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LOADING AND ELUTION CHARACTERISTICS OF SOME NATURAL AND
SYNTHETIC ZEOLITES

L. L. Ames, Jr. and K. C. Knoll

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LOADING AND ELUTION CHARACTERISTICS OF SOME NATURAL AND
SYNTHETIC ZEOLITES

L. L. Ames and K. C. Knoll

INTRODUCTION

The zeolites are a family of well defined, hydrated aluminosilicates closely related to each other in chemical composition (4). Alkali or alkaline earth metal cations are necessary to electrostatically balance $(AlO_4)^{-5}$ substitutions for $(SiO_4)^{-4}$ tetrahedra within the zeolite crystal lattice, and are stoichiometrically exchangeable for other cations with little or no lattice expansion or contraction.

There are at least three mechanisms responsible for the ion exchange characteristics of zeolites. They include cation size-crystal lattice interactions, charge distribution interactions and cation-anionic site-water interactions within the zeolite crystal lattice (2). Cation size requirements were minimized as a mechanism by choosing zeolites whose effective pore diameter allow exchange of all cations used in this study. Often the larger cations can be excluded from exchange sites due to their inability to diffuse through the zeolite crystal lattice and into the exchange sites.

A second mechanism of charge distribution is due to the rigid spatial distribution of anionic sites on the crystal lattice. Often high field strength or highly charged cations cannot effectively electrostatically neutralize the anionic charges. The net result can be exclusion of the rare earths, for example, from exchange sites even though diffusion through the zeolite crystal lattice is possible.

The third mechanism involves the electrostatic interaction of incoming cation, internal or zeolitic water, and anionic site within the zeolite lattice. Lower field

strength cations attract water within the zeolite lattice to a lesser extent, can approach the anionic site more closely, and are therefore preferentially held on anionic exchange sites.

In many cases, zeolites are able to remove small quantities of radioisotopes from macroconcentrations of other cations (5), because of the above three sieve mechanisms. Zeolites also exhibit high radiation (9) and thermal (7) stabilities, enabling them to function as storage media from which the adsorbed radionuclides can subsequently be eluted. Prior to storage, the loaded zeolite column must be dried by heating (13). During storage, the column will be subjected to heat generated by radioactive decay. Thus the zeolite must not allow a significant release of radioactivity during drying, and yet must permit the elution of adsorbed radioisotopes after heating.

OBJECT

The purpose of this document is to furnish equilibrium zeolite loading results and elution data from loaded and heated zeolite columns to facilitate choice of an appropriate zeolite to fill a given application.

SUMMARY

The cation exchange characteristics of several commercially available zeolites were evaluated. Equilibrium loading characteristics and exchange capacities were determined experimentally in the binary systems cesium-lithium, cesium-sodium, cesium-potassium, cesium-rubidium, cesium-hydrogen, cesium-ammonium, strontium-

magnesium, strontium-calcium, strontium-barium and strontium-hydrogen for the zeolites Linde AW-300, Linde AW-400, Linde AW-500, Linde 4AXW, Linde 13X, Norton Zeolon and clinoptilolite.

Exchangeable cation fractions in the equilibrium solutions were varied from 0.2 to 0.00001, while the total normality of the system constituents was held constant. Results were plotted as equivalent fraction of the traced cation on the zeolite at equilibrium vs. the fraction of traced cation remaining in the equilibrium solution. If an influent composition is known, and falls in the systems listed above, zeolite column loading may be read directly in univalent-univalent or divalent-divalent systems. However, in the strontium-sodium or strontium-hydrogen systems, a k value must be used because squared terms appear in the equilibrium expression as follows:

$$2 \text{ Na-zeolite} + \text{Sr-solution} \rightleftharpoons 2 \text{ Na-solution} + \text{Sr-zeolite},$$

or,

$$k = \frac{(\text{Sr-zeolite})(\text{Na-solution})^2}{(\text{Na-zeolite})^2(\text{Sr-solution})}$$

The k for a given cation is a function

of zeolite loading with that cation, i.e., the greater the strontium to sodium ratio of the influent solution, the smaller the k value. Several column 50 per cent loading capacities were determined to randomly test the validity of the above curves to predict column capacities.

Several columns loaded with known amounts of cesium and strontium radioisotopes were heated to 600 C for 24 hours to simulate a zeolite packaging dehydration cycle. Losses of cesium and strontium activity during the 24 period were determined. Elution of the remaining activity on the columns was accomplished with various eluting agents including dilute HNO_3 , NH_4NO_3 , CaCl_2 and $\text{La}(\text{NO}_3)_3$, and elution curves calculated for Linde AW-500, Linde 13X, Linde 4A, Norton Zeolon and Decalso.

METHODS OF INVESTIGATION

The clinoptilolite used in this study was obtained from the Hector, California deposits of the Baroid Division of National Lead. The as-received clinoptilolite ore consisted of 85 to 95 per cent by weight pure clinoptilolite, and 5 to 15 per cent by weight unaltered volcanic glass plus quartz and feldspar along with lesser amounts of calcite and montmorillonitic clays (1). Carbonates and clays in the clinoptilolite were removed or destroyed by a ten per cent nitric acid wash prior to use.

Several synthetic zeolites as one-sixteenth inch diameter, sodium-based pellets were supplied by the Linde Company of Tonawanda, New York, including 13X, 4AXW, AW-300, AW-400 and AW-500. The Norton Company of Worcester, Massachusetts, supplied one-eighths inch, hydrogen-based Zeolon pellets. Table I gives the silica to alumina ratio of the above zeolites along with their structural types, weight per cent of binder and effective pore diameter at 25C for the sodium form. Effective pore diameter refers to the apparent size of the smallest zeolite crystal lattice channel through which the cations must diffuse in going to and from anionic exchange sites.

TABLE IZeolite Properties (6,7,8)

<u>Zeolite</u>	<u>SiO₂/Al₂O₃</u>	<u>Per cent binder</u>	<u>Structural type</u>	<u>Effective pore diameter, Å</u>
Linde 4AXW	2	8	(no natural counterpart)	4
Linde 4A	2	20	" " " "	4
Linde 13X	2.8	20	Faujasite	7.4
Linde AW-300	9-10	20	Mordenite	4
Linde AW-400	6-7	20	Erionite	4
Linde AW-500	4-5	20	Chabazite	4-5
Norton Zeolon	10	unknown	Mordenite	9-10
Clinoptilolite	8-10	5-15	Clinoptilolite	> 4

Cation exchange capacities were determined by a double tracing technique. Weighed, sodium-based zeolite samples were contacted with a solution containing 0.1N CsCl plus 0.1N NaCl plus Cs¹³⁴ to determine cesium removal at 27C. Cesium-based zeolites were then contacted with a solution containing 0.1N CsCl plus 0.1N NaCl plus Na²² to determine sodium removal. The solution to zeolite ratios were adjusted to yield reliable Cs¹³⁴ and Na²² counting rates between original and final contacting solutions. All zeolite samples were weighed after being water-saturated and oven-dried at 55C. At least two days of contact time with shaking were allowed to attain zeolite-solution equilibrium. Other results were obtained in a similar manner.

With the cesium-sodium system, for example, two grams of sodium-based zeolite were contacted with 80 ml of solution at 27C. The ratios of cesium to cesium-plus-sodium in the contacting solutions were varied from 1×10^{-1} to 5×10^{-5} , while the total solution normality was held constant. Four to six zeolite samples with different cesium to cesium-plus-sodium ratios, in duplicate, were shaken for three days, centrifuged and counted to determine the amount of cesium remaining in the equilibrium solution. Using the zeolite capacity, the equivalent fraction of cesium on the zeolite could then be computed by assuming that the cesium removed from the equilibrium solution was adsorbed by the zeolite.

Results were plotted as equivalent fraction of cesium on the zeolite vs. equivalent fraction of cesium in the equilibrium solution. This type of plot allows the reader to determine zeolite column loading directly in univalent-univalent or divalent-divalent systems, if the cesium to cesium plus sodium ratio of the influent solution is known. The influent solution in the column loading system is synonymous with the

same composition of equilibrium solution in an equilibrium system. Zeolite loads in univalent-divalent systems cannot be directly determined from the equilibrium data (See Figures 11 and 12). The use of a k value (14) is required. A k is defined, in a strontium-sodium system, for example, as

$$k = \frac{\left(\frac{Z_{\text{meq Sr}}}{\text{meq (Sr + Na)}} \right) \left(\frac{S_{\text{meq Na}}}{\text{meq (Na + Sr)}} \right)^2}{\left(\frac{Z_{\text{meq Na}}}{\text{meq (Na + Sr)}} \right)^2 \left(\frac{S_{\text{meq Sr}}}{\text{meq (Sr + Na)}} \right)}$$

where Z is the zeolite and S is the equilibrium solution.

Equilibrium distributions were determined at total normalities of 1.0 and 0.1 for the systems cesium-sodium, cesium-hydrogen and strontium-sodium to confirm that total system normality had little or no effect on equilibrium distributions over this range. However, several systems, such as cesium-lithium, show considerable divergence above a normality of one (10). The reader should take care when extrapolating the data in this report to greater than a total system normality of one.

Zeolites used in the equilibrium experiments were based with saturated chloride solutions of the desired cation. HNO_3 was used in ten per cent solution to hydrogen base clinoptilolite, Zeolon and Linde AW-300. Fifty gram portions were placed in 400 ml beakers and appropriate chloride solutions added. The contacting chloride solutions were changed six times at 45-minute intervals. A seventh and final solution was contacted for two days. The variously-based zeolites were then thoroughly washed until chloride could not be detected in the wash water with AgNO_3 solution.

After loading, all zeolites were checked by X-ray diffraction to assure that no major structural changes had occurred. Only Linde 4A in the strontium-barium system

was found to have altered to a barium analog (7).

Kinetic loading curves were obtained by continuously recording the loading of a zeolite shallow bed with the apparatus shown in Figure 15 (3).

Vycor columns were prepared for the heating and elution studies from 9 mm diameter tubing approximately 11 cm in length. One end of the column terminated in a ground glass joint and the other end was sealed (see Figure 19). The columns contained three grams of clinoptilolite or two and one-half grams of the other zeolites. The columns were loaded with a 0.1N cesium or strontium solution traced with the appropriate radioisotope, washed with water and placed in a tube furnace at 600C for 24 hours. A scrubber train was attached during heating to determine heating losses. The three stages of the scrubber were sodium hydroxide solution, nitric acid and a C.W.S. filter. The sum of the radioactivity in the scrubbers was measured and assumed to represent the total activity released during the 24 hours of heating.

After cooling, the column was eluted by one of several methods and the recovered activity measured. Column eluting agents included HNO_3 , NH_4NO_3 , CuCl_2 and $\text{La}(\text{NO}_3)_3$. Hydrogen and cupric cations were chosen for trial as eluting agents because of favorable equilibrium distribution coefficients in strontium systems. Ammonium cations could be easily separated from cesium, and were tested for that reason. Lanthanum, as a trivalent cation, should easily elute cesium from Decalso if the cesium were not chemically combined with the Decalso during the heating process. The sum of the radioactivity remaining on the column, eluted from the column and on the scrubber train represented the total column load.

RESULTS AND DISCUSSION

Equilibrium zeolite data are given for the systems cesium-lithium, cesium-sodium, cesium-potassium, cesium-rubidium, cesium-ammonium, cesium-hydrogen, strontium-magnesium, strontium-calcium, strontium-barium, strontium-hydrogen and strontium-sodium in Figures 1 through 14. Cesium and strontium kinetic loading data are shown in Figures 16 and 17. Figure 18 shows cesium removal from a 1965 proposed Purex formaldehyde-treated waste composition (12).

Table II lists the zeolite capacity results obtained by double tracing. Linde 4A and 4AXW are the same zeolite, except that the latter contains less binder.

TABLE IIZeolite Cation Exchange Capacities

<u>Zeolite</u>	<u>Capacity, meq/g</u>
Linde 4AXW	3.9
Linde 4A	3.5
Linde 13X	3.6
Linde AW-500	2.2
Linde AW-400	2.0
Linde AW-300	1.6
Zeolon	1.9
Clinoptilolite	1.7

Table III compares predicted zeolite loading computed from the equilibrium and capacity data with actual zeolite loading values obtained in column experiments with comparable flow rates at 27C. Note that the predicted load of Linde 4A in one-sixteenth inch pellets does not agree with the actual loading capacity. Equilibrium was not attained between contacting solution and zeolite under the experimental conditions.

TABLE III

Predicted and Actual Column Capacities

Zeolite	Influent solution	Col. wt./g	Grain size range, mm	Predicted load, meq/g	Actual load, meq/g
Linde 13X	0.002N Sr^{++} + Sr^{85} + 0.01N Na^+	5.5	0.4-0.7	3.4 (Sr)	3.2 (Sr)
Linde 4A	0.002N Sr^{++} + Sr^{85} + 0.01N Na^+	5.5	0.4-0.7	3.5 (Sr)	3.4 (Sr)
Linde 4A	0.07 Sr^{++} + Sr^{85} + 0.1N Na^+	5.5	0.4-0.7	3.5 (Sr)	3.5 (Sr)
Linde 4A	0.07N Sr^{++} + Sr^{85} + 0.1N Na^+	50	1/16" pellets (1.6 mm)	3.5 (Sr)	0.9 (Sr)
Linde 4A	0.07N Sr^{++} + Sr^{85} + 0.1N Na^+	50	1/16" pellets	3.5 (Sr)	1.8 (Sr, 65C)
Clinoptilolite	0.01N Cs^+ + Cs^{134} + 1.0N Na^+	50	0.25 - 1.00	0.75 (Cs)	0.73 (Cs)
Clinoptilolite	0.01N Cs^+ + Cs^{134} + 0.5N Na^+	50	0.25 - 1.00	1.02 (Cs)	1.01 (Cs)
Linde AW-500	0.001N Cs^+ + Cs^{134} + 1.0N NH_4^+	50	1/16" pellets	0.037 (Cs)	0.039 (Cs)
Linde 4AXW	0.001N Cs^+ + Cs^{134} + 1.0N NH_4^+	50	1/16" pellets	0.011 (Cs)	0.011 (Cs)
Linde AW-500	0.001N Cs^+ + Cs^{134} + 1.0N K^+	50	1/16" pellets	0.040 (Cs)	0.050 (Cs)
Clinoptilolite	0.01N Cs^+ + Cs^{134} + 1.0N K^+	50	0.25 - 1.00	0.143 (Cs)	0.145 (Cs)
Linde AW-400	0.001N Cs^+ + Cs^{134} + 1.1N Na^+	50	1/16" pellets	0.240 (Cs)	0.241 (Cs)

Only under the condition of equilibrium between zeolite and solution passing through the column can the equilibrium data be used to predict column loads. Figure 16 shows that no kinetic problems should be encountered in dilute cesium loading, and none were found. The strontium loading problem with larger grain sizes of Linde 4A and 13X was further substantiated by loading kinetics as seen in Figure 17. If strontium packaging influents of similar chemical composition are contemplated, a compromise between influent flow rate, temperature and zeolite grain size will be necessary to attain full loading. Changing only the zeolite grain size, for example, from 1.6 mm (one-sixteenth inches) in diameter to a size range of 0.4 to 0.7 mm is sufficient to attain full loading with a steep breakthrough.

Figure 18 indicates that with a reasonable flow rate, clinoptilolite can be used to remove and concentrate cesium radioisotopes from projected 1965, formaldehyde-treated, Purex waste.

A summary of cesium or strontium losses during heating and the elution results are given in Table IV. All elutions were at room temperature.

TABLE IV

Effect of Heat on Loss and Elution of Radioactivity from Zeolites

<u>Zeolite</u>	<u>Per cent loss static conditions</u>	<u>Eluting agent</u>	<u>Per cent eluted</u>	<u>Cation eluted</u>
Clinoptilolite	0.09	2N NH ₄ NO ₃	96.6	Cs
Linde AW-500	0.0	2N NH ₄ NO ₃	86.00	Cs
Linde AW-500	0.0	1N HNO ₃	90.0	Cs
Linde 13X	0.0	2N NH ₄ NO ₃	62.0	Cs
Linde 13X	0.0	0.5N HNO ₃	97.0	Cs
Linde 13X	0.0	2N HNO ₃	99.0	Cs
Norton Zeolon	---	2N NH ₄ NO ₃	99.0	Cs
Decalco	0.016	2N NH ₄ NO ₃	38.0	Cs
Decalco	0.0	6N La(NO ₃) ₃	22.0	Cs
Linde 4A	0.01	1N HNO ₃	99.0	Sr

The amount of cesium or strontium lost during heating is expressed as a per cent of the total load on the zeolite. A value of 0.0 indicates that the counting rates of the scrubbers and filter were at background levels with 60-minute counts. It is possible that part of the radioactivity released during heating represents loading solution present due to incomplete washing of the zeolite column.

Figures 20 through 23 show per cent of radioisotope eluted vs. throughput of eluting agent for the same zeolite columns reported in Table IV. Strontium elution from Linde 4A was very slow with CuCl_2 . Except for clinoptilolite, Zeolon and Linde AW-300, 1N HNO_3 elutions remove aluminum from the zeolite crystal lattice. If 0.01 to 0.05N HNO_3 is used as an eluting agent, a larger volume of eluting solution is required but the zeolite remains intact.

CONCLUSIONS

The zeolites, as inorganic cation exchange media, offer certain advantages over organic or other exchange media for extraction and/or storage of radioisotopes. The advantages include high radiation, thermal, and in some instances, acid resistivity. In addition, stored radioisotopes may be eluted from the zeolites by one of several eluting agents. The zeolite eluting agent used would depend upon how the eluted radioisotope is to be utilized. The data presented here on the ion exchange and elution properties of several commercially available zeolites should help to choose an appropriate zeolite to fill a given application.

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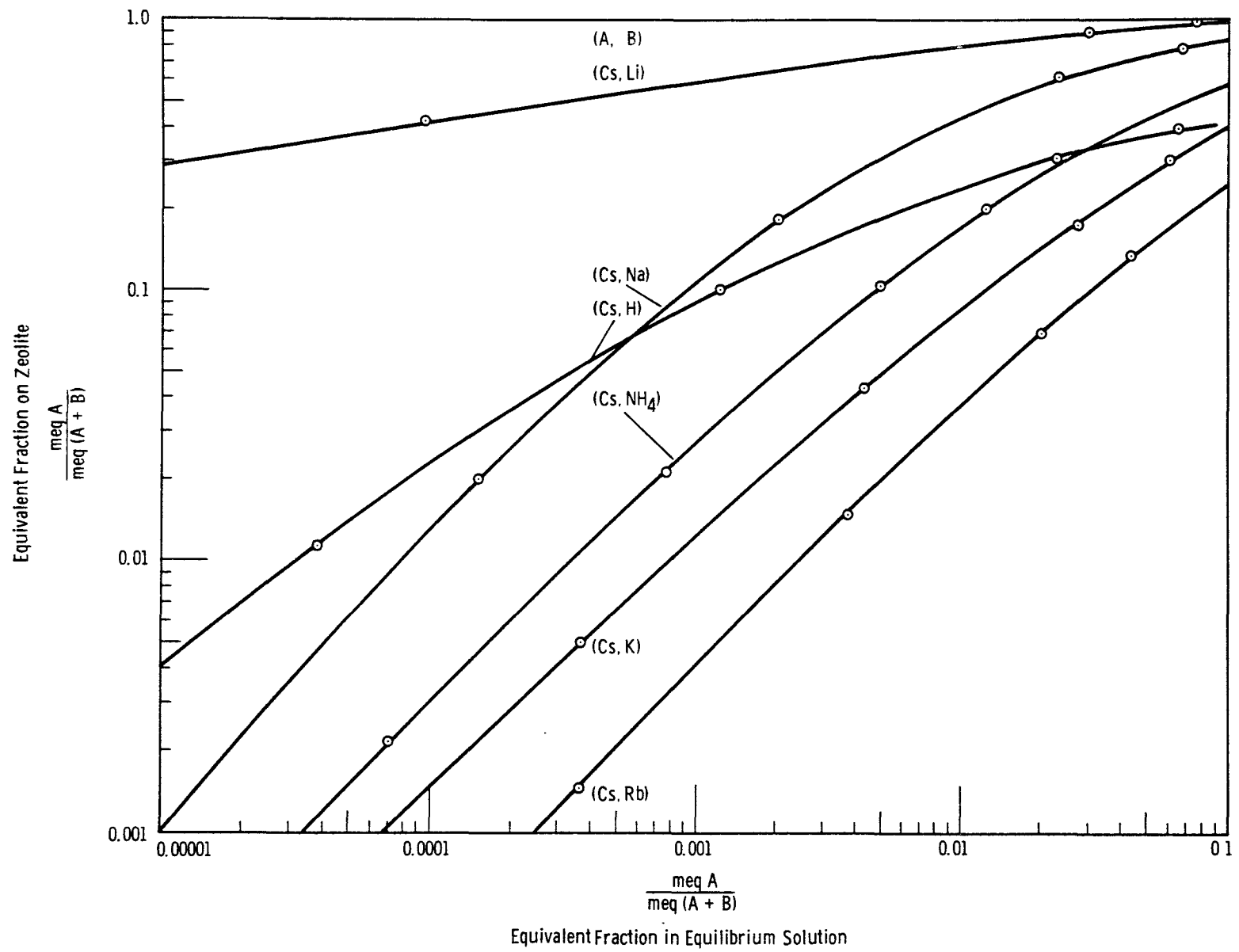


Figure 1. The effect of univalent cations on the equilibrium cesium loading of clinoptilolite.

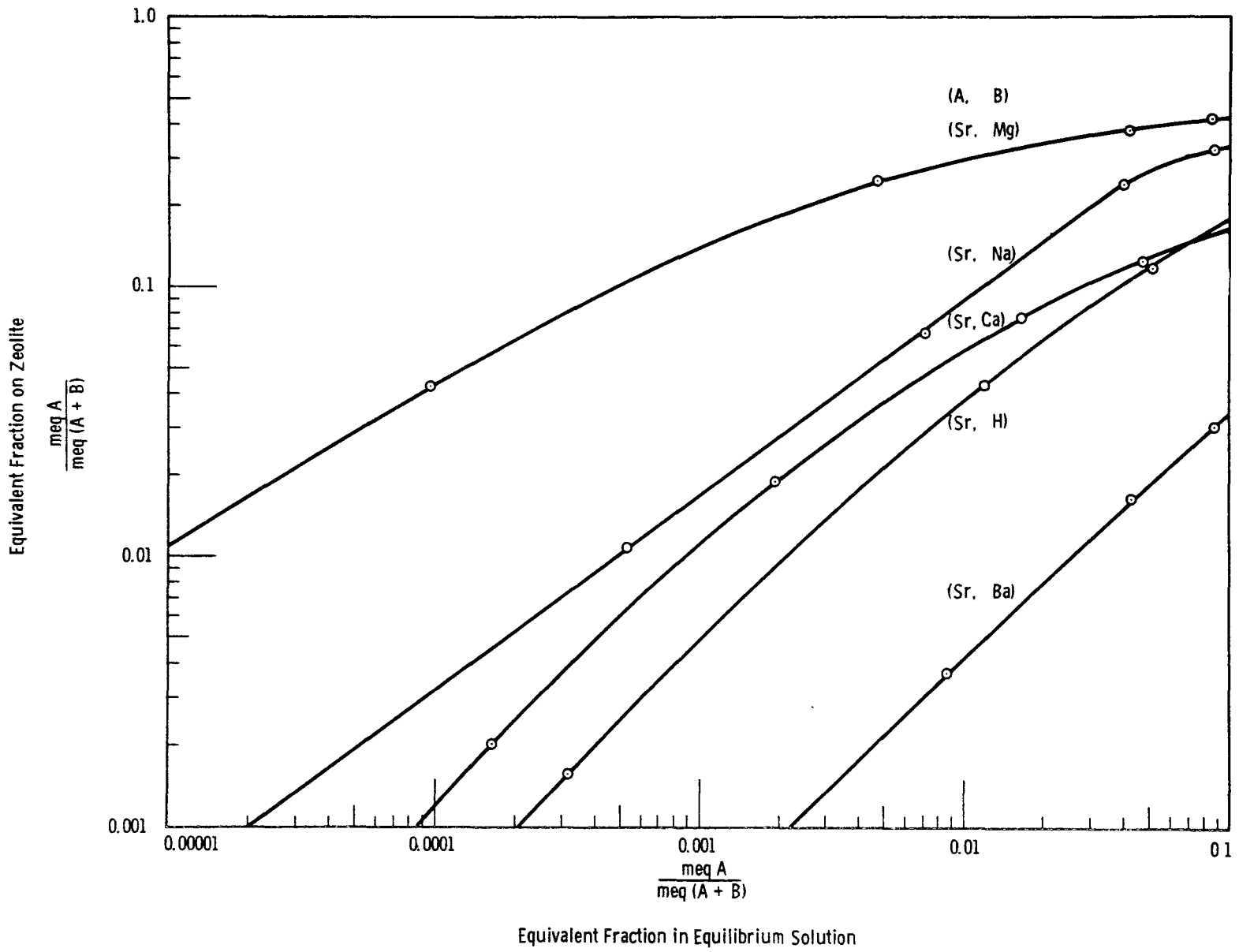


Figure 2. The effect of the alkaline earth metals, hydrogen and sodium on the equilibrium strontium loading of clinoptilolite.

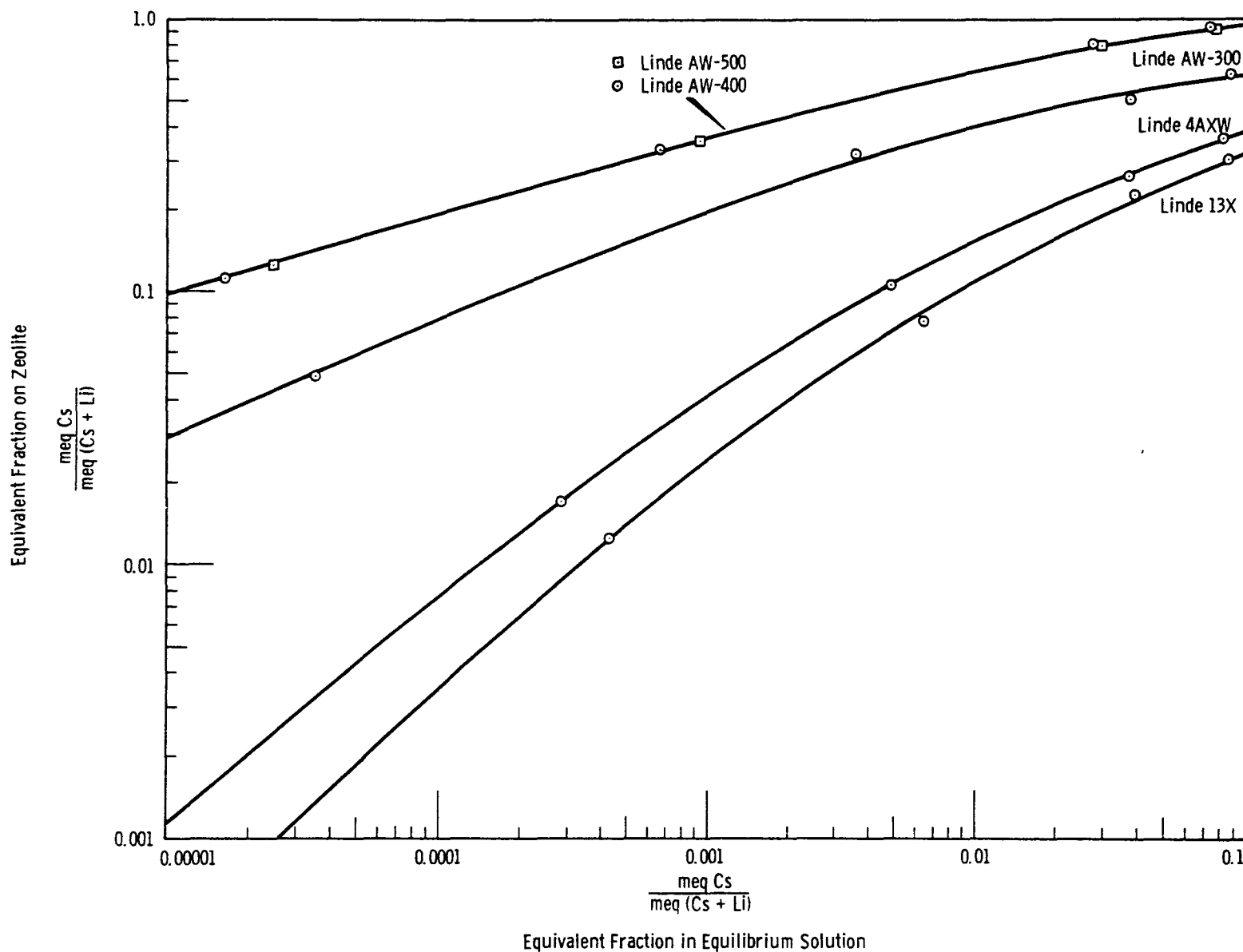


Figure 3. The effect of lithium on the equilibrium cesium loading of Linde 4AXW, 13X, AW-300; AW-400 and AW-500

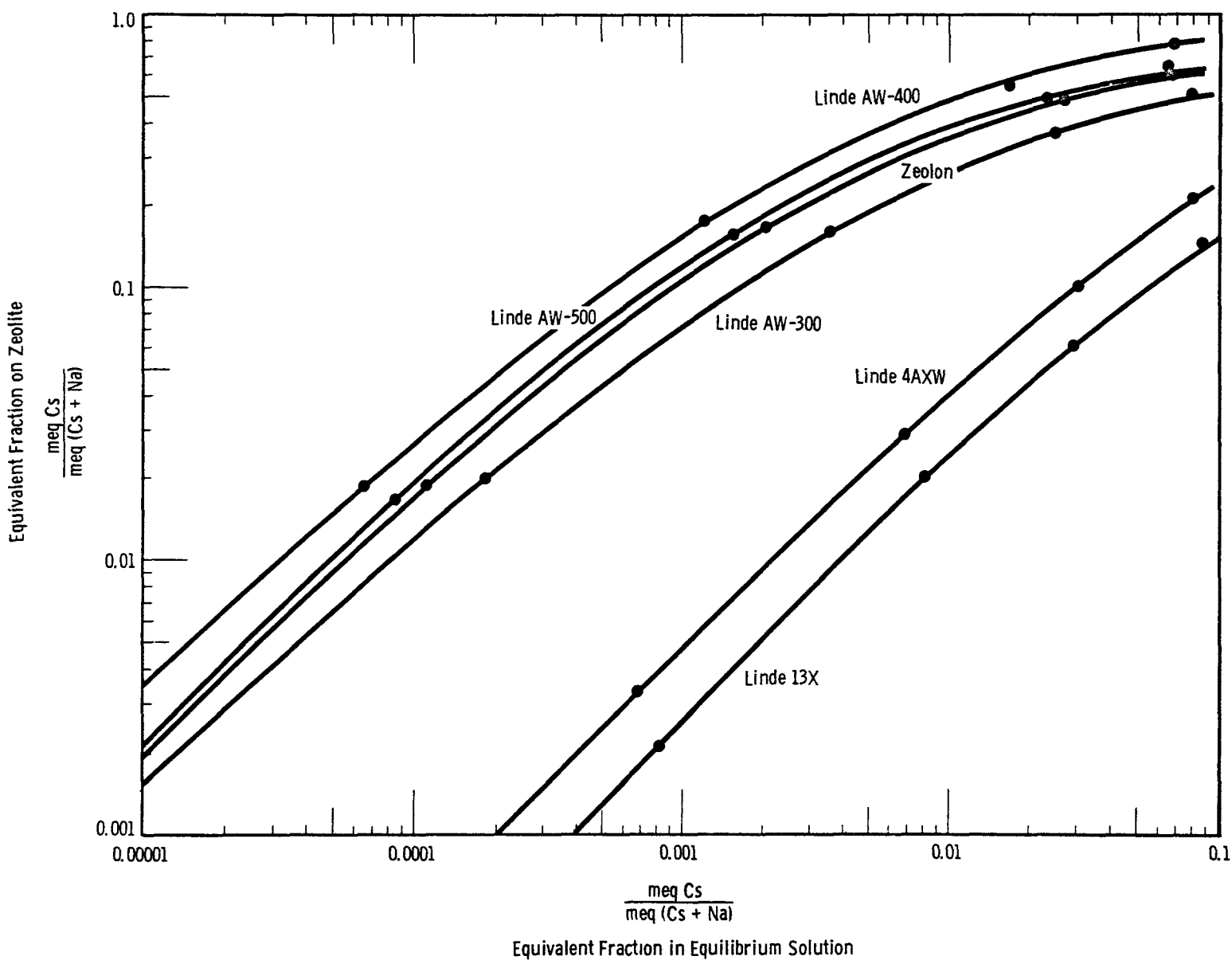


Figure 4. The effect of sodium on the equilibrium cesium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500 and Norton Zeolon.

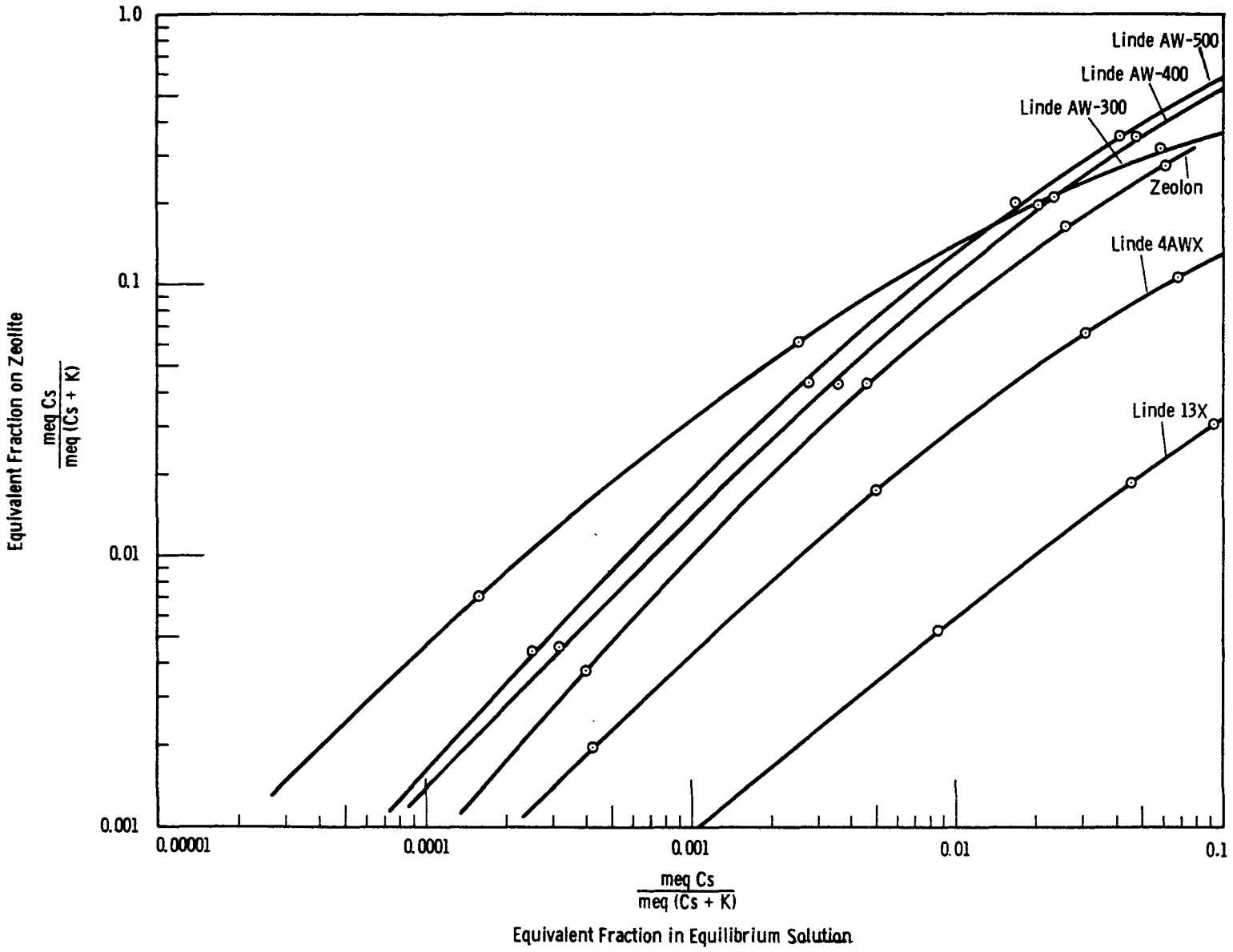


Figure 5. The effect of potassium on the equilibrium cesium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500 and Norton Zeolon.

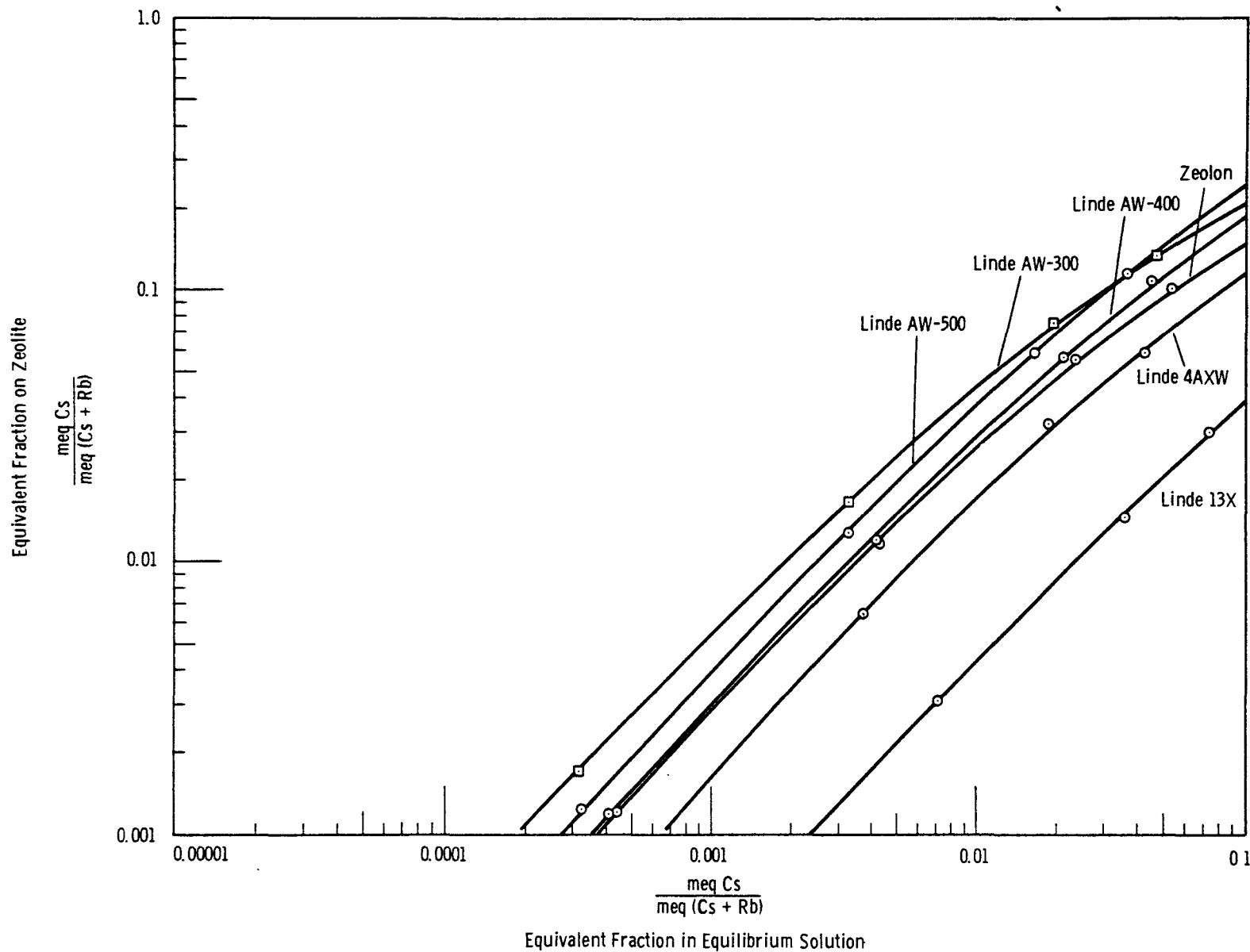


Figure 6. The effect of rubidium on the equilibrium cesium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500 and Norton Zeolon.

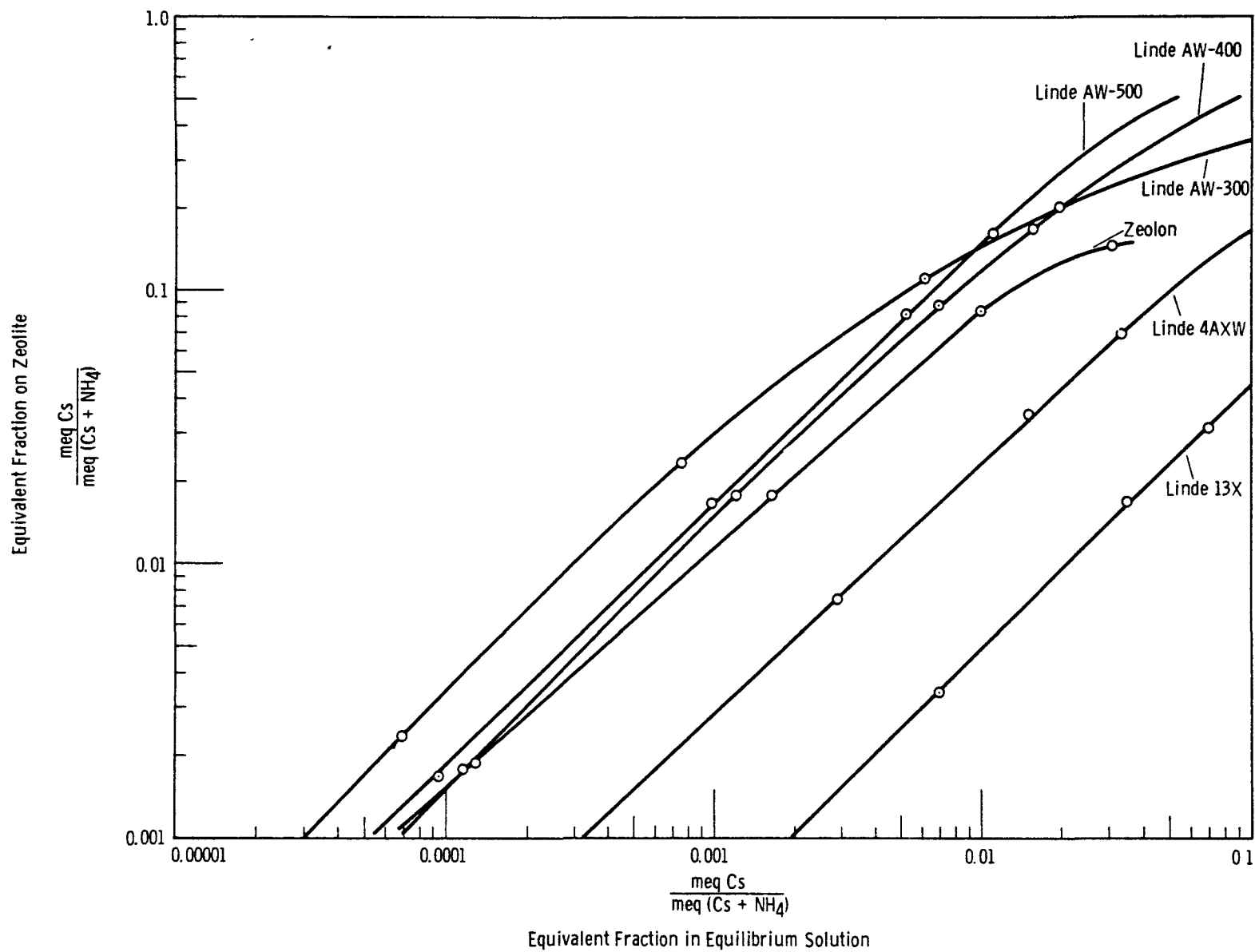


Figure 7. The effect of ammonium ion on the equilibrium cesium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500, and Norton Zeolon.

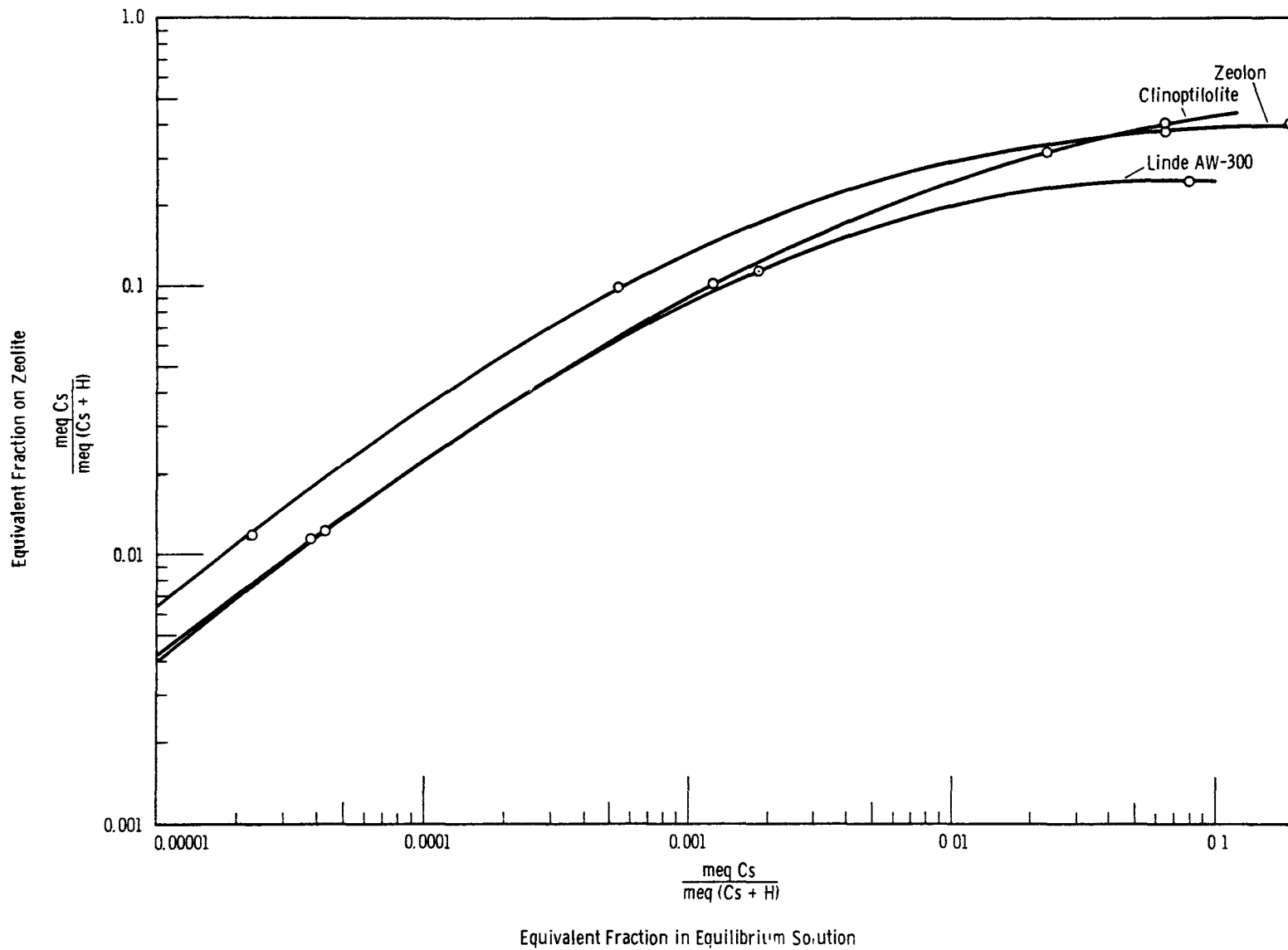


Figure 8. The effect of hydrogen on the equilibrium cesium loading of clinoptilolite, Norton Zeolon and Linde AW-300.

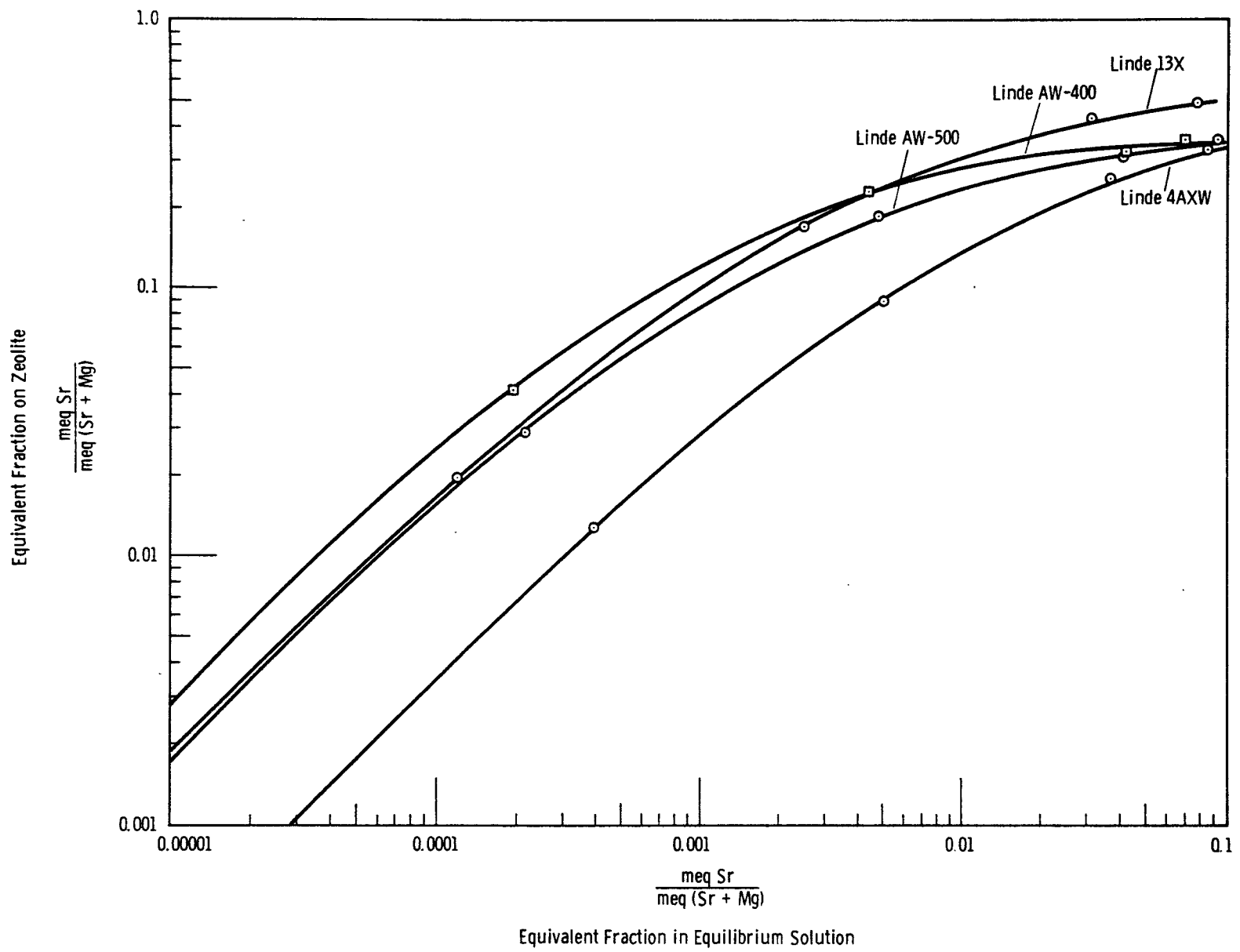


Figure 9. The effect of magnesium on the equilibrium strontium loading of Linde 4AXW, 13X, AW-400 and AW-500.

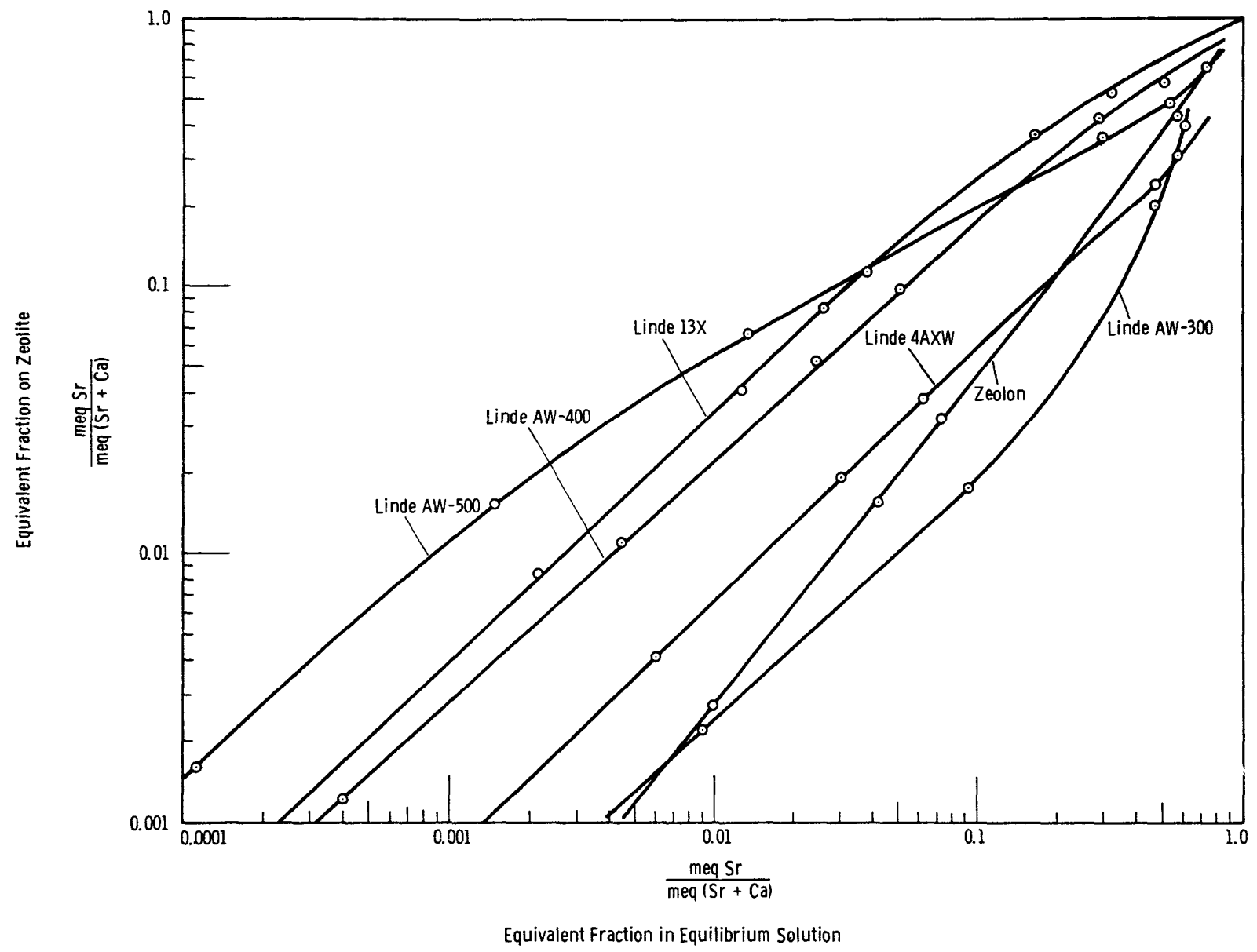


Figure 10. The effect of calcium on the equilibrium strontium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500 and Norton Zeolon.

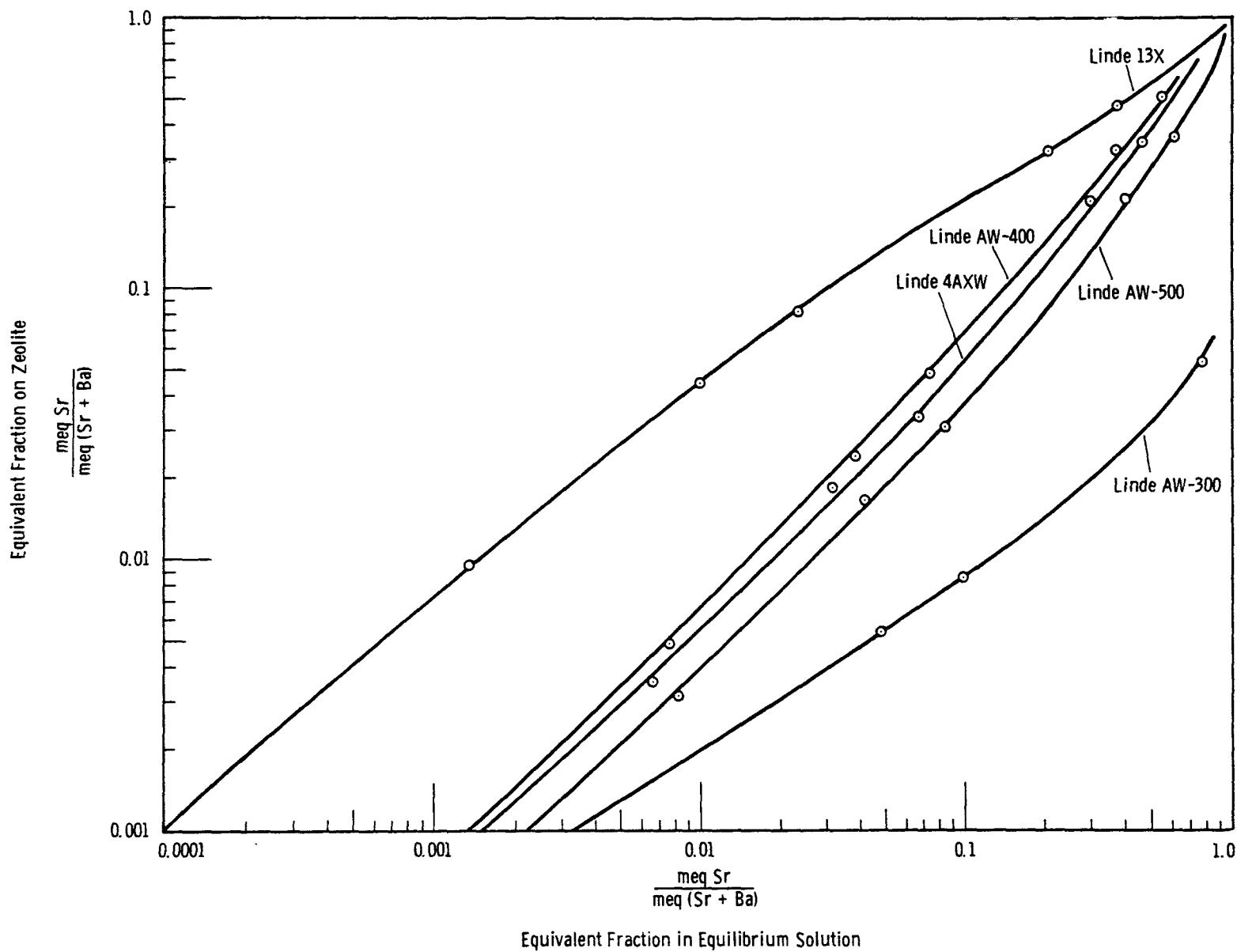


Figure 11. The effect of barium on the equilibrium strontium loading of Linde 4AXW, 13X, AW-300, AW-400, and AW-500.

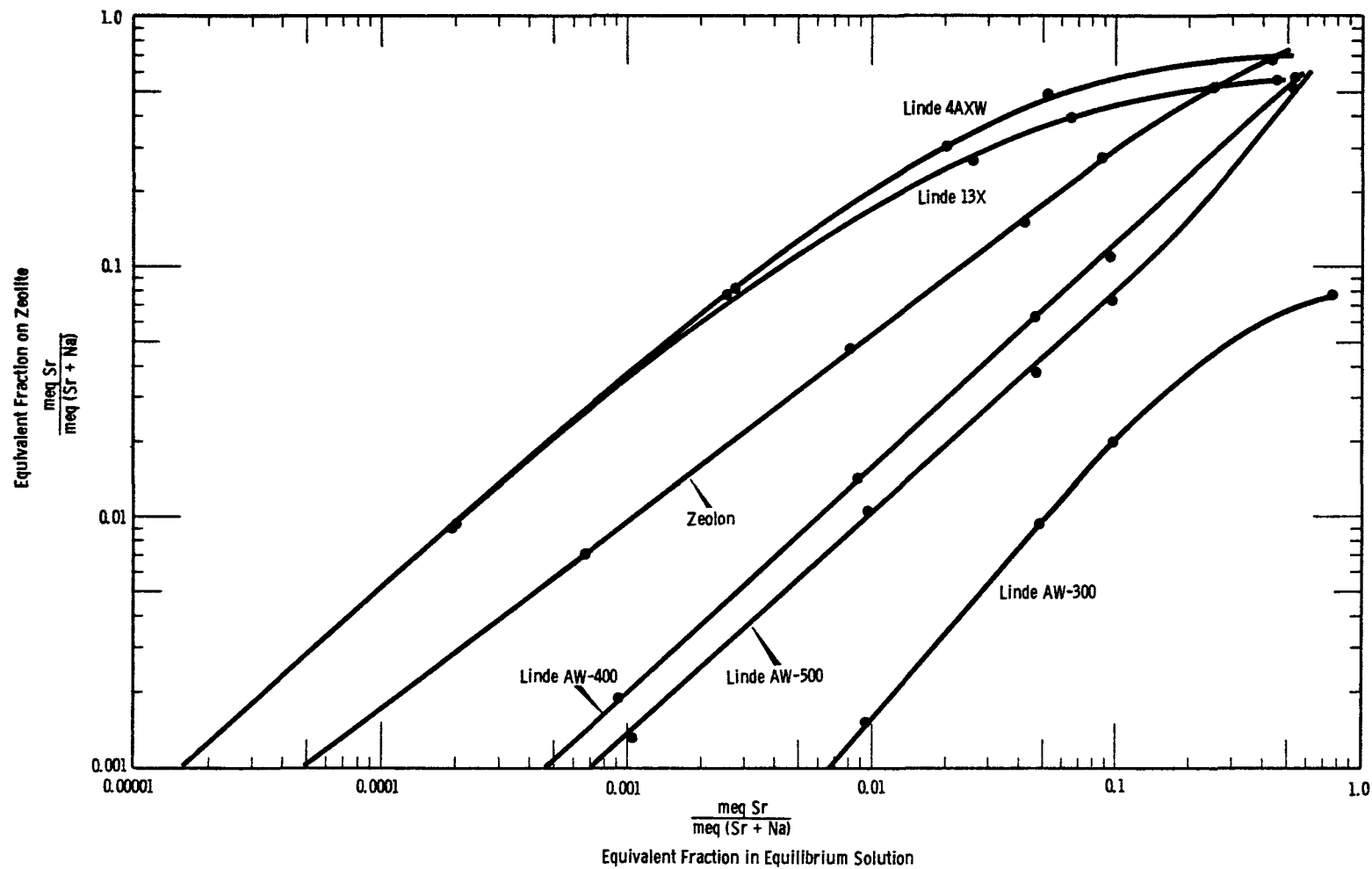


Figure 12, The effect of sodium on the equilibrium strontium loading of Linde 4AXW, 13X, AW-300, AW-400, AW-500 and Norton Zeolon.

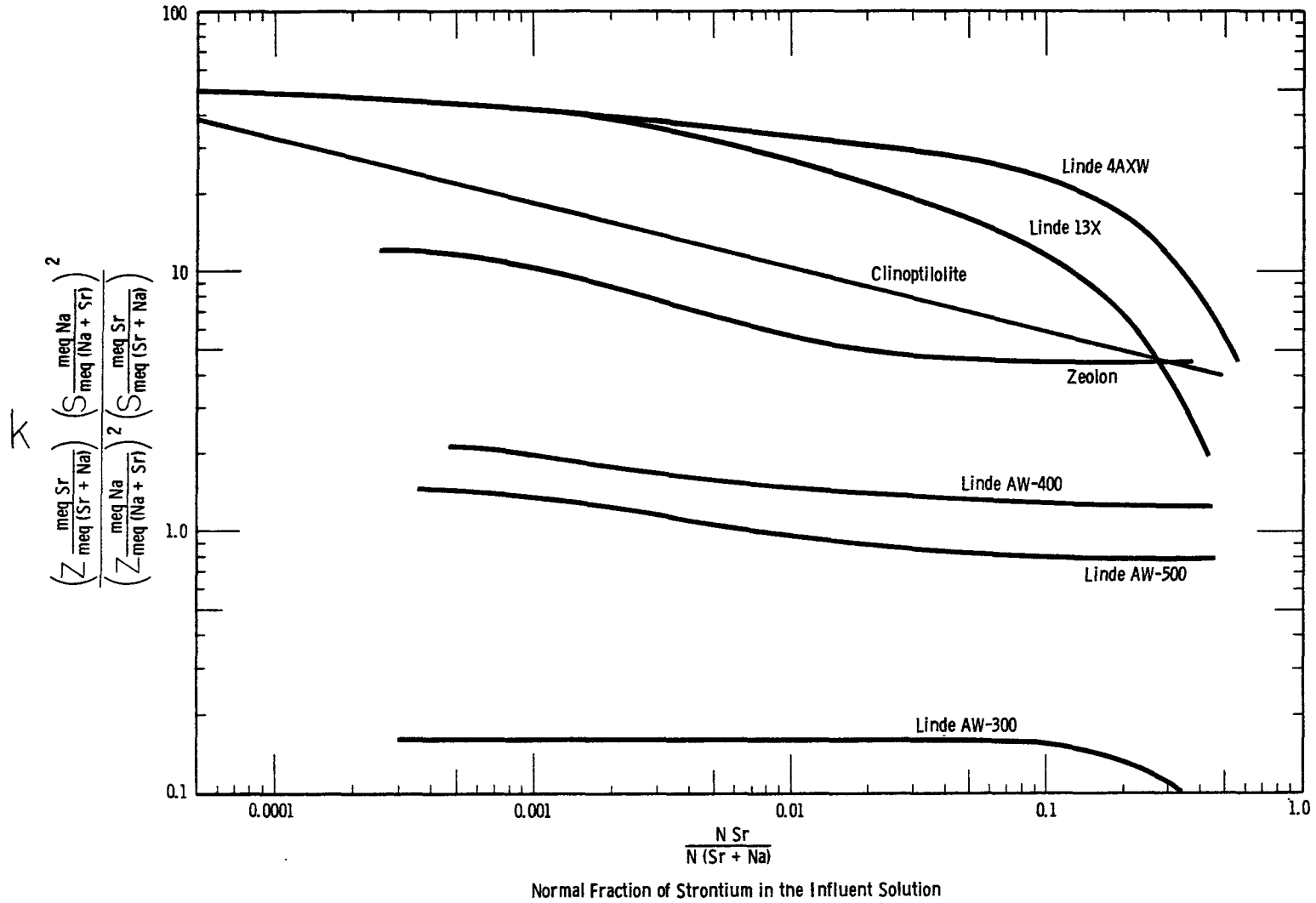


Figure 13. The effect of sodium on the strontium equilibrium constant, k , of Linde 4AXW, 13X, AW-300, AW-400, AW-500, Norton Zeolon and clinoptilolite.

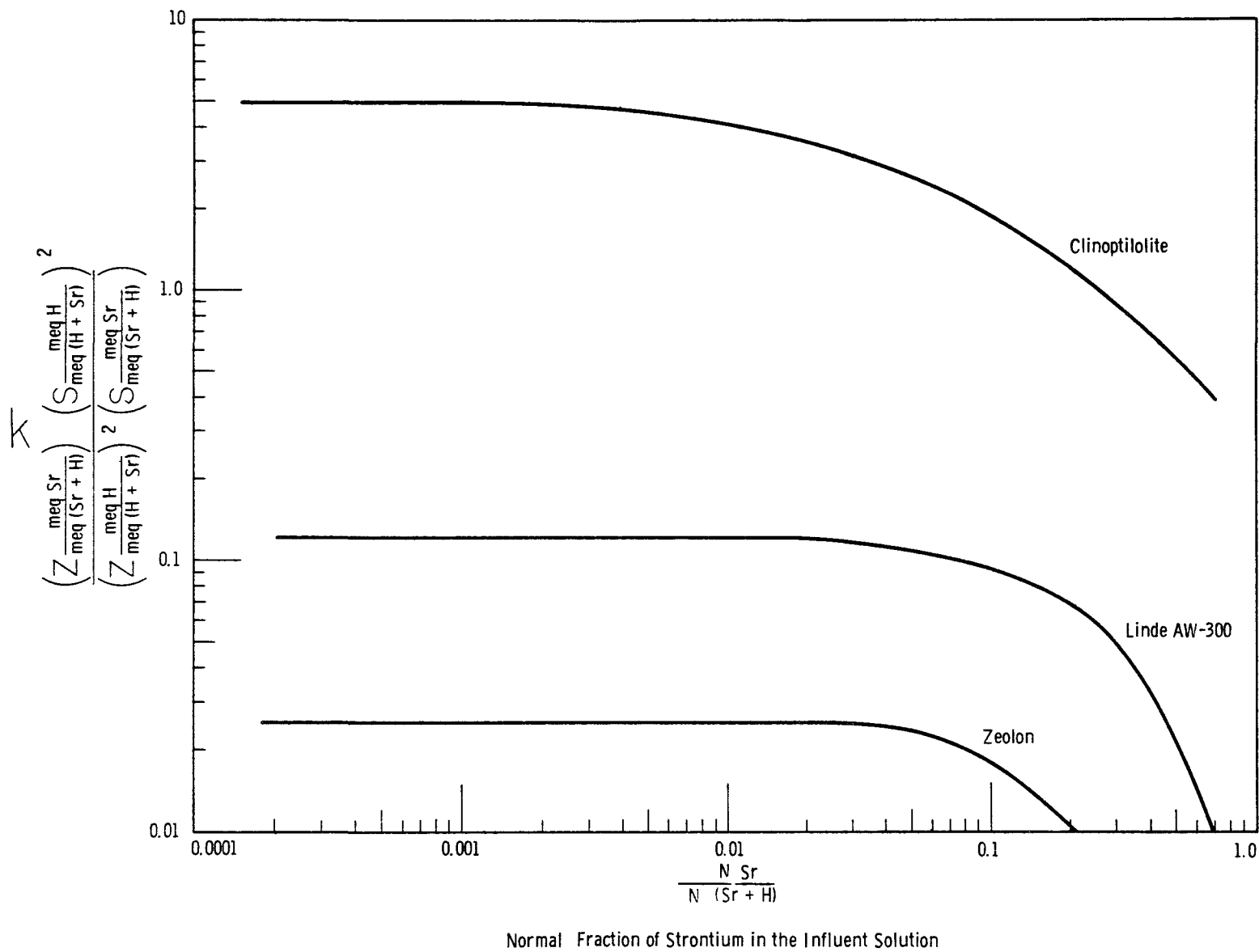


Figure 14. The effect of hydrogen on the strontium equilibrium constant, k , of Linde AW-300, Norton Zeolon and clinoptilolite.

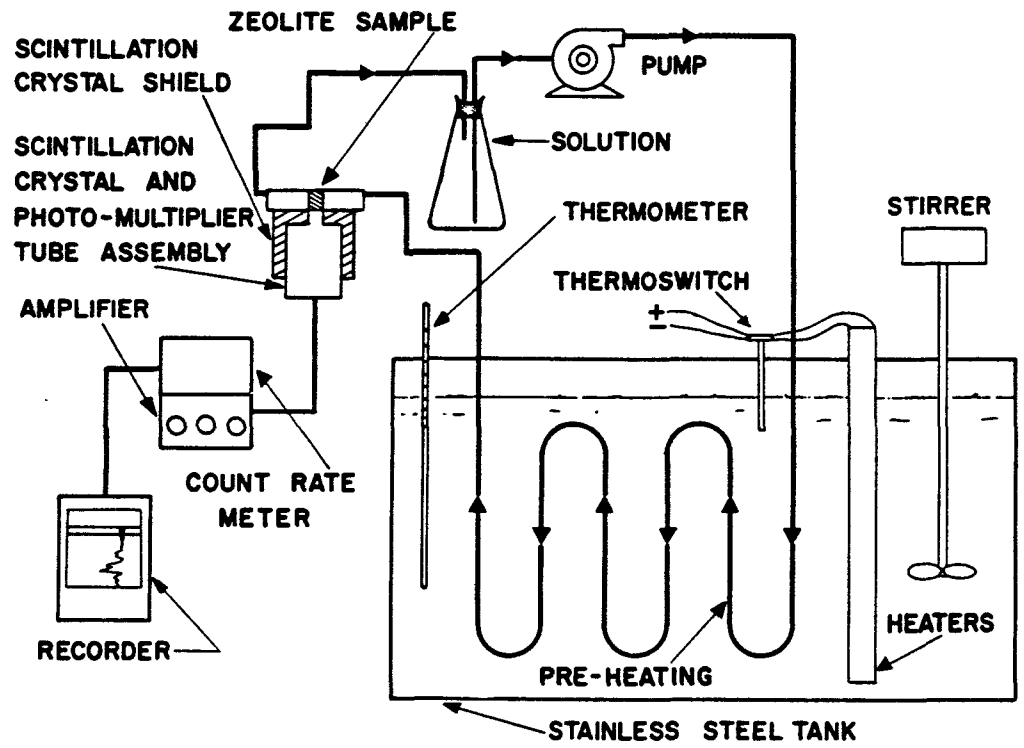


Figure 15. Shallow bed loading apparatus.

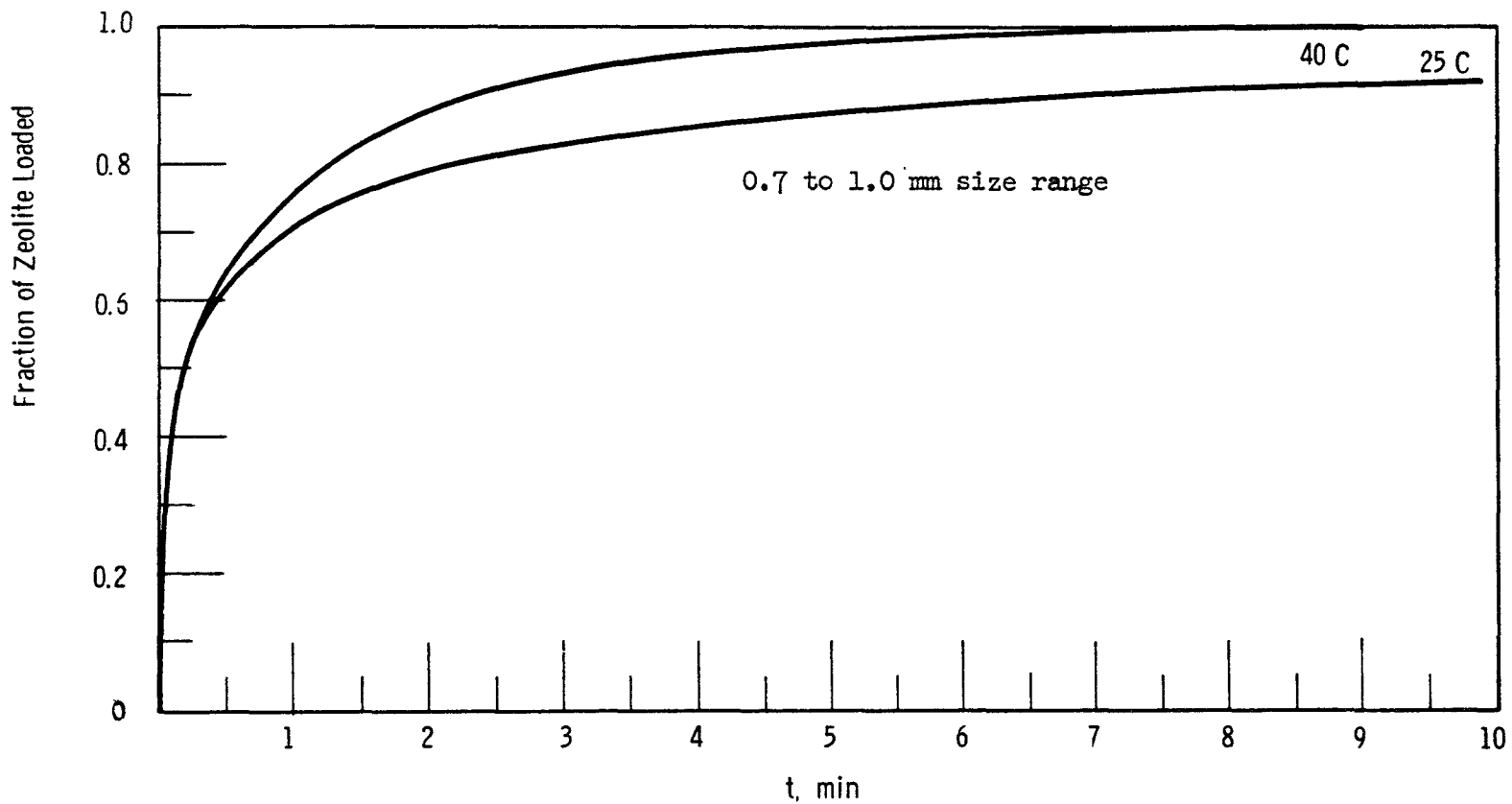


Figure 16. Cesium loading on Linde 13X vs. time from a 0.1N CsCl solution.

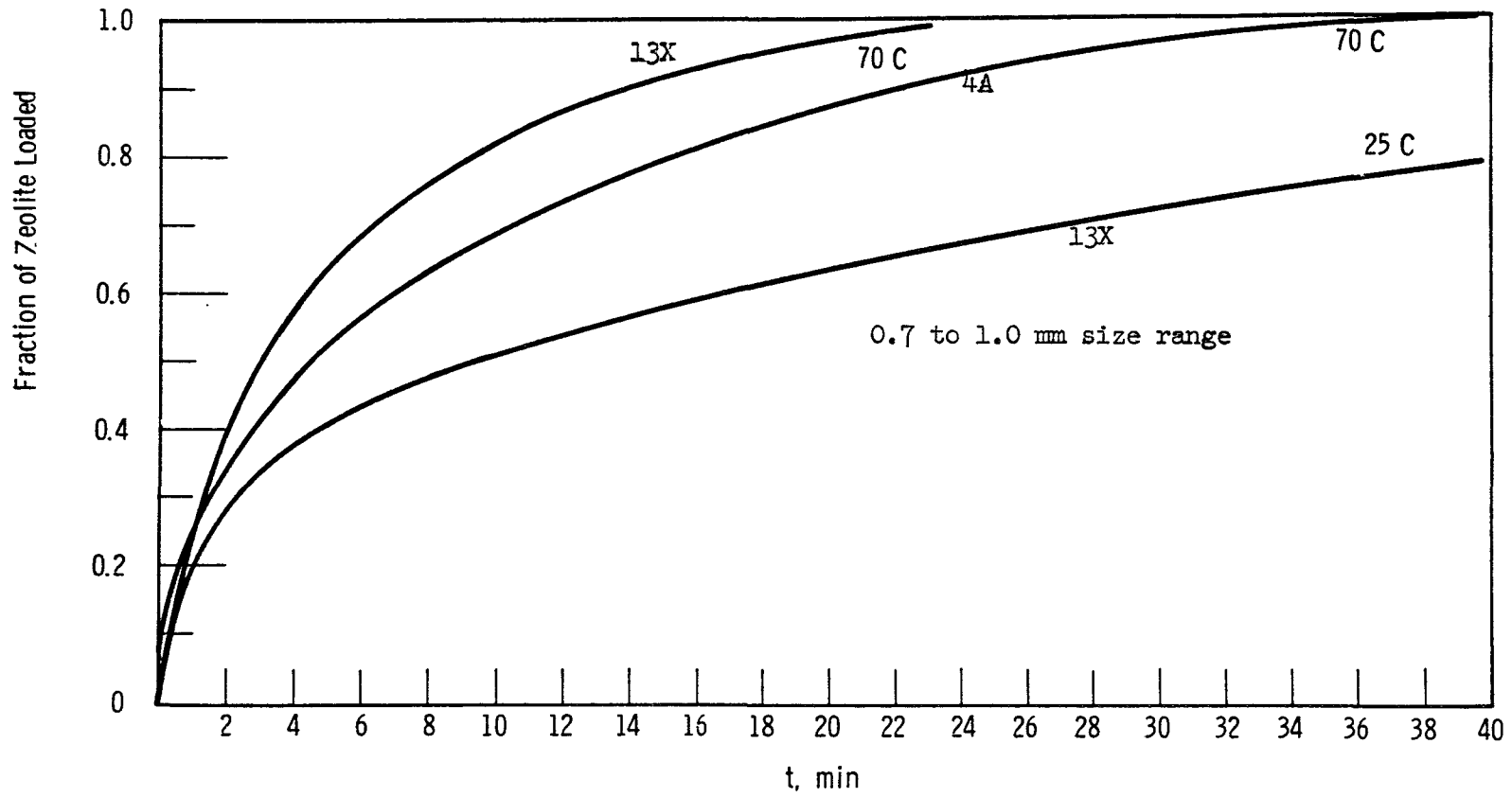


Figure 17. Strontium loading on Linde 13X and 4A vs. time from a 0.1N SrCl₂ Solution.

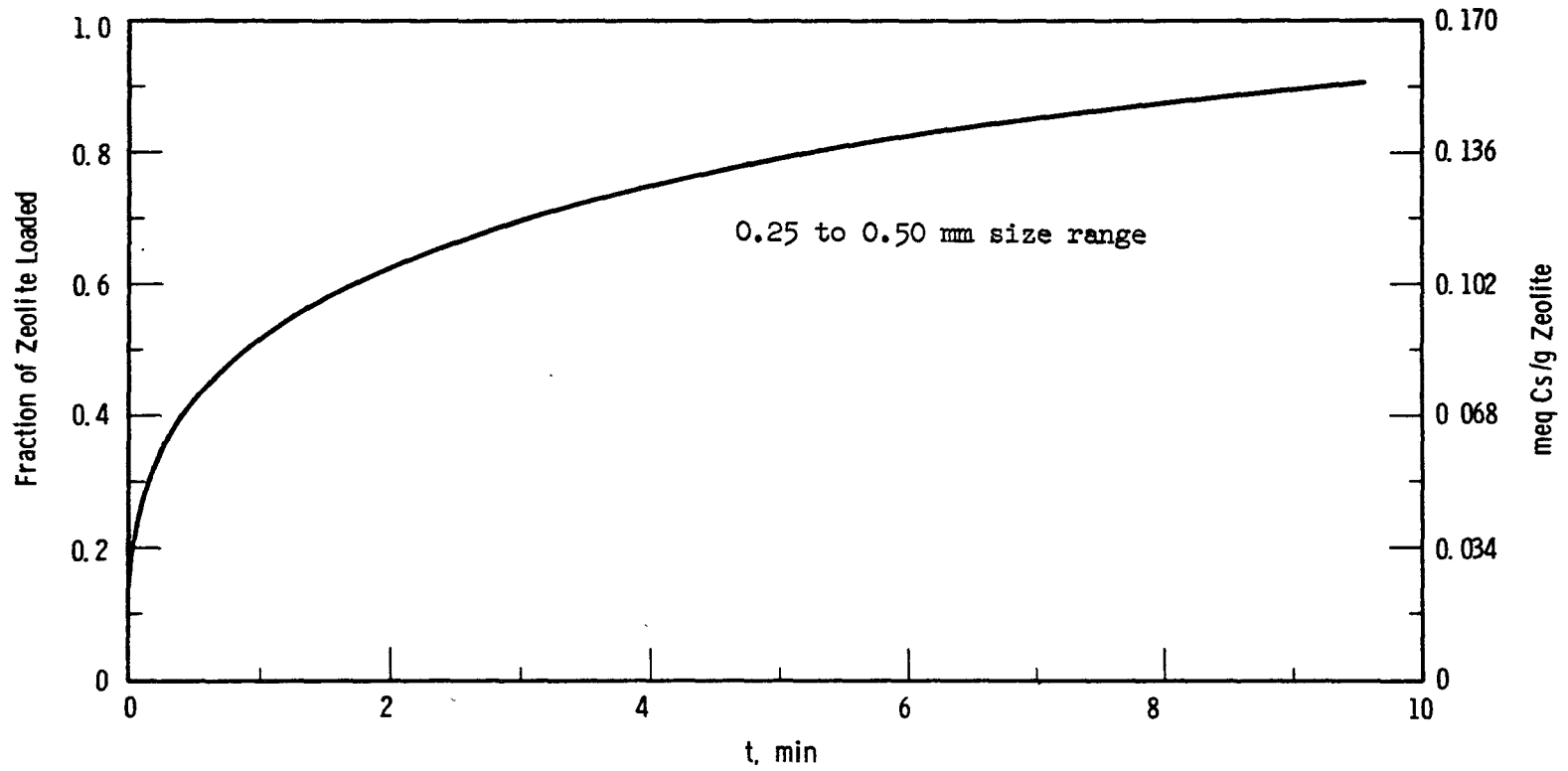


Figure 18. Cesium loading on clinoptilolite vs. time from a simulated 1965, formaldehyde-treated, Purex waste (12).

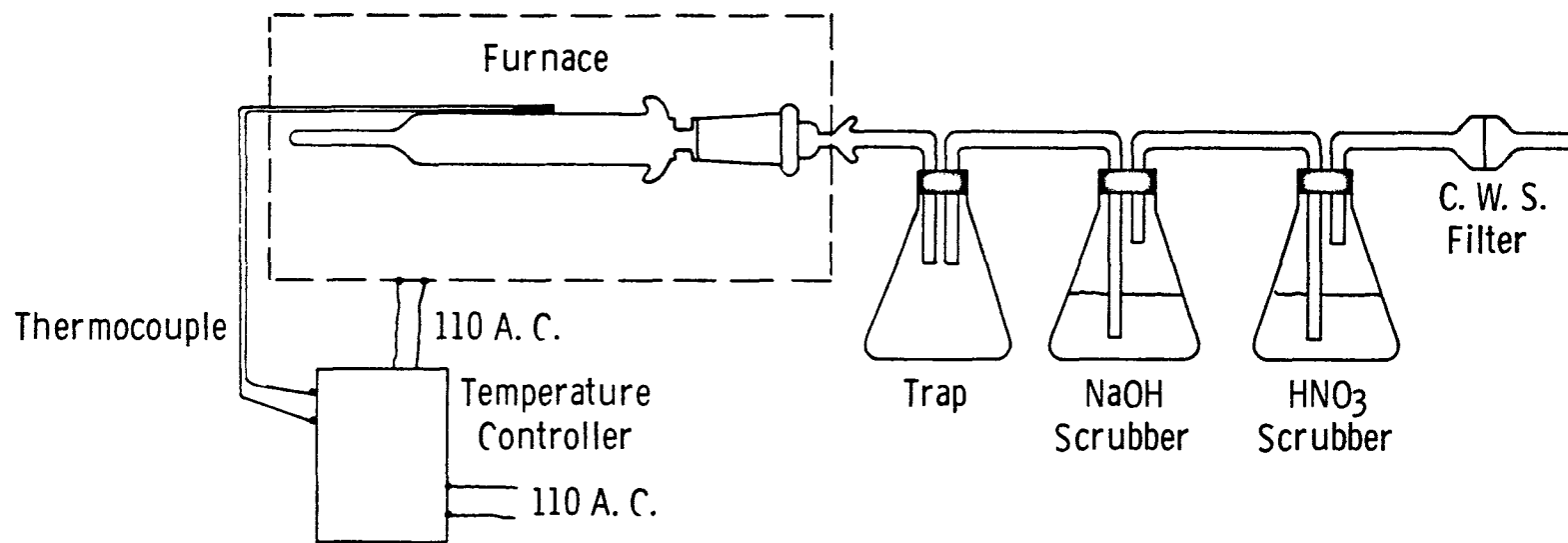


Figure 19. Zeolite column heating apparatus.

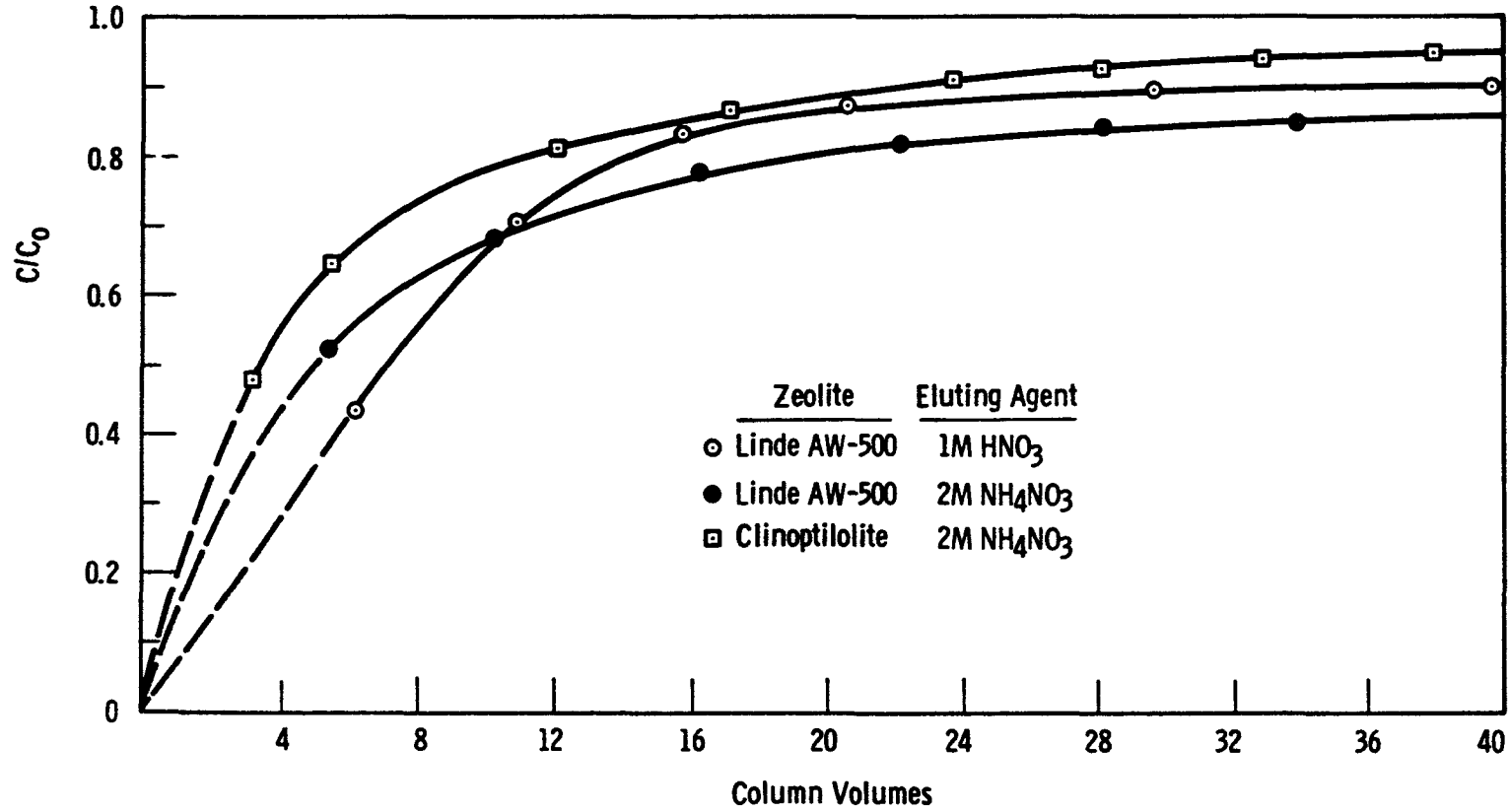


Figure 20. Elution of cesium from Linde AW-500 and clinoptilolite.

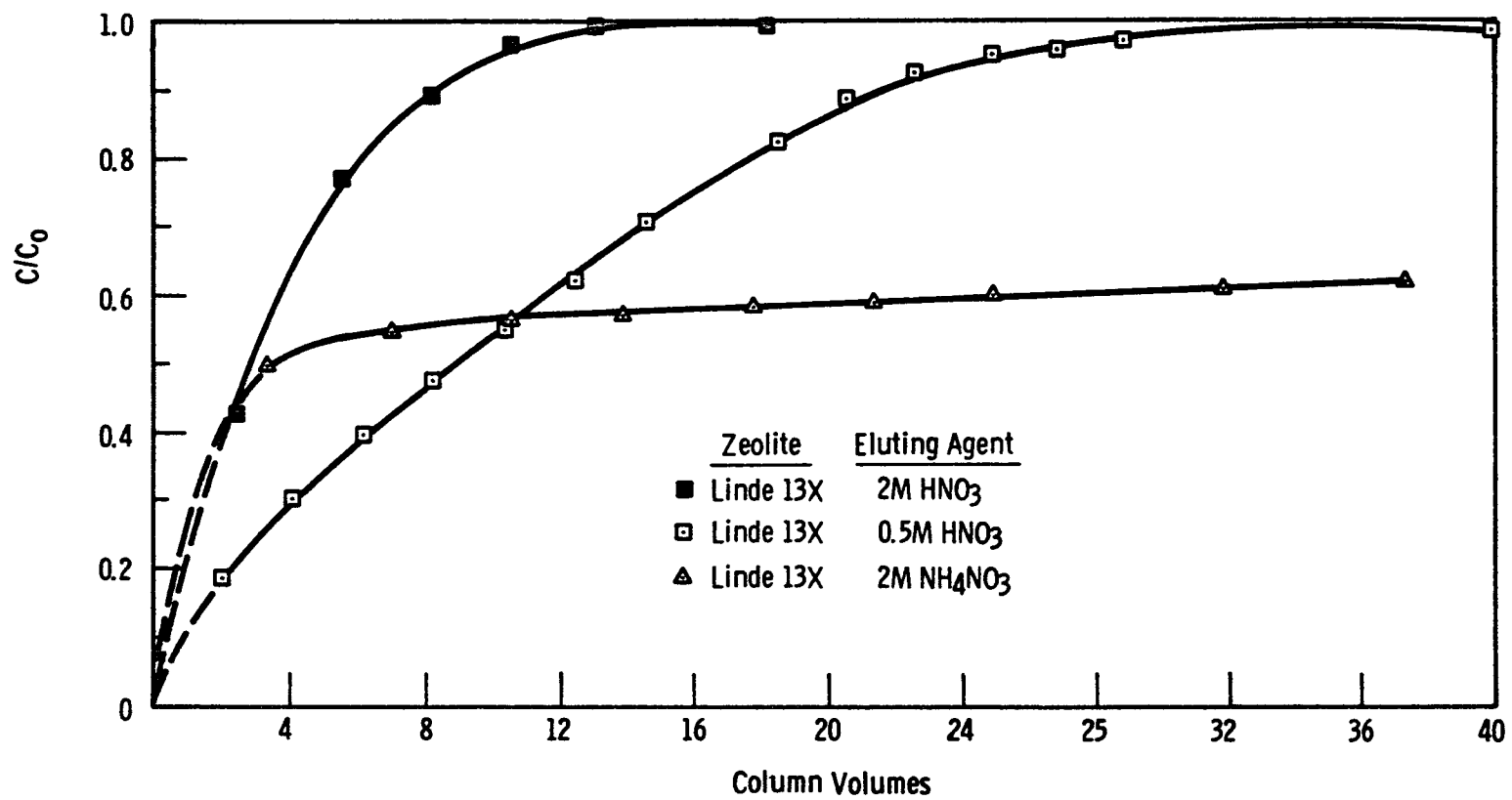


Figure 21. Elution of cesium from Linde 13X.

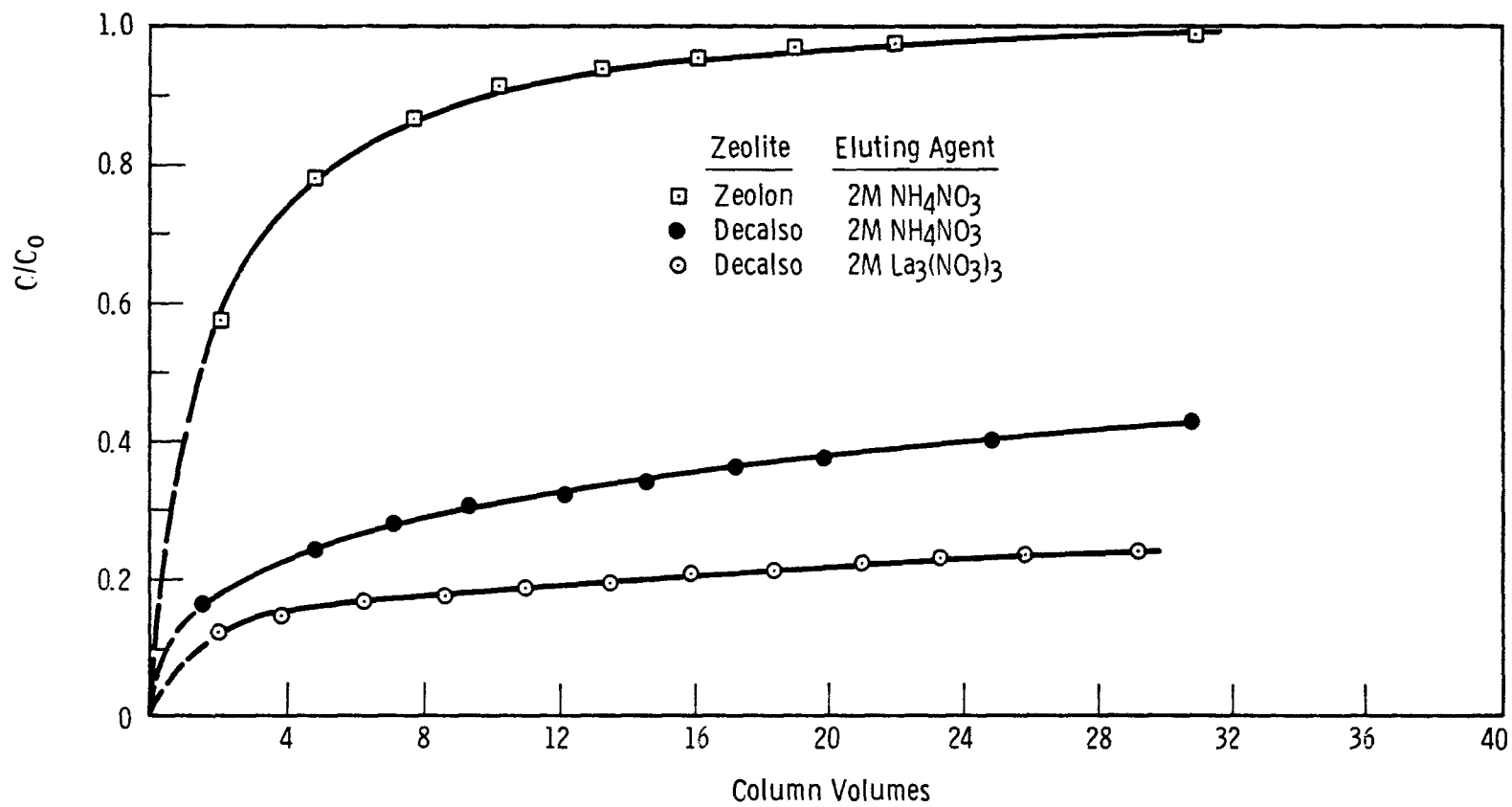


Figure 22. Elution of cesium from Norton Zeolon and Decalso.

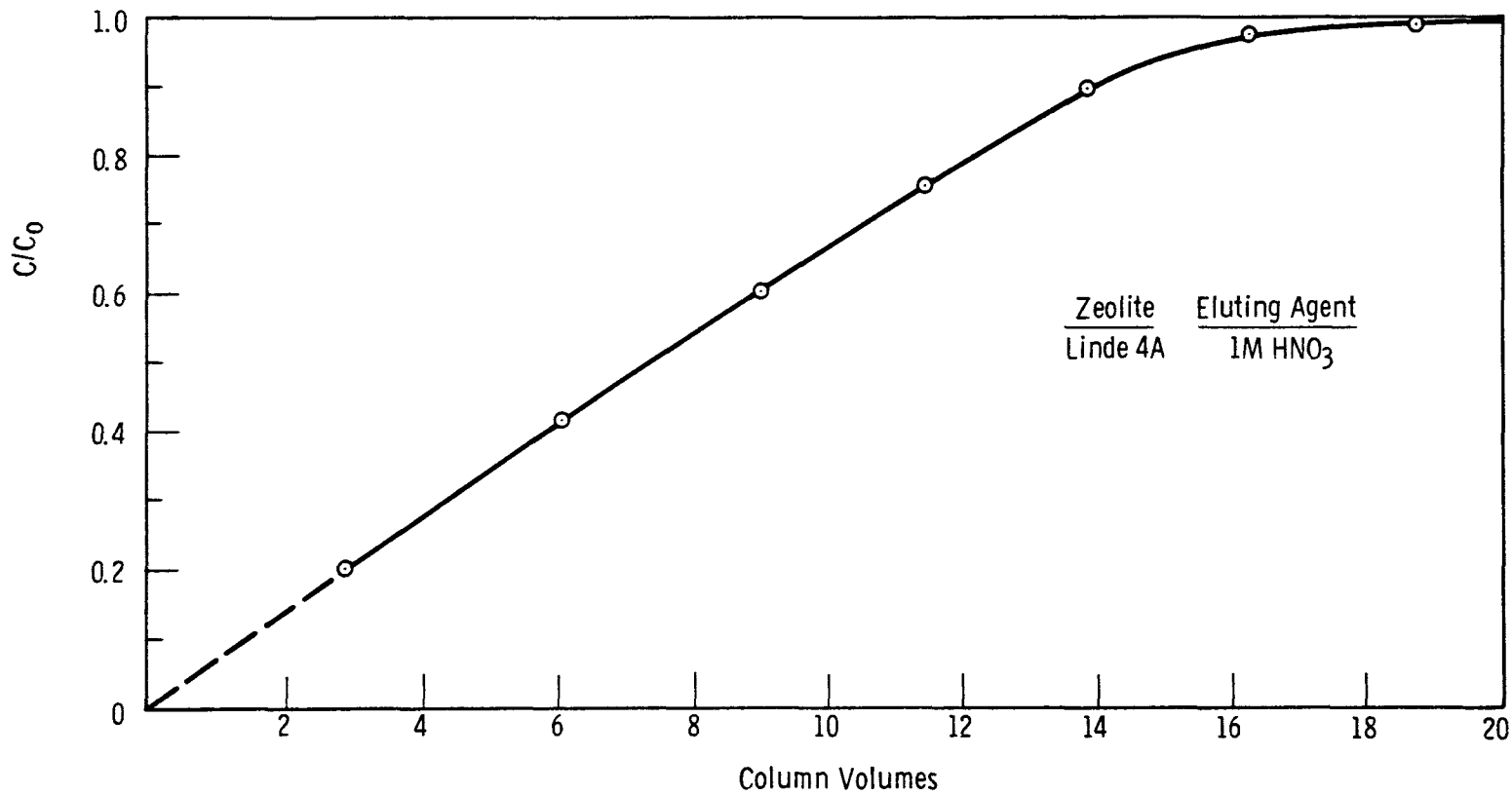


Figure 23. Elution of strontium from Linde 4A.