

NOV 19 1963

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HW-76449

ION EXCHANGE SEPARATION OF CESIUM FROM ALKALINE WASTE

SUPERNATANT SOLUTIONS

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Process Research and Development  
Chemical Effluents Technology  
CHEMICAL LABORATORY

**MASTER**

HANFORD LABORATORIES

January 30, 1963

Facsimile Price \$ 3.60  
Microfilm Price \$ 1.13  
Available from the  
Office of Technical Services  
Department of Commerce  
Washington 25, D. C.

HANFORD ATOMIC PRODUCTS OPERATION

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ION EXCHANGE SEPARATION OF CESIUM FROM ALKALINE WASTE  
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INTRODUCTION

Cesium-137 is the major radionuclide in aged high-level alkaline supernatant wastes which requires removal to permit in-tank solidification of the waste. The removal of this isotope from stored alkaline wastes is thus a key step in the current Chemical Processing Department waste management program (1). Accelerated use of Cs<sup>137</sup> for large scale irradiation sources also focuses attention on methods for its recovery from presently stored wastes. Cesium recovered from aged wastes is preferable to that from fresh wastes because the shorter-lived Cs<sup>134</sup> is partially decayed.

Possible processes for cesium separation from alkaline supernatant waste include ferrocyanide precipitation (2), solvent extraction (3), and ion exchange. Earlier studies indicated that an ion exchange process might be developed for the separation of cesium from high-level wastes (4). An ion exchange process was developed for Purex acid waste (5); a similar process has now been developed for cesium separation from Purex alkaline supernatant wastes. In addition to high radiation and thermal stability the ion exchange medium must have a high selectivity for cesium over sodium for this latter application. Good physical form, low solubility, and ready availability are further desirable characteristics. Several inorganic exchangers meet these requirements. Some phenolic resins may also be satisfactory. Studies on cesium separation from Purex alkaline supernatant waste with several ion exchangers are reported in this document.

SUMMARY

Ion exchange, with inorganic zeolites or phenolic resins, provides a good means of separating cesium from Purex alkaline supernatant waste. High cesium loadings are obtained with AW-400, a synthetic zeolite manufactured by the Linde Company. A disadvantage of the AW-400 at present is the poor quality of the binder. However, efforts are being made to obtain a more suitable binder. Optimum loading conditions appear to be a flow rate of one to two column volumes of actual waste per hour, with 20-50 mesh exchanger at a temperature of 25C. A "scrub" or sodium removal step can be accomplished with several solutions; dilute ammonium carbonate solutions are preferred for aged alkaline supernatant wastes.

Cesium is eluted with a solution of ammonium carbonate and ammonium hydroxide. Exchangers that are most selective for cesium and have the highest loadings also require the greatest elution volumes. Ammonium carbonate can be volatilized by heating, leaving a cesium solution that is very satisfactory for subsequent packaging operations. Under practical conditions a Cs/Na D F of  $10^3$  and cesium recovery of > 99 percent can be obtained.

RESULTSLaboratory Studies

Most of the laboratory experiments were carried out with a synthetic waste to which "cold" cesium and Cs<sup>137</sup> or Na<sup>22</sup> tracer were added. The composition of the synthetic Purex alkaline supernatant waste used in the experiments is given in Table I.

TABLE IComposition of Synthetic Purex Alkaline Supernatant Waste

<u>Constituent</u>	<u>Molarity</u>
Na <sup>+</sup>	9.0
NO <sub>3</sub> <sup>-</sup>	2.6
NO <sub>2</sub> <sup>-</sup>	4.2
CO <sub>3</sub> <sup>=</sup>	1.0
PO <sub>4</sub> <sup>=</sup>	0.028
SO <sub>4</sub> <sup>=</sup>	0.09
Cs <sup>+</sup>	100 percent

The waste solution was, in most cases, made up to half of the concentration to provide the equivalent of a 1:1 dilution of the waste. However more recent analysis of actual wastes indicate sodium molarities of about 5 rather than the 9 reported earlier. On this basis the 1:1 dilution of synthetic wastes of composition listed in Table I, probably is typical of actual waste compositions.

Sodium is the only major cation present, thus a sodium-cesium competition is the system of interest. Estimates of total cesium loading level in the presence of sodium can be made with equilibrium data of Ames and Knoll (6).

The columns used for most of the laboratory studies were made of glass tubing, 0.8 cm inside diameter and 20 cm long, with a 10 ml column volume of exchanger, (20-50 mesh in most cases). The columns were immersed in a constant temperature bath which was at 25°C for most loadings and washings and 55°C for elutions. The influent was fed to the column at a constant flow rate using positive displacement pumps. The effluent was collected with automatic fraction collectors and counted for Cs<sup>137</sup> or Na<sup>22</sup>.

Loading Variables

Cesium breakthrough curves for several exchangers (plotted on logarithmic probability paper) are shown in Figure 1. Duolite C-3 (a phenolic resin manufactured by Chemical Process Co.) performed quite poorly with the pH-10 waste solution. However, when an excess of sodium hydroxide is added to the waste, the cesium selective phenolic sites are activated and cesium loading is improved. A Bio-Rad product, Bio-Rex 40 (data not shown) is a modified C-3 phenolic resin and performs in a similar manner to the C-3. Decalso, a synthetic aluminosilicate gel (Permutit Co.) which was used with this waste in a previous study (7), yields a 50 percent cesium breakthrough at seven to eight column volumes (not shown here). Clinoptilolite, a natural zeolite, gives a 50 percent breakthrough at about 20 column volumes of actual waste while the new synthetic zeolites Zeolon (Norton Co.), AW-500 and AW-400 (Linde Co.) yield cesium loadings to 50 percent breakthrough of 30, 41, and 48 column volumes, respectively. All of the materials had similar kinetics, as indicated by the slopes of the curves for the same flow rates and particle sizes.

Cesium breakthrough curves for AW-400 of various particle sizes, are shown in Figure 2. The 1/16-inch pellets were crushed and sieved to obtain the desired particle size ranges. Since the same pelleting process and bonding agent is used for AW-500 as for AW-400, the particle size variable should have similar effects with AW-500 also. In general, the pelleted materials show less flattening of the breakthrough curves with increasing particle size than the naturally-cemented materials such as clinoptilolite. Duolite C-3 has a large particle size effect.



Figure 1. Cesium Breakthrough Curves for Several Exchangers Using Synthetic Purex Alkaline Supernatant Waste.

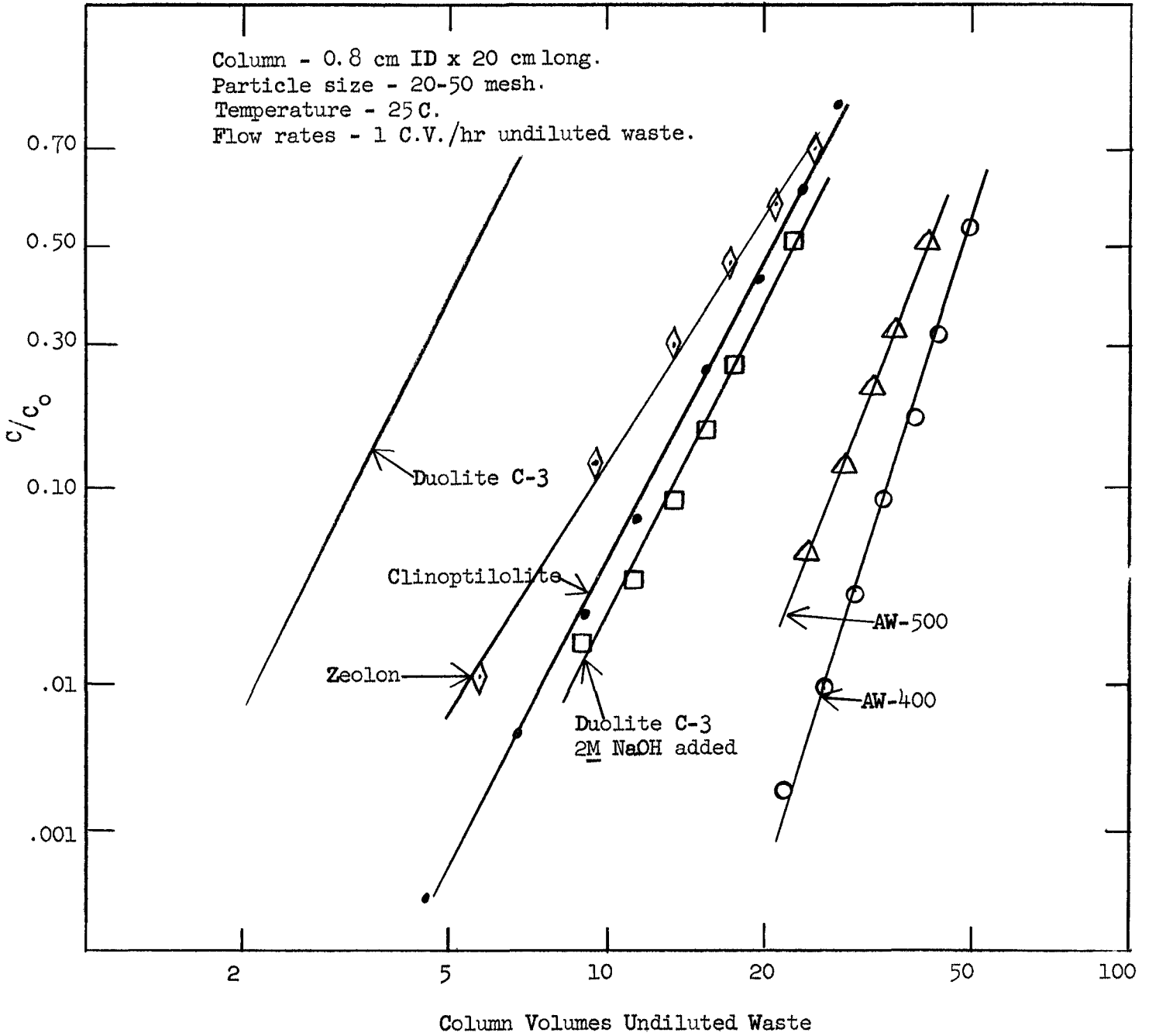
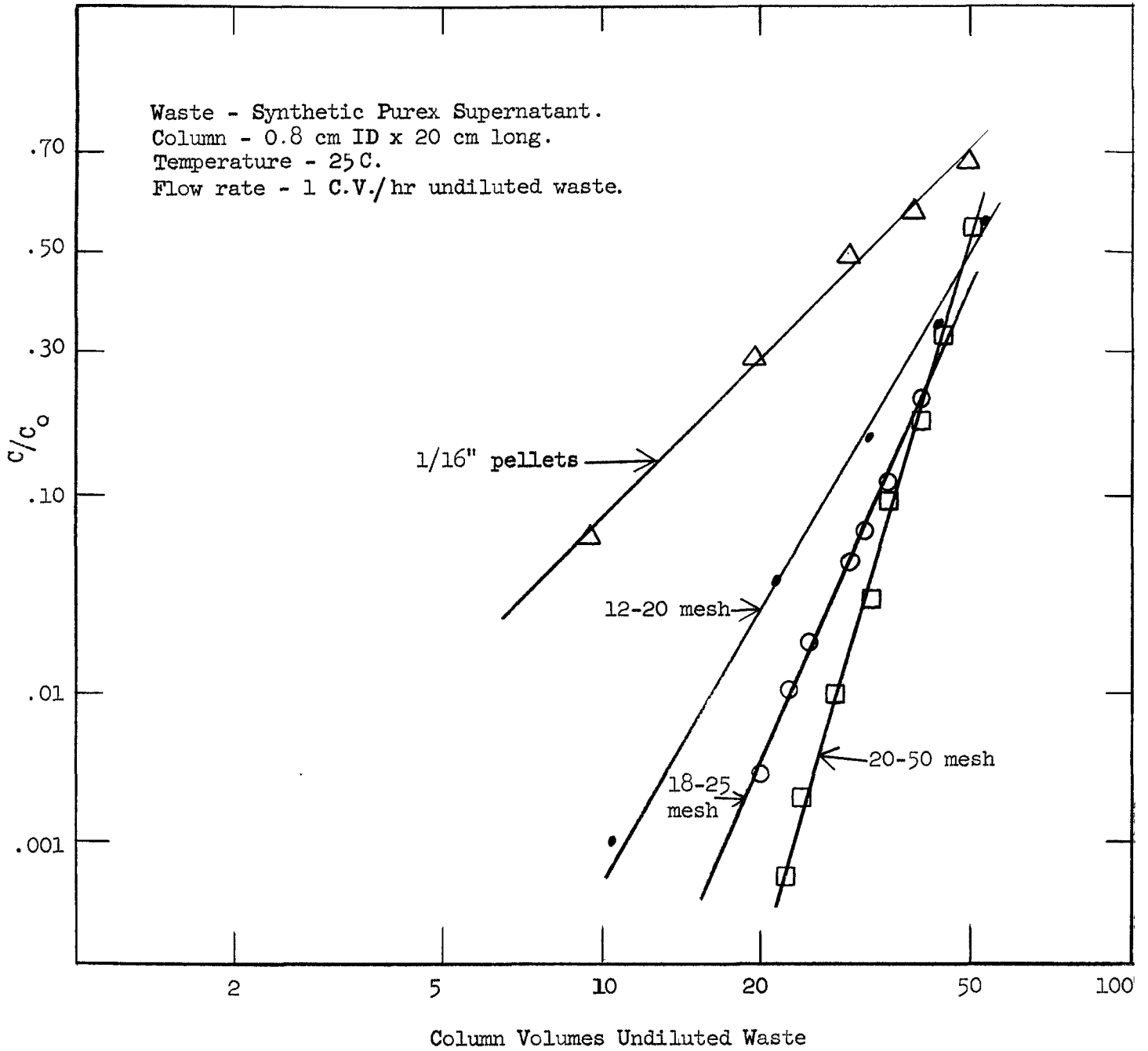


Figure 2. Effect of AW-400 Particle Size on Cesium Breakthrough Curves.



The effect of flow rate on cesium breakthrough for AW-400 is illustrated in Figure 3. The term "column volumes per hour" is convenient because it can be extended to any column size and geometry. The flow rates in Figure 3 of 0.5, 1, 2, 3, and 4 column volumes/hour refer to throughputs of undiluted waste. The 1:1 dilution was actually fed to the columns at double flow rates; other data indicate that the curves would be very close to those shown had undiluted waste been used. Flow rates resulting in comparable curve slopes are somewhat lower than those used for formaldehyde-treated waste (FIW) (5) as would be expected because of the higher salt content of the supernatant waste.

The effect of temperature on sodium-cesium competition for exchanger sites has been studied in several experiments (6, 9). For the present study, only one elevated temperature column was run to verify previous work. This is shown in Figure 4 and re-emphasizes the fact that temperatures should be held as low as possible during column loading and washing. The temperature effect is essentially the same for all exchangers because it is a function of ionic hydration in solution and not related to properties of the exchange site.

For one AW-400 column experiment the diluted synthetic Purex alkaline supernatant waste was concentrated to a volume only slightly greater than that of the undiluted waste. (Precipitation occurred if evaporation was carried to the undiluted volume). For this experiment, the cesium breakthrough curve, column volumes of undiluted waste plotted vs.  $C/C_0$ , was identical with those for the 1:1 dilutions. Experiments with Duolite C-3 at several dilutions indicated no effect of influent dilution on column capacity. For clinoptilolite, early work indicated a sizable

Figure 3. Effect of Flow Rate on Cesium Breakthrough from AW-400

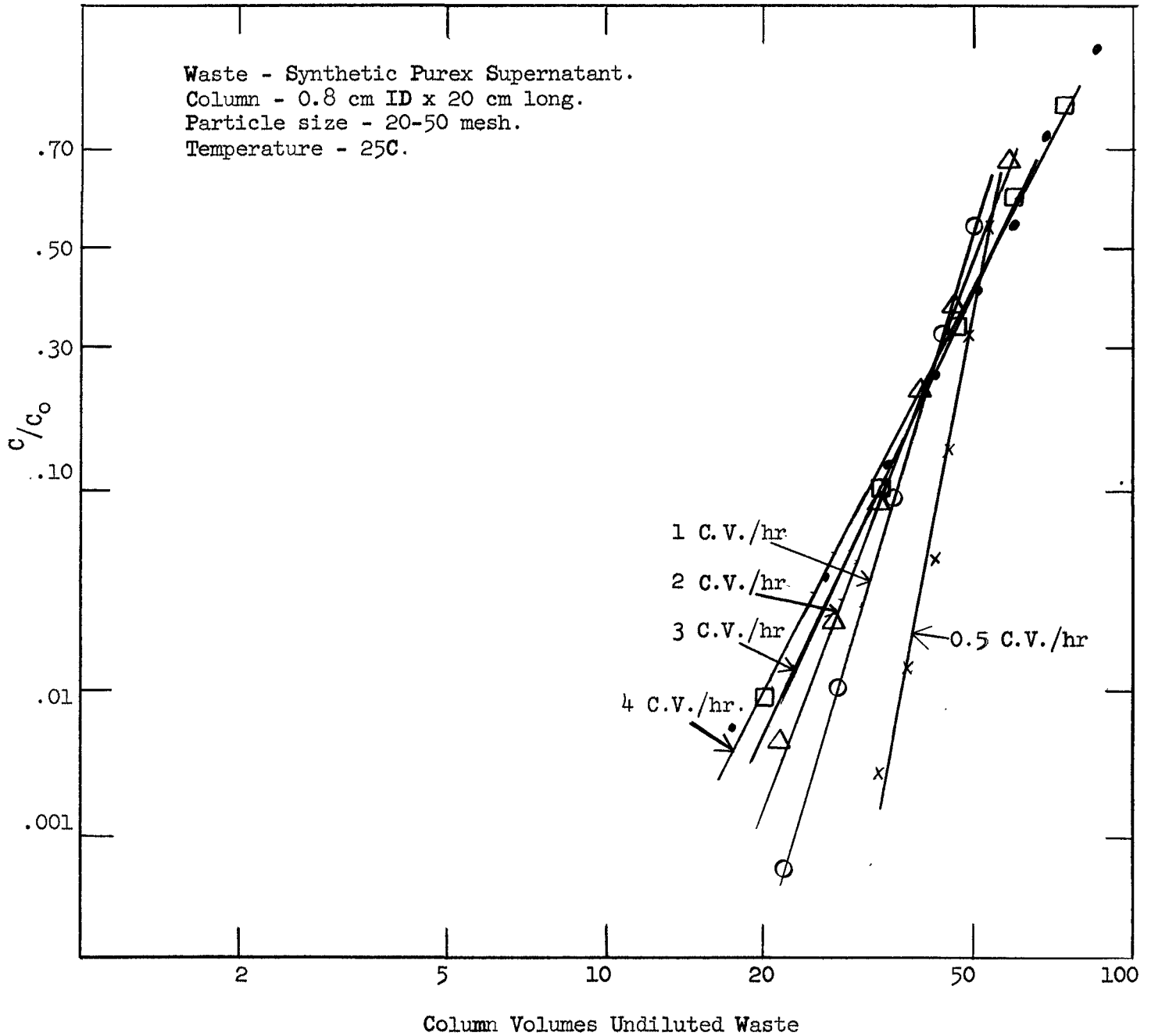
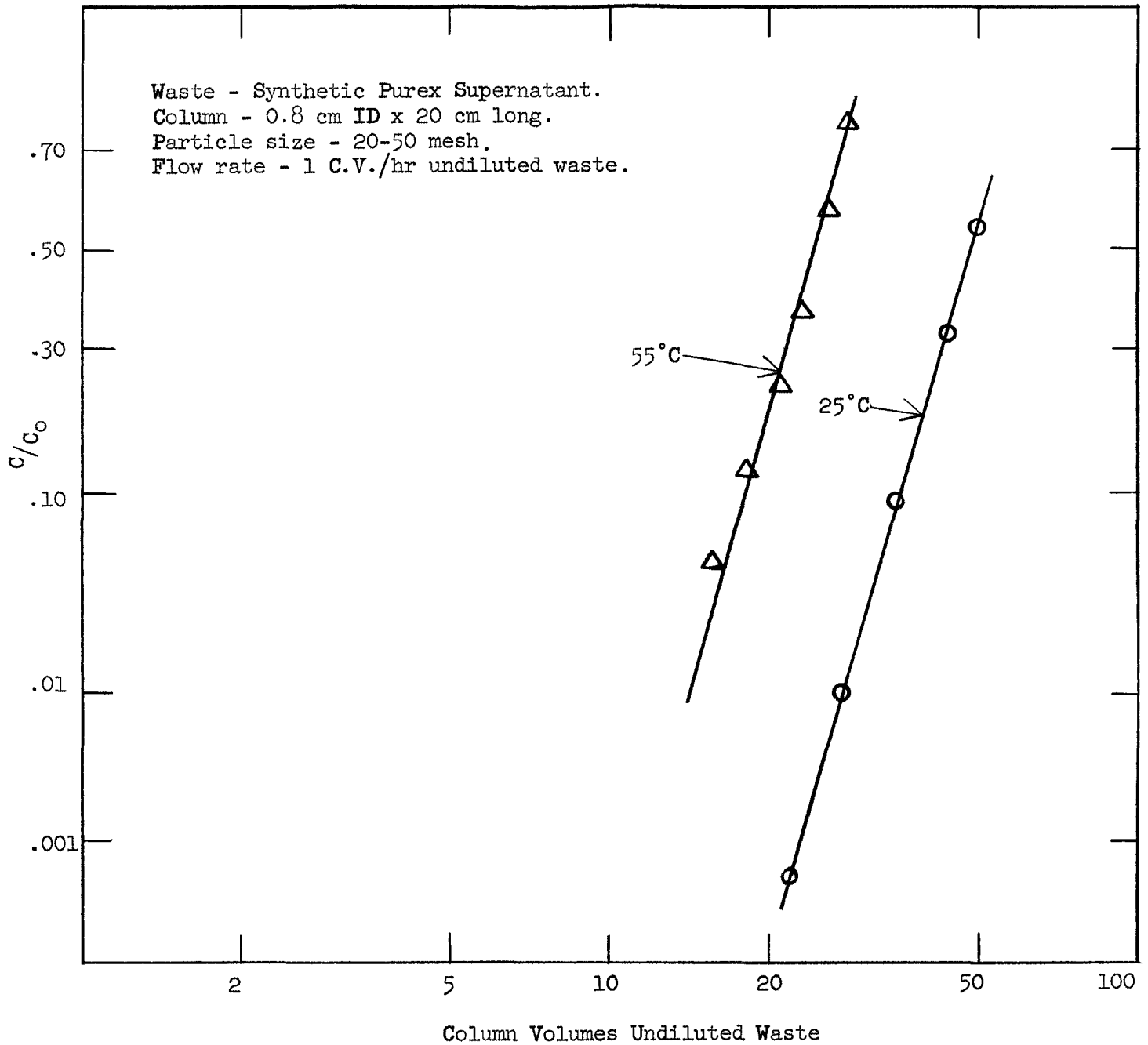


Figure 4. Effect of Loading Temperature on Cesium Breakthrough from AW-400



dilution effect on cesium loading (4). However, subsequent work (5, 9) has shown that if the throughput rate of undiluted waste remains the same for various dilutions, only a small cesium capacity increase is obtained with dilution. For applications where it is also desirable to remove strontium and other isotopes from the waste, dilution favors polyvalent ion sorption as would be predicted from mass action considerations.

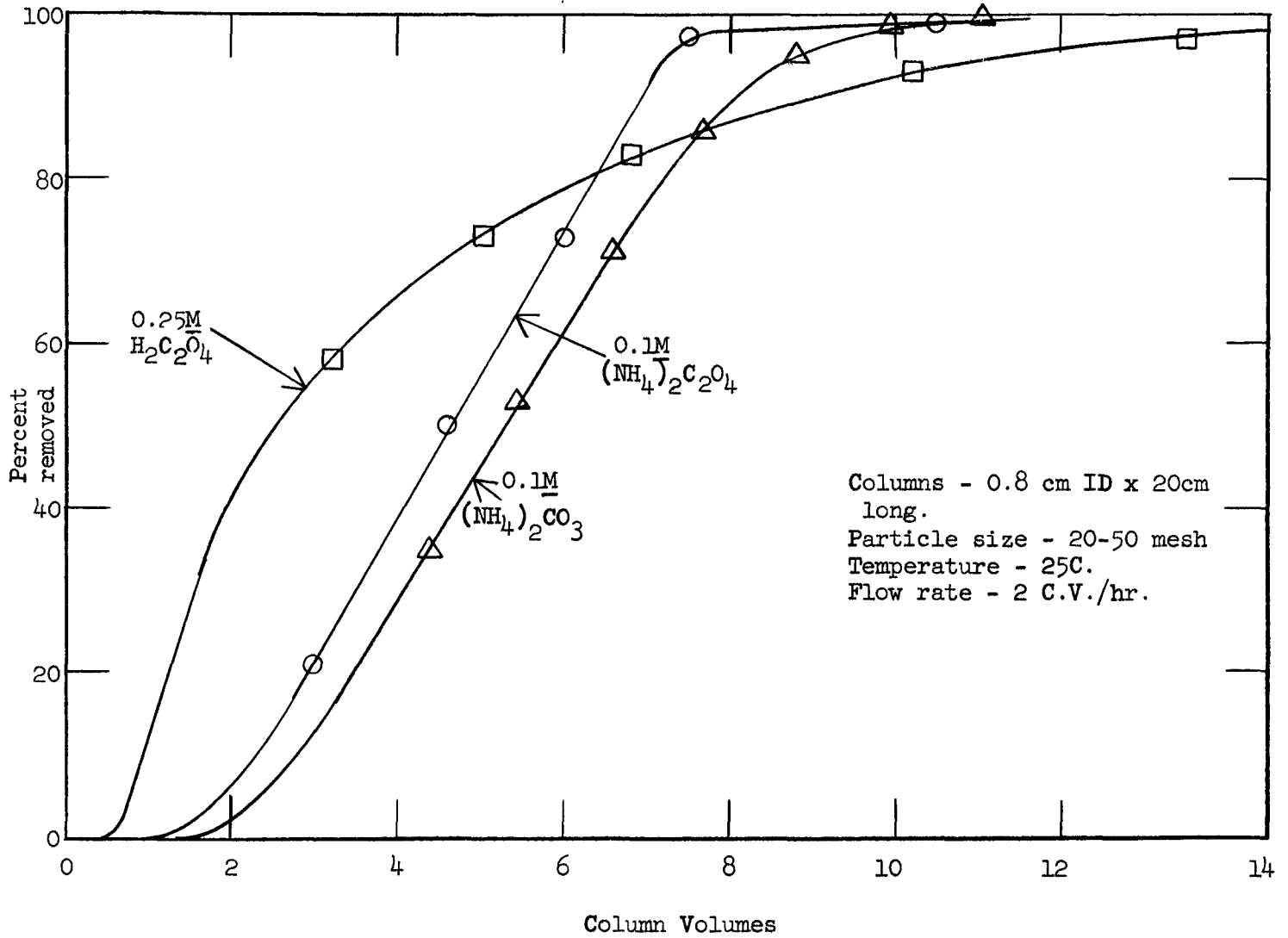
The uptake of several isotopes on AW-400 from 1:1 Purex alkaline supernate has been studied. Cesium, strontium, cerium, zirconium-niobium, and sodium equilibrium  $K_d$ 's are 91, 1.6, 4.4, 1.8, and 1.5 respectively. In addition most of the strontium, zirconium-niobium, and rare earths are in the sludge rather than in the supernate.

#### Wash and Scrub Studies

Data for washing, removing sodium and zirconium-niobium, and elution are essentially the same for loadings of cesium in any type of waste solution. Thus, data in a previous document on acid wastes is approximately applicable (5). Some additional data have been obtained since that document was written.

For flow sheets in which alkaline solutions such as Purex alkaline supernatant wastes are used, nonacidic solutions are advantageous for sodium removal. Two of these have been tried, dilute solutions of ammonium carbonate and of ammonium oxalate. Data on sodium removal from clinoptilolite by these chemicals and oxalic acid are shown in Figure 5. The dilute alkaline reagents are superior to the oxalic acid as they approach 100 per cent removal more rapidly and lower solution concentrations are used.

Figure 5. Removal of Sodium from Clinoptilolite with Various "Scrub" Solutions.



Data on sodium removal from AW-400 exchanger, including a higher concentration of ammonium carbonate are shown in Figure 6. For the higher concentrations it would probably be worthwhile to decrease the flow rate to avoid the slow approach to 100 percent removal.

Dilute ammonium carbonate would be the least costly solution for a flow sheet employing aged alkaline waste. However, if enough zirconium-niobium were present that removal was required, ammonium oxalate is capable of removing a part of it as shown in Figure 7. Only a part of the zirconium-niobium not removed by ammonium oxalate would be removed by the ammonium carbonate eluting solution. In the acid flow sheets (i.e., FTW) use of ammonium oxalate eliminates the need for the ammonium hydroxide wash which was to prevent gassing if ammonium carbonate were added to the acid mineral.

Figure 8 data show sodium removal with oxalic acid of three concentrations. Oxalic acid solutions would be useful in flow sheets with acid waste where a high degree of zirconium-niobium removal is necessary. The 0.25M concentration is probably adequate.

Cesium losses during sodium removal are dependent on the degree of cesium loading as discussed previously (5), and shown in Figure 9. Losses are not greatly different using the dilute alkaline scrub solutions than with nitric or oxalic acids. In a practical operation, loading would be stopped at less than one percent breakthrough so that cesium loss would be nil.

Sodium and zirconium-niobium removal data are quite similar for different exchangers. Clinoptilolite, AW-400, AW-500, and Zeolon exhibit only small differences so that data for one are at least approximately applicable to the others.



Figure 6. Removal of Sodium from AW-400 with Various "Scrub" Solutions.

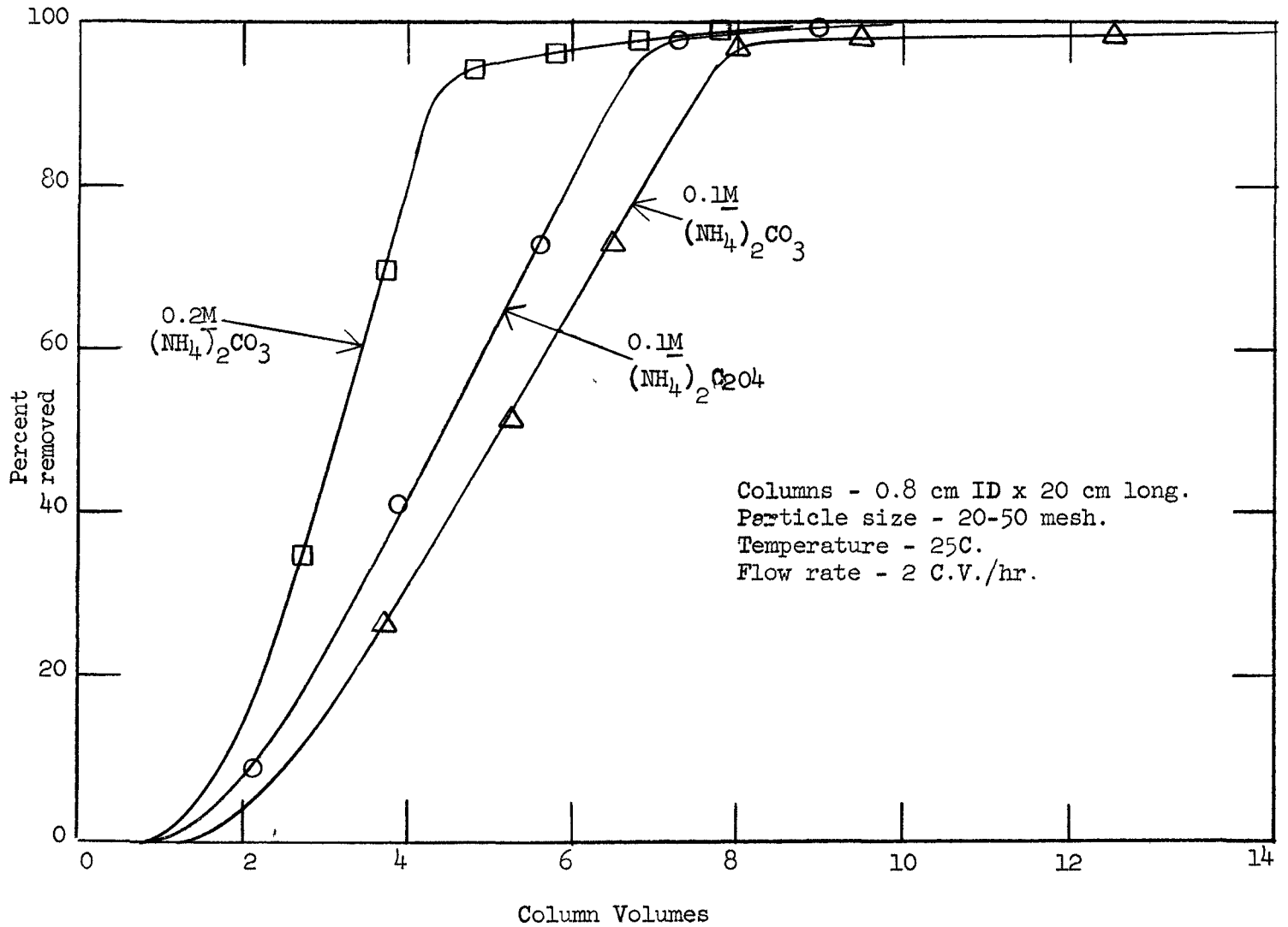


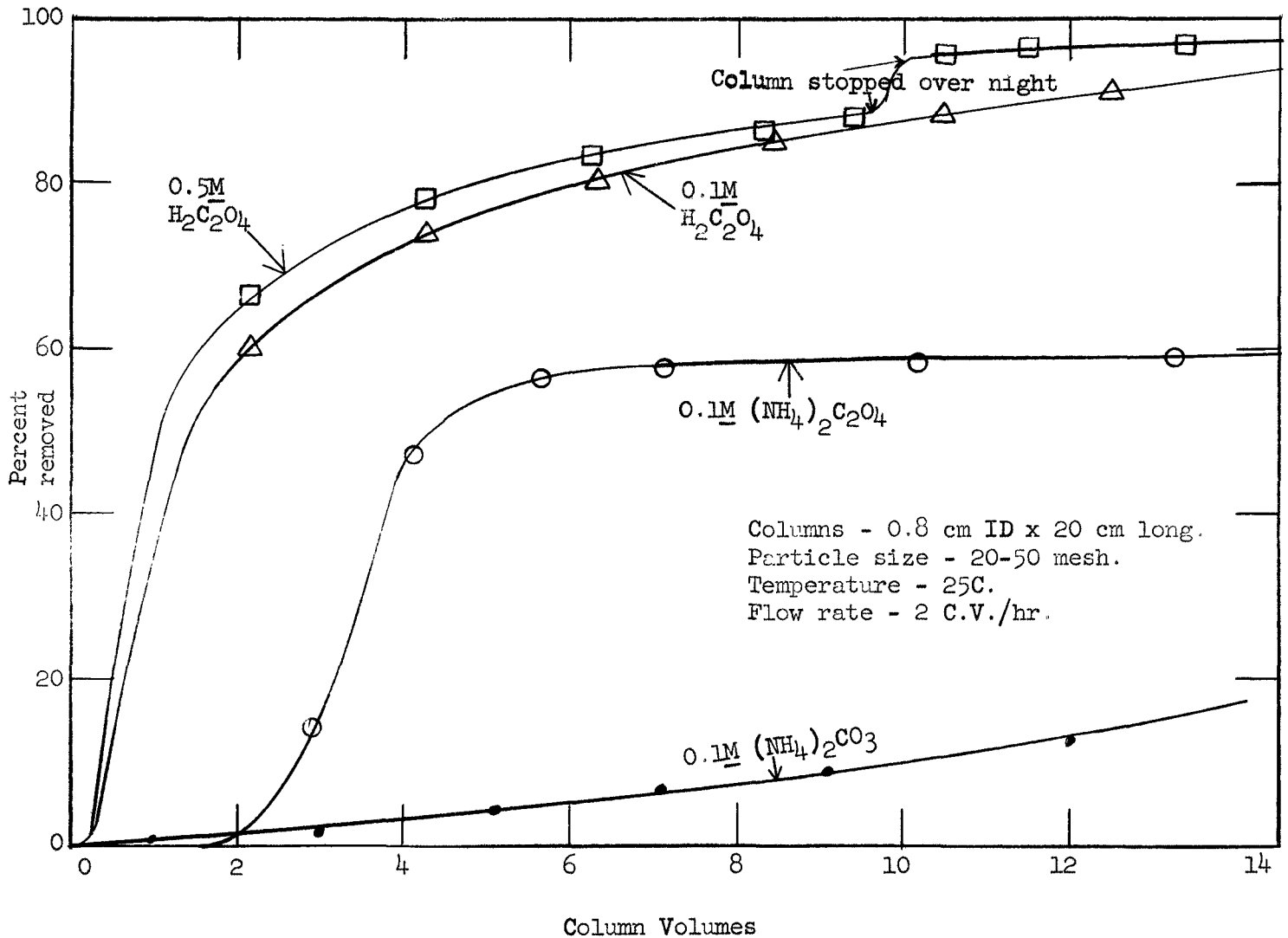
Figure 7. Removal of  $Zr^{95}$ - $Nb^{95}$  from Clinoptilolite by Four Solutions.

Figure 8. Removal of Sodium from Clinoptilolite with Various Concentrations of Oxalic Acid.

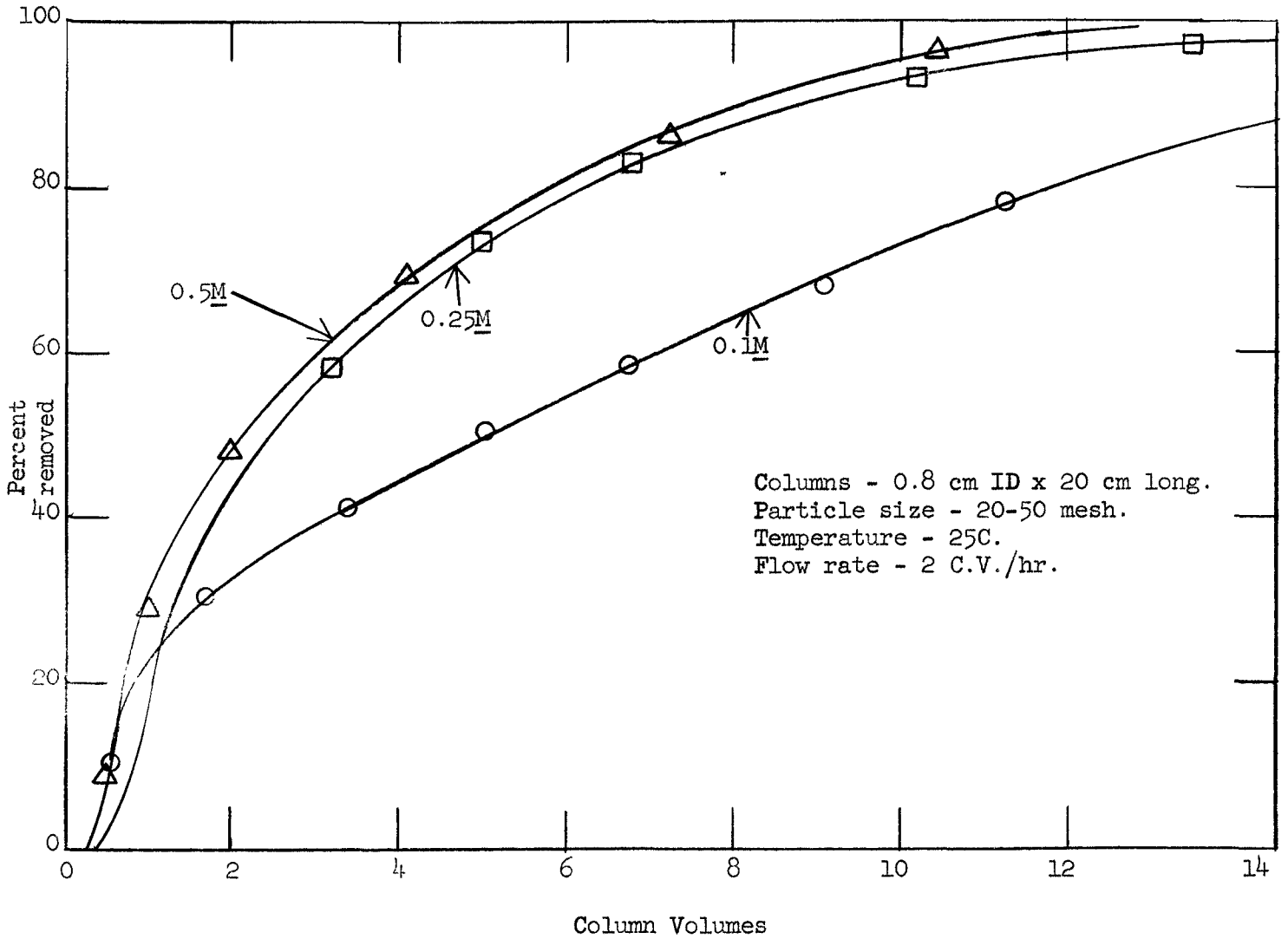
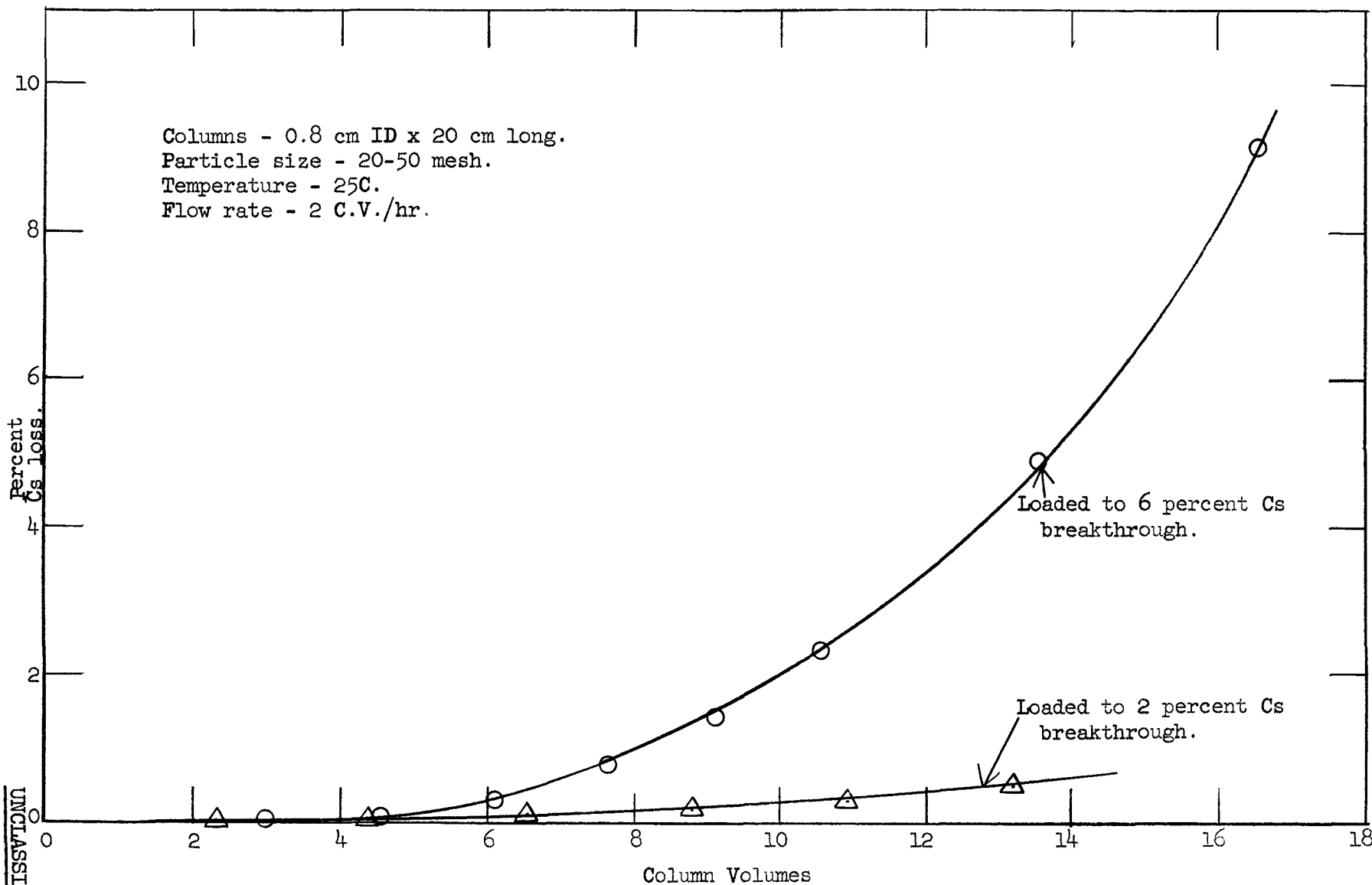


Figure 9. Cesium Loss from Clinoptilolite to 0.1M (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> Scrub Solution for Two Cesium Breakthrough Levels.



Elution Studies

Elution data previously reported (5) for clinoptilolite would be approximately applicable for an alkaline supernatant flow sheet. Comparison of elution of cesium from three exchangers is shown in Figure 10. Preliminary data indicate that elution of cesium from AW-500 is essentially the same as from AW-400. Both materials require considerably more elution volume than do clinoptilolite and Duolite C-3. There is a 4 to 8 percent fraction of the cesium that does not elute from AW-400 and AW-500 and is probably retained by the binder. This fraction does not appear to lower the capacity on subsequent loading cycles nor does it build up any further; therefore, for practical purposes it can be ignored. This fraction was not included in the calculations for the AW-400 curve in Figure 10. Elution of cesium from Zeolon is being investigated.

Cesium elution curves from clinoptilolite by three concentrations of elution solution are shown in Figure 11. The elution solution used is made up with 75 percent of the ammonium ions added as carbonate and 25 percent as hydroxide (5) As would be expected, the dilute solutions elute cesium more effectively/mole of ammonium. The advantage of lower volumes outweighs the loss in efficiency and thus the 8N solution is the choice eluting solution.

The effect of particle size and flow rate on cesium elution from Duolite C-3 is shown in Figure 12. It can be seen that the elution is limited by kinetics and is considerably improved by decreased particle sizes and flow rates. The effect of flow rate on the cesium elution from clinoptilolite is shown in Figure 13. For this exchanger elution flow rate would appear to have no effect to 4 column

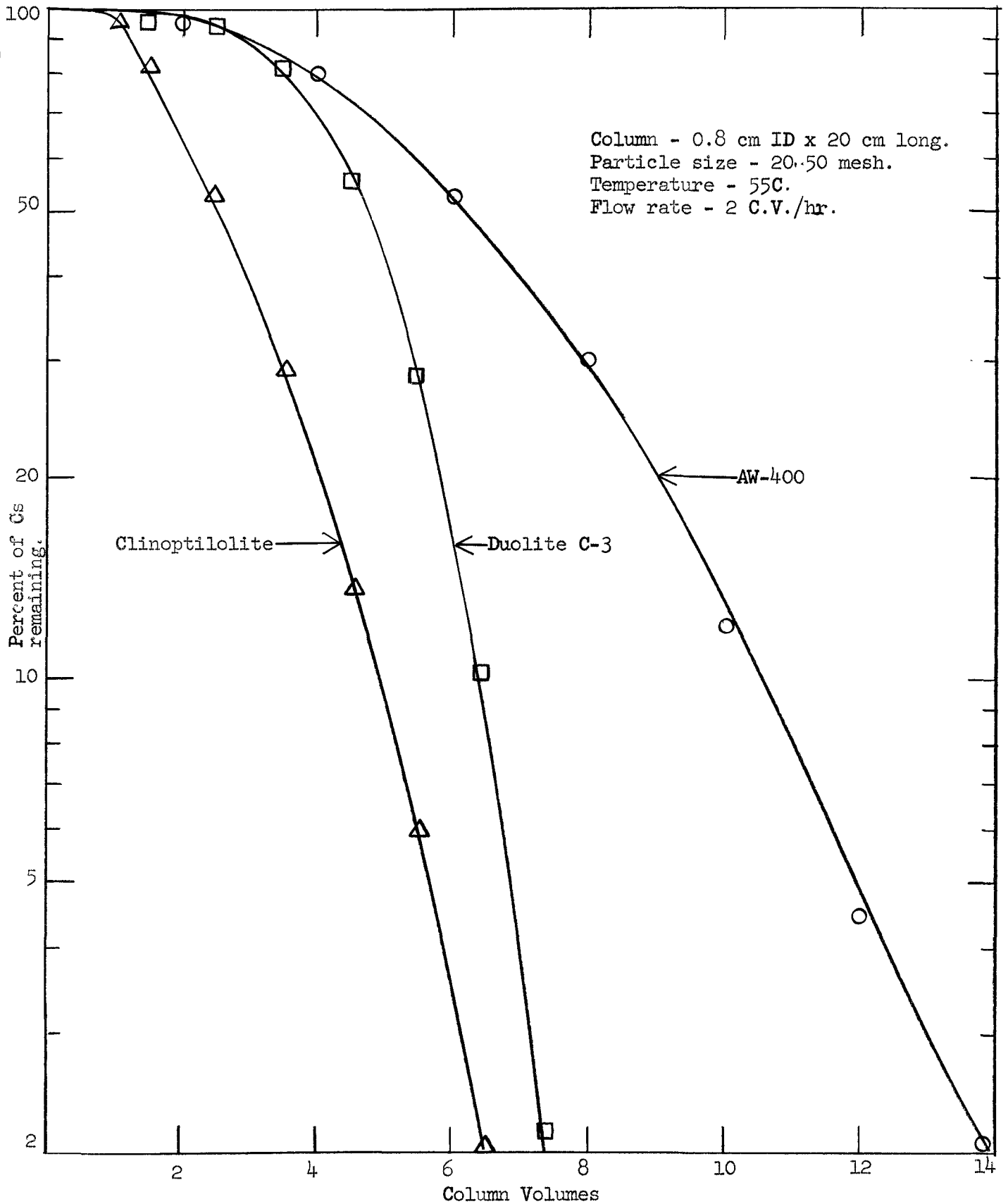


Figure 10. Elution of Cesium from Three Exchangers with  $2M (NH_4)_2CO_3$ .

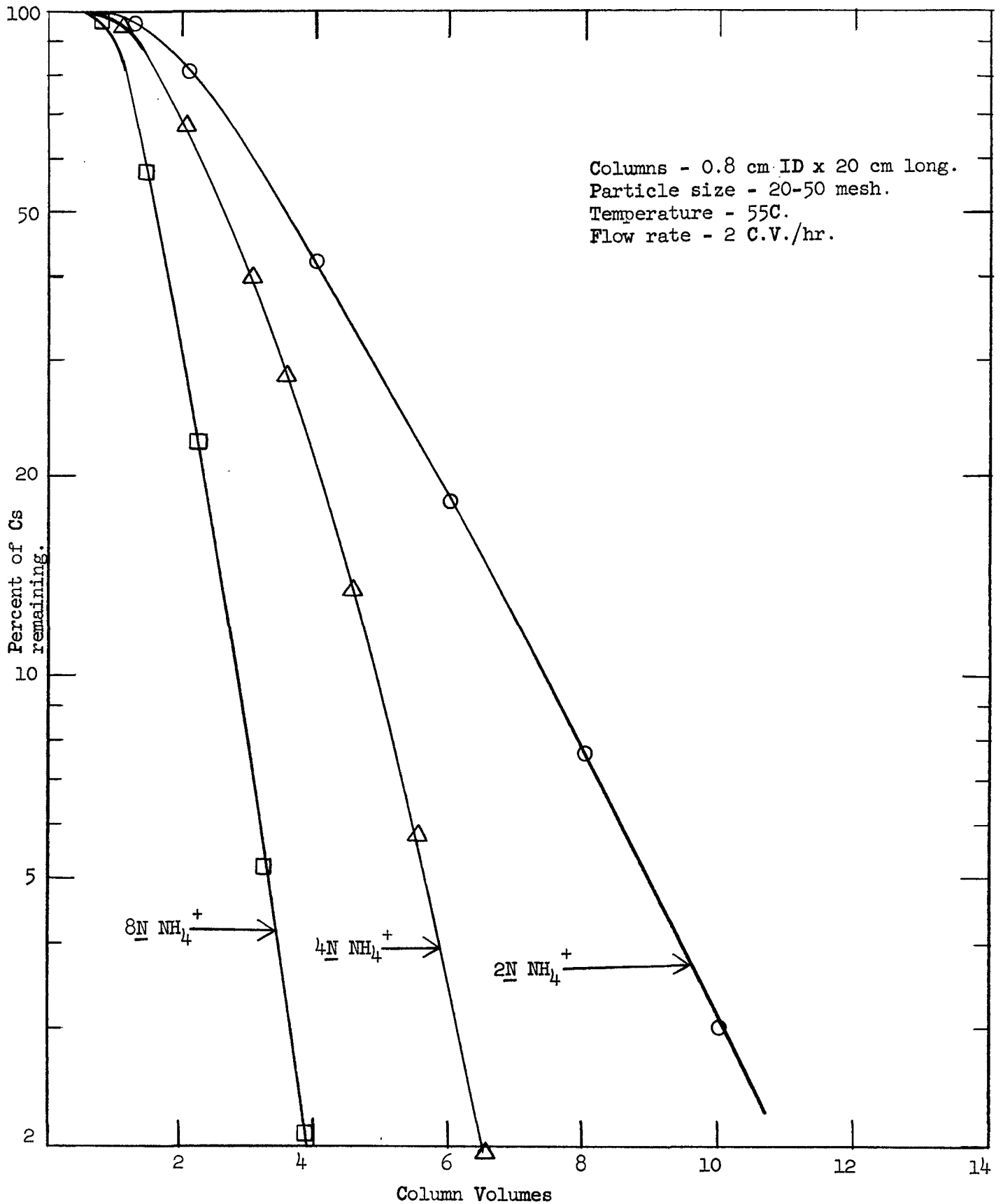


Figure 11. Elution of Cesium from Clinoptilolite by Three Concentrations of  $(\text{NH}_4)_2\text{CO}_3 - \text{NH}_4\text{OH}$  Solutions.

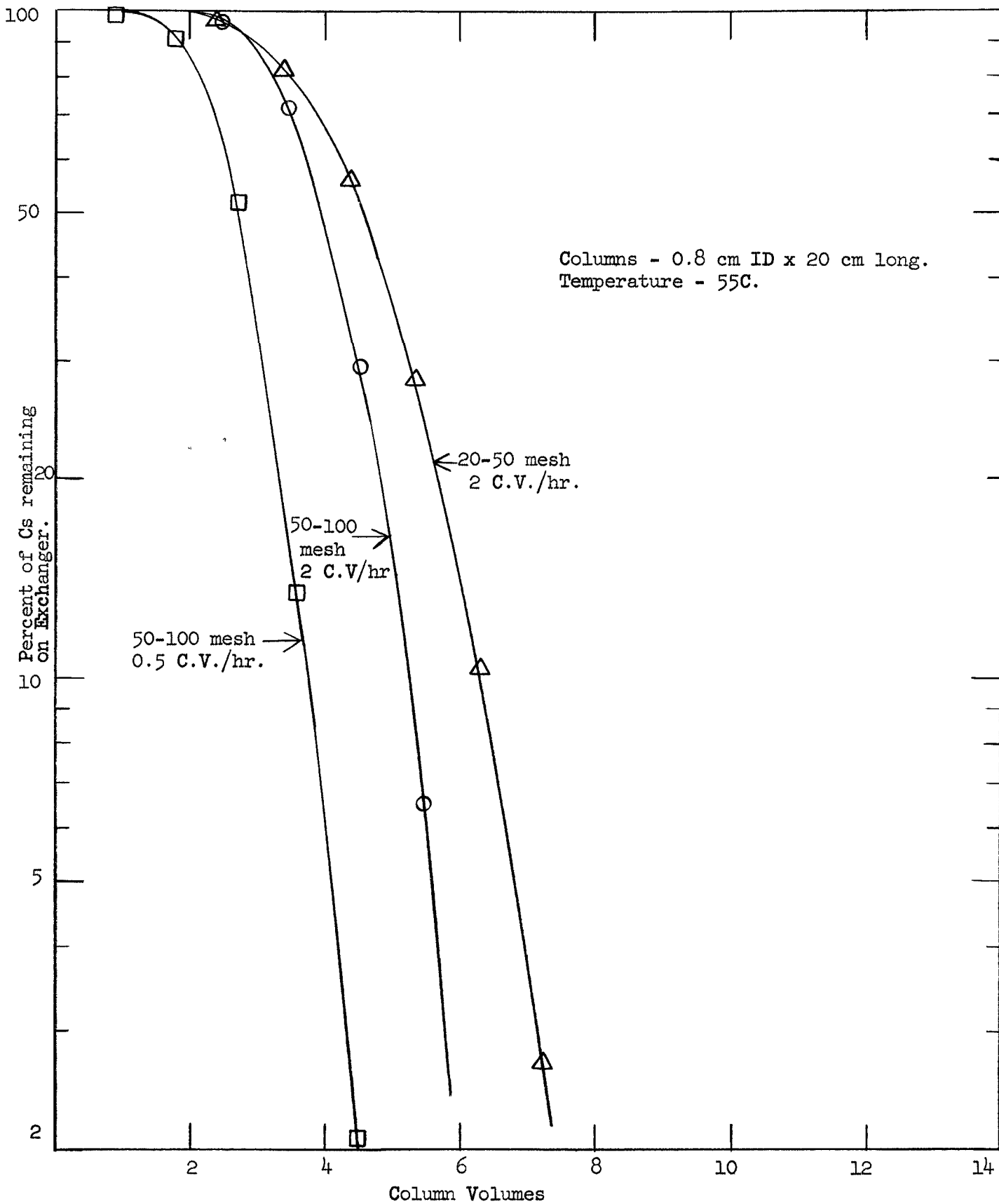


Figure 12. Elution of Cesium from Duolite C-3 with  $2M (NH_4)_2CO_3$ .



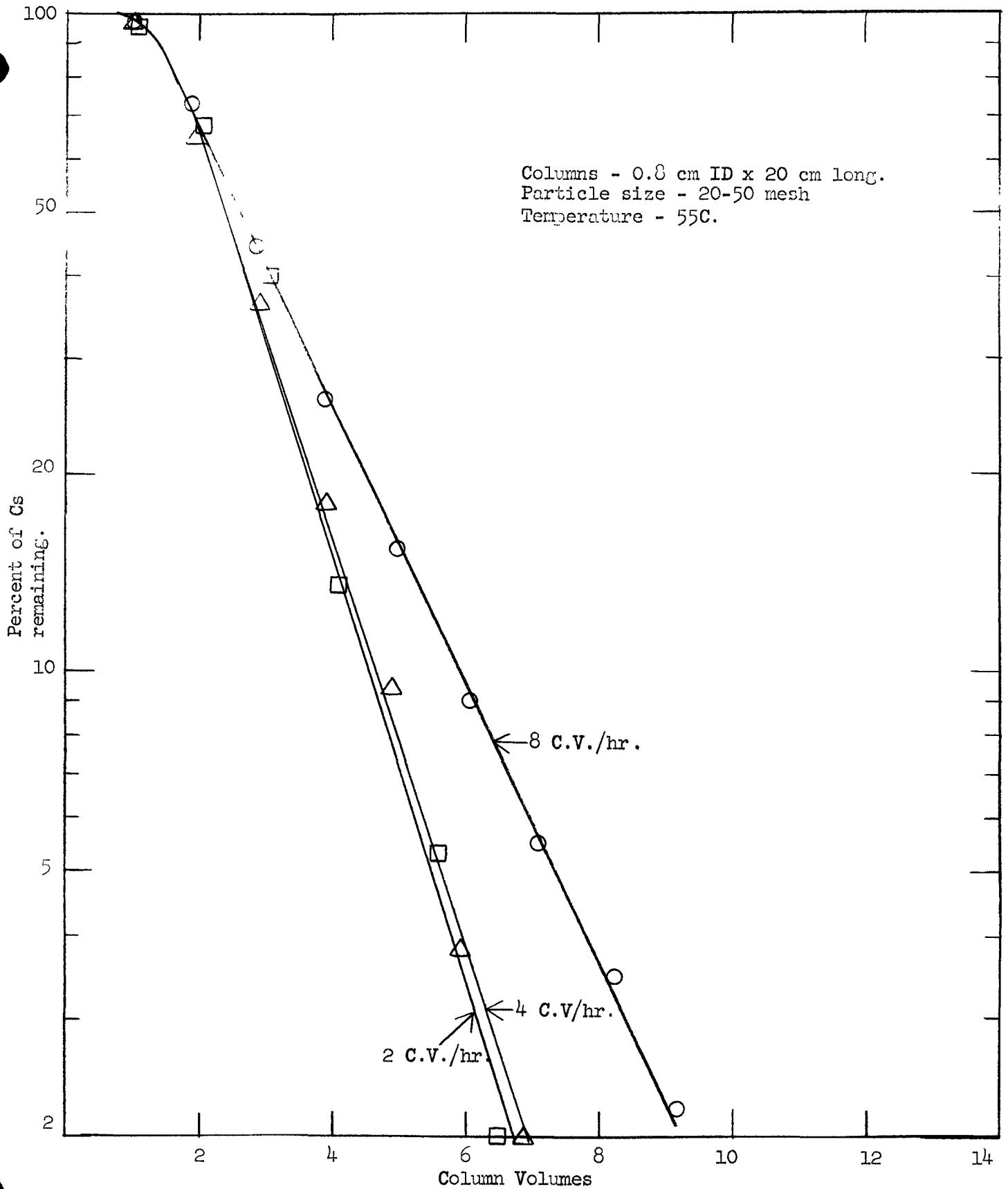


Figure 13. Effect of Flow Rate on Elution of Cesium from Clinoptilolite with 4N (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> Solution.

volumes per hour and only a small effect to 8 column volumes per hour. Similar data for AW-400 are not yet complete.

### Hot Cell Experiments

#### (1) Clinoptilolite

A hot cell experiment was run in A-cell of the 325-A High-Level Radiochemistry facility using actual Purex alkaline supernatant waste and a column filled with 20-50 mesh clinoptilolite. The column was two inches in diameter and filled to the four-foot level with the mineral. The column was filled by slurrying the clinoptilolite through a 3/8-inch line into the column. The influent used was 1:1 dilution of waste taken from the Purex 103-A tank. Flow rate was maintained at 1.3 ml/min/cm<sup>2</sup> (0.63 column volumes/hour) and the temperature was controlled at 20C by a water jacket. The column run proceeded smoothly with no measurable pressure drop across the column.

Cesium breakthrough was first detected at 14 column volumes of undiluted waste (1 percent breakthrough) and reached 50 percent breakthrough at 18 column volumes. These data indicate a considerably steeper breakthrough curve than found in laboratory work, and the volume to 50 percent breakthrough was about ten percent lower. This difference was probably caused by a lesser degree of packing in the hot cell column, resulting in less total weight of mineral/unit volume than the laboratory column. The porosity, however, would be higher, resulting in a longer residence time. It is believed that the results of the hot cell column corroborate laboratory data very well.

The column in the hot cell was washed with water and then eluted with 5M  $\text{NH}_4\text{NO}_3$  at 60C. The peak concentration of cesium in the eluate was nearly 100 times that of the influent and more than 99 percent of the cesium was eluted in a three-column volume fraction which contained 30 times the cesium concentration of the influent.

Analyses for other isotopes indicated that concentration of cesium in the eluate was at least 1000-fold higher than any other radionuclide. The waste used was about four years old.

## (2) Duolite C-3

Two hot cell experiments were run in the same facility, using 20-100 mesh Duolite C-3 phenolic resin and actual Purex 103-A tank waste.

In the first experiment the waste was used with no pretreatment. The column was two inches in diameter and seven feet long and the flow rate was 4 ml/min/cm<sup>2</sup>, (1.14 column volumes per hour). A cesium breakthrough of 50 percent was obtained at 13 column volumes of actual waste and 75 percent breakthrough at 18 column volumes when the experiment was stopped. A cesium D F across the column of > 500 was obtained for the sample taken at 7 column volumes. The volume to cesium breakthrough was greater with this actual waste than for the simulated waste (Figure 1). This difference could easily have been due to small differences in buffer capacity between the synthetic and the actual waste.

Elution of cesium from the Duolite C-3 resin was accomplished with 1M  $\text{HNO}_3$  after washing with water and a small volume of 0.25M  $\text{HNO}_3$ . The elution required nearly 20 column volumes of the 1M  $\text{HNO}_3$  and proceeded smoothly. However, upon

subsequent water washing, gassing was observed and the column plugged. This was probably due to some attack on the resin by the nitric acid.

In the second hot-cell experiment with Duolite C-3, the waste was butted to 2M NaOH, diluted slightly to prevent precipitation, and run in a similar manner to the first column. A 10 percent cesium breakthrough was reached at 17 column volumes of actual waste and 50 per cent breakthrough at 24 to 25 column volumes. These results checked well with laboratory data (Figure 1).

The column was washed with water and with 0.125M  $H_2SO_4$ , and then the cesium was eluted with 1M  $H_2SO_4$ . About 15 column volumes were required for elution. No attack of the resin was discernible with the sulfuric acid.

### (3) AW-400

A column experiment was conducted in the 222-U shielded hood facility, using AW-400 and actual Purex 103-A tank waste. The column measured 1 cm in diameter and 14.8 cm in length and contained 9.4g of 14-30 mesh AW-400. Waste was pumped through the column at a flow rate of 1.53 column volumes/hour. Because the waste was diluted slightly in transferring and storage, the flow rate is equivalent to 1.0 column volume / hour of undiluted waste.

Cesium breakthrough to 0.5 percent was reached at 24.3 column volumes of undiluted waste at which time the loading cycle was terminated. Breakthrough curves for the principal radioactive fission products, are shown in Figure 14. The cesium breakthrough compares favorably with simulated waste experiments.

Upon completion of the loading cycle the column was washed with 4 column volumes of water. Eight column volumes of a 0.1M  $(NH_4)_2CO_3$  scrub solution were

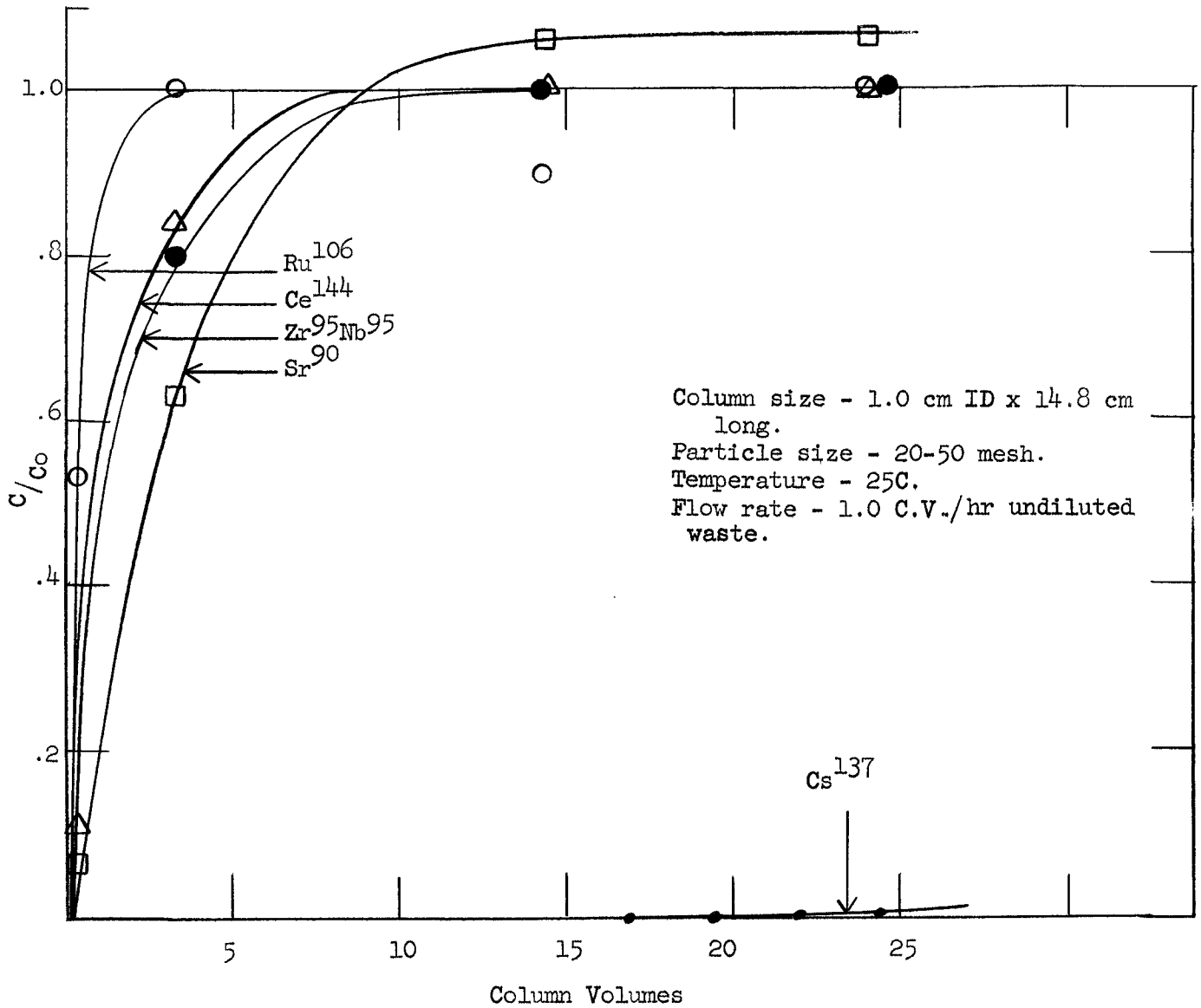


Figure 13. Fission Product Breakthrough Curves for AW-400 Using Actual 103-A Tank Supernatant Waste.

pumped through the column to elute sodium from the exchanger. The cesium was then eluted with 24.8 column volumes of  $3N(NH_4)_2CO_3 \cdot 1N NH_4OH$ . The large elution volume was necessary to assure elution of cesium since equipment failure allowed the temperature to fall below 55C during the initial part of the elution cycle.

Radiochemical analyses of the waste feed solution, scrub, and eluate are shown in Table II. Decontamination factors from cesium are also recorded.

TABLE II

Concentration of Fission Products in Purex 103-A Tank Waste Feed, Scrub and Eluate

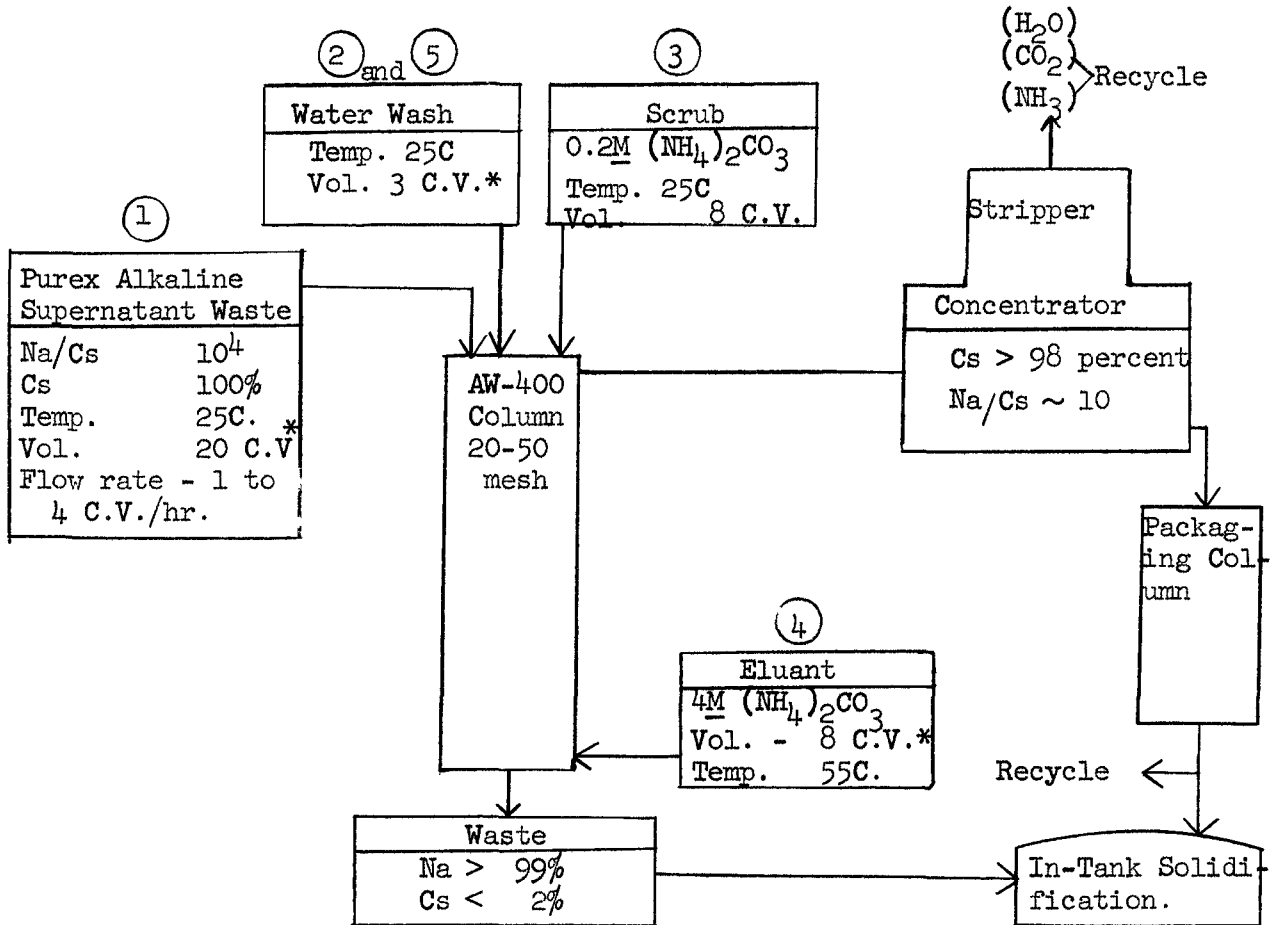
<u>Isotope</u>	<u>Feed</u> mc/l	<u>Scrub</u> mc/l	<u>Eluate</u> mc/l	<u>DF from Cesium</u>
Cs <sup>137</sup>	1800	0.64	2700	--
Ce <sup>144</sup>	360	3.4	24	22
Sr <sup>89, 90</sup>	36	0.64	1.1	49
Ru <sup>106</sup>	18	0.24	0.047	590
Zr <sup>95</sup>	22	0.48	0.56	59
Nb <sup>95</sup>	49	2.0	3.9	19

DISCUSSION

Based on the data presented in this document, a flow sheet has been designed for separation of cesium from a Purex alkaline supernatant waste. This flow sheet is shown in Figure 15. Similar flow sheets can be designed to fit other specific processing conditions.

The AW-400 synthetic zeolite is a leading candidate for the exchanger to remove cesium from alkaline wastes. However, it is still an experimental product of the Linde Co. and may not be put on the open market. The binder used is also poor from

Figure 15. Flow Sheet for Separation of Cesium from Purex Alkaline Supernatant Waste by Ion Exchange



\*C.V. - Column Volumes

several standpoints. Efforts are being made to obtain this exchanger bonded with known superior binders. The next choice is AW-500 which is commercially available, but which has the same binder as AW-400. Duolite C-3 resin would be the next choice; it has the advantage of good elution and ready availability, but the disadvantages of low capacity, poor kinetics in large particle sizes and a requirement of butting the waste with caustic.

The water wash following loading may not be necessary for this waste but is inexpensive and actually removes over half of the sodium from the bed, lightening the load on the scrub solution. A water wash between elution and reloading is desirable to remove excess ammonium ions from the pores of the exchanger and to prevent the loss of cesium to waste when elution is not carried to completion.

The amount of "scrub" solution necessary is a function of the sodium DF needed. As evidenced from Figure 6, about 5 column volumes of 0.02M  $(\text{NH}_4)_2\text{CO}_3$  would be sufficient to remove 95 percent of the sodium on the bed. This yields a final Na/Cs ratio of 20 to 40. On the other hand, if 10 or 12 column volumes of scrub are used Na/Cs ratios of 3 to 8 can be achieved.

The amount of elution solution used can also be varied. If 6 column volumes are used, the loading cycle must be shortened somewhat to make up for the cesium that is not eluted. Cesium losses to waste are minimized by elution with 8 to 10 column volumes. If elution is carried far enough it is not necessary to use reverse flow during elution.

Seven and thirteen loading and eluting cycles have been accomplished with AW-400 and AW-500, respectively, using synthetic waste with a flow sheet similar



to that shown in Figure 16. A slight dissolution of iron from the binders and a very small decrease in cesium loading over many cycles are the only observed effects. The physical appearance after the recycling was not altered. Strong iron complexing agents such as oxalic acid cannot be used for an extended period with AW-400 and AW-500 as they attack the binder. Efforts are being made to improve the binding materials.

ACKNOWLEDGMENT

The cooperation and help of E. J. Wheelwright, L. A. Bray, and R. L. Moore in the hot cell experiments is gratefully acknowledged. D. E. Clark and R. G. Henderson assisted with the laboratory experiments.

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