AECU-3348

A Study of Granular Ion Exchange

Summary Report

December 1, 1952, to September 30, 1953

Westinghouse Electric Corporation

Atomic Power Division

Subcontract No. 14-316

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DISCLAIMER

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I. SUMMARY

The present report summarizes the experimental work carried out by Ionics, Incorporated, under Task I of WAPD Subcontract 14-316 from December 1, 1952, to September 30, 1953. The four main categories of this section of the experimental program were as listed below.

(A) Investigation of the thermal stability of ion-exchange resins.

(B) Investigation of pH control of solutions using ion-exchange resin bed columns. In these studies various anionic resin forms were used in conjunction with cationic resin in the lithium ion form.

(C) Investigation of demineralization performance at high throughput rates of ion-exchange beds in the hydrogen and hydroxyl resin forms. This work was a continuation of studies carried out at Ionics on mixed bed demineralization (4).

(D) Analysis of resins and water from WAPD experimental loops.

A. Thermal Stability of Ion-Exchange Resins

Results from the thermal stability study program have demonstrated that both Amberlite IR-120 and Dowex-50 resin in the lithium ion form are more stable than the hydrogen form of these resins. The thermal stability of these two resins has been shown to be equivalent. At a temperature of 150° C. the lithium form of the IR-120 resin is predicted to lose half its capacity in 300 days. At this temperature the half-life of the hydrogen form of this resin is predicted to be 100 days. The hydrogen form of IR-120 resin decomposes at two different rates. The more rapid decomposition is associated with 15% of the resin capacity. Half-life values presented in this report are based on the slower decomposition reaction. An estimate of capacity loss at time t must include a factor which corrects for the initial 15% loss in capacity.

The phosphate form of Amberlite IRA-400 has been shown to be much more stable than the hydroxyl form. Half the capacity of this resin in the phosphate form is lost in 35 days at a temperature of 134° C. Hydrolysis of phosphate to acid phosphates also occurs. The resin in the hydroxyl form loses half its capacity in 0.5 days at a temperature of 132° C. The phosphate form of Amberlite IRA-400 at 100° C. is about as stable as the hydrogen form of IR-120 at 150° C., and the lithium form of IR-120 at 180° C. At these temperatures the predicted half-life for all of these resin forms is the order of 100 days.

On the basis of these results, it can be predicted that the temperature limitation for stable operation of ion-exchange equipment in the STR loop may be raised from 60° C. to about 90° C. by replacing the hydrogen-hydroxyl bed presently used with a lithium-phosphate bed. The use of a single resin type (hydrogen form) for demineralization may also be feasible. In this case a temperature as high as 130° to 140° C. can be employed safely.

B. Control of pH by Ion-Exchange Beds

The phosphate, carbonate, and hydroxide form, respectively, of IRA-400 in combination with Amberlite IR-120 in the lithium form have been used in the pH control studies. The development of methods for attaining a high pH effluent (9-11) during demineralization of 100 ppm. NaCl was the object of this phase of the program.

In the first experiment, varying amounts of resin in the hydrogen form were added to a lithium-hydroxide bed in an attempt to control the effluent pH. The hydrogen ion, however, selectively exchanged with metallic ions in solution to provide a neutral effluent and did not provide the moderately high pH desired. There was no leakage of lithium-hydroxide until a major fraction of resin in the hydrogen form had reacted.[1] The effluent pH of the lithium-hydroxide bed remained constant at about 11 until the bed was exhausted.

Use of phosphate and carbonate forms of IRA-400 resin in conjunction with IR-120 resin in the lithium form did not provide rigid pH control. Hydrolysis of the phosphate and carbonate forms of the resin to acid phosphates and bicarbonate occurred during the resin preparation. As a result, the effluent pH from these resin beds varied during an experiment.

Hydrolysis of resin in the phosphate and carbonate forms which occurs when these resin forms are washed free of the salt used in their preparation can be minimized by using dilute solutions of these salts as the leaching agent. However, the hydrolysis of these resin forms during bed operation cannot be inhibited. As a result, variation of the pH of solution effluent is to be expected during treatment of salt solution in these resin bed systems.

The lithium-hydroxide bed seems most useful for pH control on the basis of this phase of the study. The thermal instability of the hydroxyl resin form, however, is an undesirable property of this system, while the lithium-phosphate bed is better in this respect.

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[1] On the basis of this result, the employment of partial regeneration of exhausted mixed beds to permit reuse of these beds appears to be practical.

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C. Demineralization at High Rates of Throughput

Investigation of the demineralization performance of mixed hydrogen-hydroxyl resin beds at very rapid throughput rates has shown that this manner of operation is satisfactory. For example, at a flow rate of about 20 gal./ft^2 min., only 23% of the bed capacity was unused at 10% breakthrough. This result was predicted from correlation of data obtained from an earlier investigation by Ionics, Incorporated (4).

D. Analysis of Samples from Loop Tests

With regard to the analytical program, mixed resin samples from the M-3 experimental loop dated April 3, 1953, April 30, 1953, and June 15, 1953, and an M-3 loop water sample dated June 17, 1953, have been analyzed during the period of this study program. The results are presented in a later section of this report.

E. Miscellaneous Studies

Several short-term studies conducted during this work period yielded the following results:

(1) A monobed containing uniformly mixed resins in the hydrogen and hydroxyl form did not noticeably classify after 15 1/2 hours vibration at a frequency which produced an acceleration 1.7-1.8 times as great as gravity.

(2) Cation-exchange resin in the thorium form did not exhibit anion-exchange properties as it was expected to. On the basis of membrane potential measurements made at Ionics (8), it was believed that a cation-exchange resin in the thorium form would behave as an anion exchanger.

(3) The purity of lithium form resin prepared at Ionics exceeded Navy specifications of 0.3% sodium (equivalent basis).

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II. INTRODUCTION

A. The Thermal Stability of Ion-Exchange Resins

An earlier investigation by Ionics, Incorporated, of the stability of various resins at high temperature had indicated that Amberlite IRA-400 and Dowex-1 and Amberlite IR-120 and Dowex-50 are the most stable synthetic organic resins which can be used above room temperature for mixed bed demineralization (4). The hydroxyl form of the anion exchangers is much less stable than the hydrogen form of the cation exchangers, however, and limits the system temperature to 120°-140° F. when these resin forms are employed. As a result, a large amount of cooling is necessary to operate a mixed bed exchanger in the STR.

The possibility of increasing the thermal stability of the ion-exchange system by employment of different resin forms was investigated. The stability of the lithium form of IR-120 and the phosphate form of IRA-400 was tested for use in a mixed bed. Each of these salt forms was expected to be thermally more stable than the respective hydrogen and hydroxyl resin forms. Operation of a hydrogen bed alone for demineralization seemed feasible, since the major corrosion products are probably present as basic oxides, and the thermal stability of the hydrogen form of Amberlite IR-120 was also studied.

B. The Control of System pH by Mixed Ion-Exchange Resin Bed Systems

Data obtained by WAPD (1) had indicated that the corrosion of stainless steel was minimized in a slightly basic (pH 9-10) system. An ion-exchange system which effectively removed corrosion products while maintaining the pH of the system at a value of about 9-10 was therefore sought. Use of the carbonate and phosphate forms of IRA-400 in combination with the lithium form of IR-120 was investigated. Column operation

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with respect to demineralization and pH control was tested using these resin forms. The hydroxyl form of IRA-400 in combination with the IR-120 resin in the lithium form was also investigated.

C. <u>The Demineralization Performance of Mixed Hydrogen-Hydroxyl Form Resin Beds Exposed to Rapid Flow Conditions</u>

The effect of certain operating variables on the demineralization of sodium chloride solution has been studied earlier at Ionics (<u>4</u>). Correlation of the data obtained facilitated prediction of demineralization performance at various operating conditions. Extrapolation of this correlation indicated that satisfactory operation might be obtained at much higher flow rates than had previously been anticipated. P. Cohen and V. W. Thompson of WAPD requested additional data (<u>2</u>) in the high flow rate region to confirm this prediction.

D. <u>Analysis of Resins and Solution from WAPD Experimental Loops Using a Lithium-Hydroxide Mixed Bed</u>

The presence of small amounts of lithium in samples from corrosion study tests at WAPD which used experimental loops and a lithium-hydroxide ion exchanger prevented accurate analysis of the corrosion elements in these samples by spectrographic techniques at WAPD (<u>9</u>). This analytical difficulty had not been anticipated, and standardized methods for analysis of the corrosion elements by other methods were not available to WAPD. To avoid delay in the WAPD corrosion study program, Ionics was requested by P. Cohen (<u>2</u>, <u>9</u>) to analyze resin and solution samples from these tests. Analyses of resin and solution samples from WAPD experimental loops were conducted at Ionics, Incorporated.

III. THERMAL STABILITY STUDIES

A. Procedure

Small, accurately weighed samples of resin (about 2 g.) were sealed under 5 cc. of water in Pyrex glass tubes. The glass tubes were enclosed in steel tubes containing water and were heated at a fixed temperature in a laboratory drying oven. Samples were removed periodically for capacity determination. Loss of capacity was taken to be a measure of the resin decomposition. The resins studied were Amberlite IR-120 in the hydrogen and the lithium forms and Amberlite IRA-400 in the phosphate form. The temperatures covered were 150° C., 180° C., and 190° C., using hydrogen form IR-120; 150° C. and 180° C., using lithium form IR-120; and 100° C. and 134° C., using phosphate form IRA-400.

B. Results

The results of the various tests are summarized in a semi-logarithmic plot of the reciprocal of the fraction of initial resin capacity remaining after a given time of heating (ln Co/C) versus the time of heating in days (Figure 1). In Figure 2 the same method of plotting is used on an expanded scale to compare the decomposition rates at 150° C. of the lithium form of Amberlite IR-120 and the hydrogen form of Amberlite IR-120. With this type of plot a first order decomposition reaction is indicated by a straight line, and in such a case a half-life value can be obtained directly for a given resin form at a given temperature. The experimental points plotted in Figures 1 and 2 are listed in Table AII, which is in the Appendix.

First order decomposition reactions were indicated for most of the resin forms studied. The decomposition of the hydrogen form of IR-120, however, appeared to be more complex. By assuming the decay of this resin

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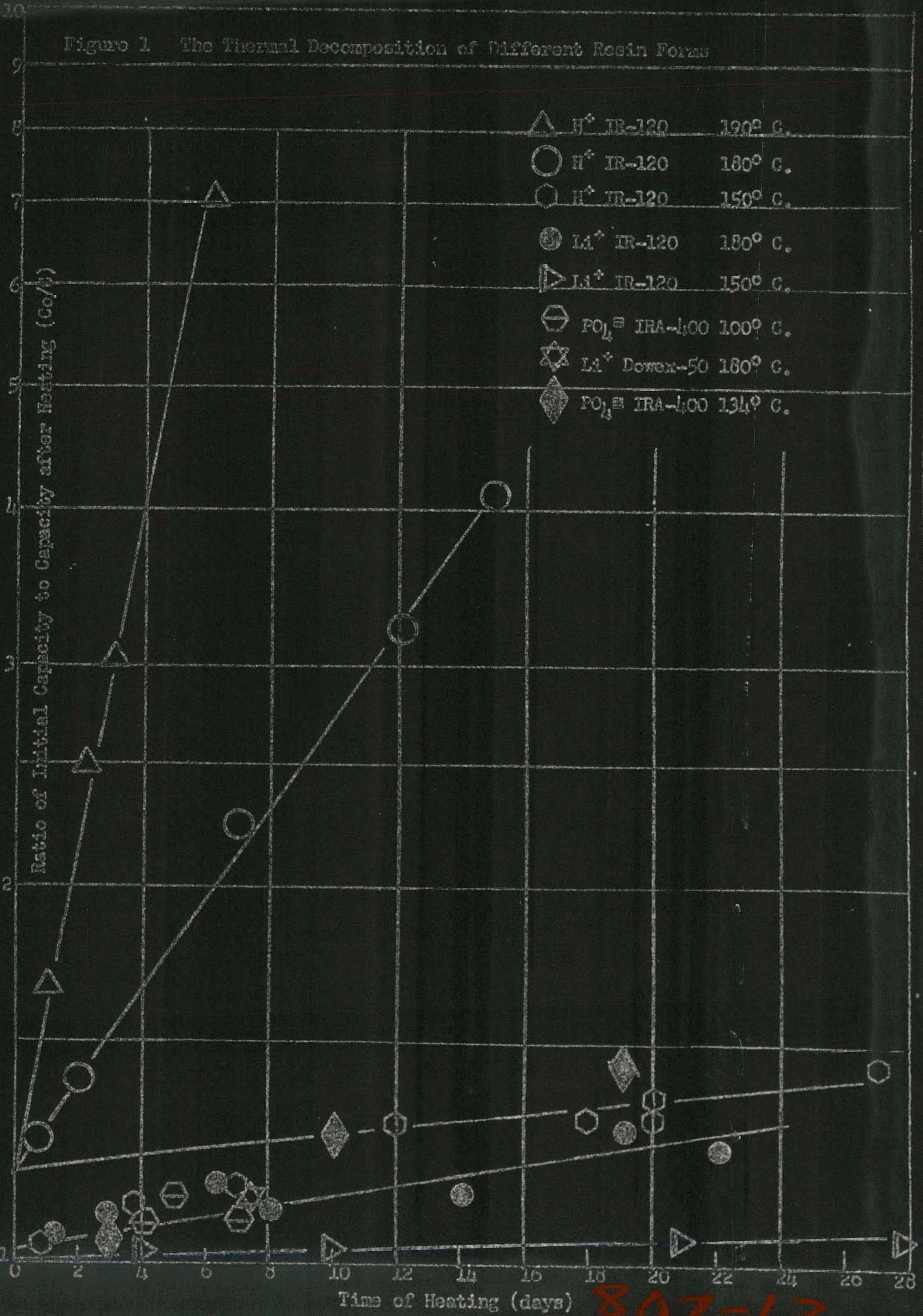
Figure 1 The Thermal Decomposition of Different Resin Forms

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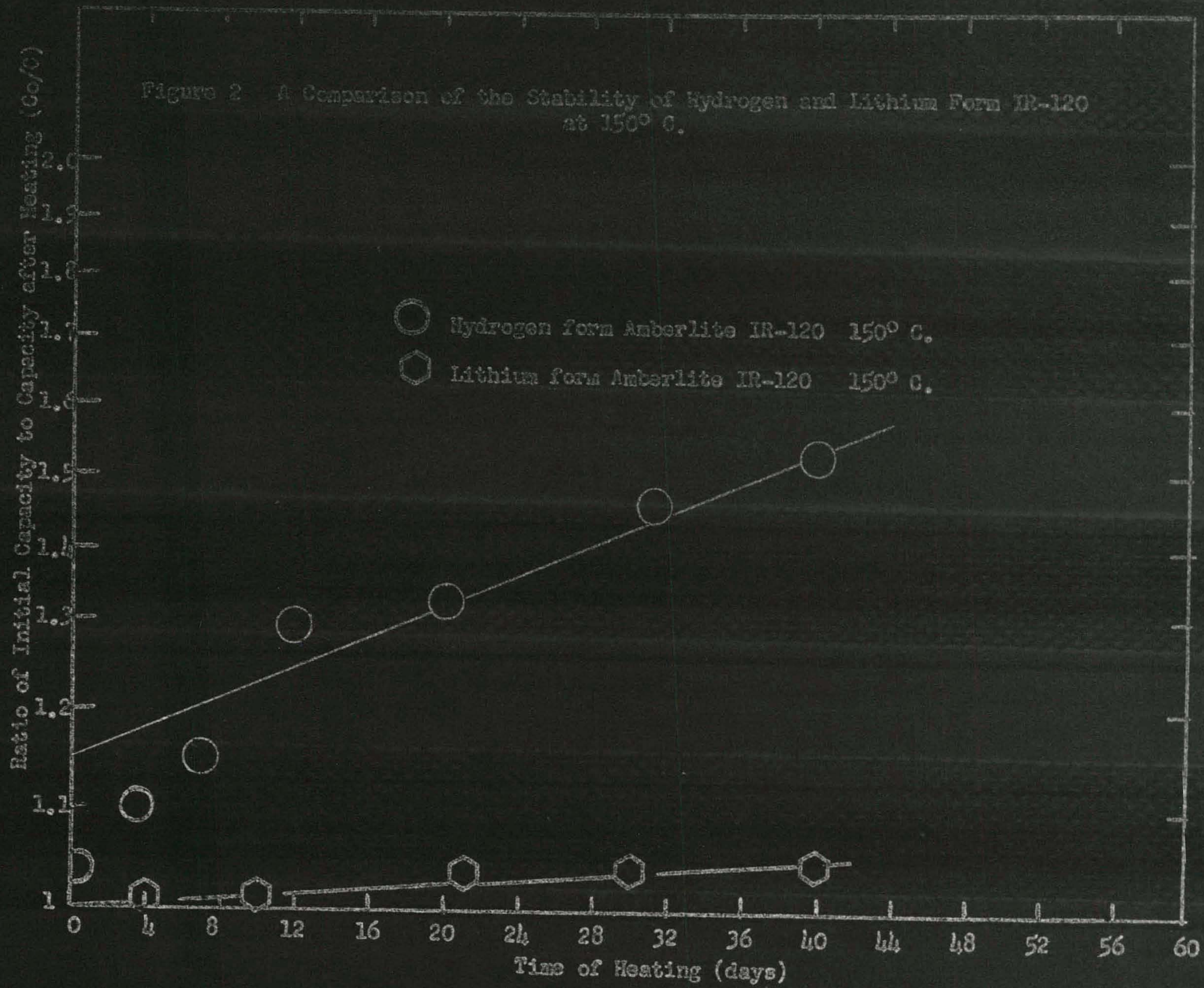

Figure 2 A Comparison of the Stability of Hydrogen and Lithium Form IR-120 at 150° C.

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form to occur by two different paths (about 15% of the resin capacity being lost more rapidly than the other 85%), the semilogarithmic plot resolves into two straight lines, one line intercepting the origin. At 150° C. the first decay reaction is slowed and is not complete before 10-12 days. At the higher temperatures (180° C. and 190° C.) decomposition by the more rapid path occurs in less than a day.

In the case of the thermal stability studies with lithium form resin, only a simple decay pattern is observed. The chemical analogue of Amberlite IR-120, Dowex-50, in the lithium form gives identical results to Amberlite IR-120 after 7 days' and 35 days' heating at 180° C.

In the case of phosphate form Amberlite IRA-400, the decomposition is first order. The decomposition measurements are based upon ion-exchange capacity remaining after a given time of heating rather than upon equivalents of phosphate remaining on the resin, since the ionic form of the resin is not well known. There is evidence that the phosphorus (as a result of hydrolysis) can be present as a mixture of $PO_4^{\equiv}$ and $HPO_4^{=}$ in the resin matrix (see Section V).

Resin half-lives have been calculated using the straight lines shown in Figure 1, and the equation $\log C_o/C = kt$, where C_o is the initial resin capacity, C is the resin capacity at time t, and k is the first order decomposition rate constant. The half-life values which have been computed are presented in Table I.

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Table I

Estimated Half-Life Values for Various Resin Forms[1]

Resin and Form	Temp. °C.	Decomposition Rate Constant (days^{-1})	Resin Half-Life Calc. in Days from Rate Constant
IRA-400			
OH^-	60	2×10^{-4}	1900
OH^-	117	1×10^{-1}	2.5
OH^-	132	6×10^{-1}	0.5
IRA-400			
$PO_4^{\equiv}$	100	2×10^{-3}	130
$PO_4^{\equiv}$	134	9×10^{-3}	35
IR-120			
H^+	150	3×10^{-3}	100
H^+	180	4×10^{-2}	8
H^+	190	1×10^{-1}	2
IR-120			
Li^+	150	1×10^{-3}	300
Li^+	180	2×10^{-3}	130

1. Calculation of the half-life of the more rapid reaction observed with the hydrogen form IR-120 has not been attempted. The half-life values are listed together with resin half-lives and decomposition rate constants estimated for the hydroxyl form of IRA-400. These values, obtained in an earlier study (4), are included in this table for comparison and for the sake of completeness.

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IV. EFFLUENT PH CONTROL STUDIES OF ION-EXCHANGE RESIN COLUMNS

A. Procedure

Well-mixed resin beds (about 1 foot in length and 1 inch in diameter) containing Amberlite IRA-400 in the forms to be tested were treated under substantially air-free conditions with 100 ppm. NaCl solution. Prior to use, the beds were grounded to remove their electrostatic charge, thereby effecting a densely packed bed. The pH of effluent samples was measured with a Beckmann pH meter. In some cases the effluent solutions were sampled and analyzed for sodium content with a Baird flame photometer.

The resin beds first tested for pH control of effluent were mixtures of lithium and hydrogen form cation resin (IR-120) in varying ratios with hydroxyl form anion resin (IRA-400). The study was extended to cover carbonate and phosphate anion resin forms used in conjunction with lithium cation form. The phosphate form of Amberlite IRA-400 was the only form tested at several different flow rates and temperatures.

B. Results

The experimental results are expressed in Figures 3, 4, and 5. The figures are plots of the variation in the effluent solution pH with time for different resin mixtures, temperatures, and flow rates.

The size of the resin beds was kept constant, and the method of plotting is uniform, so that the curves shown in Figures 3, 4, and 5 are directly comparable with each other. The columns used were necessarily small, and since 100 ppm. NaCl solution represents a higher ionic solution concentration than is likely to be encountered in the actual WAPD

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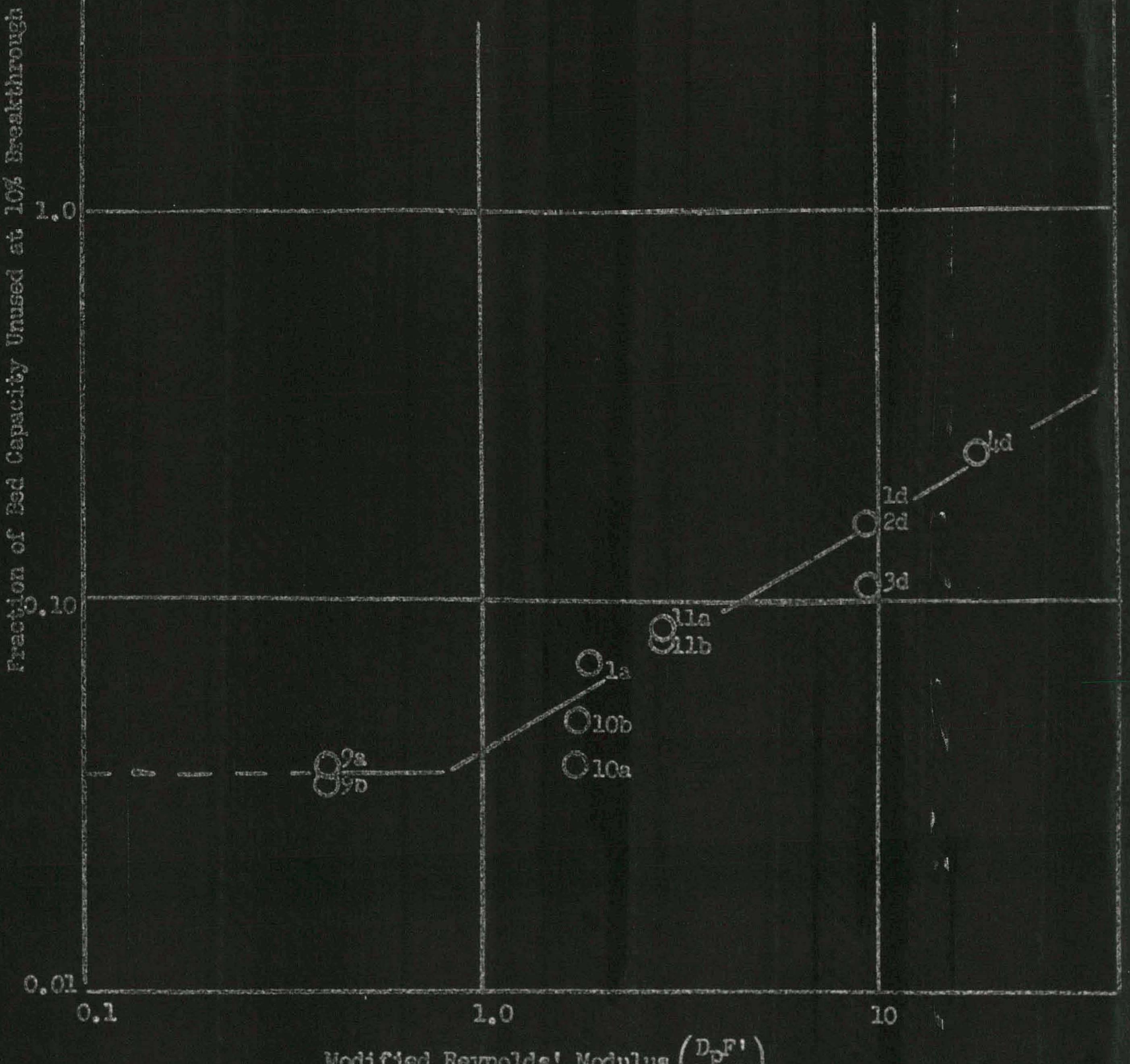
Figure 6 A Graphical Correlation of the Demineralization Performance of a Mixed Bed



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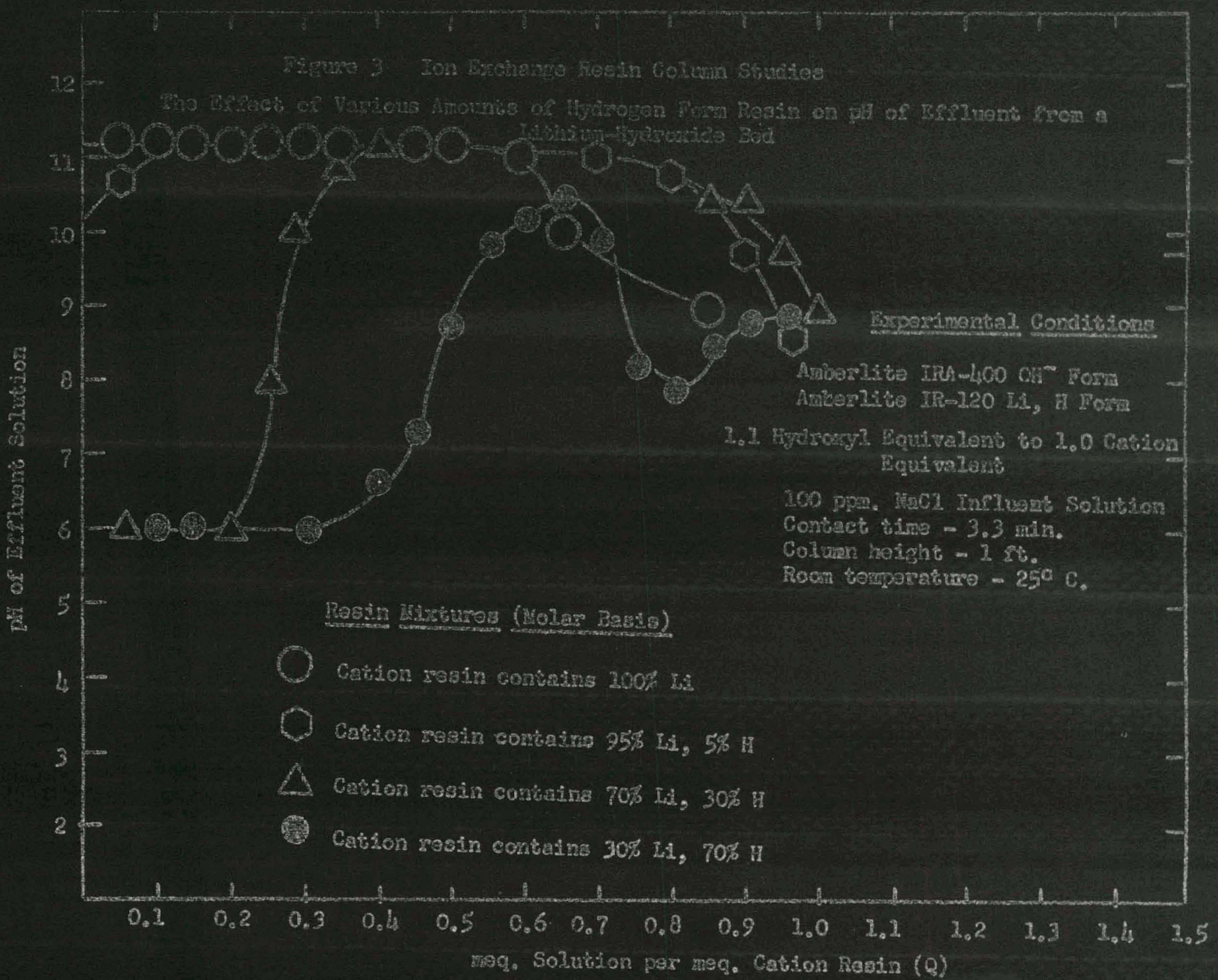

Figure 3 Ion Exchange Resin Column Studies

The Effect of Various Amounts of Hydrogen Form Resin on pH of Effluent from a Lithium-Hydroxide Bed

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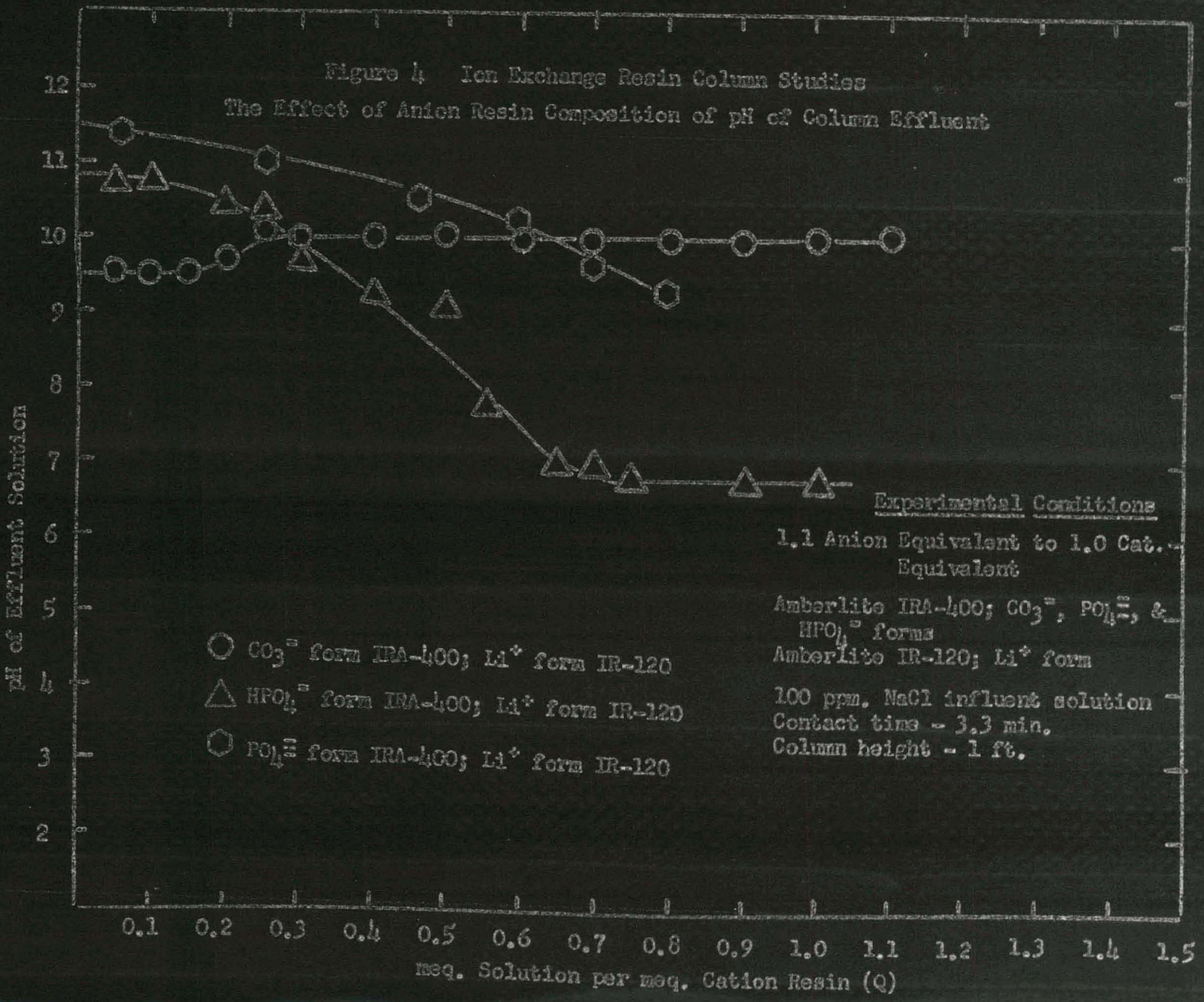

Figure 4 Ion Exchange Resin Column Studies

The Effect of Anion Resin Composition of pH of Column Effluent

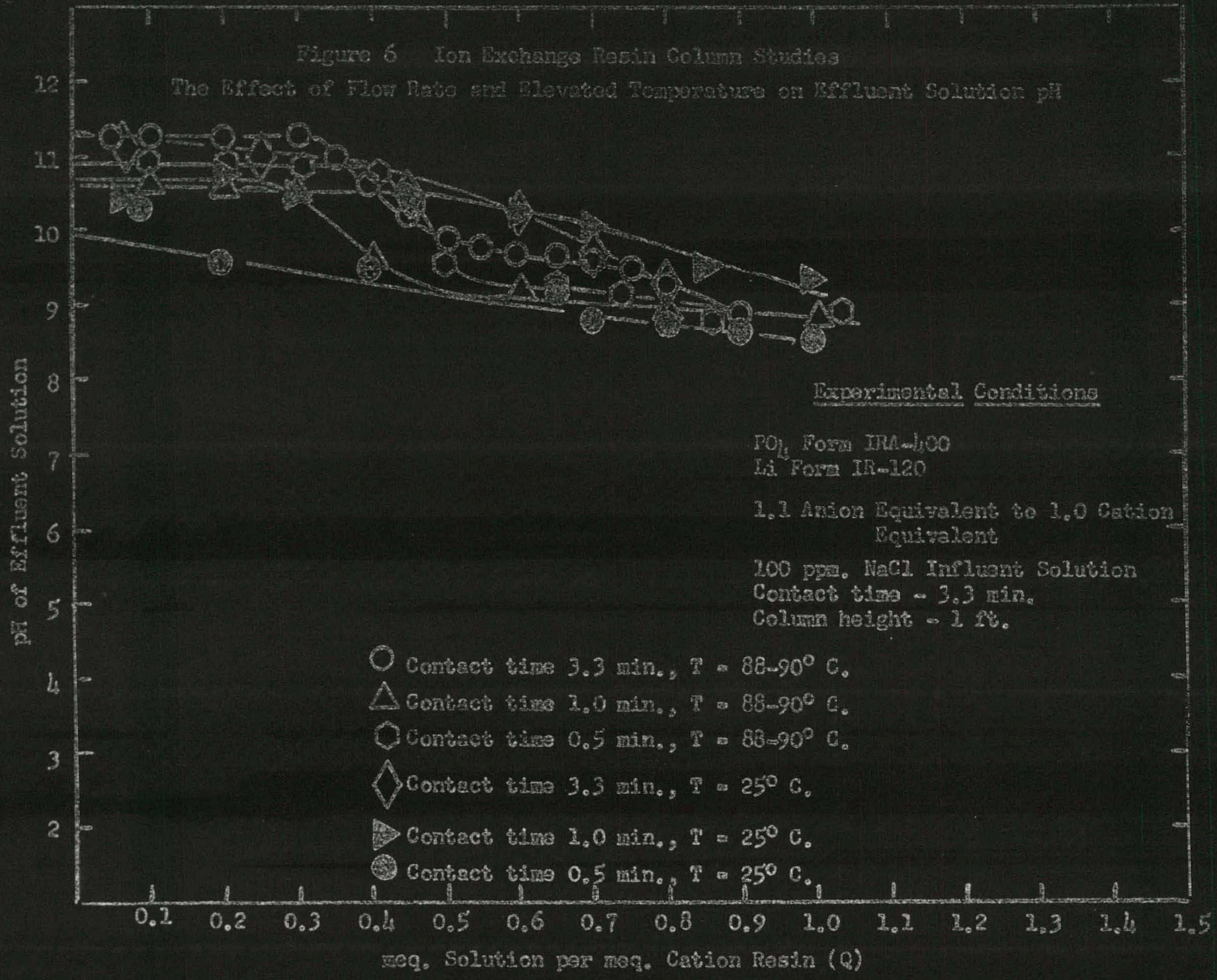

Figure 6 Ion Exchange Resin Column Studies

The Effect of Flow Rate and Elevated Temperature on Effluent Solution pH

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loop ion-exchange bed, the results should be taken as only a qualitative picture of effects to be expected when different column sizes and flow rates are used.

In Figure 3 are presented the effluent solution pH histories obtained using resin mixtures containing, initially, lithium or lithium and hydrogen in the cation fraction and hydroxyl in the anion fraction. There is an exchange of dissolved sodium for resin-bound hydrogen and/or lithium. Simultaneously, an exchange of dissolved chloride for the resin-bound hydroxyl ion occurs. It will be noted that when hydrogen is present, it tends to exchange first, producing a neutral or nearly neutral effluent solution during the first part of the run. Where hydrogen form cation resin is absent, the pH is a measure of the efficiency of the anion-exchange reactions.

Figure 4 shows the pH histories of effluent solutions obtained by passing 100 ppm. NaCl solution through Li form IR-120 resin combined with either the carbonate, the $HPO_4^=$, or the $PO_4^{\equiv}$ forms of IRA-400. Constant effluent pH values of about 10, 11, and 9 were predicted for the carbonate, phosphate, and acid phosphate forms, respectively. Instead, the pH pattern was variable in all of the runs.

The slightly low pH of the effluent at the start of the run with the $Li^+-CO_3^=$ resin bed can be attributed to some hydrolysis of carbonate to bicarbonate ion. The pH pattern of effluent in the other two runs may also be explained by hydrolysis. In the run with anion form resin prepared from tri-sodium phosphate, the eventual reduction of effluent pH from a value of about 11 to 9 indicates the presence of $PO_4^{\equiv}$, OH, and $HPO_4^=$ ions. Similarly, the reduction in pH of effluents

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from sodium acid phosphate suggests the presence in the resin of some OH^- and $H_2PO_4^-$ ions as well as some $HPO_4^=$ ion.

The effect of temperature and flow rate on the pH control performance of lithium-phosphate beds is shown in Figure 5. The slight variation in effluent pH pattern as a function of either temperature or flow rate is within the limits of experimental control. This result appears to indicate that pH control is not strongly dependent on temperature or flow rate.

The efficiencies of the lithium-sodium exchange in the runs which are listed in Table AI of the Appendix have been evaluated graphically according to the method of computation which is outlined in Ionics' Summary Report of December 1, 1952 (4). The results indicate that the demineralization is less efficient than in the mixed bed which uses resins in the hydrogen and hydroxyl forms.

V. MIXED RESIN BED DEMINERALIZATION STUDIES

During ion-exchange demineralization a dissolved salt consisting of a cation and an anion component is exchanged by contact with resin-bound hydrogen ion and with resin-bound hydroxyl ion in such a manner that neutralization occurs, and pure water results. The distinction is therefore plain between the experiments to be described, and those described in the preceding section where sodium and chloride ions in solution were exchanged, respectively, for lithium ion and some other anion.

The factors affecting mixed resin bed demineralization were summarized in Ionics' Report of December 1, 1952 (4). The results of numerous laboratory column experiments are also described therein. This earlier work has now been further amplified by a series of laboratory column experiments which were designed to provide a basis for estimation of ion-exchange column performance at very rapid flow rates.

A. Procedure

As in the previously reported demineralization experiments, hydrogen form Amberlite IR-120 was used in conjunction with hydroxyl form Amberlite IRA-400. The resin beds were discharged by grounding with a copper wire, and the air-free mixed resin beds were tested in Pyrex glass columns by passing 100 ppm. NaCl solution down flow under known temperature conditions and at fixed flow rates. Samples of the effluent solutions were periodically collected and analyzed for sodium content using the Baird flame photometer. Conventional ion-exchange column breakthrough curves were constructed and graphically analyzed according to the method referred to in the preceding section. Two-foot resin columns were used in order to eliminate the effect of column length (4).

B. Results

The results obtained and a comparison with earlier results are shown graphically in Figure 6 (see Table AIII, Appendix, for tabulated values). The runs numbered 1-d, 2-d, 3-d, and 4-d represent the new data.

In Figure 6, b, the fraction of the bed capacity unused at 10% breakthrough, is plotted against a modified Reynolds' modulus using a log-log scale. The linear solution velocity (related to the volumetric flow rate and to the resin-solution contact time for a given size of resin bed) is expressed by the modified Reynolds' modulus, D_pF/μ, where D_p is the average resin particle diameter, 1.93×10^{-3} ft., μ is 2.35 lb. per hr.ft., and F is the space velocity factor expressed as lb./ft^2.hr.

The demineralization performance of the mixed resin beds studied in the hydrogen and hydroxyl forms at varying linear velocity of solution throughput can be estimated by comparison of the various b values obtained in the graph. For example, the effect of elevated temperature is indicated by the lower b value of run No. 3-d as compared to run Nos. 1-d and 2-d. In run No. 3-d, which was conducted at 75° C., the b value is 0.11 as compared with 0.16 for the b value obtained at 25° C. The difference is small but experimentally significant and indicates that performance in this region is improved by raising the temperature of the system. This result is to be expected, since mass transfer rate is increased with temperature.

Extrapolation of the data obtained in the earlier study predicted very well the performance observed at higher flow rates.

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VI. ANALYSIS OF SAMPLES FROM WAPD EXPERIMENTAL LOOP TESTS

A. Procedure

The resins as received from WAPD were separated into the anion and cation components by flotation in a glycerol-water mixture. The cation and anion components were eluted in separate glass columns with 2N HCl (about 900 cc./column) followed by 12N HCl (about 100 cc.). The eluates were tested for Na and Li with the Baird flame photometer. Combined calcium and magnesium expressed as calcium carbonate was determined by a versene titration. The solutions were concentrated by boiling, and the Fe was removed by ether extraction for analysis.

It was established that Ni, Mn, and Co can be separated quantitatively in strong hydrochloric acid solution by the technique of Kraus and Moore (3). In this method the Mn and Co are absorbed on an anion-exchange resin as chloride complexes from strong HCl solution and are separated chromatographically on elution with HCl of diminishing concentration. The method permits concentration of the metals for spectrophotometric determination using a Beckmann DU spectrophotometer. The anion resin fraction of June 18, 1953, was tested together with an unused blank resin sample for carbonate, hydroxyl and chloride content by titration of a saturated $NaNO_3$ eluate solution.

B. Results

The complete results of analyses of mixed resins from the M-3 experimental loop dated April 3, 1953, April 30, 1953, and June 18, 1953, and of an M-3 loop water sample dated June 17, 1953, are listed in Table II.

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Table II

Analytical Results

a) M-3 Loop Water Sample of June 17, 1953

pH	10.3
Li	1.0×10^{-4} N
Na	0 ($> 10^{-6}$ N)
Fe	1.0 mg./1. (1 ppm.)
Ni	0
Cr	0
Mn	0
Co	0

b) M-3 Loop Resin Samples

	Cation Apr. 3	Anion Apr. 3	Cation Apr. 30	Anion Apr. 30	Anion June 18	Anion June 18(blank)
Volume (cc.)	36	72	31	71		
OH^-					2.5 meq/bone dry resin gm	2.4 meq/bone dry resin gm
$CO_3^{=}$					0.54 meq/bdg	0.52 meq/bdg
Cl^-					1.7 meq/bdg	1.7 meq/bdg
	mg. per total resin sample					
Li	450 mg	0	407 mg	0		
Na	2.9 mg	0	6.9 mg	0		
Ca Mg As $CaCO_3$	78 mg	0	98 mg	0		
Fe	2.2 mg	5.0 mg	0.7 mg	0.14 mg		
Ni	0	trace	0	trace		
Co	0.01 mg ± 0.007	0.003 mg ± 0.007	0 >0.007 mg	----		
Cr	0 (>0.0005 mg)	0 (>0.0005 mg)	0 (>0.0005 mg)	0.015 mg ± 0.0005 mg		
Mn	0.50 mg	0.13 mg	0.50 mg	0.09 mg		

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VII. MISCELLANEOUS STUDIES

Several additional tasks that are not included under the four main categories of the experimental program were completed during this work period. These shorter-term experiments have been reported elsewhere (5, 6, 7). A summary of these experiments is presented below.

Two runs were made to determine whether classification of a hydrogen-hydroxyl bed occurred as a result of exposure to vibrations of high frequency (5). In these experiments the mixed bed endured an acceleration of 1.7-1.8 times the acceleration due to gravity as requested by Mr. Cohen of WAPD in accordance with Navy specifications. The two vibration-classification runs used a well-packed, one-foot mixed bed 4 1/4" in diameter and a full amplitude of 0.068" and 0.04" at a frequency of 1350 and 1750 per minute, respectively. Resin classification did not occur after 15 1/2 hours vibration under each condition. This information supplements data obtained during an earlier investigation by Ionics, Incorporated, or resin bed classification as a result of exposure to vibration (4).

Studies at Ionics, Incorporated, (8) had shown that a cation-exchange membrane in the thorium form exhibits membrane potentials which are characteristic of an anion-exchange membrane. On the basis of this observation, it was believed that a cation resin in the thorium form might behave as an anion exchanger. If this were the case, the quaternary amine type of anion-exchange resin could be replaced by a cation-exchange resin in the thorium form to provide a mixed bed which is thermally stable. The thorium form of Amberlite IR-120 was studied to see if it exhibited anion-exchange properties. Experiments using sodium hydroxide and sodium chloride showed that the thorium form of IR-120 did not act as an anion-exchange resin (6).

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Finally, a cubic foot of lithium form Amberlite IR-120 was prepared for WAPD (7). The sodium content was 0.08% to 0.12% on an equivalent basis. No hydrogen was observed. The purity of the lithium form resin exceeded Navy specifications of 0.3% sodium (equivalent basis).

VIII. APPENDIX

A. The Demineralization Efficiency of Mixed Beds in Various Ionic Forms

The relative efficiencies of the cation-exchange reaction in the runs carried out are represented by the b values (calculated according to the graphical method discussed in an earlier report) (4) listed below in Table AI. The term b represents the fraction of the cation-exchange capacity of the resin column which is unused at 10% influent cation breakthrough. The b values obtained in the present studies are somewhat higher than those previously obtained under identical experimental conditions using mixed resin beds initially in the hydrogen and hydroxyl forms. The b values listed in Table AI range in general between 0.25 and 0.35. The b value obtained using the hydroxyl and the hydrogen resin forms was less than 0.10.

Table AI

Sodium Breakthrough Characteristics of Amberlite IRA-400, Amberlite IR-120 Mixed Resin Beds (cf. Figures 3 - 6)

Contact Time (minutes)	b Value	I Values	t (° C.)	Resin Bed Composition (Ratio: 1.1 anion eq. to 1.0 cation eq.)
3.3	0.26	0.99	25	100% Li cation; OH anion
3.3	0.29	1.10	25	95% Li, 5% H cation; OH anion
3.3	0.25	1.04	25	70% Li, 30% H cation; OH anion
3.3	0.27	1.02	25	30% Li, 70% H cation; OH anion
3.3	0.27	1.13	25	Li cation; CO_3 anion
3.3	0.15	0.96	25	Li cation; HPO_4 anion
1.0	0.34	0.98	25	Li cation; PO_4 anion
0.5	0.67	0.95	25	Li cation; PO_4 anion
3.3	0.25	0.95	90	Li cation; PO_4 anion
1.0	0.40	1.0	90	Li cation; PO_4 anion
0.5	0.45	0.97	90	Li cation; PO_4 anion

Table AI also lists I values for the various runs. The term I, as explained in Ionics' Summary Report of December 1, 1952 (4), represents the exactness of fit between the experimental data and the empirical mathematical theory used in the computation of b. An I value of 1.0 corresponds to an exact

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correlation between experiment and theory. A deviation of the I value from 1.0, which is greater than ± 0.07, is probably due to imperfect experimentation or inappropriate application of the graphical method, since the maximum experimental errors likely to be encountered are expected to give a fluctuation in I of ±7%.

B. Summary of Results

Table AII

Thermal Stability Data

Resin and Form	Experimental Temperature (° C.)	Time of Heating (days)	C/C_o	C_o/C
IRA-400 $PO_4^{\equiv}$	100	4	0.94	1.07
		5	0.89	1.13
		7	0.95	1.07
	134	3	0.95	1.05
		4	0.92	1.08
		10	0.79	1.26
		19	0.70	1.43
IR-120 H^+	150	0.7	0.96	1.04
		3.8	0.89	1.12
		7	0.86	1.16
		12	0.78	1.29
		20	0.77	1.31
		31	0.69	1.45
		40	0.66	1.52
IR-120 H^+	180	0.7	0.79	1.26
		2	0.71	1.41
		7	0.45	2.24
		12	0.31	3.24
		15	0.24	4.17
IR-120 H^+	150	4	0.99	1.01
		10	0.98	1.025
		21	0.95	1.05
		20	0.95	1.05
		40	0.935	1.07
		80	0.91	1.10
IR-120 Li^+	180	1	0.96	1.04
		3	0.92	1.09
		8	0.90	1.11
		14	0.88	1.14

Table AIII

Demineralization Performance Studies with 100 ppm. NaCl at 25° C.

Run	Space Velocity lb./ft^2. hr.	Modified Reynolds' Modulus D_pF/μ	L Length of Col. (ft.)	b	I	Contact Time (min)
9-a	501	0.42	2.9	0.035	0.98	20.8
9-b	501	0.42	2.1	0.038	0.97	15.4
10-a	2,110	1.75	3.0	0.038	0.98	5.4
10-b	2,110	1.75	2.0	0.050	0.97	3.5
1-a	2,300	1.9	2.8	0.068	1.00	4.5
11-a	3,500	2.9	3.15	0.078	0.97	3.2
11-b	3,500	2.9	2.2	0.079	0.94	2.2
1-d	11,600	9.65	2.0	0.16	1.01	0.76
2-d	11,600	9.65	2.0	0.16	1.01	0.76
3-d*	11,600	9.65	2.0	0.11	1.01	0.76
4-d	21,900	18.0	2.0	0.23	—	0.41

* Temperature of run was 75° C.

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C. References

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