Volume at start of cycle - 18.5 cc
Volume at end of cycle - 16.0 cc
Percent lost during cycle - 13.5
Percent lost over three cycles - 32.5



Cesium Breakthrough Curve - Run 23

Run 24

Repetitive runs to test the stability of the zeolite ion exchanger (AW-500) with feed saturated with B Plant 1AX; fourth cycle.

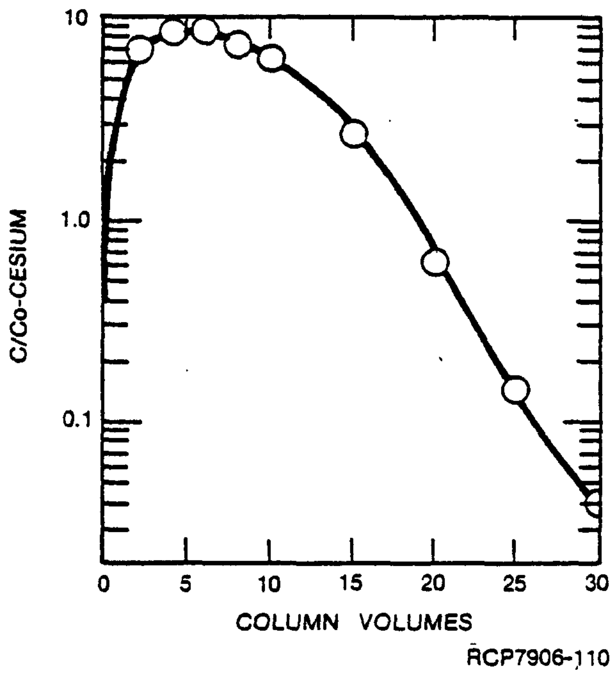
	Feed	Product
^{137}Cs	1089 μCi/ℓ	3010 μCi/ℓ
^{106}Rh	168 μCi/ℓ	---
^{144}Ce	52 μCi/ℓ	---
Cs^{+}	1.07×10^{-3}M	---
Na^{+}	1.83M	4.91×10^{-3}M
Fe^{3+}	0.104M	1.38×10^{-5}M
Al^{3+}	0.115M	$<9.31 \times 10^{-4}$M
pH	4.5	---

Scrub: 0.15M $(NH_4)_2CO_3$

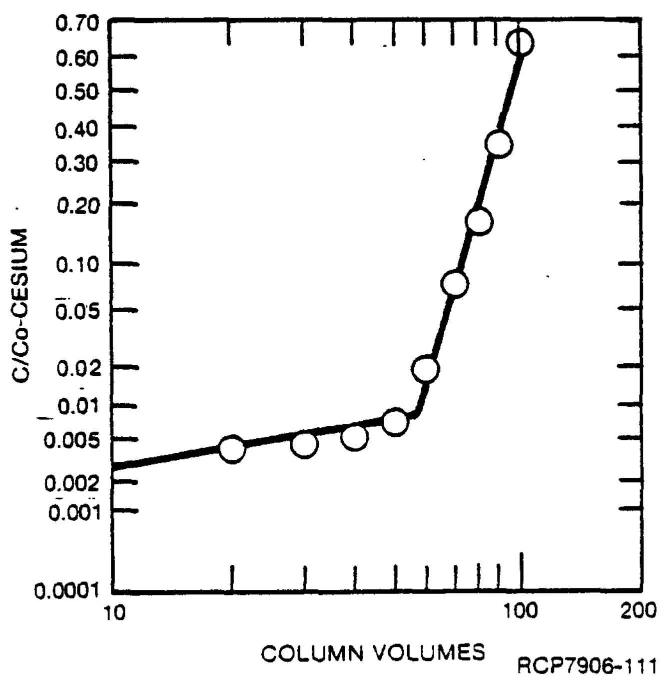
Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	0	0	173	1.03	51	0.98		E-2	10600	9.72
L-20	4	0.004	172	1.02	51	0.98		E-4	11700	10.7
L-30	5	0.004	169	1.01	49	0.94		E-6	8390	7.71
L-40	5	0.004	171	1.02	49	0.94		E-8	4740	4.36
L-50	7	0.007	166	0.99	46	0.88		E-10	2560	2.35
L-60	21	0.019	174	1.04	48	0.92		E-15	581	0.53
L-70	80	0.074	171	1.02	46	0.88		E-20	184	0.17
L-80	180	0.17	175	1.04	45	0.87		E-25	70	0.064
L-90	371	0.34	172	1.02	44	0.85		E-30	37	0.034
L-100	691	0.64	173	1.03	45	0.87				
W-1,2	250	0.23	64	0.38	14	0.27				
S-1-10	546	0.50					0.080			

Volume at start - 20 cc
Volume at start of cycle - 16 cc
Volume at end of cycle - 0.5 cc
Percent lost during cycle - 97
Percent lost over four cycles - 98



Cesium Breakthrough Curve - Run 24



Cesium Elution Curve - Run 24

Run 25

Test the effectiveness of aluminum as a blocking agent to inhibit the deterioration of AW-500; first cycle.

	Feed*	Product
^{137}Cs	1080 μCi/ℓ	3380 μCi/ℓ
^{106}Rh	167 μCi/ℓ	---
^{144}Ce	49 μCi/ℓ	---
Cs^+	9.71×10^{-4} M	---
Na^+	1.92M	1.42×10^{-3} M
Fe^{3+}	0.090M	2.68×10^{-4} M
Al^{3+}	0.121M	$<2.34 \times 10^{-4}$ M
pH	3.9	---

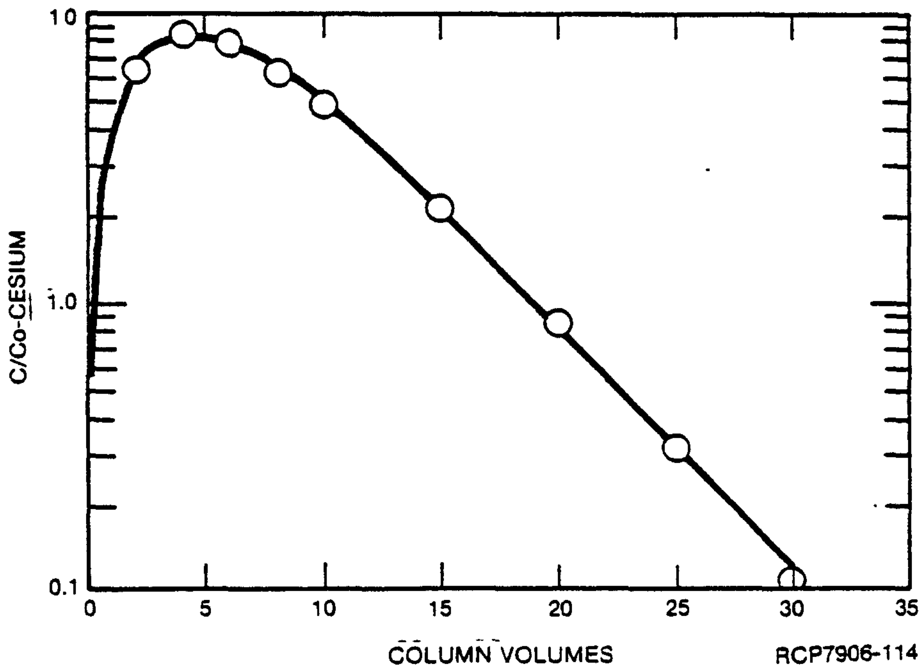
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

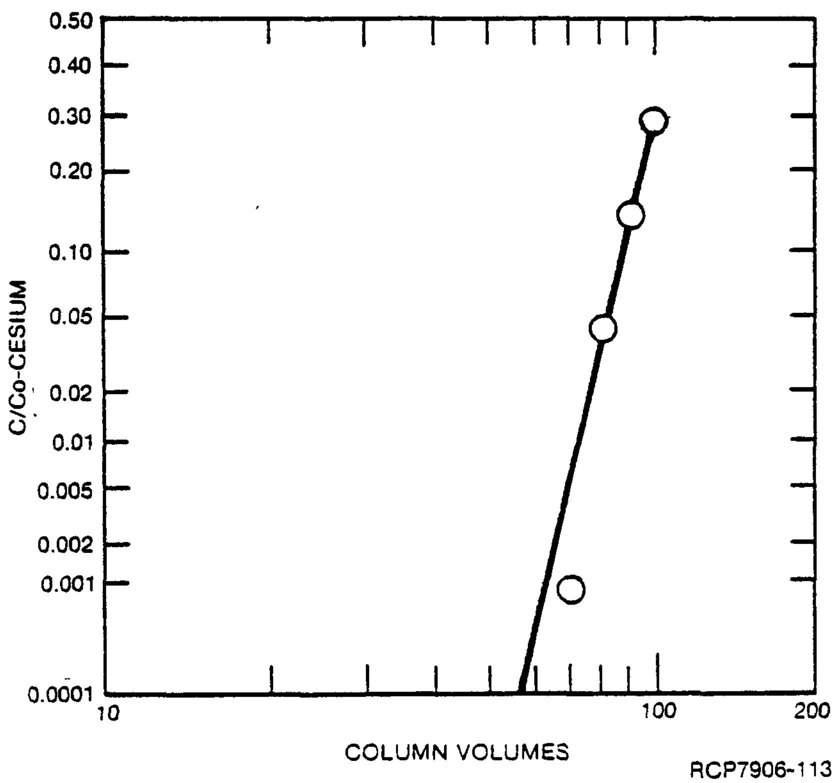
Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	0	0	157	0.94	47	0.97		E-2	6760	6.25
L-20	0	0	162	0.97	49	0.99		E-4	8800	8.21
L-30	0	0	145	0.86	46	0.93		E-6	8500	7.86
L-40	0	0	131	0.78	46	0.93		E-8	6720	6.21
L-50	0	0	131	0.78	48	0.98		E-10	5270	4.87
L-60	0	0	126	0.75	46	0.94		E-15	2290	2.11
L-70	9	0.008	122	0.73	46	0.94		E-20	916	0.85
L-80	47	0.043	128	0.77	48	0.98		E-25	347	0.32
L-90	151	0.14	130	0.78	50	1.01		E-30	123	0.11
L-100	309	0.29	125	0.77	48	0.98				
W-1,2	142	0.13								
S-1-10	220	0.20					0.118			

*Saturated with B Plant 1AX (0.5M HDEHP, 0.3M TBP, NPH)

Volume of AW-500 at start - 20 cc



Cesium Breakthrough Curve - Run 25



Cesium Elution Curve - Run 25

Run 26

Test the effectiveness of aluminum as a blocking agent to inhibit the deterioration of AW-500; second cycle.

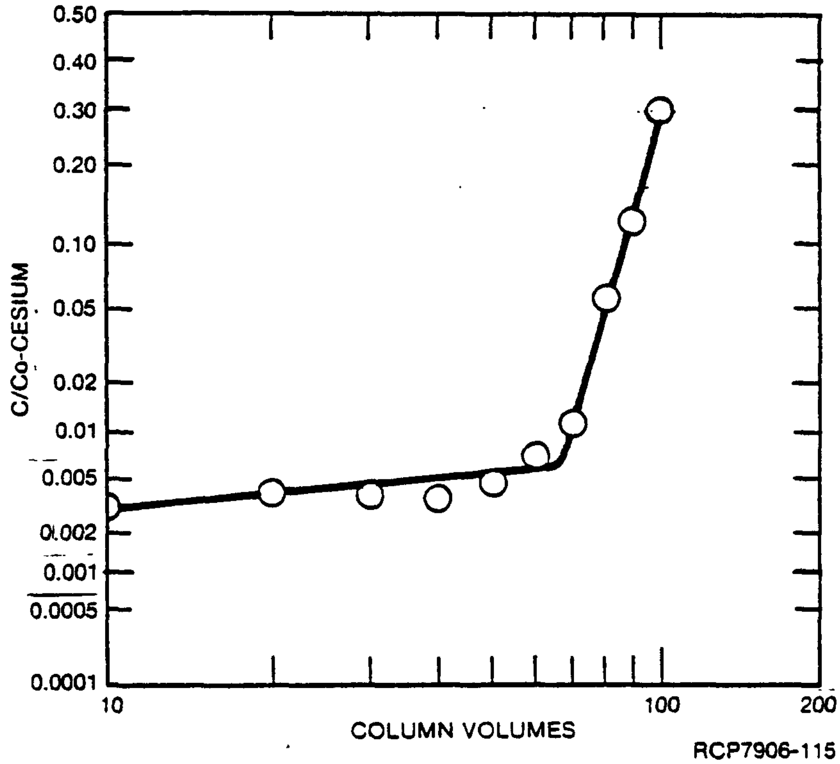
	Feed*	Product
^{137}Cs	1110 μCi/ℓ	3810 μCi/ℓ
^{106}Rh	165 μCi/ℓ	---
^{144}Ce	49 μCi/ℓ	---
Cs^{+}	9.71×10^{-4} M	---
Na^{+}	1.88M	1.00×10^{-3} M
Fe^{3+}	0.097M	1.09×10^{-4} M
Al^{3+}	0.137M	2.08×10^{-4} M
pH	3.6	---

Scrub: 0.15M $(NH_4)_2CO_3$

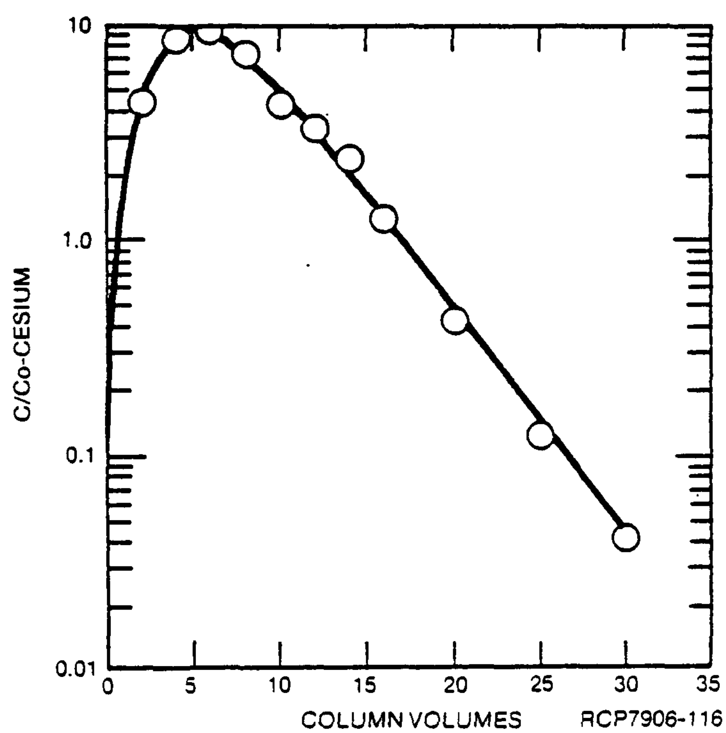
Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co		μCi/ℓ	C/Co
L-10	3	0.003	126	0.78	48	0.99	E-2	4750	4.30
L-20	4	0.004	100	0.62	48	0.99	E-4	9150	8.28
L-30	4	0.004	94	0.58	50	1.02	E-6	10300	9.36
L-40	4	0.004	92	0.57	48	1.00	E-8	7870	7.12
L-50	5	0.005	86	0.53	48	0.98	E-10	4970	4.50
L-60	8	0.007	89	0.55	49	1.00	E-12	3570	3.23
L-70	20	0.018	84	0.52	48	0.99	E-14	2580	2.33
L-80	64	0.058	84	0.52	48	0.99	E-16	1380	1.25
L-90	137	0.12	85	0.53	50	1.02	E-20	462	0.42
L-100	342	0.31	84	0.52	52	1.06	E-25	129	0.12
W-1,2	342	0.31	84	0.52	52	1.06	E-30	45	0.04
S-1-10	160	0.14	292	1.80					

*Saturated with B Plant 1AX (0.5M HDEHP, 0.3M TBP, NPH)



Cesium Breakthrough Curve - Run 26



Cesium Elution Curve - Run 26

Run 27

Test the effectiveness of aluminum as a blocking agent to inhibit the deterioration of AW-500; third cycle.

	Feed*	Product
^{137}Cs	1110 μCi/ℓ	2880 μCi/ℓ
^{106}Rh	162 μCi/ℓ	---
^{144}Ce	51 μCi/ℓ	---
Cs^{+}	9.71×10^{-4}M	---
Na^{+}	1.96M	4.07×10^{-3}M
Fe^{3+}	0.142M	4.85×10^{-4}M
Al^{3+}	0.138M	$<2.0 \times 10^{-4}$M
pH	3.81	---

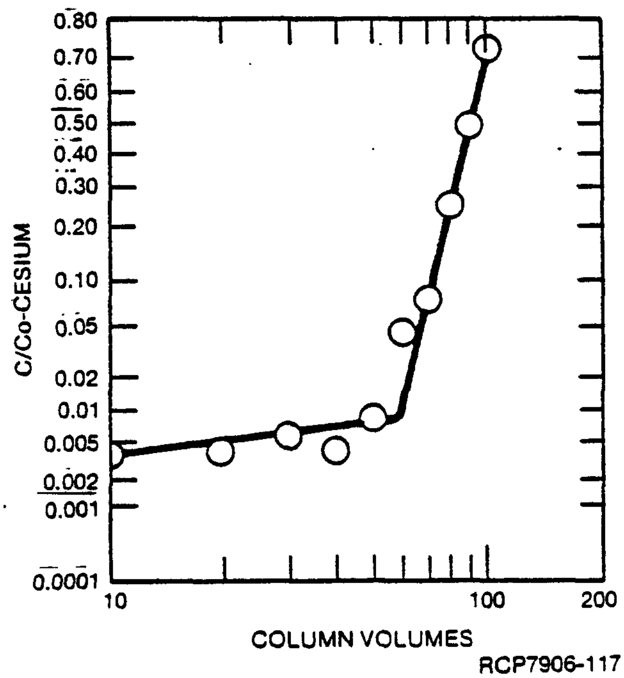
Scrub: 0.15M $(NH_4)_2CO_3$

Eluant: 3M $(NH_4)_2CO_3$ - 2M NH_4OH

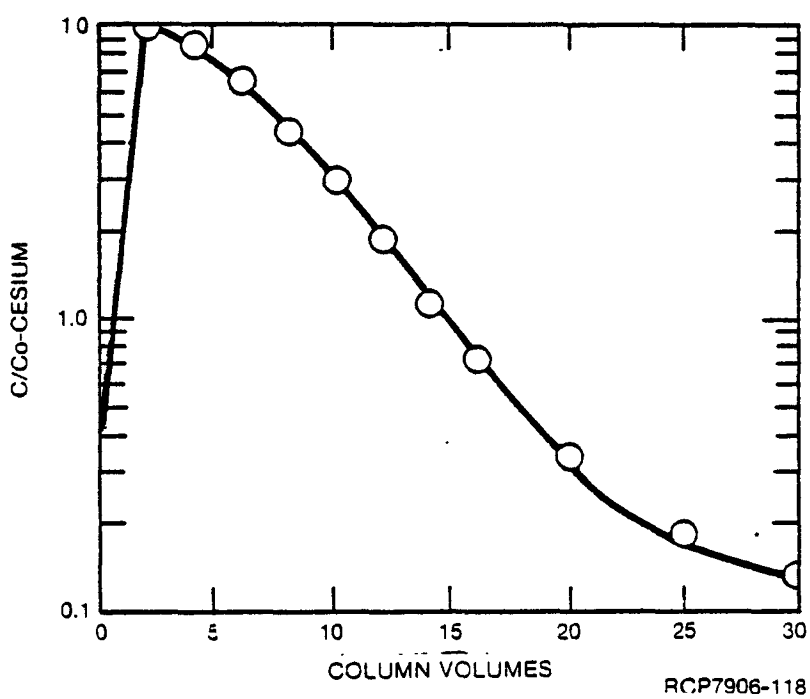
Effluent	^{137}Cs		^{106}Rh		^{144}Ce		Na, M	Effluent	^{137}Cs	
	μCi/ℓ	C/Co	μCi/ℓ	C/Co	μCi/ℓ	C/Co			μCi/ℓ	C/Co
L-10	4	0.004	138	0.85	49	0.96		E-2	11000	9.90
L-20	4	0.004	98	0.60	48	0.94		E-4	9440	8.50
L-30	6	0.005	98	0.60	51	1.00		E-6	7220	6.51
L-40	4	0.004	86	0.53	46	0.90		E-8	4740	4.27
L-50	9	0.008	86	0.53	48	0.94		E-10	3250	2.93
L-60	20	0.044	91	0.56	49	0.96		E-12	2070	1.87
L-70	86	0.077	88	0.54	49	0.96		E-14	1240	1.12
L-80	276	0.25	90	0.56	49	0.96		E-16	797	0.72
L-90	542	0.49	92	0.57	56	1.10		E-20	376	0.34
L-100	800	0.72	94	0.58	55	1.08		E-25	195	0.18
W-1,2	493	0.44	220	1.36			0.096	E-30	144	0.13

*Saturated with B Plant 1AX (0.5M HDEHP, 0.3M TBP, NPH)

Beginning volume of AW-500 - 20 cc
Volume at end of third cycle - 4.4 cc
Percent lost over three cycles - 78



Cesium Breakthrough Curve - Run 27



Cesium Elution Curve - Run 27

APPENDIX B

Internal Letter

Rockwell International

Date: February 15, 1979

No. 60140-79-069

TO: (Name, Organization Internal Address)
J. S. Buckingham
Chemical Sciences Group
222-S/TRL, 200 W
2-2487

FROM: (Name, Organization, Internal Address Phone)
D. F. Shepard
Statistical Sciences Group
271-U, 200 W
2-2492

Subject: Statistical Analysis of Data From Cesium Ion Exchanger Stability Study

Ref: Letter, J. S. Buckingham to D. F. Shepard, 11-21-78, same subject.

The statistical analysis of the data from the fourty-six stability tests on the Linde AW-500 and on the Norton Zeolon-900 zeolitic materials showed that:

1. The saturation of water with IAX did not significantly increase the rate of degradation of either AW-500 or Zeolon-900.

2. During the test period of 29 to 88 days, AW-500 degraded at a significantly (at the 99.9% level) higher rate than Zeolon-900.

3. During the test period of 29 to 74 days, the presence of chelates resulted in a doubling of the rate of degradation of AW-500.

4. During the test period of 28 to 77 days, the presence of chelates did not significantly (at the 90% level) change the rate of degradation of Zeolon-900.

5. During the test period of 28 to 99 days, the presence of AcOH significantly (at the 97.5% level) increased the degradation of Zeolon-900.

The effect on the degradation rate of water saturated with IAX compared to water only was examined using a two way analysis of variance. The saturation of the water with IAX solution did not significantly (at the 90% level) change the rate of degradation of either AW-500 or of Zeolon-900.

The data of the tests with water and water saturated with IAX were compared to the data of similar tests where all four chelates were present. For AW-500 the rate of loss per day in the absence of chelates was 0.456 ± 0.054. In the presence of chelates the rate significantly (at the 99% level) increased to 0.916 ± 0.193. For Zeolon-900 the rate of loss per day in the absence of chelates was 0.182 ± 0.060. The presence of chelates did not significantly change this rate, the value observed was 0.162 ± 0.080. The combined data, with and without chelates, gave an average rate of loss per day for Zeolon-900 of 0.172 ± 0.067.

The overall rate of degradation of the two materials was obtained by fitting all the data to the equation $\ln(\% \text{ Loss}) = a + \ln(\text{No. of Days})$. At 84 days the predicted loss of AW-500 was 73.0% (95% limits of 81.0% to 65.9%) and of Zeolon-900 was 18.5% (95% limits of 22.4% to 15.3%). The rate of loss of AW-500 was significantly (at the 99.9% level) greater than the rate of loss of Zeolon-900.

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An attempt was made to use Yates method to determine which chelate, or combination of chelates, caused an increase in the degradation of the two materials. The effect of the number of days of exposure was removed by converting all observed percent losses to the same number of days of exposure, 88 days for AW-500 and 99 days for Zeolon-900. This correction was made using the average rate of loss per day for each material. The presence of high order interaction terms prevented the determination of significant effects due to the individual chelates or combinations of chelates.

An indication of which chelates or combinations of chelates were causing an increase in the degradation of the zeolitic materials was obtained using step-wise regression. The results obtained are summarized in Table 1.

TABLE 1

Coefficients of Equation % Loss = f(No. Days, Conc. of Chelates)

Variable	Units	AW-500 Coefficient	Zeolon-900 Coefficient
Days	Number of Days	0.410	-
HEDTA	M	126.	-
EDTA	M	-469.	-
H_3Cit	M	128.	-
AcOH	M	99.	26.7
Days x Days	$(\text{No. of Days})^2$	-	0.00292
Days x EDTA	(No. of Days) x M	6.8	-
H_3Cit x AcOH	M x M	-421.	-
Constant		1.3	-1.1
R Squared		.96	.73
Standard Deviation of Residuals		5.8	5.8

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Only coefficients having an F ratio significant at the 97.5% level were included in the above table. However, the demonstration of the effect of individual chelates or combinations of chelates, on the degradation of the materials would require experimental data from tests where direct comparisons could be made.

The R squared of 0.96 shows that the coefficients identified in Table 1 account for 96% of the observed variation in percent loss of AW-500. The residual of 5.8% is the standard deviation of the difference between the percent loss calculated using the coefficients of Table 1 and the percent loss observed. This residual standard deviation is due to either experimental measurement error or degradation of the material not accounted for by the time in days, specific chelates, or combinations identified in Table 1. Step-wise regression selects those effects that best account for the observed percent loss. There is no assurance that the effects selected are in fact those responsible for the degradation of the material.

The R squared of 0.73 shows that the two coefficients identified in Table 1 account for 73% of the observed variation in percent loss of Zeolon-900. This is considerably less than the variation accounted for with AW-500. However, the standard deviation of the difference between calculated and observed percent loss was the same for Zeolon-900 as for AW-500. While the coefficient for AcOH was significant at the 97.5% level, addition of this coefficient only increased the variation in percent loss accounted for from 67% to 73% and decreased the standard deviation of the residual from 6.2% to 5.8%. Verification of an effect of this order of magnitude would require direct comparison tests similar to those made for the combination of all four chelates.

The detailed computer print outs of the statistical analysis are available in our office. If you have any questions regarding this statistical analysis of your data, please let us know.

L. P. McRae for

D. F. Shepard
Senior Statistician
Statistical Sciences Group

DFS/dm

cc: H. Babad
R. F. Carlestrom
F. M. Jungfleisch
L. P. McRae
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