

1949

Industrial uses of ion exchange resins

Donald H. White
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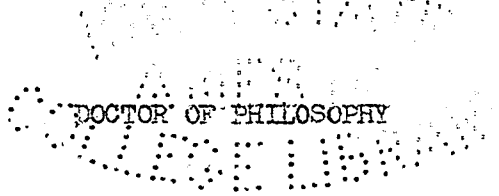
141

INDUSTRIAL USES OF ION EXCHANGE RESINS

by

Donald H. White

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of



Major Subject: Chemical Engineering

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TABLE OF CONTENTS

	Page
I. INTRODUCTION.....	1
II. HISTORICAL AND LITERATURE REVIEW.....	2
A. Terminology.....	2
B. History of Ion Exchange Materials.....	6
C. Methods of Classification.....	7
1. Classification by type of exchange reaction.....	7
2. Classification by industries.....	8
D. Review of Ion Exchange Literature.....	9
1. Theory of ion exchange.....	10
a. Mechanism of ion exchange.....	10
b. Equilibrium of ion exchange.....	12
c. Rate of ion exchange.....	17
2. Techniques of operation.....	19
3. Properties of exchange materials.....	21
E. Uses of Cation Exchange Materials.....	24
1. Preparations by cation exchange.....	24
a. Preparation of chemical products.....	24
b. Modification of materials.....	30
2. Separations by cation exchange.....	30
a. Separation of cations from anions.....	30
b. Separation of cations by differences in ionic forces... 31	31
c. Separation of cations by complex ion elution.....	36
3. Concentrations by cation exchange.....	36

T 9068 L

	Page
4. Purification by cation exchange.....	37
F. Uses of Anion Exchange Materials.....	38
1. Preparations by anion exchange.....	39
2. Separations by anion exchange.....	39
3. Concentrations by anion exchange.....	40
4. Purifications by anion exchange.....	40
G. Combined Uses of Cation and Anion Exchange Materials.....	41
H. Exchangers as Contact Materials.....	42
III. EXPERIMENTAL.....	44
A. Equilibrium of Ion Exchange.....	44
1. Preparation of materials.....	44
2. Operating procedure.....	45
3. Experimental runs.....	46
4. Analytical procedures.....	46
5. Results and correlations.....	46
B. Rate of Ion Exchange.....	64
1. Preparation of materials.....	64
2. Operating procedure.....	64
a. Combined rate of exchange and static capacity.....	64
b. Combined rate of exchange and equilibrium curves....	65
c. Combined rate of exchange and equilibrium point.....	66
d. Combined rate of exchange and equilibrium curve by pH	67
3. Experimental runs.....	67
4. Results and correlations.....	68
C. Fluidized Zeolite Fines Bed.....	123

	Page
1. Description of apparatus.....	123
2. Preparation of materials.....	124
3. Operating procedure.....	125
4. Experimental runs.....	126
5. Analytical procedures.....	126
6. Results and correlations.....	127
7. Proposed industrial unit.....	130
D. Beaded Zeolite Cation Exchanger.....	183
1. General preparation procedure.....	183
2. Experimental preparation of exchanger.....	183
3. Results.....	186
E. Process for Copper Production.....	187
1. Leaching of ore.....	187
2. Concentration and purification by cation exchange.....	191
3. Tolerance for interfering ions.....	191
4. Copper equilibrium studies.....	193
5. Distribution of copper and calcium in exchanger bed.....	194
6. Discussion on proposed process for copper production.....	195
F. Purification of Phenol Wastes.....	216
1. Laboratory investigations.....	216
2. Future possibilities for phenol recovery.....	217
IV. DISCLOSURE OF INVENTIONS.....	219
V. SUMMARY AND CONCLUSIONS.....	221
VI. BIBLIOGRAPHY.....	223
VII. ACKNOWLEDGMENT.....	276

I. INTRODUCTION

Two relatively recent events marked the beginning of real advancement in the field of ion exchange. The first event took place in 1930 when the first organic cation exchange material was discovered. This made it possible to operate a cation exchange in the acid, or, as it is commonly called, the hydrogen cycle. The second event marked the discovery of anion exchange in 1935, which made it possible to remove anions as well as cations from aqueous solutions.

It is the purpose of this investigation to (1) systematically classify the existing industrial uses of ion exchange and (2) present experimental data illustrating how ion exchange might be used more extensively.

It will be made clear that a consideration of present processes utilizing ion exchange will not suffice for a clear understanding of the field. One must become familiar with three broad classes of "exchange tools", namely, (1) theory of ion exchange, (2) techniques of operation, and (3) properties of exchange materials. Advancement in these three "exchange tools" will be a major factor in the future rate of progress of ion exchange applications.

The experimental work reported in this thesis was done prior to September 1, 1948. Likewise, the bibliography and literature review involve the reported uses of ion exchange resins up to the same date.

II. HISTORICAL AND LITERATURE REVIEW

A. Terminology

Very little standardization of test methods and terminology has been done in the field of ion exchange. Many terms now being used were originally set up for siliceous exchangers and designed for water softening practices. However, since exchangers are now available that exhibit properties entirely different from those exhibited by the siliceous exchangers, and since there are many other uses for exchangers outside of the softening field, there is a need for standardization of terminology.

No specific system of nomenclature or terminology is proposed in this study, but the use of certain terms is recommended. The broad term ion exchange, with sub-headings of cation and anion exchange, are generally accepted. The materials themselves should be called exchange materials, exchange resins, or exchangers, rather than some of the other terms found in the literature, such as organolites, base exchangers, zeolites, and the like.

It is impractical to define at this point all the terms used in ion exchange. However, a few of the more common ones are listed below, taken from an extensive glossary given in The Resinous Reporter (322):

1. ADSORBENT: A synthetic resin possessing the ability to attract and to hold charged particles.

2. **ADSORPTION:** The attachment of charged particles to the chemically active groups on the surface and in the pores of an ion exchanger.
3. **ANALYTICAL GRADE:** A specially purified form of the Amberlite ion exchange resins for use in quantitative laboratory determinations.
4. **ANION:** A negatively charged particle or ion.
5. **ATTRITION:** The rubbing of one particle against another in a resin bed; frictional wear that will affect the size of resin particles.
6. **BACKWASH:** The counter-current flow of water through a resin bed (i.e., in at the bottom of the exchange unit, out at the top) to clean and reclassify the bed after exhaustion.
7. **BED EXPANSION:** The effect produced during backwashing: the resin particles become separated and rise in the column. The expansion of the bed due to the increase in the space between resin particles may be controlled by regulating backwash flow.
8. **BREAKTHROUGH:** The first appearance in the solution flowing from an ion exchange unit of unadsorbed ions similar to those which are depleting the activity of the resin bed. Breakthrough is an indication that regeneration of the resin is necessary.
9. **CAPACITY:** The adsorption activity possessed in varying degree by ion exchange materials. This quality may be expressed as kilograins per cubic foot, gram-milliequivalents per gram, pound-equivalents per pound, gram-milliequivalents per milliliter, etc., where the numerators of these ratios represent the weight of the ions adsorbed and the denominators, the weight or volume of the adsorbent.
10. **CATION:** A positively charged particle or ion.
11. **CHEMICAL STABILITY:** Resistance to chemical change which ion exchange resins must possess despite contact with aggressive solutions.
12. **COLOR-THROW:** Discoloration of the liquid passing through an ion exchange material: the flushing from the resin interstices of traces of colored organic reaction intermediates.
13. **CYCLE:** A complete course of ion exchange operation. For instance, a complete cycle of cation exchange would involve: regeneration of the resin with acid, rinse to remove excess acid, exhaustion, backwash, and finally regeneration.

14. **DEIONIZATION:** See deashing. Deionization, a more general term than deashing, embraces the removal of all charged constituents or ionizable salts (both inorganic and organic) from solution.
15. **EFFICIENCY:** The effectiveness of the operational performance of an ion exchanger. Efficiency in the adsorption of ions is expressed as the quantity of regenerant required to effect the removal of a specified unit weight of adsorbed material, e.g., pounds of acid per kilogram of salt removed.
16. **EFFLUENT:** The solution which emerges from an ion exchange column.
17. **ELUTION:** The stripping of adsorbed ions from an ion exchange material by the use of solutions containing other ions in concentrations higher than those of the ions to be stripped.
18. **EXHAUSTION:** The state in which the adsorbent is no longer capable of useful ion exchange; the depletion of the exchanger's supply of available ions. The exhaustion point is determined arbitrarily in terms of: (a) a value in parts per million of ions in the effluent solution; (b) the reduction in quality of the effluent water determined by a conductivity bridge which measures the resistance of the water to the flow of an electric current.
19. **FINES:** Extremely small particles of ion exchange materials.
20. **GRAINS PER GALLON:** An expression of concentration of material in solution. One grain per gallon is equivalent to 17.1 parts per million.
21. **HARDNESS:** The scale-forming and lather-inhibiting qualities which water, high in calcium and magnesium ions, possesses. Temporary hardness, caused by the presence of magnesium or calcium bicarbonate, is so called because it may be removed by boiling the water to convert the bicarbonates to the insoluble carbonates. Calcium sulfate, magnesium sulfate, and the chlorides of these two metals cause permanent hardness.
22. **HEADLOSS:** The reduction in liquid pressure associated with the passage of a solution through a bed of exchange material; a measure of the resistance of a resin bed to the flow of the liquid passing through it.

23. **HYDRAULIC CLASSIFICATION:** The rearrangement of resin particles in an ion exchange unit. As the backwash water flows up through the resin bed, the particles are placed in a mobile condition wherein the larger particles settle and the smaller particles rise to the top of the bed.
24. **HYDROGEN CYCLE:** A complete course of cation exchange operation in which the adsorbent is employed in the hydrogen or free acid form.
25. **LEAKAGE:** The phenomenon in which some of the influent ions are not adsorbed and appear in the effluent when a solution is passed through an under-regenerated exchange resin bed.
26. **PHYSICAL STABILITY:** The quality which an ion exchange resin must possess to resist changes that might be caused by attrition, high temperatures, and other physical conditions.
27. **REGENERANT:** The solution used to restore the activity of an ion exchanger. Acids are employed to restore a cation exchanger to its hydrogen form; brine solutions may be used to convert the cation exchanger to the sodium form. The anion exchanger may be rejuvenated by treatment with an alkaline solution.
28. **STATIC SYSTEM:** The batch-wise employment of ion exchange resins, wherein (since ion exchange is an equilibrium reaction) a definite endpoint is reached in which a finite quantity of all the ions involved is present. Opposed to a dynamic, column-type operation.
29. **SWELLING:** The expansion of an ion exchange bed which occurs when the reactive groups on the resin are converted from one form to another.
30. **TRETTER:** The suspended or buoyant state of resin particles in a column during backwashing; the bed is said to be in tetter when the velocity of the upflow solution maintains the particle in suspension.
31. **ZEOLITE:** Naturally-occurring hydrous silicates exhibiting limited base exchange.

B. History of Ion Exchange Materials

As previously indicated, the discovery of an acid-resistant organic cation exchange material in 1931 was a landmark in the evolution of ion exchange (428). This material was made from brown coal or lignite by Borrowman. Before this time, the only cation exchangers known were the natural zeolites, greensands, and synthetic inorganic siliceous zeolites, all of which deteriorated in the presence of acidic or alkaline solutions. Borrowman's discovery allowed operation in the hydrogen cycle, such that all cations in an aqueous solution could be removed by exchange for the hydrogen in the acid-regenerated cation exchanger. Such operation results in an acid solution.

In 1935 Adams and Holmes (1) discovered that a water-insoluble resin containing active amino groups could be used to adsorb or exchange acids from aqueous solutions. Thus, it became possible for the first time to use cation and anion exchange materials in series to remove all ionized electrolytes from an aqueous solution. The effluent water from such a "deionizing" or "demineralizing" process is equivalent to distilled water.

Under the basic Adams and Holmes patents, as sole licensees in the United States, The Resinous Products and Chemical Company began in 1939 to investigate the synthesis and production of ion exchange resins (322). Thus, strictly speaking, Amberlite IR-1, the cation exchanger, and Amberlite IR-3, the anion exchanger, were the first synthetic resin exchangers commercially produced in this country. Zeo-Karb, which was produced some time previously by Permutit, was not strictly a synthetic resin. It was made by sulfonating coal, following the basic discovery

of Borrowman.

It was soon apparent that many of the uses of ion exchangers in the process industries required special resins. Some of these applications required exchange reactions of precise selectivity. Other uses demanded exceedingly rapid exchange rates. Also, elution efficiency became a necessity in still other exchange reactions. As a result, in the period 1946 to 1948, numerous "tailor-made" resins appeared on the market. Each such resin had some specific property making it the best resin for a particular use. However, all resins are still basically similar.

Progress in the development of better and cheaper anion exchangers has been much slower than for cation exchangers. As a result, there are only two or three suitable anion exchangers on the market today, and these cost several times as much as the cation exchangers.

C. Methods of Classification

No satisfactory classification of all phases of ion exchange has been given. Most publications classify ion exchange according to (a) type of exchange reaction (197), or (b) type of industry utilizing the exchangers (324). The former is preferable, but is not extensive enough.

1. Classification by type of exchange reaction

In this study the existing classification systems are modified in order to make them more comprehensive and understandable. This modified system has been used in the literature review that follows in a later section. This classification consists of three headings for the "exchange

tools", followed by four headings for the ion exchange applications.

These are as follows:

- a. Theory of ion exchange.
- b. Techniques of operation.
- c. Properties of exchange materials.
- d. Cation exchange.
- e. Anion exchange.
- f. Combined cation and anion exchange.
- g. Exchangers as contact materials.

The theory of ion exchange includes mechanisms, exchange equilibria and rates. Techniques of operation involve column, batch, fluidized bed, revolving drum, and the like. Properties of exchange materials include both chemical and physical characteristics. The next two headings, cation and anion exchange are classified according to the following operations: preparations, separations, concentrations, and purifications. Combined cation and anion exchange includes deionization of water, and certain processes such as the purification of sugar juice solutions. Exchangers as contact materials are discussed regarding their use as catalysts, solid acids and bases, and the like.

2. Classification by industries

From a theoretical or development point of view, a classification by industries is not as satisfactory as one based on the type of exchange reaction. However, from a sales point of view, a knowledge concerning which industries are already using ion exchange can be very useful. A

partial list of industries using ion exchange is as follows (324):

- | | |
|------------------------------|-------------------------|
| 1. Alkaloids | 26. Minerals |
| 2. Amino acids | 27. Municipal waters |
| 3. Beverages (soft drinks) | 28. Non-ferrous metals |
| 4. Breweries | 29. Oils |
| 5. Biologicals | 30. Paper mills |
| 6. Canned juices | 31. Paints |
| 7. Canneries | 32. Petroleum producing |
| 8. Catalysts | 33. Pharmaceuticals |
| 9. Ceramics | 34. Pigments |
| 10. Chemicals, water soluble | 35. Powerhouses |
| 11. Chemicals, fine | 36. Precious metals |
| 12. Coke oven products | 37. Radium |
| 13. Corn syrup | 38. Railroads |
| 14. Dye works | 39. Restaurants |
| 15. Electroplating | 40. Resins |
| 16. Flavoring extracts | 41. Soap and glycerine |
| 17. Foods | 42. Steamboats |
| 18. Glue and gelatin | 43. Sugars, beet & cane |
| 19. Hospitals | 44. Tanneries |
| 20. Hotels | 45. Textile mills |
| 21. Hydroponics | 46. Varnishes |
| 22. Ice plants | 47. Vinegar and cider |
| 23. Insecticides | 48. Vitamins |
| 24. Laundries | 49. Wine |
| 25. Milk and milk products | 50. Wood distillation |

D. Review of Ion Exchange Literature

The number of publications on ion exchange is increasing at an accelerated rate. It is proposed to review here some of the more important ones, particularly those concerned with the industrial uses of ion exchange resins. This review is outlined as discussed previously under section C-1, classification by type of exchange reaction.

Many good reviews have been written on ion exchange. The Resinous Products & Chemical Company published a special issue of The Resinous Reporter (322). Walton, formerly with the Permutit Company, has also written an excellent review (421). Other discussions of theory and

applications of exchangers include articles by Myers (249, 251, 253), Wiklander (434), Kunin (197), and the staff of Chemical Engineering in a "Chem & Met" Report (72). The review by Kunin was included in the third annual Unit Operations Review of Industrial and Engineering Chemistry in early 1948, indicating that ion exchange is being accepted as a unit operation.

1. Theory of ion exchange

The theory of ion exchange will be considered under (a) mechanism, (b) equilibrium, and (c) rate.

a. Mechanism of ion exchange. An ion exchanger is an insoluble solid, which is at the same time a salt, acid, or base having exchangeable ions of its own. Furthermore, it has a highly porous structure or a very large exposed surface to permit these ions to get in and out.

It has been found (149) that the necessary conditions for cation exchange in zeolite mineral structures are the presence of negative charges in portions of the lattice framework and of multiconnected channels large enough for ionic migration. For example, in the mineral natrolite, $\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10}$, the negative ions are not single $\text{Al}_2\text{Si}_3\text{O}_{10}$ groups, but an endless three-dimensional framework in which this unit occurs repeatedly, like the pattern on wallpaper. Two out of every ten oxygen atoms bear a negative charge. This particular framework has three sets of parallel channels at right angles to each other, running through the whole crystal. The sodium ions are in

these channels, such that their positive charges exactly neutralize the fixed negative charges of the aluminosilicate framework. Because of the channeled structure the sodium ions can easily move out, but when they do move out, other positively charged ions must move in to take their place. Otherwise, the negative charges of the framework would not be neutralized.

The synthetic ion exchangers, such as the sodium aluminosilicate used in water softening, are far more porous than the natural zeolites. A particle of the exchanger, magnified sufficiently, would resemble a sponge whose walls were studded at intervals with fixed negative charges. To balance these charges, positive sodium ions hover in the internal passages and on the surface. In contact with a calcium chloride solution, calcium ions can enter and diffuse through the exchanger particle. For every calcium ion entering a particle, two sodium ions must leave to preserve electrical neutrality. It makes no difference what kind of positive ions are in the exchanger channels, so long as the positive charges always exactly balance the fixed negative charges of the exchanger.

It has been found by Jenny and Overstreet (172), and Walton (422) that, in general, the higher the valence of an ion and the less hydrated it is in solution, the more the ion is attracted to an exchanger. Correlations between equilibrium constant and ionic radius have been made by Nachod and Wood (260). Up to a point, the larger the unsolvated ion, the better it is held by an exchanger. There is very little heat of reaction in ion exchange, as indicated by temperature having very

little influence on the equilibrium of exchange. Ion exchange can be a very fast process, but, of course, the rate of exchange depends greatly on the porosity of the exchanger. For a porous, rapidly acting exchanger, temperature has little or no effect on the rate of exchange, showing that no great energy of activation is needed.

If the relative affinity for the resin or the concentration of the ion introduced in the solution phase is sufficiently greater than that of the ion originally combined with the resin, replacement will be nearly complete. Russell found that the effective concentration of an ion may be lowered by complex formation. Spedding and associates (378) point out that in the separation of the rare earths by ion exchange, after the pH is suitably adjusted, competition is set up for the rare earth ions between the citrate complexes and the active centers of the resin. Therefore, as the citrate solution washes the rare earths down the column, each rare earth ion is exchanged many times. Since the equilibrium constants for the rare earth citrate complexes vary slightly among the different rare earths, their rates of travel down the column differ sufficiently to lead to their separation.

Typical exchange reactions are given on Figures 1 and 2, applying particularly to the softening of water.

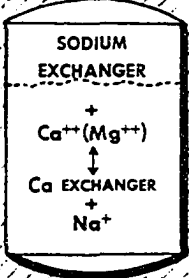
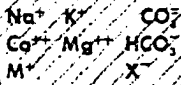
b. Equilibrium of ion exchange. Since ion exchange appears to be a typical reversible reaction, particularly for the alkali metal and alkaline earth cations, its equilibrium can be expressed by a modified mass action law. Walton (420) has utilized such an

EXCHANGE PROCESSES

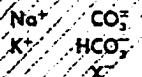
FIGURE I

SODIUM CYCLE

HARD WATER

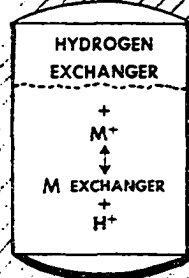
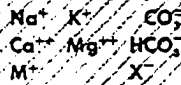


SOFT WATER

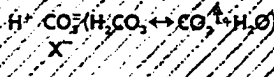


HYDROGEN CYCLE

RAW WATER

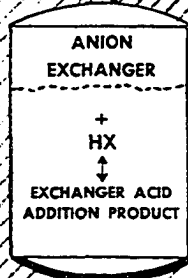


TREATED WATER

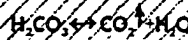


ACID ADSORPTION

INFLUENT



EFFLUENT



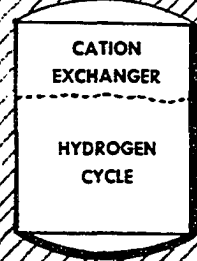
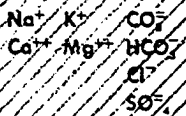
(M⁺ = Na⁺, K⁺, Ca⁺⁺, Mg⁺⁺, Fe⁺⁺⁺, etc. X⁻ = Cl⁻, SO₄²⁻, NO₃⁻, etc.)

REDUCTION OF ALKALINITY

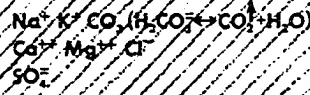
FIGURE II

(PARTIAL SOFTENING)

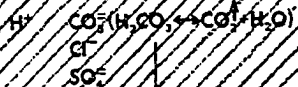
RAW WATER



TREATED WATER



EFFLUENT



BY-PASS

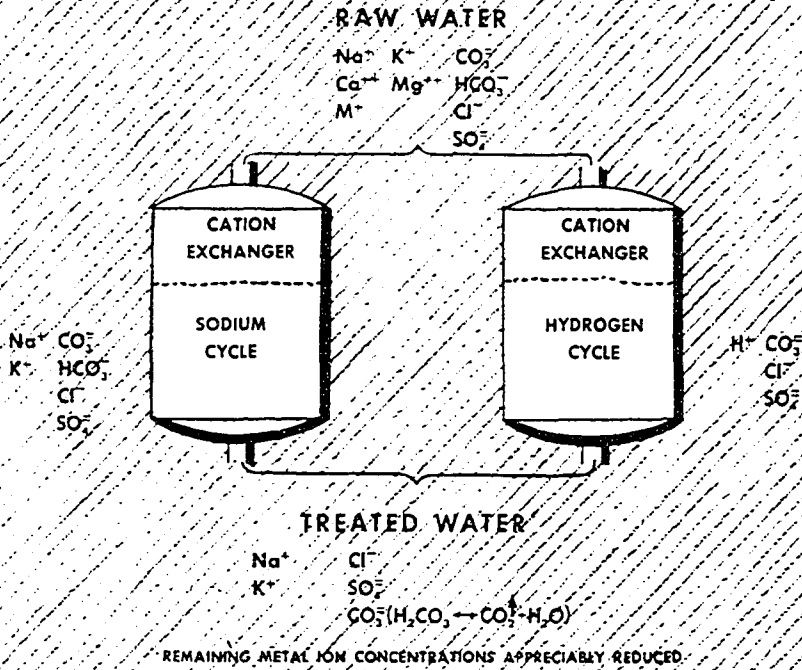
ALL METAL ION CONCENTRATIONS APPRECIABLY REDUCED

Fig. 1. Typical ion exchange operations.

Reproduced from: The Amberlites. Bulletin. p.4. Philadelphia. 1947.

REDUCTION OF ALKALINITY

FIGURE III
(COMPLETE SOFTENING)



DEIONIZING

FIGURE IV

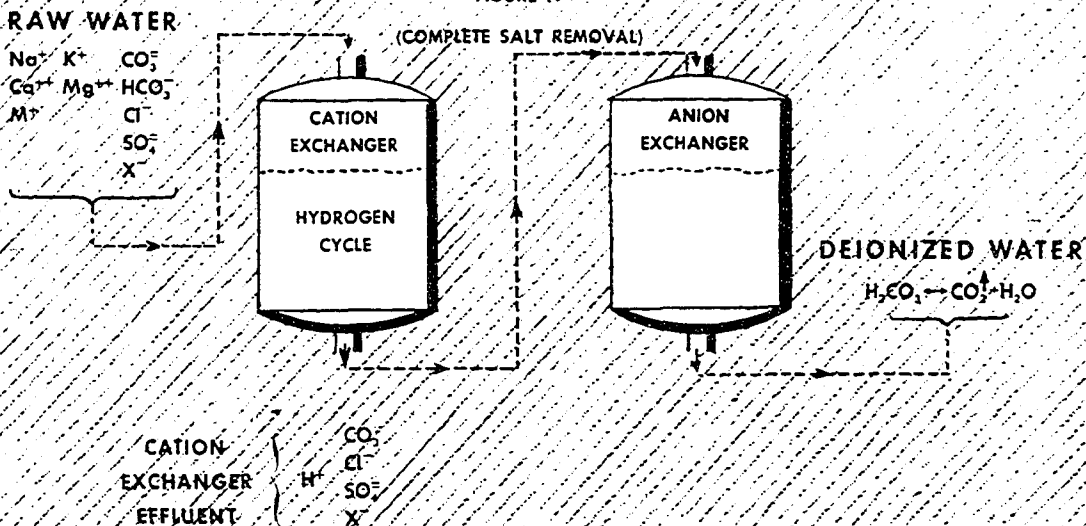
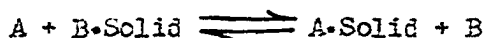


Fig. 2. Typical softening and deionizing operations.
Reproduced from: The Amberlites. Bulletin. p.5. Philadelphia. 1947.

equation, the derivation of which is as follows:

Defining A as the cations originally in solution and B as the cations originally in the exchanger solid, the reaction can be expressed by the equation



The equilibrium constant is then

$$K = \frac{B \cdot A_{\text{solid}}}{A \cdot B_{\text{solid}}} = (B/A)_{\text{soln.}} \cdot (A/B)_{\text{solid}}$$

and rearranging,

$$(A/B)_{\text{solid}} = K \cdot (A/B)_{\text{soln.}}$$

Assuming that the action law, where $(A/B)_{\text{soln.}}$ is to a power of one, will not hold, a modified, empirical equation can be used, where $(A/B)_{\text{soln.}}$ is to some power p. The resulting equation is $(A/B)_{\text{solid}} = K \cdot (A/B)_{\text{soln.}}^p$.

Walton's data for a Zeo-Karb cation exchanger, utilizing this modified equation, is shown on Figure 3. The law of mass action was nearly obeyed for sodium-potassium, sodium-calcium, and calcium-barium, but for sodium-hydrogen and calcium-hydrogen there was considerable deviation. These differences were explained by assuming the Zeo-Karb exchanger to consist of two or more solid acids of different dissociation constants. The exchanges were found to be reversible except for sodium-calcium, where there was hysteresis in approaching the equilibrium from opposite directions.

Myers and others (254) presented this equilibrium as adsorption isotherms. Bauman and Eichhorn (25) have applied the Donnan membrane equilibrium to cation exchange studies. Graham and Horning (128)

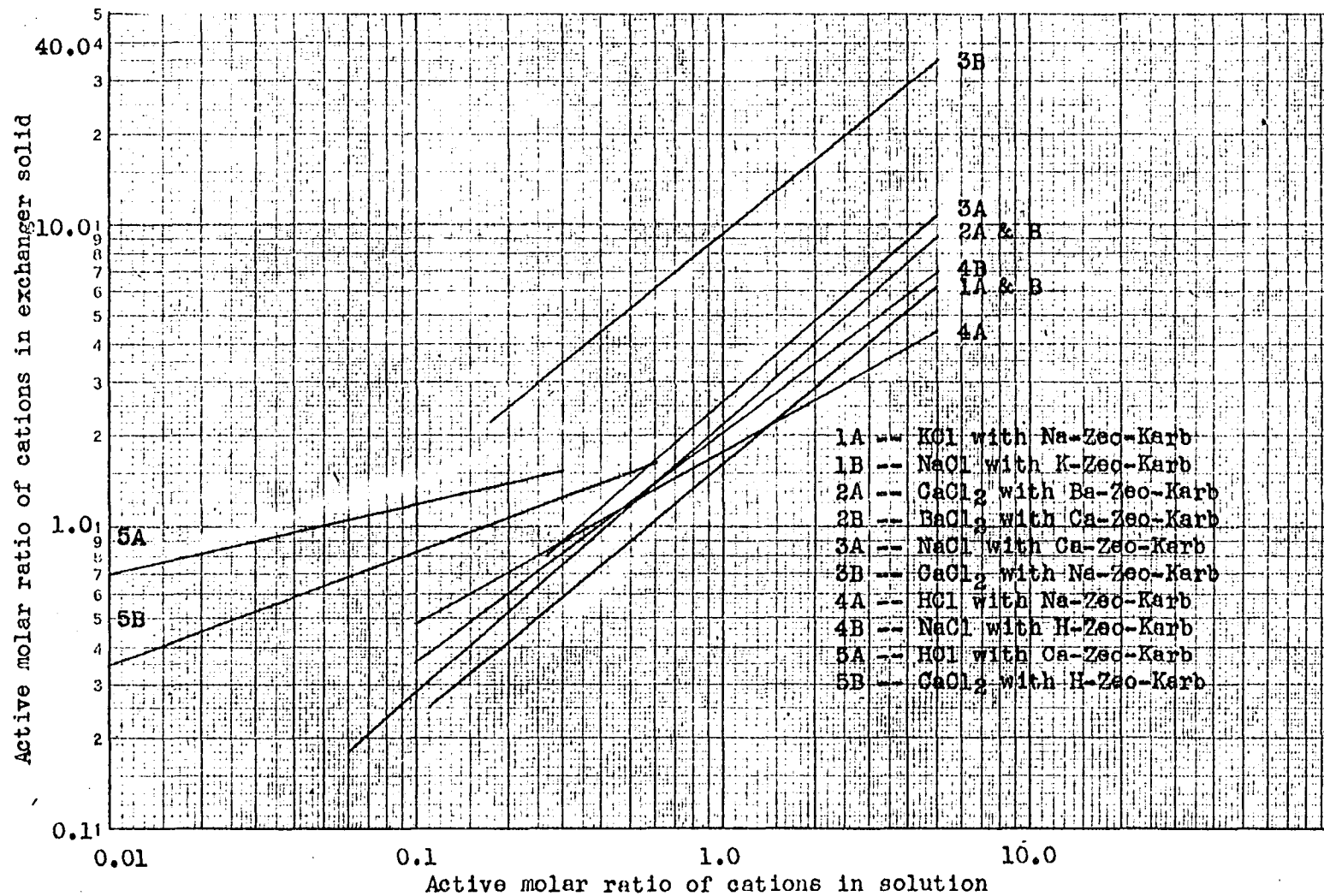


Fig. 3. Equilibrium of alkali metal and alkaline earth cations with Zeo-Karb.

have observed the phenomenon of anion exchange to occur with hydrous alumina. An excellent study on equilibrium in anion exchange resins has been made by Kunin and Myers (199). Other studies on ion exchange equilibrium include those by Beaton and Furnas (31), Boyd, Schubert and Adamson (50), Tompkins and Mayer (404), and Kielland (185).

c. Rate of ion exchange. The rate of ion exchange has been found to vary with both the type of exchanger and operating conditions. Nachod and Wood (259, 260) have reported that the rates for cation exchangers follow a second order bimolecular reaction. Their results are summarized in Table 1 for the rate constant in equation

$$k = \frac{2.303 t}{a-b} \ln \frac{b(a-x)}{a(b-x)}$$

where k = rate constant, $\text{meg.}^{-1} \times \text{l.} \times \text{min.}^{-1}$

a = ions originally in solution, meg.

b = ions from cation exchanger in maximum amount released when equilibrium is reached, meg.

x = ions exchanged at a given time, t , meg.

They also found that temperature had no effect on the rate of exchange in the 27 to 60°C. range. However, a temperature coefficient of 0.91 per degree was found in the 0 to 27°C. range. It was suggested that the latter may be due to a lower diffusion rate at the lower temperature.

Boyd, Adamson and Myers (51) have reported equations capable of describing the rate of exchange of the alkali metal cations by Amberlite IR-1. These equations were based on a diffusion mechanism

Table 1. Summary of experimental rate of ion exchange constants reported by Nachod and Wood

Exchange material	Commercial name	Source	Exchange reaction	Temp., °C.	Rate constant (k in meq. ⁻¹ x l. x min. ⁻¹)
Sulfonated coal	Zeo-Karb	The Permutit Company	H ₂ Z+Ca ⁺⁺	27 & 60	6.6 x 10 ⁻¹
Resin A	Amberlite IR-100	Resinous Products	H ₂ Z+Ca ⁺⁺	27	6.6 x 10 ⁻¹
Resin B	Ionac C	American Cyanamid Co.	H ₂ Z+Ca ⁺⁺	27	1.5 x 10 ⁻¹
Resin C	Duolite	Chemical Process Co.	H ₂ Z+Ca ⁺⁺	27	5.8 x 10 ⁻¹
Sulfonated coal	Zeo-Karb	The Permutit Company	H ₂ Z+Ca ⁺⁺	0	1.7 x 10 ⁻¹
Sulfonated coal	Zeo-Karb	The Permutit Company	CaZ+2H ⁺	27	8.4 x 10 ⁻¹
Sulfonated coal	Zeo-Karb	The Permutit Company	Na ₂ Z+Ca ⁺⁺	27	6.0 x 10 ⁻¹
Resin A	Amberlite IR-100	Resinous Products	Na ₂ Z+Ca ⁺⁺	27	2.9 x 10 ⁻¹
Greensand type	Zeo-Dur	The Permutit Company	Na ₂ Z+Ca	27	1.6
Synthetic siliceous gel	Decalso	The Permutit Company	Na ₂ Z+Ca ⁺⁺	27	2.6 x 10 ⁻¹
Resin D	De-Acidite	The Permutit Company	R ₃ N+HCl	27	1.6 x 10 ⁻³
Resin E	Amberlite IR-4	Resinous Products	R ₃ N+HCl	27	1.5 x 10 ⁻⁴

and a bimolecular chemical rate equation. The experimental results revealed two rate processes governing:

- a. With solutions 0.1 M in total electrolyte, or greater, the rate was controlled by diffusion in and through the adsorbent particle.
- b. With solutions 0.003 M or less the rate was limited by diffusion through a liquid film at the periphery of the particle.

Differential rates of exchange over one-half inch beds of zeolite were obtained by du Domaine, Swain, and Hougen (97). Other studies on ion exchange rates include those of Jenny (170), Davis (89), and Tompkins and Mayer (404).

2. Techniques of operation

The column method of operation generally is employed because it is continuous (except for regeneration), is controlled readily, and utilizes the exchanger to its fullest capacity. In a static system, such as the batch process, a definite end point is reached at which there is a finite quantity of all involved ions present in the solution being treated. In the column method of operation, a dynamic system, the countercurrent principle is effective because the ion concentration of the solution passing down the bed is constantly being reduced and, at the same time, the solution is contacting fresh exchanger. Thus, there is no possibility of reaching an equilibrium condition.

Although the column method of operation generally is employed in the usual applications of ion exchange resins, the batch method should be given careful consideration. It is much simpler and can often be carried out with existing equipment. Because head loss factors do not enter into consideration, the batch process has proved to be valuable in the treatment of viscous solutions. It should be remembered, however, that the batch process is a static process, such that at equilibrium a finite quantity of all the ions involved will remain.

Theoretically, the ion exchange reactions should take place with equal efficiency whether the solution is passed upflow or downflow through the column bed. In practice, however, downflow operation is generally preferable because any flow rate, up to a practical maximum, can be used with no danger of carrying the exchange resin out of the column.

Continuous operation of a column, without a period of time for regeneration, was studied as far back as 1921 by Nordell (266, 267, 268). He obtained patents upon apparatus, which in general were too unwieldy and elaborate to be put into practical use. A comparison can be made with fluidized solids beds, which had their beginning in the 1910 to 1920 period, but were not made practical until their recent applications in petroleum cracking.

Various techniques for regeneration of the ordinary type bed have been the subject of many patents. These have been concerned with the regeneration of cation exchangers (133, 256), anion exchangers (41, 256), and deionizing units (98, 307).

An interesting technique was described recently (322), which involves a mixed-bed deionization. Two exchangers, one containing strongly acidic groups and the other strongly basic substituents, were mixed intimately in definite ratios to remove both cations and anions simultaneously from solution. The two exchangers were then separated by ordinary hydraulic classification, through a difference in density. No data have been given, from which one might evaluate the economics of this process. If it is found to be practical from an economic standpoint, it would have an important advantage by removing all salts from solution at a neutral pH of seven. Hence, both acid- and alkali-sensitive systems can be treated easily for the removal of all ionized constituents.

Various other types of equipment have been used or proposed for ion exchange operations. For example, Pattock and Meier have used a revolving drum (282). Cochrane (76), Liquid Conditioning Corporation (211), The Permutit Company (286), and others, furnish various types of equipment for ion exchange installations.

3. Properties of exchange materials

The types of cation and anion exchangers which have been described in the literature can be classified on the basis of the functional group. These have been classified by Myers (251), as listed in Table 2.

Table 2. Types of cation and anion exchangers

Functional group	Principle region of application
	Acid resins:
—SO ₃ H (nuclear)	Very low pH
—CH ₂ SO ₃ H	Low pH
—COOH	Neutral solutions
—OH (phenolic)	High pH
—CH ₂ OH	Not yet investigated
—CH ₂ SH	Not yet investigated
	Basic resins:
—NH ₂ (aromatic)	Acid solutions
—NH ₂ (aliphatic)	Acid and neutral solutions
≡NH (aromatic and aliphatic)	Not fully investigated
≡N (aromatic and aliphatic)	Not fully investigated

It became apparent from early work on the synthetic-type exchangers, most of which were modifications of the Adams and Holmes resins, that chemical and physical properties could be varied to give certain desired results. Hence, the various functional groups in Table 2 were investigated with respect to ion exchange selectivity and adsorption on ions over the entire pH range. Physical and chemical properties, in addition to mechanical means in processing, were utilized to give good chemical and mechanical stability.

The Resinous Products and Chemical Company have introduced three new ion exchangers, which revolutionize exchange concepts (322). Amberlite IR-120 is a strong-acid type, bead-form cation exchanger, which has an exchange capacity four times that of the old Amberlite 100. It also possesses outstanding chemical stability and high temperature resistance. It will find application in processes involving treatment

of solutions of high solids content, because of its high capacity. The second new exchanger is Amberlite IRC-50, which is the first exchanger to derive its exchange activity entirely from weakly-acidic carboxylic groups. It gives a very high chemical efficiency in regeneration, although its rate of exchange is exceedingly slow. The third new development resulted in the strongly-basic anion exchanger, Amberlite IRA-400, which behaves as solid caustic with only its hydroxyl ions in solution. The extraordinarily rapid rate of exchange of this resin, in addition to its ability to remove anions from acid, neutral, and even mildly-alkaline solutions, greatly expands the field of application for ion exchange methods.

Another development that has attracted as much attention as the new Amberlite resins is the new beaded form of exchangers. The spherical shape and uniformity of bead size provide a more evenly-graded bed classification than has been possible with the standard granular types. The formation of fissures or channels within the bed is less likely to occur, thereby assuring a maximum exchange effectiveness. Losses of exchange resin by attrition are greatly reduced. Also, increased capacity per unit volume is usually obtained, due to the large number of capillaries existing in the spherical bead. There is every reason to believe that any exchanger, which involves a gel or resin stage in its formation, could be made into a beaded form. One method of bead formation is illustrated by that used for TCC catalyst, which is an activated alumina contained in silica gel (301).

Figures 4 and 5 summarize the properties of ion exchange resins, and illustrate the importance of this phase of the ion exchange field. Figure 5 shows a typical apparatus, which may be used for either a cation or an anion exchanger.

E. Uses of Cation Exchange Materials

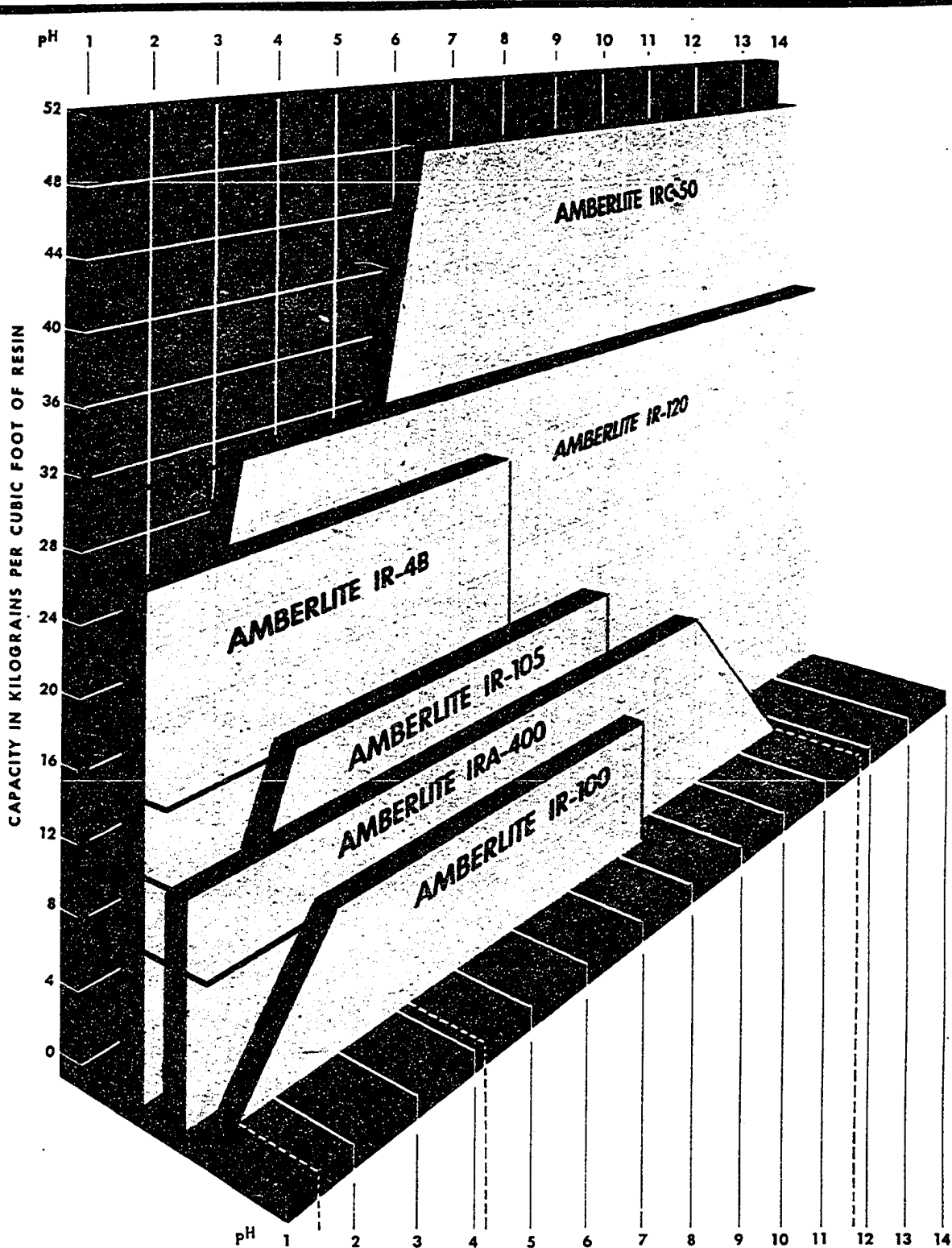
Cation exchange has been known for nearly a century. The first important application was that of softening water. This use is still the major one for cation exchange materials, but a great diversification of application followed Borrowman's discovery (428) in 1930 that a suitable organic cation exchange resin could be made from brown coal or lignite. This was the first acid-resistant exchanger, which made possible the use of a hydrogen cycle by acid regeneration.

1. Preparations by cation exchange

Preparations by means of the mechanism of cation exchange can be classified into two groups, namely, preparation of chemical products and modification of various materials.

a. Preparation of chemical products. Early attempts of preparing chemicals by cation exchange centered on salts. This was due to fact that hydrogen cycle operation and also anion exchange were not yet discovered.

The more recent applications usually have taken advantage of the fact that acids are formed by exchange with a cation exchanger in the



OPERATING CAPACITY IN RELATION TO pH

No longer is pH a factor that limits the usefulness of ion exchange. Cation adsorption was once confined to the pH range 1.5 to 8.5; now cation exchange may take place in the extended pH range of 1.5 to 14. Anion exchange, once practical in media of pH 1 to 7, now may be adapted to the wide range of 1 to 11. In this 3-dimensional representation, the capacities of the cation exchangers are compared at a constant regeneration level of approximately 0.6 pounds of appropriate regenerant per kilograin of cations adsorbed. The capacities of the anion exchangers are based on regeneration levels of 7.5 pounds of sodium carbonate per cubic foot of Amberlite IR-48 and 12 pounds of sodium hydroxide per cubic foot of Amberlite IRA-400.

Fig. 4. Operating capacity of the amberlites in relation to pH.
Reproduced from: The Resinous Reporter 9, No. 4, 12 (1948).

AMBERLITE SYNTHETIC RESIN ION EXCHANGERS

CHARACTERISTIC	IR-100	IR-105	IR-120	IRC-50	IR-48	IRA-400																																																																																				
Density (lb./ft. ³ as shipped).....	48	47	48	46	32	42																																																																																				
Moisture Content (%).....	43-45	40-50	35-45	30-40	40-45	45-55																																																																																				
Effective Size (mm.).....	0.35-0.45	0.4-0.6	0.4-0.6	0.35-0.50	0.4-0.55	0.38-0.60																																																																																				
Uniformity Coefficient (max.).....	1.8	1.7	2.0	2.0	2.0	2.0																																																																																				
Screen Grading (wet).....	20-40 mesh	20-50 mesh	16-50 mesh	16-50 mesh	20-50 mesh	20-50 mesh																																																																																				
Typical Wet Screen Analysis.....	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>3.2</td></tr> <tr><td>30</td><td>10.1</td></tr> <tr><td>40</td><td>17.5</td></tr> <tr><td>50</td><td>24.0</td></tr> <tr><td>60</td><td>29.2</td></tr> <tr><td>70</td><td>33.0</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	3.2	30	10.1	40	17.5	50	24.0	60	29.2	70	33.0	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>10.0</td></tr> <tr><td>30</td><td>45.8</td></tr> <tr><td>40</td><td>31.2</td></tr> <tr><td>50</td><td>12.8</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	10.0	30	45.8	40	31.2	50	12.8	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>25.0</td></tr> <tr><td>30</td><td>48.0</td></tr> <tr><td>40</td><td>27.0</td></tr> <tr><td>50</td><td>0.0</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	25.0	30	48.0	40	27.0	50	0.0	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>17.0</td></tr> <tr><td>30</td><td>45.0</td></tr> <tr><td>40</td><td>31.5</td></tr> <tr><td>50</td><td>5.5</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	17.0	30	45.0	40	31.5	50	5.5	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>22.0</td></tr> <tr><td>30</td><td>42.0</td></tr> <tr><td>40</td><td>31.0</td></tr> <tr><td>50</td><td>2.0</td></tr> <tr><td>60</td><td>1.0</td></tr> <tr><td>70</td><td>1.5</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	22.0	30	42.0	40	31.0	50	2.0	60	1.0	70	1.5	<table border="1"> <tr><th>Size of Mesh</th><th>% Retained</th></tr> <tr><td>10</td><td>0.0</td></tr> <tr><td>20</td><td>2.0</td></tr> <tr><td>30</td><td>10.0</td></tr> <tr><td>40</td><td>25.0</td></tr> <tr><td>50</td><td>40.0</td></tr> <tr><td>60</td><td>17.0</td></tr> <tr><td>70</td><td>12.0</td></tr> </table>	Size of Mesh	% Retained	10	0.0	20	2.0	30	10.0	40	25.0	50	40.0	60	17.0	70	12.0
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pH Range.....	1-8.5	1-8.5	1-14	1-14	1-14	1-14																																																																																				
Maximum Temperature, °F.....	120	120	200	200	95	95																																																																																				
Minimum Bed Depth* (inches).....	24	24	24	24	24	24																																																																																				
Regeneration Concentration.....	10% NaCl 2% H ₂ SO ₄	10% NaCl 2% H ₂ SO ₄	10% NaCl 2% H ₂ SO ₄	20% NaCl 1% NaOH 1% H ₂ SO ₄	2-4% Na ₂ CO ₃	5-10% NaOH																																																																																				
Regeneration Level: lb./ft. ³ of resin.....	1.8	1.8	1.8	5.0-15.0	7.5	12.0																																																																																				
Regenerant.....	NaCl	NaCl	NaCl	NaCl, NaOH, H ₂ SO ₄	Na ₂ CO ₃	NaOH																																																																																				
Exchange Capacity (kg./ft. ³).....	2.0	2.0	3.2	20-100	28	12																																																																																				
Rinse Requirements (gal./ft. ³).....	40	40	50	30	75	40																																																																																				

* In some instances, shallower beds have been used successfully by flow rate adjustment. For all resins a Regeneration Flow Rate of 1 gal./min./ft.³ and a Service Flow Rate of 2 gal./min./ft.³ are recommended.

† True Void Volume (%) = 100 * [1 - (Volume of water actually displaced by a given quantity of resin) / (Volume occupied by resin bed backwashed and in place in column)]

NOTE: In a summary of this type it is impossible to provide complete data on the operational performance of these resins. Prior to actual investigation of these products, the individual technical bulletins should be consulted.

Fig. 5. Summary of properties of the amberlite ion exchange resins. Reproduced from: The Resinous Reporter 9, No. 4, 13 (1948).

TYPICAL AMBERLITE UNIT

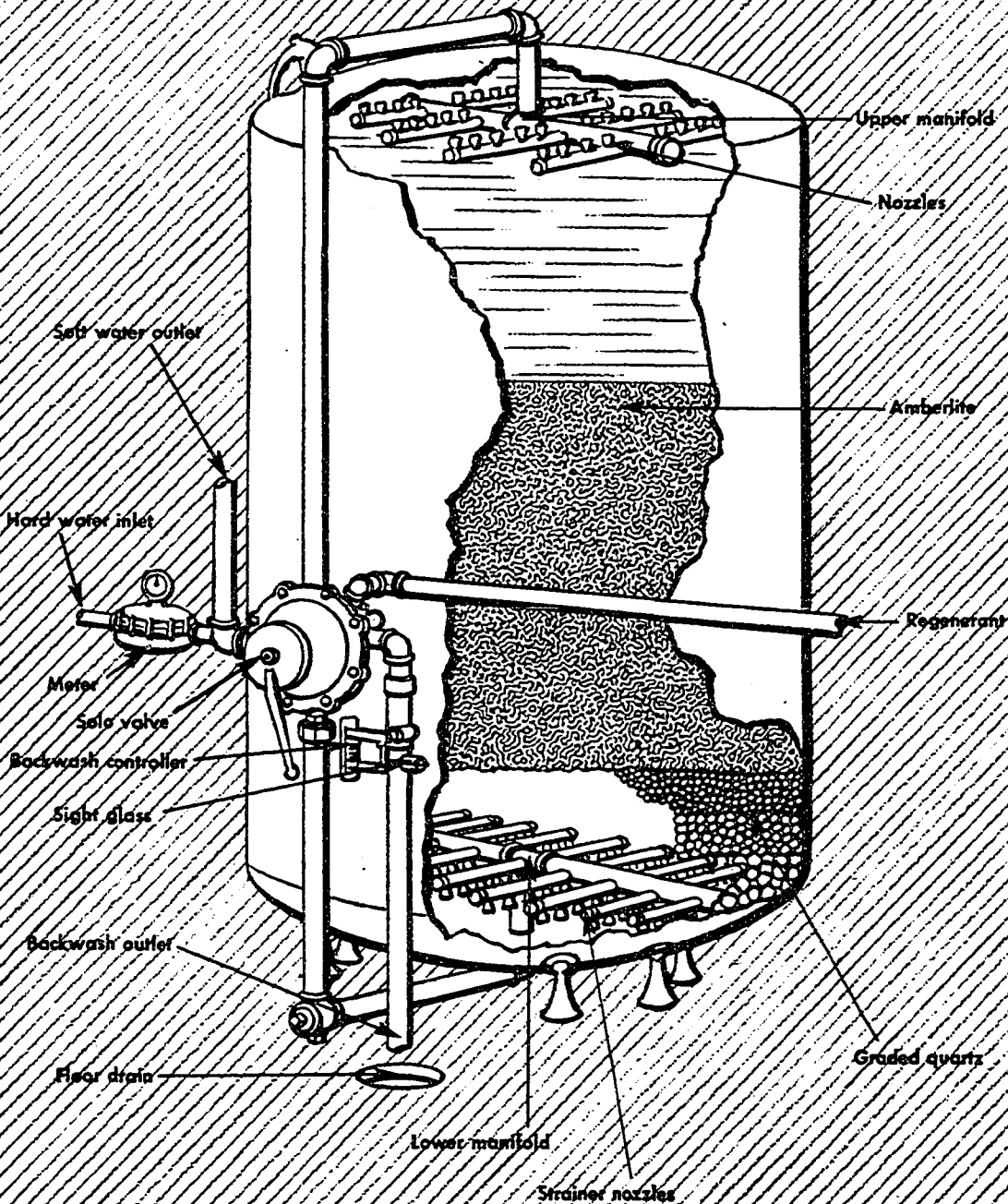
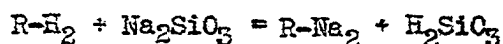


Fig. 6. Typical apparatus for ion exchange operations.
Reproduced from: The Amberlites. Bulletin. p.3. Philadelphia. 1947.

hydrogen cycle. Attempts to produce useful acids were made by Mathers and Yancy (225). However, the resulting solutions were more dilute than desirable, and the acid ions used in activating the exchanger contaminated the product solution. The esterification of butanol and oleic acid has been accomplished in the presence of an acid-form cation exchange resin as catalyst (389).

A method of preparing and purifying hydrous oxide sols has been devised by Bird and Ryznar of National Aluminate Corporation (343). Using a cation exchange material, Na^+ , Ca^{++} , Mg^{++} , Al^{+++} , and other metal ions are removed and replaced with an H^+ ion. For example, relatively pure silica sols are obtained by passage of a dilute solution of sodium silicate through an acid-regenerated bed. The sodium ion in the sodium silicate is then replaced by a hydrogen ion to give a silicic acid sol in the effluent:



In plant operation, solutions of sodium silicate, having as high as 3.5 per cent silica, have been passed through the cation exchange resin without difficulty with a flow rate of about one gallon per square foot of cross-sectional area per minute. A typical analysis of the effluent obtained is 2.5 per cent silica and 0.05 per cent sodium oxide.

By allowing some sodium silicate to pass into the effluent after the bed capacity is exhausted, the sol can be stabilized by the alkalinity from the sodium silicate. It can then be concentrated by evaporation to a silica content as high as 20 per cent, having only 0.37 per cent Na_2O , with no danger of gel formation.

Other sols can be prepared in this same manner. The soluble salt of any insoluble acidic oxide can be used to prepare the corresponding oxide sol. Ammonium metavanadate solution gives colloidal vanadium pentoxide. A tungstic oxide sol containing 99 per cent tungstic acid and 0.5 per cent sodium oxide can be made. Dilute ferric sulfate solution, passed through an alkali-regenerated anion exchanger, gives a red ferric oxide sol, which is described in more detail under the uses of anion exchangers.

An interesting example showing the effect of different types of cation exchange resins on the final composition of a sol was obtained by passing a solution of sodium molybdate through the acid-regenerated beds of two different materials. When a clear colorless solution of sodium molybdate was passed through a sulfonated phenol-formaldehyde resin, the effluent contained colloidal yellow molybdic acid, but when the sodium molybdate solution was passed through a sulfonated coal exchanger, the effluent contained colloidal molybdenum blue complex. Since there is some question as to the chemical constitution of this molybdenum blue complex, closer study of the reactions may lead to a more complete understanding of this compound as well as the exchanger itself.

The preparation of other chemicals have been described, but in general do not appear economically feasible. For example, Yen (440) described a Chinese process for the preparation of ammonium sulfate from wine, apparently for use as a fertilizer.

b. Modification of materials. A typical example of how materials are modified by cation exchangers is the readjustment of salts in milk. Lyman, Browne and Otting (216) reduced the calcium content of milk by cation exchange to give an edible product of improved digestibility for infants. A patent by Lyman (215) has been assigned to M & R Dietetic Laboratories, Inc. on this process. A specific case was described, whereby a skim milk containing 9.40 per cent total solids, 0.1919 per cent CaO and 0.2403 per cent P_2O_5 , was passed through an inorganic cation exchanger. The effluent contained 0.1289 and 0.1671 per cent CaO and P_2O_5 , respectively. The ratio of CaO to P_2O_5 was reduced from 0.797 to 0.771, indicating that most of the removal of ions was due to adsorption, rather than true ion exchange.

Finely divided inorganic zeolitic exchanger has been used to reduce the viscosity of drilling muds (68). Other modifications include salt adjustments in the blood (393) and removal of sodium from the human body (92).

2. Separations by cation exchange

The separation of cations is based on differences in those properties that affect cation exchange. These properties include valence, size, and type of ion or complex ion, and the characteristics of the cation exchange material.

a. Separation of cations from anions. In most applications of cation exchange materials, certain cations are being separated from

anions. For example, even in water softening, calcium and magnesium are being separated from the anions, such as chlorides, of the water. However, this section of ion exchange classification is being limited to those cases where certain specific cations are being separated from certain anions.

Ion exchangers are useful analytical tools for the separation of cations from anions. A notorious difficulty in quantitative analysis is the exact determination of sulfate in solutions which also contain iron or aluminum. Barium sulfate precipitated from such solutions is always contaminated with iron or aluminum, while if the trivalent metals are precipitated first as hydrous oxides, these hydrous oxides carry down sulfate with them. If the solution is passed through a hydrogen ion exchanger (421), the metal ions are retained by the exchanger, and the sulfate passed on as sulfuric acid, in which the sulfate ion is easily and accurately determined.

Many other analytical separations are possible along similar lines. Samuelson of Sweden has been very active in this field, and has published numerous papers in the last few years (340, 341, 344, 345, 346, 347, 348, 349, 350, 351, 352, 353, 354, 355).

b. Separation of cations by differences in ionic forces. Since ions of higher valence are retained more firmly than ions of lower valence under identical conditions, it is general practical to effect a separation of such ions by means of cation exchangers. The usual water softening process illustrates this, whereby divalent calcium and magnesium ions displace monovalent sodium ions from the exchanger.

Cations of strong bases can be separated from those of weaker bases. One of the most interesting developments is in the separation of amino acids. These can be anions, cations, or neutral, depending on the pH. For example, using both cation and anion exchangers, it is fairly simple to separate lysine, $\text{H}_2\text{N}^+(\text{CH}_2)_4\text{CHNH}_2\text{COOH}$, glycine, $\text{H}_2\text{N}^+\text{CH}_2\text{COOH}$, and aspartic acid, $\text{HOOC}\cdot\text{CH}_2\text{CHNH}_2\text{COOH}$ from one another. In approximately neutral solution (pH 6) the first forms cations, the third anions, and the second neutral molecules (actually zwitterions, $\text{H}_3\text{N}^+\text{CH}_2\text{COO}^-$). Pure amino acids produced in this way from protein hydrolyzates are used for intravenous feeding. Englis and Fiess (104) also studied the exchange and separation of glycine, leucine norleucine, phenylalanine, tryptophan, hydroxyproline, glutamic acid, asparagine, and lysine hydrochloride. Laboratory data for another process for the separation of amino acids (402) are given in Figure 7.

Considerable work has been done on the separation of metal ions. Data have been given (249) on the removal of iron from aluminum sulfate solutions. A tower containing 800 grams of Amberlite IR-1 (sodium form) was used for purification of a 33° Baume alum solution (approximately 25 per cent aluminum sulfate) containing 125 mg. ferric oxide per 100 ml. The first 250 ml. portion of the effluent contained no alum, since it was required to convert the sodium derivative of the resin to the aluminum salt. The first portion of effluent contained only 60 mg. Fe_2O_3 per 100 ml., compared with the 125 mg. per 100 ml. initial value. The amount of Fe_2O_3 increased to 77 mg. per 100 ml. solution after 1500 ml. effluent, and to 120 mg. per 100 ml. solution

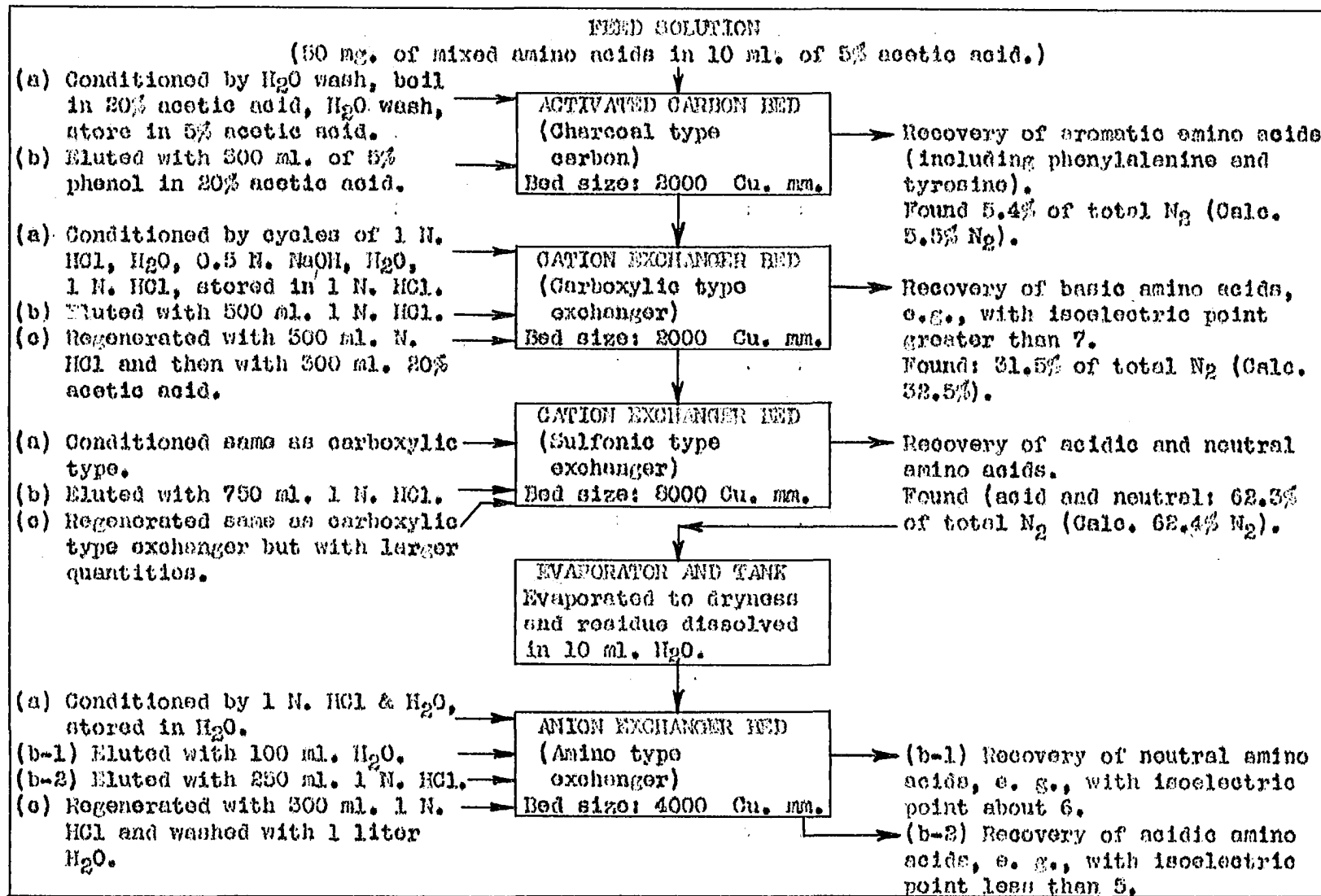


Fig. 7. Separation of amino acids by ion exchange.

after 3300 ml. effluent. Thus, a high efficiency of iron removal was effected, when the concentration of aluminum ion is considered.

Another interesting application is the removal of copper from rayon wastes, in which the concentration of ammonium salts is high. The waste contains 4.8 p.p.m. copper (as CuSO_4) and 2750 p.p.m. ammonia (as $(\text{NH}_4)_2\text{SO}_4$) at a pH of approximately five. Using the static exchange technique, rather than column operation, the copper content was reduced from 4.8 to 2.0 p.p.m., even in the presence of the high ammonium ion concentration. However, the capacity of the resin for copper under these conditions was only one third of that obtained when the concentration of other salts is negligible. Further pilot plant studies are being made upon this process, which appears to be economically practical.

A study of the recovery of zinc from mine waters has been made, but the economics of the process were not favorable. The mine water studied had the following composition (in p.p.m.): zinc 137.0, calcium 147.7, magnesium 17.7, iron and aluminum 8.0, copper and lead less than 0.1 each. Column studies with Amberlite H-IR-1 (254) showed that there was a slight preferential adsorption of calcium over zinc, as indicated by 3.76 per cent calcium and 3.21 per cent zinc in the first section of the column. However, such a slight preferential adsorption would not be economical in practice.

Some separation is possible between certain metal ions having the same valence but different atomic weights. An I.G. Farbenindustrie patent (249) gives data on the partial separation of copper and zinc.

Kozak and Walton (192) have reported attempts to separate metal ions by cation exchange. Their best results were obtained with an equimolar solution of silver and copper sulfates. After passing the solution containing the two salts through the exchanger, they were eluted by 2M sulfuric acid. In the first 600 ml. of effluent, 79.9 mol per cent of the total silver and copper was the latter, whereas the next 700 ml. of effluent contained 97.8 per cent silver and 2.2 per cent copper. Interesting results were obtained with equimolar solutions of zinc and cadmium sulfates. When a solution containing each salt in 0.01M strength was used, the eluted solution was slightly richer in zinc, having 52.6 per cent zinc and 47.4 per cent cadmium on a molar basis. When the strength of each salt is 0.1M, the eluted solution is definitely richer in cadmium, containing 64.1 per cent cadmium and 35.9 per cent zinc on a molar basis.

Reid (309) has proposed a multistage system, utilizing cation exchange and regeneration in each stage, for the concentration of radium in a radium-barium mixture. Using a solution consisting of a mixture of 0.15M HCl, 0.046M BaCl₂, and 0.104 x 10⁻⁸M RaCl₂, equilibrium values were obtained. It was predicted from these values that the radium-barium ratio of a solution could be increased from 2 x 10⁻⁶ to 100 with less than 200 stages, and an additional stripping section of 100 stages would save all but 0.01 per cent of the radium. It appears that a considerable concentration of radium can be effected by this method, but it is doubtful if a radium-barium ratio of 100 could be achieved. It would be unusual for two cations, having such similar

properties as do radium and barium, to have an ion exchange equilibrium curve allowing such a high radium-barium ratio. A close examination of Reid's experimental data, run only on low radium-barium ratios, indicates that his equilibrium "constant", calculated on actual concentrations rather than "active concentrations", is actually varying in value. Reid made a good suggestion that possibly a combination of the ion exchange method for lower radium concentrations with a fractional crystallization method for the higher concentrations would be practical for radium refining.

c. Separation of cations by complex ion elution. By lowering the effective concentration of an ion by means of complex formation, this ion can then be effectively replaced from its resin by a relatively dilute solution of a second cation. Spedding and co-workers (378, 379) and others (143, 404) have utilized a citrate complex for the separation of the rare earths. Conditions were given for the separation of cerium and yttrium by adsorption on Amberlite IR-1 and elution with 5 per cent citric acid-ammonium citrate solutions. Although this process is probably too expensive for the usual industrial applications of ion exchange, it is excellent for a number of difficult separations involving valuable products.

3. Concentrations by cation exchange

Dilute solutions of metal salts can be passed through a cation exchanger bed to remove the metallic ions. Regeneration by rather strong acid results in a definite concentrating effect with respect to the metal.

Pattock and Meier (282) have patented a process for working up complex ammoniacal solutions of metals, based on German processes and economy. They found that dilute ammoniacal copper, zinc, cadmium, or silver solutions could be worked up economically in Germany by effecting the regeneration with a pH above 4.5. This method was found to be advantageous for ammonium salts of volatile acids as regenerating agents.

Dilute solutions of Schweizer's reagent were passed through a cation exchanger to remove the copper as tetra-, tri- or diamines. A large portion of the copper was then eluted with an ammonium carbonate solution of 20 to 40 per cent (pH of 9.5), with the amine complex being exchanged for ammonia. It was found that partial regeneration by means of ammonium sulfate or chloride, instead of the ammonium carbonate, made possible the precipitation of basic copper sulfate or chloride.

Certain organic materials can be concentrated by the use of cation exchange materials. For example, Bennett and Nees (38,262) have assigned patents to The Great Western Sugar Company for the recovery of betaine and betaine salts from sugar beet wastes.

4. Purification by cation exchange

Water for domestic and industrial use continues to be the most important product purified by means of cation exchange. Olson (274, 275), Streicher (387) and others have discussed the use of cation exchangers for municipal water supplies. The advantages and disadvantages of processes for the softening of industrial and boiler feed waters have been discussed adequately by Applebaum (8), Bird (42), Burrell (62),

Brown (58), Classen (74), Clark (73), Collins (77) and some twenty to thirty other men.

A large number of chemical processes utilize cation exchange to purify valuable products. In the pharmaceutical field, these include the removal of nickel from sorbitol (321), preparation of cinchona alkaloids (13), recovery of nicotine from tobacco wastes (398), and barium salts from adenosine triphosphate (297). Patents have been issued for the removal of salts from petroleum oils to Jones (176) and Schutze (361) of Standard Oil Development Company. Dahlberg (84) patented a process (assigned to The Great Western Sugar Company) for purifying a portion of a sugar juice solution, which in turn is blended with the remaining portion of original alkaline juice. Usually, however, an anion exchanger is used in conjunction with the cation exchanger for purification of sugar juices, instead of the blending operation proposed by Dahlberg.

F. Uses of Anion Exchange Materials

The experimental work and industrial applications of anion exchange materials are not nearly as extensive as for the cation exchangers. This is due chiefly to two factors: (a) Anion exchange is a relatively new principle, and (b) the anion exchange resins are two to four times greater in price.

It should be noted that most investigators have considered anion exchange to be an adsorption mechanism. They assumed that the acid salt

of the exchanger was formed in a manner analogous to the formation of salts by organic amines. However, Kunin and Myers (199) have provided experimental evidence supporting the theory that ion exchange is the true mechanism. This supports the earlier conclusions of Wiklander, Griessbach, and Jenny that true anion exchange can occur.

1. Preparations by anion exchange

The anion exchange resin can be used as a "carrier" for various acids. For example, Gaddis (113) has used an anion exchanger to hold hydrogen sulfide until needed for the determination of Group II ions in qualitative analysis. Likewise, these exchangers have been used to provide nitrates, and the like, to nutrient solutions being used for plant nutrition.

The salts of amines can be modified by anion exchange resins. In the process for manufacturing streptomycin, one step involves converting the sulfate salt of streptomycin to the chloride.

2. Separations by anion exchange

The separation of acids can be achieved by anion exchange. This is analogous to the separation of cations by the principle of cation exchange. Hesler and Behrman have assigned a patent to Infilco, Inc. (153) for a method of freeing a lactic acid solution from sulfuric and hydrochloric acids. Saturating the anion exchanger with lactic acid before beginning the treatment of impure acid solution provides the proper equilibrium conditions in the bed for optimum purification.

A wide range of organic acids may similarly be purified and freed from relatively smaller amounts of stronger inorganic acids. For example, citric acid can be prepared from lemons by neutralizing the citric acid in the juice with lime, separating the calcium citrate, treating the calcium citrate with sulfuric acid to set free citric acid again, and then freeing the citric acid of the excess sulfuric acid by anion exchange.

3. Concentrations by anion exchange

A number of valuable materials have been concentrated by means of anion exchange. Nachod has assigned a patent to The Permutit Company (257), which described a process for the recovery of precious metals. Gold and the metals of the platinum group (iridium, osmium, palladium, rhodium, and ruthenium) were removed from dilute solutions as complex anions by means of anion exchange resins. The metal was then recovered by either burning the resin or eluting with an alkali. When the latter method is used, a substantial part of the metal is tenaciously retained by the anion exchanger.

4. Purifications by anion exchange

One of the most important applications of anion exchange is the removal of traces of acids in the purification steps for valuable products. In the pharmaceutical field, acidic materials have been removed from vitamin B solutions (218) and d-tubocurarine (24). The first process is described in a patent assigned to Vico Products Co. and the latter in a patent assigned to E. R. Squibb & Sons. Many

similar applications are described in the literature.

G. Combined Uses of Cation and Anion Exchange Materials

The combined use of cation and anion exchange materials involves the formation of acids by cation exchange in the hydrogen cycle in the first step, followed by the acid removal in the second step. Thus, with raw water as the feed, a completely "deionized" or "demineralized" water is obtained. It is equivalent to distilled water in quality. Bachman (20), Morrison (239), Riley (332), Lindsay (210), and others, have discussed the economics of deionized water. In general, it is very much cheaper than the standard multiple-effect evaporation.

The deionization process has been applied to the process industries. Intensive interest has been centered upon this process in the sugar industry. With present standard methods of purifying sugar juice, both cane and beet, it is possible to obtain a juice approximately 90 per cent pure and to recover 83.5 to 85 per cent as pure sugar (72). However, with the ion exchange process it is possible to remove a greater proportion of these non-sugars to produce a juice of 95.5 to 98.5 per cent purity. This gives a 95 per cent or better recovery as white sugar. This ion exchange treatment also removes 60 to 90 per cent of the organic non-sugar solids, which affect taste, odor and color.

A large number of patents have been granted recently on processes for purifying sugar juices. A partial list of such patents is given in Table 3.

Table 3. Patents pertaining to purification of sugar juices

Reference No.	U.S. Patent No.	Inventors	Company
84	2,559,902	Dahlberg	Great Western Sugar Co.
365	2,365,221	Shafor	The Dorr Co., Inc.
306	2,366,650	Rawlings, et al	The Dorr Co., Inc.
414	2,388,194	Vallez	Infilco, Inc.
34	2,388,222	Behrman	Infilco, Inc.
55	2,388,224	Behrman	Infilco, Inc.
367	2,391,649	Shafor	The Dorr Co., Inc.
305	2,391,843	Rawlings	The Dorr Co., Inc.
131	2,402,960	Gustafson, et al	Infilco, Inc.
132	2,403,177	Gustafson	Infilco, Inc.
33	2,413,676	Behrman, et al	Infilco, Inc.
307	2,413,784	Rawlings	The Dorr Co., Inc.
304	2,413,844	Rawlings	The Dorr Co., Inc.

H. Exchangers as Contact Materials

Ion exchange materials have been used as catalysts. The inorganic aluminosilicate type was studied in Russia by Bitepazh (44) for use as a catalyst in petroleum cracking. The cracking catalytic activity could be correlated with the exchangeable hydrogen or aluminum ions in the exchanger catalyst. The high catalytic action was found to be due to the exchange of hydrogen ions for the original sodium ions in the gel zeolite. Sodium and potassium ions could be adsorbed on the negatively charged surface in numbers sufficiently large to block the surface and catalytic action. This does not apply to hydrogen ions on account of their exceptionally small size and high mobility, which results in large areas of the surface being accessible. Displacement of sodium by

trivalent cations, such as aluminum, requires only one-third the number of ions, which also results in a major part of the surface being accessible. Calcium and magnesium gave intermediate results, compared with sodium and aluminum.

Other special products were made from ion exchangers by Riley, the patents for which were assigned to The Permutit Company. A copper-containing zeolite (331) was effective as a fungicide, and a nicotine-containing resin (330) acted as an insecticide.

III EXPERIMENTAL

Studies on ion exchange were made to illustrate (a) the "exchange tools" and (b) the possibilities for new industrial uses. Under "exchange tools", theory was represented by studies on equilibria and rates of ion exchange, techniques of operation by a fluidized bed study, and properties of exchange materials by the preparation of a beaded inorganic zeolite exchanger. A process for copper production was studied as a typical cation exchange application. Similarly, the purification of phenol wastes was studied as a typical anion exchange application.

A. Equilibrium of Ion Exchange

As noted in the literature review, the equilibria of various systems involving alkali metal and alkaline earth cations have been studied rather thoroughly. Therefore, the equilibria of nickel with Zeo-Karb cation exchanger was chosen for this investigation.

1. Preparation of materials

Zeo-Karb cation exchanger, obtained from The Permutit Company, was screened to -20 + 35 mesh. A batch of "standard" sodium Zeo-Karb was prepared by agitation with eight times its volume of a 5 per cent solution of sodium sulfate. This operation was done three times, followed by thorough washing with distilled water. The resulting Na-Zeo-Karb was air dried, and stored in an air-tight bottle for future use.

To determine the amount of exchangeable sodium in the standard Na-Zeo-Karb, five gram samples were agitated with three successive 100 ml. portions of 0.1005 N HCl. The resulting effluent was filtered, evaporated to dryness to remove the excess hydrochloric acid, and re-dissolved in distilled water. The chloride content was determined by Mohr method, from which the amount of exchangeable sodium was calculated as 0.0299 grams per gram of Na-Zeo-Karb.

A standard nickel exchanger, Ni-Zeo-Karb, was prepared in a manner similar to that for Na-Zeo-Karb. The nickel content was checked by two methods. It was removed from the resin by repeated treatment with nitric acid and analyzed colorimetrically. An exchanger sample was also ignited, residue dissolved in nitric acid, and the nickel determined colorimetrically. The calculated results indicated the exchangeable nickel as 0.150 grams per gram of Ni-Zeo-Karb.

2. Operating procedure

In order to obtain sufficient data for an equilibrium curve, five samples were run simultaneously. A weighed amount of standard exchanger was placed in each of five 250 ml. rubber-stoppered bottles. Also, 100 ml. of a known concentration of the other ion being studied was added to each bottle. These samples were then tumbled end over end in an attachment to a motor-driven ball mill for a period of 15 minutes to reach equilibrium. The resulting equilibrium mixture was allowed to settle, and the supernatant liquid filtered for analysis.

3. Experimental runs

Nickel nitrate solution was contacted with standard Na-Zeo-Karb in Run 1 until equilibrium conditions were obtained. The equilibrium was approached from the opposite direction in Run 2 by contacting sodium chloride solution with standard Ni-Zeo-Karb. The experimental data and calculated results are given in Tables 4 through 7.

4. Analytical procedures

The nickel concentration in the solution at equilibrium was determined colorimetrically, based on the procedure outlined in Snell and Snell (375) for the dimethylglyoxime method. This procedure was modified as follows: A standard 6 inch test tube was filled half full of the solution being tested, which ranged from 0.5 up to 20 ppm nickel. Two drops of saturated bromine water was added, allowed to stand 15 seconds, at which time 2 drops of 6N ammonium hydroxide were added. Three drops of indicator were then added and the sample set aside for five minutes. A photometer reading was then taken, which could be used to read the ppm nickel from a photometer calibration curve. A typical calibration curve for this method is shown on Figure 8.

5. Results and correlations

The experimental data were correlated by plotting the ratio of nickel to sodium ions in the solution at equilibrium against the nickel to sodium ratio in the solid cation exchanger. These plots were made on log-log paper, resulting in straight lines.

Usually, it is necessary to plot the "active" molar concentrations in order to obtain straight lines, but, as can be seen from examining Figures 13 through 17, these data could be correlated also on weight and actual molar concentrations. This can be attributed chiefly to the fact that rather dilute solutions were used in these studies.

The active molar ratios listed in Tables 5 and 7, and used for the correlations in Figures 15 and 16, were calculated from the mean activity coefficients plotted in Figure 12. These in turn have been calculated by use of the Debye-Huckel equation, with corrections for ionic size.

The most important point to note in this equilibrium study on nickel-sodium ions is the fact that the same equilibrium was not attained by approaching it from opposite directions. Figure 15 shows the correlation for what might be called the "forward" run, whereby nickel nitrate solution is contacted with Na-Zeo-Karb until equilibrium is attained. Figure 16 gives the correlation for the "reverse" run, whereby a sodium chloride solution is contacted with a standard Ni-Zeo-Karb exchanger until equilibrium is attained. In the forward run, for an active molar ratio of 30 for nickel to sodium in solution, the ratio in the solid is 0.0155 at equilibrium. In the reverse run, for the same ratio of 30 in solution, the ratio in the solid is 30,000 at equilibrium. It can be seen immediately that the cation exchange takes up a normal amount of nickel, but holds on to it very tenaciously in the reverse reaction. This behavior of nickel metal is in contrast to that observed for the alkali metal and alkaline earth cations, which give the same equilibrium

when approached from both directions.

This preference for nickel over sodium is also clearly illustrated by examination of the slope of the straight line plot of Figure 16, which represents the power p in the modified mass action equation of

$$(A/B)_{\text{solid}} = K \cdot (A/B)_{\text{soln.}}^p$$

whereas the value of p is less than 1.0 for all the alkali metal and alkaline earth cations, it has a value of 1.84 for this correlation on nickel. Thus, with the active ratio of nickel to sodium in the solution to the 1.84 power, it can be seen that a much lower ratio of Ni/Na in the solution is required in order to obtain a given ratio on the solid, in comparison with the alkali metals and alkalize earths.

It should be noted that equivalent exchange was assumed between the nickel and sodium for the calculations of the above-mentioned correlation. In view of the fact that the exchanger has a great affinity for nickel in comparison to sodium, it is doubtful that an equivalence of nickel was displaced from the exchange by each equivalence of sodium in the reverse run.

An equilibrium correlation for sodium chloride solution contacted with H-Zeo-Karb is shown on Figure 17. The data for this plot were taken from the rate of exchange Runs 7a, 8a, and 12a of the next section. Since these points represent the end conditions of 30 minute rate of exchange runs, they are true equilibrium points. It is important to note that this type of run can be used to determine the rate of exchange and at the same time one point on an equilibrium curve.

Data from the literature were included in Figures 9, 10, and 11 on the activity coefficients for various electrolytes. Since these activity coefficients represent the "active" concentrations, particularly at the higher solution concentrations, they denote a trend or effect that one can expect to be exerted on the equilibrium of the ions under consideration.

Table 4. Equilibrium of nickel nitrate solution with Na-Zeo-Karb (Run 1)^a

Test No.	Weight of Na-Zeo-Karb, g. ^b	Vol. of Ni(NO ₃) ₂ solution, ml. ^c	Before exchange		At exchange equilibrium ^d			
			Na in exchanger, meq.	Ni ⁺⁺ in solution, meq.	In exchanger		In solution	
					Na, meq.	Ni, meq.	Na ⁺ , meq.	Ni ⁺⁺ , meq. ^e
1	1.000	100.0	1.30	0.668	0.6544	0.6544	0.6544	13.62 x 10 ⁻³
2	2.000	100.0	2.60	0.668	1.933	0.6593	0.6593	8.86 x 10 ⁻³
3	3.000	100.0	3.90	0.668	3.232	0.6646	0.6646	3.40 x 10 ⁻³
4	5.000	100.0	6.50	0.668	5.832	0.6658	0.6658	2.21 x 10 ⁻³
5	7.000	100.0	9.10	0.668	8.432	0.6661	0.6661	1.91 x 10 ⁻³

^aSamples tumbled in 250 ml. stoppered bottles for 10 minutes.

^bNa-Zeo-Karb contained 0.0299 grams exchangeable sodium per gram dry exchanger.

^cStandard Ni(NO₃)₂ solution contained 196 p.p.m. of nickel.

^dEquivalent exchange assumed between sodium and nickel.

^eNickel in solution determined colorimetrically by dimethylglyoxime method.

Table 5. Equilibrium calculations for nickel nitrate solution with Na-Zeo-Karb (Run 1)

Test No.	Activity coefficients ^a		Weight ratios		Molar ratios		Active molar ratios	
	Ni ⁺⁺	Na ⁺	In exchanger	In solution	In exchanger	In solution	In exchanger ^b	In solution
			Ni/Na	Ni ⁺⁺ /Na ⁺	Ni/Na	Ni ⁺⁺ /Na ⁺	Ni/(Na) ²	^a Ni ⁺⁺ / ^a Na ⁺
1	0.843	0.917	1.319	0.0266	0.517	0.0104	0.815	160.0
2	0.844	0.918	0.429	0.0172	0.168	0.0067	0.0882	101.9
3	0.844	0.918	0.263	0.0065	0.103	0.0025	0.0318	38.52
4	0.844	0.918	0.145	0.0042	0.057	0.0016	0.00978	24.95
5	0.844	0.918	0.101	0.0036	0.039	0.0017	0.000468	21.55

^aActivity coefficients calculated by Debye-Huckel equation, with correction for ionic size.

^bRatio in exchanger solid based on total millimols of nickel and sodium.

Table 6. Equilibrium of sodium chloride solution with Ni-Zeo-Karb (Run 2)^a

Test No.	Weight of Ni-Zeo-Karb, g. ^b	Vol. of NaCl solution, ml. ^c	Before exchange		At exchange equilibrium ^d			
			Ni in exchanger, meq.	Na ⁺ in solution, meq.	In exchanger		In solution	
					Ni, meq.	Na, meq.	Ni ⁺⁺ , meq. ^e	Na ⁺ , meq.
1	0.500	200.0	2.56	2.140	2.551	9.26 x 10 ⁻³	9.26 x 10 ⁻³	2.131
2	1.000	200.0	5.11	2.140	5.098	11.86 x 10 ⁻³	11.86 x 10 ⁻³	2.128
3	1.500	200.0	7.67	2.140	7.658	12.27 x 10 ⁻³	12.27 x 10 ⁻³	2.128
4	2.000	200.0	10.22	2.140	10.207	13.29 x 10 ⁻³	13.29 x 10 ⁻³	2.127
5	2.500	200.0	12.78	2.140	12.766	14.18 x 10 ⁻³	14.18 x 10 ⁻³	2.126

^aSamples tumbled in 250 ml. stoppered bottles for 10 minutes.

^bNi-Zeo-Karb contained 0.150 grams exchangeable nickel per gram dry exchanger.

^cStandard NaCl solution was 0.0107 in normality.

^dEquivalent exchange assumed between sodium and nickel.

^eNickel in solution determined colorimetrically by dimethylglyoxime method.

Table 7. Equilibrium of sodium chloride solution with Ni-Zeo-Karb (Run 2)

Test No.	Activity coefficients ^a		Weight ratios		Molar ratios		Active molar ratios	
	Ni ⁺⁺	Na ⁺	In exchanger	In solution	In exchanger	In solution	In exchanger ^b	In solution
			Ni/Na	Ni ⁺⁺ /Na ⁺	Ni/Na	Ni ⁺⁺ /Na ⁺	Ni/(Na) ²	^a Ni ⁺⁺ / ^a Na ⁺
1	0.812	0.899	352.0	0.0055	137.9	0.00217	14,860.	20.42
2	0.812	0.899	548.5	0.0071	215.0	0.00278	18,130.	26.20
3	0.812	0.899	797.0	0.0073	312.0	0.00288	25,460.	27.12
4	0.812	0.899	981.2	0.0079	384.2	0.00312	28,870.	29.41
5	0.812	0.899	1150.5	0.0085	450.5	0.00334	31,700.	31.40

^aActivity coefficients calculated by Debye-Huckel equation, with correction for ionic size.

^bRatio in exchanger solid based on total millimols of nickel and sodium.

105

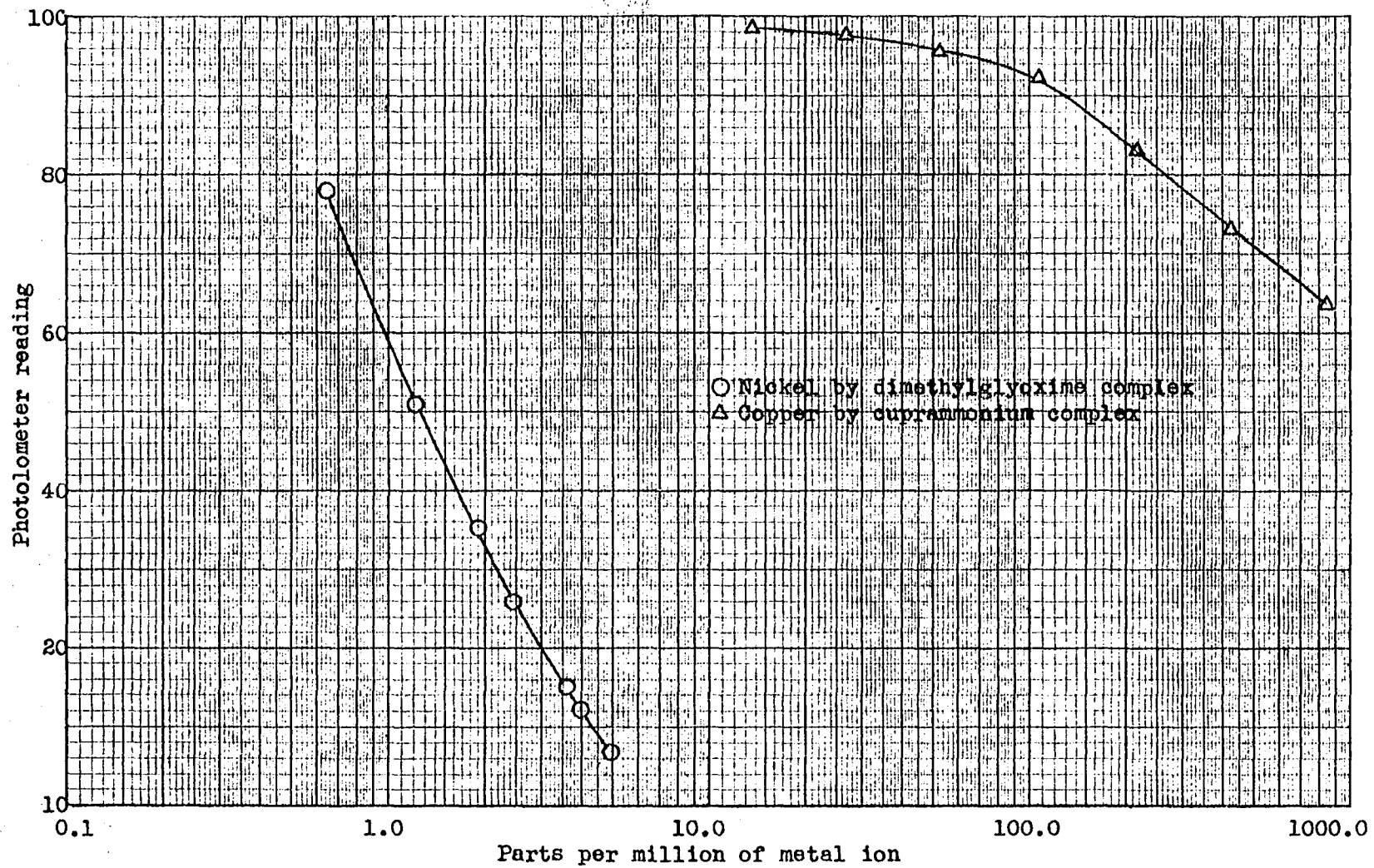


Fig. 8. Calibration curves for colorimetric analytically methods.

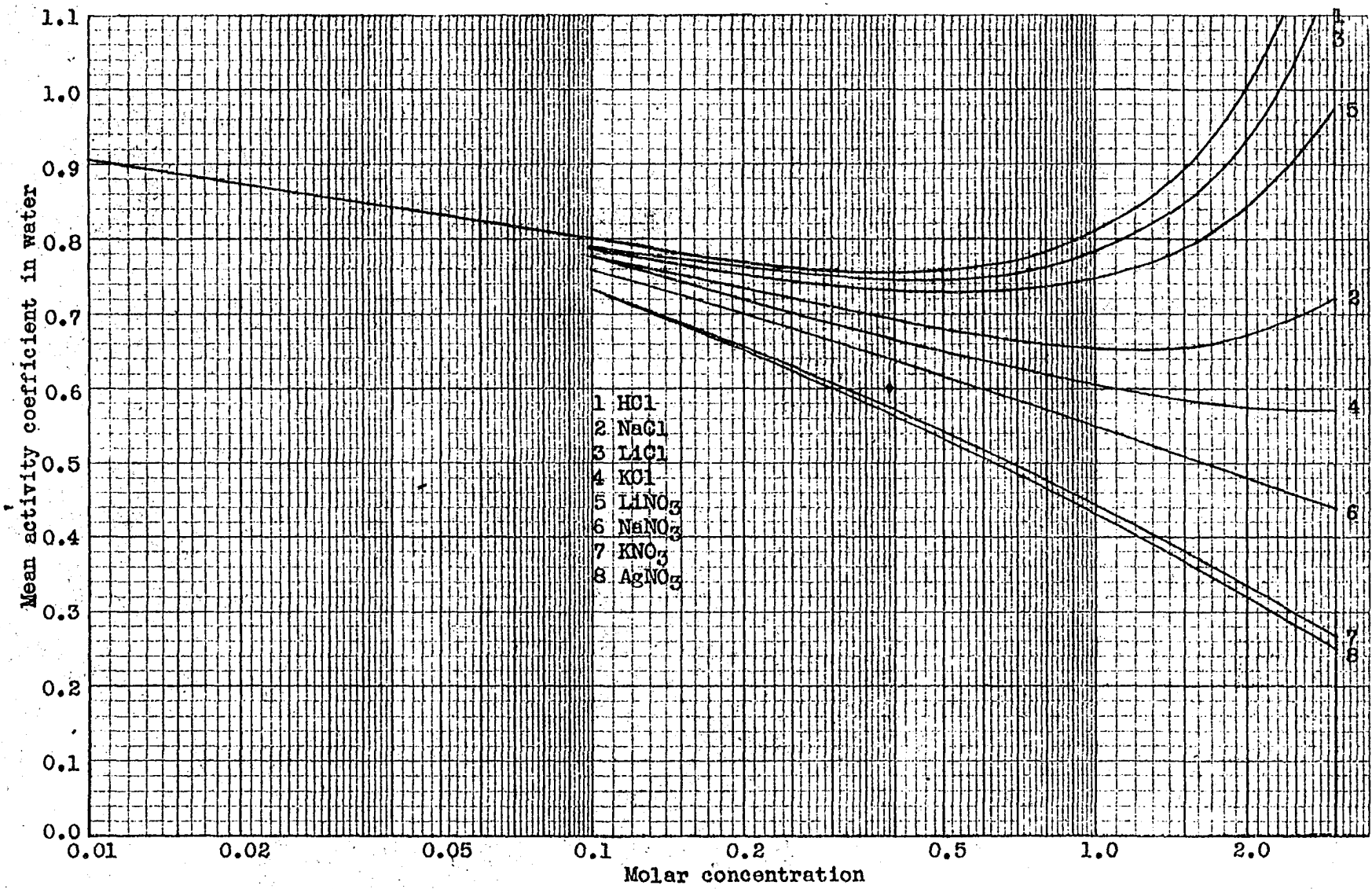


Fig. 9. Mean activity coefficients of 1-1 electrolytes at 25°C.

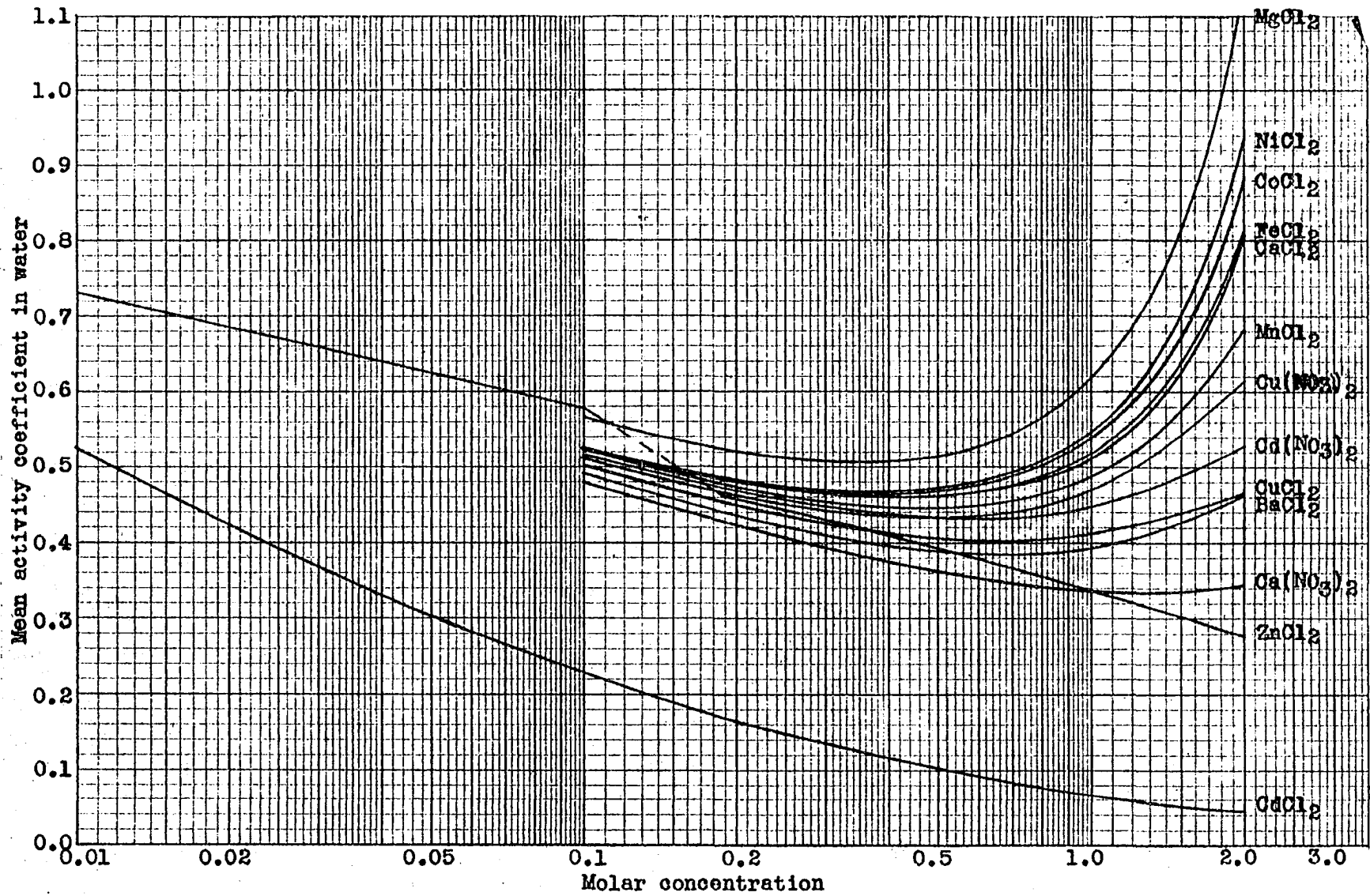


Fig. 10. Mean activity coefficients of 2-1 electrolytes at 25°C.

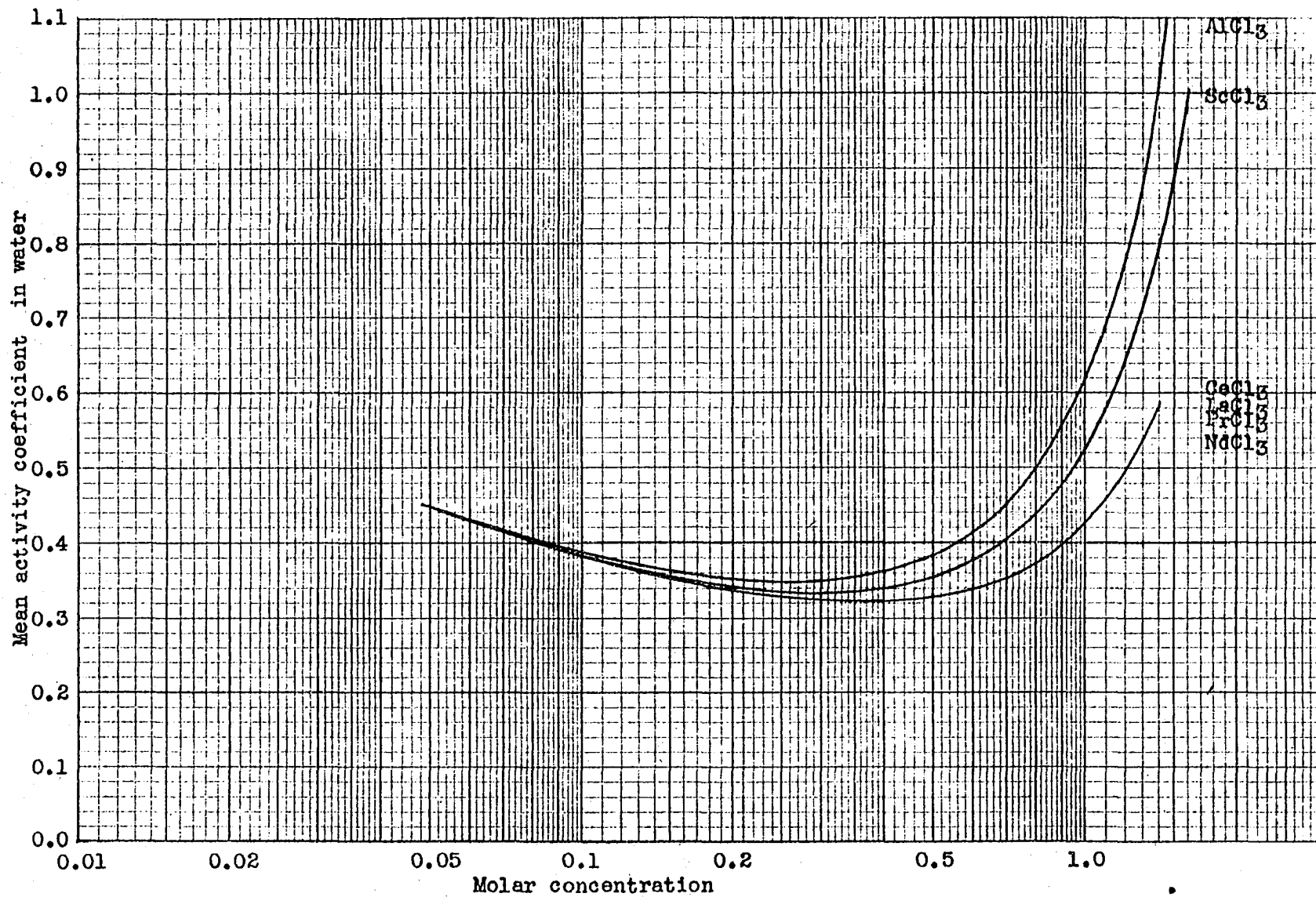


Fig. 11. Mean activity coefficients of 3-1 electrolytes at 25°C.

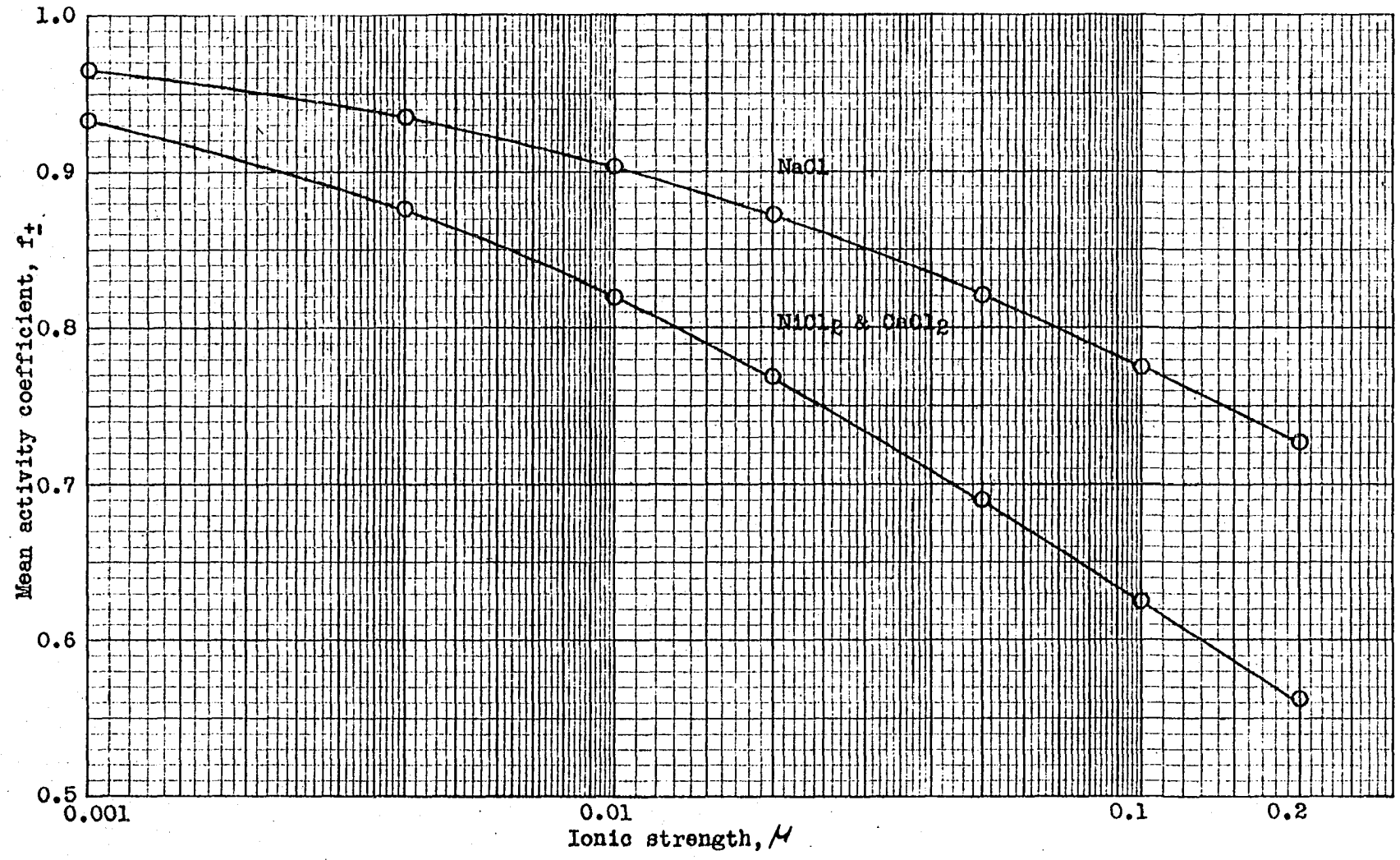


Fig. 12. Calculated mean activity coefficients for various ionic strengths of solution.

12

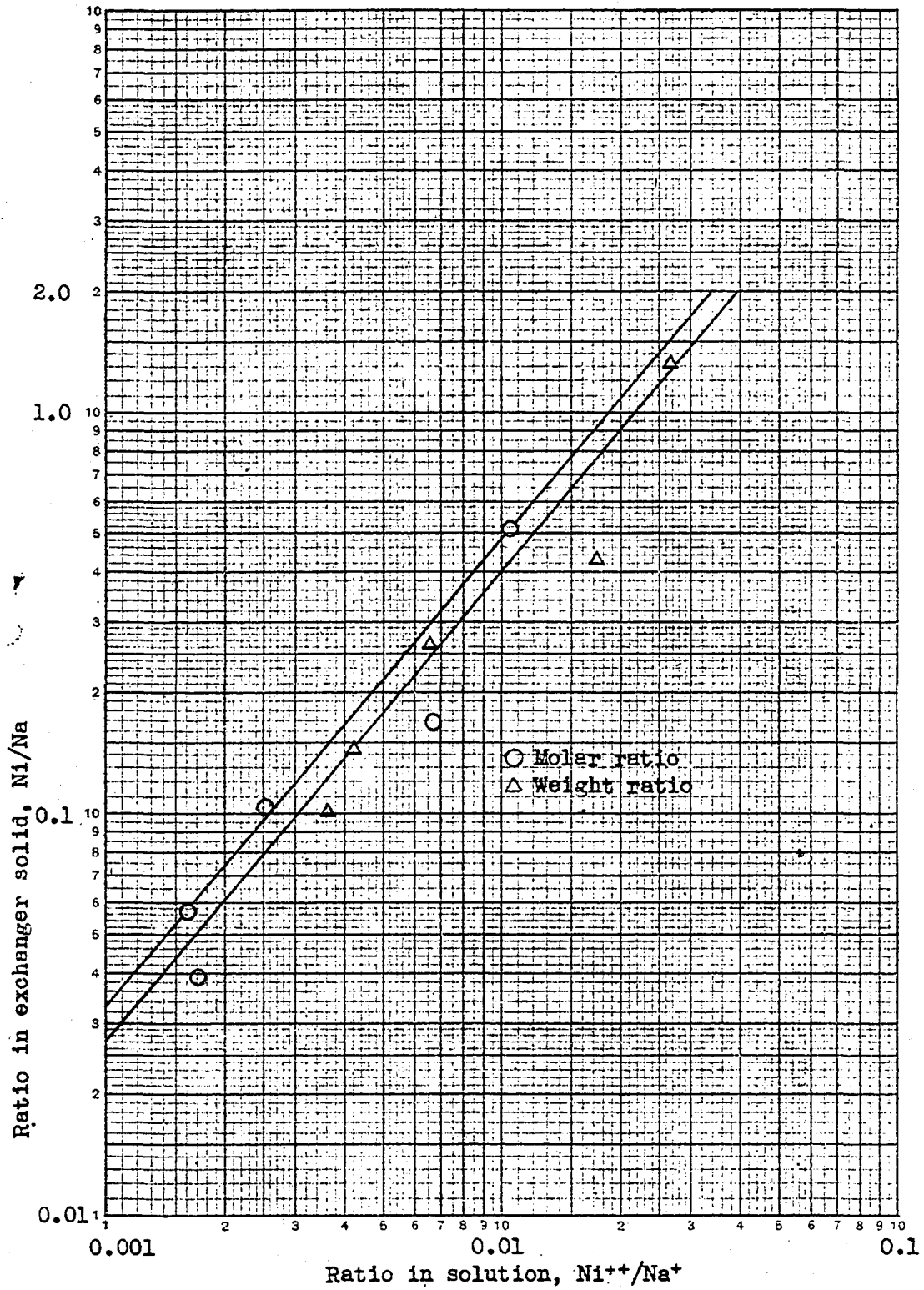


Fig. 13. Equilibrium correlation of nickel nitrate solution with Na-Zeo-Karb.

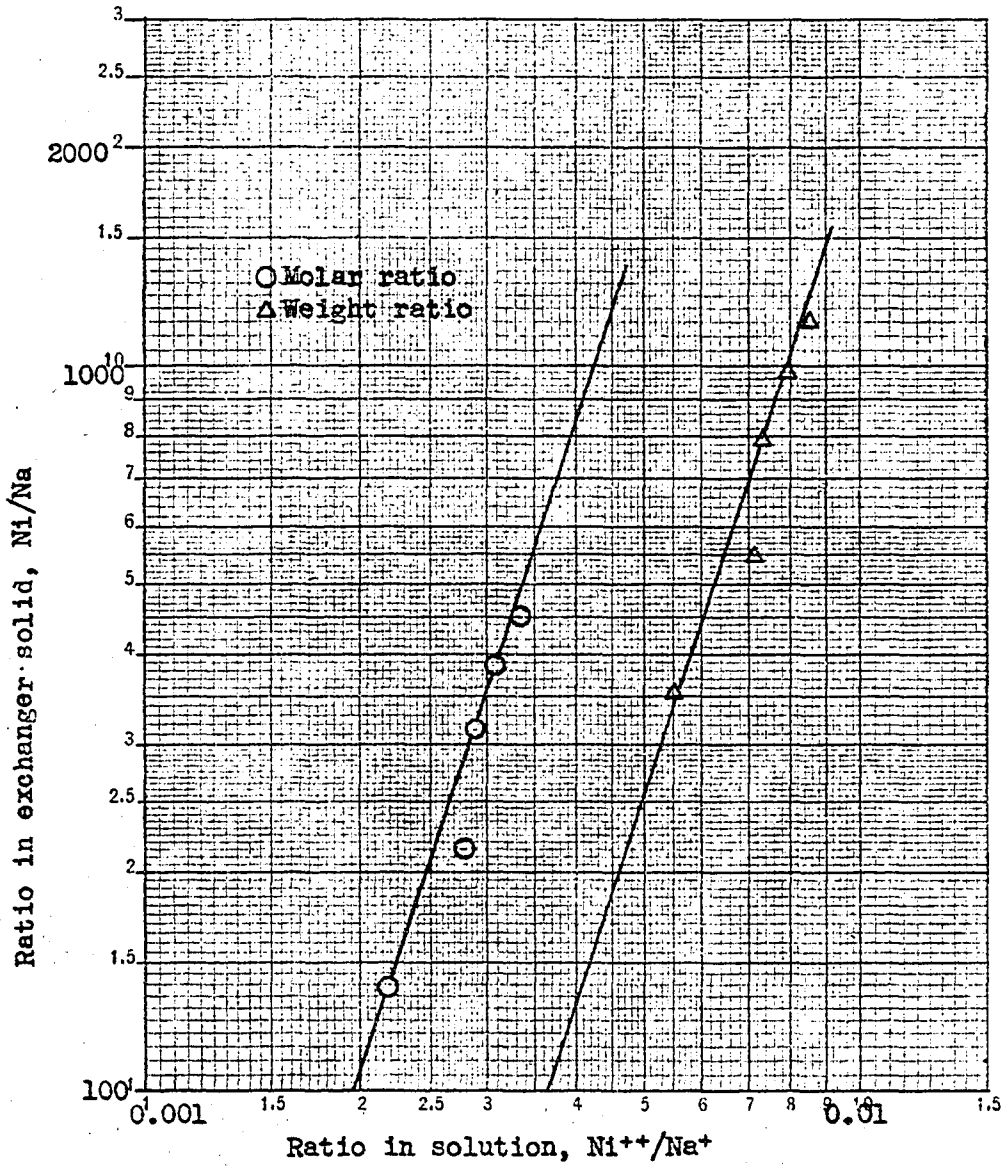


Fig. 14. Equilibrium correlation of sodium chloride solution with Ni-Zeo-Karb.

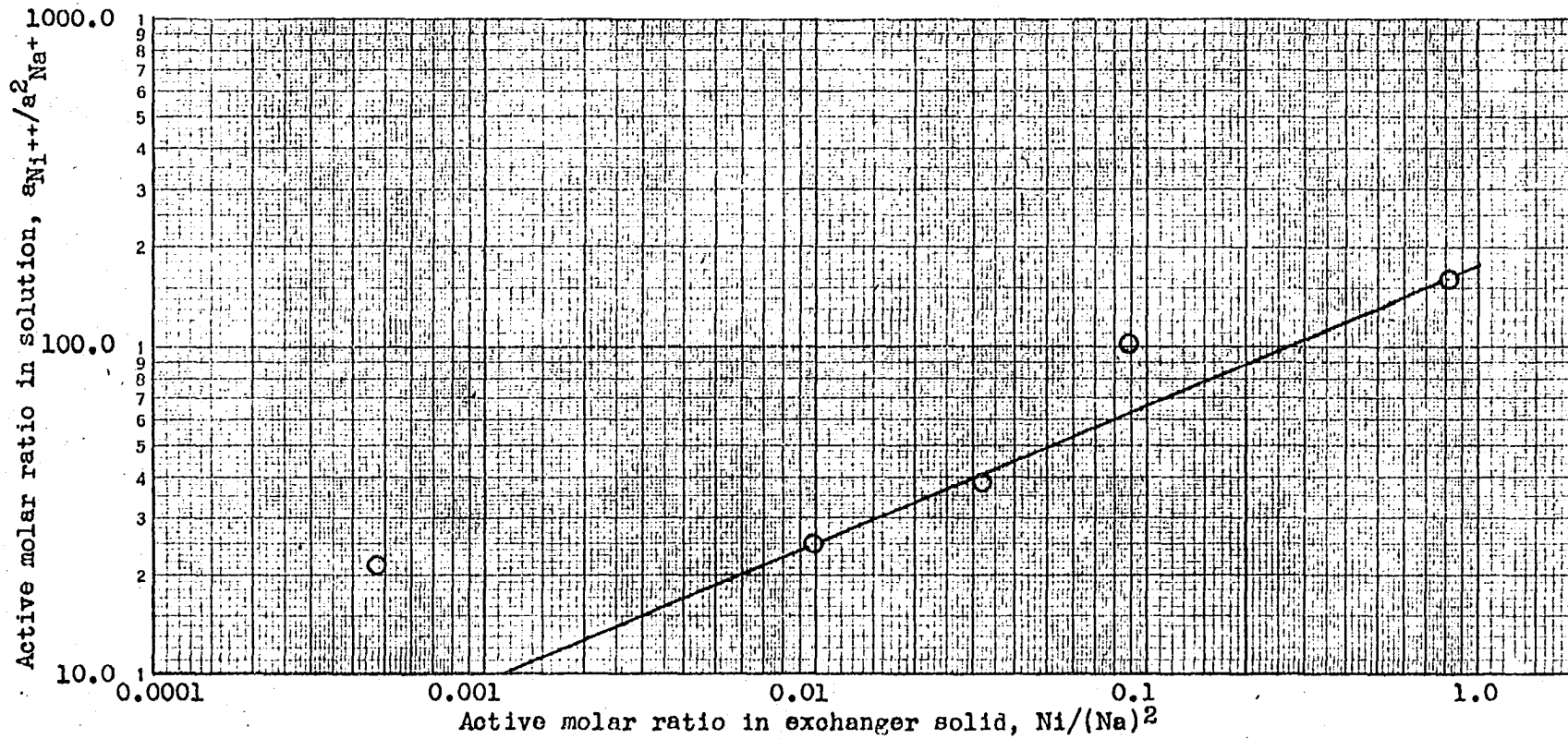


Fig. 15. Equilibrium correlation of nickel nitrate solution with Na-Zeo-Karb.

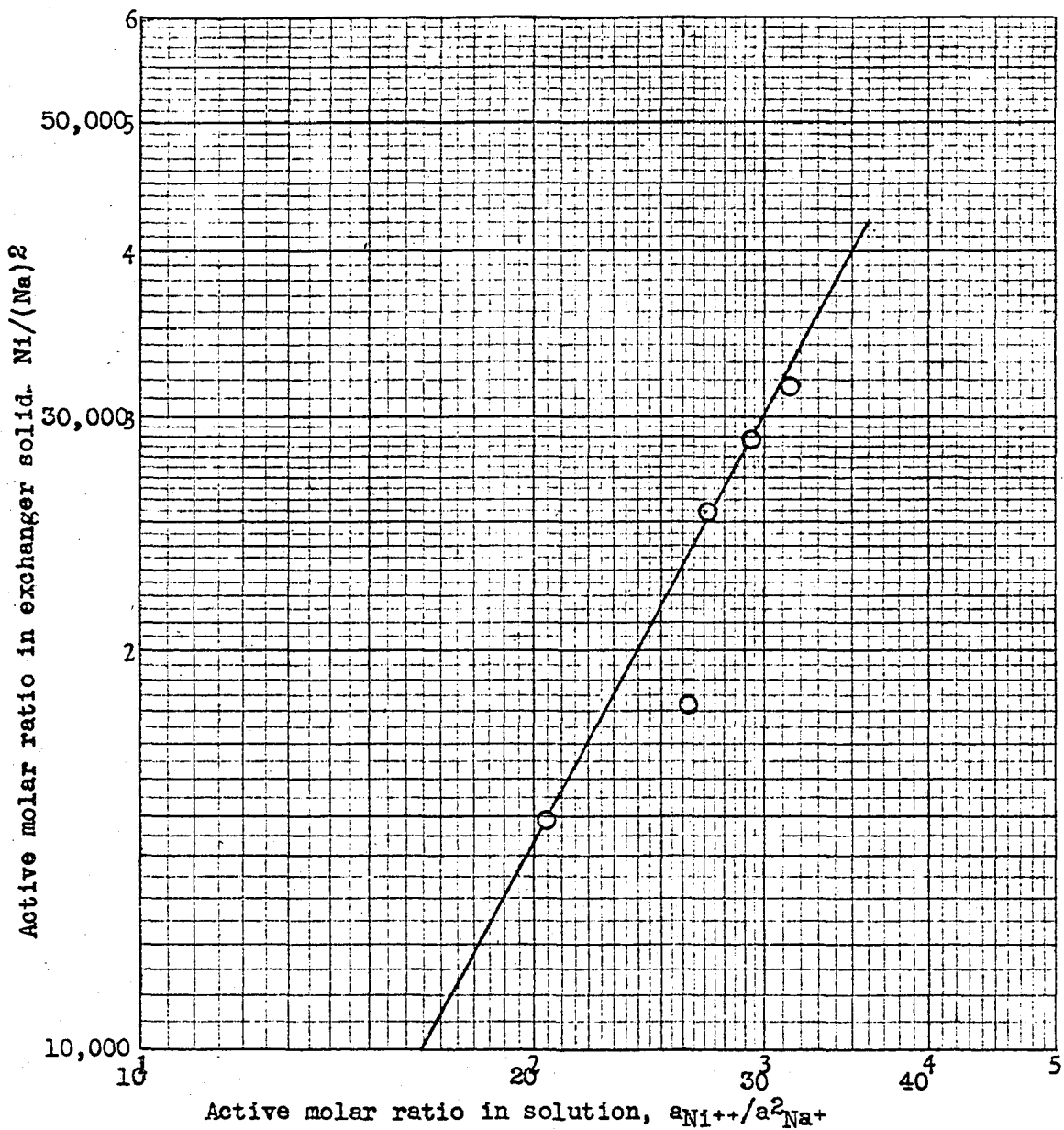


Fig. 16.. Equilibrium correlation of sodium chloride solution with Ni-Zeo-Karb.

16

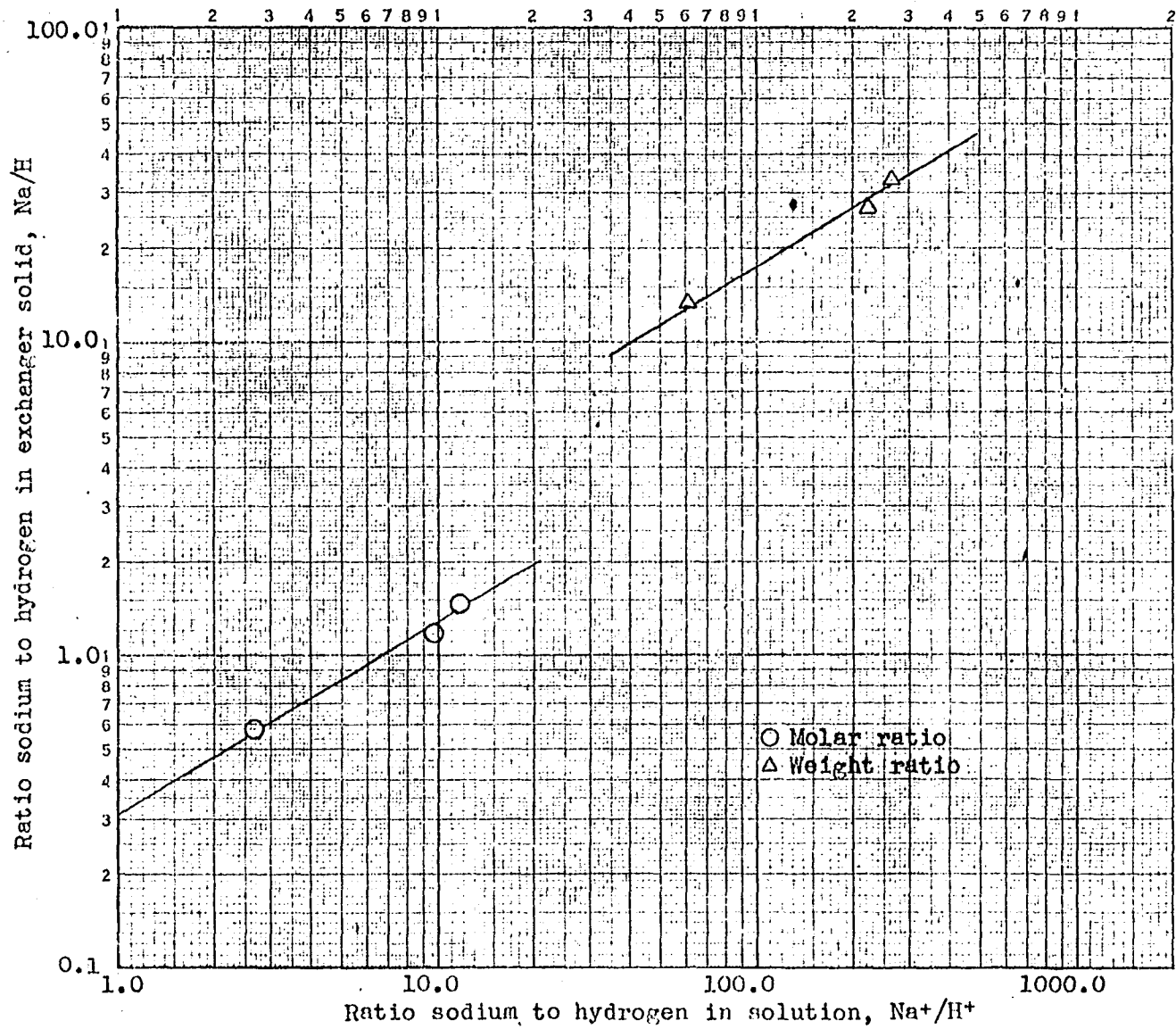


Fig. 17. Equilibrium correlation of sodium chloride solution with H-Zeo-Karb.

B. Rate of Ion Exchange

The rate of exchange for Zeo-Karb was determined for various conditions and by different experimental procedures. Data were obtained for the rates of exchange of other cation exchange resins. Correlations have been made in order to postulate a mechanism for the rate of exchange.

1. Preparation of materials

A batch of standard Na-Zeo-Karb, other than the batch made for equilibria studies, was prepared in the usual manner. The amount of exchangeable sodium was checked by two methods. It was eluted from the exchanger by means of 0.1005 N HCl, evaporated to dryness and analyzed in the usual manner. A check was made by igniting a sample of standard exchanger, and dissolving the ash in hydrochloric acid, which was then analyzed for sodium as previously described.

A batch of standard H-Zeo-Karb was then made from a portion of the above Na-Zeo-Karb. Again the exchanger was analyzed for residual sodium, from which the exchangeable hydrogen was calculated by assuming equivalent exchange.

2. Operating procedure

Several different experimental procedures were used in these studies, the details of which are described in the following sections.

a. Combined rate of exchange and static capacity. The ordinary static test for determining exchanger capacity has been modified so that

rates of cation exchange can be determined simultaneously with the static capacity. Essentially, this new method involves noting the period of time required for each constant increment of sodium hydroxide to be absorbed by the exchanger (actually, reaching an equilibrium). The detailed procedure of a typical run (Run 3 in Table 8) is as follows: After two or three preliminary runs to condition the resin, a regeneration is made by treating a one gram sample of exchanger with exactly 50 ml. of 4.6 per cent HCl. The regeneration period is carried out by constant stirring for exactly 30 minutes, after which the exchanger is immediately washed with distilled water until colorless to phenol red. Then 50 ml. of distilled water is added to a dry beaker. The moist sample is filtered to remove most of its surface water, and weighed in order to determine the amount of free water still on the test sample. The moist sample is placed in the 50 ml. of water in a 150 ml. beaker, where a constant stirring speed can be maintained. Five drops of phenol red are added to the mixture being stirred. The test proper is now ready to proceed. A wide-mouthed pipette is used to add 0.5 ml. increments of 0.0995 N NaOH. A stop-watch is snapped as the first portion of the increment goes into the solution. It is stopped at the end of the increment period, as denoted by change in color of the indicator. The rates of exchange become slower as the exchanger becomes saturated with sodium ions, or, thinking of it from the equilibrium standpoint, as the ratio of sodium to hydrogen becomes increasingly large. The final endpoint, of course, represents the static exchange capacity, which also represents one point on an equilibrium curve.

b. Combined rate of exchange and equilibrium curves. It is possible to modify the above procedure even further, although no experimental data

were taken to illustrate this procedure. It will be noted that the one equilibrium point of the above procedure represents a low sodium to hydrogen ratio in the solution. In fact, very often, it is too small to be an accurate equilibrium point. Therefore, this modified procedure proposes that several additional equilibrium points be determined at the end of the rate of exchange period. This can be done by adding four or five well-chosen increments of stronger NaOH standard solution, and taking out samples for analysis after each increment has been allowed to come to equilibrium.

c. Combined rate of exchange and equilibrium point. This procedure differs from the "combined rate of exchange and static capacity" in that the final equilibrium point represents a higher sodium to hydrogen ratio, and is, therefore, a more accurate point. The detailed procedure of a typical run (Runs 7a and 7b in Tables 12 and 13) is as follows: A 150 ml. volume of test solution is placed in a dry, 200 ml. beaker, which is equipped for constant stirring. A standard cation exchange resin, or one regenerated in the standard manner, is weighed (usually 6 gram sample), washed with distilled water, filtered to a low free moisture content, and added to the test solution to start a run proper. At timed intervals of 1, 1, 1, 2, 3, 4, 8 and 10 minutes, two ml. samples are withdrawn for analysis. At the end of the final interval a 50 ml. sample is withdrawn for analysis, and the exchanger is filtered and washed so that it can be used in further runs. It will then start out with the same ratio of ions in it as were present at the end of the previous run.

d. Combined rate of exchange and equilibrium curve by pH. A

procedure was developed to avoid the troublesome removal of periodic samples and the complicated calculations required for such runs. This procedure is typified by Runs 14a and 14b, tabulated in Tables 26 and 27, and is as follows: The test solution and standard cation exchanger are added in the usual manner to a beaker equipped with a stirrer. The electrodes of a pH meter are also placed within the beaker and in contact with the solution. Hence, as soon as the standard exchanger is added to the solution, instantaneous pH reading can be taken, indicating the rate of exchange in the first few critical seconds. After equilibrium has been attained, a second increment of solution is added. This procedure can be continued until five or six rate curves and equilibrium points are obtained. A solution sample was taken at the end of the last increment for analysis, although it would have been preferable to take samples for analysis at the end of each increment, at which point equilibrium is attained.

3. Experimental runs

Combined rate of exchange and static capacity were investigated in Runs 3 through 6 (Tables 8 through 11). Rate of exchange was combined with a realistic equilibrium point in each of Runs 7a and 7b through 12a (Tables 12 through 22, except 19 and 21). The remaining runs of this section, up to and including Run 23b and Table 45, present data taken by the pH and multiple increment method. These also include data on various cation exchange resins, in addition to various solution concentrations and types of cations.

4. Results and correlations

Several factors affecting the rate of exchange were illustrated in the runs combining rate of exchange and static capacity. A comparison of Figures 18 and 19 indicates that the amount of washing sample after regeneration affects the rate of exchange. Washing until acid-free to phenolphthalein in Figure 19 (Run 4) gives an initial increment time of 40 seconds compared with 52 seconds for Run 3 in Figure 18, which was not washed as thoroughly (acid-free to methyl orange). Since both of these times are relatively slow, in comparison with chemical reactions, it is believed that free acid in the exchanger must be diffusing to its surface and then reacting chemically with the NaOH in the solution. This theory is further verified by the fact that a similar run, not shown here, gave almost instantaneous increment times for the first part of the run. This fast rate was due to a relatively large amount of free acid being present in or on the exchanger.

Figure 20 represents graphically the effect of a driving force in the solution. Extrapolation of the upper 0.5 ml. increment curve, back one increment, gives a time of approximately 41 seconds. The same increment number on the lower 1.0 increment curve has an increment time of 50 seconds. Hence, only nine seconds were required to reduce the driving from approximately 0.00663 N NaOH to 0.00331 N NaOH, with the remaining 41 seconds required to reduce the concentration down to the color change of the phenol red end point.

The critical nature of the stirring speed is clearly illustrated in Figure 21. Each section of the curve represents a different stirring

speed. The breaks are definite and occur at the beginning of the increment having a different speed. Hence, it is apparent that diffusion of ions to and from the exchanger is exerting a major influence on the rate of exchange. This is particularly true at the beginning of a run, where the ratio of hydrogen to sodium on the exchanger is very low.

A correlation for the rate of exchange has been made, based on the data in Runs 7a and 7b, and the similar runs that follow them. Since these runs were run with a large initial volume of solution, generally 150 ml. to 200 ml., and had only small samples withdrawn for analyses, the solution concentration remains nearly constant throughout the run. Corrections can be made for the changes in concentrations caused by the withdrawal of samples. This was done in calculating the data for the milliequivalents of exchange that had taken place at any given time. Likewise, the actual solution concentrations are listed, based on the results from the analysis samples. However, in correlating these data to obtain straight line plots of equations from which rate of exchange constants are available, these minor changes in solution concentration were ignored.

The basis of this correlation is that the rate of exchange varies as some function of the amount of exchange still available on the exchanger. This is a modification of the second order bimolecular equation of Nachod and Wood (259). The solution concentration was assumed to be constant. Hence, the following equation was proposed:

$$dx/dt = K(b-x)^n$$

where the terms are defined as

$$dx/dt = \text{rate of exchange, meq./gram resin/min.}$$

$$K = \text{rate of exchange constant, including the effects,}$$

if any, due to concentration in the solution.

$(b-x)$ = approach to equilibrium, where b is the maximum exchange capacity of resin at equilibrium, and x is the amount of exchange at given time t .

n = power exponent of $(b-x)$, indicating the degree of influence, which the "residual capacity" can exert.

In order to correlate these data in the form of this equation, the exchange, x , was plotted against time. The slopes were determined at various points along the curve to get instantaneous rates of exchange. These values are listed in Table 47 for the various runs. These instantaneous rates of exchange, dx/dt , were then plotted against $(b-x)$, the approach to equilibrium. These plots are given on Figures 23 and 24. The power, n , of $(b-x)$ was next determined from the slopes of these straight line correlations. Thence, the rate of exchange constant, K , was determined.

The values of n , the power to which $(b-x)$ is raised, and the rate of exchange constants, K , as obtained in this correlation are as follows:

Run	7a	$n = 1.00$	$K = 0.1338$
	8a	1.00	0.192
	9a	2.24	9.65
	10a	2.07	3.45
	11a	1.32	1.06
	12a	1.65	6.90
	7b	2.00	8.20
	8b	1.15	0.44
	9b	3.24	10.00
	12b	2.14	7.75

The fact that the "rate constants" are not constant for runs having same ions indicates that other factors are involved in the value of K .

Dividing K by the average solution concentration for each run does not bring the values much closer together. The equilibrium values at the end

of Runs 7a, 8a, and 12a, all of which involve sodium chloride solutions in contact with H-Zeo-Karb, were examined. The equilibrium plot of these three points shows that Run 7a, although having the lowest solution concentration, was represented by the middle point on the straight equilibrium line. This means that a different amount of excess sodium chloride was used in this run, which resulted in a different set of ion ratios at equilibrium. This indicates that the ratio of ions on the solid is important, rather than just the ion being removed from solution (as indicated by the approach to equilibrium). Dividing the K values of Runs 7a, 8a, and 12a by the sodium to hydrogen ratio in the exchanger at final equilibrium give 0.1133, 0.1192 and 11.82, respectively. These data are insufficient to say which of the above values are in error, if at all. The other rate values cannot be considered in this comparison, because they were run with ions other than sodium.

It should be noted that for Runs 7a and 8a for sodium chloride solutions in contact with standard H-Zeo-Karb, the value of the power n was 1.0. Thus, the equation of the correlation for these two runs give straight lines on semi-log paper. These runs were correlated by plotting the fractional approach to equilibrium, $(b-x)/b$, against time as shown on Figure 22.

It is believed that most rate of exchange data must be taken under conditions similar to those of a desired operation. On the other hand, equilibria data can be utilized under all circumstances. Hence, a procedure, such as the modified pH method, can be of great utility in collecting a maximum of data with a minimum of effort and time. It is

preferable to analyze solution and exchanger samples wherever possible in these studies. It is particularly true for the metal ions, which do not exhibit equivalent exchange.

Table 8. Rate of exchange of H-Zeo-Karb at constant incremental concentration in solution (Run 3)^{a, b}

Test No.	Std. NaOH increment ^c	Differential	Cumulative	Time, sec.	Test No.	Std. NaOH increment ^c	Differential	Cumulative	Time, sec.
1	0.5	0.5	0.5	52.0	26	0.5	13.0	13.0	50.0
2	0.5	0.5	1.0	52.0	27	0.5	13.5	13.5	52.0
3	0.5	0.5	1.5	52.0	28	0.5	14.0	14.0	52.0
4	0.5	0.5	2.0	52.0	29	0.5	14.5	14.5	53.0
5	0.5	0.5	2.5	52.0	30	0.5	15.0	15.0	54.5
6	0.5	0.5	3.0	52.0	31	0.5	15.5	15.5	58.0
7	0.5	0.5	3.5	50.0	32	0.5	16.0	16.0	61.0
8	0.5	0.5	4.0	49.0	33	0.5	16.5	16.5	64.0
9	0.5	0.5	4.5	50.0	34	0.5	17.0	17.0	67.0
10	0.5	0.5	5.0	49.0	35	0.5	17.5	17.5	69.5
11	0.5	0.5	5.5	48.0	36	0.5	18.0	18.0	74.0
12	0.5	0.5	6.0	48.0	37	0.5	18.5	18.5	80.0
13	0.5	0.5	6.5	48.5	38	0.5	19.0	19.0	105.0
14	0.5	0.5	7.0	48.5	39	0.5	19.5	19.5	84.0
15	0.5	0.5	7.5	48.0	40	0.5	20.0	20.0	100.0
16	0.5	0.5	8.0	47.0	41	0.5	20.5	20.5	115.0
17	0.5	0.5	8.5	47.0	42	0.5	21.0	21.0	125.0
18	0.5	0.5	9.0	47.0	43	0.5	21.5	21.5	145.0
19	0.5	0.5	9.5	46.0	44	0.5	22.0	22.0	171.0
20	0.5	0.5	10.0	47.0	45	0.5	22.5	22.5	207.0
21	0.5	0.5	10.5	46.0	46	0.5	23.0	23.0	240.0
22	0.5	0.5	11.0	46.0	47	0.5	23.5	23.5	300.0
23	0.5	0.5	11.5	47.0	48	0.5	24.0	24.0	360.0
24	0.5	0.5	12.0	46.0	49	0.5	24.5	24.5	480.0
25	0.5	0.5	12.5	48.0	50	0.5	25.0	25.0	900.0

^aDry H-Zeo-Karb sample weighed 2.000 grams; sample (after use in preceding run) is regenerated by stirring with 50 ml. of 4.59% HCl for 45 minutes, and washed until acid-free to methyl orange.

^bInitially, 50 ml. distilled water added to sample in 150 ml. sample.

^cStandard NaOH was 0.0995 in normality.

Table 9. Effect of initial washing on rate of exchange of H-Zeo-Karb (Run 4)^{a,b}

Test No.	Std. NaOH increment ^c	Differential	Cumulative	Time, sec.	Test No.	Std. NaOH increment ^c	Differential	Cumulative	Time, sec.
1	0.5	0.5	0.5	40.0	30	0.5	15.0	15.0	47.0
2	0.5	0.5	1.0	40.0	31	0.5	15.5	30.5	45.0
3	0.5	0.5	1.5	40.0	32	0.5	16.0	46.5	48.0
4	0.5	0.5	2.0	40.0	33	0.5	16.5	63.0	49.0
5	0.5	0.5	2.5	40.0	34	0.5	17.0	80.0	53.0
6	0.5	0.5	3.0	40.0	35	0.5	17.5	97.5	54.0
7	0.5	0.5	3.5	40.0	36	0.5	18.0	115.5	57.0
8	0.5	0.5	4.0	40.0	37	0.5	18.5	134.0	61.0
9	0.5	0.5	4.5	41.0	38	0.5	19.0	153.0	65.0
10	0.5	0.5	5.0	41.5	39	0.5	19.5	172.5	70.0
11	0.5	0.5	5.5	41.0	40	0.5	20.0	192.5	75.0
12	0.5	0.5	6.0	40.0	41	0.5	20.5	213.0	82.0
13	0.5	0.5	6.5	40.0	42	0.5	21.0	234.0	83.0
14	0.5	0.5	7.0	40.5	43	0.5	21.5	255.5	90.0
15	0.5	0.5	7.5	40.5	44	0.5	22.0	277.5	93.0
16	0.5	0.5	8.0	41.0	45	0.5	22.5	300.0	97.0
17	0.5	0.5	8.5	40.5	46	0.5	23.0	323.0	109.0
18	0.5	0.5	9.0	42.0	47	0.5	23.5	346.5	117.0
19	0.5	0.5	9.5	41.0	48	0.5	24.0	370.5	125.0
20	0.5	0.5	10.0	42.0	49	0.5	24.5	395.0	140.0
21	0.5	0.5	10.5	42.0	50	0.5	25.0	420.0	155.0
22	0.5	0.5	11.0	41.5	51	0.5	25.5	445.5	190.0
23	0.5	0.5	11.5	42.0	52	0.5	26.0	471.5	195.0
24	0.5	0.5	12.0	41.5	53	0.5	26.5	498.0	225.0
25	0.5	0.5	12.5	42.0	54	0.5	27.0	525.0	225.0
26	0.5	0.5	13.0	42.5	55	0.5	27.5	552.5	255.0
27	0.5	0.5	13.5	44.0	56	0.5	28.0	580.5	320.0
28	0.5	0.5	14.0	43.5	57	0.5	28.5	609.0	375.0
29	0.5	0.5	14.5	45.0	58	0.5	29.0	638.0	480.0

^aDry H-Zeo-Karb sample weighed 2.000 grams; sample (after use in preceding run) is regenerated by stirring with 50 ml. of 4.59% HCl for 45 minutes, and washed until acid-free to phenolphthalein.

^bInitially, 50 ml. distilled water added to sample in 150 ml. sample.

^cStandard NaOH was 0.0995 in normality.

Table 10. Effect of incremental concentration in solution on rate on rate of exchange of H-Zeo-Karb (Run 5)^{a,b}

Test No	Std. NaOH increment ^c		Time, sec.	Test No	Std. NaOH increment ^c		Time, sec.
	Differential	Cumulative			Differential	Cumulative	
1	0.5	0.5	0.1	21	0.6	17.6	71.5
2	0.5	1.0	0.1	22	0.4	18.0	63.0
3	0.5	1.4	0.1	23	0.5	18.5	76.0
4	0.5	2.0	0.1	24	0.5	19.0	87.0
5	1.0	3.0	0.1	25	0.5	19.5	75.0
6	1.0	4.0	1.0	26	0.6	20.1	113.0
7	1.0	5.0	1.0	27	0.4	20.5	114.0
8	1.0	6.0	2.0	28	0.5	21.0	127.0
9	1.0	7.0	8.0	29	0.5	21.5	132.0
10	1.0	8.0	8.0	30	0.5	22.0	187.0
11	1.0	9.0	9.0	31	0.55	22.55	197.0
12	1.0	10.0	14.0	32	0.45	23.0	195.0
13	1.0	11.0	15.0	33	0.50	23.5	202.0
14	1.0	12.0	20.0	34	0.50	24.0	231.0
15	1.0	13.0	30.0	35	0.50	24.5	256.0
16	1.0	14.0	40.0	36	0.50	25.0	275.0
17	1.0	15.0	49.0	37	0.50	25.5	290.0
18	1.0	16.0	50.0	38	0.50	26.0	294.0
19	0.5	16.5	42.5	39	0.50	26.5	315.0
20	0.5	17.0	56.2	40	0.50	27.0	1200.0

^aDry H-Zeo-Karb sample weighed 2.000 grams, and contained 0.865 meq. exchangeable and 0.035 meq. free hydrogen per gram dry H-Zeo-Karb.

^bInitially, 50 ml. distilled water added to sample in 150 ml. sample.

^cStandard NaOH was 0.0995 in normality.

Table 11. Effect of agitation on rate of exchange of H-Zeo-Karb (Run 6)^{a,b}

Test No.	Std. NaOH increment ^c		Time, sec. ^d	Test No.	Std. NaOH increment ^c		Time, sec.
	Differential	Cumulative			Differential	Cumulative	
1	0.5	0.5	6.0 ^F	28	0.5	14.0	33.0
2	0.5	1.0	6.0	29	0.5	14.5	35.0
3	0.5	1.5	6.0	30	0.5	15.0	38.0
4	0.5	2.0	7.0	31	0.5	15.5	38.0
5	0.5	2.5	7.4	32	0.5	16.0	40.0
6	0.5	3.0	8.4	33	0.5	16.5	43.0
7	0.5	3.5	9.0	34	0.5	17.0	57.0 ^{VS}
8	0.5	4.0	9.6	35	0.5	17.5	61.0
9	0.5	4.5	12.5 ^M	36	0.5	18.0	61.0
10	0.5	5.0	12.6	37	0.5	18.5	72.0
11	0.5	5.5	15.0	38	0.5	19.0	73.0
12	0.5	6.0	15.0	39	0.5	19.5	85.0
13	0.5	6.5	15.5	40	0.5	20.0	81.0 ^F
14	0.5	7.0	15.6	41	0.5	20.5	77.0
15	0.5	7.5	16.0	42	0.5	21.0	84.0
16	0.5	8.0	16.6	43	0.5	21.5	99.0
17	0.5	8.5	22.5 ^S	44	0.5	22.0	125.0
18	0.5	9.0	22.7	45	0.5	22.5	135.0
19	0.5	9.5	23.2	46	0.5	23.0	140.0
20	0.5	10.0	23.4	47	0.5	23.5	165.0
21	0.5	10.5	27.0	48	0.5	24.0	180.0
22	0.5	11.0	27.0	49	0.5	24.5	255.0 ^S
23	0.5	11.5	29.0	50	0.5	25.0	330.0
24	0.5	12.0	33.0	51	0.5	25.5	390.0
25	0.5	12.5	27.0	52	0.5	26.0	450.0
26	0.5	13.0	29.0	53	0.5	26.5	570.0
27	0.5	13.5	31.0	54	0.5	27.0	900.0

^aDry H-Zeo-Karb sample weighed 2.000 grams; sample (after use in preceding run) is regenerated by stirring with 50 ml. of 4.59% HCl for 45 minutes, and washed until acid-free to methyl orange.

^bInitially, 50 ml. distilled water added to sample in 150 ml. sample.

^cStandard NaOH was 0.0995 in normality.

^dSpeed of stirring indicated as follows: F for fast; M for medium; S for slow; and VS for very slow.

Table 12. Exchange of sodium from 0.0182 M NaCl solution with H-Zeo-Karb (Run 7a)^{a,b,c,d}.

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 5.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 5.00 ml. sample (smoothed), ml.	H ion removed by 5.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	301.1	0.50	0.50	0.0050	0.301	0.301	0.266
2	1	2	296.1	0.66	0.66	0.0066	0.391	0.396	0.361
3	1	3	291.1	0.76	0.69	0.0069	0.402	0.414	0.379
4	2	5	286.1	0.73	0.73	0.0073	0.417	0.436	0.401
5	3	8	281.1	0.75	0.77	0.0077	0.432	0.458	0.423
6	4	12	276.1	0.80	0.80	0.0080	0.442	0.476	0.441
7	8	20	271.1	0.84	0.84	0.0084	0.455	0.497	0.462
8	10	30	266.1	0.85	0.85	0.0085	0.453	0.503	0.468

^aDry H-Zeo-Karb sample weighed 1.0 grams, and contained 0.865 meq. exchangeable and 0.035 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 1.1 grams.

^cOriginal volume of 0.0182 M NaCl solution was 300.0 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.085 meq. (out of 0.120 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 5.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.035 meq. free hydrogen on 1.0 gram H-Zeo-Karb samples.

Table 13. Exchange of hydrogen from 0.0090 N HCl solution with Na-Zeo-Karb (Run 7b)^{a,b,c,d}

Test interval, No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 5.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 5.00 ml. sample (smoothed), ml.	H ion removed by 5.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion, incl. sample, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	171.2	3.60	3.60	0.0360	1.233	1.233	0.297
2	1	2	166.2	3.27	3.27	0.0327	1.085	1.121	0.419
3	1	3	161.2	3.14	3.14	0.0314	1.010	1.079	0.451
4	2	5	156.2	3.03	3.03	0.0303	0.946	1.046	0.484
5	3	8	151.2	2.99	2.99	0.0299	0.905	1.035	0.495
6	4	12	146.2	2.97	2.97	0.0297	0.868	1.028	0.502
7	8	20	141.2	3.00	2.94	0.0294	0.830	1.020	0.510
8	10	30	136.2	2.93	2.93	0.0293	0.798	1.017	0.513

^aNa-Zeo-Karb sample weighed 1.0 grams (as dry H-Zeo-Karb), and contained 0.468 meq. exchangeable sodium per gram dry H-Zeo-Karb.

^bWater on moist Na-Zeo-Karb sample weighed 1.2 grams.

^cOriginal volume of 0.0090 N HCl solution was 170.0 ml.

^dFree acid washed from sample until trace only remaining.

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 5.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals difference between column nine and the 1.530 meq. original H ion.

Table 14. Exchange of sodium from 0.1018 M NaCl solution with H-Zeo-Karb (Run 8a)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ² ml.	Volume NaOH for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ⁶	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exohanged from resin, meq. ^h
1	1	1	201.5	1.43	1.43	0.0143	1.442	1.442	1.362
2	1	2	199.5	1.48	1.48	0.0148	1.476	1.490	1.410
3	1	3	197.5	1.58	1.51	0.0151	1.491	1.520	1.440
4	2	5	195.5	1.56	1.56	0.0156	1.524	1.568	1.488
5	3	8	193.5	1.59	1.58	0.0158	1.529	1.589	1.509
6	4	12	191.5	1.58	1.59	0.0159	1.523	1.599	1.519
7	8	20	189.5	1.60	1.605	0.01605	1.521	1.613	1.533
8	10	30	187.5	1.61	1.61	0.0161	1.509	1.617	1.537

^aDry H-Zeo-Karb sample weighed 3.0 grams, and contained 0.865 meq. exchangeable and 0.027 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 4.5 grams.

^cOriginal volume of 0.1018 M NaCl solution was 200.0 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.280 meq. (out of 0.360 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.080 meq. free hydrogen on 3.0 gram H-Zeo-Karb.

Table 15. Exchange of hydrogen from 0.0307 N HCl solution with Na-Zeo-Karb (Run 8b)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion, incl. sample, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	151.9	4.50	4.50	0.0450	3.42	3.42	1.18
2	1	2	149.9	4.33	4.33	0.0433	3.25	3.30	1.30
3	1	3	147.9	4.17	4.17	0.0417	3.08	3.17	1.43
4	2	5	145.9	4.04	4.04	0.0404	2.95	3.08	1.52
5	3	8	143.9	3.84	3.94	0.0394	2.84	3.01	1.59
6	4	12	141.9	3.89	3.89	0.0389	2.76	2.96	1.64
7	8	20	139.9	3.87	3.87	0.0387	2.70	2.94	1.66
8	10	30	137.9	3.89	3.86	0.0386	2.66	2.94	1.66

^aNa-Zeo-Karb sample weighed 3.0 grams (as dry H-Zeo-Karb), and contained 0.512 meq. exchangeable sodium per gram dry H-Zeo-Karb.

^bWater on moist Na-Zeo-Karb sample weighed 1.9 grams.

^cOriginal volume of 0.0307 N HCl solution was 150.0 ml.

^dFree acid washed from sample until trace only remaining.

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals difference between column nine and the 4.60 meq. original H ion.

Table 16. Exchange of calcium from 0.100 M CaCl₂ solution with H-Zeo-Karb (Run 9a)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	152.1	4.57	4.57	0.0457	3.47	3.47	3.21
2	1	2	150.1	5.02	5.02	0.0502	3.76	3.81	3.55
3	1	3	148.1	5.26	5.26	0.0526	3.89	3.99	3.73
4	2	5	146.1	5.43	5.43	0.0543	3.96	4.11	3.85
5	3	8	144.1	5.50	5.51	0.0551	3.97	4.17	3.91
6	4	12	142.1	5.55	5.58	0.0558	3.97	4.23	3.97
7	8	20	140.1	5.73	5.69	0.0569	3.98	4.29	4.03
8 ⁱ	10	30	138.1	5.77	5.77	0.0577	3.98	4.35	4.09

^aDry H-Zeo-Karb sample weighed 6.0 grams, and contained 0.865 meq. exchangeable and 0.043 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 2.1 grams.

^cOriginal volume of 0.100 M CaCl₂ solution was 150 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.459 meq. (out of 0.720 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 5.00 ml. volume of

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.26 meq. free hydrogen on 6.0 gram H-Zeo-Karb samples.

ⁱAt end of Test No. 8, solution analyzed 0.0546 M CaCl₂ (by soap titration).

Table 17. Exchange of hydrogen from 0.0902 N HCl solution with Ca-Zeo-Karb (Run 9b)^{a,b,c,d}

Test interval, No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion incl. sample, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	133.8	15.44	15.44	0.1544	10.33	10.33	1.58
2	1	2	131.8	14.89	14.89	0.1489	9.95	10.10	1.81
3	1	3	129.8	14.78	14.78	0.1478	9.56	9.86	2.05
4	2	5	127.8	14.69	14.69	0.1469	9.38	9.83	2.08
5	3	8	125.8	14.61	14.65	0.1465	9.21	9.81	2.10
6	4	12	123.8	14.63	14.63	0.1463	9.05	9.79	2.12
7	8	20	121.8	14.64	14.61	0.1461	8.90	9.79	2.12
8 ⁱ	10	30	119.8	14.60	14.60	0.1460	8.75	9.79	2.12

^aCa-Zeo-Karb sample weighed 6.0 grams, (as dry H-Zeo-Karb), and contained 0.341 meq. exchangeable calcium per gram dry H-Zeo-Karb.

^bWater on moist Ca-Zeo-Karb sample weighed 1.8 grams.

^cOriginal volume of 0.0902 N HCl solution was 132.0 ml.

^dFree acid washed from sample until trace only remaining.

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals difference between column nine and the 11.91 meq. original H ion.

ⁱAt end of Test No. 8, solution analyzed 0.0143 M CaCl₂ (by soap titration).

Table 18. Exchange of copper from 0.100 M CuCl_2 solution with H-Zeo-Karb (Run 10a)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	152.1	4.84	4.84	0.0484	3.68	3.68	3.54
2	1	2	150.1	5.54	5.54	0.0554	4.16	4.21	4.07
3	1	3	148.1	5.86	5.86	0.0586	4.35	4.46	4.32
4	2	5	146.1	6.12	6.12	0.0612	4.47	4.64	4.50
5	3	8	144.1	6.34	6.33	0.0633	4.56	4.79	4.65
6	4	12	142.1	6.42	6.42	0.0642	4.56	4.85	4.71
7	8	20	140.1	6.55	6.55	0.0655	4.59	4.94	4.80
8	10	30	138.1	6.68	6.68	0.0668	4.61	5.03	4.89

^aDry H-Zeo-Karb sample weighed 6.0 grams, and contained 0.865 meq. exchangeable and 0.023 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 2.1 grams.

^cOriginal volume of 0.100 M CuCl_2 solution was 150.0 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.583 meq. (out of 0.720 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.14 meq. free hydrogen on 6.0 gram H-Zeo-Karb samples.

ⁱAt end of test No. 8, solution analyzed 0.0715 M CuCl_2 (by colorimetric method).

Table 19. Exchange of hydrogen from HCl solution with Cu-Zeo-Karb (Run 10b)^a

Vol. HCl, ml. ^b	100.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5 ^c	
Corr. of pH reading	0.00		0.00		0.00		0.00		0.00	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
10	2.04	0.00956	1.91	0.0131	1.79	0.0174	1.58	0.0289	1.50	0.0351
20	2.07	0.00891	1.92	0.0127	1.80	0.0170	1.61	0.0269	1.51	0.0342
30	2.12	0.00793	1.95	0.0118	1.81	0.0167	1.62	0.0262	1.51	0.0342
40	2.14	0.00755	1.97	0.0113	1.81	0.0167	1.62	0.0262	1.52	0.0334
50	2.16	0.00721	1.97	0.0113	1.82	0.0162	1.62	0.0262	1.52	0.0334
60	2.17	0.00705	1.98	0.0110	1.80	0.0170	1.62	0.0262	1.53	0.0327
80	2.17	0.00705	1.99	0.0107	1.82	0.0162	1.62	0.0262	1.52	0.0327
90	2.18	0.00688	1.99	0.0107	1.83	0.0158	1.63	0.0256	1.52	0.0327
120	2.19	0.00672	1.99	0.0107	1.80	0.0170	1.64	0.0250	1.53	0.0327
180	2.20	0.00658	1.99	0.0107	1.82	0.0162	1.66	0.0238	1.56	0.0303
240	2.21	0.00642	2.00	0.0105	1.83	0.0158	1.67	0.0232	1.56	0.0303
300	2.21	0.00642	1.98 ^d	0.0110 ^d	1.84	0.0154	1.67	0.0232	1.57	0.0297
420	2.22	0.00627	--	---	--	---	--	---	--	---
480	--	---	--	---	1.84	0.0154	--	---	1.59	0.0283

^aDry Cu-Zeo-Karb sample weighed 6.00 grams (as H-Zeo-Karb), and contained 2.7 grams free moisture.

^bThe HCl solution additions were 0.0090 N for first increment, and 0.0902 N for last four increments.

^cAt end of last increment, solution analyzed 0.0014 N CuCl₂ (by colorimetric method).

^dMeter was found out of calibration, and was adjusted at this point.

Table 20. Exchange of cadmium from 0.100 M CdCl₂ solution with H-Zeo-Karb (Run 11a)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	152.1	3.53	3.53	0.0353	2.68	2.68	2.51
2	1	2	150.1	4.02	4.02	0.0402	3.02	3.06	2.89
3	1	3	148.1	4.21	4.21	0.0421	3.12	3.20	3.03
4	2	5	146.1	4.31	4.45	0.0445	3.25	3.37	3.20
5	3	8	144.1	4.58	4.58	0.0458	3.30	3.46	3.29
6	4	12	142.1	--	4.63	0.0463	3.29	3.50	3.33
7	8	20	140.1	4.69	4.67	0.0467	3.27	3.52	3.35
8	10	30	138.1	4.66	4.68	0.0468	3.23	3.53	3.36

^aDry H-Zeo-Karb sample weighed 6.0 grams, and contained 0.865 meq. exchangeable and 0.028 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 2.1 grams.

^cOriginal volume of 0.100 M CdCl₂ solution was 150 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.549 meq. (out of 0.720 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.17 meq. free hydrogen on 6.0 gram H-Zeo-Karb samples.

Table 21. Exchange of hydrogen from HCl solution with Cd-Zeo-Karb (Run 11b)^a

Vol. HCl, ml. ^b	100.0		10.0		25.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.01		0.01		0.02		0.02	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
10	2.06	0.00912	2.00	0.0102	1.82	0.0158	1.54	0.0303	1.48	0.0351
20	2.08	0.00871	2.00	0.0102	1.61	0.0262	1.51	0.0327	1.48	0.0351
30	2.12	0.00793	2.00	0.0102	1.63	0.0250	1.52	0.0319	1.48	0.0351
40	2.13	0.00774	2.00	0.0102	1.66	0.0232	1.53	0.0311	1.48	0.0351
50	2.14	0.00755	2.00	0.0102	1.66	0.0232	1.53	0.0311	1.49	0.0342
60	2.14	0.00755	2.01	0.0100	1.67	0.0228	1.53	0.0311	1.49	0.0342
80	2.16	0.00721	2.03	0.00957	1.68	0.0222	1.55	0.0297	1.49	0.0342
100	2.17	0.00705	--	---	1.69	0.0217	1.56	0.0289	1.50	0.0334
120	2.18	0.00688	2.03	0.00957	1.69	0.0217	1.55	0.0297	1.50	0.0334
180	2.19	0.00672	2.03	0.00957	1.69	0.0217	1.57	0.0283	1.50	0.0334
240	2.18	0.00688	2.03	0.00957	1.70	0.0212	1.56	0.0289	1.50	0.0334
300	2.19	0.00672	2.03	0.00957	--	---	1.58	0.0277	1.50	0.0334
360	2.19	0.00672	--	---	1.69	0.0277	1.58	0.0277	1.50	0.0334
420	--	---	--	---	1.70	0.0212	--	---	--	---

^aDry Cd-Zeo-Karb sample weighed 6.00 grams (as H-Zeo-Karb), and contained 2.1 grams free moisture.

^bThe HCl solution additions were 0.0090 N for first increment, and 0.0902 N for last four increments.

Table 22. Exchange of sodium from 0.050 M NaCl solution with H-Zeo-Karb (Run 12a)^{a,b,c,d}

Test No.	Stirring interval, min.	Total stirring time, min.	Volume solution (before sample), ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample, ml.	Volume NaOH (0.0100 N) for 2.00 ml. sample (smoothed), ml.	H ion removed by 2.00 ml. sample, meq. ^e	Cumulative H ion in solution, meq. ^f	Cumulative H ion from resin, meq. ^g	Cumulative H ion exchanged from resin, meq. ^h
1	1	1	152.3	2.54	2.54	0.0254	1.934	1.934	1.762
2	1	2	150.3	2.61	2.61	0.0261	1.960	1.960	1.788
3	1	3	148.3	2.66	2.66	0.0266	1.970	2.021	1.849
4	2	5	146.3	2.65	2.70	0.0270	1.974	2.052	1.880
5	3	8	144.3	2.66	2.72	0.0272	1.961	2.066	1.894
6	4	12	142.3	2.73	2.73	0.0273	1.940	2.072	1.900
7	8	20	140.3	2.74	2.74	0.0274	1.920	2.079	1.907
8	10	30	138.3	2.75	2.75	0.0275	1.901	2.087	1.915

^aDry H-Zeo-Karb sample weighed 6.0 grams, and contained 0.865 meq. exchangeable and 0.029 meq. free hydrogen per gram dry H-Zeo-Karb.

^bWater on moist H-Zeo-Karb sample weighed 2.3 grams.

^cOriginal volume of 0.050 M NaCl₂ solution was 150 ml.

^dTotal free acid washed from H-Zeo-Karb sample was 0.548 meq. (out of 0.720 meq. total free acid).

^eColumn seven equals column six times 0.0100 normality of standard NaOH solution.

^fColumn eight equals the product of columns seven and four, divided by the 2.00 ml. volume of analytical samples.

^gColumn nine equals column eight plus cumulative of previous values of column seven.

^hColumn ten equals column nine minus 0.172 meq. free hydrogen on 6.0 gram H-Zeo-Karb samples.

Table 23. Exchange of hydrogen from HCl solution with Na-Zeo-Karb (Run 12b)^{a, b}

Time, sec.	pH	HCl N. ^c	Time, sec.	pH	HCl N. ^c
10	1.12	0.0884	160	1.21	0.0711
20	1.16	0.0801	170	1.21	0.0711
30	1.18	0.0764	180	1.21	0.0711
40	1.19	0.0745	210	1.21	0.0711
50	1.19	0.0745	240	1.20	0.0728
60	1.20	0.0728	300	1.20	0.0728
70	1.20	0.0728	360	1.22	0.0694
80	1.20	0.0728	420	1.22	0.0694
90	1.20	0.0728	480	1.225	0.0685
100	1.21	0.0711	540	1.22	0.0694
110	1.21	0.0711	600	1.23	0.0677
120	1.21	0.0711	900	1.22	0.0694
130	1.21	0.0711	1200	1.20	0.0728
140	1.21	0.0711	1500	1.22	0.0694
150	1.21	0.0711	1800	1.23	0.0677

^aDry Na-Zeo-Karb sample weighed 6.000 grams (as H-Zeo-Karb), and contained 2.1 grams free moisture.

^bThe HCl solution was 0.0902 in normality and 110.0 ml. in volume.

^cNo correction required for salt effect in converting from pH reading.

Table 24. Exchange of lithium from LiCl solution with H-Zeo-Karb (Run 13a)^{a, b}

Vol. LiCl, ml. ^c	9.1		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	---	---	---	---	---	---	---	---	2.03	0.00957
10	---	---	2.17	0.00705	2.07	0.00871	2.02	0.00979	2.01	0.0100
20	2.39	0.00421	2.11	0.00811	2.04	0.00933	2.00	0.0102	2.00	0.0102
30	2.32	0.00497	2.10	0.00832	2.03	0.00957	1.99	0.0105	2.00	0.0102
40	2.30	0.00521	2.09	0.00851	2.03	0.00957	1.98	0.0107	2.00	0.0102
50	2.28	0.00546	2.09	0.00851	2.02	0.00979	1.98	0.0107	1.99	0.0105
60	2.27	0.00559	2.08	0.00871	2.03	0.00957	1.98	0.0107	2.00	0.0102
80	2.27	0.00559	2.08	0.00871	2.01	0.0100	1.98	0.0107	---	---
100	2.25	0.00584	2.08	0.00871	2.00	0.0102	---	---	1.99	0.0105
120	2.25	0.00584	2.07	0.00891	2.00	0.0102	1.98	0.0107	1.99	0.0105
180	2.23	0.00613	2.07	0.00891	2.00	0.0102	1.98	0.0107	1.98	0.0107
240	2.23	0.00613	---	---	2.00	0.0102	---	---	2.00	0.0102
300	---	---	---	---	---	---	---	---	1.99	0.0105
420	---	---	---	---	---	---	---	---	1.98	0.0107

^aDry H-Zeo-Karb sample weighed 6.00 grams, and contained 0.865 meq. exchangeable and 0.095 meq. free hydrogen per gram dry H-Zeo-Karb. Water on moist sample weighed 2.1 grams.

^bInitial vol. distilled water added to resin sample was 90.9 ml.

^cThe LiCl solution additions were 0.1178 N for all increments.

Table 25. Exchange of hydrogen from HCl solution with Li-Zeo-Karb (Run 13b)^a

Vol. HCl, ml. ^b	100.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.00		0.00		0.00	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	--	---	1.98	0.0110	1.87	0.0144	1.63	0.0256	1.53	0.0327
10	2.21	0.00642	2.03	0.00979	1.88	0.0140	1.64	0.0250	1.54	0.0319
20	2.23	0.00546	2.07	0.00891	1.91	0.0131	1.64	0.0250	1.54	0.0319
30	2.37	0.00441	2.09	0.00851	1.92	0.0127	1.65	0.0244	1.54	0.0319
40	2.42	0.00394	2.11	0.00811	1.93	0.0124	1.66	0.0238	1.54	0.0319
50	2.44	0.00376	2.13	0.00774	1.93	0.0124	1.67	0.0232	1.55	0.0311
60	2.44	0.00376	2.13	0.00774	1.94	0.0122	1.67	0.0232	1.55	0.0311
80	2.47	0.00351	2.13	0.00774	1.95	0.0118	1.67	0.0232	1.56	0.0303
100	2.49	0.00333	2.14	0.00755	--	---	--	---	--	---
120	2.49	0.00333	2.14	0.00755	1.96	0.0116	1.68	0.0228	1.58	0.0303
150	2.51	0.00319	--	---	--	---	--	---	--	---
180	2.51	0.00319	2.15	0.00737	1.96	0.0116	1.68	0.0228	1.56	0.0303
240	2.51	0.00319	2.16	0.00721	1.96	0.0116	1.69	0.0222	--	----
300	2.52	0.00312	2.16	0.00721	--	---	--	---	1.56	0.0303
360	--	---	--	---	--	---	1.69	0.0222	--	----
420	2.52	0.00312	2.16	0.00721	--	---	--	---	--	----

^a Dry Li-Zeo-Karb sample weighed 6.00 grams (as H-Zeo-Karb), and contained 2.1 grams free moisture.

^b The HCl solution additions were 0.0090 N for first increment, and 0.0902 N for last four increments.

Table 26. Exchange of nickel from NiCl₂ solution with H-Zeo-Karb (Run 14a)^{a, b}

Vol. NiCl ₂ , ml. ^c	9.1		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5 ^d	
Corr. of pH reading	0.01		0.02		0.03		0.05		0.07	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.24	0.00584	---	---	1.74	0.0183	1.79	0.0154	1.79	0.0146
10	2.19	0.00658	1.86	0.0140	1.73	0.0187	1.78	0.0158	1.79	0.0146
20	2.05	0.00913	1.81	0.0158	1.72	0.0192	1.76	0.0167	1.78	0.0151
30	1.98	0.0107	1.79	0.0167	1.71	0.0197	1.75	0.0170	1.78	0.0151
40	1.96	0.0113	1.79	0.0167	1.71	0.0197	1.75	0.0170	1.78	0.0151
50	1.93	0.0122	1.78	0.0170	1.71	0.0197	1.74	0.0174	1.78	0.0151
60	1.93	0.0122	1.78	0.0170	1.71	0.0197	1.74	0.0174	1.78	0.0151
80	1.91	0.0127	1.77	0.0174	1.69	0.0207	1.74	0.0174	1.78	0.0151
100	1.91	0.0127	1.73	0.0192	1.69	0.0207	1.72	0.0183	1.78	0.0151
120	1.88	0.0137	1.76	0.0178	1.70	0.0201	1.72	0.0183	1.78	0.0151
150	1.91	0.0127	1.75	0.0183	1.69	0.0207	1.72	0.0183	1.78	0.0151
180	1.91	0.0127	1.72	0.0197	1.69	0.0207	1.72	0.0183	1.78	0.0151
240	1.91	0.0127	1.71	0.0201	1.69	0.0207	1.72	0.0183	1.78	0.0151
300	1.91	0.0127	1.72	0.0197	--	---	--	---	1.78	0.0151

^aDry H-Zeo-Karb sample weighed 6.00 grams, and contained 0.865 meq. exchangeable and 0.08 meq. free hydrogen per gram dry H-Zeo-Karb. Water on moist sample weighed 1.9 grams.

^bInitial vol. distilled water added to resin sample was 90.9 ml.

^cThe NiCl₂ solution additions were 0.201 N for all increments.

^dAt end of last increment, solution analyzed 0.0740 N NiCl₂ (by colorimetric method).

Table 27. Exchange of hydrogen from HCl solution with Ni-7eo-Karb (Run 14b)^a

Vol. HCl, ml. ^b	100.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5 ^c	
Corr. of pH reading	0.00		0.01		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.20	0.00658	1.82	0.0158	1.74	0.0192	1.56	0.0297	1.49	0.0351
10	2.19	0.00672	1.89	0.0134	1.76	0.0183	1.56	0.0297	1.50	0.0342
20	2.12	0.00703	1.91	0.0127	1.78	0.0174	1.58	0.0283	1.51	0.0334
30	2.12	0.00793	1.92	0.0124	1.78	0.0174	1.59	0.0277	1.51	0.0334
40	2.15	0.00737	1.93	0.0122	1.79	0.0170	1.59	0.0277	1.52	0.0327
50	2.16	0.00721	1.93	0.0122	1.79	0.0170	1.59	0.0277	1.52	0.0327
60	2.18	0.00688	1.94	0.0118	1.79	0.0170	1.59	0.0277	1.52	0.0327
80	2.19	0.00672	1.94	0.0118	1.79	0.0170	1.59	0.0277	1.52	0.0327
100	2.19	0.00672	1.94	0.0118	1.80	0.0167	1.61	0.0262	1.52	0.0327
120	2.19	0.00672	1.94	0.0118	1.79	0.0170	1.61	0.0262	1.52	0.0327
150	2.19	0.00672	1.94	0.0118	1.81	0.0162	1.61	0.0262	1.52	0.0327
180	2.19	0.00672	1.94	0.0118	1.80	0.0167	1.62	0.0256	1.52	0.0327
240	2.19	0.00672	1.95	0.0116	1.80	0.0167	1.62	0.0256	1.52	0.0327
300	2.19	0.00672	1.95	0.0116	1.80	0.0167	1.62	0.0256	--	---

^aDry Ni-Zeo-Karb sample weighed 6.00 grams (as H-Zeo-Karb), and contained 2.1 grams free moisture.

^bThe HCl solution additions were 0.0090 N for first increment, and 0.0902 N for last four increments.

^cAt end of last increment, solution analyzed 0.0064 N NiCl₂ (by colorimetric method).

Table 28. Exchange of cobalt from CoCl_2 with H-Zeo-Karb (Run 15a)^{a, b}

Vol. CoCl_2 , ml. ^c	9.1		10.0		10.0		25.0		25.0		25.0	
Increment No.	1		2		3		4		5		6	
Conn. of pH reading	0.02		0.03		0.04		0.05		0.07		0.08	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	--	---	2.32	0.00497	2.06	0.00832	1.99	0.00957	1.90	0.0113	1.88	0.0140
10	2.68	0.00205	2.19	0.00672	2.01	0.00933	1.94	0.0107	1.82	0.0118	1.87	0.0118
20	2.48	0.00327	2.13	0.00774	1.98	0.0100	1.90	0.0118	1.84	0.0131	1.87	0.0118
30	2.38	0.00412	2.09	0.00851	1.96	0.0105	1.88	0.0124	1.85	0.0127	1.87	0.0118
40	2.32	0.00474	2.08	0.00871	1.93	0.0113	1.86	0.0131	1.86	0.0124	1.87	0.0118
50	2.30	0.00497	2.06	0.00913	1.93	0.0113	1.85	0.0134	1.86	0.0124	1.87	0.0118
60	2.29	0.00508	2.04	0.00891	1.92	0.0116	1.84	0.0137	1.86	0.0124	1.87	0.0118
80	2.28	0.00521	2.02	0.00933	1.92	0.0116	1.83	0.0140	1.86	0.0124	1.87	0.0118
100	2.27	0.00532	2.00	0.00979	1.92	0.0116	1.84	0.0137	1.86	0.0124	1.87	0.0118
120	2.26	0.00546	2.01	0.00957	1.92	0.0116	1.84	0.0137	1.86	0.0124	1.87	0.0118
180	2.26	0.00546	2.01	0.00957	1.92	0.0116	1.83	0.0140	1.83	0.0134	1.85	0.0124
240	2.24	0.00572	2.01	0.00957	1.91	0.0118	1.83	0.0140	1.83	0.0134	--	--
300	2.24	0.00572	1.99	0.0100	1.91	0.0118	1.83	0.0140	1.83	0.0134	1.84	0.0127
360	--	---	1.99	0.0100	1.90	0.0122	1.82	0.0144	1.83	0.0134	--	--
420	2.24	0.00572	2.00	0.00979	1.89	0.0124	--	--	--	--	1.84	0.0127
540	--	---	2.00	0.00979	1.89	0.0124	--	--	--	--	1.83	0.0131
780	--	---	--	---	--	--	--	--	--	--	1.81	0.0137
1380	--	---	--	---	--	--	--	--	--	--	1.78	0.0147

^aDry H-Zeo-Karb sample weighed 6.00 grams, and contained 0.865 meq. exchangeable and 0.086 meq. free hydrogen per gram dry H-Zeo-Karb. Water on moist sample weighed 4.1 grams.

^bInitial vol. distilled water added to resin sample was 90.9 ml.

^cThe CoCl_2 solution additions were 0.0888 N for all increments.

Table 29. Exchange of hydrogen from HCl solution with Co-Zeo-Karb (Run 15b)^a

Vol. HCl, ml. ^b	100.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.00		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.00	0.0105	1.86	0.0147	1.71	0.0212	1.51	0.0342	1.46	0.0378
10	2.02	0.0100	1.88	0.0140	1.73	0.0201	1.56	0.0303	1.47	0.0369
20	2.11	0.00811	1.89	0.0137	1.75	0.0192	1.56	0.0303	1.48	0.0360
30	2.13	0.00774	1.89	0.0137	1.76	0.0187	1.59	0.0297	1.48	0.0360
40	2.14	0.00755	--	---	1.77	0.0183	1.57	0.0297	1.48	0.0360
50	2.15	0.00737	1.90	0.0134	1.77	0.0183	1.57	0.0297	1.48	0.0360
60	2.16	0.00721	1.90	0.0134	1.78	0.0178	1.58	0.0289	1.48	0.0360
80	2.18	0.00688	1.91	0.0131	1.78	0.0178	1.57	0.0297	1.48	0.0360
100	2.18	0.00688	1.91	0.0131	1.78	0.0178	1.57	0.0297	1.48	0.0360
120	2.18	0.00688	1.92	0.0127	1.78	0.0178	1.58	0.0289	1.49	0.0351
150	2.18	0.00688	1.92	0.0127	1.78	0.0178	1.58	0.0289	1.49	0.0351
180	2.18	0.00688	1.92	0.0127	1.78	0.0178	1.58	0.0289	1.49	0.0351
240	2.18	0.00688	1.92	0.0127	1.78	0.0178	1.58	0.0289	1.50	0.0342
300	2.18	0.00688	1.92	0.0127	1.78	0.0178	--	---	1.50	0.0342
420	2.18	0.00688	--	---	--	---	--	---	1.50	0.0342

^a Dry Co-Zeo-Karb sample weighed 6.00 grams (as H-Zeo-Karb), and contained 2.0 grams free moisture.

^b The HCl solution additions were 0.0090 N for first increment, and 0.0902 N for last four increments.

Table 30. Exchange of sodium from NaCl solution with Amberlite H-IRC-50 (Run 16a)^{a, b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	3.45	0.000361	3.20	0.000642	3.16	0.000688	3.19	0.000642	3.19	0.000642
10	3.41	0.000398	3.19	0.000658	3.15	0.000704	3.18	0.000658	3.18	0.000658
20	3.35	0.000457	3.18	0.000673	3.15	0.000704	3.18	0.000658	3.18	0.000658
30	3.31	0.000500	3.18	0.000673	3.14	0.000721	3.17	0.000673	3.18	0.000658
40	3.29	0.000523	3.17	0.000688	3.14	0.000721	3.17	0.000673	3.18	0.000658
50	3.28	0.000535	3.17	0.000688	3.14	0.000721	3.16	0.000688	3.18	0.000658
60	3.27	0.000548	3.16	0.000704	3.14	0.000721	3.16	0.000688	3.18	0.000658
80	3.24	0.000587	3.16	0.000704	3.13	0.000737	3.15	0.000704	3.17	0.000673
100	3.23	0.000601	3.15	0.000721	3.12	0.000755	3.15	0.000704	3.17	0.000673
120	3.22	0.000613	3.15	0.000721	3.12	0.000755	3.15	0.000704	3.17	0.000673
150	3.21	0.000628	3.15	0.000721	3.12	0.000755	3.15	0.000704	3.17	0.000673
180	3.20	0.000642	3.12	0.000773	3.12	0.000755	3.14	0.000721	3.16	0.000688
240	3.19	0.000658	3.12	0.000773	3.12	0.000755	3.13	0.000737	3.16	0.000688
300	3.18	0.000673	3.12	0.000773	3.12	0.000755	3.13	0.000737	3.16	0.000688
420	3.18	0.000673	3.13	0.000755	3.12	0.000755	3.12	0.000755	3.16	0.000688
540	3.18	0.000673	--	---	--	---	--	---	3.16	0.000688

^aDry H-IRC-50 sample weighed 6.00 grams, and contained no free moisture.

^bInitial vol. distilled water (of 6.21 pH) added to resin sample was 100.0 ml.

^cThe NaCl solution additions were 0.1281 N for all increments.

Table 31. Exchange of hydrogen from HCl solution with Amberlite Na-IRC-50 (Run 16b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		25.0		10.0		25.0	
Increment No.	1		2		3		4		5	
Gorr. of pH reading	0.00		0.00		0.00		0.00		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.08	0.00871	1.87	0.0144	1.61	0.0269	1.05	0.0934	0.70	0.234
10	2.09	0.00851	1.87	0.0144	1.61	0.0269	1.05	0.0934	--	---
20	2.09	0.00850	1.87	0.0144	1.61	0.0269	1.06	0.1020	--	---
30	2.10	0.00832	1.87	0.0144	1.61	0.0269	1.06	0.1020	0.68	0.245
40	2.11	0.00811	1.87	0.0144	1.61	0.0269	--	---	--	---
50	2.12	0.00793	1.87	0.0144	1.61	0.0269	--	---	--	---
60	2.12	0.00793	1.87	0.0144	1.61	0.0269	1.06	0.1020	0.68	0.245
80	2.12	0.00793	1.88	0.0140	1.60	0.0277	--	---	--	---
100	2.12	0.00793	1.88	0.0140	1.60	0.0277	--	---	--	---
120	2.13	0.00774	1.88	0.0140	1.60	0.0277	1.06	0.1020	0.68	0.245
150	2.13	0.00774	1.88	0.0140	1.60	0.0277	--	---	--	---
180	2.13	0.00774	1.88	0.0140	--	---	--	---	--	---
240	2.13	0.00774	1.88	0.0140	1.60	0.0277	1.06	0.1020	0.68	0.245
300	2.14	0.00755	1.88	0.0140	--	---	--	---	0.68	0.245
420	2.15	0.00737	1.90	0.0134	1.60	0.0277	1.06	0.1020	0.68	0.245
600	2.15	0.00737	1.90	0.0134	1.60	0.0277	1.06	0.1020	0.68	0.245

^aDry Na-IRC-50 sample weighed 6.00 grams (as H-IRC-50), and contained 3.90 grams free moisture.

^bInitial vol. distilled water (of 6.13 pH) added to resin sample was 100.0 ml., after which pH was 5.32.

^cThe HCl solution additions were 0.1000 N for first three increments, and 1.2597 N for last two increments.

Table 32. Exchange of sodium from NaCl solution with Amberlite H-IR-100 (Run 17a)^{a, b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3d		4d		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	3.2	0.000642	2.30	0.00521	2.05	0.00913	2.09	0.00832	2.03	0.00957
10	2.9	0.00128	2.25	0.00584	2.00	0.0102	2.08	0.00850	2.02	0.00979
20	2.63	0.00241	2.22	0.00627	1.98	0.0107	2.02	0.00979	2.01	0.0100
30	2.54	0.00298	2.12	0.00793	1.98	0.0107	1.85	0.0147	1.99	0.0105
40	2.51	0.00319	2.11	0.00811	1.97	0.0110	1.81	0.0162	1.99	0.0105
50	2.47	0.00351	2.10	0.00832	1.96	0.0113	1.81	0.0162	1.99	0.0105
60	2.46	0.00359	2.10	0.00832	1.94	0.0118	1.81	0.0162	1.99	0.0105
80	2.40	0.00412	2.05	0.00933	1.91	0.0127	1.69	0.0217	1.99	0.0105
100	2.39	0.00421	2.03	0.00979	1.88	0.0137	1.80	0.0167	1.99	0.0105
120	2.38	0.00432	2.02	0.0100	1.85	0.0147	1.81	0.0162	1.99	0.0105
150	2.38	0.00432	2.02	0.0100	1.82	0.0158	1.59	0.0277	1.99	0.0105
180	2.37	0.00441	2.01	0.0102	2.02	0.00979	1.99	0.0105	1.99	0.0105
240	2.32	0.00497	2.00	0.0105	2.02	0.00979	1.98	0.0107	1.99	0.0105
300	2.31	0.00508	2.00	0.0105	2.02	0.00979	1.74	0.0192	1.99	0.0105
420	2.20	0.00658	2.00	0.0105	2.03	0.00957	1.99	0.0105	2.00	0.0102
540	2.20	0.00658	--	---	--	---	--	---	2.01	0.0100

^aDry H-IR-100 sample weighed 6.001 grams, and contained 7.50 grams free moisture.

^bInitial vol. distilled water (of 6.57 pH) added to resin sample was 100.0 ml., after which pH was 4.10.

^cThe NaCl solution additions were 0.1281 N for all increments.

^dLoose terminal on pH meter was discovered, accounting for these erratic readings.

Table 33. Exchange of hydrogen from HCl solution with Amberlite Na-IR-100 (Run 17b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		10.0		10.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.00		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.22	0.00627	2.06	0.00913	1.95	0.0118	1.88	0.0137	1.70	0.0212
10	2.24	0.00599	2.08	0.00871	1.96	0.0116	1.88	0.0137	1.71	0.0207
20	2.32	0.00497	2.12	0.00793	1.98	0.0110	1.89	0.0134	1.72	0.0201
30	2.38	0.00432	2.13	0.00774	2.00	0.0105	1.89	0.0134	1.72	0.0201
40	2.42	0.00394	2.15	0.00737	2.00	0.0105	1.90	0.0131	1.72	0.0201
50	2.43	0.00385	2.16	0.00721	2.01	0.0102	1.91	0.0127	1.73	0.0197
60	2.45	0.00368	2.17	0.00705	2.01	0.0102	1.91	0.0127	1.73	0.0197
80	2.46	0.00359	2.17	0.00705	2.01	0.0102	1.92	0.0124	1.73	0.0197
100	2.46	0.00359	2.18	0.00688	2.01	0.0102	1.92	0.0124	1.73	0.0197
120	2.46	0.00359	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.73	0.0197
150	2.46	0.00359	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.74	0.0192
180	2.47	0.00351	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.74	0.0192
240	2.48	0.00342	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.74	0.0192
300	2.48	0.00342	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.74	0.0192
420	2.48	0.00342	2.18	0.00688	2.02	0.0100	1.92	0.0124	1.74	0.0192

^aDry Na-IR-100 sample weighed 6.001 grams (as H-IR-100), and contained 2.30 grams free moisture.

^bInitial vol. distilled water (of 6.13 pH) added to resin sample was 100 ml., after which pH was 4.19.

^cThe HCl solution additions were 0.1000 N for all increments.

Table 34. Exchange of sodium from NaCl solution with Dow H-MX-30 (Run 18a)^{a,b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.00		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.70	0.00205	2.18	0.00688	2.02	0.0100	2.01	0.0100	1.94	0.0118
10	2.45	0.00368	2.13	0.00774	2.01	0.0102	1.99	0.0105	1.91	0.0127
20	2.35	0.00463	2.09	0.00850	1.99	0.0107	1.94	0.0118	1.89	0.0134
30	2.28	0.00546	2.06	0.00913	1.99	0.0107	1.93	0.0122	1.87	0.0140
40	2.27	0.00559	2.04	0.00957	1.98	0.0110	1.91	0.0127	1.87	0.0140
50	2.25	0.00584	2.03	0.00979	1.98	0.0110	1.90	0.0131	1.88	0.0137
60	2.24	0.00599	2.03	0.00979	1.98	0.0110	1.90	0.0131	1.83	0.0154
80	2.23	0.00627	2.02	0.0100	1.97	0.0113	1.90	0.0131	1.83	0.0154
100	2.21	0.00642	2.02	0.0100	1.97	0.0113	1.90	0.0131	1.89	0.0134
120	2.21	0.00642	2.02	0.0100	1.97	0.0113	1.89	0.0134	1.89	0.0134
150	2.21	0.00642	2.02	0.0100	1.97	0.0113	1.89	0.0134	1.89	0.0134
180	2.21	0.00642	2.02	0.0100	1.97	0.0113	1.89	0.0134	1.89	0.0134
240	2.21	0.00642	2.02	0.0100	1.97	0.0113	1.84	0.0151	1.89	0.0134
300	2.21	0.00642	2.02	0.0100	1.90	0.0134	1.89	0.0134	1.89	0.0134
420	2.21	0.00642	2.02	0.0100	1.91	0.0131	1.89	0.0134	1.89	0.0134
480	2.21	0.00642	2.02	0.0100	1.91	0.0131	--	---	--	---

^aDry H-MX-30 sample weighed 6.000 grams, and contained 4.80 grams free moisture.

^bInitial vol. distilled water (of 6.12 pH) added to resin sample was 100.0 ml., after which pH was 4.39.

^cThe NaCl solution additions were 0.1281 N for all increments.

Table 35. Exchange of hydrogen from HCl solution with Dow Na-MX-30 (Run 18b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		25.0		10.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.00		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.10	0.00832	1.95	0.0118	1.68	0.0228	1.07	0.0975	0.68	0.245
10	2.17	0.00705	2.00	0.0105	1.69	0.0222	1.08	0.0950	0.69	0.240
20	2.25	0.00584	2.05	0.00933	1.72	0.0207	1.08	0.0950	0.69	0.240
30	2.32	0.00497	2.07	0.00891	1.73	0.0201	1.08	0.0950	0.69	0.240
40	2.36	0.00453	2.08	0.00871	1.74	0.0197	1.08	0.0950	0.69	0.240
50	2.38	0.00432	2.11	0.00811	1.75	0.0192	1.09	0.0928	0.69	0.240
60	2.41	0.00402	2.12	0.00793	1.76	0.0187	1.09	0.0928	0.69	0.240
80	2.42	0.00394	2.12	0.00793	1.77	0.0183	1.10	0.0905	0.69	0.240
100	2.42	0.00394	2.13	0.00774	1.77	0.0183	1.10	0.0905	0.69	0.240
120	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240
150	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240
180	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240
240	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240
300	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240
420	2.44	0.00376	2.13	0.00774	1.78	0.0178	1.11	0.0884	0.69	0.240

^aDry Na-MX-30 sample weighed 8.000 grams (as H-MX-30), and contained 4.30 grams free moisture.

^bInitial vol. distilled water (of 6.13 pH) added to resin sample was 100.0 ml., after which pH was 4.77.

^cThe HCl solution additions were 0.1000 N for first three increments, and 1.2597 N for last two increments.

Table 36. Exchange of sodium from NaCl solution with Amberlite H-IRC-50 (Run 19a)^{a,b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr of pH reading	0.00		0.01		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	4.32	0.0000488	3.78	0.000165	3.59	0.000256	3.59	0.000256	3.54	0.000287
10	4.20	0.0000642	3.75	0.000177	3.58	0.000262	3.58	0.000262	3.54	0.000287
20	4.08	0.0000846	3.73	0.000185	3.57	0.000268	3.57	0.000268	3.53	0.000293
30	4.02	0.0000972	3.72	0.000189	3.57	0.000268	3.56	0.000274	3.53	0.000293
40	3.98	0.000107	3.71	0.000194	3.56	0.000274	3.55	0.000280	3.53	0.000293
50	3.96	0.000112	3.70	0.000197	3.56	0.000274	3.54	0.000287	3.52	0.000300
60	3.93	0.000119	3.69	0.000202	3.56	0.000274	3.54	0.000287	3.52	0.000300
80	3.89	0.000132	3.68	0.000207	3.54	0.000287	3.52	0.000300	3.52	0.000300
100	3.87	0.000137	3.68	0.000207	3.53	0.000293	3.52	0.000300	3.51	0.000308
120	3.86	0.000140	3.67	0.000212	3.53	0.000293	3.51	0.000308	3.51	0.000308
150	3.83	0.000151	3.67	0.000212	3.53	0.000293	3.51	0.000308	3.51	0.000308
180	3.82	0.000154	3.66	0.000217	3.53	0.000293	3.51	0.000308	3.51	0.000308
240	3.80	0.000162	3.65	0.000222	3.53	0.000293	3.50	0.000314	3.50	0.000314
300	3.79	0.000165	3.64	0.000228	3.53	0.000293	3.50	0.000314	3.50	0.000314
420	3.78	0.000168	3.62	0.000238	3.53	0.000293	3.50	0.000314	3.50	0.000314
720	--	---	3.56	0.000274	--	---	--	---	3.50	0.000314

^aDry H-IRC-50 sample weighed 6.004 grams, and contained 3.60 grams free moisture.

^bInitial vol. distilled water (of 6.82 pH) added to resin sample was 100.0 ml., after which pH was 4.67.

^cThe NaCl solution additions were 0.1281 N for all increments.

Table 37. Exchange of hydrogen from HCl solution with Amberlite Na-IRC-50 (Run 19b)^{a, b}

Vol. HCl, ml. ^c	10.0		25.0		10.0		5.0	
Increment No.	1		2		3		4	
Corr. of pH reading	0.00		0.00		0.00		0.00	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.08	0.00871	1.66	0.0238	1.04	0.0957	---	---
10	2.08	0.00871	1.67	0.0232	1.05	0.0934	0.42	0.466
20	2.09	0.00851	1.67	0.0232	1.03	0.0979	0.42	0.466
30	2.10	0.00832	1.67	0.0232	1.03	0.0979	0.42	0.466
40	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.42	0.466
50	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.42	0.466
60	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.42	0.466
80	2.09	0.00851	1.67	0.0232	1.04	0.0957	0.41	0.478
100	2.11	0.00811	1.67	0.0232	1.01	0.116	0.40	0.489
120	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.40	0.489
150	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.40	0.489
180	2.11	0.00811	1.67	0.0232	1.04	0.0957	0.40	0.489
240	2.12	0.00793	1.67	0.0232	1.04	0.0957	0.40	0.489
300	2.13	0.00774	1.67	0.0232	1.04	0.0957	0.40	0.489
360	2.13	0.00774	1.67	0.0232	1.04	0.0957	0.40	0.489
420	2.14	0.00755	1.67	0.0232	1.04	0.0957	0.40	0.489
540	2.15	0.00737	1.67	0.0232	1.04	0.0957	0.40	0.489
600	2.15	0.00737	1.67	0.0232	1.04	0.0957	0.40	0.489

^aDry Na-IRC-50 sample weighed 6.004 grams (as H-IRC-50), and contained 4.60 grams free moisture.

^bInitial vol. distilled water (of 6.12 pH) added to resin sample was 100 ml., after which pH was 4.98.

^cThe HCl solution additions were 0.100 N for first two increments, 1.2597 N for third, and 12.55 N for last increment.

Table 38. Exchange of sodium from NaCl solution with Amberlite H-IR-100 (Run 20a)^{a, b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	3.14	0.000737	2.61	0.00252	2.49	0.00327	2.42	0.00385	2.39	0.00402
10	2.91	0.00127	2.57	0.00278	2.43	0.00376	2.40	0.00402	2.35	0.00453
20	2.77	0.00174	2.51	0.00319	2.41	0.00394	2.37	0.00432	2.33	0.00474
30	2.69	0.00210	2.49	0.00333	2.40	0.00412	2.35	0.00453	2.33	0.00474
40	2.66	0.00225	2.49	0.00333	2.39	0.00412	2.33	0.00474	2.32	0.00486
50	2.65	0.00230	2.48	0.00342	2.39	0.00412	2.32	0.00486	2.31	0.00497
60	2.64	0.00236	2.47	0.00351	2.39	0.00412	2.32	0.00486	2.31	0.00497
80	2.62	0.00247	2.46	0.00359	2.38	0.00421	2.32	0.00486	2.31	0.00497
100	2.61	0.00252	2.46	0.00359	2.38	0.00421	2.31	0.00497	2.31	0.00497
120	2.61	0.00252	2.46	0.00359	2.38	0.00421	2.31	0.00497	2.31	0.00497
150	2.61	0.00252	2.45	0.00368	2.38	0.00421	2.31	0.00497	2.31	0.00497
180	2.61	0.00252	2.45	0.00368	2.38	0.00421	2.31	0.00497	2.31	0.00497
240	2.62	0.00247	2.45	0.00368	2.38	0.00421	2.31	0.00497	2.31	0.00497
300	2.61	0.00252	2.45	0.00368	2.38	0.00421	2.31	0.00497	2.31	0.00497
420	2.63	0.00241	2.47	0.00351	2.38	0.00421	2.31	0.00497	2.31	0.00497

^aDry H-IR-100 sample weighed 6.00 grams, and contained 3.10 grams free moisture.

^bInitial vol. distilled water (of 6.62 pH) added to resin sample was 100 ml., after which pH was 4.46.

^cThe NaCl solution additions were 0.1281 N for all increments.

Table 39. Exchange of hydrogen from HCl solution with Amberlite Na-IR-100 (Run 20b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.27	0.00559	2.18	0.00688	2.07	0.00871	1.86	0.0144	1.76	0.0183
10	2.34	0.00474	2.22	0.00627	2.08	0.00851	1.86	0.0144	1.77	0.0188
20	2.47	0.00351	2.27	0.00559	2.11	0.00793	1.87	0.0140	1.78	0.0174
30	2.52	0.00312	2.28	0.00546	2.12	0.00774	1.88	0.0137	1.78	0.0174
40	2.57	0.00278	2.29	0.00532	2.14	0.00737	1.88	0.0137	1.78	0.0174
50	2.58	0.00271	2.31	0.00508	2.15	0.00721	1.89	0.0134	1.79	0.0170
60	2.59	0.00266	2.32	0.00497	2.15	0.00721	1.89	0.0134	1.79	0.0170
80	2.59	0.00266	2.32	0.00497	2.16	0.00705	1.90	0.0131	1.79	0.0170
100	2.61	0.00252	2.33	0.00486	2.18	0.00672	1.91	0.0127	1.79	0.0170
120	2.62	0.00247	2.33	0.00486	2.17	0.00688	1.91	0.0127	1.79	0.0170
150	2.63	0.00241	2.35	0.00463	2.16	0.00705	1.91	0.0127	1.80	0.0167
180	2.64	0.00236	2.35	0.00463	2.18	0.00672	1.92	0.0124	1.80	0.0167
240	2.66	0.00225	2.35	0.00463	2.19	0.00658	1.92	0.0124	1.80	0.0167
300	2.67	0.00220	2.35	0.00463	2.19	0.00658	1.92	0.0124	1.80	0.0167
480	2.68	0.00214	2.35	0.00463	2.19	0.00658	1.92	0.0122	1.81	0.0162

^aDry Na-IR-100 sample weighed 6.00 grams (as H-IR-100), and contained 3.00 grams free moisture.

^bInitial vol. distilled water (of 5.98 pH) added to resin sample was 100.0 ml., after which pH was 4.98.

^cThe HCl solution additions were 0.1000 N for all increments.

Table 40. Exchange of sodium from NaCl solution with Dow H-MX-30 (Run 21a)^{a,b}

Vol. NaCl, ml. ^c	10.0		10.0		10.0		25.0		25.0		25.0	
Increment No.	1		2		3		4		5		6	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	3.09	0.000829	2.68	0.00214	2.43	0.00376	2.41	0.00394	2.34	0.00463	2.35	0.00453
10	2.83	0.00152	2.57	0.00278	2.41	0.00394	2.38	0.00421	2.32	0.00486	2.34	0.00463
20	2.72	0.00196	2.50	0.00327	2.38	0.00421	2.34	0.00463	2.31	0.00508	2.32	0.00486
30	2.68	0.00214	2.48	0.00342	2.38	0.00432	2.31	0.00497	2.30	0.00508	2.31	0.00497
40	2.63	0.00241	2.47	0.00351	2.38	0.00432	2.31	0.00497	2.30	0.00508	2.31	0.00497
50	2.62	0.00247	2.46	0.00359	2.37	0.00432	2.30	0.00508	2.30	0.00508	2.31	0.00497
60	2.61	0.00252	2.45	0.00368	2.37	0.00432	2.30	0.00508	2.29	0.00521	2.31	0.00497
80	2.60	0.00259	2.44	0.00376	2.35	0.00453	2.30	0.00508	2.29	0.00521	2.31	0.00497
100	2.59	0.00266	2.44	0.00376	2.35	0.00453	2.29	0.00521	2.29	0.00521	2.31	0.00497
120	2.60	0.00259	2.44	0.00376	2.35	0.00453	2.29	0.00521	2.29	0.00521	2.32	0.00486
150	2.60	0.00259	2.44	0.00376	2.35	0.00453	2.28	0.00532	2.29	0.00521	2.32	0.00486
180	2.59	0.00266	2.44	0.00376	2.35	0.00453	2.28	0.00532	2.29	0.00521	2.32	0.00486
240	2.59	0.00266	2.44	0.00376	2.35	0.00453	2.28	0.00532	2.29	0.00521	2.32	0.00486
300	2.59	0.00266	2.44	0.00376	2.35	0.00453	2.29	0.00521	2.29	0.00521	2.32	0.00486
420	2.59	0.00266	2.44	0.00376	2.37	0.00432	2.30	0.00508	2.30	0.00508	2.32	0.00486
600	--	---	--	---	--	---	--	---	--	---	2.33	0.00474
900	--	---	--	---	--	---	--	---	--	---	2.34	0.00463
1200	--	---	--	---	--	---	--	---	--	---	2.38	0.00421
1500	--	---	--	---	--	---	--	---	--	---	2.38	0.00421

^aDry H-MX-30 sample weighed 6.00 grams and contained 4.10 grams free moisture.

^bInitial vol. distilled water (of 6.50 pH) added to resin sample was 100.0 ml., after which pH was 3.96.

^cThe NaCl solution additions were 0.1281 N for all increments.

Table 41. Exchange of hydrogen from HCl solution with Dow Na-MX-30 (Run 21b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		25.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.01		0.01		0.01	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.15	0.00737	2.18	0.00688	1.89	0.0134	1.82	0.0158	1.73	0.0197
10	2.34	0.00474	2.21	0.00642	1.92	0.0124	1.82	0.0158	1.76	0.0182
20	2.47	0.00351	2.28	0.00546	1.98	0.0107	1.83	0.0154	1.77	0.0178
30	2.57	0.00278	2.32	0.00497	1.99	0.0105	1.84	0.0151	1.78	0.0174
40	2.61	0.00252	2.36	0.00453	1.99	0.0105	1.86	0.0144	1.78	0.0174
50	2.66	0.00225	2.37	0.00441	2.00	0.0102	1.87	0.0140	1.78	0.0174
60	2.68	0.00214	2.38	0.00432	2.01	0.0100	1.87	0.0140	1.78	0.0174
80	2.70	0.00205	2.39	0.00421	2.02	0.00979	1.87	0.0140	1.78	0.0174
100	2.71	0.00201	2.39	0.00421	2.03	0.00957	1.87	0.0140	1.78	0.0174
120	2.72	0.00196	2.39	0.00421	2.03	0.00957	1.87	0.0140	1.78	0.0174
150	2.72	0.00196	2.40	0.00412	2.04	0.00933	1.88	0.0137	1.78	0.0174
180	2.72	0.00196	2.41	0.00402	2.05	0.00913	1.88	0.0137	1.78	0.0174
240	2.73	0.00191	2.42	0.00394	2.06	0.00891	1.88	0.0137	1.78	0.0174
300	2.74	0.00187	2.43	0.00385	2.06	0.00891	1.88	0.0137	1.78	0.0174
480	2.75	0.00183	2.44	0.00376	2.07	0.00871	1.89	0.0134	1.79	0.0170

^aDry Na-MX-30 sample weighed 6.00 grams (as H-MX-30), and contained 8.00 grams free moisture.

^bInitial vol. distilled water (of 6.67 pH) added to resin sample was 100 ml., after which pH was 4.92.

^cThe HCl solution additions were 0.1000 N for all increments.

Table 42. Exchange of nickel from NiCl₂ solution with Amberlite H-IR-100 (Run 22a)^{a, b}

Vol. NiCl ₂ , ml. ^c	2.0		2.0		10.0		10.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.01		0.02		0.05		0.07		0.10	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.70	0.00201	2.05	0.00891	1.75	0.0170	1.55	0.0262	1.58	0.0228
10	2.44	0.00368	2.00	0.0100	1.70	0.0192	1.54	0.0269	1.58	0.0228
20	2.24	0.00584	1.92	0.0122	1.63	0.0228	1.54	0.0269	1.57	0.0232
30	2.17	0.00688	1.88	0.0134	1.61	0.0238	1.54	0.0269	1.57	0.0232
40	2.13	0.00755	1.87	0.0137	1.59	0.0250	1.54	0.0269	1.57	0.0232
50	2.12	0.00774	1.86	0.0140	1.58	0.0256	1.54	0.0269	1.57	0.0232
60	2.10	0.00811	1.86	0.0140	1.57	0.0262	1.54	0.0269	1.57	0.0232
80	2.10	0.00811	1.86	0.0140	1.55	0.0277	1.54	0.0269	1.57	0.0232
100	2.10	0.00811	1.86	0.0140	1.54	0.0283	1.54	0.0269	1.57	0.0232
120	2.10	0.00811	1.86	0.0140	1.53	0.0289	1.54	0.0269	1.57	0.0232
150	2.10	0.00811	1.85	0.0144	1.53	0.0289	1.54	0.0269	1.57	0.0232
180	2.10	0.00811	1.85	0.0144	1.52	0.0297	1.50	0.0297	1.57	0.0232
240	2.10	0.00811	1.85	0.0144	1.52	0.0297	1.50	0.0297	1.57	0.0232
300	2.10	0.00811	1.85	0.0144	1.52	0.0297	1.50	0.0297	1.57	0.0232
420	2.10	0.00811	1.85	0.0144	1.52	0.0297	1.50	0.0297	1.57	0.0232

^aDry H-IR-100 sample weighed 6.00 grams, and contained no free moisture.

^bInitial vol. distilled water (of 6.20 pH) added to resin sample was 100.0 ml., after which pH was 4.03.

^cThe NiCl₂ solution additions were 0.606 N for all increments.

^dAt end of last increment, solution analyzed 4.46 grams nickel per liter (by Ni-dimethylglyoxime precipitate).

Table 43. Exchange of hydrogen from HCl solution with Amberlite Ni-IR-100 (Run 22b)^{a, b}

Vol. HCl, ml. ^c	10.0		10.0		25.0		5.0		25.0	
Increment No.	1		2		3		4		5 ^d	
Corr. of pH reading	0.00		0.01		0.02		0.03		0.07	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.12	0.00793	1.91	0.0127	1.70	0.0207	--	---	1.20	0.0613
10	2.14	0.00755	1.94	0.0118	1.70	0.0207	1.37	0.0449	0.97	0.0957
20	2.16	0.00721	1.96	0.0113	1.71	0.0201	1.37	0.0449	0.85	0.143
30	2.17	0.00705	1.98	0.0107	1.71	0.0201	1.38	0.0438	0.85	0.143
40	2.18	0.00688	1.99	0.0105	1.71	0.0201	1.38	0.0438	0.85	0.143
50	2.19	0.00672	1.99	0.0105	1.72	0.0197	1.39	0.0428	0.85	0.143
60	2.19	0.00672	1.99	0.0105	1.72	0.0197	1.39	0.0428	0.85	0.143
80	2.19	0.00672	1.99	0.0105	1.73	0.0192	1.39	0.0428	0.85	0.143
100	2.20	0.00658	1.99	0.0105	1.73	0.0192	1.39	0.0428	0.85	0.143
120	2.20	0.00658	1.99	0.0105	1.73	0.0192	1.39	0.0428	0.85	0.143
150	2.20	0.00658	1.99	0.0105	1.73	0.0192	1.39	0.0428	0.85	0.143
180	2.21	0.00642	1.99	0.0105	1.73	0.0192	1.40	0.0417	0.85	0.143
240	2.21	0.00642	1.99	0.0105	1.73	0.0192	1.40	0.0417	0.85	0.143
300	2.21	0.00642	1.99	0.0105	1.73	0.0192	1.40	0.0417	0.85	0.143
420	2.21	0.00642	2.00	0.0102	1.73	0.0192	1.40	0.0417	0.86	0.139

^aDry Ni-IR-100 sample weighed 6.00 grams (as H-IR-100), and contained no free moisture.

^bInitial vol. distilled water (of 6.18 pH) added to resin samples was 100.0 ml., after which pH was 4.27.

^cThe HCl solution additions were 0.1000 N for first three increments, and 1.2597 N for last two increments.

^dAt end of last increment, solution analyzed 0.6502 grams nickel per liter (by Ni-dimethylglyoxime precipitate).

Table 44. Exchange of calcium from CaCl_2 with Amberlite H-IR-100. (Run 23a)^{a,b}

Vol. CaCl_2 , ml. ^c	10.0		10.0		10.0		25.0		25.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.02		0.03		0.04		0.05		0.06	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.87	0.00132	2.10	0.00774	1.88	0.0127	1.79	0.0154	1.71	0.0188
10	2.49	0.00319	2.04	0.00891	1.85	0.0137	1.75	0.0170	1.71	0.0188
20	2.28	0.00521	1.97	0.0105	1.81	0.0151	1.71	0.0187	1.71	0.0188
30	2.19	0.00642	1.93	0.0116	1.79	0.0158	1.70	0.0192	1.70	0.0187
40	2.16	0.00688	1.92	0.0118	1.78	0.0162	1.69	0.0197	1.70	0.0187
50	2.14	0.00721	1.91	0.0122	1.77	0.0167	1.68	0.0201	1.70	0.0187
60	2.13	0.00737	1.89	0.0127	1.77	0.0167	1.68	0.0201	1.70	0.0187
80	2.11	0.00774	1.86	0.0137	1.76	0.0170	1.68	0.0201	1.68	0.0197
100	2.11	0.00774	1.85	0.0140	1.75	0.0174	1.68	0.0201	1.68	0.0197
120	2.11	0.00774	1.85	0.0140	1.75	0.0174	1.67	0.0207	1.68	0.0197
150	2.11	0.00774	1.86	0.0137	1.75	0.0174	1.67	0.0207	1.68	0.0197
180	2.11	0.00774	1.86	0.0137	1.75	0.0174	1.66	0.0212	1.68	0.0197
240	2.11	0.00774	1.86	0.0137	1.75	0.0174	1.66	0.0212	1.68	0.0197
300	2.11	0.00774	1.86	0.0137	1.75	0.0174	1.66	0.0212	1.68	0.0197
420	2.11	0.00774	1.87	0.0134	1.75	0.0174	1.66	0.0212	1.65	0.0212

^aDry H-IR-100 sample weighed 6.00 grams, and contained no free moisture.

^bInitial vol. distilled water (of 6.23 pH) added to resin sample was 100.0 ml., after which pH was 3.72.

^cThe CaCl_2 solution additions were 0.200 N for all increments.

Table 45. Exchange of hydrogen from HCl solution with Amberlite Ca-IR-100 (Run 23b)^{a,b}

Vol. HCl, ml. ^c	10.0		10.0		5.0		2.0		5.0	
Increment No.	1		2		3		4		5	
Corr. of pH reading	0.00		0.00		0.02		0.05		0.07	
Time, sec.	pH	N.	pH	N.	pH	N.	pH	N.	pH	N.
5	2.13	0.00774	1.90	0.0134	1.55	0.0297	0.72	0.203	0.50	0.326
10	2.13	0.00774	1.90	0.0134	1.31	0.0531	0.73	0.198	0.37	0.445
20	2.14	0.00755	1.91	0.0131	1.32	0.0518	0.73	0.198	0.35	0.466
30	2.15	0.00737	1.91	0.0131	1.32	0.0518	0.77	0.181	0.34	0.478
40	2.15	0.00737	1.91	0.0131	1.33	0.0505	0.78	0.177	0.32	0.500
50	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.31	0.512
60	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.30	0.526
80	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.79	0.172	0.30	0.526
100	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.79	0.172	0.30	0.526
120	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.79	0.172	0.31	0.512
150	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.31	0.512
180	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.31	0.512
240	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.31	0.512
300	2.16	0.00721	1.91	0.0131	1.33	0.0505	0.78	0.177	0.31	0.512
420	2.16	0.00721	1.91	0.0131	1.33	0.0505	--	--	0.31	0.512

^aDry Ca-IR-100 weighed 6.00 grams (as H-IR-100), and contained no free moisture.

^bInitial vol. distilled water (of 6.17 pH) added to resin sample was 100.0 ml., after which pH was 4.38.

^cThe HCl solution additions were 0.1000 N for first two increments, 1.2597 N for third, and 12.55 N for last two increments.

Table 46. Equilibrium calculations for sodium chloride with H-Zeo-Karb

Run No.	Ion in solution	Vol. of solution, ml.	Before exchanger		At exchange equilibrium			
			H or Na in exchanger, meq.	Na ⁺ or H ⁺ in solution, meq.	In exchanger		In solution	
					H, meq.	Na, meq.	H ⁺ , meq.	Na ⁺ , meq.
7a	Na ⁺	266.1	0.865	4.877	0.397	0.468	0.453	4.409
8a	Na ⁺	187.5	2.595	19.057	1.058	1.537	1.509	17.520
12a	Na ⁺	138.3	5.190	6.981	3.275	1.915	1.901	5.066
7b	H ⁺	136.2	0.468	1.311	0.910	-0.045	0.798	0.513
8b	H ⁺	137.9	1.537	4.318	2.716	-0.121	2.660	1.658
12b	H ⁺	112.1	1.915	9.922	5.798	-0.608	7.399	2.523

Table 47. Rate of exchange calculations to engineering units

Run No.	Time, min.	meq./g.	Rates of exchange		
			dx/dt , meq./g./min.	meq. per hr.-lb.	lb. per hr.-lb.
7a	1	0.266	0.01570	0.942	21.67
	2	0.361	0.01443	0.867	19.93
	3	0.379	0.01212	0.728	16.76
	5	0.401	0.00927	0.557	12.80
	8	0.423	0.00622	0.373	8.58
	12	0.441	0.00301	0.180	4.14
	20	0.462	0.00126	0.0758	1.746
7b	1	0.297	0.412	2.47	2.49
	2	0.419	0.049	2.93	2.96
	3	0.451	0.0237	1.42	1.43
	5	0.484	0.0087	0.522	0.527
	8	0.495	0.00217	0.150	0.151
	12	0.502	0.0012	0.0722	0.728
	20	0.510	0.0007	0.0420	0.424
8a	1	0.454	0.02634	1.58	36.32
	2	0.470	0.02208	1.32	30.34
	3	0.480	0.01862	1.12	25.74
	5	0.496	0.01307	0.783	18.02
	8	0.503	0.00777	0.467	10.73
	12	0.506	0.00389	0.233	5.36
	20	0.511	0.00093	0.0558	1.284
8b	1	0.393	0.0854	5.12	5.17
	2	0.433	0.0383	2.30	2.32
	3	0.477	0.0206	1.23	1.24
	5	0.507	0.0130	0.780	0.787
	8	0.530	0.00576	0.347	0.350
	12	0.547	0.00194	0.117	0.118

Table 47 (continued)

Run No.	Time, min.	meq./g.	Rates of exchange		
			dx/dt, meq./g./min.	meq. per hr.-lb.	lb. per hr.-lb.
1	0.535	0.259	7.60	304.6	
2	0.592	0.0387	1.16	46.50	
3	0.622	0.0198	0.593	23.75	
5	0.642	0.00462	0.138	5.53	
8	0.652	0.00299	0.0897	3.59	
12	0.662	0.0019	0.0570	2.28	
20	0.672	0.0010	0.0300	1.202	
1	0.263	0.0438	2.63	2.65	
2	0.302	0.0304	1.82	1.84	
3	0.342	0.01785	1.07	1.08	
5	0.347	0.00491	0.295	0.298	
8	0.350	0.00140	0.0842	0.849	
12	0.353	0.00025	0.0150	0.0151	
1	0.590	0.1600	4.80	304.8	
2	0.678	0.0537	1.61	102.3	
3	0.720	0.0262	0.787	50.0	
5	0.750	0.0123	0.368	23.35	
8	0.775	0.00482	0.145	9.22	
12	0.785	0.00189	0.0567	3.600	
20	0.800	0.00154	0.0462	2.932	
1	0.418	0.1020	3.070	345.10	
2	0.4817	0.0362	1.080	121.40	
3	0.505	0.0203	0.610	68.60	
5	0.533	0.00846	0.255	28.67	
8	0.548	0.00339	0.102	11.47	
12	0.555	0.00105	0.031	3.54	
1	0.294	0.01210	0.727	16.720	
2	0.298	0.00427	0.257	5.920	
3	0.308	0.00174	0.104	2.392	
5	0.313	0.00050	0.0300	0.690	
8	0.316	0.00018	0.0108	0.249	
12	0.317	0.00008	0.0048	0.110	

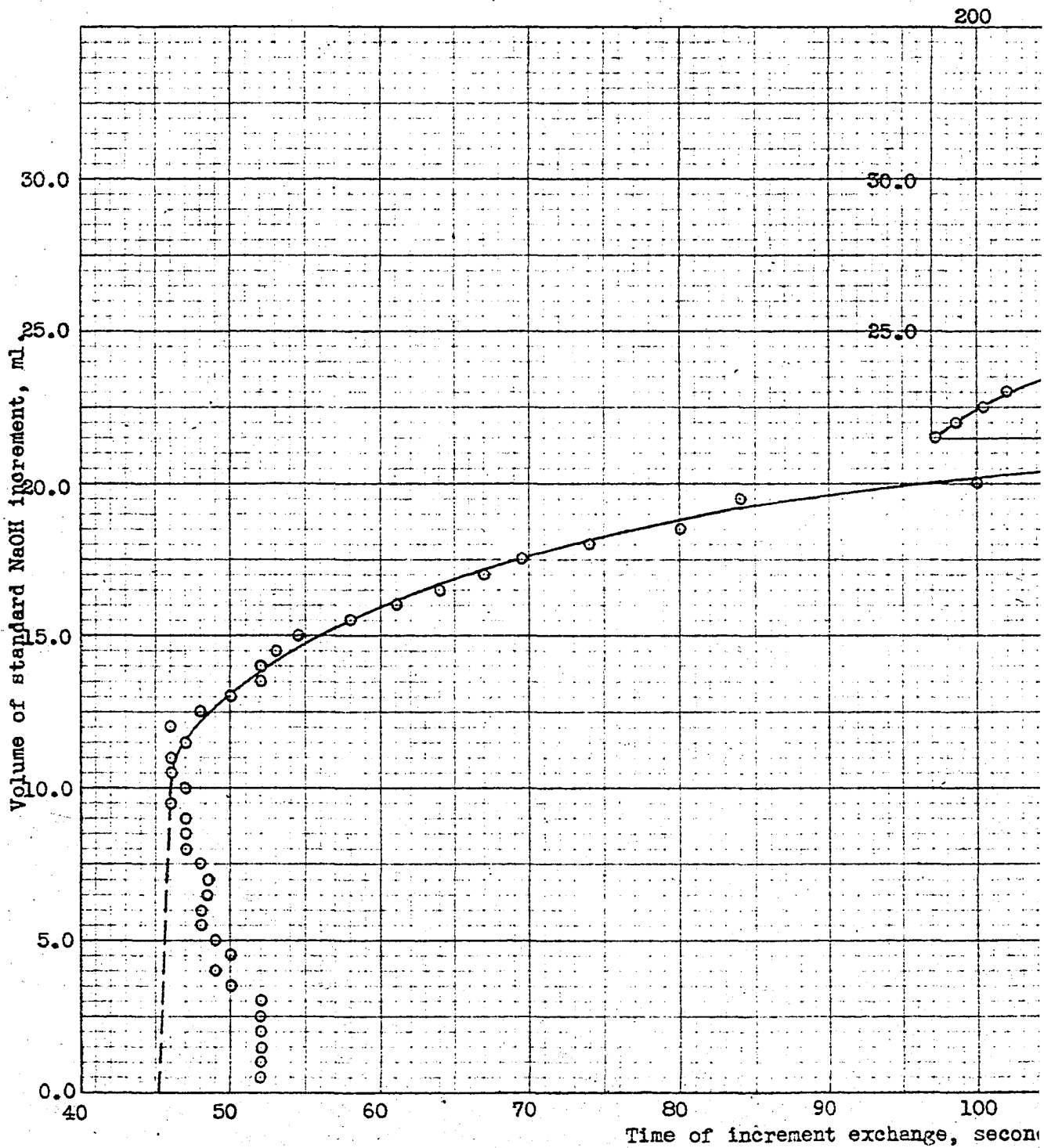
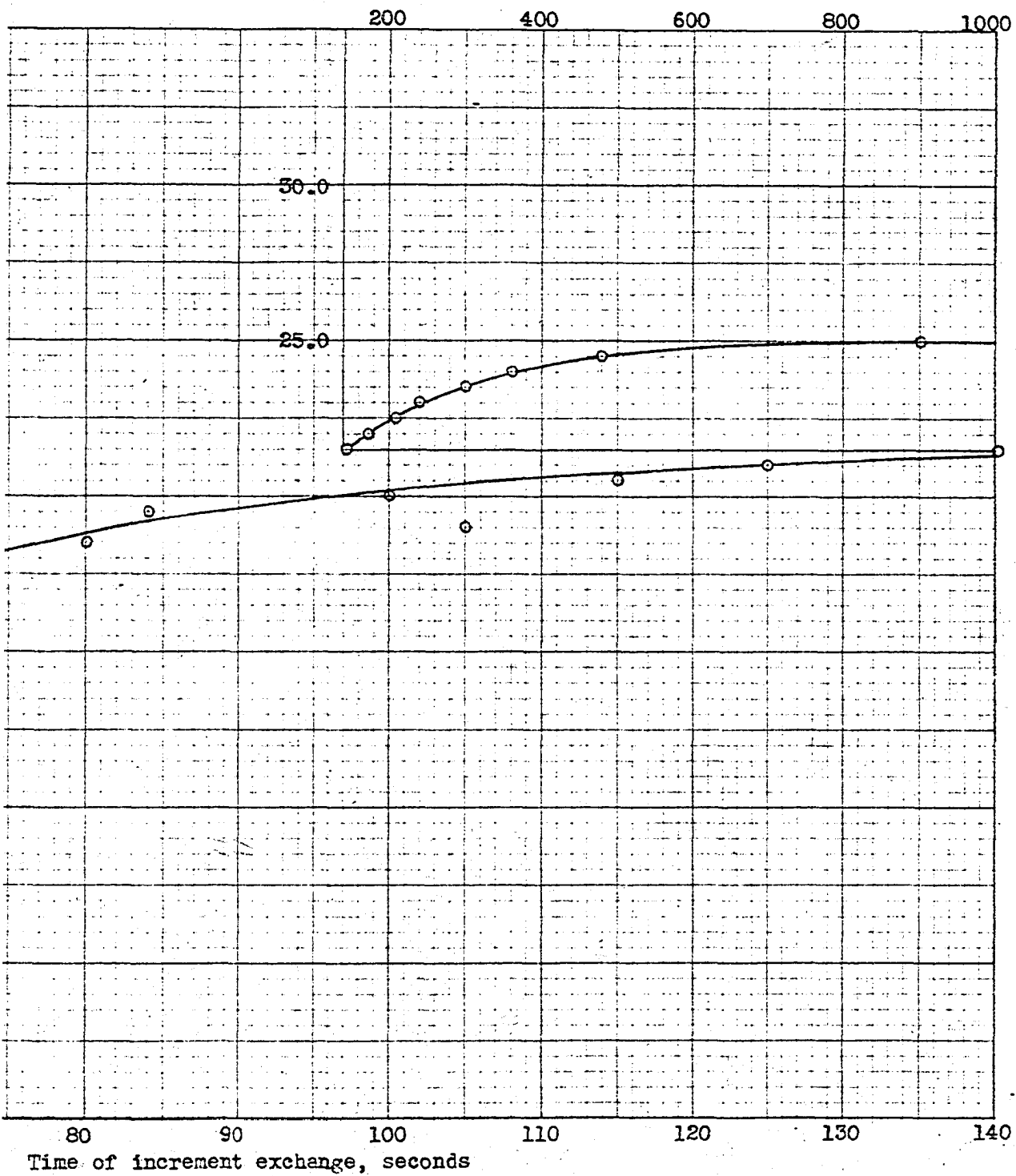


Fig. 18. Typical curve for rate of exchange of H-Zeo-Karb at constant i



exchange of H-Zeo-Karb at constant incremental concentration in solution.

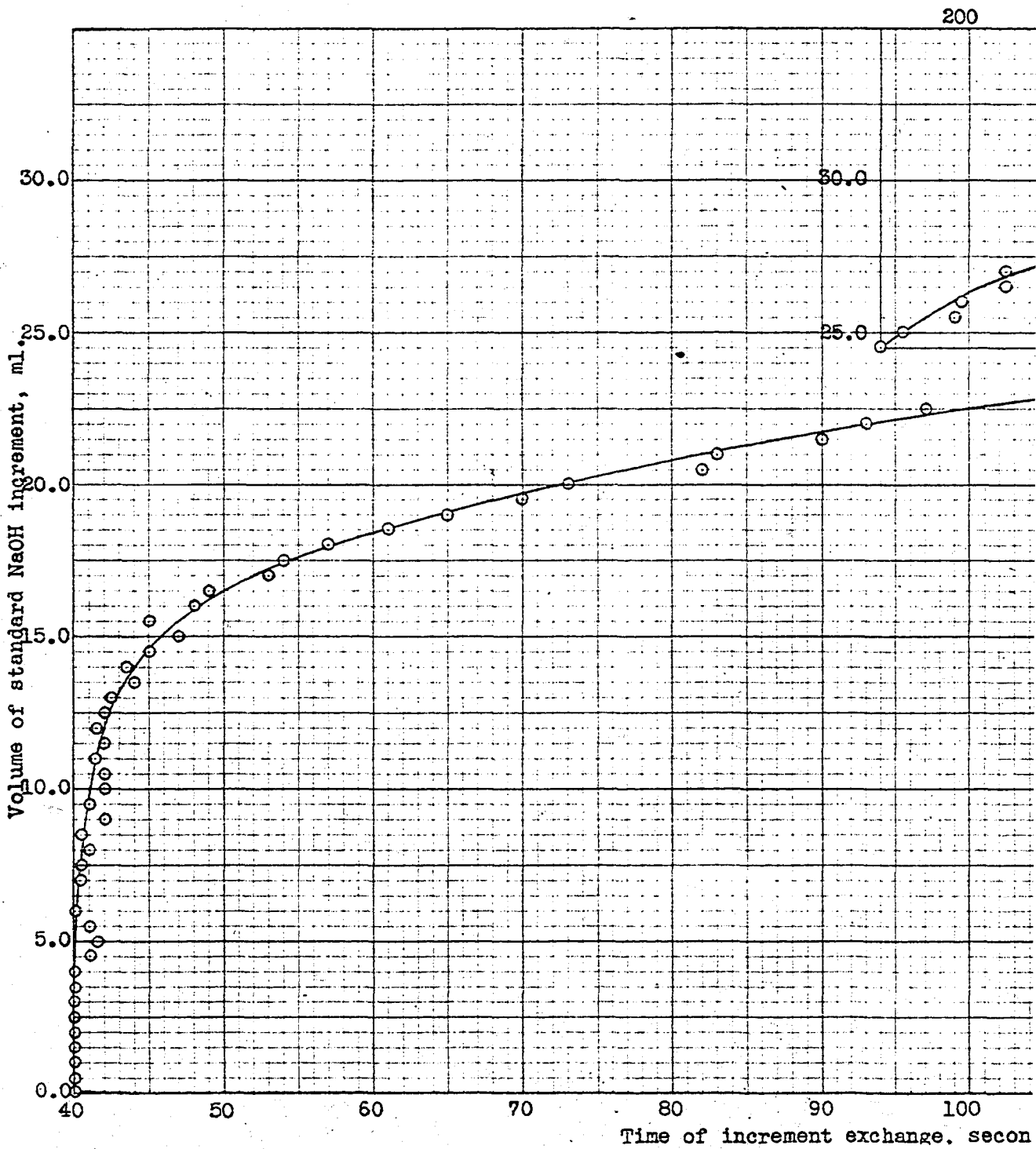
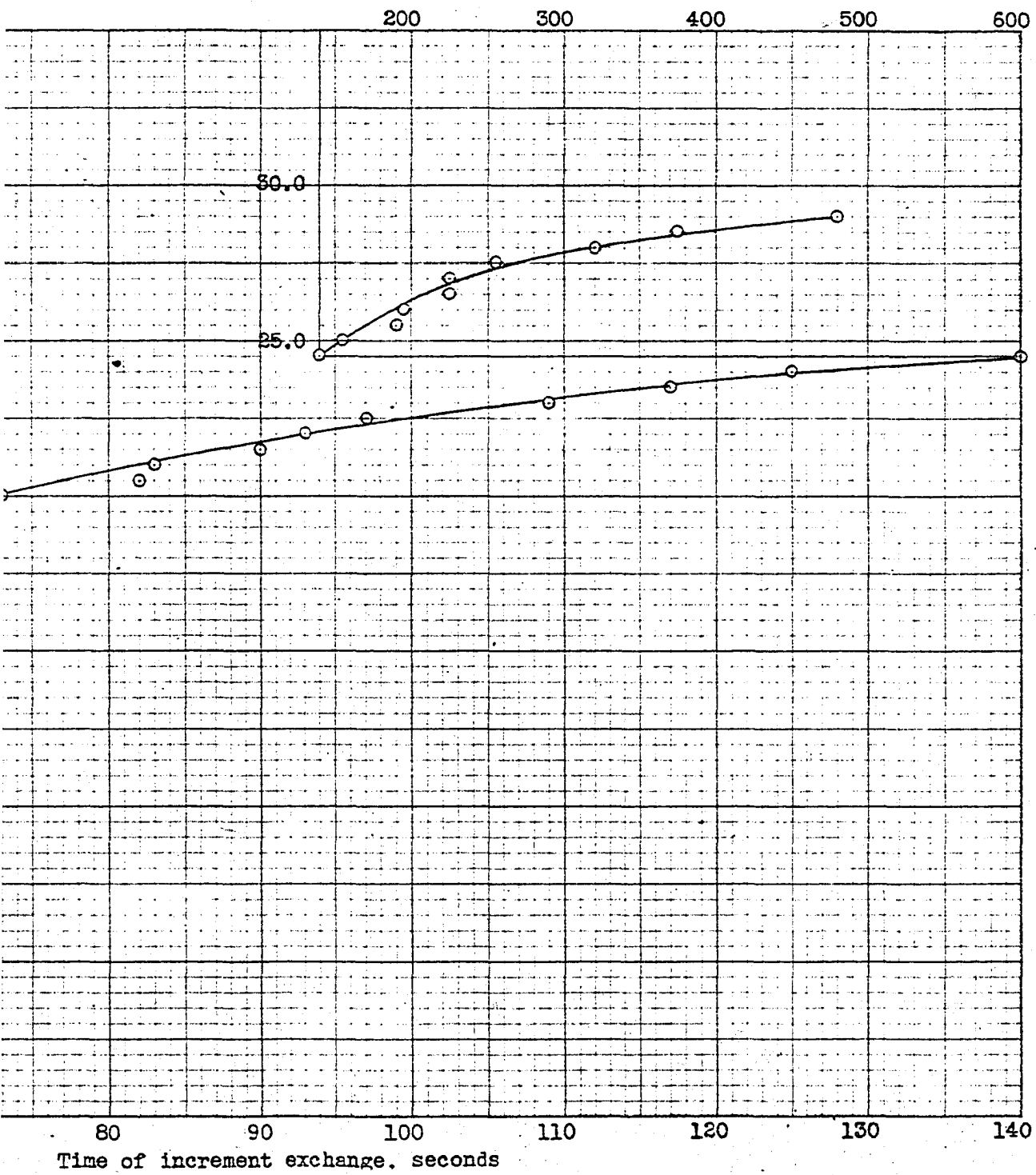


Fig. 19. Effect of initial washing on rate of exch



Effect of initial washing on rate of exchange of H-Zeo-Karb.

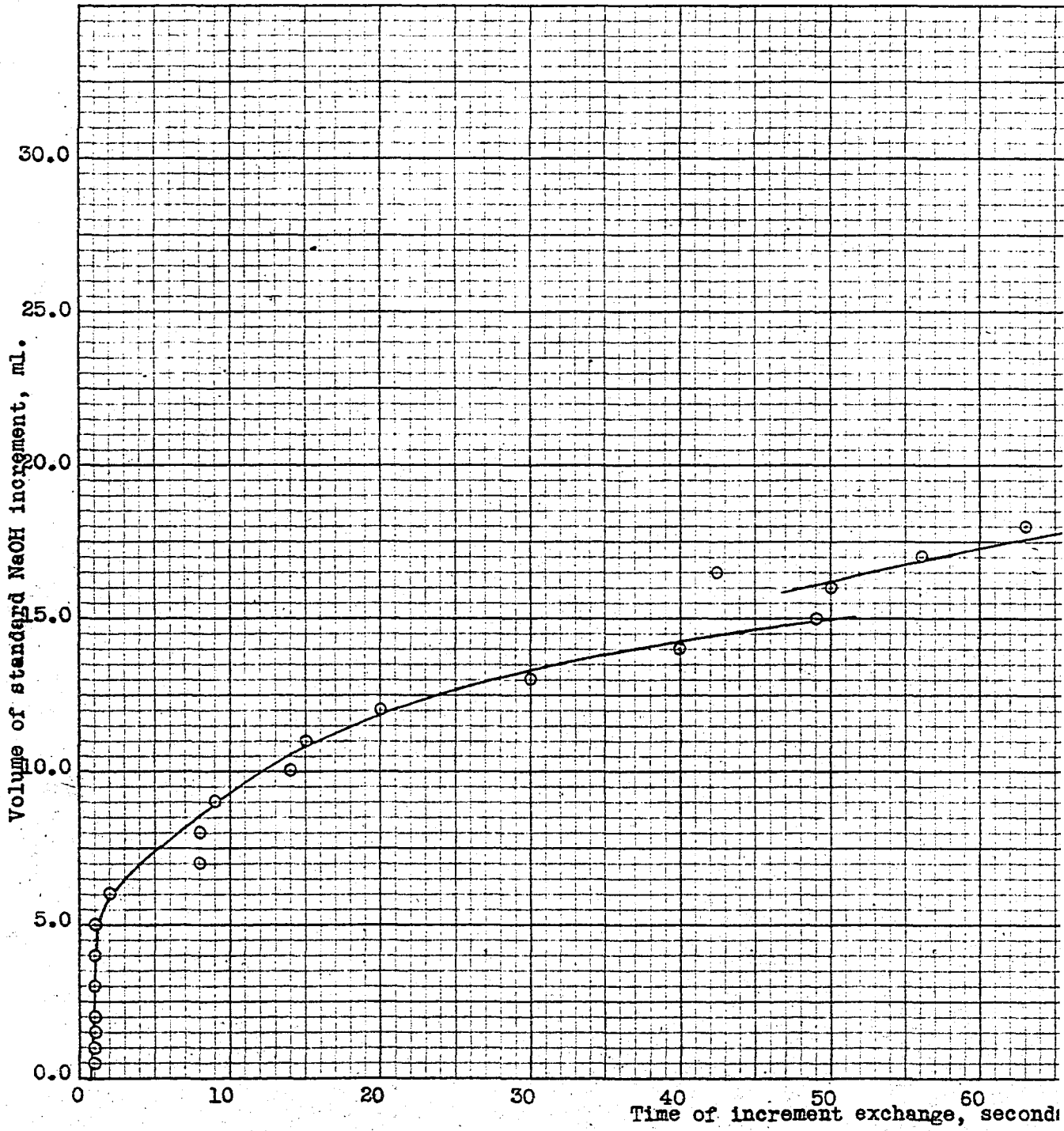
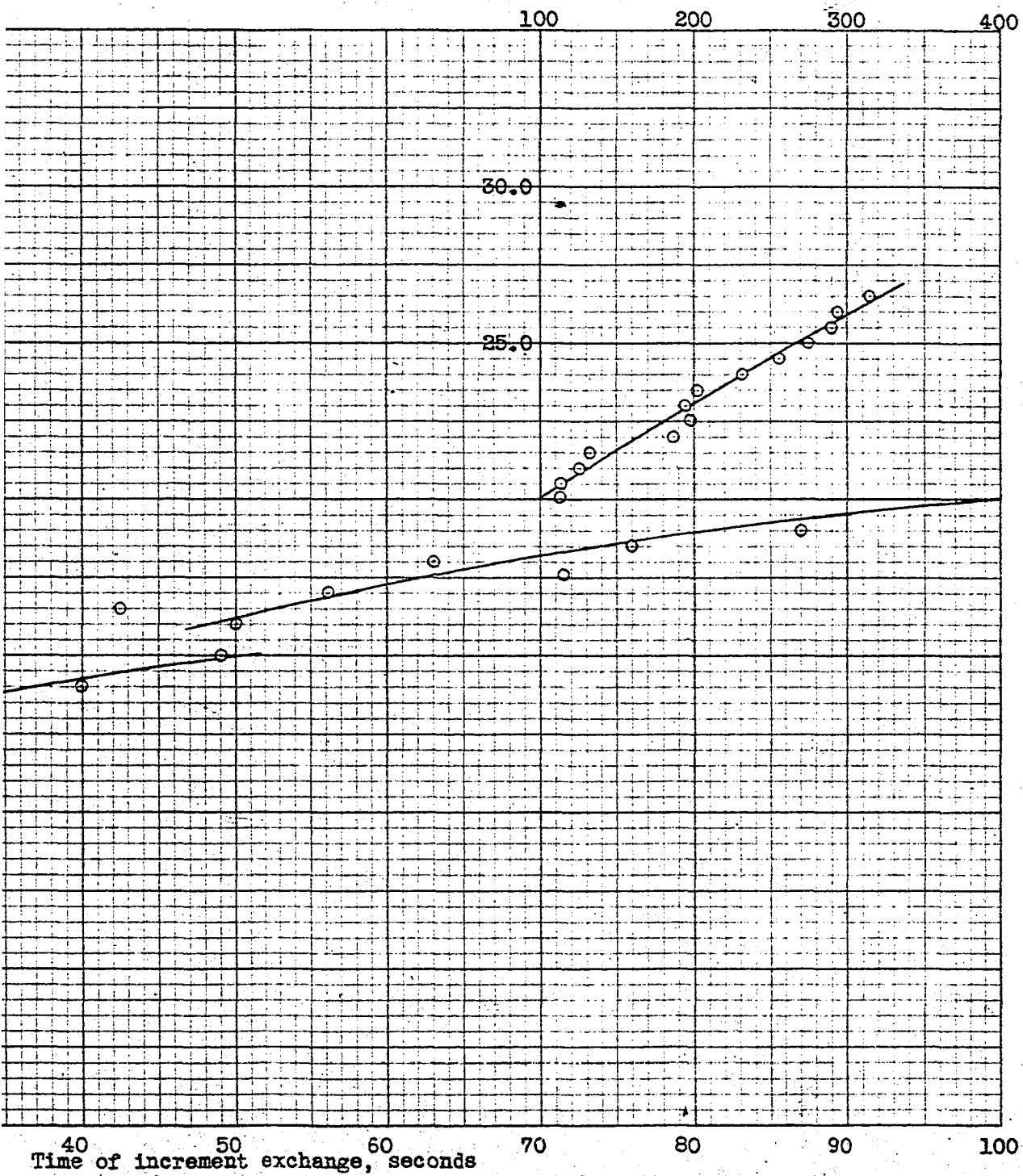


Fig. 20. Effect of incremental concentration in solution on



Experimental concentration in solution on rate of exchange of H-Zeo-Karb.

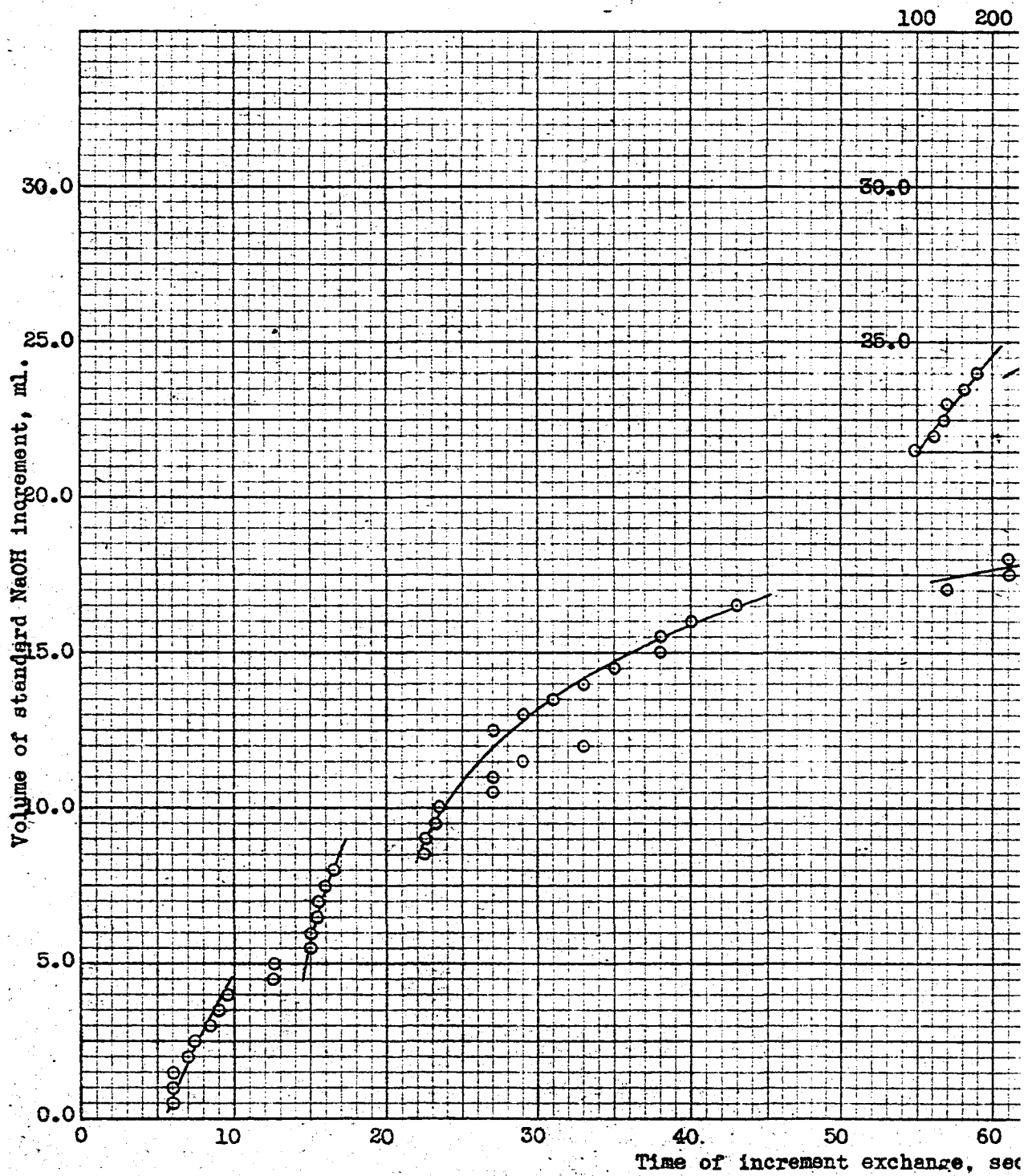
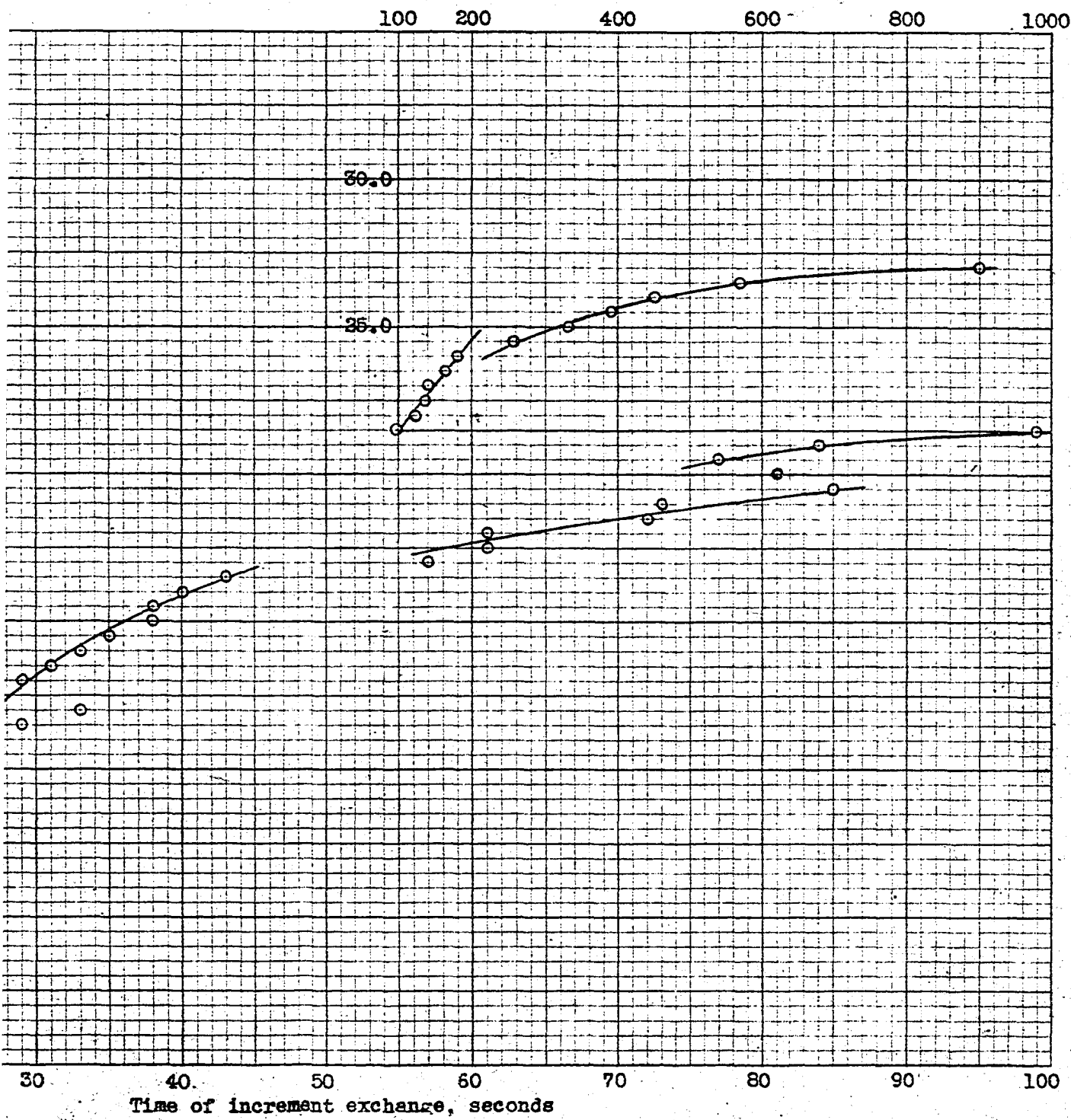


Fig. 21. Effect of agitation on rate of exchange



21. Effect of agitation on rate of exchange of H-Zeo-Karb.

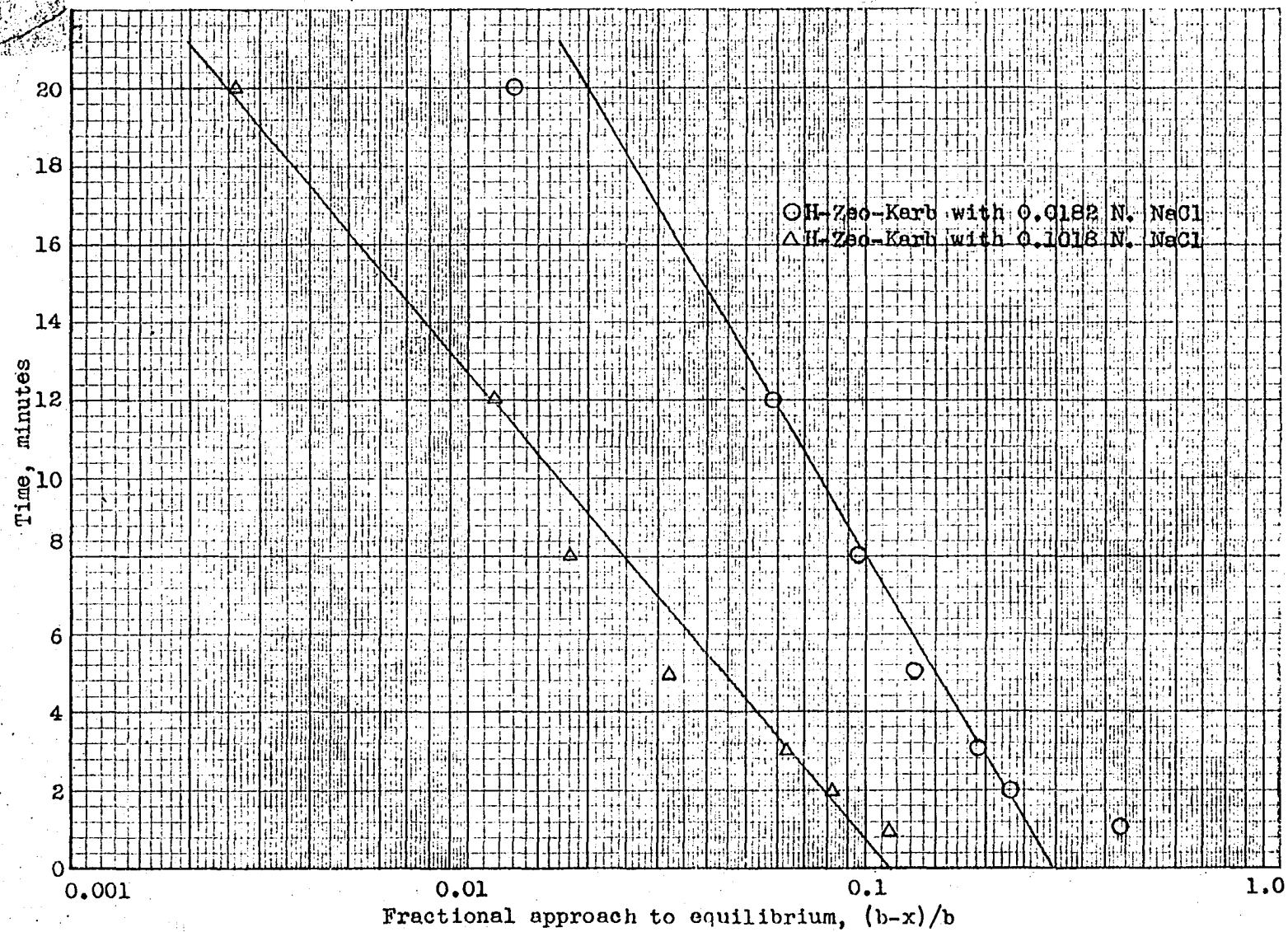
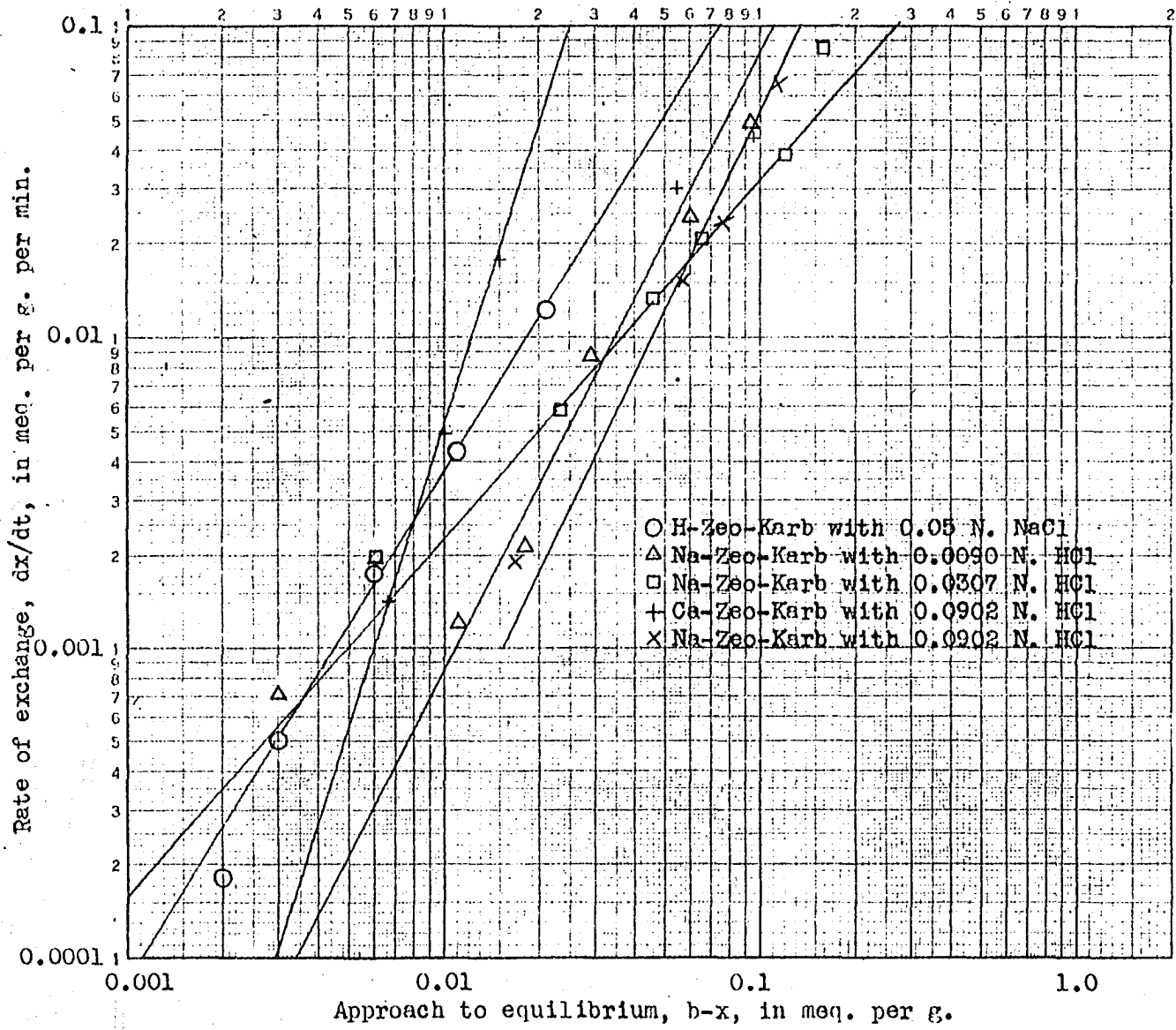


Fig. 22. Equilibrium correlations for rate of exchange of H-Zeo-Karb with sodium chloride solutions.



2.3

Fig. 23. Equilibrium correlations for rates of exchange of various Zeo-Karbs.

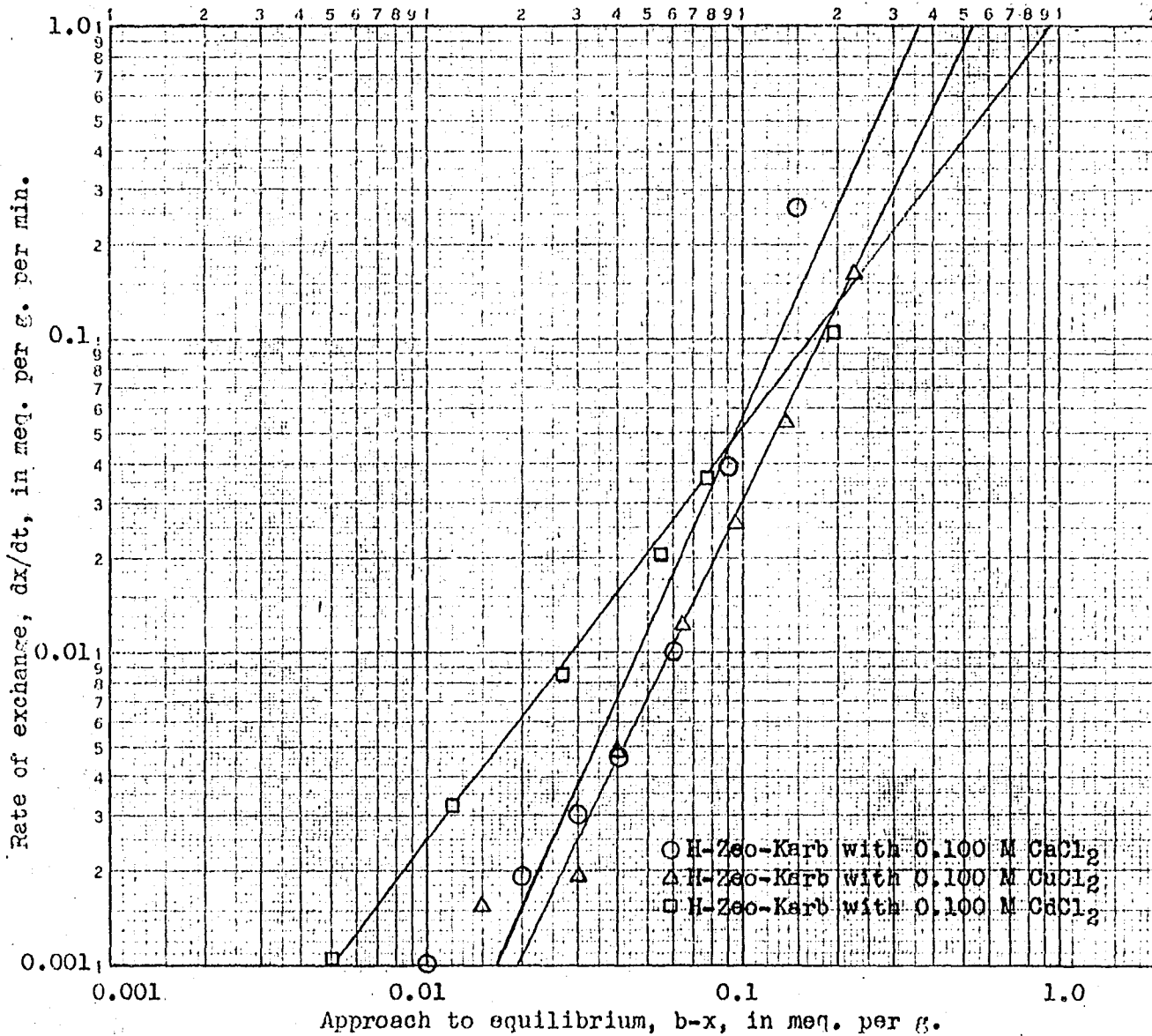


Fig. 24. Equilibrium correlations for rates of exchange of various Zeo-Karbs.

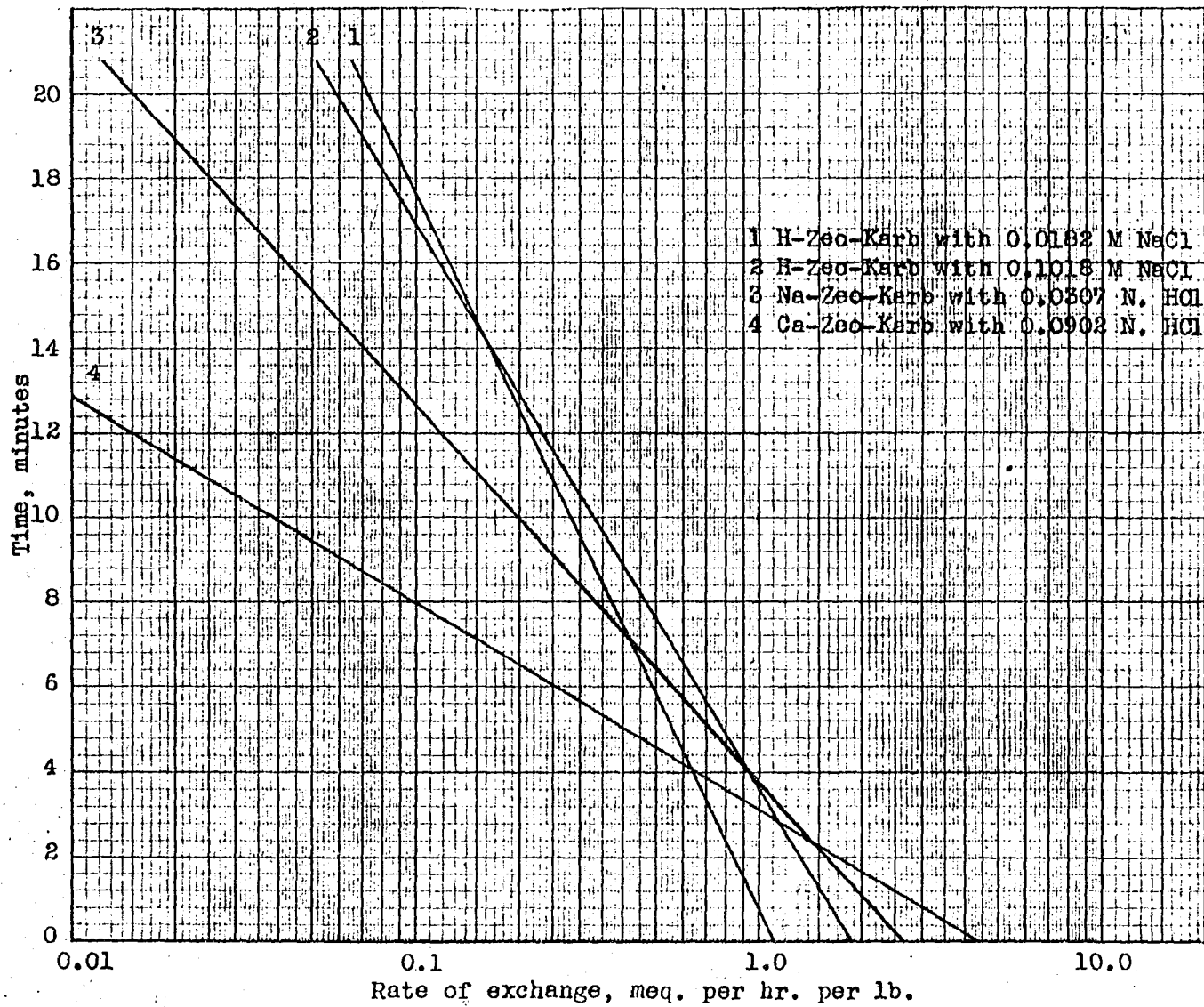


Fig. 25. Equilibrium correlations for rates of exchange of various Zeo-Karbs.

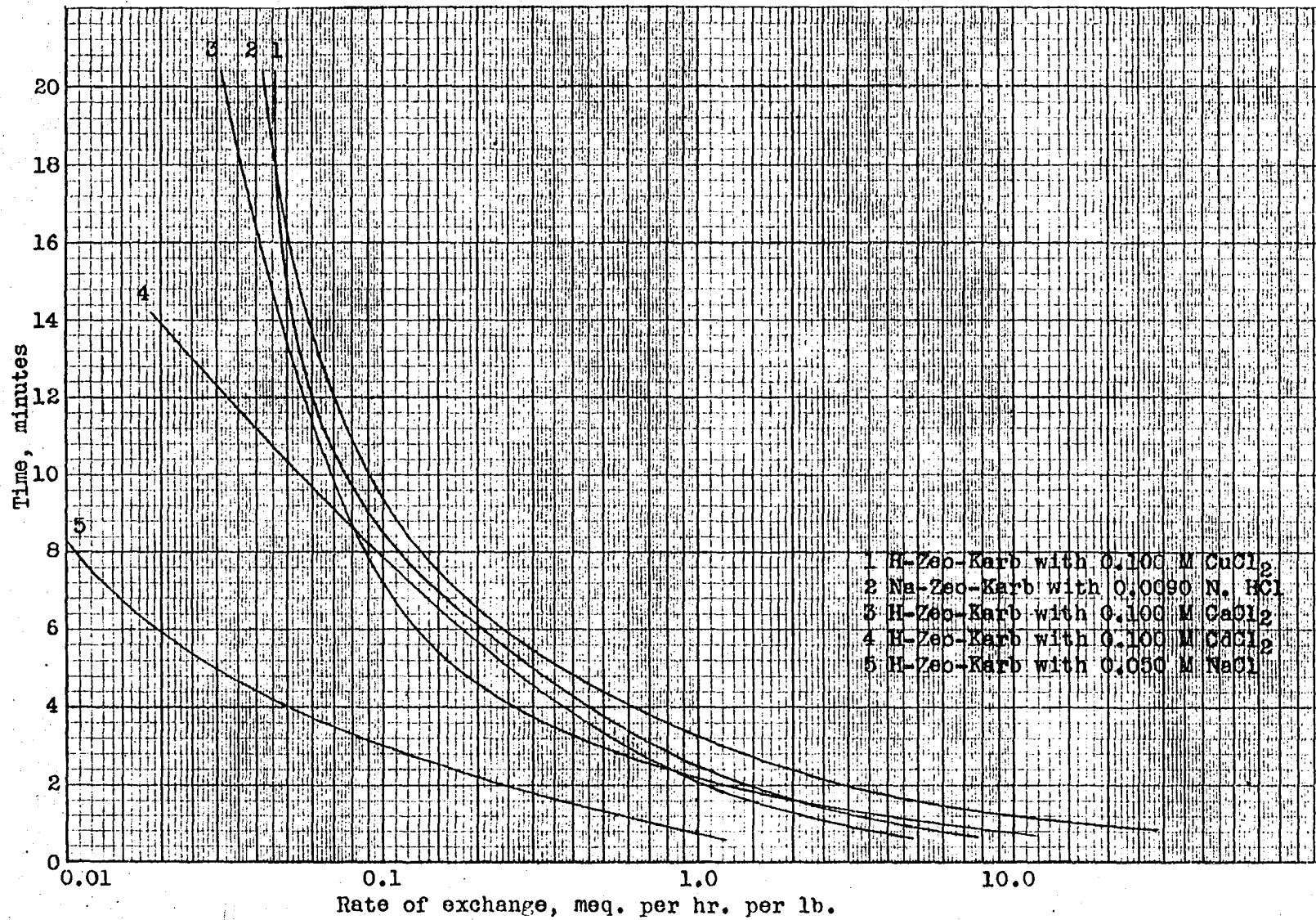


Fig. 26. Equilibrium correlations for rates of exchange of various Zeo-Karb.

C. Fluidized Zeolite Fines Bed

These studies were made to demonstrate the advantages of a fluidized bed for ion exchange operations. This technique of operation would seem to have particular utility for the finer or denser materials. Hence, the zeolite fines, which are normally waste material, were used in this investigation. Thus, a technique of operation and a utilization of a waste cation exchanger are combined.

1. Description of apparatus

The main part of the fluidization apparatus consisted of a standard two inch pyrex pipe, five feet in diameter. It was adequately supported in a vertical position by wall clamps. The details, as shown in Figure 27, are as follows: A conical bottom connection allowed proper entrance conditions for the water. A one-inch discharge line was provided at the top, which assured an adequate cross-sectional area with very low pressure drop. The conical bottom was connected to a tap water outlet by means of a flexible rubber hose. It should be noted that an open vent was provided at the top of the column for charging zeolite samples. Hence, the removal of pipe flanges was not required. An inclined manometer was attached by rubber hose to the upstream side of the apparatus, with the other end open to the atmosphere. A calibrated container and drain pipe were provided, but are not shown on Figure 27.

2. Preparation of materials

The zeolite fines used in this study were obtained from the Culligan Zeolite Company, and are representative of their fines. This material is too fine for normal bed operation, and, as a result, millions of pounds have been wasted.

A screen analysis was obtained by running a 200 gram sample for 45 minutes in a standard Ro-Tap machine. The data on this analysis are listed in Table 48 and shown graphically in Figure 28.

Table 48. Dry screen analysis of zeolite fines using Ro-Tap machine (Run 24)

Mesh No.	Weight material retained by corresponding mesh, g.	
	Differential per cent	Cumulative per cent
28	0.1	0.1
35	25.8	25.9
48	42.2	68.1
60	8.6	76.9
80	18.4	95.3
100	2.7	98.0
150	1.5	99.5
200	0.3	99.8
Pan	0.2	100.0

An important factor in any fluidized bed, particularly one desiring high rates of flow, is the density of the material. It was anticipated that the zeolite fines would permit a rather high rate of water flow through a fluidized bed, since they are denser than most cation exchangers. The bulk, dry, density of these fines is approximately 35

pounds per cu. ft., but the density of the actual particles is relatively high. A hydraulic classification of these zeolite fines was made by placing a sample in the fluidized bed, and increasing the rate of flow by increments. Each differential of flow rate caused a portion of the zeolite fines to be carried out of the bed. Each portion of fines was collected, filtered, dried and weighed. These data are given in Table 49 and presented graphically in Figure 29.

An examination of Figure 29 indicated that in practice it might be desirable to discard a portion of the finer material. Therefore, a large batch of zeolite fines was prepared for future use by screening through 50 mesh. The 68.6 per cent by weight portion retained on the 50 mesh screen was held for the future runs of this study, and the material through 50 mesh was discarded.

3. Operating procedure

For the runs in which the flow characteristics of the fluidized bed were observed, the exchanger was added in increments of 100 grams. It was added to the column through the top vent, utilizing a glass funnel. In a typical run measuring pressure drop and related data, the flow rate was set at a low valve initially. Readings were made and the flow rate increased slightly for another set of readings. A zero reading on the manometer was obtained at the beginning and end of each run.

In a typical softening run, the hard tap water standing on the bed was first displaced by distilled water. This was done for experimental purposes only, in order to observe rinse water requirements. A

regeneration step following, using a 4 gram NaCl per 100 ml. water solution. This regenerating solution was added slowly to the bed by gravity flow from a one gallon bottle supported above the apparatus. The excess salt solution was then washed from the bed. The run proper was then ready to begin. The flow rate was set at some constant, predetermined value. It was checked during the run by stopwatch and adjusted when necessary. The soft water effluent was collected in calibrated containers, with 250 ml. samples taken out of each liter of effluent. The run is terminated when the hardness of the effluent water approaches that of the tap water feed.

4. Experimental runs

Data on the rate of approach to equilibrium are given in Table 51 for this fluidized zeolite fines bed. Flow characteristics, including flow rates, bed heights, densities, and pressure drops, are presented in Tables 52 through 61. Then, the operations of rinsing, regenerating and softening are considered in Tables 62 through 71.

5. Analytical procedures

The only analysis of importance used in this study of a fluidized bed was that for hardness, utilizing a standard soap solution. The effluent from the bed was tested periodically throughout each run in order to follow the process of exhausting the bed capacity. The standardization of the soap solution, utilizing tap water, is shown in the following Table 50.

Table 50. Standardization of soap solution using tap water (Run 26)

Volume tap water, ml.	Volume distilled water, ml.	Volume st'd. soap soln., ml.	Hardness, grains per gal. (as CaCO ₃)
10	40	1.70	4.82
10	40	1.72	4.82
20	30	3.20	9.64
20	30	3.16	9.64
30	20	4.75	14.47
30	20	4.78	14.47
40	10	6.21	19.28

^aBased on hardness of 24.1 grains per gal. (as CaCO₃) for campus water, as determined with standard CaCl₂ solution versus st'd. soap solution.

6. Results and correlations

It can be seen from the hydraulic classification of Figure 29 that over 90 per cent of the zeolite fines, as received, were retained in the bed up to a velocity of 50 gal. per min. per sq. ft. of cross-sectional area. This flow rate is ten times the normal rate of flow utilized in softening by the usual fixed bed. However, this is not the entire story, as will be seen in results further along in this section.

The reproducibility of experimental data for pressure drops is illustrated in Figure 31. Also, it should be noted that the first run shown on this graph was made immediately after the dry zeolite sample was placed in the tower, whereas the zeolite for run 2 has soaked in water overnight. Therefore, it is evident that very little, if any, "water-logging" of zeolite occurs.

The data for pressure drops through the fluidized zeolite fines bed

are given in Figure 32. The regular spacing between curves indicates that the pressure drop is proportional to the weight of the bed. Approximately 0.9 inches water pressure drop per 100 grams of zeolite was observed. A portion of the pressure drop can probably be attributed to the increased bed density, and thus can be considered as part of the hydrostatic head.

The rinsing and regeneration operations that must be carried out in conjunction with exchanger beds are clearly illustrated in graph form on Figure 34. The initial period, up to almost one gallon of effluent, shows the regeneration salt solution displacing the distilled water that was standing on the bed. It is interesting to note that the peak concentration of the excess regeneration salt solution is within 3 per cent of the original concentration entering the column. The amount of rinse water cannot be taken directly from this figure, because it represents that required for a very thin bed. The curve, from the peak of the effluent to the point where there is negligible salt remaining, represents an "end effect" that depends upon the degree of mixing. Hence, the rinse water requirements for a column can be estimated by adding this end effect to the volume of solution standing on the bed at the end of a regeneration period.

The zeolite fines bed used in these studies was thoroughly exhausted by the large quantities of hard water that passed through it during the runs on flow characteristics. Therefore, the first three softening runs "conditioned" the bed. It can be seen on Figure 35 that a certain operating capacity has been approached in the third run of this series. As the bed is conditioned, the slope of the curve is greater, giving more ef-

ficient softening, and the break-through point is further to the right, indicating greater capacity. It might be mentioned at this point that the break-through point is surprisingly sharp for this type of operation. There was so much turbulence in the bed, that one would have predicted such a break-through point for very tall beds only. The softening capacities (determined by areas above curves and below line representing the 24.1 grains per gallon tap water) were 17,500, 19,250, and 21,800 grains per cuft., respectively, for these three conditioning runs.

Figure 36 shows the softening capacities of Runs 43, 45, and 47, all of which were maintained at the same flow rate. The capacities of these runs were 21,800, 22,750, and 17,550 grains per cu. ft., respectively. It is believed that the last value is lower than it should be.

Figure 37 illustrates that different flow rates, varying from 0.2 to 8.2 gal. per min. per sq. ft., had little effect upon the softening capacity. The highest rate curve does show a break-through point that is considerably lower than the others. These runs indicate that the flow rates were not high enough for the rate of cation exchange to become critical.

A correlation of bed density with various flow rates is given on Figure 38. The fact that most of the points were on or near the curve shows that bed height measurements under steady operation are a fair indication of the flow rate through the bed.

A further correlation was made to show the critical flow rate at which a given size of material would be carried from the bed. This is plotted on Figure 39.

The bed densities, flow rate, and volume of water softened in unit

time were used to establish an optimum rate of flow. The volume of water softened varies directly with the flow rate. Likewise, the volume of bed required for a given amount of exchanger varies with the rate. An optimum rate of flow of 13.0 gal. per min. per sq. ft. is shown on Figure 40.

7. Proposed industrial unit

It is believed that an industrial unit employing the fluidized bed technique and waste zeolite fines could be made economical and practical. It would have the economical features of a cheap exchanger and large through-put per unit volume of equipment. Two to three times the usual flow rates are possible. The bed operation can be made "fool-proof" by installing "exchanger collectors" or separators on all effluent lines. Also, the process could be made continuous, if desired, by having a similar fluidized bed column operating on the regeneration cycle.

As an example, two 3 ft. x 20 ft. towers, one softening and one regenerating, utilizing 30 to 50 mesh zeolite fines having 18,000 grains per cu. ft. capacity, could soften 905 gpm of 24.1 grain water. The zeolite circulation rate would be 1.21 cu. ft. per minute, based on a "static" density of 31.8 lbs. per cu. ft. The residence time of the zeolite in the bed would be 45.7 minutes. The height of the static bed during shut-downs would be 7.86 feet.

In addition to the advantages previously mentioned, the use of zeolite fines gives higher capacity per cubic foot of exchanger. On the other

hand, the abrasion of the zeolite might prove to be a disadvantage. It should be checked in a pilot unit over an extended period of time. Likewise, careful consideration should be given to the design of the unit, in order to keep it simple for operation and yet not using an excessive amount of auxiliary equipment.

Table 49. Hydraulic classification of zeolite fines (Run 25)

Test No.	Interval time, min.	Water flow rate ^a			Material carried out of column		
		Observed, ht., in.	Calculated, gal.	gal./min./ft. ²	Weight, g.	Differential per cent	Cumulative per cent
1	21	5.38	1.34	2.9	0.5	0.67	0.67
2	47	43.07	10.68	10.4	1.0	1.34	2.01
3	29	72.20	17.91	28.3	25.3	33.92	35.93
4	22	97.38	24.19	50.3	41.0	54.95	90.88
5	14	104.31	25.91	84.4	6.8	9.12	100.00

^aTap water used throughout run.

Table 51. Rate of approach to steady flow rate
in fluidized zeolite bed (Run 27)

Test No.	Interval time, min.	Water flow rate			Height of bed	
		Observed, ml./min. ^a	Calculated, gal./min. ^a gal./min./ft. ²		Observed, in.	Calc., ^b in.
1	0	---	---	---	18.8	23.0
	5	---	---	---	18.6	22.8
	5	90.2	0.0238	1.09	18.6	22.8
	5	---	---	---	18.6	22.8
	10	---	---	---	18.5	22.7
	10	85.3	0.0225	1.03	18.5	22.7
	8	---	---	---	18.4	22.6
	5	---	---	---	18.2	22.4
	15	---	---	---	18.2	22.4
2	0	---	---	---	20.1	24.3
	2	---	---	---	20.0	24.2
	5	---	---	---	19.9	24.1
	12	131.5	0.0347	1.59	19.6	23.8
	8	---	---	---	19.4	23.6
	8	114.2	0.0302	1.38	19.4	23.6
	15	116.8	0.0308	1.41	19.4	23.6
3	0	---	---	---	21.9	26.1
	1	---	---	---	21.9	26.1
	1	---	---	---	21.8	26.0
	6	---	---	---	21.4	25.6
	3	196.1	0.0508	2.32	21.0	25.2
	7	---	---	---	20.5	24.7
	10	161.6	0.0427	1.95	20.5	24.7
	5	---	---	---	20.1	24.3
	10	150.0	0.0396	1.81	20.1	24.3
4	0	---	---	---	24.2	28.4
	1	---	---	---	24.1	28.3
	3	---	---	---	23.8	28.0
	14	283.3	0.0748	3.42	23.2	27.4
	10	---	---	---	22.8	27.0
	5	266.6	0.0705	3.22	22.7	26.9
	16	237.0	0.0626	2.86	21.8	26.0
	6	---	---	---	21.5	25.7

Table 51. (Continued)

Test No.	Interval time, min.	Water flow rate			Height of bed	
		Observed, ml./min. ^a	Calculated, gal./min. ^a gal./min./ft. ²		Observed, in.	Calc., ^b in.
5	0	---	---	---	30.9	35.1
	3	---	---	---	30.9	35.1
	5	---	---	---	30.6	34.8
	9	452.0	0.1192	5.45	30.4	34.6
6	0	---	---	---	31.5	35.7
	6	---	---	---	31.5	35.7
	3	---	---	---	31.4	35.6
	4	495.0	0.1308	5.98	31.3	35.5
	8	---	---	---	31.0	35.2
	7	---	---	---	31.5	35.7
	2	---	---	---	31.4	35.6
	2	514.0	0.1357	6.20	31.4	35.6
	7	---	---	---	30.7	34.9
	5	---	---	---	31.1	35.3
	1	492.0	0.1300	5.95	31.1	35.3
	5	---	---	---	31.1	35.3
	3	---	---	---	31.0	35.2
	2	490.0	0.1293	5.92	31.0	35.2
	3	---	---	---	30.5	34.7
4	---	---	---	30.3	34.5	
7	0	---	---	---	33.4	37.6
	3	---	---	---	33.2	37.4
	2	523.3	0.1382	6.33	33.1	37.5
	3	---	---	---	32.9	37.1
	5	---	---	---	32.8	37.0
	3	513.3	0.1358	6.21	32.5	36.7
	3	---	---	---	32.5	36.7
	4	510.0	0.1349	6.17	32.5	36.7
	3	---	---	---	32.5	36.7
	3	508.3	0.1343	6.15	32.3	36.5
	3	---	---	---	32.2	36.4

Table 51. (Continued)

Test No.	Interval time, min.	Water flow rate			Height of bed	
		Observed, ml./min. ^a	Calculated, gal./min. ^a gal./min./ft. ²		Observed, in.	Calc., ^b in.
8	0	---	---	---	42.3	46.4
	3	---	---	---	42.1	46.2
	3	---	---	---	42.0	46.1
	3	---	---	---	41.9	46.0
	2	753.5	0.1990	9.10	41.8	45.9
	3	---	---	---	41.8	45.9
	3	---	---	---	41.8	45.9
	2	753.5	0.1990	9.10	41.7	45.8
	8	---	---	---	41.4	45.5
9	0	---	---	---	53.5	57.6
	1	---	---	---	53.4	57.5
	2	---	---	---	53.0	57.1
	2	---	---	---	52.6	56.7
	1	---	---	---	52.5	56.6
	1	983.3	0.2600	11.90	52.3	56.4
	5	---	---	---	52.1	56.2
	2	---	---	---	52.1	56.2
	2	976.7	0.2382	11.81	52.4	56.5
	4	---	---	---	52.6	56.7
	4	---	---	---	52.7	56.8
	3	---	---	---	52.9	57.0
	1	983.3	0.2600	11.90	52.9	57.0
5	---	---	---	52.9	57.0	

^aVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^bIncludes an equivalent height for zeolite fines in cone at bottom of column.

Table 52. Flow characteristics for 300 grams zeolite fines in fluidized bed (Run 28)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated		Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	ΔH ,	
		gal./min.	gal./min./ft. ²			in. H ₂ O		in. H ₂ O	
1	93.3	0.025	1.13	7.3	11.5	31.6	37.0	5.34	3.72
2	119.2	0.031	1.44	7.5	11.7	31.0	37.5	5.42	3.80
3	203.2	0.054	2.45	8.3	12.5	29.0	36.2	5.23	3.61
4	381.2	0.101	4.62	10.5	14.7	24.7	35.5	5.13	3.51
5	707.6	0.187	8.57	17.0	21.1	17.2	35.5	5.13	3.51
6	1003.0	0.265	12.16	23.6	27.6	13.1	35.3	5.10	3.48
7	1143.0	0.302	13.85	32.2	36.1	10.0	35.4	5.11	3.49
8	1563.0	0.413	18.93	47.0	50.7	7.2	35.2	5.08	3.46
9	0.0	0.000	0.00	7.3	11.5	31.6	11.2	1.62	0.00

^aThe zeolite fines were -30 + 50 mesh, and were soaked in water at least 12 hours before run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

Table 53. Flow characteristics for 400 grams zeolite fines in fluidized bed (Run 29)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated gal./min	gal./min./ft. ²	Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	in. H ₂ O	Δ H, in. H ₂ O
1	112.4	0.0298	1.36	10.9	15.1	32.0	13.3	5.92	4.70
2	195.8	0.052	2.37	12.1	16.3	29.7	12.8	5.70	4.48
3	322.8	0.085	3.91	14.1	18.3	26.4	12.6	5.61	4.39
4	438.5	0.116	5.33	16.7	20.9	23.2	12.6	5.61	4.39
5	611.8	0.162	7.41	21.2	25.3	19.1	12.68	5.64	4.42
6	768.0	0.203	9.32	24.4	28.5	17.0	12.6	5.61	4.39
7	1018.0	0.269	12.32	32.2	36.2	13.4	12.63	5.62	4.40
8	1257.5	0.332	15.21	40.5	44.4	10.9	12.63	5.62	4.40
9	1480.0	0.391	17.88	58.0	61.8	7.8	12.65	5.63	4.41
10	0.0	0.000	0.0	11.0	15.2	31.8	2.75	1.22	0.00

^aThe zeolite fines were -30 + 50 mesh, and were soaked in water at least 18 hours before run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.4968, with respect to horizontal line.

Table 54. Flow characteristics for 500 grams zeolite fines in fluidized bed (Run 30)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated gal./min.	gal./min./ft. ²	Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	in. H ₂ O	ΔH, in. H ₂ O
1	118.3	0.032	1.43	14.5	18.7	32.4	15.23	6.78	5.60
2	173.1	0.046	2.10	16.2	20.4	29.7	15.05	6.70	5.52
3	268.4	0.071	3.25	17.7	21.9	27.7	14.7	6.55	5.37
4	348.0	0.092	4.21	19.8	24.0	25.3	14.6	6.50	5.32
5	502.6	0.133	6.08	24.1	28.3	21.4	14.8	6.58	5.40
6	715.6	0.189	8.67	30.5	34.6	17.5	14.75	6.57	5.39
7	984.0	0.260	11.92	40.5	44.5	13.6	14.70	6.55	5.37
8	1241.0	0.328	15.03	52.5	56.4	10.8	14.65	6.52	5.34
9	0.0	0.000	0.00	14.4	18.6	32.6	2.65	1.18	0.00

^aThe zeolite fines were -30 + 50 mesh, and were soaked in water at least 18 hours before run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.4968, with respect to horizontal line.

Table 55. Flow characteristics for 600 grams zeolite fines in fluidized bed (Run 31)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated		Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	in. H ₂ O	ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²						
1	100.0	0.026	1.21	18.8	23.0	31.6	17.2	7.66	6.58
2	146.3	0.039	1.77	20.0	24.2	30.0	17.35	7.72	6.64
3	273.7	0.072	3.31	23.1	27.3	26.6	16.8	7.48	6.40
4	416.4	0.110	5.04	27.4	31.6	23.0	16.85	7.50	6.42
5	586.4	0.155	7.09	34.0	38.1	19.6	16.8	7.48	6.40
6	748.8	0.198	9.07	39.8	43.9	16.6	16.75	7.46	6.38
7	904.0	0.239	10.92	46.0	50.0	14.5	16.65	7.42	6.34
8	1014.0	0.268	12.28	52.0	56.0	13.0	16.65	7.42	6.34
9	0.0	0.000	0.00	19.3	23.5	30.9	2.43	1.08	0.00

^aThe zeolite fines were -30 + 50 mesh, and were not allowed to soak in water prior to run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.4968, with respect to horizontal line.

Table 56. Flow characteristics for 600 grams zeolite fines in fluidized bed (Run 32)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated		Observed, in.	Calc. ^c , in.		Mano. rdg. ^e		ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²			obser.	in. H ₂ O		
1	0.0	0.0000	0.00	18.4	22.6	32.1	2.48	1.10	0.00
2	51.0	0.0135	0.62	18.4	22.6	32.1	13.10	5.83	4.73
3	25.8	0.0068	0.31	18.4	22.6	32.1	9.33	4.15	3.05
4	9.2	0.0024	0.11	18.4	22.6	32.1	6.09	2.71	1.61

^aThe zeolite fines were -30+50 mesh, and were soaked in water at least 12 hours before run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.4968, with respect to horizontal line.

Table 57. Flow characteristics for 200 grams zeolite fines in fluidized bed (Run 33)^a

Test No.	Water flow rate			Pressure drop		
	Observed, in./min. ^b	Calculated		Meno. rdg. ^{c, d}		ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²	obser.	in. H ₂ O	
1	15.85	0.00419	0.19	27.0	3.90	2.39
2	15.33	0.00405	0.19	25.5	3.68	2.17
3	15.74	0.00416	0.19	27.0	3.90	2.39
4	16.28	0.00430	0.20	26.5	4.12	2.61
5	16.52	0.00436	0.20	29.0	4.19	2.68
6	16.51	0.00436	0.20	27.4	3.96	2.45
7	16.51	0.00436	0.20	27.4	3.96	2.45
8	16.48	0.00435	0.20	27.5	3.97	2.46
9	16.48	0.00435	0.20	27.3	3.96	2.45
10	16.50	0.00436	0.20	27.5	3.97	2.46

^aThe zeolite fines were -30+50 mesh, and were soaked in water at least 12 hours before run.

^bVolume of water measured in a graduated cylinder for a convenient period of time by use of stop-watch.

^cManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

^dZero manometer reading assumed 10.44, equivalent to 1.51 inches water.

Table 58. Flow characteristics for water in two inch I.D. column (Run 34)^a

Test No.	Water flow rate			Pressure drop		
	Observed, in./min. ^b	Calculated		Mano. rdg. ^c		ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²	obser.	in. H ₂ O	
1	11.97	2.97	136.0	39.1	5.64	4.13
2	11.40	2.83	129.6	36.9	5.33	3.82
3	10.85	2.69	123.3	34.7	5.02	3.51
4	10.27	2.55	116.8	32.4	4.68	3.17
5	9.70	2.41	110.3	30.6	4.42	2.91
6	8.545	2.07	94.8	27.9	4.03	2.52
7	7.865	1.88	86.0	25.8	3.73	2.22
8	6.875	1.70	78.2	24.2	3.50	1.99
9	6.365	1.58	72.4	22.8	3.29	1.78
10	5.70	1.41	64.8	21.37	3.09	1.58
11	5.393	1.34	61.4	20.97	3.03	1.52
12	5.167	1.28	58.8	20.45	2.95	1.44
13	4.933	1.22	56.0	19.90	2.87	1.36
14	4.527	1.12	51.4	19.27	2.78	1.27
15	4.293	1.07	48.8	18.83	2.72	1.21
16	3.892	0.966	44.20	18.17	2.62	1.11
17	3.780	0.938	43.00	17.83	2.58	1.07
18	3.490	0.867	39.72	17.47	2.52	1.01
19	3.200	0.794	36.39	16.98	2.45	0.94
20	2.940	0.730	33.46	16.63	2.40	0.89

Table 58. (Continued)

Test No.	Water flow rate			Pressure drop		
	Observed, in./min. ^b	Calculated		Mano. rdg. ^c		ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²	obser.	in. H ₂ O	
21	2.740	0.680	31.15	16.29	2.35	0.84
22	2.530	0.628	28.80	15.92	2.30	0.79
23	2.283	0.567	25.98	15.61	2.24	0.73
24	2.154	0.534	24.48	15.39	2.22	0.71
25	1.925	0.479	21.87	15.04	2.17	0.66
26	1.671	0.415	19.02	14.72	2.13	0.62
27	1.397	0.347	15.88	14.32	2.07	0.56
28	1.367	0.339	15.52	13.75	1.98	0.55
29	1.121	0.278	12.75	13.53	1.95	0.52
30	0.941	0.233	10.69	13.28	1.92	0.49
31	0.789	0.196	8.97	13.01	1.88	0.45
32	0.706	0.175	8.03	12.83	1.85	0.42
33	0.510	0.127	5.80	12.58	1.82	0.39
34	0.198	0.049	2.25	11.78	1.70	0.37
35	0.0	0.000	0.00	10.44	1.51	0.00

^aThe zeolite fines were -30+50 mesh, and were soaked in water at least 12 hours before run.

^bHeight of water in inches was measured in a calibrated container for a convenient period of time.

^cManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

Table 59. Flow characteristics for 100 grams zeolite fines in fluidized bed (Run 35)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated		Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	in. H ₂ O	Δ H, in. H ₂ O
		gal./min.	gal./min./ft. ²						
1	0.624	0.155	7.09	3.4	7.5	16.1	23.10	3.34	1.75
2	1.322	0.328	15.03	9.4	13.3	9.1	22.50	3.25	1.66
3	1.680	0.417	19.13	18.0	21.7	5.6	22.65	3.27	1.68
4	1.989	0.493	22.59	36.0	39.4	3.1	22.90	3.31	1.72
5	2.096	0.521	23.85	45.0	48.4	2.5	22.90	3.31	1.72
6	0.392	0.097	4.46	1.4	5.6	21.6	24.10	3.48	1.89
7	0.000	0.000	0.00	--	--	--	10.79	1.59	0.00

^aThe zeolite fines were -30+50 mesh, and were not allowed to soak in water prior to run.

^bHeight of water in inches was measured in a calibrated container for a convenient period of time.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

Table 60. Flow characteristics for 100 grams zeolite fines in fluidized bed (Run 36)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated		Observed, in.	Calc. ^c , in.		Mano. rdg. ^e		ΔH , in. H ₂ O
		gal./min.	gal./min./ft. ²			obser.	in. H ₂ O		
1	0.398	0.099	4.53	1.3	5.5	22.0	23.8	3.44	1.89
2	1.168	0.290	13.29	6.7	10.7	11.3	22.4	3.24	1.69
3	1.593	0.395	18.11	13.1	16.9	7.2	22.3	3.22	1.67
4	1.900	0.472	21.61	23.5	27.0	4.5	22.65	3.27	1.72
5	2.214	0.549	25.15	48.0	51.3	2.4	22.7	3.28	1.73
6	0.000	0.000	0.00	--	--	--	10.75	1.55	0.00

^aThe zeolite fines were -30+50 mesh, and were soaked in water at least 12 hours before run.

^bHeight of water in inches was measured in a calibrated container for a convenient period of time.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

Table 61. Flow characteristics for 200 grams zeolite fines in fluidized bed (Run 37)^a

Test No.	Water flow rate			Height of bed		Density ^d of bed, lb./ft. ³	Pressure drop		
	Observed, in./min. ^b	Calculated gal./min.	gal./min./ft. ²	Observed, in.	Calc. ^c , in.		Mano. rdg. ^e obser.	in. H ₂ O	4H, in. H ₂ O
1	0.103	0.024	1.12	3.5	7.7	31.4	31.5	4.55	3.05
2	0.273	0.068	3.10	4.4	8.6	28.2	30.0	4.33	2.83
3	0.567	0.141	6.44	7.9	12.1	20.0	29.0	4.19	2.69
4	1.244	0.309	14.17	17.6	21.5	11.2	28.3	4.08	2.58
5	1.525	0.379	17.33	25.5	29.3	8.3	28.3	4.08	2.58
6	1.842	0.457	20.95	40.0	43.6	5.6	28.55	4.13	2.63
7	1.883	0.467	21.40	43.0	47.6	5.1	28.5	4.12	2.62
8	0.000	0.000	0.00	3.1	7.3	33.2	10.39	1.50	0.00

^aThe zeolite fines were -30+50 mesh, and were soaked in water at least 12 hours before run.

^bHeight of water in inches was measured in a calibrated container for a convenient period of time.

^cIncludes an equivalent height for zeolite fines in cone at bottom of column.

^dBased on calculated height of bed.

^eManometer inclined to form angle having tangent of 0.1459, with respect to horizontal line.

Table 62. Rinsing of hard water from fluidized zeolite bed (Run 38)

Test No.	Wash water volume ^a		Effluent ^b volume, gal.	Effluent hardness ^c	
	Observed, liters	Calc., gal.		Soap, ml.	Gr. per gal.
1	0.50	0.13	0.00	--	--
2	1.00	0.26	0.00	--	--
3	1.50	0.40	0.00	--	--
4	2.00	0.53	0.00	--	--
5	2.50	0.66	0.13	4.62	14.1
6	3.00	0.79	0.26	3.68	11.1
7	3.50	0.92	0.40	2.61	7.7
8	4.00	1.06	0.53	2.04	5.9
9	4.50	1.19	0.66	1.41	3.9
10	5.00	1.32	0.79	1.32	3.6
11	5.50	1.45	0.92	0.87	2.2
12	6.00	1.58	1.06	1.00	2.6
13	6.50	1.72	1.19	1.15	3.0
14	7.00	1.85	1.32	0.90	2.3
15	7.50	1.98	1.45	0.78	1.9
16	8.00	2.11	1.58	0.64	1.5
17	8.50	2.24	1.72	0.61	1.4
18	9.00	2.38	1.85	0.63	1.5
19	9.50	2.51	1.98	0.61	1.4
20	10.00	2.64	2.11	0.59	1.3
21	10.50	2.78	2.24	0.54	1.2
22	11.00	2.90	2.38	0.51	1.1

^aDistilled water used as wash.

^bZero effluent (initially) due to starting with drained bed.

^cZeolite bed had been in contact with campus water containing 24.1 grains hardness per gal. (as CaCO₃).

Table 65. Rinsing of hard water from fluidized zeolite bed (Run 39)

Test No.	Wash water volume ^a		Effluent volume, gal.	Effluent hardness ^b	
	Observed, liters	Calc., gal.		Soap, ml.	Gr. per gal.
1	0.50	0.13	0.13	6.64	20.4
2	1.00	0.26	0.26	6.20	19.0
3	1.50	0.40	0.40	6.04	18.5
4	2.00	0.53	0.53	5.56	17.0
5	2.50	0.66	0.66	4.62	14.1
6	3.00	0.79	0.79	3.74	11.3
7	3.50	0.92	0.92	2.35	6.9
8	4.00	1.06	1.06	1.74	4.8
9	4.50	1.19	1.19	1.57	4.4
10	5.00	1.32	1.32	1.06	2.8
11	5.50	1.45	1.45	0.84	2.1
12	6.00	1.58	1.58	0.61	1.4
13	6.50	1.72	1.72	0.50	1.0
14	7.00	1.85	1.85	0.42	0.8
15	7.50	1.98	1.98	0.42	0.8
16	8.00	2.11	2.11	0.41	0.8
17	8.50	2.24	2.24	0.40	0.7
18	9.00	2.38	2.38	0.31	0.4
19	9.50	2.51	2.51	0.30	0.4
20	10.00	2.64	2.64	0.26	0.3
21	10.50	2.78	2.78	0.28	0.3
22	11.00	2.90	2.90	0.27	0.3
23	11.50	3.04	3.04	0.26	0.3
24	12.00	3.17	3.17	0.23	0.2
25	12.50	3.30	3.30	0.22	0.2
26	13.00	3.44	3.44	0.23	0.2
27	13.50	3.57	3.57	0.21	0.1
28	14.00	3.70	3.70	0.19	0.1
29	14.50	3.83	3.83	0.19	0.1
30	15.00	3.96	3.96	0.17	0.0

^aDistilled water used as wash.

^bZeolite bed had been in contact with campus water containing 24.1 grains hardness per gal. (as CaCO₃).

Table 64. Regeneration and rinsing of fluidized zeolite bed (Run 40)

Test No.	Effluent volume		Flow rate		Salts in effluent ^b		
	Observed, liters	Calc., gal.	Observed ml./min.	Calc., gal./min.	St'd AgNO ₃ ml.	Chloride, g. per meq.	As NaCl ^c , 100 g. H ₂ O
1	0.50	0.13	---	---	0.30	0.06	0.0007
2	1.00	0.26	83.0	0.022	0.30	0.06	0.0007
3	1.50	0.40	56.0	0.015	0.25	0.05	0.0006
4	2.00	0.53	33.0	0.009	0.27	0.06	0.0007
5	2.50	0.66	56.0	0.015	0.50	0.10	0.0012
6	3.00	0.79	71.0	0.019	0.70	0.14	0.0016
7	3.50	0.92	---	---	14.30	28.9	0.538
8	4.00	1.06	250.0	0.066	12.75	257.5	3.015
9	4.50	1.19	---	---	16.40	331.5	3.881
10	5.00	1.32	125.0	0.033	15.50	313.0	3.665
11	5.50	1.45	---	---	13.40	270.7	3.167
12	6.00	1.58	---	---	11.18	225.5	2.641
13	6.50	1.72	167.0	0.044	9.47	191.2	2.235
14	7.00	1.85	---	---	7.90	159.7	1.870
15	7.60	2.00	122.0	0.032	6.08	122.9	1.438
16	8.20	2.16	---	---	4.23	85.5	1.000
17	8.80	2.32	---	---	2.84	57.4	0.672
18	9.40	2.48	---	---	2.07	41.8	0.490
19	9.90	2.61	---	---	1.50	30.3	0.355
20	10.40	2.74	---	---	1.18	23.8	0.279
21	10.90	2.88	100.0	0.026	8.00	16.2	0.190
22	11.40	3.01	125.0	0.033	5.80	11.7	0.137
23	11.90	3.14	100.0	0.026	4.19	8.47	0.099
24	12.40	3.28	100.0	0.026	3.27	6.61	0.077
25	12.90	3.41	83.0	0.022	2.40	4.85	0.057
26	13.40	3.54	71.0	0.019	1.83	3.70	0.0433
27	13.90	3.67	71.0	0.019	1.25	2.53	0.0296
28	14.40	3.80	---	---	1.10	2.22	0.0260
29	14.90	3.93	100.0	0.026	0.85	1.72	0.0201
30	15.40	4.07	166.0	0.044	6.20	1.25	0.0146

^aFirst 3500 ml. of column feed was salt solution (4 grams NaCl per 100 ml.) for regeneration, followed by distilled water wash.

^bInitial effluent low in solids, because it consists chiefly of water from previous wash.

^cExpressed as NaCl, but actually consists of a mixture of NaCl, MgCl₂ and CaCl₂.

Table 65. First run for conditioning of fluidized zeolite bed (Run 41)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	0.50	0.132	90.0	167.0	0.37	0.6
2	1.00	0.264	60.0	250.0	0.33	0.5
3	1.75	0.462	60.0	250.0	0.36	0.6
4	2.25	0.595	60.0	250.0	0.35	0.6
5	2.75	0.727	30.0	500.0	0.22	0.15
6	3.25	0.859	35.5	422.0	0.35	0.6
7	3.75	0.991	32.0	469.0	0.35	0.6
8	4.25	1.125	39.6	379.0	0.32	0.5
9	4.75	1.254	37.0	406.0	0.41	0.75
10	5.25	1.390	37.0	406.0	0.44	0.9
11	5.75	1.519	--	---	0.34	0.5
12	6.25	1.655	--	---	0.40	0.7
13	6.75	1.783	--	---	0.40	0.7
14	7.25	1.92	36.0	417.0	0.37	0.6
15	7.75	2.15	36.5	411.0	0.39	0.7
16	8.25	2.18	--	---	0.36	0.6
17	8.75	2.31	36.5	411.0	0.38	0.7
18	9.25	2.45	--	---	0.41	0.75
19	9.75	2.58	37.0	406.0	0.34	0.5
20	10.25	2.71	--	---	0.33	0.5
21	10.75	2.84	37.3	402.0	0.33	0.5
22	11.25	2.98	--	---	0.34	0.5
23	11.75	3.10	37.6	399.0	0.35	0.6
24	12.25	3.24	--	---	0.35	0.6
25	12.75	3.37	38.6	389.0	0.33	0.5
26	13.25	3.50	--	---	0.35	0.6
27	13.75	3.64	--	---	0.37	0.6
28	14.25	3.77	--	---	0.38	0.7
29	15.25	4.03	--	---	0.36	0.6
30	16.25	4.30	38.2	393.0	0.43	0.85

Table 65. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	17.25	4.56	38.0	395.0	0.46	0.9
32	18.25	4.82	37.4	401.0	0.38	0.7
33	19.25	5.09	--	--	0.44	0.9
34	20.25	5.35	38.4	391.0	0.43	0.85
35	21.25	5.62	37.5	400.0	0.44	0.9
36	22.25	5.88	37.0	406.0	0.43	0.85
37	23.25	6.15	38.0	395.0	0.42	0.8
38	24.25	6.41	--	--	0.48	1.0
39	25.25	6.67	--	--	0.58	1.3
40	26.25	6.94	39.0	385.0	0.53	1.15
41	27.25	7.10	37.8	397.0	0.64	1.5
42	28.25	7.46	--	--	0.59	1.3
43	29.25	7.73	37.8	397.0	0.66	1.5
44	30.25	8.00	--	--	0.70	1.7
45	31.25	8.26	38.5	390.0	0.60	1.35
46	32.25	8.52	--	--	0.60	1.35
47	33.25	8.79	38.4	391.0	0.68	1.6
48	34.25	9.05	38.5	390.0	0.72	1.7
49	35.25	9.31	--	--	0.76	1.9
50	36.25	9.58	39.0	385.0	0.82	2.0
51	37.25	9.85	39.8	377.0	0.82	2.0
52	38.25	10.10	39.7	378.0	0.82	2.6
53	39.25	10.37	--	--	1.00	2.3
54	40.25	10.62	39.7	378.0	0.91	2.6
55	41.25	10.90	34.5	435.0 ¹	1.00	3.1
56	42.25	11.17	38.0	395.0	1.17	3.1
57	43.25	11.43	38.3	392.0	--	--
58	44.25	11.69	--	--	1.13	3.0
59	45.25	11.96	39.0	385.0	--	--
60	46.25	12.22	--	--	1.21	3.3

Table 65. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
61	47.25	12.49	39.1	384.0	1.36	3.75
62	48.25	12.75	38.8	387.0	1.39	3.8
63	49.25	13.01	39.3	382.0	1.32	3.6
64	50.25	13.28	39.8	377.0	1.41	3.9
65	51.25	13.53	39.0	385.0	1.51	4.2
66	52.25	13.80	39.2	383.0 ⁱ	1.66	4.7
67 ^a	53.25 ^a	14.08 ^a	33.0 ^a	455.0 ^a	1.67 ^a	4.7 ^a
68	54.25	14.33	--	---	1.54	4.3
69	55.25	14.60	30.0	500.0	1.49	4.2
70	56.25	14.87	31.0	485.0	1.52	4.25
71	57.25	15.13	30.0	500.0	1.53	4.25
72	58.25	15.40	39.0	385.0	1.59	4.5
73	59.25	15.66	37.0	406.0	1.72	4.9
74	60.25	15.91	--	---	1.74	4.95
75	61.25	16.19	35.0	429.0	1.62	4.7
76	62.25	16.45	--	---	1.81	5.2
77	63.25	16.70	34.0	441.0	1.75	5.0
78	64.25	16.98	33.0	283.0	2.13	6.2
79	65.25	17.23	45.0	334.0	2.10	6.1
80	66.25	17.50	47.0	319.0	1.94	5.6
81	67.25	17.77	38.0	395.0	2.18	6.4
82	68.25	18.02	38.0	395.0	2.22	6.5
83	69.25	18.29	38.8	387.0	2.38	7.0
84	70.25	18.55	37.0	406.0	2.40	7.05
85	71.25	18.81	37.3	402.0	2.70	8.0
86	72.25	19.09	37.2	404.0	2.65	7.8
87	73.25	19.35	37.0	406.0	2.75	8.15
88	74.25	19.61	36.5	411.0	3.04	9.1
89	75.25	19.88	36.0	417.0	2.81	8.5
90	76.25	20.14	36.0	417.0	3.45	10.4

Table 65. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
91	77.25	20.40	35.2	426.0	3.64	11.0
92	78.25	20.66	36.2	415.0	3.62	10.6
93	79.25	20.94	36.0	417.0	3.76	11.35
94	80.25	21.20	35.2	426.0	--	---
95	81.25	21.46	36.8	408.0	3.82	11.55
96	82.25	21.73	37.0	406.0	--	---
97	83.25	22.00	37.4	402.0	4.16	12.6
98	84.25	22.26	37.6	399.0	--	---
99	85.25	22.52	37.0	406.0	4.42	13.45
100	86.25	22.79	37.4	402.0	--	---
101	87.25	23.05	--	---	4.52	13.75
102	88.25	23.31	37.2	402.0	--	---
103	89.25	23.58	36.5	411.0	4.72	14.4
104	90.25	23.84	38.2	393.0	--	---
105	91.25	24.10	38.0	395.0	4.98	15.2
106	92.25	24.38	37.0	406.0	--	---
107	93.25	23.64	37.2	404.0	5.02	15.35
108	94.25	24.90	37.0	406.0	--	---
109	95.25	25.17	37.2	404.0	5.26	16.1
110	96.25	25.43	37.2	404.0	--	---
111	97.25	25.70	37.0	406.0	5.38	16.5
112	98.25	25.96	37.2	404.0	--	---
113	99.25	26.22	37.0	406.0	5.34	16.35
114	100.25	26.50	37.0	406.0	--	---
115	101.25	26.77	36.8	408.0	5.62	17.2
116	102.25	27.03	37.0	406.0	--	---
117	103.25	27.29	37.0	406.0	5.92	18.3
118	104.25	27.55	36.6	410.0	--	---
119	105.25	27.80	36.7	409.0	6.00	18.45
120	106.25	28.07	36.2	415.0	--	---

Table 65. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
121	107.25	28.33	37.0	406.0	6.42	19.8
122	108.25	28.60	36.4	412.0	6.04	18.6
123	109.25	28.86	37.4	401.0	6.32	19.4
124	110.25	29.13	40.0	375.0	5.94	18.3
125	111.25	29.40	39.0	385.0	6.04	18.6
126	112.25	29.66	36.8	408.0	6.16	19.0
127	113.25	29.93	36.2	415.0	6.06	18.6
128	114.25	30.20	37.5	400.0	5.90	18.1
129	115.25	30.46	39.0	385.0	5.86	18.0
130	116.25	30.73	39.0	385.0	5.92	18.2
131	117.25	31.00	40.5	371.0	6.00	18.45

^aRun stopped for four days at this point.

ⁱIncreased rate of flow by opening valve slightly.

Table 66. Second run for conditioning of fluidized zeolite bed (Run 42)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	2.0	0.53	60.0	250.0	0.29	0.4
2	4.0	1.06	38.0	395.0	0.23	0.2
3	6.0	1.59	38.4	391.0	0.22	0.2
4	8.0	2.11	43.0	349.0	0.23	0.2
5	10.0	2.64	36.0	417.0	0.22	0.2
6	12.0	3.17	37.0	406.0	0.24	0.2
7	14.0	3.70	37.0	406.0	0.21	0.1
8	15.0	4.23	36.8	408.0	0.22	0.2
9	18.0	4.76	38.0	395.0	0.26	0.3
10	20.0	5.29	39.0	385.0	0.22	0.2
11	22.0	5.82	39.8	377.0	0.20	0.1
12	24.0	6.35	41.0	366.0	0.23	0.2
13	26.0	6.88	39.6	379.0	0.21	0.1
14	28.0	7.40	42.0	358.0	0.25	0.3
15	30.0	7.94	39.0	385.0 ⁱ	0.22	0.2
16	32.0	8.46	36.0	407.0	0.22	0.2
17	34.0	8.99	36.0	417.0	0.21	0.1
18	36.0	9.53	36.4	412.0	0.24	0.2
19	38.0	10.03	37.0	406.0	0.21	0.1
20	40.0	10.57	36.2	415.0	0.23	0.2
21	42.0	11.10	37.0	406.0	0.22	0.2
22	44.0	11.62	37.0	406.0	0.24	0.2
23	46.0	12.15	39.4	381.0	0.25	0.3
24	48.0	12.69	38.0	395.0	0.27	0.3
25	50.0	13.21	39.0	385.0	0.29	0.4
26	52.0	13.75	38.0	395.0	0.29	0.4
27	54.0	14.27	36.6	410.0	0.30	0.4
28	56.0	14.80	37.2	404.0	0.31	0.4
29	58.0	15.33	37.3	402.0	0.31	0.4
30	60.0	15.85	38.4	391.0	0.30	0.4

Table 66. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	62.0	16.58	39.0	385.0	0.35	0.6
32	64.0	16.90	35.8	420.0 ¹	0.41	0.8
33	66.0	17.44	36.2	415.0	0.38	0.7
34	68.0	17.97	35.6	422.0	0.40	0.7
35	70.0	18.50	35.0	429.0	0.45	0.9
36	72.0	19.02	35.0	429.0	0.44	0.9
37	74.0	19.52	36.0	417.0	0.49	1.0
38	76.0	20.04	36.2	415.0	0.47	0.9
39	78.0	20.60	36.2	415.0	0.55	1.2
40	80.0	21.12	37.2	404.0	0.62	1.4
41	82.0	21.65	36.4	412.0	0.70	1.7
42	84.0	22.20	37.2	404.0	0.82	2.0
43	86.0	22.73	36.4	412.0	0.94	2.4
44	88.0	23.25	36.2	415.0	1.01	2.6
45	90.0	23.78	35.4	424.0	1.17	3.2
46	92.0	24.30	36.3	414.0	1.23	3.4
47	94.0	24.82	37.6	399.0	1.43	4.0
48	96.0	25.37	37.6	399.0	1.60	4.5
49	98.0	25.90	37.0	406.0	1.68	4.8
50	100.0	26.40	36.5	411.0	1.92	5.5
51	102.0	26.94	35.4	424.0	2.26	6.6
52	104.0	27.47	35.0	429.0	2.71	8.0
53	106.0	28.00	37.6	399.0	3.50	10.55
54	108.0	28.53	32.0	469.0	--	--
55	110.0	29.06	32.0	469.0	3.76	11.4
56	112.0	29.60	33.0	455.0	--	--
57	114.0	30.12	33.4	449.0	4.26	13.0
58	116.0	30.65	33.1	454.0	--	--
59	118.0	31.20	34.0	441.0	4.44	13.5
60	120.0	31.73	34.0	441.0	--	--

Table 66. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ³	Gr. per gal.
61	122.0	32.23	34.2	439.0	4.54	13.8
62	124.0	32.78	34.1	440.0	--	---
63	126.0	33.30	33.6	447.0	4.84	14.8
64	128.0	33.82	33.4	449.0	5.18	15.9
65	130.0	34.37	33.5	448.0	5.22	15.95
66	132.0	34.90	33.6	447.0	5.36	16.4
67	134.0	35.40	33.8	444.0	5.56	17.0
68	136.0	35.95	34.1	440.0	5.84	17.9
69	138.0	36.48	34.6	434.0	5.76	17.7
70	140.0	37.00	34.6	434.0	6.04	18.6
71	142.0	37.53	34.7	433.0	6.00	18.45
72	144.0	38.06	35.0	429.0	6.34	19.5
73	146.0	38.60	35.5	423.0	--	---
74	148.0	39.10	35.6	422.0	--	---
75	150.0	39.63	35.3	426.0	6.36	19.6
76	152.0	40.20	35.4	424.0	6.46	19.9
77	154.0	40.73	35.2	426.0	6.64	20.4
78	156.0	41.23	35.2	426.0	6.72	20.7
79	158.0	41.76	36.2	415.0	6.80	20.95
80	160.0	42.30	36.8	409.0	7.04	21.7
81	162.0	42.83	37.0	406.0	7.20	22.2
82	164.0	43.38	36.5	411.0	6.98	21.5
83	166.0	43.90	36.4	412.0	6.88	21.2
84	168.0	44.43	37.0	406.0	7.02	21.6
85	170.0	44.98	37.2	404.0	7.40	21.4
86	172.0	45.48	37.2	404.0	7.56	23.3
87	174.0	46.00	36.4	412.0	7.06	21.8
88	176.0	46.53	37.3	403.0	7.12	21.9
89	178.0	47.05	37.5	400.0	7.50	23.1
90	180.0	47.60	--	---	7.44	23.0

ⁱIncreased rate of flow by opening valve slightly.

Table 67. Third run for conditioning of fluidized zeolite bed (Run 43)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	3.0	0.79	16.9	887.0	0.28	0.4
2	6.0	1.58	16.7	898.0	0.14	0.0
3	9.0	2.38	18.7	801.0 ^d	0.18	0.0
4	12.0	3.17	37.4	401.0 ^d	0.18	0.0
5	15.0	3.96	39.0	385.0	0.12	0.0
6	18.0	4.76	39.0	385.0	0.19	0.1
7	21.0	5.55	35.2	426.0 ⁱ	0.16	0.0
8	24.0	6.34	35.6	422.0	0.15	0.0
9	27.0	7.13	35.6	422.0	0.13	0.0
10	50.0	7.93	35.5	411.0	0.17	0.0
11	35.0	8.72	38.0	395.0	0.12	0.0
12	56.0	9.51	36.0	417.0 ⁱ	0.15	0.0
13	39.0	10.30	35.2	426.0	0.19	0.1
14	42.0	11.10	35.0	429.0	0.14	0.0
15	45.0	11.89	--	---	0.11	0.0
16	48.0	12.69	34.2	439.0 ⁱ	0.16	0.0
17	51.0	13.48	33.0	455.0	0.20	0.1
18	54.0	14.27	32.8	458.0	0.17	0.0
19	57.0	15.05	32.0	469.0	0.17	0.0
20	60.0	15.85	32.4	464.0	0.14	0.0
21	63.0	16.64	31.8	472.0	0.15	0.0
22	66.0	17.43	34.8	431.0 ^d	0.17	0.0
23	69.0	18.21	34.8	431.0	0.20	0.1
24	72.0	19.00	34.8	451.0	0.18	0.1
25	75.0	19.80	34.9	430.0	0.18	0.1
26	78.0	20.60	36.4	413.0	0.17	0.0
27	81.0	21.40	37.4	401.0	0.15	0.0
28	84.0	22.18	31.8	472.0 ⁱ	0.21	0.15
29	87.0	22.96	32.2	466.0	0.29	0.4
30	90.0	23.78	32.4	464.0	0.29	0.4

Table 67. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	93.0	24.57	32.6	461.0	0.28	0.4
32	96.0	25.37	31.5	477.0	0.38	0.7
33	99.0	26.17	31.4	479.0	0.51	1.1
34	102.0	26.96	31.2	481.0	0.75	1.8
35	105.0	27.76	31.0	484.0	1.22	3.3
36	108.0	28.55	31.2	481.0	2.12	6.0
37	111.0	29.34	31.1	483.0	2.40	7.05
38	114.0	30.13	31.0	484.0	3.86	11.7
39	117.0	30.91	33.4	450.0 ^d	4.21	12.8
40	120.0	31.70	33.8	444.0	2.56 ^a	15.2
41	123.0	32.50	34.4	436.0	2.91	17.4
42	126.0	33.29	34.4	436.0	2.69	16.0
43	129.0	34.09	34.4	436.0	2.93	17.5
44	132.0	34.87	35.0	429.0	3.03	18.2
45	135.0	35.67	35.0	429.0	3.19	19.1
46	138.0	36.46	35.2	426.0	3.15	18.8
47	141.0	37.35	35.6	422.0	3.15	18.8
48	144.0	38.04	36.9	407.0	3.16	18.9
49	147.0	38.83	31.0	485.0 ⁱ	3.20	19.2
50	150.0	39.62	35.8	419.0 ^d	3.27	19.6
51	153.0	40.4	36.0	417.0	3.17	19.0
52	156.0	41.2	33.4	450.0 ⁱ	3.35	20.1
53	159.0	42.0	33.8	444.0	3.30	19.8
54	162.0	42.8	34.4	437.0	3.47	20.8
55	165.0	43.6	34.5	435.0	3.31	19.9
56	168.0	44.4	34.4	437.0	3.34	20.0
57	171.0	45.2	34.7	433.0	3.44	20.7
58	174.0	46.0	36.4	412.0	3.46	20.8
59	177.0	46.8	34.7	433.0 ⁱ	3.47	20.9
60	180.0	47.6	34.6	434.0	3.46	20.8

Table 67. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
61	183.0	48.4	35.6	422.0	3.55	21.5
62	186.0	49.2	36.0	417.0	3.53	21.3
63	189.0	50.0	33.7	445.0 ¹	3.46	20.8
64	192.0	50.8	33.8	444.0	3.49	21.0
65	195.0	51.5	33.8	444.0	3.55	21.4
66	198.0	52.3	34.0	441.0	3.47	20.9
67	201.0	53.1	34.4	437.0	3.56	21.4
68	204.0	53.9	35.0	429.0	3.52	21.2
69	207.0	54.7	35.0	429.0	3.51	21.2
70	210.0	55.5	35.1	430.0	3.50	21.1
71	213.0	56.3	32.7	459.0 ¹	3.48	21.0
72	216.0	57.1	33.4	450.0	3.51	21.2
73	219.0	57.9	33.6	447.0	3.50	21.1
74	222.0	58.7	33.0	455.0	3.50	21.1
75	225.0	59.5	--	---	3.55	21.4

^aSamples from this point are 25.0 ml. in volume.

^dDecreased rate of flow by closing valve slightly.

¹Increased rate of flow by opening valve slightly.

Table 68. Softening capacity at high flow rate with 200 grams zeolite fines in fluidized bed (Run 44)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	5.0	0.79	15.2	986.0	0.42	0.8
2	6.0	1.58	16.0	937.0	0.71	1.7
3	9.0	2.38	20.8	720.0	0.70	1.7
4	12.0	3.17	20.6	728.0	0.59	1.3
5	15.0	3.96	20.6	728.0	0.66	1.6
6	18.0	4.76	20.9	718.0	0.80	2.0
7	21.0	5.55	20.8	720.0	0.94	2.4
8	24.0	6.34	20.8	720.0	1.34	3.7
9	27.0	7.13	21.0	714.0	1.42	3.9
10	30.0	7.93	21.2	707.0	1.86	5.3
11	33.0	8.72	21.5	704.0	2.25	6.6
12	36.0	9.51	21.6	694.0	1.70 ^a	9.7
13	39.0	10.30	22.2	675.0	2.12	12.3
14	42.0	11.10	22.0	681.0	2.46	14.4
15	45.0	11.89	21.8	688.0	2.75	16.3
16	48.0	12.69	22.0	681.0	2.98	17.8
17	51.0	13.48	21.9	685.0	3.13	18.8
18	54.0	14.27	21.8	688.0	3.09	18.4
19	57.0	15.05	21.8	688.0	3.29	19.8
20	60.0	15.85	22.0	681.0	3.43	20.6
21	63.0	16.64	22.1	678.0	3.40	20.4
22	66.0	17.43	22.4	669.0	3.49	21.0
23	69.0	18.21	22.5	666.0	3.57	21.5
24	72.0	19.00	23.0	651.0	3.60	21.7
25	75.0	19.80	20.9	718.0 ⁱ	3.59	21.6
26	78.0	20.60	21.4	700.0	3.58	21.6
27	81.0	21.40	21.5	697.0	3.63	22.0
28	84.0	22.19	21.9	685.0	3.65	22.0
29	87.0	22.98	22.0	681.0	3.76	22.8
30	80.0	23.78	22.0	681.0	3.77	22.8

Table 68. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	83.0	24.57	22.0	681.0	3.77	22.8
32	86.0	25.37	22.4	669.0	3.81	23.0
33	89.0	26.17	21.9	685.0	3.83	23.2
34	92.0	26.96	22.2	675.0	3.73	22.6
35	95.0	27.76	--	---	3.84	23.2

^aSamples from this point are 25.0 ml. in volume.

ⁱIncreased rate of flow by opening valve slightly.

Table 69. Softening capacity at intermediate flow rate with 200 grams zeolite fines in fluidized bed (Run 45)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	3.0	0.79	37.2	404.0	0.42	0.8
2	6.0	1.58	34.6	454.0	0.20	0.1
3	9.0	2.38	35.4	424.0	0.20	0.1
4	12.0	3.17	37.2	404.0	0.19	0.1
5	15.0	3.96	37.0	406.0	0.24	0.2
6	18.0	4.76	38.2	393.0	0.32	0.5
7	21.0	5.55	36.0	417.0	0.42	0.8
8	24.0	6.34	36.6	410.0	0.57	1.3
9	27.0	7.13	35.0	429.0	0.83	2.1
10	30.0	7.93	35.4	424.0	1.30	3.6
11	33.0	8.72	35.4	424.0	2.51	6.8
12	36.0	9.51	33.5	448.0 ⁱ	1.91 ^a	11.0
13	39.0	10.30	33.8	444.0	2.48	14.6
14	42.0	11.10	34.0	441.0	2.71	16.0
15	45.0	11.89	34.3	458.0	3.01	16.4
16	48.0	12.69	34.8	431.0	3.08	16.8
17	51.0	13.48	35.2	426.0	3.21	17.6
18	54.0	14.27	32.8	458.0 ⁱ	3.30	19.8
19	57.0	15.05	33.4	449.0	3.45	20.8
20	60.0	15.85	34.2	439.0	3.41	20.5
21	63.0	16.64	34.7	432.0	3.51	21.2
22	66.0	17.43	35.0	429.0	3.49	21.0
23	69.0	18.21	36.0	417.0	3.54	21.4
24	72.0	19.00	33.0	455.0 ⁱ	3.49	21.0
25	75.0	19.80	34.0	441.0	3.72	22.4
26	78.0	20.60	33.4	450.0	3.67	22.2
27	81.0	21.40	34.2	439.0	3.72	22.4
28	84.0	22.19	34.0	441.0	3.73	22.4
29	87.0	22.98	34.5	435.0	3.84	23.2
30	90.0	23.78	34.9	430.0	3.69	22.3

Table 69. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	93.0	24.57	35.0	429.0	3.82	23.1
32	96.0	25.37	31.9	471.0 ⁱ	3.61	21.8
33	99.0	26.17	32.4	463.0	3.56	21.4
34	102.0	26.96	32.0	469.0	3.81	23.0
35	105.0	27.76	--	---	3.77	22.8

^aSamples from this point are 25.0 ml. in volume.

ⁱIncreased rate of flow by opening valve slightly.

Table 70. Softening capacity at low flow rate with 200 grams zeolite fines in fluidized bed (Run 46)

Test No.	Effluent volume		Flow rate	Hardness	
	Observed, liters	Calc., gal.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	3.15	0.83	70.0	0.45 ^a	0.9
2	4.15	1.10	--	0.30	0.4
3	5.15	1.36	--	0.17	0.0
4	6.15	1.62	--	0.14	0.0
5	11.07	2.92	41.0	0.16	0.0
6	13.69	3.62	32.8	0.15	0.0
7	14.72	3.89	17.2	0.18	0.0
8	19.17	5.06	12.9	0.17	0.0
9	27.73	7.32	11.4	0.18	0.0
10	31.11	8.22	16.1	0.41	0.8
11	32.21	8.51	--	1.03	2.7
12	33.24	8.78	--	1.17	3.1
13	34.24	9.05	--	1.43	4.0
14	34.95	9.23	12.8	1.78	5.1
15	35.62	9.40	6.4	1.90	5.5
16	36.57	9.65	4.5	2.13	6.2
17	37.47	9.90	1.6	3.59	10.8
18	38.34	10.12	--	4.42	13.4

^aAll samples were taken from the mixed volume increments, and hence should be plotted at midpoints of volume increments.

Table 71. Softening capacity at intermediate flow rate in fluidized bed with two plates holding 200 grams zeolite fines each (Run 47)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
1	3.0	0.79	31.0	484.0	0.34	0.5
2	6.0	1.58	32.0	469.0 ^d	0.27	0.3
3	9.0	2.38	33.6	447.0 ^d	0.14	0.0
4	12.0	3.17	35.8	419.0	0.17	0.0
5	15.0	3.96	36.2	415.0	0.18	0.0
6	18.0	4.76	35.4	424.0	0.18	0.0
7	21.0	5.55	34.8	431.0	0.15	0.0
8	24.0	6.34	36.7	409.0 ^d	0.19	0.05
9	27.0	7.13	36.0	417.0	0.19	0.05
10	30.0	7.93	37.0	406.0	0.19	0.05
11	33.0	8.72	36.6	410.0	0.19	0.05
12	36.0	9.51	37.0	406.0	0.18	0.0
13	39.0	10.30	35.1	428.0	0.19	0.05
14	42.0	11.10	36.4	412.0	0.29	0.4
15	45.0	11.89	35.8	419.0	0.28	0.4
16	48.0	12.69	37.7	398.0	0.35	0.6
17	51.0	13.48	37.5	400.0 ⁱ	0.41	0.8
18	54.0	14.27	45.0	334.0 ^d	0.45	0.9
19	57.0	15.05	30.5	492.0 ⁱ	0.98	2.55
20	60.0	15.85	39.6	379.0 ^d	1.45	4.0
21	63.0	16.64	43.0	349.0	1.98	5.7
22	66.0	17.43	47.0	319.0	2.82	8.4
23	69.0	18.21	36.8	408.0	2.10 ^a	12.2
24	72.0	19.00	38.4	391.0	2.40	14.1
25	75.0	19.80	45.0	334.0	2.34	13.7
26	78.0	20.60	40.0	375.0	2.58	15.2
27	81.0	21.40	36.0	417.0	2.86	17.0
28	84.0	22.19	47.0	319.0	3.08	18.4
29	87.0	22.98	50.0	300.0	3.21	19.2
30	90.0	23.78	40.0	375.0	3.49	21.0

Table 71. (Continued)

Test No.	Effluent volume		Flow rate		Hardness	
	Observed, liters	Calc., gal.	Observed, sec./250 ml.	Calc., ml./min.	Soap, ml. ^a	Gr. per gal.
31	93.0	24.57	50.0	300.0	3.60	21.7
32	96.0	25.37	50.0	300.0	3.58	21.6
33	99.0	26.17	45.0	334.0	3.61	21.8
34	102.0	26.96	45.0	334.0	3.60	21.7
35	105.0	27.76	37.0	406.0	3.66	22.1

^aSamples from this point are 25.0 ml. in volume.

^dDecreased rate of flow by closing valve slightly.

ⁱIncreased rate of flow by opening valve slightly.

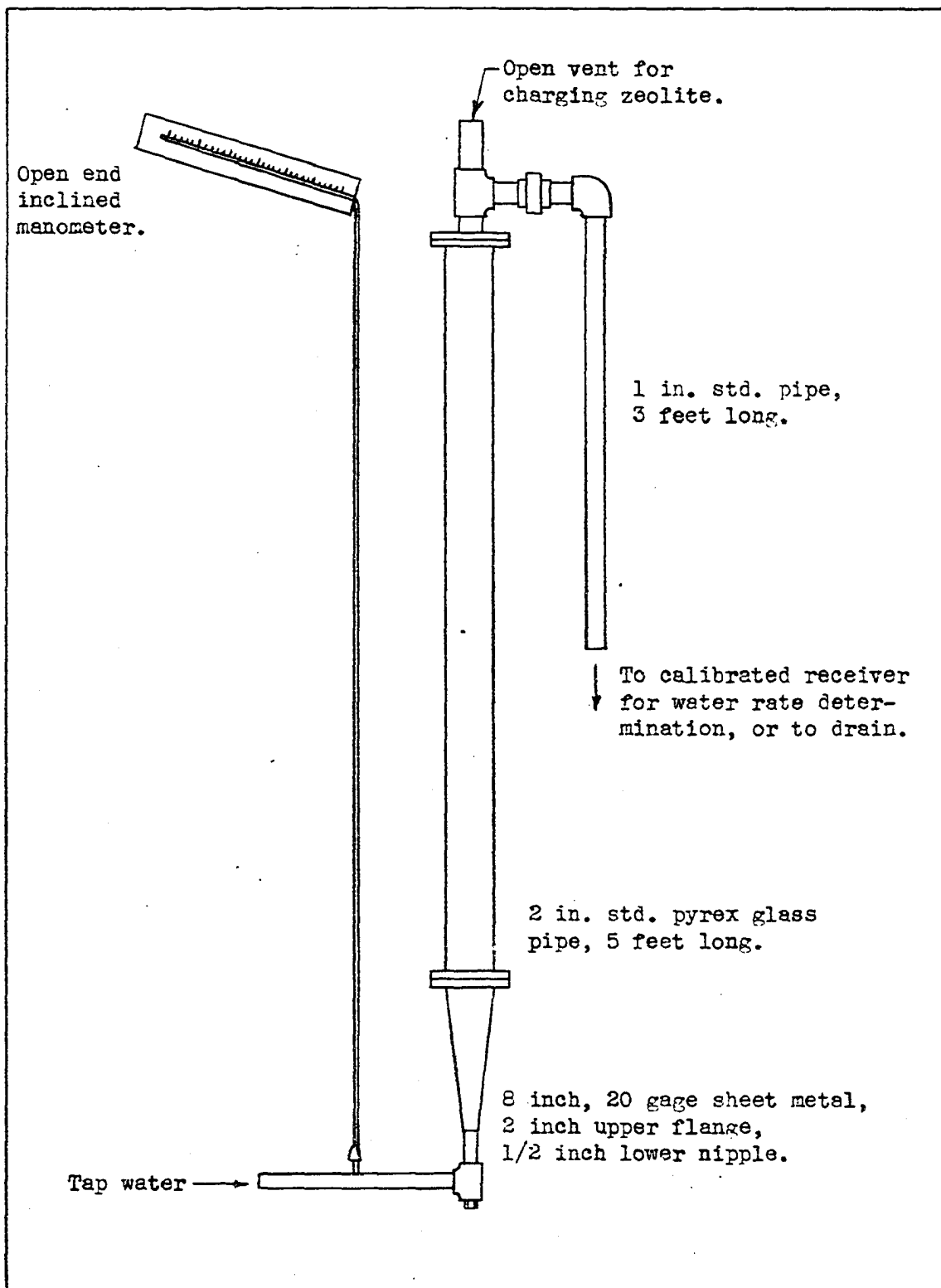


Fig. 27. Experimental apparatus for fluidization of zeolite fines bed.

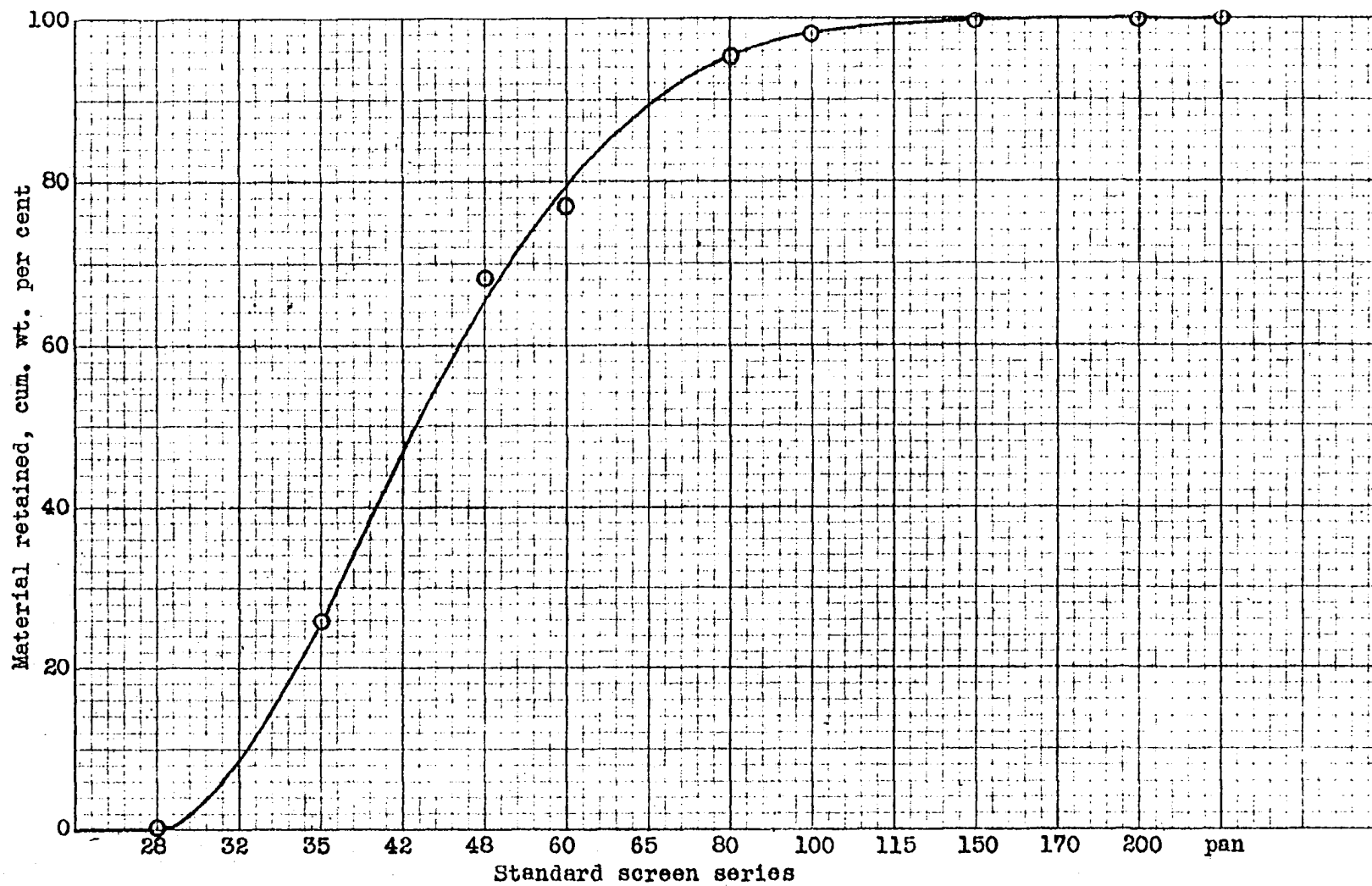


Fig. 28. Standard screen analysis of zeolite fines.

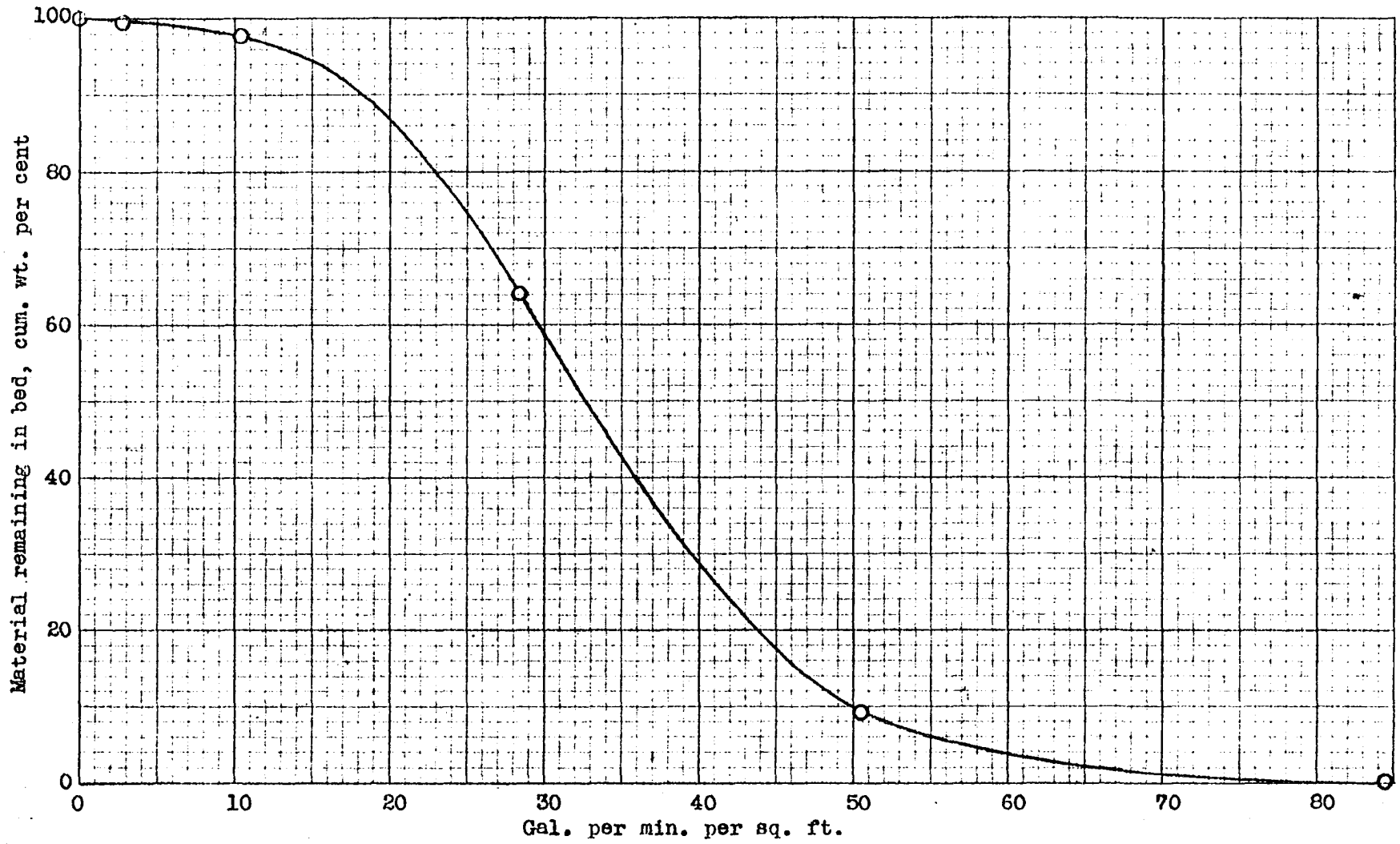


Fig. 29. Hydraulic classification of zeolite fines.

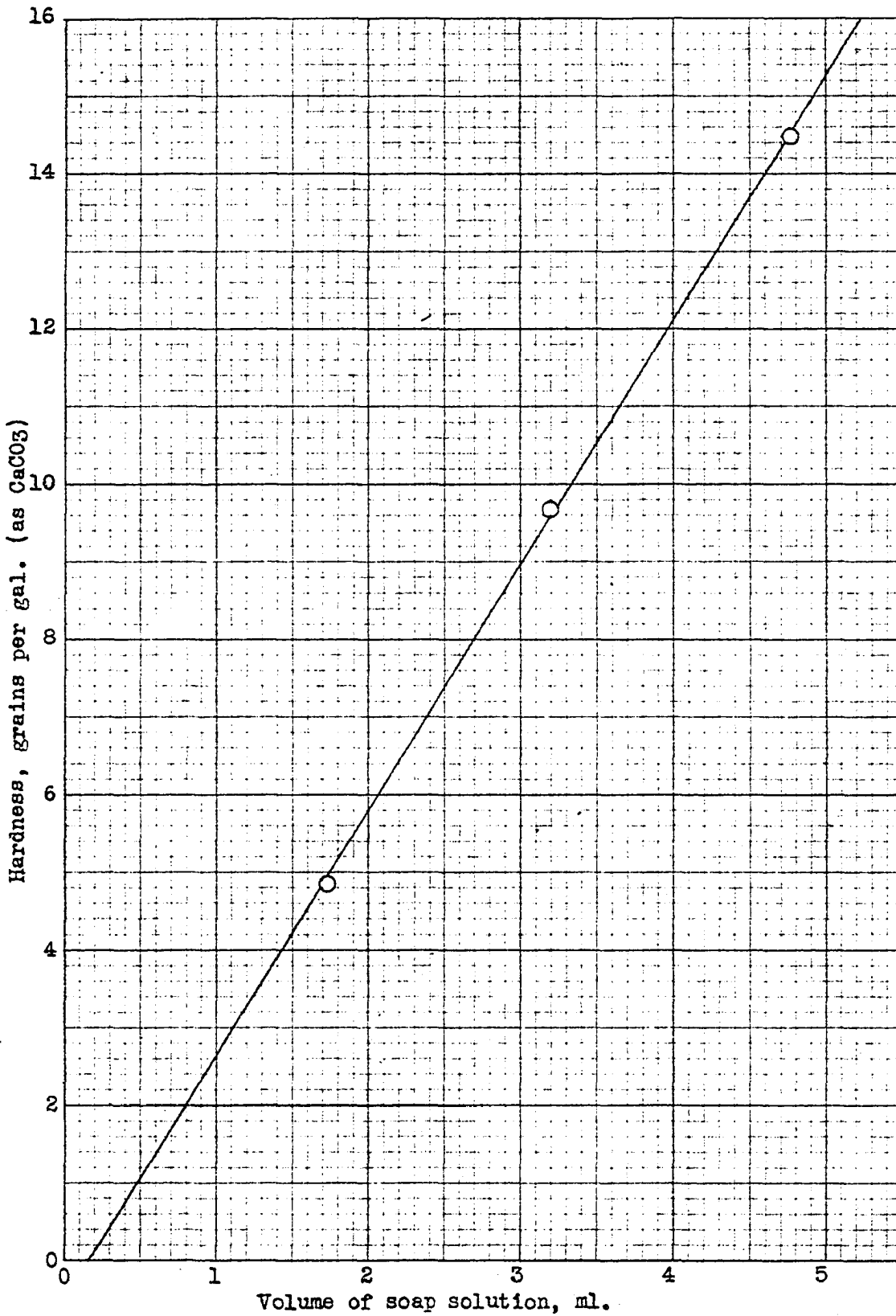


Fig. 30. Calibration of standard soap solution.

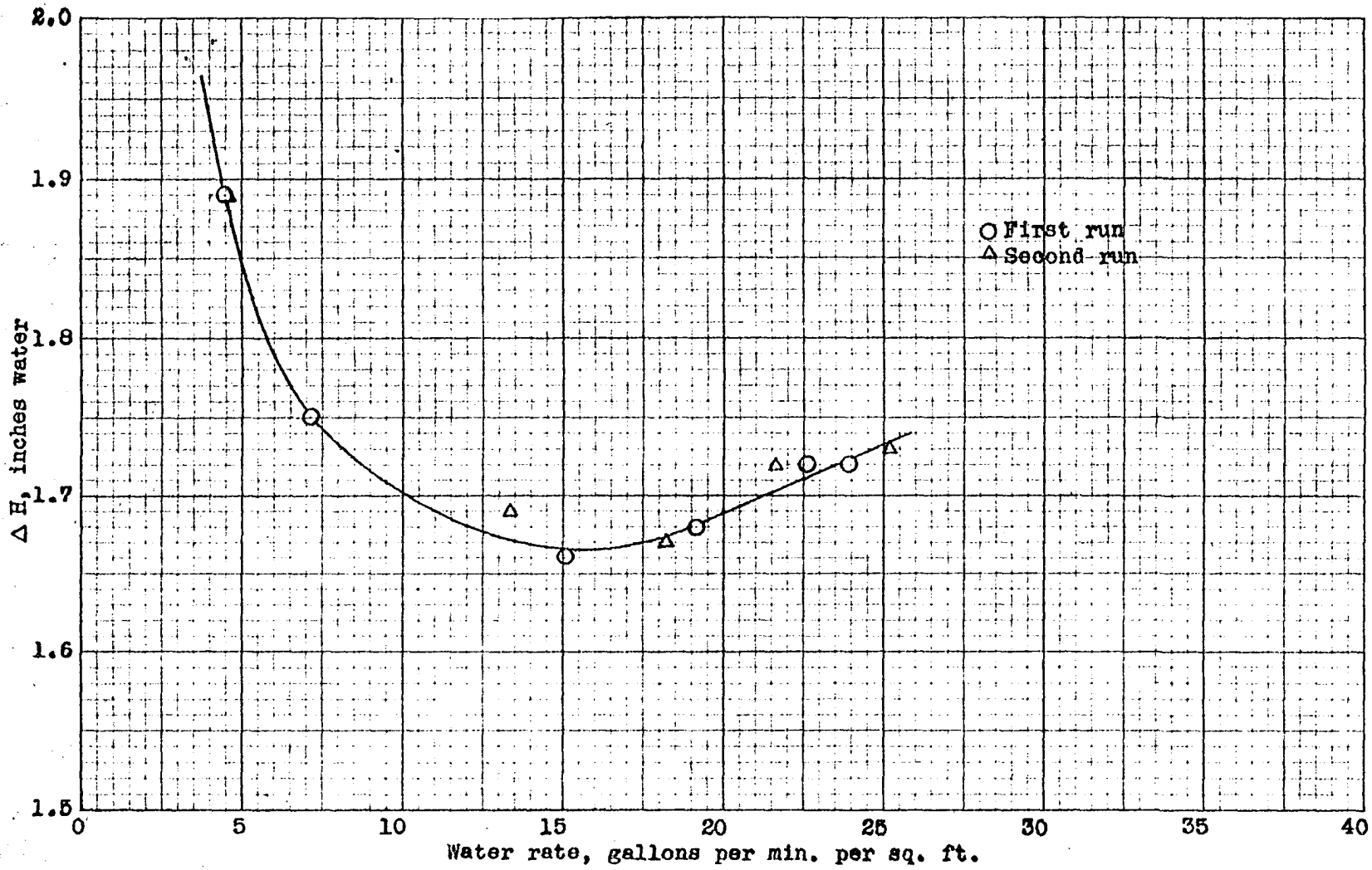


Fig. 31. Reproducibility of experimental data in fluidized zeolite bed.

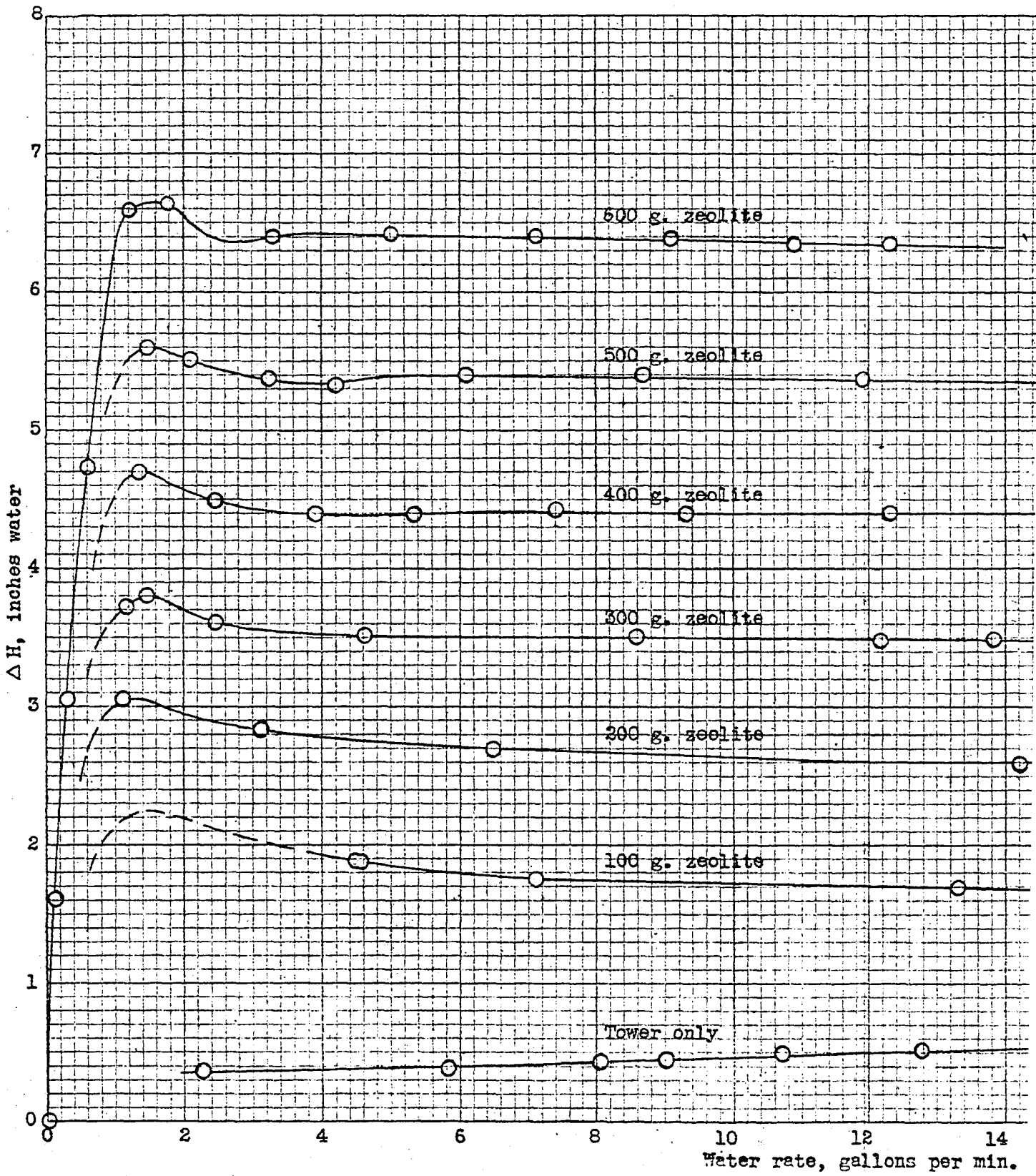
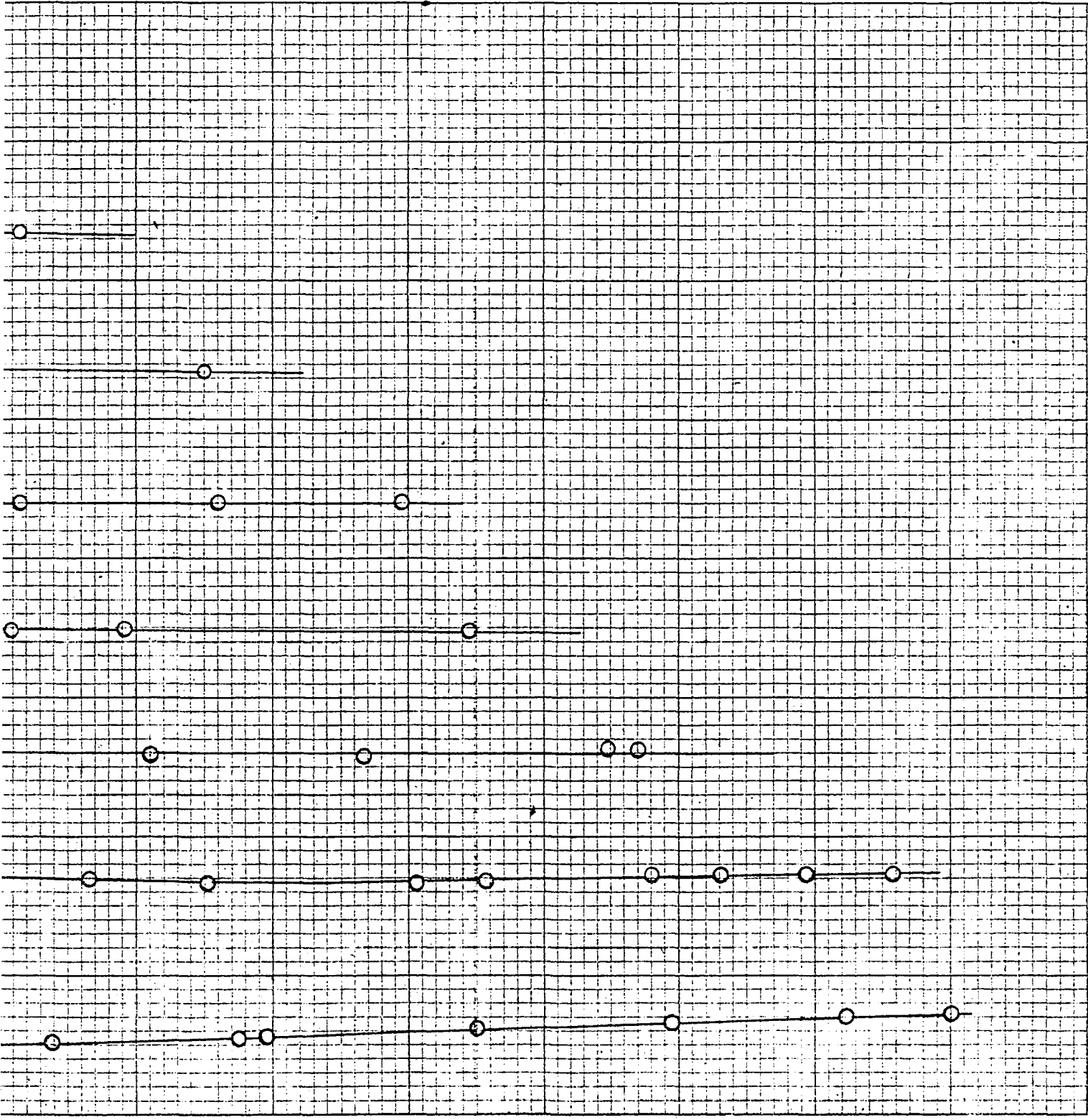


Fig. 32. Pressure drop in fluidized zeolite bed.



ons per min. per sq. ft.

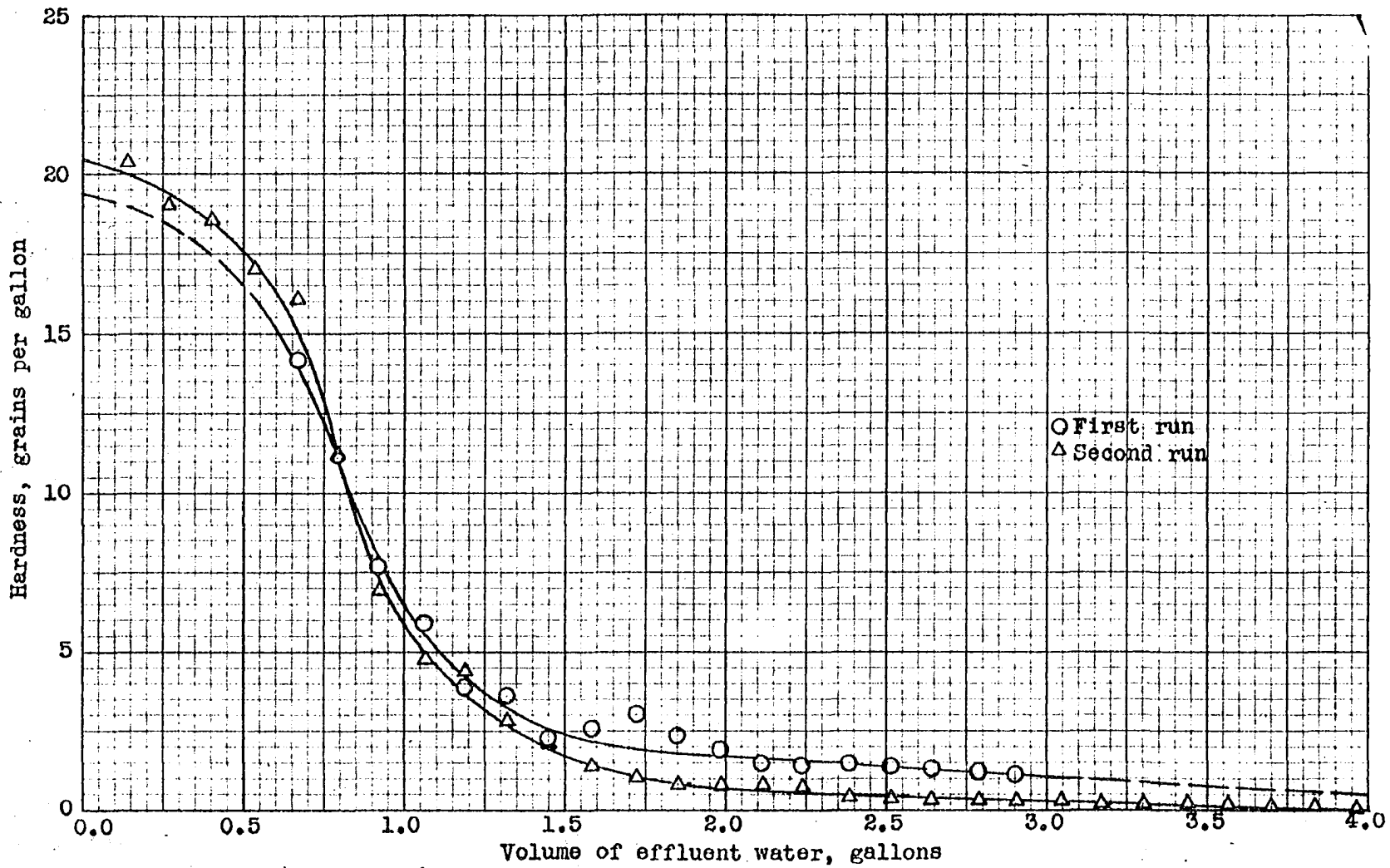


Fig. 33. Typical curves of rinsing operation in fluidized zeolite bed.

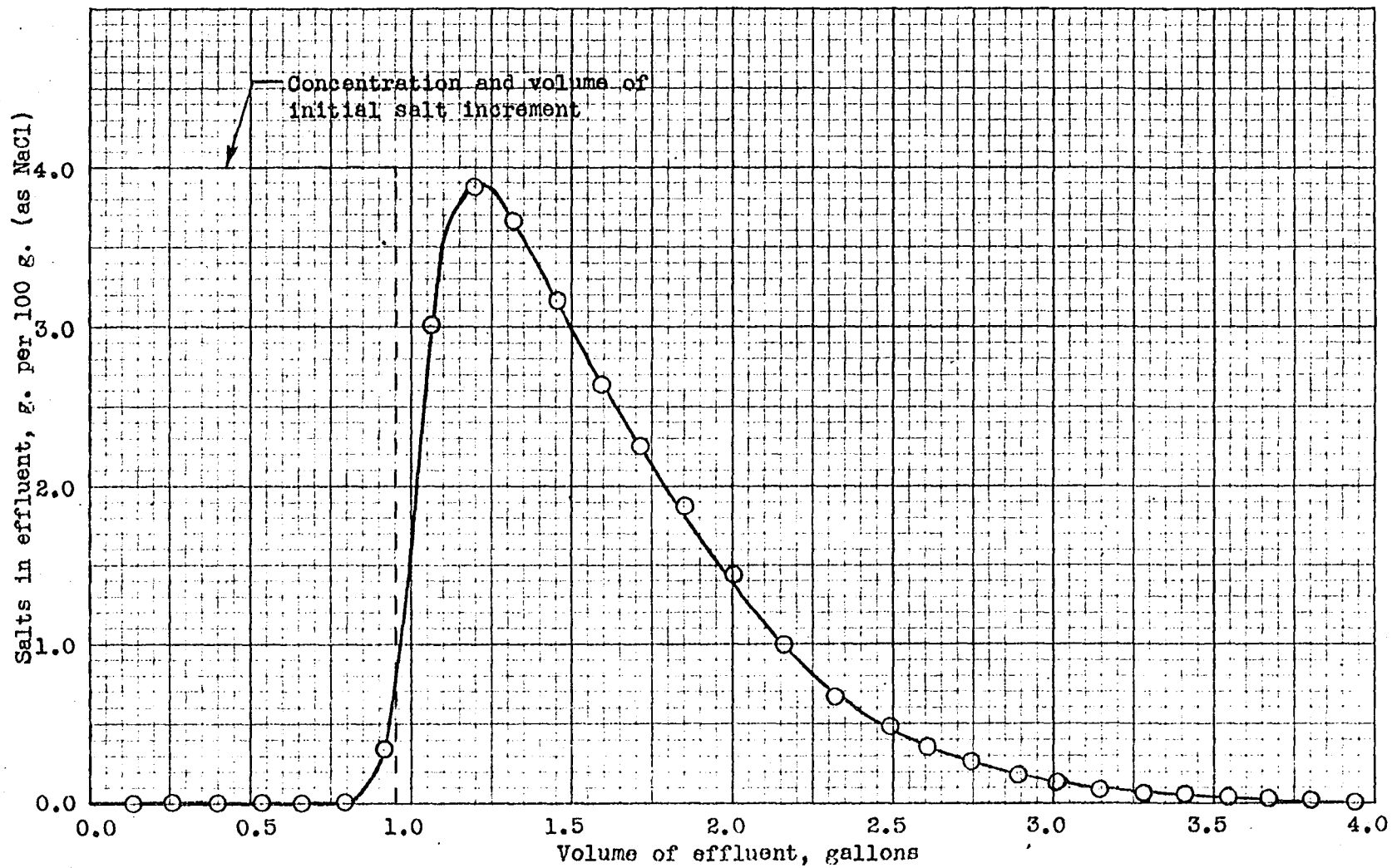


Fig. 34. Regeneration and rinsing of fluidized zeolite bed.

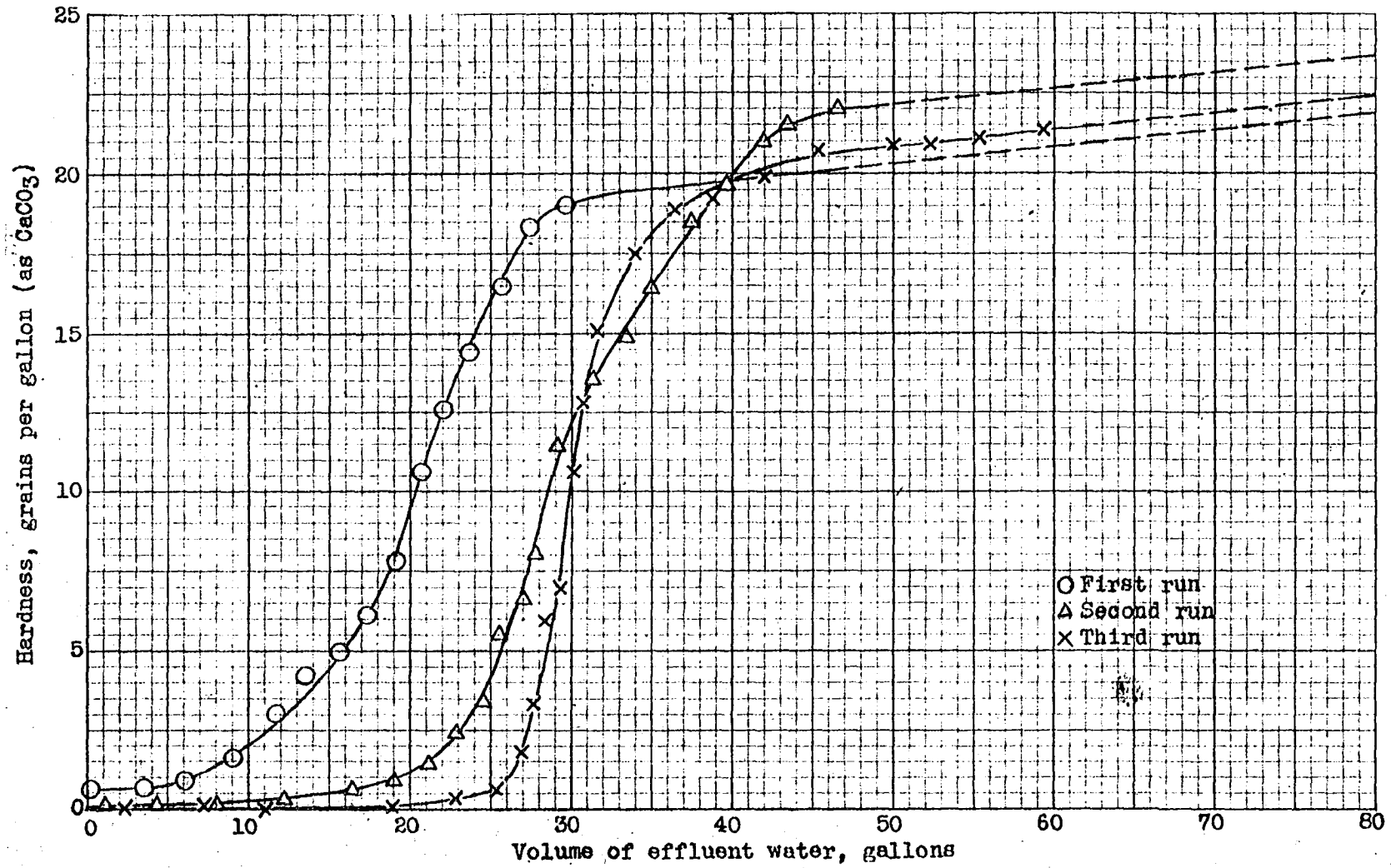


Fig. 35. Conditioning of fluidized zeolite bed.

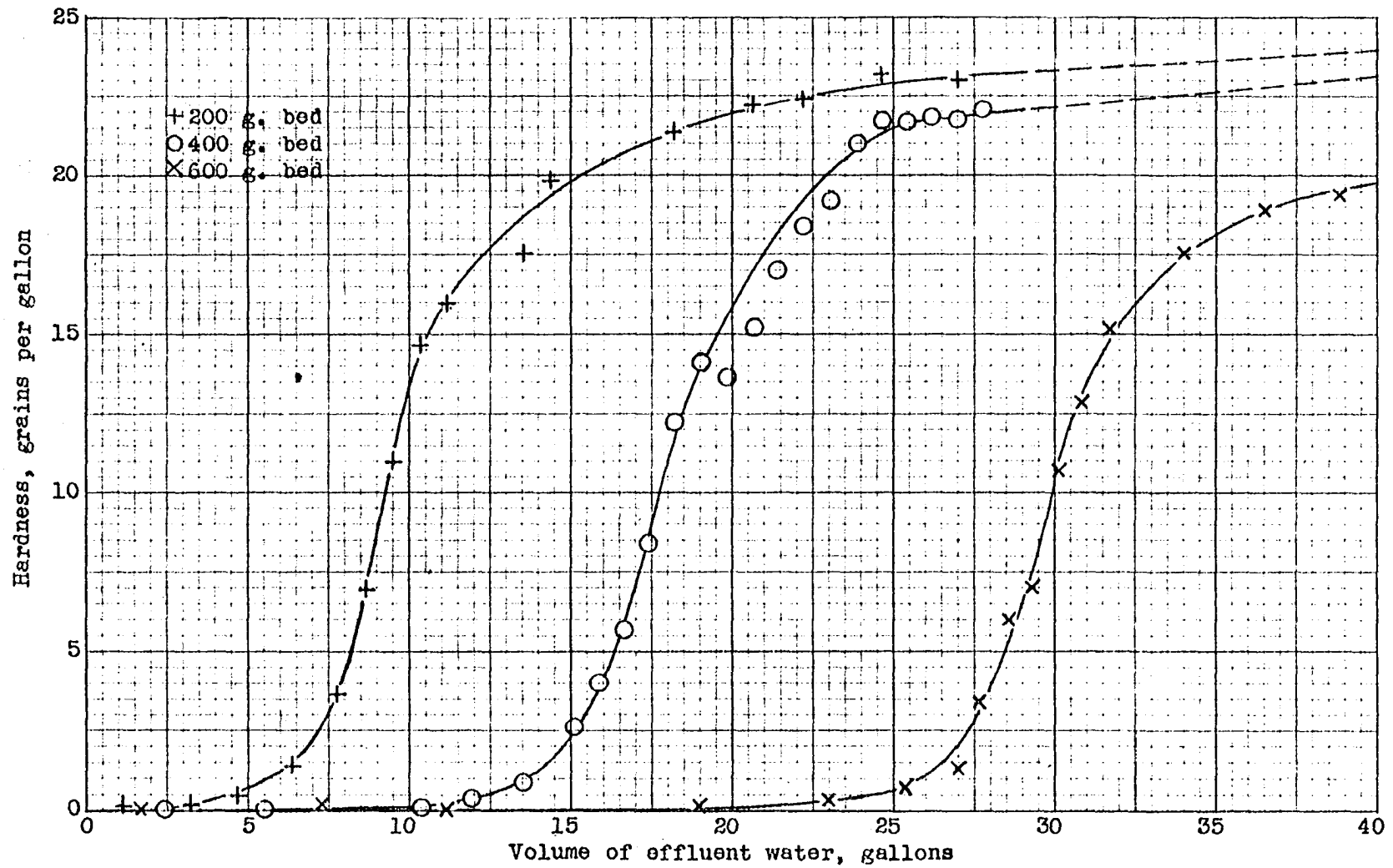


Fig. 36. Softening capacities of various sizes of fluidized zeolite beds.

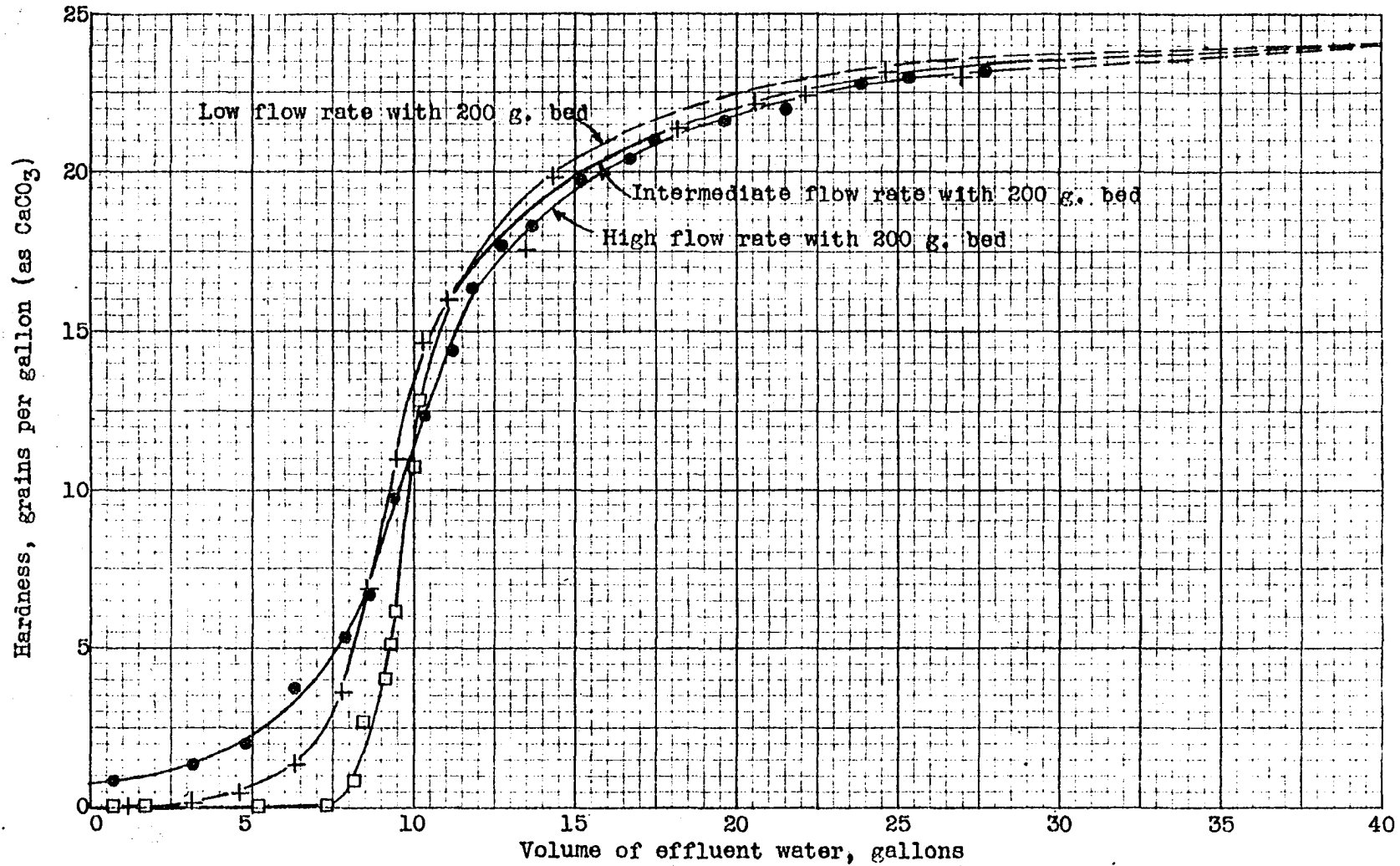


Fig. 37. Effect of rate of flow on softening capacity in fluidized zeolite bed.

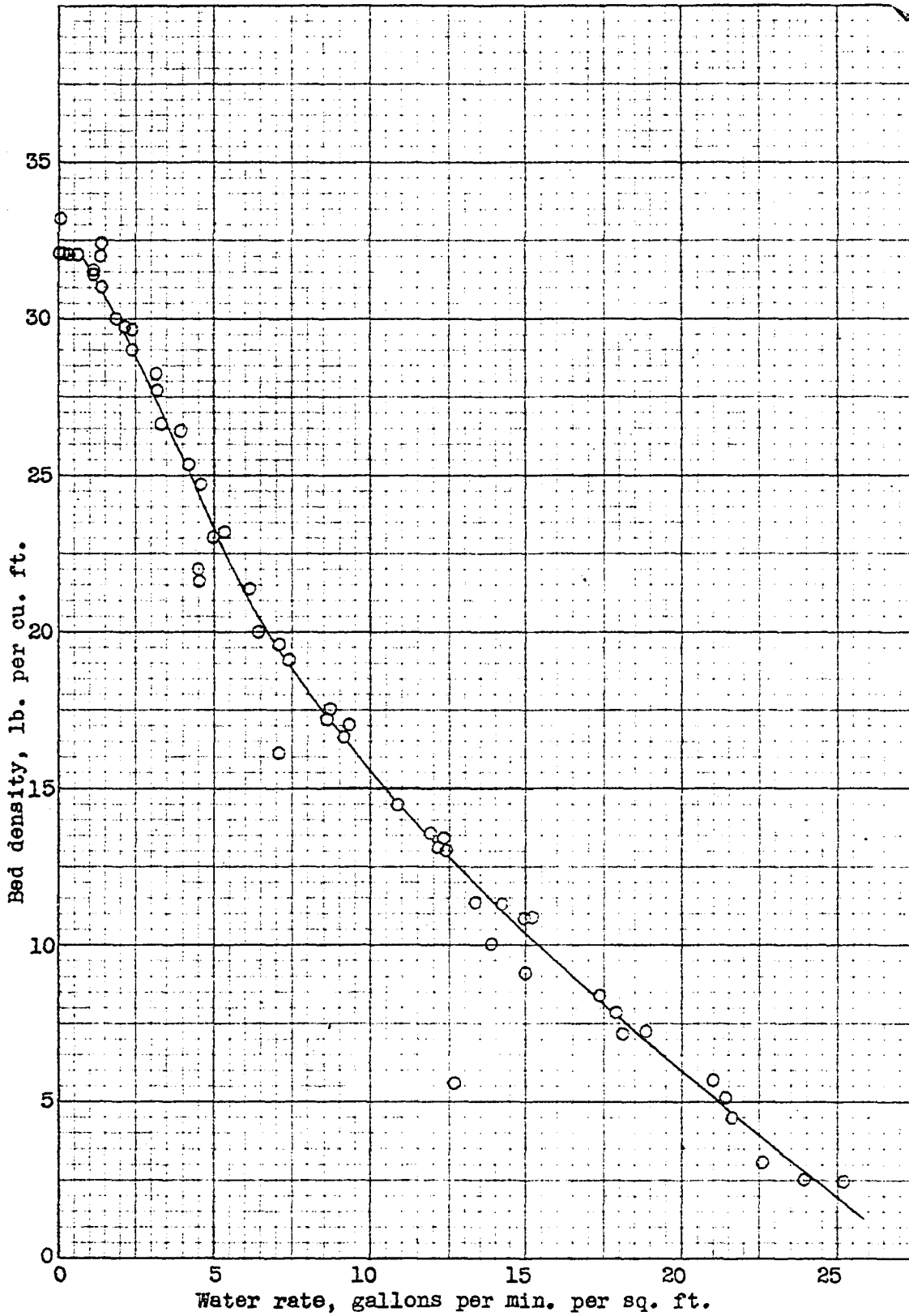


Fig. 38. Density of fluidized zeolite bed at various rates of flow.

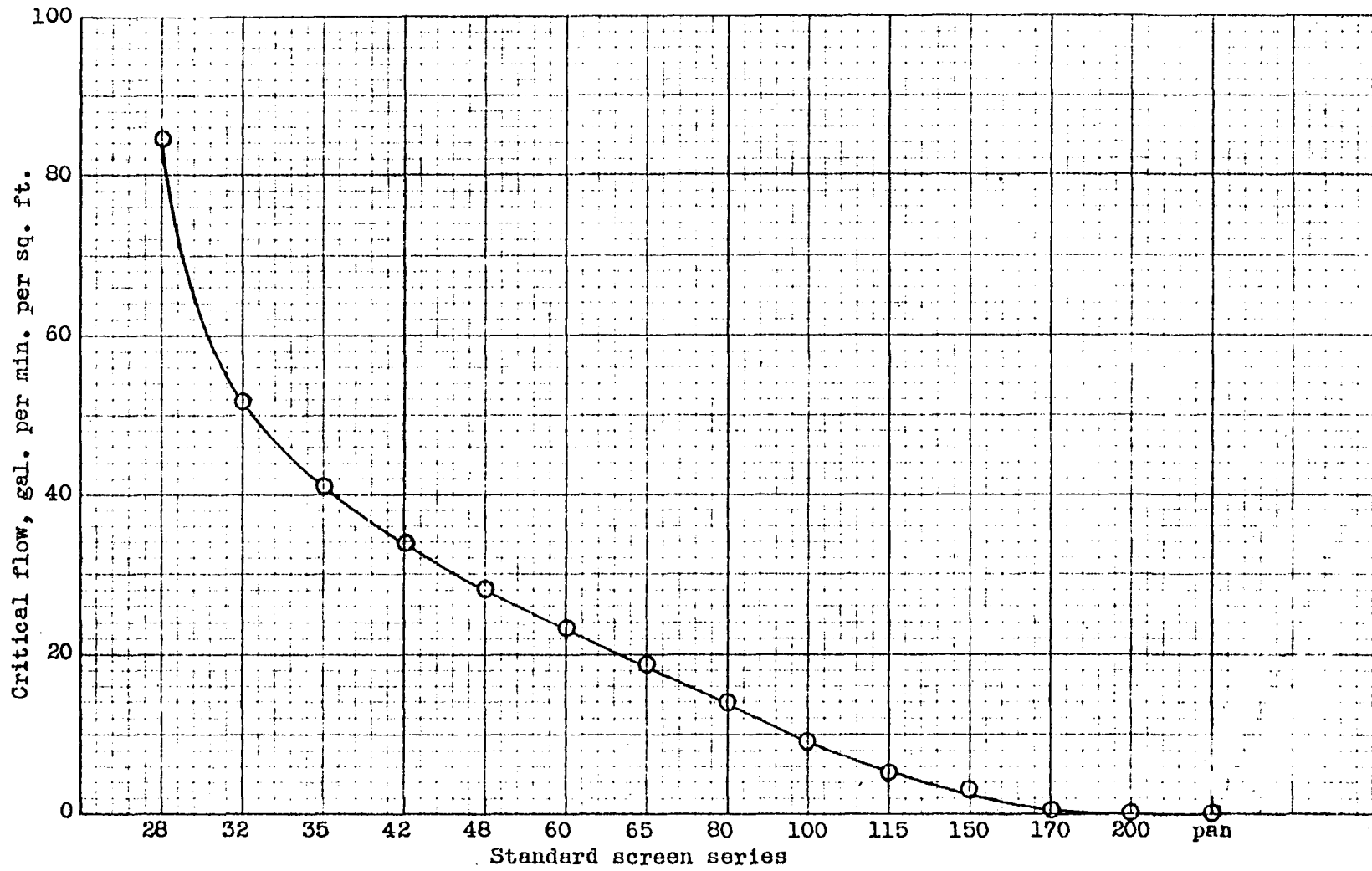
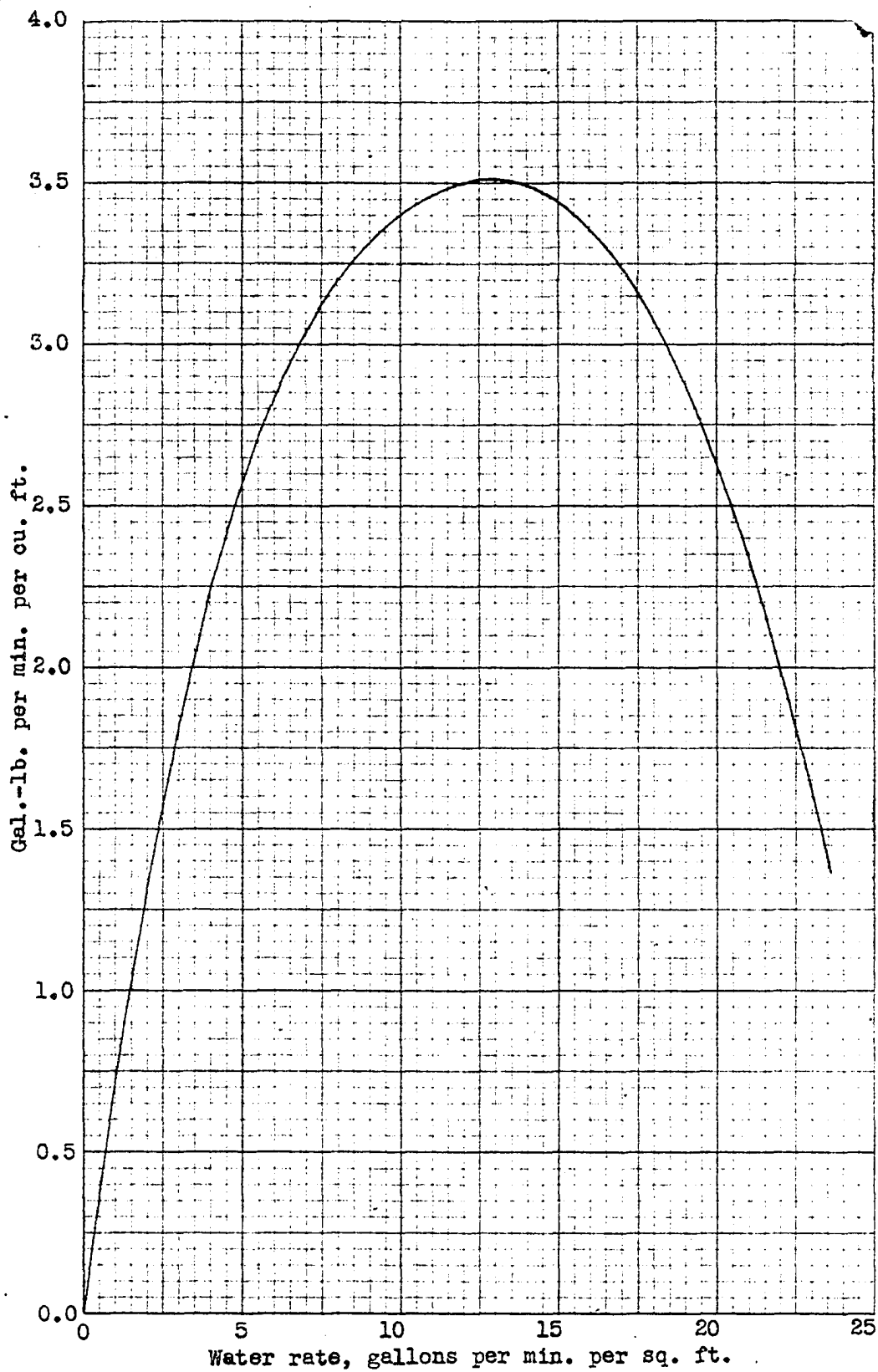


Fig. 39. Critical flow rates for various sizes of zeolite in fluidized zeolite bed.



40
Fig. 40. Optimum rate of flow in experimental fluidized zeolite bed.

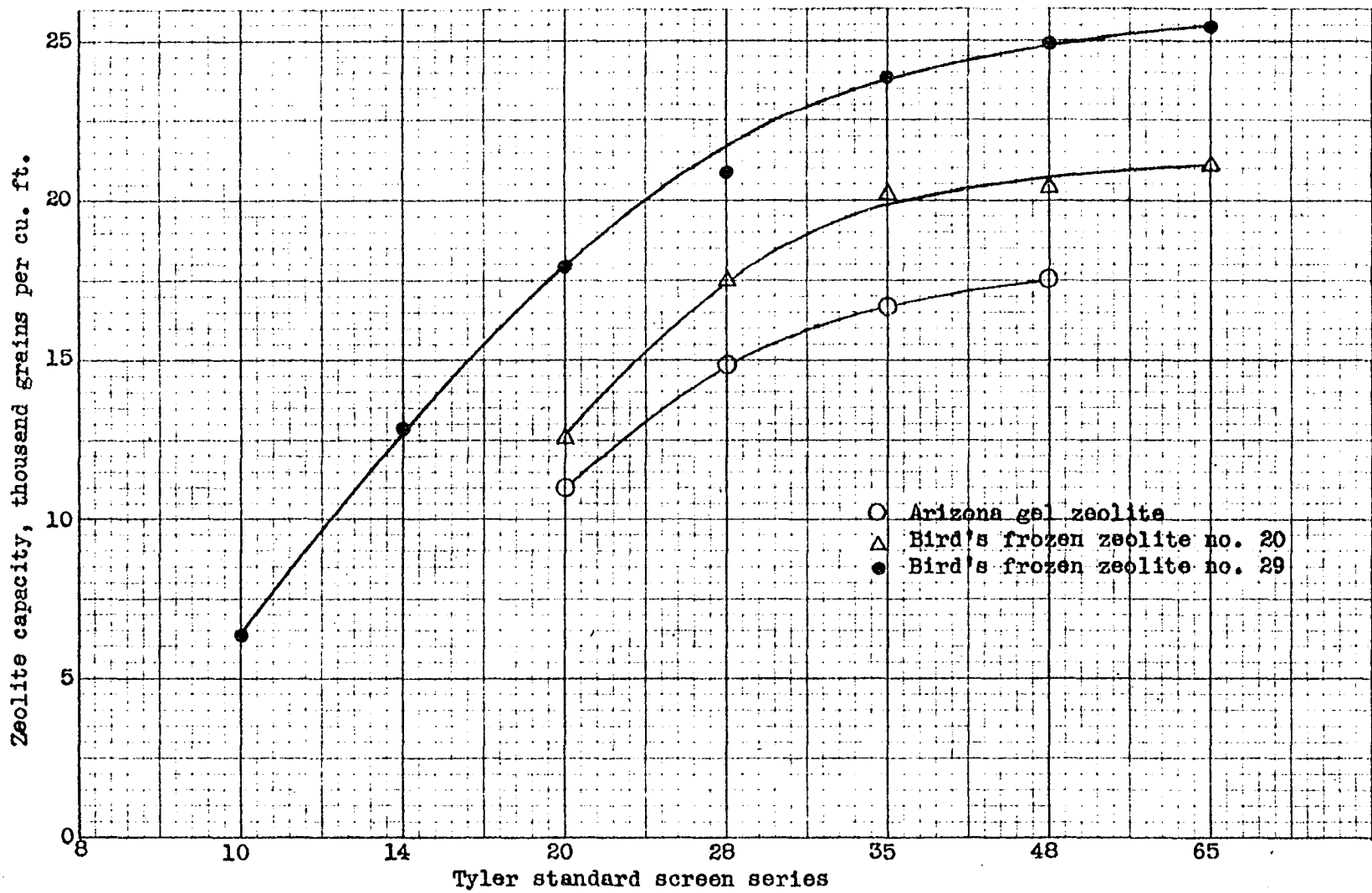


Fig. 41. Effect of zeolite size on softening capacity of various zeolites.

D. Beaded Zeolite Cation Exchanger

A preliminary study was made to determine the feasibility of producing the synthetic gel-type zeolite in a beaded form. It was believed that such a material would have better physical properties, such as resistance to mechanical attrition, and possibly higher capacity than the ordinary granular zeolite exchanger.

1. General preparation procedure

It was believed that the principles involved in making beaded catalyst for the FCC Process for cracking petroleum oils (301) could be utilized in making a beaded aluminosilicate zeolite cation exchanger. Accordingly, the following operations were involved in this preparation:

- a. Prepare a gel from sodium aluminate and sodium silicate.
- b. Transfer the gel before, during, or after initial setting to an inert medium to form spherical droplets of the soft gel.
- c. Provide sufficient time in the inert medium for the gel to become stiff.
- d. Remove soft beads from inert medium and dry.
- e. Temper dried beads to relieve strains set up in the drying operation.

2. Experimental preparation of exchanger

The gel was prepared by mixing equal portions of 0.247 molar sodium aluminate and 1.48 molar sodium silicate solutions. This resulted in a gel with a six to one $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio and a setting time of 90 to 100

seconds.

Various inert media were used, including petroleum ether, distillate No. 2, a heavy mineral oil, glycerin, and alcohol. The glycerin proved to be the best for laboratory scale work.

Although several procedures were found to be successful in preparing the soft beads of gel, the most convenient method for the preparation of small batches was as follows: A standard one inch pyrex pipe, two feet in length, was filled with the mineral oil to within a few inches of the top (held in a vertical position and rubber-stoppered at the bottom). The sodium aluminate and sodium silicate solutions were quickly mixed in a beaker and then added dropwise to the mineral oil in the pyrex pipe. Approximately 20 ml. of reactants could be added before the first beads reached the bottom of the pipe. The top of the pipe was then stoppered and turned end over end to keep the beads in suspension until sufficient time was allowed for setting into a stiff gel. The suspended beads were then poured out into flat enameled pans, after which they were separated from the oil by filtration through coarse filter paper by gravity.

Modifications in procedure gave beads of different physical properties. Typical examples are as follows:

- a. Preheating the mineral oil to 70°C. gave a faster setting time in the inert medium, and resulted in better beads. Preheating the reactants was not practical for laboratory work, because it gave insufficient time for thoroughly mixing reactants before initial setting time.

- b. The size of the beads varied with the rate of pouring reactants into inert medium. It was found that beads with an initial diameter of approximately one-eighth inch were preferable.

Several methods of drying the beaded exchanger were investigated.

The drying operation was a very critical step, because of the large decrease in volume as the gel, containing over 90 per cent water initially, was dehydrated. It is believed that many strains are set up during this operation. Methods of drying included the following:

- a. Air drying gave some beads with apparently good physical properties, but a high percentage of the beads cracked during the operation.
- b. A single bead was dried in a vacuum over a water atmosphere until it reduced in size from one-half to one-eighth inch diameter. Further drying in the air caused it to crack. The undried core had a white, translucent color, whereas the outer part (two-thirds of the distance to the center) was perfectly transparent and very hard.
- c. Batches were dried in an insulated, electrically heated box, in which a high humidity was maintained by the use of a saturated solution of ammonium sulfate. Drying temperatures ranging from room temperature to 150°C. were satisfactory, and gave the desired slow drying.
- d. Beads, with oil still on the surface, were placed in ethyl alcohol to remove water and possibly the mineral oil also. The beads became more clear in color and higher in strength within

a few minutes. However, the resulting beads could not be air dried, because the alcohol evaporated so rapidly that the beads cracked in the operation.

The beaded product was transparent for sizes of 1/32 inch or smaller in diameter. The larger beads were translucent, indicating incomplete drying in the core.

3. Results

Satisfactory beaded form for the alumino-silicate zeolite exchanger was obtained by several procedures. However, very slow rates of drying under controlled conditions were necessary.

Since small batches of only a few grams each were produced in these preliminary studies, the static tests on softening capacity were inconclusive. Running them side by side with a Doucil sample (a granular synthetic gel zeolite), their exchange capacities were approximately 10 per cent lower than that of Doucil. However, this was very encouraging, considering the fact that some residual mineral oil was apparently on the beaded samples. There is every reason to expect that further work on these beaded materials will result in higher softening capacities, in addition to better physical properties, than possessed by the standard granular zeolite. Although the inorganic zeolite exchanger has lost much of its importance because of the acid-resistant resins, nevertheless, it maintains a position in the water softening field.

Production of a beaded exchanger on a commercial scale in continuous operation should be simpler than batchwise in the laboratory. Better

control of the dry conditions in a continuous system is anticipated. Likewise, it should be noted that the setting time can be controlled by the concentrations of the reactants. Whereas a setting time of 90 to 100 seconds is required for laboratory manipulation, a large scale, continuous plant could operate on a setting time of a few seconds. This short period of time can be attained by temperature and concentration adjustments.

E. Process for Copper Production

The economics of using ion exchange resins in most metallurgical processes can be questioned. However, a process has been developed for copper production, which merits serious consideration. It consists essentially of three steps, namely, (1) leaching of ore with dilute sulfurous acid, (2) concentrating and separating from impurities by means of cation exchange resin, and (3) acid recycling from electrolytic cells for use in eluting copper from the exchanger beds.

1. Leaching of ore

Although there may be many types of ores that could be used in this process, this study was limited to a consideration of the low grade oxidized copper ores, which contain one per cent or less of copper. There is considerable data available on the leaching operations of these ores. The following information was obtained from Booth Engineers, Salt Lake City, (49):

The information I have available may not be sufficient for your requirements because of the fact that my method of leaching the copper ore is probably not the most desirable leaching method when the solutions are to be used with Cation Exchange Materials or electrolytic precipitation. Practically all my leaching experiments have been done on -20 mesh material. It is well known that this particular ore can be leached with weak sulphuric acid solution at $3/8$ inch crushing. I have some information on work of this sort done by others on the deslimed $3/16$ inch material and some solution analysis under these conditions. Attached to this letter is some information utilizing this type of treatment and showing analysis of solutions resulting from the treatment of -65 mesh material and $3/6$ inch /65 mesh. It is possible that you could arrive at a synthetic solution made up in accordance with the solution analysis as shown on this work. Since the fine ore represented 10.5% of the original ore, the solution resulting from the fine ore treatment should bear this relationship to the solution resulting from the deslimed coarse ore.

In the work that I have done on this ore, complete solution analysis were not obtained, but in acid treating -20 mesh ore agitated for four hrs, 80.2% of the copper was dissolved and the resulting solutions contained 0.405 grams Al_2O_3 per liter and 0.36 grams Fe per liter, the leaching was done in a 3 to 1 pulp and by calculation the solution should have contained 2.7 grams copper per liter. This was a bottle agitation leaching test and the leaching solution at the start contained 26.6 lbs H_2SO_4 per ton of solution. At the end of the treatment the solution titrated 10.0 lbs. H_2SO_4 so that 49.8 lbs. H_2SO_4 were consumed per ton of ore treated.

Any process of practical consideration on this ore would have to provide for low acid consumption, possibly a net acid consumption of from 18 to 25 lbs of H_2SO_4 per ton of ore treated would be of interest.

Coarse Ore Leaching Preceded by Desliming - For these tests, the $1/2$ " material was crushed to pass a $3/16$ " screen. The minus -65- mesh material was screened out, somewhat duplicating the desliming operation which generally precedes coarse ore leaching. A screen-assay analysis follows:

<u>Size of Material</u>	<u>Weight %</u>	<u>Copper %</u>	<u>Total Cu %</u>
$3/16$ " -65 mesh	89.05	0.67	81.5
Minus 65 mesh	10.95	1.23	18.5

The plus-65-mesh material was leached with 4.40 percent H_2SO_4 , and the results are in Table 1.

TABLE 1 - LEACHING OF OXIDE COARSE ORE WITH DILUTE SULFURIC ACID AFTER DESLIMING

	<u>Aerated</u>	<u>Not Aerated</u>
Leaching Time	1 day	1 day
Lbs. H_2SO_4 consumed/ton of ore	25.72	27.64
Lbs. H_2SO_4 /lb. Cu in solution	2.43	2.71
Total Cu in solution-Percent	80.64	77.59

Leaching Time	2 days	2 days
Lbs. H_2SO_4 /ton of ore	29.60	31.52
Lbs. H_2SO_4 /lb. Cu in solution	2.85	3.00
Total Cu in solution-Percent	79.12	80.03

Leaching time	3 days	3 days
Lbs. H_2SO_4 consumed/ton of ore	25.33	30.36
Lbs. H_2SO_4 /lb. Cu. in solution	2.27	2.86
Total Cu in solution-Percent	84.90	80.64

A screen-assay analysis of the tailings from the three-day coarse ore aerated leaching test is given in Table 2. The high Cu content of the minus-100-mesh material is probably due to poor washing.

TABLE 2 - SCREEN*ASSAY ANALYSIS OF COARSE ORE TAILINGS AFTER LEACHING WITH DILUTE SULPHURIC ACID

<u>Size Product</u>	<u>Weight-Percent</u>		<u>Cu Percent</u>	<u>Total Cu Percent</u>
	<u>Direct</u>	<u>Cumulative</u>		
3/16" - 10 mesh	70.0	70.0	0.10	61.0
10 - 20 mesh	14.3	84.3	0.12	14.8
20 - 100 mesh	9.6	93.9	0.10	8.4
Minus 100 mesh	6.1	100.0	0.30	15.8

Low H_2SO_4 consumption in coarse ore leaching is due to low iron solution as shown again by comparison of leach solution analyses in Table 3.

TABLE 3 - COMPARISON OF LEACH SOLUTION ANALYSES WITH
COARSE ORE AND FINE ORE LEACHING.

	Fine Ore	Deslimed Coarse Ore, Leached 3 Days with Aeration
Leach solution H ₂ SO ₄ -Percent	4.52	4.40
Total Cu in solution-Percent	82.33	84.90
H ₂ SO ₄ consumption Lbs./ton of Ore by titration method	60.80	25.33
H ₂ SO ₄ consumption Lbs./Lb. Cu In solution	4.83	2.27
Cu in solution-Percent	0.315	0.279
CaO in solution-Percent	0.070	0.050
Al ₂ O ₃ in solution-Percent	0.104	0.100
Iron in solution-Percent	0.640	0.070

An ore sample also was obtained from Booth Engineers. It was crushed and screened to -20 + 55 mesh. A series of samples were leached with sulfuric acid ranging in concentrations from 0.75 to 2.00 per cent by weight. The leaching device consisted of a rack with bottles turned by a small electric motor with a reducer to give a speed of approximately 20 r.p.m. The leaching periods ranged from one to three days. The longer leaching periods and higher acid concentrations leached out slightly higher percentages of the copper. The pH of the final leach solutions are given in Figure 43. These values were higher than those reported in the literature, which may be due to the buffering action of carbonates from the ore. This ore sample contained on a weight basis the following:

Au	Ag	Total Cu	Ox. Cu	Fe	CaO	MgO	S
0.003	0.02	1.01	0.81	2.9	0.28	0.51	0.66

It was found that excellent leaching of this ore could also be done with sulfurous acid. A dilute sulfurous acid solution of 0.233 N was made by bubbling cylinder SO₂ through water. A 60 gram ore sample

was leached for five hours with 130 ml. of the 0.253 N sulfurous acid. The leach solution analyzed 3040 ppm copper (0.304 per cent), and gave a 2.6 pH reading. Thus, slightly over 65 per cent of the copper in the ore was leached out with this dilute acid.

2. Concentration and purification by cation exchange

A synthetic leach effluent, containing 0.30 per cent copper, was passed through a bed of H-Dow MX(Super) cation exchange resin. When the effluent showed a color change from colorless to faint blue the resin was assumed exhausted. The absorbed copper was eluted from the resin with a 10 per cent sulfuric acid solution. The major portion of the original copper was concentrated in the effluent as approximately 8 per cent copper sulfate. Hence, the copper concentration was increased nearly twenty-seven fold over the original concentration of the synthetic leach solution.

The main impurities leached out with the copper from the ore are magnesium, iron and aluminum. There is the possibility that ion exchange can separate a considerable amount of these impurities from copper. This can be shown more clearly after the data in the following sections on interfering ions, equilibrium and column distribution of cations have been considered.

3. Tolerance for interfering ions

A study was made to determine the concentration of impurities (interfering ions) that could be tolerated in a copper leach solution, without materially reducing the exchanger capacity for copper. A series of synthetic test solutions were made up, using mixtures of copper and

iron, copper and magnesium, and copper and aluminum. The concentration of copper was held constant at 2 per cent by weight in these three series, with varying concentrations of interfering ions. These test solutions were added by means of a burette to a 10 ml. sample of H-Dow MX(Super), which was agitated in a water solution by an electric stirrer. A period of time was allowed after each addition for exchange to reach equilibrium. An indicator of potassium ferro-cyanide was used to determine the presence of any residual copper in the solution. When copper was indicated in the solution after a ten minute period of continuous stirring without test additions, the run was terminated. Runs 48, 49, and 50, together with Tables 73 through 78, list the experimental data and calculated results for these studies.

In general, aluminum causes the greatest magnesium intermediate, and ferrous iron the least interference, upon the exchange capacity for copper alone. As shown on Figure 45, the amount of interference levels off at the higher percentages of interfering ions. The interference by magnesium becomes practically constant for 10 per cent or higher magnesium. Aluminum interference levels off at 20 per cent, whereas ferrous iron appears to level off at 50 per cent, if at all. The percentages of interfering ions are better correlated on a mol basis, as shown on Figure 46.

It is interesting to note that at high percentages of interfering ions, aluminum is definitely the most troublesome. However, at low percentages, it becomes the least troublesome. The transition point for this phenomenon occurs at approximately 21 mol per cent of interfering ion.

The effects from all three ions are about equal at this point.

4. Copper equilibrium studies

In order to learn more concerning the properties of copper in relation to ion exchange, an equilibrium study was made. The agitation by tumbling of samples, as described for the nickel equilibrium studies, was used to establish equilibrium of the copper chloride solution and Na-Geo-Karb.

After equilibrium had been established, the effluent solution was analyzed for copper colorimetrically. A method based on the formation of the cuprammonium complex was used. The copper chloride equilibrium solution sample was measured into a clean, dry beaker and an equal portion of 1.5 N ammonium hydroxide solution was added. This provided an excess of ammonium ions over that complexed with the copper present at these low concentrations. Photometer readings were taken, and used to obtain the copper concentration in ppm from a calibration curve. A typical calibration curve for this method is shown on Figure 8.

As in the nickel equilibrium studies, a straight correlation was obtained by plotting the copper to sodium ratio in solution against the copper to sodium ratio in the solid exchanger on log-log paper. Likewise, the correlation could be made on a basis of active molar, molar, or weight ratios with equal facility. These correlations are shown on Figure 42. Again, as in the case of nickel, the exchanger has a greater affinity for the copper than for the sodium. This is shown by the fact that the slope of the straight line plot for the active molar ratios is 1.21, which equals the power p of the copper to sodium ratio in the solution. Thus, in com-

parison with Walton's reported values for the alkali metal and alkaline earth cations, it can be seen that the exchangers have a much greater affinity for the copper. In comparison with nickel, using the power p as an index of exchanger affinity for a given cation, it is evident that nickel would be held more strongly than copper by a cation exchange resin.

5. Distribution of copper and calcium in exchanger bed

Data were obtained on the distribution of copper and calcium in an Amberlite IR-100 tower bed, as shown in Tables 81 and 82. Equimolar concentrations of copper chloride and calcium chloride (or 63.6 per cent copper and 36.4 per cent calcium on a weight basis of the cations) were fed to the column. It can be seen graphically in Figure 49 that the copper was distributed down the bed, ranging from 10.6 at the top to 0.8 mg. copper per cc. resin at the bottom. It should be noted that the average concentration for each of five bed sections has been plotted. Hence, a differential portion of the bed at the top should contain as much as 12 mg. per cc., and at the bottom very little, if any, copper. This latter point is verified by the fact that no trace of copper was detected in the effluent during the column run.

The copper has concentrated in the top of the tower as approximately 80 to 85% of the total cations by weight. On an equivalent basis, Table 82 indicates that the copper has concentrated from an original 1:1 ratio with the calcium to a 3.08 ratio when the column was shut down. Likewise, the calcium has concentrated in the bottom of the tower to a 7.18 equivalent ratio of calcium to copper.

6. Discussion on proposed process for copper production

The unique advantages and possibilities of this copper production process are apparent after a consideration of the data presented in this section. These points can be summarized as follows:

- a. The acid consumption is not as critical as for the standard sulfuric acid leaching process, due to the use of cheap sulfurous acid. Such acid could be made available with a minimum of equipment at sulfite smelting plants.
- b. Leaching with dilute acid is no disadvantage in a cation exchange process, because the final solution concentration is dependent upon elution techniques rather than original concentration. On the other hand, there is a lower economical limit for acid concentration in the standard leaching processes, since costly evaporation must be used to remove the water from the dilute acid.
- c. Smaller amounts of impurities are leached out by the use of weaker acids, which give less trouble in the purification steps for electrolytic refining.
- d. There are many indications that cation exchange upon a copper solution, containing magnesium, iron, and aluminum impurities, will effect a further purification of the copper. Equilibrium and column studies both support this point.
- e. Cation exchange concentrates the copper leach effluent such that no evaporation is required, regardless of the strength of leaching acid.

f. Recycle acid from electrolytic cells, even with copper in it, can be used for elution of the exchanger to give an even stronger copper makeup solution.

It must be emphasized, however, that many points must be tested experimentally before this process can be considered practical. These include such items as exchanger life, residual copper on exchanger beds, more detailed study on the effects of impurities, elution techniques, and corrosion of sulfurous acid and sulfite solutions.

Table 73. Interference by magnesium upon exchange capacity for copper (Run 48)

Test No.	Resin sample, ml. ^a	Test solution ^b				Resin capacity ^c Vol. soln., ml.
		Cu/Mg ratio		Magnesium content		
		Weight	Molar	Wt. %	Mol %	
1	10.0	0.31	0.1175	76.32	89.40	14.0
2	10.0	4.00	1.517	20.00	39.75	14.0
3	10.0	5.00	1.896	16.67	34.54	14.0
4	10.0	6.00	2.275	14.28	30.52	14.0
5	10.0	7.00	2.654	12.50	27.35	14.0
6	10.0	8.00	3.034	11.11	24.78	14.0
7	10.0	9.00	3.414	10.00	22.67	14.2
8	10.0	10.00	3.792	9.09	20.87	16.5
9	10.0	20.00	7.584	4.76	11.64	20.0
10	10.0	40.00	15.170	1.60	6.18	20.0
11	10.0	100.00	37.920	0.99	2.57	20.3

^aExchange resin volume measured under water in a graduated cylinder.

^bCopper concentration held constant at 2.00 per cent by weight throughout run.

^cBased on "break-through" point of detecting copper in solution (after stirring 10 minutes) by use of potassium ferrocyanide test.

Table 74. Interference by iron upon exchange capacity for copper (Run 49)

Test No.	Resin sample, ml. ^a	Test solution ^b				Resin capacity ^c Vol. soln., ml.
		Cu/Fe ratio		Iron content		
		Weight	Molar	Wt. %	Mol %	
1	10.0	1.0	0.878	50.00	53.25	15.0
2	10.0	5.0	4.390	16.67	18.54	20.5
3	10.0	20.0	17.57	4.76	5.38	26.0
4	10.0	25.0	21.95	3.85	4.36	29.5
5	10.0	30.0	26.34	3.23	3.66	27.0
6	10.0	40.0	35.14	1.60	2.77	29.5

^aExchange resin volume measured under water in a graduated cylinder.

^bCopper concentration held constant at 2.00 per cent by weight throughout run.

^cBased on "break-through" point of detecting copper in solution (after stirring 10 minutes) by use of potassium ferrocyanide test.

Table 75. Interference by aluminum upon exchange capacity for copper (Run 50)

Test No.	Resin sample, ml. ^a	Test solution ^b				Resin capacity ^c Vol. soln., ml.
		Cu/Al ratio		Aluminum content		
		Weight	Molar	Wt. %	Vol. %	
1	10.0	1.0	0.424	30.00	70.22	6.6
2	10.0	4.0	1.697	30.00	27.07	7.8
3	10.0	15.0	6.39	6.25	15.59	20.0
4	10.0	20.0	8.48	4.76	10.54	22.5
5	10.0	25.0	10.62	3.85	8.61	24.5
6	10.0	30.0	12.73	3.25	7.26	25.5

^aExchange resin volume measured under water in a graduated cylinder.

^bCopper concentration held constant at 2.00 per cent by weight throughout run.

^cBased on "break-through" point of detecting copper in solution (after stirring 10 minutes) by use of potassium ferrocyanide test.

Table 76. Calculated results for interference by magnesium upon exchange capacity for copper (Run 48)

Test No.	Weight exchange		Molar exchange		Equivalent exchange		Total exchange, Cu ⁺⁺ + Mg ⁺⁺ , meq.
	Cu ⁺⁺ , g.	Mg ⁺⁺ , g.	Cu ⁺⁺ , millimols	Mg ⁺⁺ , millimols	Cu ⁺⁺ , meq.	Mg ⁺⁺ , meq.	
1	0.2800	0.9020	4.405	37.090	8.810	74.180	82.990
2	0.2800	0.0700	4.405	2.881	8.810	5.762	14.572
3	0.2800	0.0561	4.405	2.307	8.810	4.614	13.424
4	0.2800	0.0466	4.405	1.915	8.810	3.830	12.640
5	0.2800	0.0400	4.405	1.645	8.810	3.290	12.100
6	0.2800	0.0351	4.405	1.443	8.810	2.886	11.696
7	0.2840	0.0315	4.470	1.296	8.940	2.592	11.532
8	0.3300	0.0328	5.190	1.348	10.380	2.696	13.076
9	0.4000	0.0200	6.285	0.823	12.570	1.646	14.216
10	0.4000	0.0070	6.285	0.288	12.570	0.576	13.146
11	0.4060	0.0030	6.380	0.123	12.760	0.246	13.006

Table 77. Calculated results for interference by iron upon exchange capacity for copper (Run 49)

Test No.	Weight exchange		Molar exchange		Equivalent exchange		Total exchange, Cu ⁺⁺ + Fe ⁺⁺ , meq.
	Cu ⁺⁺ , g.	Fe ⁺⁺ , g.	Cu ⁺⁺ , millimols	Fe ⁺⁺ , millimols	Cu ⁺⁺ , meq.	Fe ⁺⁺ , meq.	
1	0.3000	0.3000	4.723	5.370	9.446	10.740	20.186
2	0.4100	0.0820	6.452	1.469	12.904	2.938	15.842
3	0.5200	0.0260	8.180	0.466	16.360	0.932	17.292
4	0.5900	0.0240	9.280	0.429	18.560	0.858	19.418
5	0.5400	0.0178	8.495	0.319	16.990	0.638	17.628
6	0.5900	0.0090	9.280	0.161	18.560	0.322	18.882

Table 78. Calculated results for interference by aluminum upon exchange capacity for copper (Run 50)

Test No.	Weight exchange		Molar exchange		Equivalent exchange		Total exchange, Cu ⁺⁺ + Al ⁺⁺⁺ , meq.
	Cu ⁺⁺ , g.	Al ⁺⁺⁺ , g.	Cu ⁺⁺ , millimols	Fe ⁺⁺⁺ , millimols	Cu ⁺⁺ , meq.	Fe ⁺⁺⁺ , meq.	
1	0.1320	0.1320	2.075	4.890	4.150	14.670	18.820
2	0.1560	0.0390	2.454	1.444	4.908	4.332	9.240
3	0.4000	0.0270	6.285	1.000	12.570	3.000	15.570
4	0.4700	0.0230	7.390	0.852	14.780	2.556	17.336
5	0.4900	0.0200	7.715	0.741	15.430	2.223	17.653
6	0.5100	0.0170	8.024	0.630	16.048	1.890	17.938

Table 79. Equilibrium of copper chloride solution with Na-Zeo-Karb (Run 51)^a

Test No.	Weight of Na-Zeo-Karb g. ^b	Vol. of CuCl ₂ solution, ml. ^c	Before exchange		At exchange equilibrium ^d			
			Na in exchanger, meq.	Cu ⁺⁺ in solution, meq.	In exchanger		In solution	
					Na, meq.	Cu, meq.	Na ⁺ , meq.	Cu ⁺⁺ , meq. ^e
1	0.500	100.0	0.65	2.048	0.081	0.569	0.569	1.479
2	1.000	100.0	1.30	2.048	0.297	1.003	1.003	1.045
3	2.000	100.0	2.60	2.048	1.010	1.590	1.590	0.458
4	4.000	100.0	5.20	2.048	3.267	1.933	1.933	0.115
5	6.000	100.0	7.80	2.048	5.818	1.982	1.982	0.066

^aSamples tumbled in 250 ml. stoppered bottle for 20 minutes.

^bNa-Zeo-Karb contained 0.0299 grams exchangeable sodium per gram dry exchanger.

^cStandard CuCl₂ solution contained 651 p.p.m. of copper.

^dEquivalent exchange assumed between sodium and copper.

^eCopper in solution determined colorimetrically by cupraammonium complex method.

Table 80. Equilibrium correlation of copper chloride solution with Na-Zeo-Karb (Run 51)

Test No.	Activity coefficients		Weight ratios		Molar ratios		Active molar ratios	
	Cu ⁺⁺	Na ⁺	In exchanger	In solution	In exchanger	In solution	In exchanger ^b	In solution
			Cu/Na	Cu ⁺⁺ /Na ⁺	Cu/Na	Cu ⁺⁺ /Na ⁺	Cu/(Na) ²	^a Cu ⁺⁺ / ^a Na ⁺
1	0.740	0.853	9.73	3.590	3.520	1.300	43.30	231.3
2	0.746	0.858	4.66	1.444	1.688	0.522	5.68	52.7
3	0.756	0.864	2.18	0.398	0.787	0.144	0.781	9.17
4	0.763	0.869	0.82	0.082	0.296	0.030	0.0905	1.55
5	0.763	0.870	0.47	0.046	0.170	0.017	0.0293	0.85

^aActivity coefficients calculated by Debye-Huckel equation, with correction for ionic size.

^bRatio in exchanger solid based on total millimols of nickel and sodium.

Table 81. Removal of copper and calcium from equimolar chloride solution by tower bed of Amberlite IR-100 (Run 52)^a

Test No.	Cumulative volume of effluent, ml.	Vol. .0995 N. NaOH for 50 ml. eff., ml.	Copper in effluent, meq. ^b	Calcium in effluent	
				Soap, ml. ^c	Calcium, mg. ^d
1	100	2.30	0.0	0.40	1.10
2	200	2.79	0.0	0.40	1.10
3	300	2.75	0.0	0.30	0.63
4	400	2.75	0.0	0.30	0.63
5	500	2.75	0.0	0.20	0.0
6	600	3.00	0.0	0.20	0.0
7	700	2.65	0.0	0.20	0.0
8	817	2.80	0.0	0.20	0.0
9	927	2.79	0.0	0.20	0.0
10	1032	2.79	0.0	0.20	0.0
11	1132	2.77	0.0	0.19	0.0
12	1232	2.87	0.0	0.20	0.0
13	1332	3.58	0.0	0.20	0.0
14	1432	2.86	0.0	0.20	0.0
15	1532	3.00	0.0	0.19	0.0
16	1632	2.81	0.0	0.21	0.0
17	1732	2.85	0.0	0.20	0.0
18	1832	2.89	0.0	0.19	0.0
19	1933	2.80	0.0	0.20	0.0
20	2033	2.85	0.0	0.19	0.0

Table 81 (continued)

Test No.	Cumulative volume of effluent, ml.	Vol. .0995 N. NaOH for 50 ml. eff., ml.	Copper in effluent, meq. ^b	Calcium in effluent	
				Soap, ml. ^c	Calcium, mg. ^d
21	2141	2.89	0.0	0.19	0.0
22	2244	2.82	0.0	0.20	0.0
23	2344	2.80	0.0	0.20	0.0
24	2444	2.86	0.0	0.17	0.0
25	2544	2.80	0.0	0.17	0.0
26	2644	2.90	0.0	0.20	0.0
27	2744	2.88	0.0	0.17	0.0
28	2844	2.85	0.0	0.18	0.0
29	2954	2.93	0.0	0.16	0.0
30	3061	2.94	0.0	0.17	0.0
31	3176	2.90	0.0	0.17	0.0
32	3286	2.89	0.0	0.30	0.63
33	3386	2.86	0.0	0.32	0.68
34	3495	2.75	0.0	0.50	1.58
35	3575	2.54	0.0	0.59	2.03

^aFeed solution contained 3.40 meq. each of CuCl₂ and CaCl₂ per liter.

^bEffluent tested colorimetrically for copper.

^cStandard soap test on 50 ml. sample.

^dCalcium content in 100 ml. increment of effluent.

Table 82. Distribution of copper and calcium in Amberlite IR-100 tower bed (Run 53)^a

Section No. ^b	Height of section, in.	Vol. of section, cc.	Total vol. of HCl wash, ^c ml.	Copper distribution ^d			Calcium distribution ^e		
				p.p.m.	meq.	mg.	Soap, ml.	meq.	mg.
1	1.30	6.85	51.0	145.0	2.28	72.5	1.50	0.74	14.8
2	1.20	6.33	47.0	108.0	1.70	54.0	1.39	0.66	13.2
3	1.25	6.59	49.0	108.0	1.70	54.0	1.46	1.24	24.8
4	1.90	10.00	74.0	77.0	1.21	38.5	1.86	1.14	22.8
5	1.25	6.59	49.0	10.5	0.17	5.2	0.73	1.22	24.4
Total	6.90	36.36	270.0	---	7.06	224.2	--	5.00	100.0

^aFeed solution, containing 3.40 meq. each of CuCl_2 and CaCl_2 per liter, was passed through column until calcium appeared in effluent at 3575 ml.

^bFirst section is at top of column.

^cWash, or regeneration, HCl was 0.0902 in normality.

^dCopper determined colorimetrically in wash solution.

^eCalcium determined by soap titration, with correction for presence of CuCl_2 .

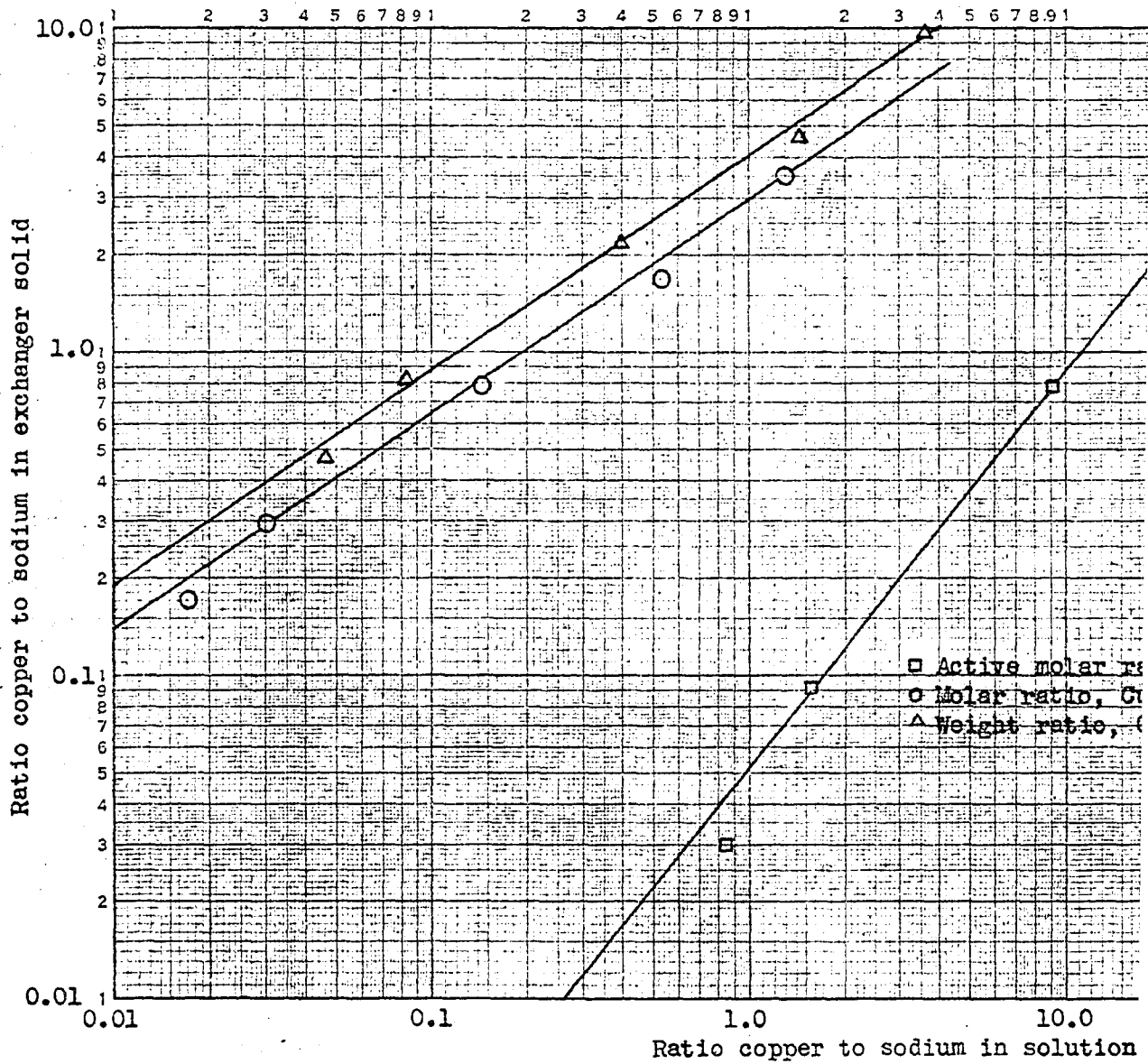
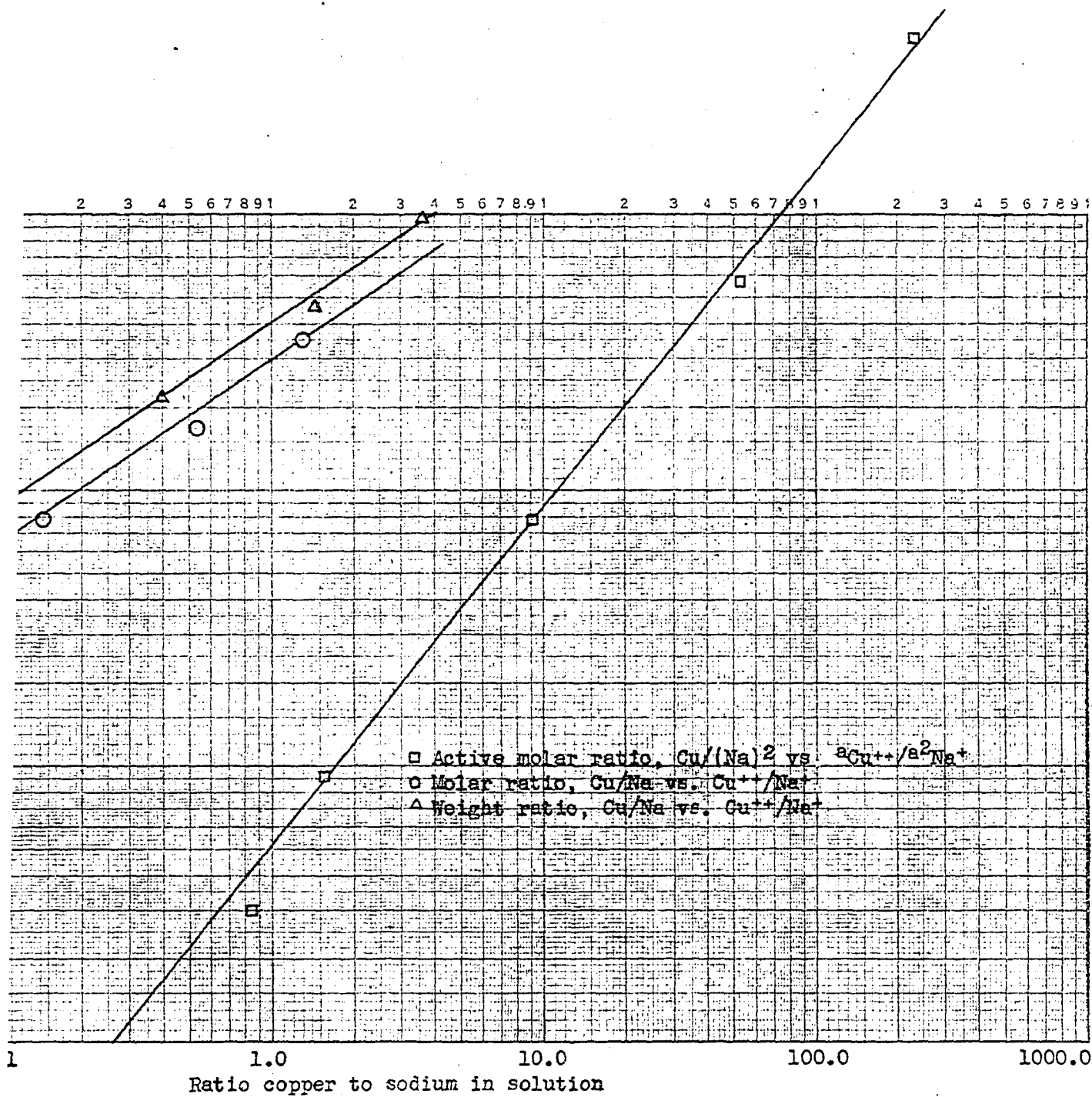


Fig. 42. Equilibrium correlation of copper chloride solution w



Equilibrium correlation of copper chloride solution with Na-Zeo-Karb.

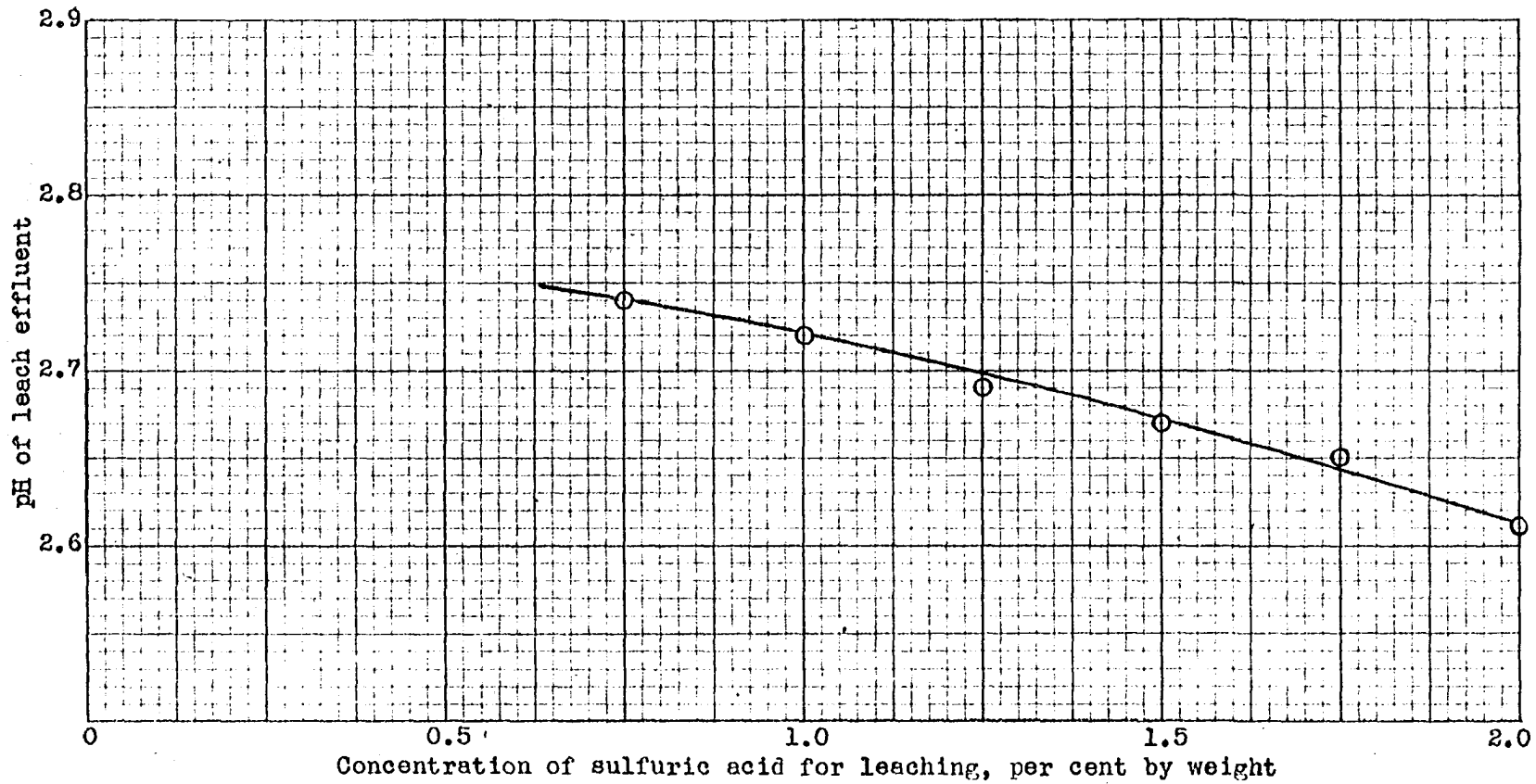


Fig. 43. Effect of sulfuric acid concentration on the leaching of copper ore.

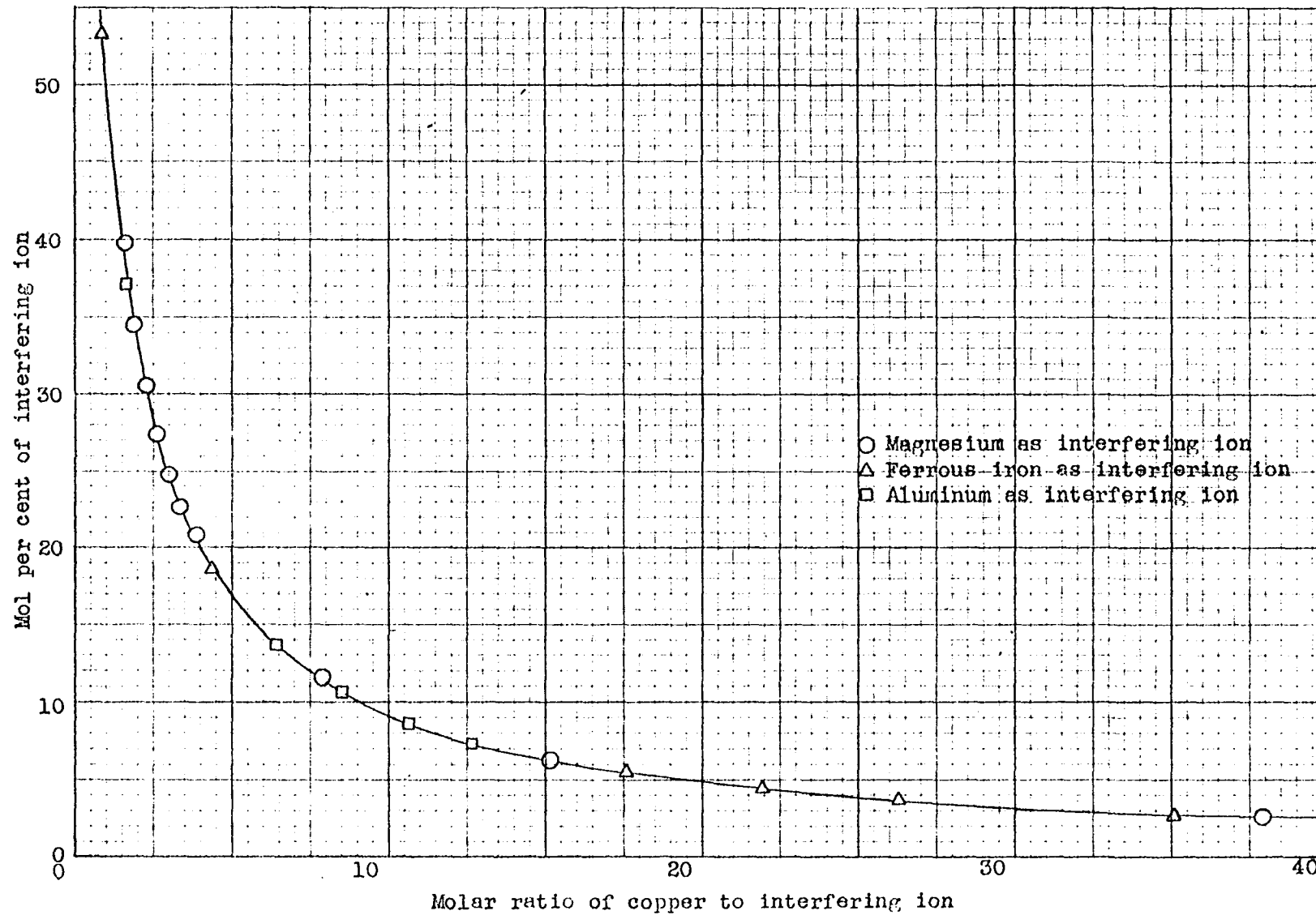


Fig. 44. Relationship between interfering ion and its ratio to copper.

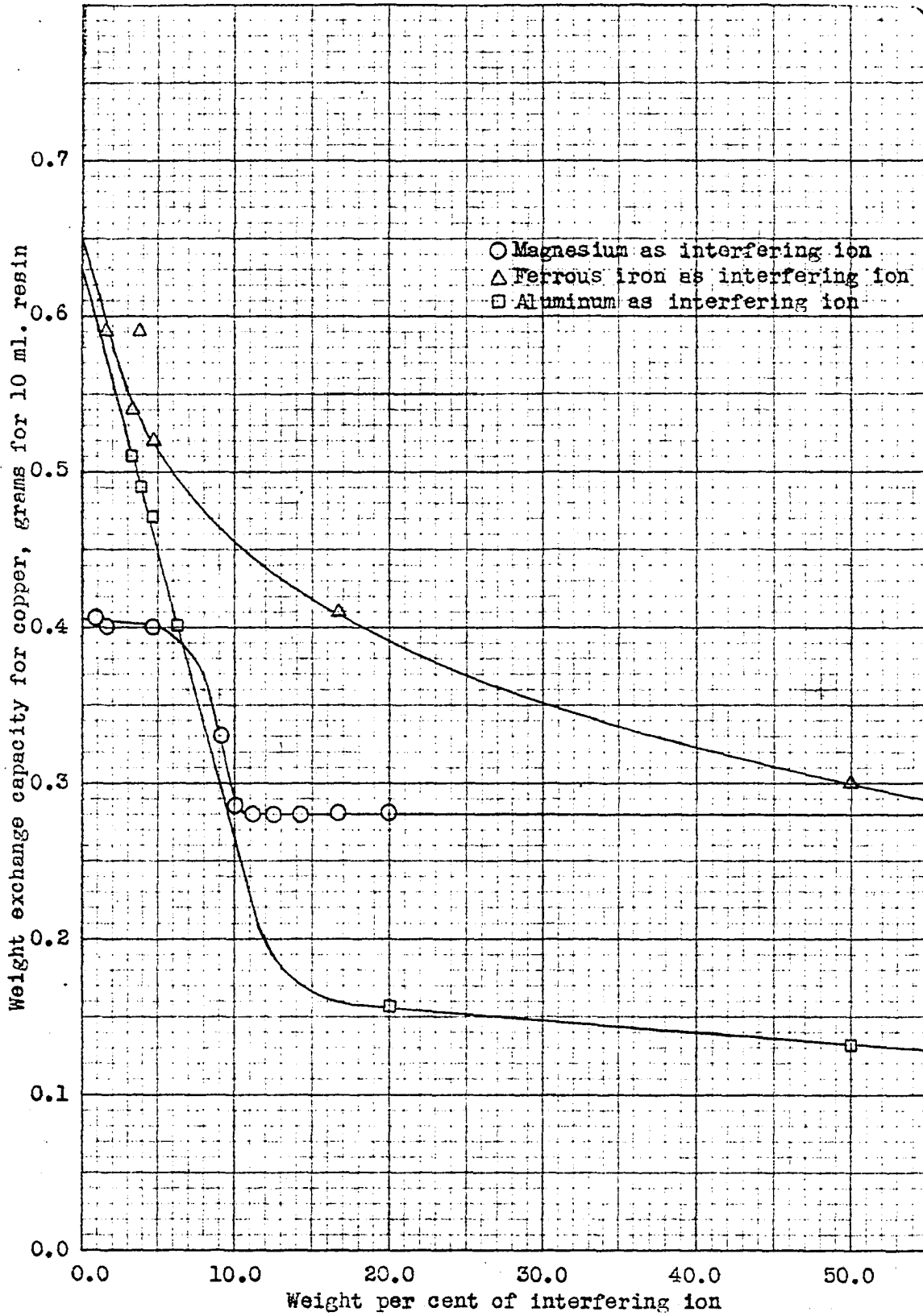


Fig. 45. Effect of interfering ions on weight exchange capacity for copper.

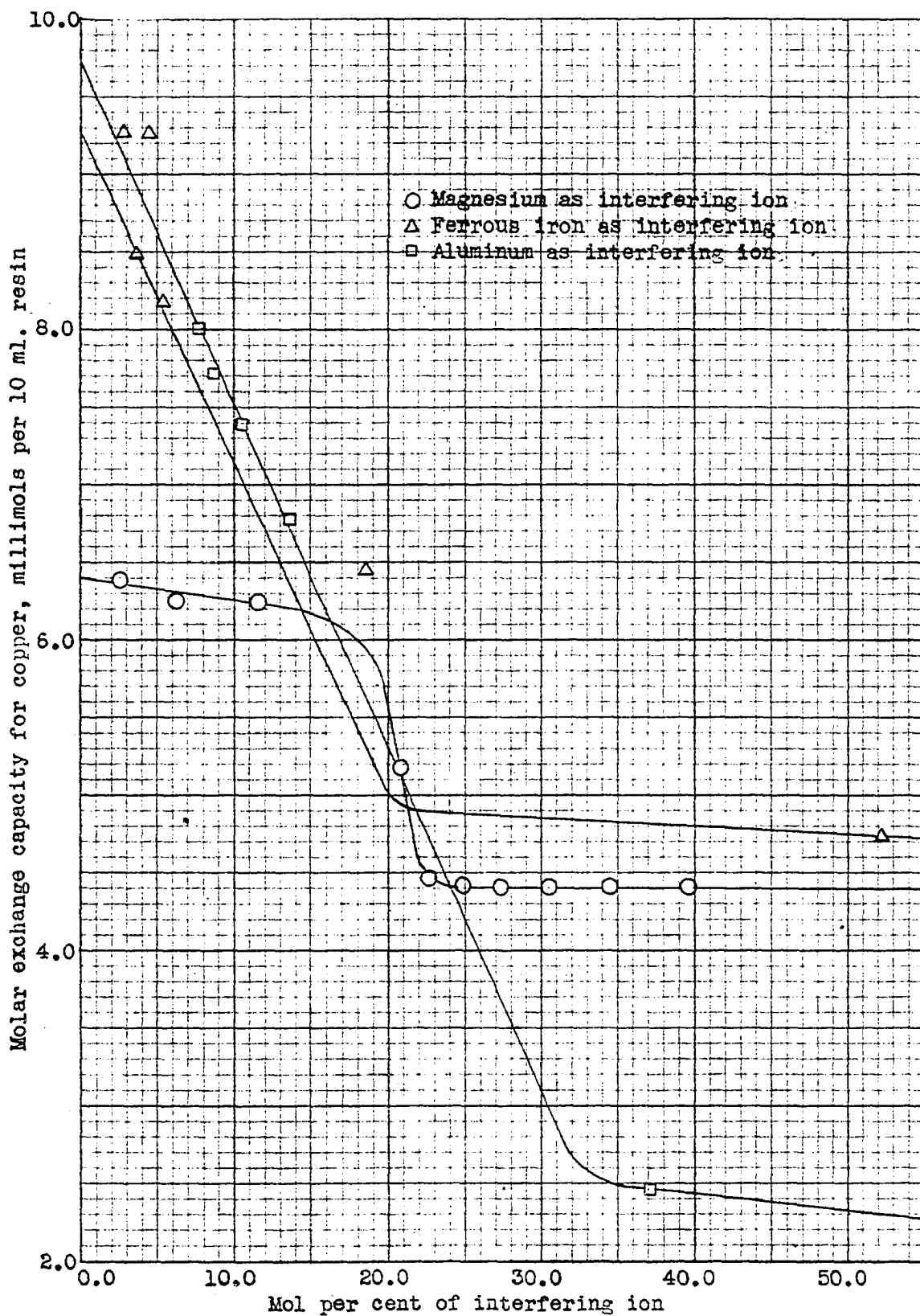


Fig. 46. Effect of interfering ions on molar exchange capacity for copper.

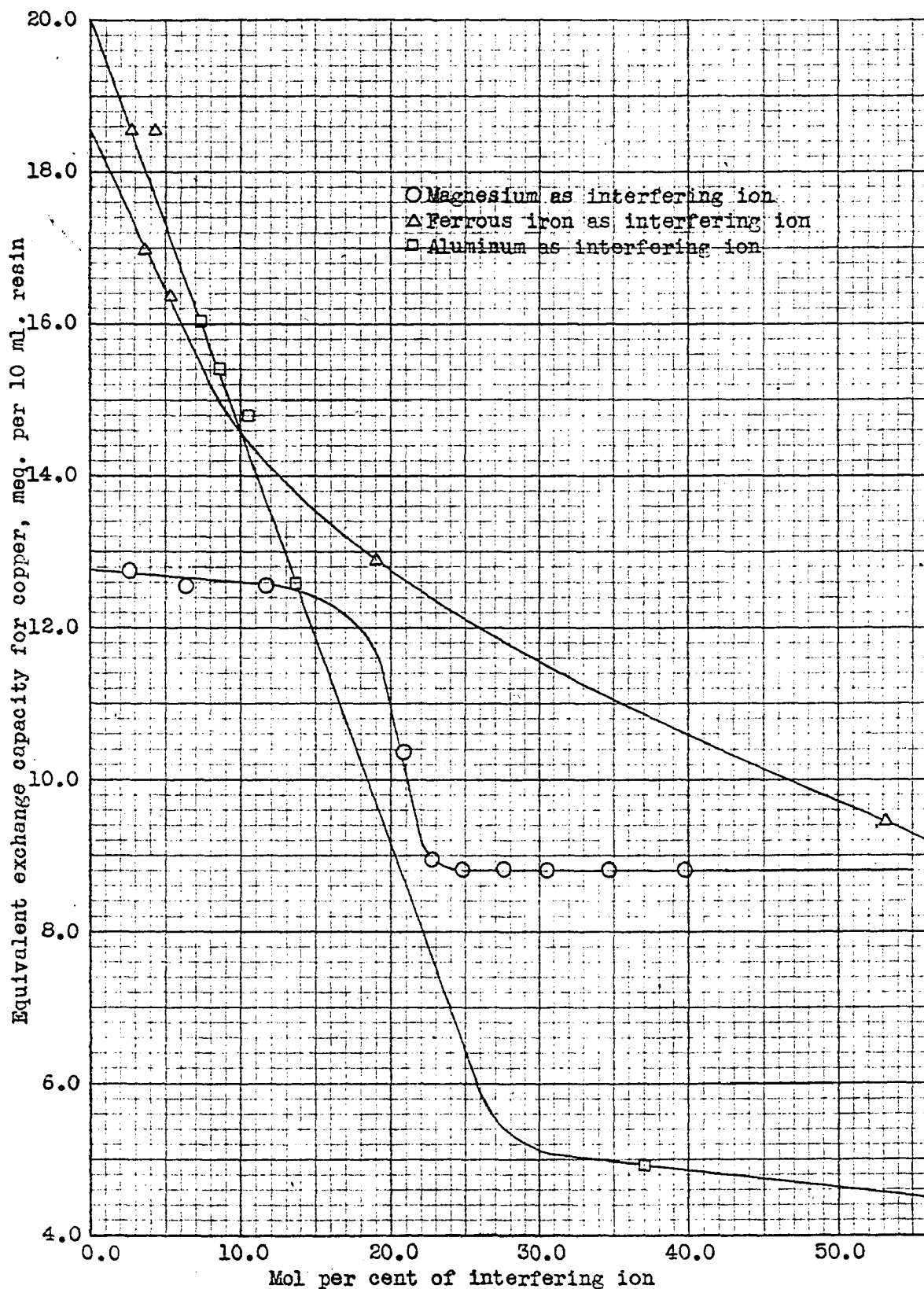


Fig. 47. Effect of interfering ions on equivalent exchange capacity for copper.

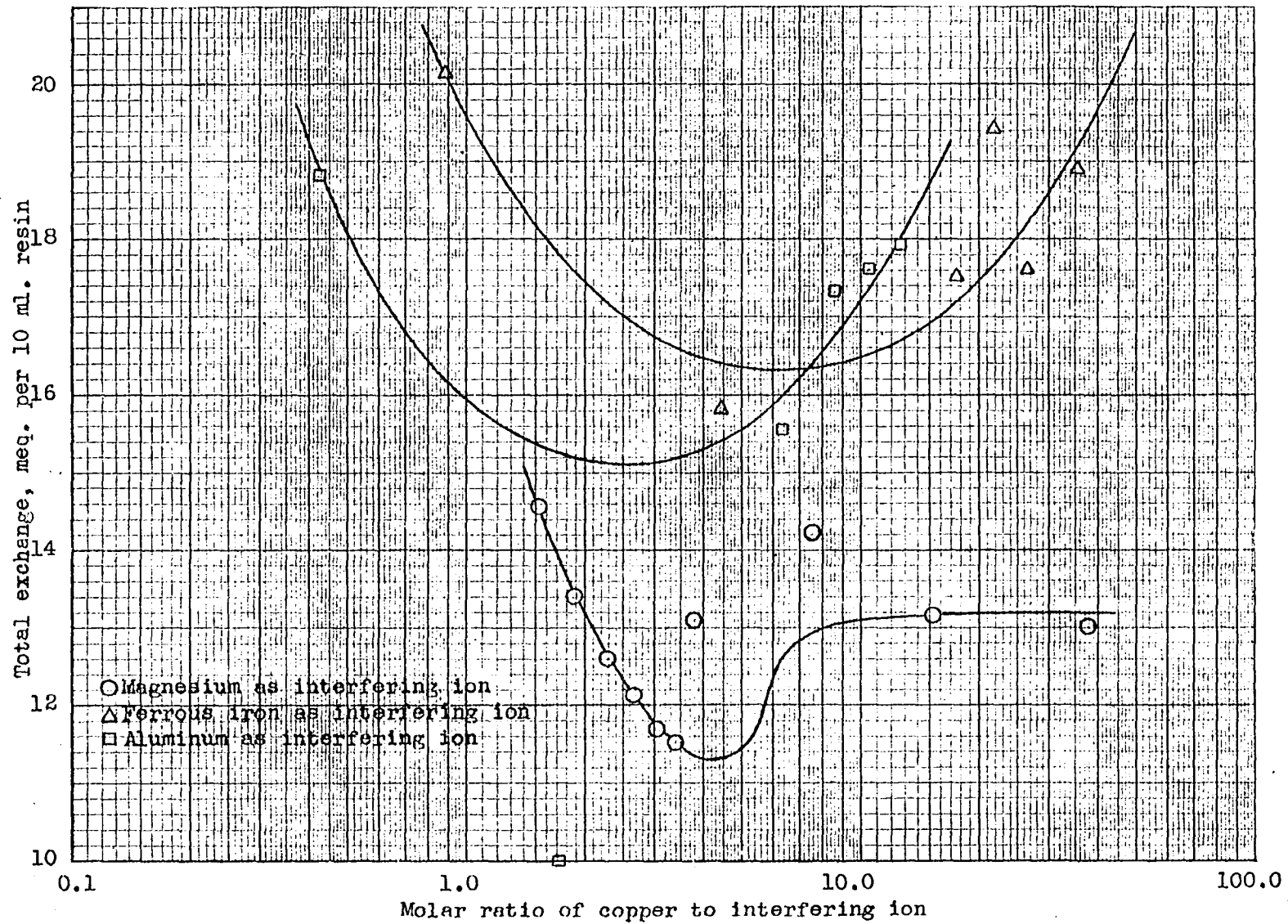


Fig. 48. Total exchange capacity for copper and interfering ion.

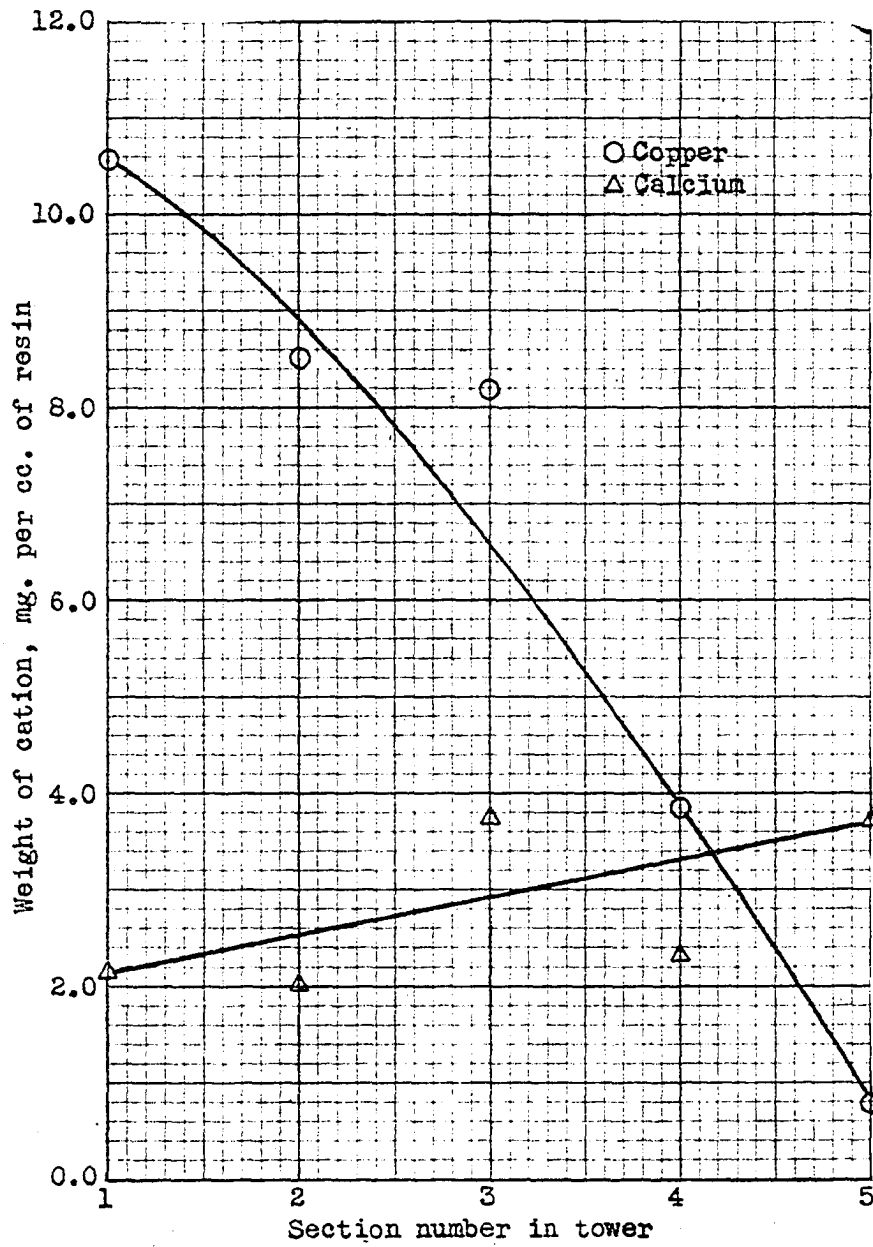


Fig. 49. Weight distribution of copper and calcium in tower.

F. Purification of Phenol Wastes

One of the common applications of anion exchange resins is for the removal of traces of inorganic acids from various solutions. The possibility of removing the weakly-acidic phenols from waste solutions by anion exchange was considered. Such a recovery of phenol might pay for part or all of the expenses normally incurred in preventing stream pollution, and the like, by phenol around coke ovens and refineries.

1. Laboratory investigations

Preliminary studies were made on the adsorption of phenol, utilizing Amberlite IR-4 of Resinous Products & Chemical Company and A-2 of Chemical Process Company. Unfortunately, neither exchanger possessed basic properties of sufficient strength to effectively remove phenol from solutions.

Using a 3 gram sample of Amberlite IR-4, increment volumes of a 200 ppm synthetic phenol solution were added according to the standard static test procedure. The pH readings of this run are shown on Figure 50. The pH values indicate partial removal of the phenol. This was verified by withdrawing a sample for analysis. The total capacity of this run was quite high, being 0.234 grams of phenol per gram of resin. However, at no time in the run was complete removal of phenol effected. A bed operation was then tried, in the hopes that leakage of the phenol through the bed could be prevented. Again, considerable phenol was found in the effluent from the very first period. The same results were obtained with A-2 anion exchanger.

2. Future possibilities for phenol recovery

Although the results of this study on phenol recovery was disappointing, the present state of knowledge on the subject illustrates what can be expected in general from ion exchange in the future. At the time this study was just completed, Resinous Products & Chemical Company announced that an anion exchanger with strongly-basic properties had been prepared. There is every reason to believe that this resin, Amberlite IRA-400, would be very successful in the recovery of phenol. A sample was ordered, but never tested. Hence, this case history illustrates clearly how ion exchange applications will follow closely on the heels of new developments in "exchange tools", one of which is the properties that can be expected from the exchange materials themselves.

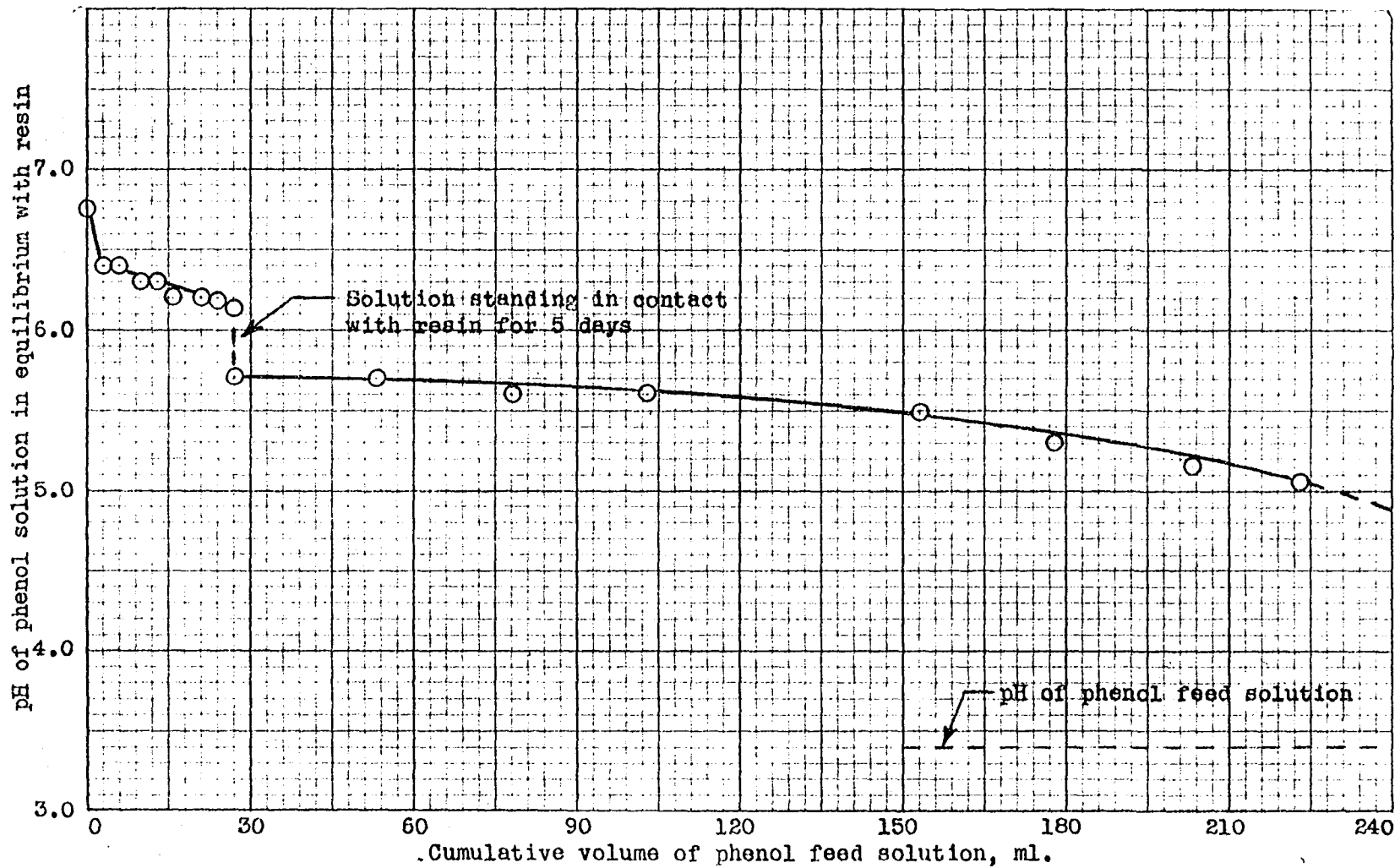


Fig. 50. Phenol adsorption by anion exchange.

IV. DISCLOSURE OF INVENTIONS

A summary is hereby given for the outstanding processes and other inventions that are disclosed in this thesis, and which should warrant consideration from an application for patent standpoint. This summary does not necessarily include all patentable inventions disclosed in this study.

1. The new beaded, inorganic, aluminosilicate cation exchanger has merit, particularly from the standpoint of mechanical properties and increased capacity.
2. The "process for copper production" combines the advantages of cation exchange and leaching by sulfurous acid. This process has been a joint development by R. B. Holt and D. H. White.
3. The utilization of zeolite fines by the fluidized bed technique has the advantages of cheap exchanger and large through-put per unit volume of equipment.
4. Although no laboratory experiments have been performed, it is proposed to separate cadmium ions from other metal ions, on the basis of the extremely low activity possessed by cadmium ions, particularly as the concentration of the solution is increased. For example, at a molar concentration of 2.0, CdCl_2 has an activity coefficient of about 0.05, compared with a value of 0.28 for ZnCl_2 and 0.94 for NiCl_2

at the same molar concentration. The "active molar" concentrations of CdCl_2 will be very low, such that impurities can be more readily removed by cation exchangers, than for those ions possessing similar activity coefficients.

V. SUMMARY AND CONCLUSIONS

1. The "exchange tools" and industrial uses of ion exchange have been classified for better understanding and usefulness.
2. Equilibria studies indicated that the metallic ions, such as copper and nickel, do not undergo equivalent cation exchange.
3. Rate of exchange studies revealed that diffusion of cations to and from the exchanger surface can be an important factor.
4. Rate of exchange data were correlated on the basis of approach to equilibrium in the solid exchanger, and it was further pointed out that the ratio of ions in the solid exchanger should be included in rate of exchange correlations.
5. A technique was developed for obtaining data simultaneously for rate of exchange, static capacity, and equilibrium points.
6. A technique of fluidized bed operation for zeolite fines was developed, which simultaneously provided a potential use for the waste zeolite fines.
7. A new, beaded, inorganic, aluminosilicate cation exchange material was prepared.

8. A "process for copper production" was developed, which combined cheap ore leaching by sulfurous acid with concentration and purification by cation exchange.
9. The purification of phenol wastes was studied; although the results were disappointing, it was pointed out that new, strongly-basic anion exchange resins, such as Amberlite IRA-400, should be suitable for this application.

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The precious metals of the platinum group and gold were recovered as complex negative ions by means of anion exchange materials.
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The rates of ion exchange and their correlation were studied.
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The rates of ion exchange and their correlation were studied.
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The applicability of an exchange unit for softening water to be used in the quality textile industry has been briefly reviewed and strongly recommended.
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A process and apparatus were described for automatically softening water for domestic use with the regeneration taking place at night.
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A process and equipment were described for continuously softening water with means of regenerating and washing the exchanger in separate tanks.

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A process was described for washing the anion exchanger after regeneration with effluent from the cation exchanger, which was obtained by continued operation after the break-through point of the hydrogen cycle. This wash water was free from alkaline earth metal ions but contained alkali metal ions.

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A process was described for the recovery of copper from cuprammonium rayon waste liquors.

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A process was described for removing the non-sugar impurities from sugar juices by means of cation and anion exchangers in order to increase the efficiency of evaporation and of crystallization as well as increasing the output of crystallized sugar while reducing its loss into molasses.
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A process was described for clarification and ionic exchange treatments of sugar juices.
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A process was described for regenerating a cation exchanger containing calcium with a NaCl solution to replace the Ca with Na, whereupon it may be regenerated with H₂SO₄ to give soluble Na₂SO₄.
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Operating data were presented for a new type of cation exchange resin.

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A review of the progress made by ion exchange in the process industries, and the contributions to this progress made by The Resinous Products & Chemical Co. were given in a special publication.
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The range of applications of the amberlite exchangers was presented.
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