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Cation exchange between solutions of ferric nitrate and DOWEX 50X-8 resin

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CATION EXCHANGE BETWEEN SOLUTIONS OF
FERRIC NITRATE AND DOWEX 50X-8 RESIN

BY

MICHAEL H. MANSKY

A THESIS

PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE

OF

MASTER OF SCIENCE IN CHEMICAL ENGINEERING

AT

NEWARK COLLEGE OF ENGINEERING

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Newark, New Jersey
1965

APPROVAL OF THESIS

FOR

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NEWARK COLLEGE OF ENGINEERING

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ABSTRACT

The rate of exchange of ferric ions from solutions of ferric nitrate and hydrogen ions from Dowex 50X-8 has been studied in an agitated bed ion exchange column over a range of bed loading conditions. Agitation has been maintained in a regime known to indicate film diffusion control of rate in small scale batch reactors.

Calculated diffusion coefficients for the column are slightly higher than those determined in the batch reactor because of greater turbulence of agitation in the column.

The assumption of previous investigators concerning linearity of equilibrium data has been found to be invalid for the system studied. Equilibrium data for the system investigated has been determined and the necessity of accuracy in its determination is discussed.

The relationship between pH, the rate monitoring technique used in these studies, and ferric ion concentration in solution has also been determined and the necessity of this determination is discussed.

A method of constructing predicted concentration-vs-time curves for agitated bed ion exchange columns is presented.

BACKGROUND AND INTRODUCTION

Several investigators have attempted to correlate steady state flow results with batch experiments for the dissolution of salts in water under conditions of turbulent agitation in a mixing vessel.^{5 8 12} The basic approach of these investigators has been to assume that the coefficient of diffusion for the solute moving from the particle of undissolved salt to the bulk of the solution is the same in a continuous flow system as in a batch system, providing that the same degree of agitation and the same particle sizes are used.

Thesis investigators at Newark College of Engineering^{7 9} have presented batch data for the transfer of various ions to cationic exchange resins under conditions of agitation that indicate rate control by a resistance that may be represented by a stagnant film surrounding the resin particle.

The original purpose of this investigation was to determine the usefulness of batch data in predicting the performance of an agitated ion exchange column with continuous flow.

During the initial stages of this investigation Marchello and Davis¹¹ published equations for predicting the concentrations of effluent leaving an agitated ion exchange column. Basically, the Marchello and Davis equation

describes a gradual increase in effluent concentration due to the decrease in the rate of ion exchange as the solute concentration of the resin increases. The equation of Marchello and Davis has been converted to a weight instead of a volume basis, and is as follows:

$$\frac{c}{c_f} = 1 - \frac{(w_r k a)}{(Q + w_r k a)} e^{-b} \quad (1)$$

where:

$$b = \frac{Q}{m w_r} \int_0^t \frac{w_r k a \, dt}{Q + w_r k a}$$

$k a$ = diffusion coefficient in liters per gram-minute

$$= \frac{\frac{dq}{dt}}{c - c^*} \quad (2)$$

q = concentration of solute in resin in meq. per gram

t = time elapsed since beginning of run in minutes

c = concentration of effluent in meq. per liter

c^* = solution concentration which would be in equilibrium with $q = m q$

m = slope of equilibrium curve in liters of solution per gram of resin

c_f = concentration of feed solution to column

w_r = grams of resin

Q = feed rate to column in liters per minute

The basic assumptions in the Marchello and Davis equation are:

1. The equilibrium curve is a straight line whose slope is known.
2. A constant diffusion coefficient, k_a , exists from the time fresh resin is charged to the column until this resin becomes totally loaded.

Rates of ion exchange are generally considered to be controlled by one or more of the following mechanisms:²

1. Diffusion of the ions across a film of stagnant liquid surrounding each bead of resin.
2. Diffusion of the ions within the particle of resin.
3. Reaction at the ion exchange site on the matrix of the resin.

In the range of solution concentrations studied in this thesis, film diffusion will be the rate controlling mechanism initially, since the resin is just starting to load and neither particle diffusion nor reaction at the ion exchange site have begun. The driving force for diffusion across the film is $c - c^*$. As the resin loads, the rate of loading $\frac{dq}{dt}$ will decrease in proportion to the decrease in driving force $c - c^*$, providing that film diffusion remains the controlling mechanism. If, however, either particle diffusion or reaction rate become controlling, the rate of loading $\frac{dq}{dt}$ will decrease at a faster rate than the driving force for film diffusion, $c - c^*$. Therefore, Marchello and Davis's assumption that k_a , the ratio of

$\frac{dq}{dt}$ to $c - c^*$, is constant, implies that the rate of ion exchange is film controlled.

Chen, Belter and French⁴ suggest a method of determining whether diffusion is film controlled. The method is to calculate k_a with a modified version of Equation (2) at several time intervals while ion exchange is proceeding. If k_a remains constant, film diffusion is the rate controlling mechanism. The modified equation is:

$$k_a = \frac{\frac{\Delta q}{\Delta t}}{c - c^*} \quad (3)$$

where:

q = concentration of solute in the resin
at time t

t = time elapsed since ion exchange began

$\frac{\Delta q}{\Delta t}$ = slope of the resin loading curve (q vs. t)
at time t

c = concentration of solute in the solution
at time t

c^* = solution concentration which would be in
equilibrium with the resin concentration
 q

EXPERIMENTAL

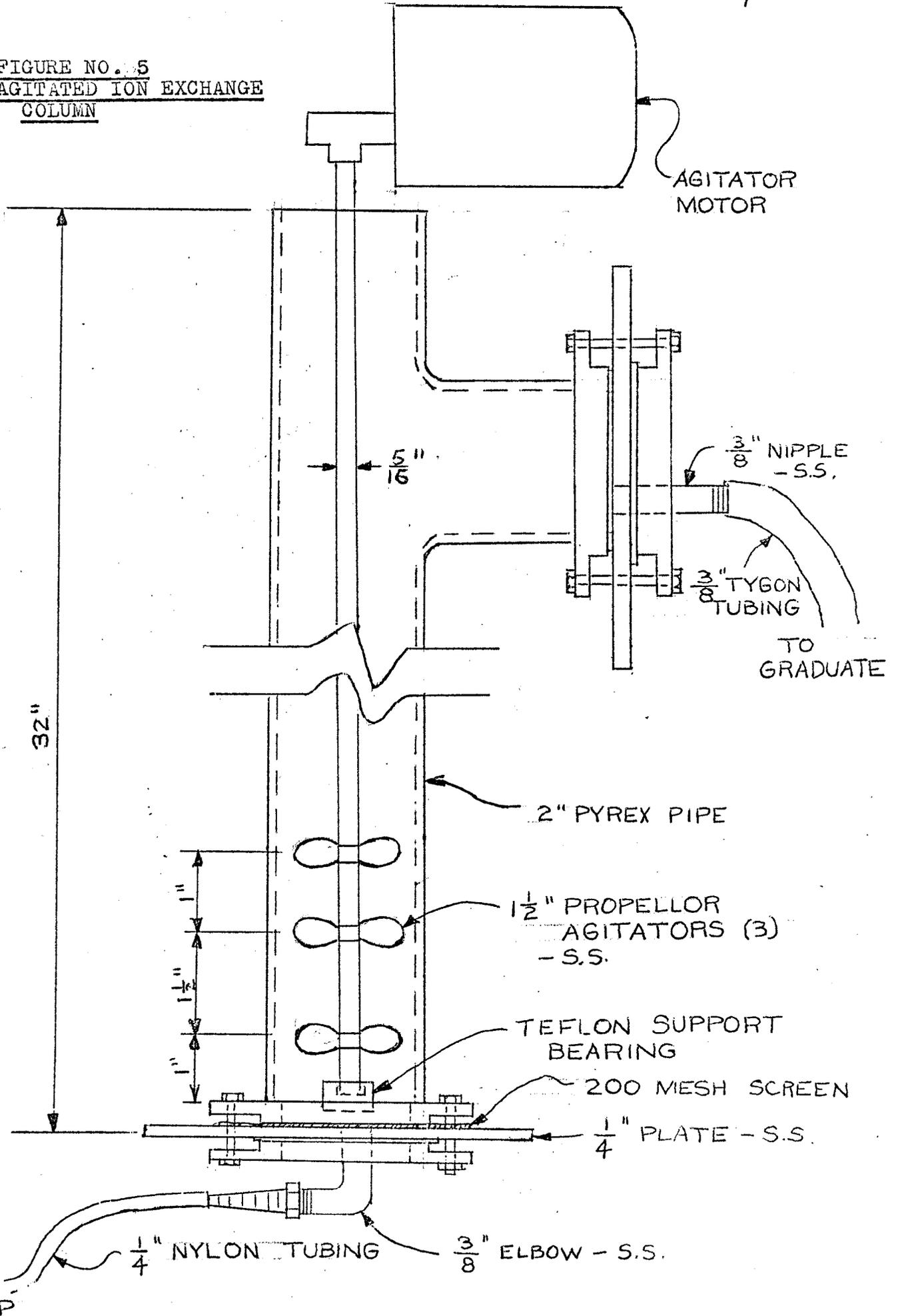
Equipment

Batch experiments were conducted in a 2000 ml. beaker, using a propellor type agitator with a variable speed drive. All experiments were made at an agitator speed of 600 RPM, as determined by a stroboscope. Temperatures varied from 72 to 78 F.

Column experiments were made in a column constructed of 2" Pyrex pipe, 32" long (see Figure 5). Feed entered the column through a 3/8" pipe nipple screwed into a blank flange at the bottom of the column; effluent left the column through a 3/8" nipple screwed into a blank flange on a side tee at the top of the column. Resin was retained in the column by 200 mesh stainless steel screening stretched over the inlet and outlet ports.

Agitation was provided by 3 1-1/2" propellor agitators mounted on a 36" long shaft driven by the same laboratory agitator motor used in the batch experiments at 600 RPM. The agitator shaft was supported at the bottom of the column by a Teflon bushing installed in the blank flange. The propellers were spaced 1 1/2" apart starting at 1" from the bottom of the shaft. During column runs, the resin stayed in suspension in the bottom half of the column.

FIGURE NO. 5
AGITATED ION EXCHANGE
COLUMN



Feed solution was pumped from a glass reservoir and into the column with a Clark-Cooper metering pump which gave flow rates reproducible to within 1 cc/min. Feed temperatures varied from 74 to 78 F. Connections from the reservoir to the pump and from the pump to the column were made with $\frac{1}{4}$ " nylon tubing. Effluent left the column through a $\frac{3}{8}$ " Tygon tubing to be collected in a 0-100 ml. graduate.

Since the effluent tubing did not flood at the flow rates used, the solution in the column only built up to the height of the outlet connection and then overflowed, free falling through the Tygon tubing. Therefore, the head space above the outlet connection was not filled with liquid and the top of the column could be left unsealed.

Preparation of Resin

Dowex 50X-8 ion exchange resin, H⁺ form, -20+ 50 mesh screen fraction, was used in all experiments. All runs were made on resin from the same 5 lb. batch. Residual acid on the resin was removed by slurring with deionized water and decanting until pH readings on the decantings indicated a constant value of about 5.5 (the pH of deionized water).

After the last decanting, the resin was partially dewatered in a vacuum filter flask. A sample was then retained for later moisture analysis by drying in a vacuum

drying oven for 18 hours. Prior to each batch or column run, wet resin was weighed out and a later correction made to convert to dry basis.

Preparation of Solution

Ferric nitrate solutions were made up by dissolving reagent grade $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ crystals in deionized water. The ferric nitrate was weighed to the nearest .001 gram on an analytical balance and the water was weighed to the nearest gram on a 500 gram balance. Batches of solution were made up in quantities ranging from 120 to 9000 grams for the various batch and column runs. All solutions were made up to contain 2.02 grams of ferric nitrate per liter of solution or to a normality of .015.

The deionized water was produced by agitating 1200 gram batches of distilled water with approximately 500 cc. of a "mixed bed" ion exchange resin for 5 minutes and then decanting off the water through a 200 mesh screen. This procedure was found to produce a resistivity of 1,000,000 ohms or more as measured with a conductivity meter. Periodically, resistivity was checked to determine whether the mixed bed resin was becoming spent. It was found that water could not be deionized and then stored for several days in polyethylene bottles, as previous experimenters⁹ had reported, without a significant decrease in purity. Consequently, water was deionized and ferric nitrate solution made up fresh for each run.

Analysis of Solution

To determine the relationship between pH and the degree of ion exchange experimentally, colorimetry was used to analyze samples of .015N ferric nitrate solution which had been contacted with the ion exchange resin in various proportions. Measurements of pH for all work described in this paper were made with a Beckman Model G pH meter zeroed in with either pH 7.0 or pH 4.0 buffer prior to each experiment. The instrument is accurate to within $\pm .02$ pH units.

The colorimetric procedure used was a modification of a procedure found in the literature.¹ The modified procedure follows:

A one ml. sample of the solution was diluted to 53 ml. total volume with distilled water. To this volume was added 10 ml. of 9.6% potassium thiocyanate solution. The percent transmission at 480 millimicrons wavelength was immediately read on a Bausch & Lomb Colorimeter. A calibration curve of percent transmission vs. concentration was initially made up with solutions of known concentration. Thereafter, unknowns were analyzed by converting transmission to concentration with this curve, which is shown in Figure No. 2.

The colorimeter analyses resulted in the experimental points shown in Figure No. 1.

Experimental Procedure

Batch runs. Ten (10) batch runs were made. In each run a known weight of resin was agitated with a known volume of .015N ferric nitrate solution and pH was read at frequent intervals by maintaining the electrodes submerged in the agitated solution. When the pH no longer changed with time, the run was considered to be in equilibrium.

FIGURE 1
IONS EXCHANGED VS
HYDROGEN ION CONC.
= .015N FERRIC NITRATE

LEGEND
 O Analysis by colorimeter
 + Theoretical - This Thesis
 X Theoretical - Previous Theses

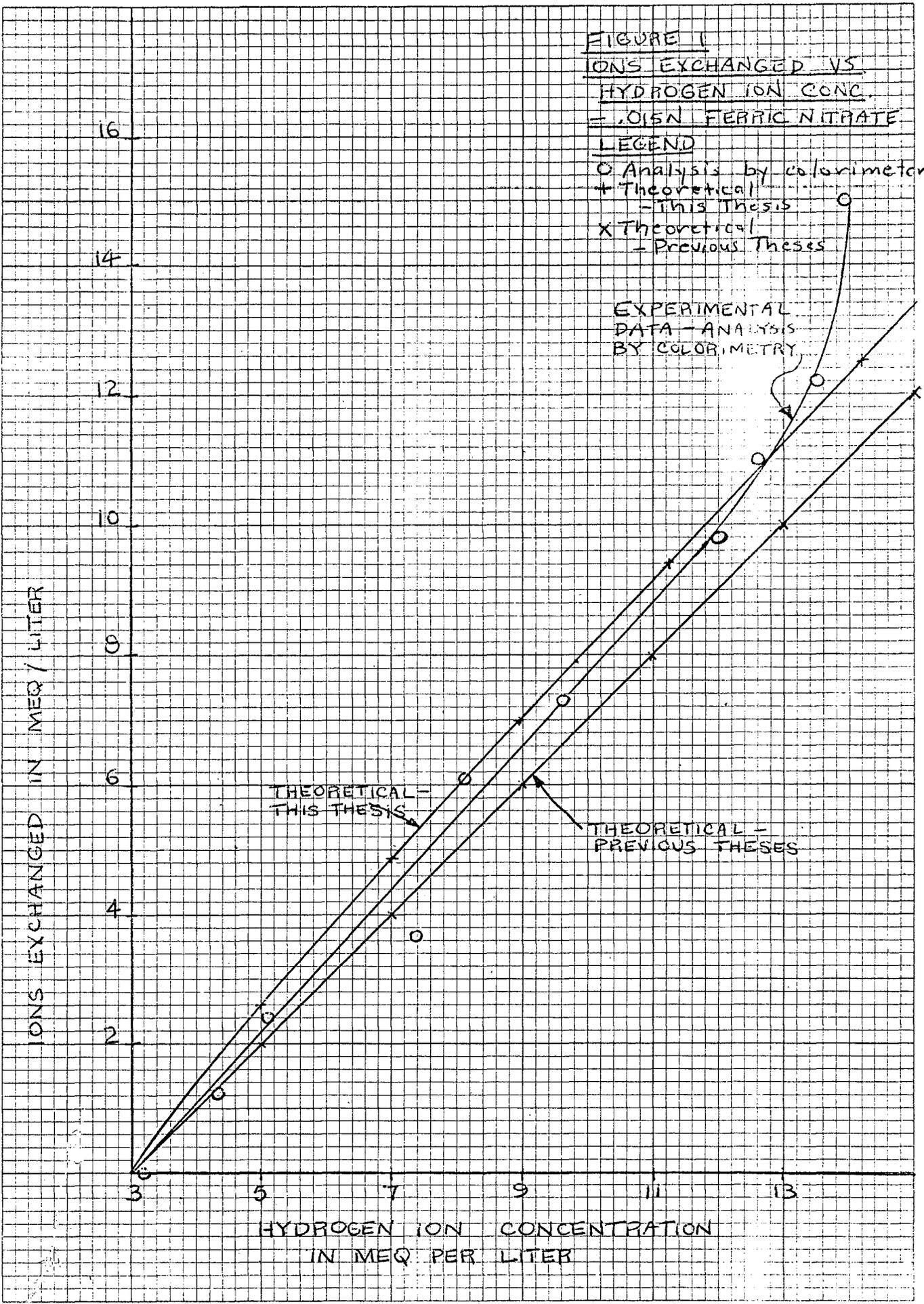
EXPERIMENTAL
 DATA - ANALYSIS
 BY COLORIMETRY

IONS EXCHANGED IN MEQ / LITER

THEORETICAL -
 THIS THESIS

THEORETICAL -
 PREVIOUS THESES

HYDROGEN ION CONCENTRATION
 IN MEQ PER LITER



Column runs. Column runs were made as follows:

1. The column was filled with .015N ferric nitrate solution.
2. The agitator was turned on.
3. The pump was started at the desired rate, rate being checked by collecting effluent in a graduate over a timed interval.
4. The resin, which was preweighed, was dropped through the top of the column.
5. Samples of effluent were collected at frequent intervals and pH readings taken.

CALCULATIONS AND RESULTS

Calculations

Calculation of q (concentration of solute in resin)

The concentration of ferric ions in the resin, q was calculated as follows for the batch runs:

$$q = \frac{(c_f - c)V}{w_r} \quad (4)$$

where:

q = concentration of ferric ions in resin
in meq. per gram at time t

c_f = concentration of ferric ions in solution
at start of experiment in meq. per liter

= 15 for all experiments

c = concentration of ferric ion in solution
at time t in meq. per liter

V = volume of solution in liters

w_r = weight of dry resin in grams

These calculations are tabulated at several time intervals for each of 7 of the batch runs in Tables Nos. 1 - 7. Plots of q vs. t (resin loading curves) are shown in Figures Nos. 6 - 12.

Calculation of q - column runs. If perfect mixing is assumed to take place in the column, a material balance around the column can be described by the following equation:

$$v \frac{dc}{dt} = Qc_f - Qc - w_r \frac{dq}{dt} \quad (5)$$

where:

V = liquid holdup in column = 1.35 liters

c = concentration of effluent at time t in meq. of ferric ion per liter = c_f when $t = 0$

c_f = feed concentration = 15.0 meq. of ferric ion per liter

Q = feed rate in liters per minute

w_r = weight of dry resin in grams

q = concentration of ferric ion in resin at time t in meq. per gram

Then, integrating and rearranging terms:

$$q = \frac{Q \int_{t=0}^{t=t} (c_f - c) dt - V \int_{c=c_f}^{c=c} dc}{w_r} \quad (6)$$

Values of q were calculated at various time intervals during each run by the following approximation of Equation

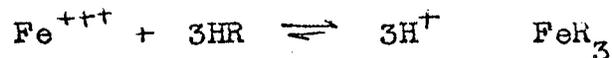
(6):

$$q = \frac{Q \sum (c_f - c) \Delta t + V(c_f - c)}{w_r} \quad (7)$$

using intervals of Δt varying from 2 to 10 minutes and averaging the value of c for each interval Δt .

A tabulation of these calculations is given in Tables Nos. 16 - 22. Plots of q vs. t (resin loading curves) for 7 column runs are shown in Figures Nos. 13 - 19.

Construction of equilibrium curve. Ion exchange equilibrium is considered to be subject to the same relationships as chemical reactions. That is, if ion exchange is described as follows:⁶



where R represents an equivalent amount of resin.

Then:

$$\frac{[\text{Fe}^{+++}]_R}{[\text{H}^+]_R^3} = K \frac{[\text{Fe}^{+++}]_S}{[\text{H}^+]_S^3} \quad (8)$$

where K is the equilibrium constant and the subscripts R and S refer to ion concentrations in the resin and solution respectively.

However, K varies as $[\text{Fe}^{+++}]_R$ varies.⁶ This variation has been approximated by Mar and David¹⁰ with a log - log plot of $\frac{[\text{Fe}^{+++}]_R}{[\text{H}^+]_R^3}$ vs. $\frac{[\text{Fe}^{+++}]_S}{[\text{H}^+]_S^3}$. Each of the 10 batch

runs described above was carried to equilibrium. Table No. 8 is a tabulation of the calculations of $\frac{[\text{Fe}^{+++}]_R}{[\text{H}^+]_R^3}$ and

$\frac{[\text{Fe}^{+++}]_S}{[\text{H}^+]_S^3}$ in which the terms are defined as follows:

TABLE NO. 8
EQUILIBRIUM DATA SUMMARY

<u>Exp.</u> <u>No.</u>	$[H^+]_S$	$[Fe^{++}]_S$	$[Fe^{++}]_R$	$[H^+]_R$	$\frac{[Fe^{++}]_S}{[H^+]_S^3}$	$\frac{[Fe^{++}]_R}{[H^+]_R^3}$
1	4.1	13.8	4.8	0.4	0.200	75.0
2	4.9	12.9	4.9	0.3	0.110	181
3	5.4	12.2	4.8	0.4	0.0776	75.0
4	5.9	11.7	4.5	0.7	0.0570	131
5	7.8	9.5	6.15	0.0	0.0201	∞
6	12.6	4.0	4.9	0.3	0.00202	202
7	12.3	4.8	2.2	3.0	0.00258	0.0814
8	9.8	6.6	2.3	2.9	0.00708	0.0942
9	15.5	0.0	0.7	4.5	0.0	0.00836
10	13.8	2.1	0.3	4.9	0.000798	0.00297

- $[H^+]_S$ = the concentration of hydrogen ion in solution at equilibrium, as measured with a pH meter. (meq. per liter)
- $[Fe^{+++}]_S$ = c, the concentration of ferric ion in solution at equilibrium, as determined by measurements of $[H^+]_S$ and the use of the experimental curve in Figure No. 1 (meq. per liter)
- $[Fe^{+++}]_R$ = q, the concentration of ferric ion in the resin at equilibrium, as determined by Equation (4). (meq. per gram)
- $[H^+]_R$ = the concentration of hydrogen ion in the resin at equilibrium (meq. per gram)
- = T. C. - $[Fe^{+++}]_R$
 where T. C. is the total capacity of the resin = 5.2 meq. per gram.¹⁴

Figure No. 3 is a plot of $\log \frac{[Fe^{+++}]_R}{[H^+]_R^3}$ vs. $\log \frac{[Fe^{+++}]_S}{[H^+]_S^3}$.

Three of the experiments have been omitted from this plot either because the value of $\frac{[Fe^{+++}]_R}{[H^+]_R^3}$ approached infinity or because the value of $\frac{[Fe^{+++}]_S}{[H^+]_S^3}$ approached zero, due to imprecision in the experimental data.

The log-log plot in Figure No. 3 has been transposed to a simple equilibrium plot of $[Fe^{+++}]_R$ vs. $[Fe^{+++}]_S$ in Figure No. 4

Calculation of k_a (diffusion coefficient). Diffusion coefficient, k_a , was calculated at several time intervals for each of the 7 batch runs and 7 column runs, using Equation (3), which is repeated below:

FIGURE NO. 3
 LOG-LOG PLOT OF
 $\frac{[\text{Fe}^{++}]_R}{[\text{H}^+]_R^3}$ vs. $\frac{[\text{Fe}^{++}]_S}{[\text{H}^+]_S^3}$

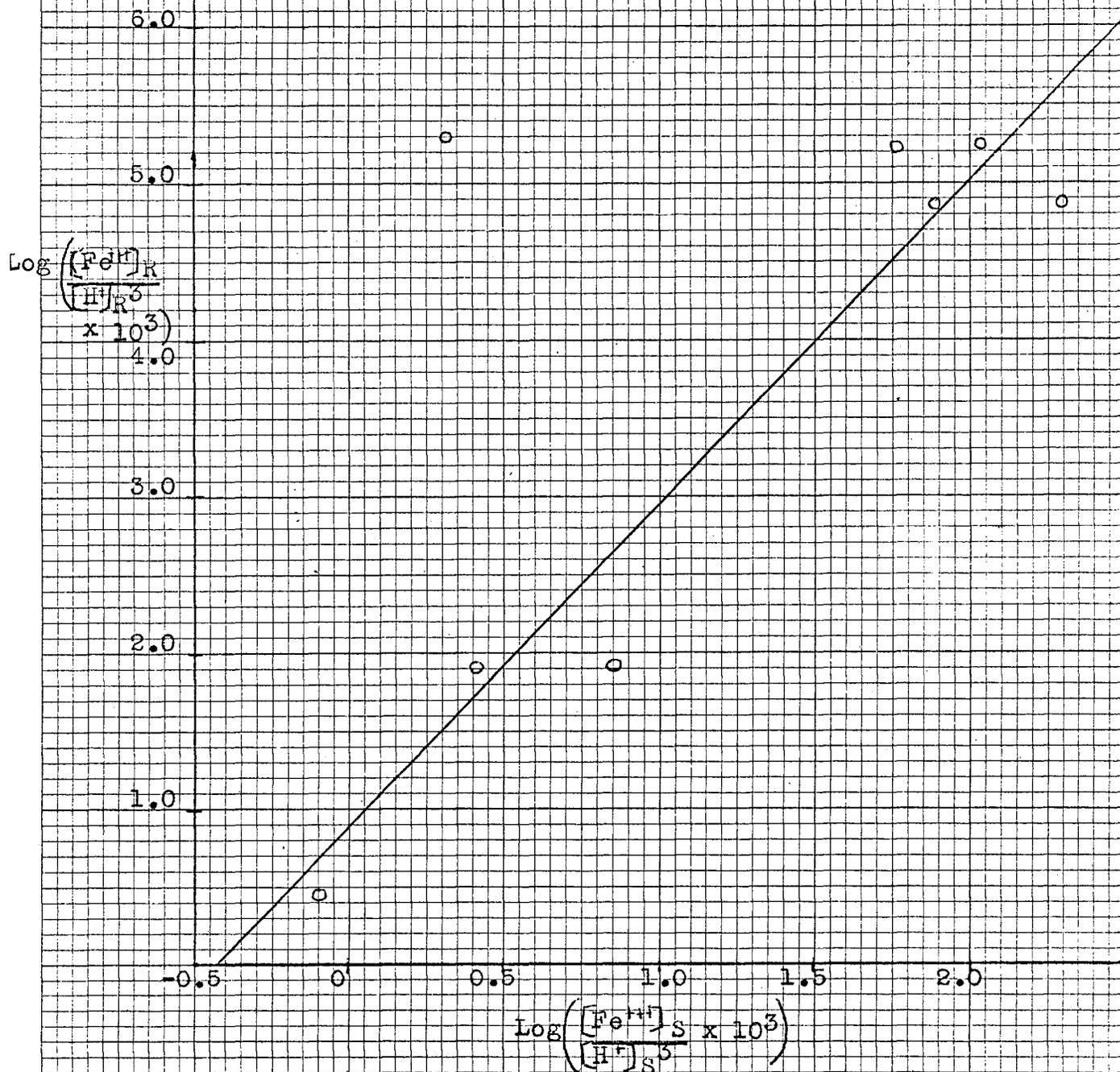


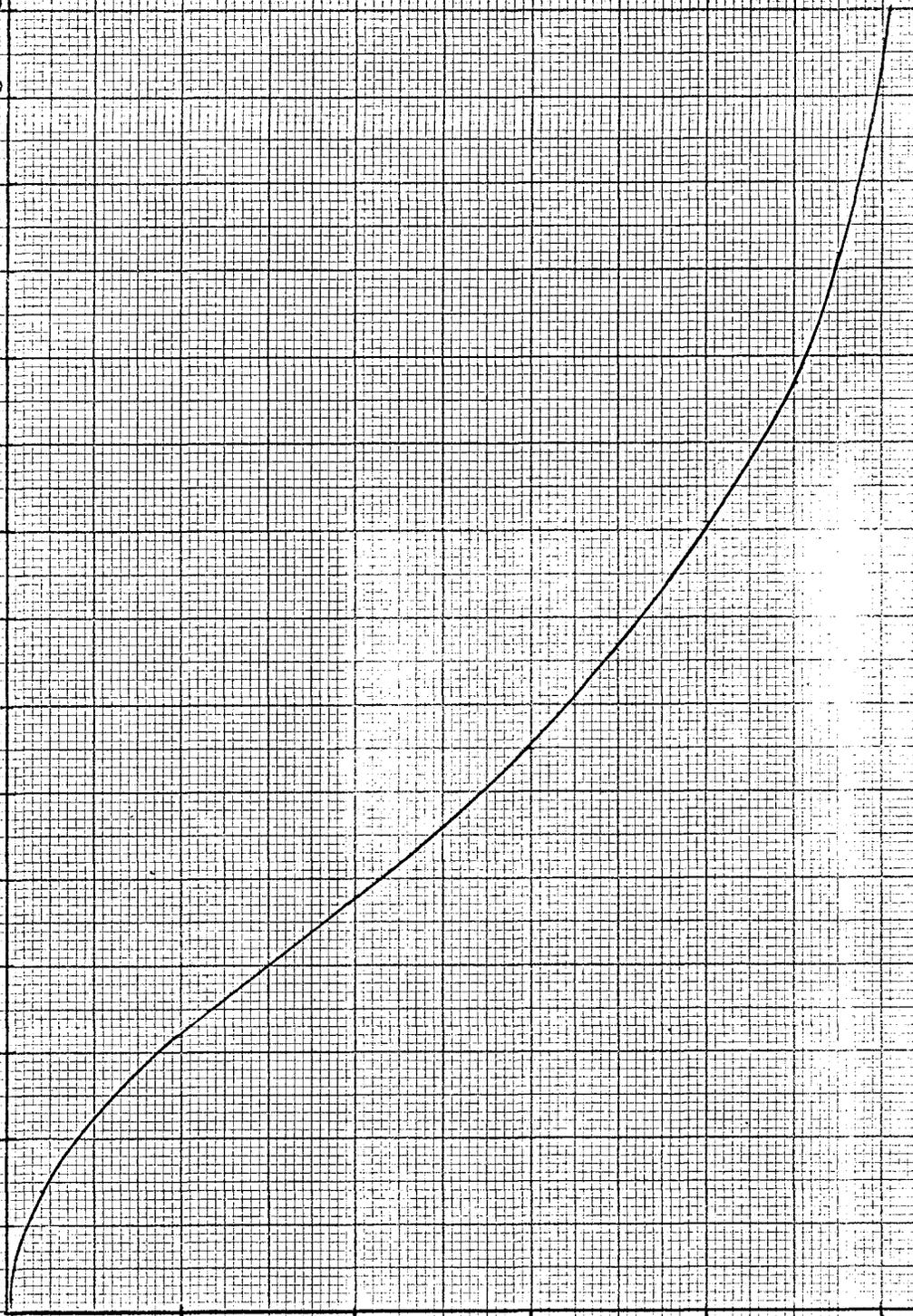
FIGURE 4
EQUILIBRIUM CURVE
FERRIC ION DISTRIBUTION
BETWEEN DOWEX 50X-8
AND 0.015N FERRIC NITRATE
SOLUTION

CONCENTRATION OF Fe^{3+} IN SOLUTION IN MEQ PER LITER

15.0
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0

CONCENTRATION OF Fe^{3+} IN RESIN IN MEQ PER GRAM

1.0 2.0 3.0 4.0 5.0



$$k_a = \frac{\frac{\Delta q}{\Delta t}}{c - c^*} \quad (3)$$

where:

q = concentration of ferric ion in the resin in meq. per gram, at time t

t = time elapsed since beginning of run in minutes

c = concentration of ferric ion in solution at time t , in meq. per liter

c^* = solution concentration which would be in equilibrium with q

$\frac{\Delta q}{\Delta t}$ = slope of the resin loading curve at time t , in meq. per gram per minute

Calculations of k_a are tabulated in Tables Nos. 9 - 15 for the batch runs and in Tables Nos. 23 - 29 for the column runs. A resin loading value $\frac{q}{q_\infty}$ is also calculated at each time interval, where q_∞ is the resin concentration which would be in equilibrium with the solution concentration c . The term $\frac{q}{q_\infty}$ is a means of expressing fractionally how close the resin is to total loading.

A composite summary of all calculations of k_a is given in Table No. 30.

TABLE NO. 30
CALCULATION OF DIFFUSION COEFFICIENT

SUMMARY OF RESULTS

BATCH RUNS

<u>Exp.</u>	<u>Wt. of Resin</u> (grams)	<u>Vol. of Solution</u> (ml.)	<u>Diffusion Coefficient (ka)</u>						
			$\frac{q}{q_{\infty}} =$ <u>0.1</u>	<u>0.2</u>	<u>0.3</u>	<u>0.4</u>	<u>0.5</u>	<u>0.6</u>	<u>Ave.</u>
1	0.292	1157	.044	.027	.021	.021	.021	.021	<u>.026</u>
2	0.545	1217	.038	.040	.040	.034	.030	.028	<u>.035</u>
3	0.745	1273	.021	.024	.026	.029	.031	.040	<u>.028</u>
4	0.886	1207	.015	.018	.018	.019	.020	.020	<u>.018</u>
7	5.59	1220	.015	.018	.019	.020	.019	.021	<u>.019</u>
8	5.41	1304	.008	.010	.009	.006	.007	.007	<u>.008</u>
10	46.6	1241	.009	.007	.006	.004	.004	-	<u>.006</u>

COLUMN RUNS

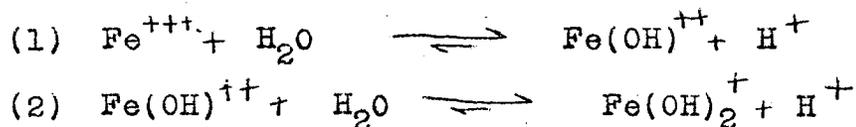
<u>Exp.</u>	<u>Wt. of Resin</u> (grams)	<u>Feed Rate</u> (ml/min)	$\frac{q}{q_{\infty}} =$						
			<u>0.1</u>	<u>0.2</u>	<u>0.3</u>	<u>0.4</u>	<u>0.5</u>	<u>0.6</u>	<u>Ave.</u>
11	0.59	10	.032	.031	.030	.026	.021	.017	<u>.026</u>
12	5.41	10	.022	.029	.032	.047	.061	.070	<u>.044</u>
13	0.60	100	.040	.044	.047	.042	.039	.036	<u>.041</u>
14	2.95	100	.030	.034	.036	.043	.048	.050	<u>.040</u>
15	5.90	100	.015	.018	.019	.018	.020	.017	<u>.018</u>
16	5.90	100	.030	.031	.031	.038	.045	.050	<u>.038</u>
17	54.1	100	.011	.011	.012	.012	.014	.019	<u>.013</u>

DISCUSSION OF RESULTS

Analysis of Ferric Nitrate Solutions

In previous theses^{7 9} it had been assumed that the increase in hydrogen ion concentration in solution, as indicated by pH, was equivalent to the amount of ferric ion picked up by the resin. However, in this investigation it was observed that the total change in hydrogen ion concentration was never more than 12 meq. per liter, regardless of how much resin was used. Since it would be expected that the degree of ion exchange would approach completion if enough resin is used, and since the total ferric ion concentration in solution is 15 meq. per liter, it should follow that the change in hydrogen ion concentration in solution should also go as high as 15 meq. per liter.

When ferric nitrate is dissolved in water, hydrolysis may be described as follows:



If this solution is then contacted with a hydrogen form ion exchange resin, the hydrogen ions in the resin should replace the ferric ions in solution. Now, however, the surplus of hydrogen ions would, according to Le Chatelier's principle, tend to reverse the above reactions. If this reversal is significant, the monitored change in hydrogen ion concentration would not be equal to the total amount of

hydrogen ion given up by the resin, as previously presumed since some of the hydrogen would be participating in the hydrolysis reaction.

If the following material balances are assumed in the solution:

$$[\text{Fe}^{+++}] = c - [\text{Fe}(\text{OH})^{++}] - [\text{Fe}(\text{OH})_2^+] - \frac{x}{3} \quad (9)$$

$$[\text{H}^+] = [\text{Fe}(\text{OH})^{++}] + [\text{Fe}(\text{OH})_2^+] + x \quad (10)$$

where:

[] = concentration of ion in brackets in gram-moles per liter

c = original total concentration of ferric nitrate in gram-moles per liter

x = amount of ion exchange which has taken place in gram-equivalents per liter

Then the following equation should describe the relationship between hydrogen ion concentration and the amount of ion exchange which has taken place:

$$x = \frac{3[\text{H}^+] - 3ac + 3abc - 2bc}{3 + ab + a - 2b} \quad (13)$$

where:

$$a = \frac{K_1}{[\text{H}^+] + K_1}$$

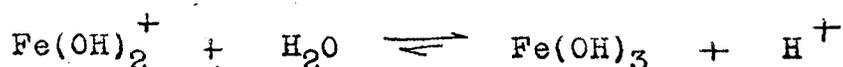
$$b = \frac{a}{\frac{K_2}{K_2} + a}$$

$$K_1 = \text{hydrolysis constant} = \frac{[\text{Fe}(\text{OH})^{++}][\text{H}^+]}{[\text{Fe}^{+++}]} \quad (11)$$

$$K_2 = \text{hydrolysis constant} = \frac{[\text{Fe}(\text{OH})_2^+][\text{H}^+]}{[\text{Fe}(\text{OH})^{++}]} \quad (12)$$

Equation (13) is a simultaneous solution of equations (9), (10), (11) and (12). The hydrolysis constants K_1 and K_2 were taken to be 3.2×10^{-3} and 0.4×10^{-3} respectively.³

This relationship between $[H^+]$ and x is plotted in Figure No. 1 and is seen to deviate somewhat from the relationship assumed in previous theses,^{7, 9} which is simply a 45 degree line. The curve described by colorimetric analyses lies about midway between the two theoretical curves up to an exchange of about 12 meq. per liter. At this point additional ion exchange produces proportionately less change in hydrogen ion concentration than either of the two theoretical curves predicts. This deviation is probably due to an incomplete description of hydrolysis as outlined in the above discourse. One possible error is the omission of a third hydrolysis reaction from the theoretical approach, as follows:



Non-linearity of Equilibrium Curve.

The equilibrium curve (Figure No. 4) is obviously not linear. Previous experimenters have also found that equilibrium curves for polyvalent ion exchange are far from being linear, particularly in the same low range of

concentrations.¹³ However, it is now impossible to use Marchello and Davis's prediction equation for column performance, which is predicated on a linear equilibrium relationship. Instead, it would now be necessary to use some numerical approximation to construct effluent concentration curves. Such an approach was taken by Chen, Belter and French⁴ in elaborating on Marchello and Davis's original work. Numerical solutions of this type call for digital computers and are beyond the time allotment for this thesis.

Diffusion coefficients. Examination of Table No. 30 reveals the following:

1. For three of the batch runs (Nos. 3, 4 and 7) and four of the column runs (Nos. 11, 14, 16 and 17) there is a definite upward trend in the value of the diffusion coefficient as ion exchange proceeds. This effect may be due to resin breakage, thus increasing the surface area available for diffusion between the resin and the solution. Inaccuracies in the equilibrium curve may also be responsible for this effect.
2. In only one of the batch runs (No. 10) and one of the column runs (No. 11) is there a consistent decrease in diffusion coefficient as ion exchange proceeds. Since the coefficient does not diminish in magnitude up to 60% loading in any of the other

16 runs, it can be concluded that diffusion through a film of solution surrounding each particle is the rate controlling mechanism, rather than diffusion within the particle or reaction at the ion exchange site. (See page 4 for the reasoning behind this conclusion).

3. The average diffusion coefficient for both the batch and column runs tends to become lower as the ratio of resin volume to solution volume exceeds .005. This effect is probably due to less efficient agitation as the volume of resin increases, so that each bead of resin is not exposed to the maximum turbulence in the surrounding solution. As proof of this hypothesis, the propellor agitator heads were relocated on the shaft to give greater turbulence to the bed of resin prior to Experiment No. 16, and values of k_a for this experiment were twice as great as for Experiment No. 15, which it duplicates.
4. The average diffusion coefficient for those column runs in which the ratio of resin volume to solution volume was low enough to allow complete agitation (Nos. 11, 12, 13, 14 and 16) is higher than for the batch runs with complete agitation (Nos. 1, 2 and 3) (.038 vs. .030). This higher value of k_a in the column runs may have been due to greater turbulence

in the column runs due to differences in geometry between the column and the beaker used for the batch runs or due to the use of three propellor agitator heads in the column runs versus only one head in the batch runs. Greater turbulence of agitation would result in a thinner film of stagnant solution around each resin particle and thus a smaller resistance to diffusion across the film.

CONCLUSIONS AND RECOMMENDATIONS

From the above results, it may be concluded that the rate of exchange of ferric and hydrogen ions between a .015N solution of ferric nitrate and Dowex 50X-8 resin, -20 50 mesh screen fraction, is controlled by film diffusion.

The magnitude of the coefficient of diffusion is between .026 and .035 liters per gram-minute in batch runs with an agitator speed of 600 RPM and between .038 and .044 liters per gram-minute in column runs with an agitator speed of 600 RPM.

The magnitude of the diffusion coefficient in both the batch runs and the column runs depends upon the degree of turbulence achieved in the agitated slurry. The degree of turbulence depends upon the ratio of resin volume to solution volume, the geometry of the vessel and the location of the agitator heads. If corrections could be made, either experimentally or mathematically, for these factors, the diffusion coefficients calculated for the column runs would more closely reproduce the values found in the batch runs.

Fore-knowledge of the diffusion coefficient from batch data should allow prediction of concentration vs. time curves for column runs at various feed rates and quantities of resin, provided that the same degree of turbulence has been maintained in batch and column systems. The method of prediction would probably have to be by numerical approximation methods, since the non-linearity of the equilibrium relation-

ship prevents the use of straight-forward mathematical formulae, such as Marchello and Davis's equation, discussed earlier in this paper.

The following numerical procedure is recommended for constructing predicted concentration-vs-time curves for agitated ion exchange columns in which diffusion rates are film controlled:

1. It is assumed that the diffusion coefficient k_a , as defined by Equation (2) or Equation (3), is known beforehand and that equilibrium data is also available.
2. Calculation of c , the effluent concentration, begins at time $t = 0$, at which time q , the concentration of solute in the resin, equals zero; also, when $t = 0$, $c^* = 0$ and $c = c_f$.
3. Calculations are made with a small time interval Δt , such as one minute.
4. Calculations begin by determining the amount of solute picked up by the resin from $t = 0$ to $t = 1$, using the following rearrangement of Equation (3):

$$(\Delta q)_1 = (k_a)(c_0 - c_0^*)\Delta t$$

5. Next, $(c)_1$, the effluent concentration change from $t = 0$ to $t = 1$, is calculated by the following modification of Equation (5):

$$(\Delta c)_1 = \frac{Q(c_f - c_0)\Delta t - w_r(\Delta q)_1}{Q\Delta t + V}$$

6. Then,

$$c_1 = c_0 + (\Delta c)_1$$

7. Steps 4, 5 and 6 are repeated to calculate c_2 , c_3 and so forth until the entire concentration-vs.-time curve has been constructed.

In the above procedure, it is assumed that the concentration is changing in incremental steps from the end of one time interval to the end of the next. To minimize the error caused by this assumption it is necessary to select values of Δt as small as possible. Consequently, the entire procedure becomes too tedious for hand calculation and calls for the use of a digital computer. Equilibrium data would have to be entered into the computer's memory.

For future investigators of the ion exchange system studied in this thesis, it would be worthwhile to develop more equilibrium data before proceeding to any kinetic studies. Some of the unexpected effects found in this work, such as the decreasing diffusion coefficient found in two of the runs, may have been due to inaccuracies in the equilibrium curve.

It would also be valuable for analytical purposes to have more information on the relationship between ferric ion concentration and hydrogen ion concentration (or pH) in a solution undergoing ion exchange.

TABLE NO. 1CALCULATION OF α EXPERIMENT NO. 1 $w_T = 0.292$ grams $V = 1.157$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>α</u>
1	2.48	3.31	14.7	1.2
2	2.48	3.31	14.7	1.2
3	2.48	3.31	14.7	1.2
4	2.47	3.39	14.6	1.6
5	2.47	3.39	14.6	1.6
10	2.42	3.80	14.2	3.2
15	2.42	3.80	14.2	3.2
20	2.40	3.98	13.8	4.8
25	2.40	3.98	13.8	4.8
30	2.39	4.10	13.8	4.8
35	2.39	4.10	13.8	4.8
40	2.39	4.10	13.8	4.8

TABLE NO. 2
CALCULATION OF q
EXPERIMENT NO. 2

$w_r = 0.545$ grams $V = 1.217$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
1	2.52	3.0	15.0	0
2	2.48	3.3	14.7	0.7
3	2.46	3.5	14.5	1.1
4	2.43	3.7	14.3	1.6
5	2.43	3.7	14.3	1.6
6	2.42	3.8	14.1	2.0
7	2.41	3.9	14.0	2.2
8	2.40	4.0	13.9	2.5
9	2.39	4.1	13.8	2.7
10	2.36	4.4	13.4	3.6
17	2.33	4.7	13.1	4.2
23	2.32	4.8	13.0	4.5
30	2.31	4.9	12.9	4.7
35	2.31	4.9	12.9	4.7
40	2.31	4.9	12.9	4.7

TABLE NO. 3
CALCULATION OF q
EXPERIMENT NO. 3

$w_r = 0.745$ grams $V = 1.273$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
1	2.51	3.1	14.9	0.2
2	2.50	3.2	14.8	0.3
3	2.49	3.2	14.8	0.3
4	2.43	3.7	14.2	1.4
5	2.42	3.8	14.1	1.5
6	2.41	3.9	14.0	1.7
7	2.40	4.0	13.9	1.9
8	2.39	4.1	13.8	2.05
9	2.37	4.3	13.5	2.6
10	2.36	4.4	13.5	2.6
15	2.31	4.9	12.8	3.8
20	2.28	5.3	12.4	4.4
25	2.27	5.4	12.2	4.8

TABLE NO. 4
CALCULATION OF q
EXPERIMENT NO. 4

$w_r = 0.886$ grams

$V = 1.207$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
1	2.50	3.2	14.9	0.1
2	2.49	3.2	14.8	0.3
3	2.48	3.3	14.7	0.4
4	2.46	3.5	14.6	0.5
5	2.43	3.7	14.3	0.95
6	2.42	3.8	14.2	1.1
7	2.41	3.9	14.0	1.4
8	2.40	4.0	13.9	1.5
9	2.39	4.1	13.8	1.6
10	2.38	4.2	13.7	1.8
15	2.32	4.8	13.0	2.7
20	2.29	5.1	12.6	3.3
25	2.27	5.4	12.3	3.7
30	2.26	5.5	12.1	3.95
35	2.24	5.8	11.8	4.4
40	2.23	5.9	11.7	4.5
45	2.23	5.9	11.7	4.5

TABLE NO. 5
CALCULATION OF q
EXPERIMENT NO. 7

$w_r = 5.59$ grams $V = 1.229$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
0.5	2.48	3.3	14.7	0.07
1.0	2.45	3.6	14.4	0.13
1.5	2.42	3.8	14.2	0.18
2.0	2.37	4.3	13.6	0.31
2.5	2.33	4.7	13.1	0.42
3.0	2.30	5.0	12.8	0.48
3.5	2.27	5.4	12.4	0.57
4.0	2.24	5.7	12.0	0.66
4.5	2.22	6.1	11.6	0.75
5.0	2.20	6.3	11.4	0.79
5.5	2.18	6.6	11.0	0.88
6.0	2.17	6.7	10.9	0.90
6.5	2.16	7.0	10.6	0.97
7.0	2.13	7.4	10.2	1.05
7.5	2.12	7.6	10.0	1.10
8.0	2.11	7.7	9.8	1.14
8.5	2.10	7.9	9.6	1.19
9.5	2.08	8.3	9.2	1.27
10.0	2.08	8.3	9.2	1.27
11.0	2.06	8.8	8.6	1.39

(continued)

TABLE NO. 5

(continued)

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>g</u>
12.0	2.05	9.0	8.0	1.54
13.0	2.04	9.1	7.8	1.58
14.0	2.03	9.4	7.6	1.63
15.0	2.02	9.6	7.3	1.69
16.0	2.01	9.8	7.1	1.74
17.0	1.99	10.2	7.1	1.74
18.0	1.98	10.4	6.8	1.80
19.0	1.98	10.4	6.8	1.80
20.0	1.97	10.7	6.6	1.85
25.0	1.93	11.7	5.4	2.11
30.0	1.91	12.3	4.8	2.24

TABLE NO. 6
CALCULATION OF q
EXPERIMENT NO. 8

$w_r = 5.41$ grams $V = 1.304$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
1	2.50	3.2	14.6	0.10
2	2.43	3.7	14.3	0.17
3	2.42	3.8	14.1	0.22
4	2.35	4.5	13.3	0.41
6	2.34	4.6	13.2	0.43
7	2.31	4.9	12.9	0.51
8	2.25	5.6	12.2	0.68
9	2.25	5.6	12.2	0.68
10	2.20	6.3	11.3	0.89
11	2.19	6.5	11.1	0.94
12	2.15	7.1	10.5	1.09
13	2.15	7.1	10.5	1.09
14	2.13	7.4	10.2	1.16
15	2.12	7.6	10.0	1.21
20	2.11	7.7	9.8	1.49
25	2.04	9.1	8.3	1.61
30	2.02	9.5	7.8	1.73
35	2.00	10.0	7.4	1.83
40	1.99	10.2	7.1	1.90
45	1.97	10.6	6.6	2.02

(continued)

TABLE NO. 6

(continued)

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>a</u>
52	1.92	12.0	5.1	2.38
55	1.95	11.2	6.0	2.17
63	1.93	11.7	5.4	2.32
65	1.93	11.7	5.4	2.32

TABLE NO. 7
CALCULATION OF q
EXPERIMENT NO. 10

$w_r = 46.6$ grams

$V = 1.241$ liters

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>q</u>
0.5	2.28	5.2	12.6	0.06
1.0	2.13	7.4	10.2	0.13
1.5	2.08	8.4	9.0	0.16
2.0	2.00	10.0	7.4	0.20
3.0	1.90	12.6	4.2	0.29
4.0	1.89	12.9	3.7	0.30
5.0	1.90	12.6	4.2	0.29
10.0	1.87	13.5	2.6	0.33
15.0	1.86	13.8	2.1	0.34

TABLE NO. 9

CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 1

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
1	0.7	14.9	2.6	12.3	.54	.044	5.1	.14
2	1.1	14.7	3.4	11.3	.30	.026	5.1	.22
3	1.4	14.6	3.9	10.7	.23	.022	5.1	.27
4	1.6	14.6	4.2	10.4	.22	.021	5.1	.31
5	1.8	14.5	4.5	10.0	.22	.022	5.1	.35
10	2.8	14.3	6.1	8.2	.175	.021	5.0	.56
15	3.6	14.1	8.0	6.1	.15	.025	5.0	.72
20	4.1	14.0	9.4	4.6	.10	.022	5.0	.82
25	4.5	13.9	10.7	3.2	.064	.020	5.0	.90
30	4.8	13.8	12.5	1.3	.040	.031	5.0	.96
35	4.9	13.8	13.2	0.6	.012	.020	5.0	.98

TABLE NO. 10

CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 2

<u>t</u>	<u>a</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta c}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	<u>$\frac{q}{q_{\infty}}$</u>
1	0.4	14.8	2.0	12.8	.49	.038	5.1	.08
2	0.9	14.6	3.0	11.6	.47	.040	5.1	.18
3	1.3	14.4	3.7	10.7	.43	.040	5.0	.26
4	1.7	14.2	4.4	9.8	.39	.040	5.0	.34
5	2.0	14.1	4.8	9.3	.32	.034	5.0	.40
6	2.3	14.0	5.2	8.8	.27	.031	5.0	.46
7	2.5	13.9	5.6	8.3	.25	.030	5.0	.50
8	2.8	13.75	6.1	7.6	.22	.029	5.0	.56
9	3.0	13.7	6.5	7.2	.20	.028	5.0	.60
10	3.2	13.6	7.0	6.6	.18	.027	4.9	.65
17	4.1	13.2	9.4	3.8	.09	.024	4.9	.84
23	4.5	13.0	10.7	2.3	.05	.022	4.9	.92
30	4.6	12.9	11.2	1.7	.01	.006	4.9	.94

TABLE NO. 11
CALCULATION OF DIFFUSION COEFFICIENT
EXPERIMENT NO. 3

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	<u>$\frac{q}{t}$</u>	<u>ka</u>	<u>q</u>	<u>$\frac{q}{q}$</u>
1	0.2	14.9	1.7	13.2	.26	.020	5.1	.06
2	0.5	14.8	2.2	12.6	.26	.021	5.1	.10
3	0.8	14.8	2.8	12.0	.26	.022	5.1	.16
4	1.1	14.2	3.6	10.6	.26	.024	5.0	.22
5	1.4	14.1	3.9	10.2	.26	.025	5.0	.28
6	1.7	14.0	4.4	9.6	.26	.027	5.0	.34
7	2.0	13.9	4.8	9.1	.26	.029	5.0	.40
8	2.2	13.8	5.1	8.7	.26	.030	5.0	.44
9	2.4	13.5	5.4	8.1	.25	.031	4.9	.49
10	2.7	13.5	6.0	7.5	.24	.032	4.9	.55
15	3.7	12.8	8.2	4.6	.18	.039	4.8	.56
20	4.4	12.4	10.3	2.1	.11	.052	4.8	.92

TABLE NO. 12
CALCULATION OF DIFFUSION COEFFICIENT
EXPERIMENT NO. 4

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	<u>$\frac{\Delta q}{\Delta t}$</u>	<u>ka</u>	<u>q_∞</u>	<u>$\frac{q}{q_{\infty}}$</u>
1	0.1	14.9	0.8	14.1	.19	.013	5.1	.02
2	0.3	14.8	1.6	13.2	.19	.014	5.1	.06
3	0.5	14.6	2.2	12.4	.19	.015	5.1	.10
4	0.7	14.5	2.6	11.9	.19	.016	5.0	.14
5	0.9	14.3	3.0	11.3	.19	.017	5.0	.18
6	1.1	14.2	3.4	10.8	.19	.018	5.0	.22
7	1.3	14.0	3.7	10.3	.18	.018	5.0	.26
8	1.4	14.0	3.9	10.1	.18	.018	5.0	.28
9	1.6	13.8	4.2	9.6	.18	.019	5.0	.32
10	1.8	13.7	4.5	9.2	.17	.018	5.0	.36
15	2.6	13.1	5.8	7.3	.15	.021	4.9	.53
20	3.3	12.6	7.2	5.4	.11	.020	4.8	.69
25	3.8	12.2	8.5	3.7	.09	.024	4.8	.79
30	4.1	12.0	9.4	2.6	.06	.023	4.7	.87
35	4.4	11.8	10.3	1.5	.035	.023	4.7	.94
40	4.5	11.7	10.7	1.0	.016	.016	4.7	.96

TABLE NO. 13
CALCULATION OF DIFFUSION COEFFICIENT
EXPERIMENT NO. 7

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{t}{q_{\infty}}$
2	0.3	13.6	1.6	12.0	.16	.013	4.9	.04
4	0.6	12.2	2.4	9.8	.16	.016	4.8	.15
6	0.9	10.9	3.0	7.9	.14	.018	4.5	.20
8	1.2	9.8	3.5	6.3	.11	.017	4.2	.27
10	1.35	8.9	3.8	5.1	.10	.020	3.9	.35
12	1.5	8.0	4.1	3.9	.080	.020	3.6	.44
14	1.7	7.4	4.3	3.1	.058	.019	3.4	.53
16	1.8	7.0	4.4	2.6	.048	.018	3.2	.63
18	1.85	6.6	4.6	2.0	.042	.021	3.0	.73
20	1.9	6.2	4.7	1.5	.036	.024	2.8	.83
22	2.0	5.9	4.8	1.1	.032	.029	2.7	.93
24	2.1	5.6	4.9	0.7	.030	.043	2.5	1.03
26	2.1	5.4	5.0	0.4	.028	.070	2.4	1.13

TABLE NO. 14CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 8

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta c}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
5	0.4	13.3	2.0	11.3	.090	.008	4.9	.08
10	0.9	11.4	3.0	8.4	.080	.010	4.6	.20
15	1.2	10.0	3.6	6.4	.063	.010	4.3	.28
20	1.4	9.2	3.9	5.3	.040	.0075	4.0	.35
25	1.6	8.3	4.2	4.1	.026	.006	3.7	.43
30	1.7	7.8	4.4	3.4	.023	.007	3.5	.49
35	1.8	7.4	4.5	2.9	.018	.006	3.4	.53
40	1.9	7.0	4.7	2.3	.017	.007	3.2	.59
45	2.0	6.7	4.8	1.9	.015	.008	3.1	.64
51.5	2.1	6.3	4.9	1.4	.013	.009	2.9	.72
55	2.1	6.1	4.9	1.2	.013	.011	2.8	.75
63	2.2	5.7	5.1	0.6	.013	.022	2.6	.85

TABLE NO. 15CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 10

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
1	0.12	10.4	0.9	9.5	.128	.014	4.4	.02
2	0.19	7.5	1.4	6.1	.059	.010	3.4	.06
3	0.24	5.8	1.5	4.3	.040	.009	2.6	.09
4	0.27	4.8	1.5	3.3	.026	.008	2.0	.14
5	0.29	4.2	1.6	2.6	.021	.008	1.6	.18
6	0.31	3.8	1.6	2.2	.014	.006	1.3	.24
7	0.32	3.4	1.7	1.7	.010	.006	1.1	.29
8	0.33	3.2	1.7	1.5	.008	.005	1.0	.32
9	0.34	2.9	1.7	1.2	.005	.004	0.8	.45
10	0.34	2.7	1.7	1.0	.004	.004	0.7	.49

TABLE NO. 16
CALCULATION OF q
EXPERIMENT NO. 11

$W_r = 0.59$ grams

$Q = 10$ ml/min.

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>g</u>
0	2.52	3.0	15.0			
1	2.51	3.1	14.9			
2	2.50	3.2	14.8	14.9	2	0.6
3	2.46	3.5	14.5			
4	2.46	3.5	14.5	14.6	2	1.2
5	2.44	3.6	14.4			
6	2.42	3.8	14.2	14.4	2	1.8
7	2.42	3.8	14.1			
8	2.41	3.9	14.0	14.1	2	2.4
9	2.41	3.9	14.0			
10	2.41	3.9	14.0	14.0	2	2.4
12.5	2.39	4.1	13.8			
15	2.37	4.3	13.6	13.8	5	3.4
17.5	2.36	4.4	13.5			
20	2.36	4.4	13.5	13.5	5	3.8
25	2.36	4.4	13.5			
30	2.36	4.4	13.5	13.5	10	4.0
35	2.36	4.4	13.5			
40	2.36	4.4	13.5	13.5	10	4.3
45	2.36	4.4	13.5			
50	2.36	4.4	13.5	13.5	10	4.5

(continued)

TABLE NO. 16
(continued)

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
55	2.36	4.4	13.5			
60	2.36	4.4	13.5	13.5	10	4.8
65	2.37	4.3	13.6			
70	2.37	4.3	13.6	13.6	10	4.7
75	2.37	4.3	13.6			
80	2.37	4.3	13.6	13.6	10	5.0
85	2.39	4.1	13.8			
90	2.39	4.1	13.8	13.7	10	4.9

TABLE NO. 17
CALCULATION OF q
EXPERIMENT NO. 12

$w_p = 5.41$ grams $Q = 10$ ml/min.

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
2.5	2.28	5.3	12.5			
5.0	2.13	7.4	10.2	12.6	5	1.2
7.5	2.03	9.4	8.0			
10.0	1.96	10.8	6.4	8.2	5	2.2
12.5	1.92	12.0	5.0			
15.0	1.89	12.8	3.9	5.1	5	3.0
17.5	1.88	13.3	3.0			
20.0	1.86	13.8	1.0	2.6	5	3.5
22.5	1.86	13.8	1.0			
25.0	1.86	13.8	1.0	1.0	5	4.0
27.5	1.86	13.8	1.0			
30.0	1.85	14.1	0	0.7	5	4.3
32.5	1.85	14.1	0			
35.0	1.85	14.1	0	0	5	4.4
37.5	1.85	14.1	0			
40.0	1.85	14.1	0	0	5	4.6
45.0	1.85	14.1	0			
50.0	1.85	14.1	0	0	10	4.9
55.0	1.85	14.1	0			
60.0	1.85	14.1	0	0	10	5.1

TABLE NO. 18
CALCULATION OF q
EXPERIMENT NO. 13

$w_p = 0.60$ grams

$Q = 100$ ml/min

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
1	2.51	3.1	14.9			
2	2.47	3.4	14.6	14.8	2	0.9
3	2.46	3.5	14.5			
4	2.44	3.6	14.4	14.5	2	1.6
5	2.44	3.6	14.4			
6	2.42	3.8	14.2	14.3	2	2.0
7	2.41	3.9	14.0			
8	2.41	3.9	14.0	14.1	2	3.0
9	2.41	3.9	14.0			
10	2.40	4.0	14.0	14.0	2	3.6
12.5	2.40	4.0	13.9			
15	2.40	4.0	13.9	13.9	5	4.6
17.5	2.41	3.9	14.0			
20	2.41	3.9	14.0	14.0	5	5.2
22.5	2.39	4.1	13.8			
25	2.35	4.5	13.4	13.7	5	5.0
27.5	2.36	4.4	13.5			
30	2.37	4.3	13.6	13.5	5	4.8
32.5	2.37	4.3	13.6			
35	2.37	4.3	13.6	13.6	5	5.2

TABLE NO. 18

(continued)

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
37.5	2.39	4.1	13.8			
40	2.39	4.1	13.8	13.7	5	5.1

TABLE NO. 19
CALCULATION OF q
EXPERIMENT NO. 14

$w_r = 2.95$ grams $Q = 100$ ml/min.

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
2	2.44	3.6	14.4			
4	2.30	5.0	12.8	14.1	4	0.9
6	2.28	5.2	12.6			
8	2.15	7.1	10.5	12.0	4	2.1
10	2.11	7.8	9.7			
12	2.09	8.1	9.4	9.9	4	3.0
14	2.09	8.1	9.4			
16	2.09	8.1	9.4	9.4	4	3.7
18	2.11	7.8	9.7			
20	2.12	7.6	10.0	9.7	4	4.0
22	2.15	7.1	10.5			
24	2.17	6.8	10.8	10.4	4	4.4
26	2.19	6.5	11.2			
28	2.21	6.2	11.5	11.2	4	4.6
30	2.24	5.8	11.9			
32	2.24	5.8	11.9	11.8	4	4.7
34	2.27	5.4	12.4			
36	2.28	5.2	12.6	12.3	4	4.8
38	2.30	5.0	12.8			
40	2.33	4.7	13.1	12.8	4	4.9

TABLE NO. 20
CALCULATION OF q
EXPERIMENT NO. 15

$w_r = 5.90$ grams

$Q = 100$ ml/min

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
5	2.24	5.8	11.9	13.4	5	0.8
10	2.15	7.1	10.5	11.2	5	1.5
15	2.11	7.8	9.7	10.1	5	2.0
20	2.11	7.8	9.7	9.7	5	2.5
25	2.08	8.3	9.2	9.4	5	2.6
30	2.17	6.7	10.9	10.0	5	3.0
35	2.21	6.1	11.6	11.2	5	3.1
40	2.27	5.4	12.4	12.0	5	3.2

TABLE NO. 21
CALCULATION OF q
EXPERIMENT NO. 16

$w_p = 5.90$ grams

$Q = 100$ ml/min.

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
1	2.39	4.1	13.8			
2	2.28	5.2	12.6	13.8	2	0.6
3	2.21	6.2	11.5			
4	2.16	6.9	10.7	11.6	2	1.2
5	2.12	7.6	10.0			
6	2.11	7.8	9.7	10.1	2	1.6
7	2.08	8.3	9.2			
8	2.06	8.7	8.7	9.2	2	1.9
9	2.05	8.9	8.5			
10	2.04	9.1	8.3	8.5	2	2.3
12.5	2.02	9.6	7.7			
15	2.02	9.6	7.7	7.9	5	3.0
17.5	2.00	10.0	7.3			
20	2.01	9.8	7.5	7.5	5	3.7
22.5	2.02	9.6	7.7			
25	2.04	9.1	8.3	7.8	5..	4.1
27.5	2.06	8.7	8.7			
30	2.11	7.7	9.7	8.9	5	4.3
32.5	2.13	7.4	10.2			

TABLE NO. 21

(continued)

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ava}</u>	<u>Δt</u>	<u>q</u>
35	2.16	6.9	10.7	10.2	5	4.5
37.5	2.19	6.5	11.2			
40	2.21	6.2	11.5	11.1	5	4.6
42.5	2.24	5.8	11.9			
45	2.26	5.5	12.3	11.9	5	4.7
47.5	2.28	5.3	12.5			
50	2.30	5.0	12.8	12.5	5	4.8
52.5	2.33	4.7	13.1			
55	2.35	4.5	13.4	13.1	5	4.8

TABLE NO. 22
CALCULATION OF q
EXPERIMENT NO. 17

$w_p = 54.1$ grams

$Q = 100$ ml/min.

<u>t</u>	<u>pH</u>	<u>[H⁺]</u>	<u>c</u>	<u>c_{ave}</u>	<u>Δt</u>	<u>q</u>
0	2.52	3.0	15.0			
1	2.24	5.8	11.9	13.5	1	0.08
2	2.06	8.7	8.7	10.3	1	0.17
3	1.98	10.5	6.7	7.7	1	0.23
4	1.94	11.5	5.7	6.2	1	0.27
5	1.92	12.0	5.1	5.4	1	0.31
6	1.91	12.3	4.7	4.9	1	0.34
7	1.90	12.6	4.2	4.4	1	0.37
8	1.89	12.8	3.9	4.0	1	0.40
9	1.89	12.8	3.9	3.9	1	0.42
10	1.89	12.8	3.9	3.9	1	0.44
20	1.89	12.8	3.9	3.9	10	0.64
30	1.89	12.8	3.9	3.9	10	0.85

TABLE NO.23

CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 11

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
2	0.6	14.75	2.4	12.35	.39	.032	5.1	.12
4	1.3	14.5	3.8	10.7	.33	.031	5.1	.26
6	1.9	14.25	4.6	9.65	.28	.029	5.0	.38
8	2.3	14.0	5.3	8.7	.19	.022	5.0	.46
10	2.6	14.0	5.8	8.2	.17	.021	5.0	.52
15	3.3	13.6	7.2	6.4	.090	.014	4.9	.67
20	3.6	13.5	8.0	5.5	.056	.010	4.9	.74
30	4.0	13.5	9.1	4.4	.034	.008	4.9	.82
40	4.3	13.5	10.0	3.5	.023	.007	4.9	.88
50	4.5	13.5	10.7	2.8	.017	.006	4.9	.92
60	4.7	13.5	11.8	1.7	.0133	.008	4.9	.96
70	4.8	13.6	12.6	1.0	.009	.009	4.9	.98
80	4.8	13.6	12.6	1.0	.006	.006	4.9	.98

TABLE NO. 24

CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 12

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
1	0.2	14.1	1.4	12.7	.24	.019	5.0	.04
2	0.5	13.2	2.2	11.0	.24	.022	4.9	.10
3	0.7	12.1	2.6	9.5	.24	.025	4.8	.13
4	1.0	11.1	3.2	7.9	.24	.030	4.6	.22
5	1.2	10.2	3.6	6.6	.24	.036	4.4	.27
6	1.5	9.2	4.1	5.1	.23	.045	4.0	.38
7	1.7	8.5	4.4	4.1	.21	.051	3.8	.45
8	1.9	7.6	4.6	3.0	.21	.070	3.5	.54
9	2.1	6.8	5.0	1.8	.20	.111	3.1	.68
10	2.3	6.4	5.3	1.1	.20	.182	2.9	.79
15	3.1	3.9	6.8	?	.13	?	1.4	?
20	3.7	2.1	8.2	?	.10	?	0.5	?
25	4.1	0.6	9.4	?	.07	?	0.1	?
30	4.3	0.0	10.0	?	.04	?	0.0	?

TABLE NO. 25CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 13

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
1	0.5	14.8	2.2	12.6	.51	.040	5.1	.10
2	0.9	14.6	3.0	11.6	.51	.044	5.1	.18
3	1.5	14.5	4.0	10.5	.49	.047	5.0	.30
4	1.9	14.4	4.6	9.8	.41	.042	5.0	.38
6	2.6	14.25	5.3	7.95	.31	.039	5.0	.52
8	3.1	14.0	6.7	7.3	.26	.036	5.0	.62
10	3.6	13.9	7.2	6.7	.21	.031	5.0	.72
15	4.3	13.9	10.0	3.9	.12	.031	5.0	.86
17	4.5	13.9	10.7	3.2	.096	.030	5.0	.90
20	4.8	14.0	12.5	1.5	.068	.045	5.0	.96
25	5.0	14.4	14.1	0.3	.046	.153	5.0	1.00
30	5.2	14.6	15.0	?				

TABLE NO. 26

CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 14

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
3	0.5	14.0	2.2	11.8	.35	.030	5.0	.10
4	0.9	13.2	3.0	10.2	.35	.034	4.9	.18
5	1.3	13.1	3.8	9.3	.33	.035	4.9	.27
6	1.6	13.0	4.2	8.8	.32	.036	4.9	.33
8	2.2	11.4	5.1	6.2	.30	.048	4.6	.48
12	3.1	10.6	6.8	3.8	.19	.050	4.5	.69
16	3.7	10.4	8.2	2.2	.14	.063	4.4	.84
20	4.1	11.2	9.4	1.8	.092	.051	4.6	.86
24	4.4	11.7	10.3	1.4	.064	.046	4.7	.94
28	4.6	12.3	11.2	1.1	.038	.035	4.8	.96
32	4.8	12.7	12.4	0.3	.026	.087	4.8	1.00
36	4.8	13.1	12.4	0.7	.012	.017	4.9	.97

TABLE NO. 27CALCULATION OF DIFFUSION COEFFICIENTEXPERIMENT NO. 15

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	<u>$\frac{q}{t}$</u>	<u>ka</u>	<u>q</u>	<u>$\frac{q}{c}$</u>
2.5	0.4	13.4	2.0	11.4	.174	.015	4.9	.08
5.0	0.8	11.9	2.8	9.1	.160	.018	4.7	.27
10.0	1.5	10.5	4.1	6.4	.124	.019	4.5	.35
12.5	1.8	10.0	4.5	5.5	.100	.018	4.3	.42
15.0	2.0	9.8	4.8	5.0	.098	.020	4.2	.43
20.0	2.5	9.8	5.6	4.2	.072	.017	4.2	.60
25.0	2.8	9.2	6.2	3.0	.056	.019	4.0	.70
30.0	3.0	10.9	6.5	4.4	.042	.010	4.5	.67
35.0	3.1	11.6	6.7	4.9	.023	.005	4.7	.66
40.0	3.2	12.4	6.9	5.5	.010	.006	4.8	.67

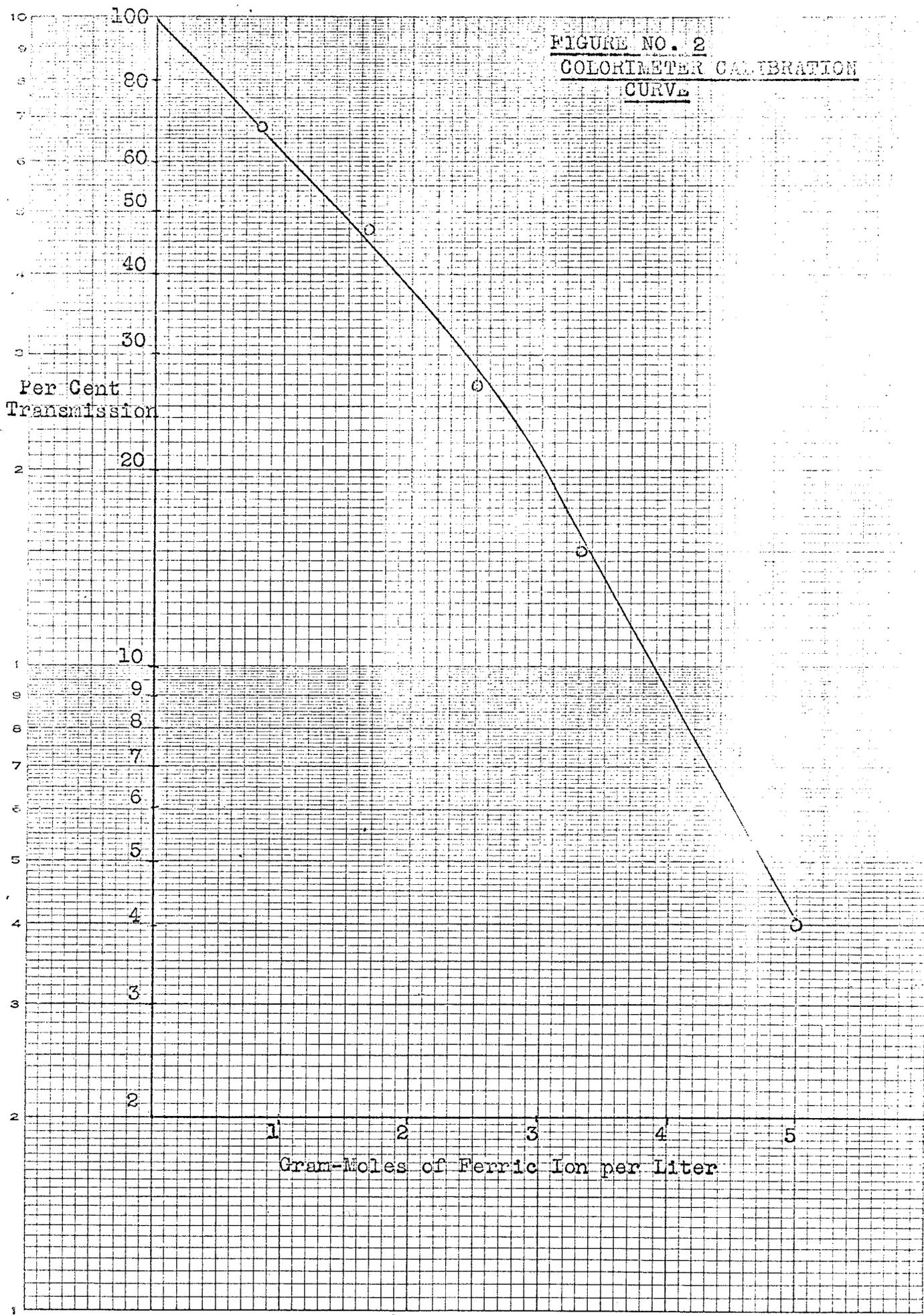
TABLE NO. 28
CALCULATION OF DIFFUSION COEFFICIENT
EXPERIMENT NO. 16.

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	<u>$\frac{q}{t}$</u>	<u>ka</u>	<u>q</u>	<u>$\frac{q}{c}$</u>
2	0.6	12.6	2.4	10.2	.31	.030	4.8	.12
4	1.2	10.6	3.5	7.1	.22	.031	4.5	.26
6	1.6	9.5	4.2	5.3	.20	.038	4.1	.38
8	1.9	8.7	4.7	4.0	.18	.045	3.9	.50
10	2.3	8.4	5.2	3.2	.16	.050	3.8	.58
15	3.0	7.7	6.6	1.1	.14	.127	3.5	.86
20	3.7	7.7	8.1	?				

TABLE NO. 29
CALCULATION OF DIFFUSION COEFFICIENT
EXPERIMENT NO. 17

<u>t</u>	<u>q</u>	<u>c</u>	<u>c*</u>	<u>c-c*</u>	$\frac{\Delta q}{\Delta t}$	<u>ka</u>	<u>q_∞</u>	$\frac{q}{q_{\infty}}$
1	.08	11.9	0.7	11.2	.083	.007	4.7	.02
2	.17	8.6	1.2	7.4	.070	.010	3.8	.04
3	.23	6.9	1.5	5.4	.055	.010	3.2	.07
4	.27	5.8	1.6	4.2	.047	.011	2.6	.10
5	.31	5.1	1.6	3.5	.036	.010	2.2	.14
6	.34	4.6	1.7	2.9	.033	.011	1.9	.18
7	.37	4.2	1.9	2.3	.028	.012	1.6	.23
8	.40	4.0	2.0	2.0	.025	.012	1.5	.27
9	.42	3.9	2.0	1.9	.022	.012	1.4	.30
10	.44	3.9	2.1	1.8	.020	.011	1.4	.31
20	.64	3.9	2.5	1.4	.020	.014	1.4	.46
30	.85	3.9	2.9	1.0	.020	.020	1.4	.61

FIGURE NO. 2
COLORIMETER CALIBRATION
CURVE



EUGENE DITZGEN CO.
MADE IN U.S.A.

NO. 340-L210 DITZGEN GRAPH PAPER
SEMI-LOGARITHMIC
2 CYCLES X 10 DIVIDERS PER INCH

Gram-Moles of Ferric Ion per Liter

Figure No. 6
RESIN LOADING CURVE
Experiment No. 1

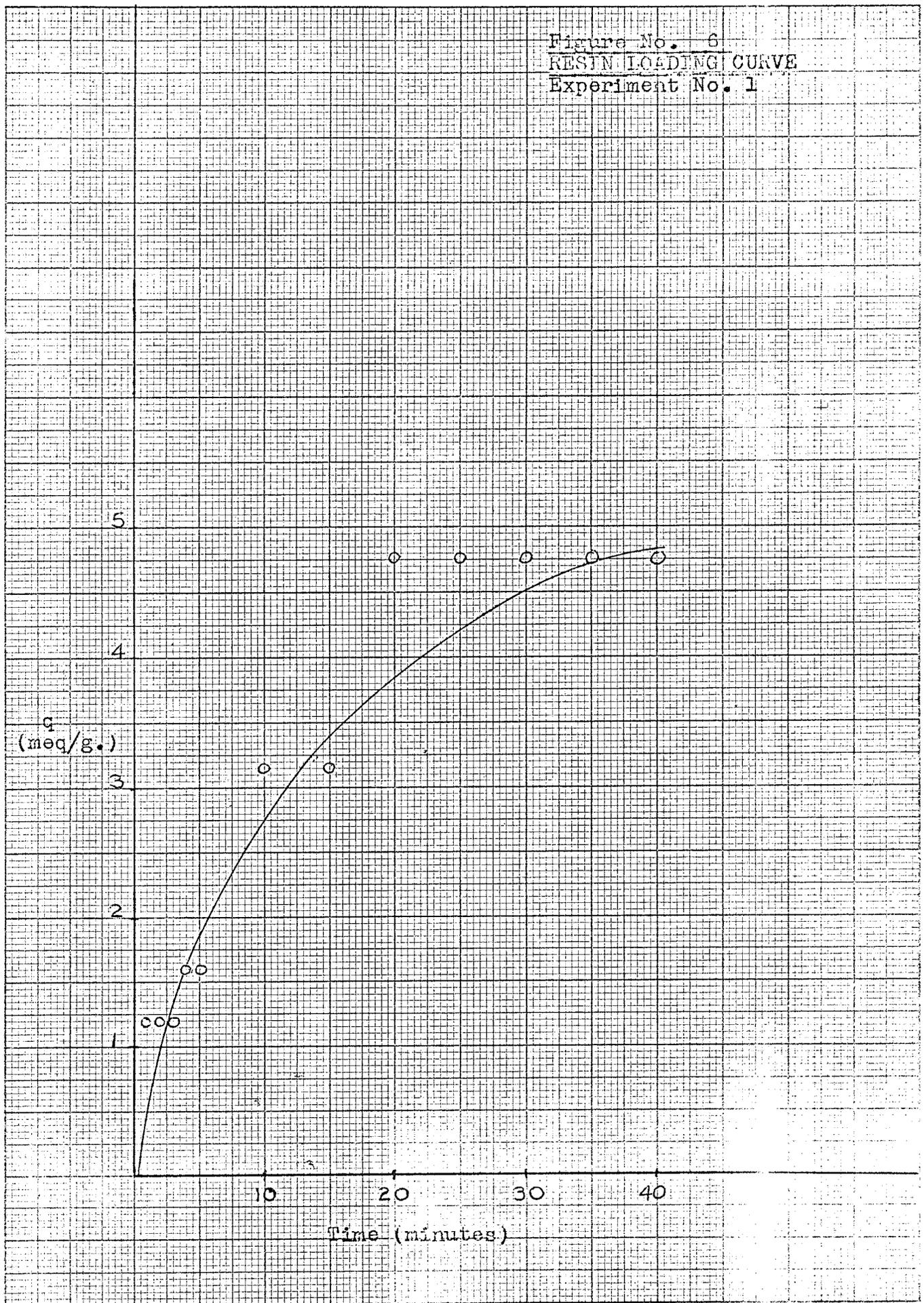


FIGURE NO. 7
RESIN-LOADING-CURVE
EXPERIMENT NO. 2

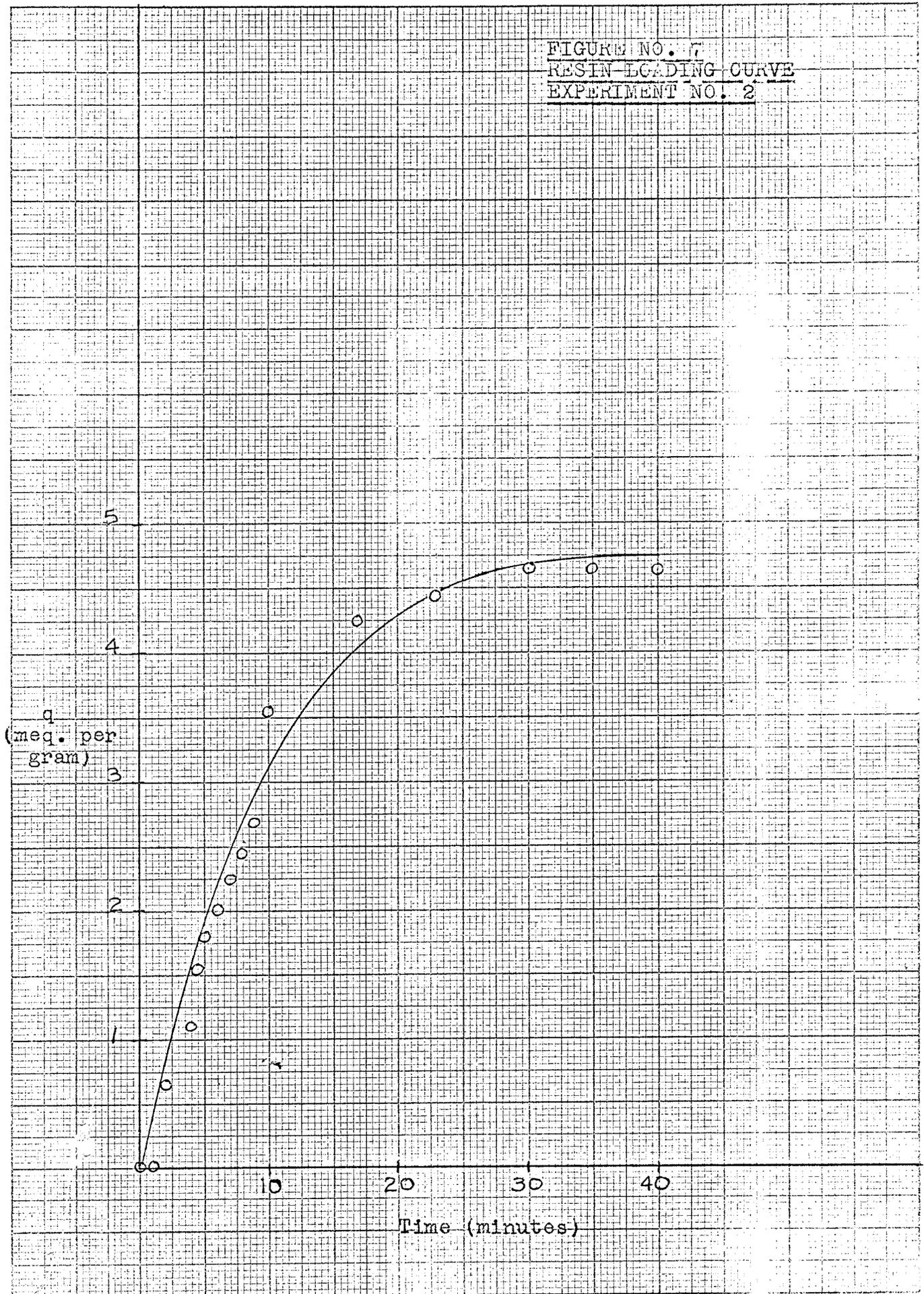
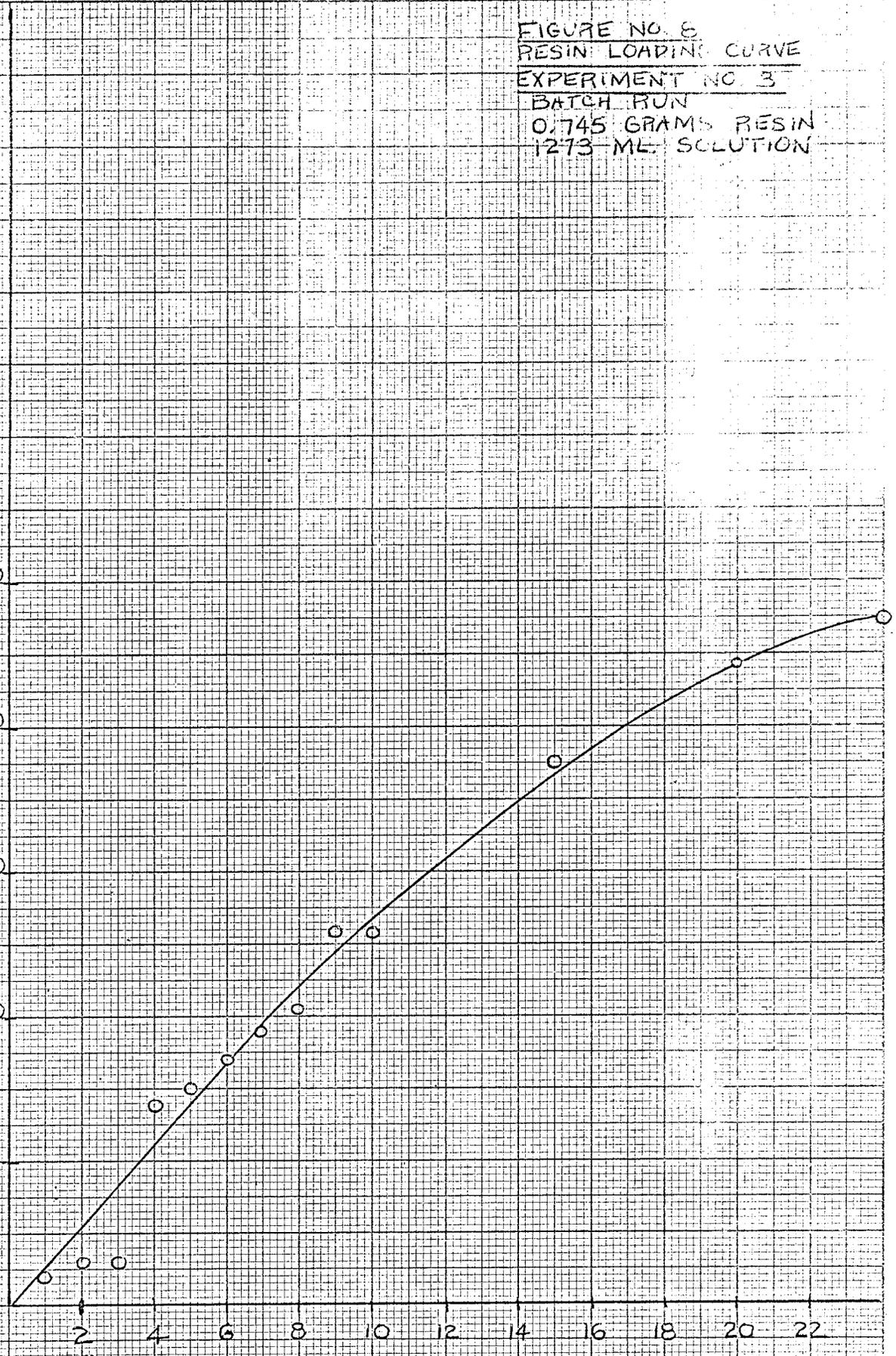


FIGURE NO. 8
RESIN LOADING CURVE
EXPERIMENT NO. 3
BATCH RUN
0.745 GRAMS RESIN
1273 ML SOLUTION

CONCENTRATION OF FERRIC ION IN RESIN IN MEQ PER GRAM



TIME IN MINUTES

FIGURE NO. 9
RESIN LOADING CURVE
EXPERIMENT NO, 4

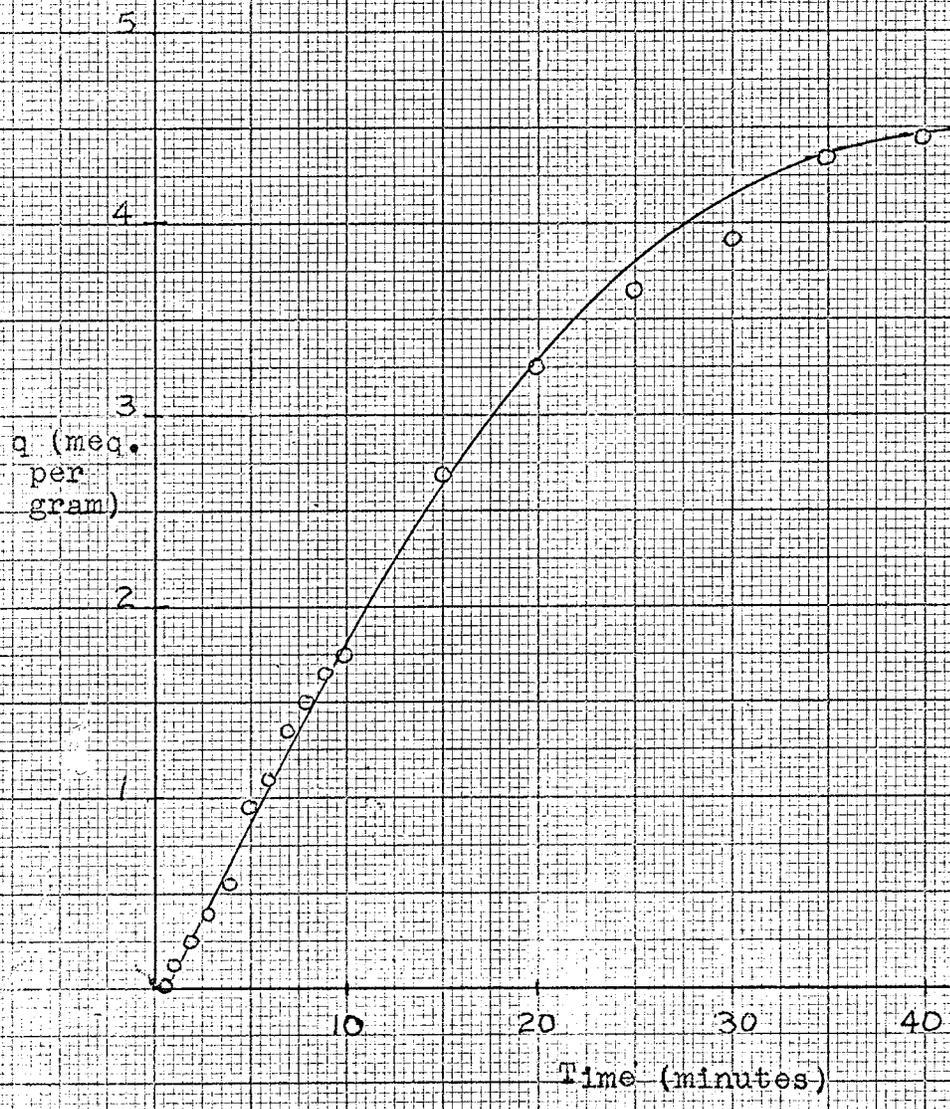


FIGURE NO. 10
RESIN LOADING CURVE
EXPERIMENT NO. 7

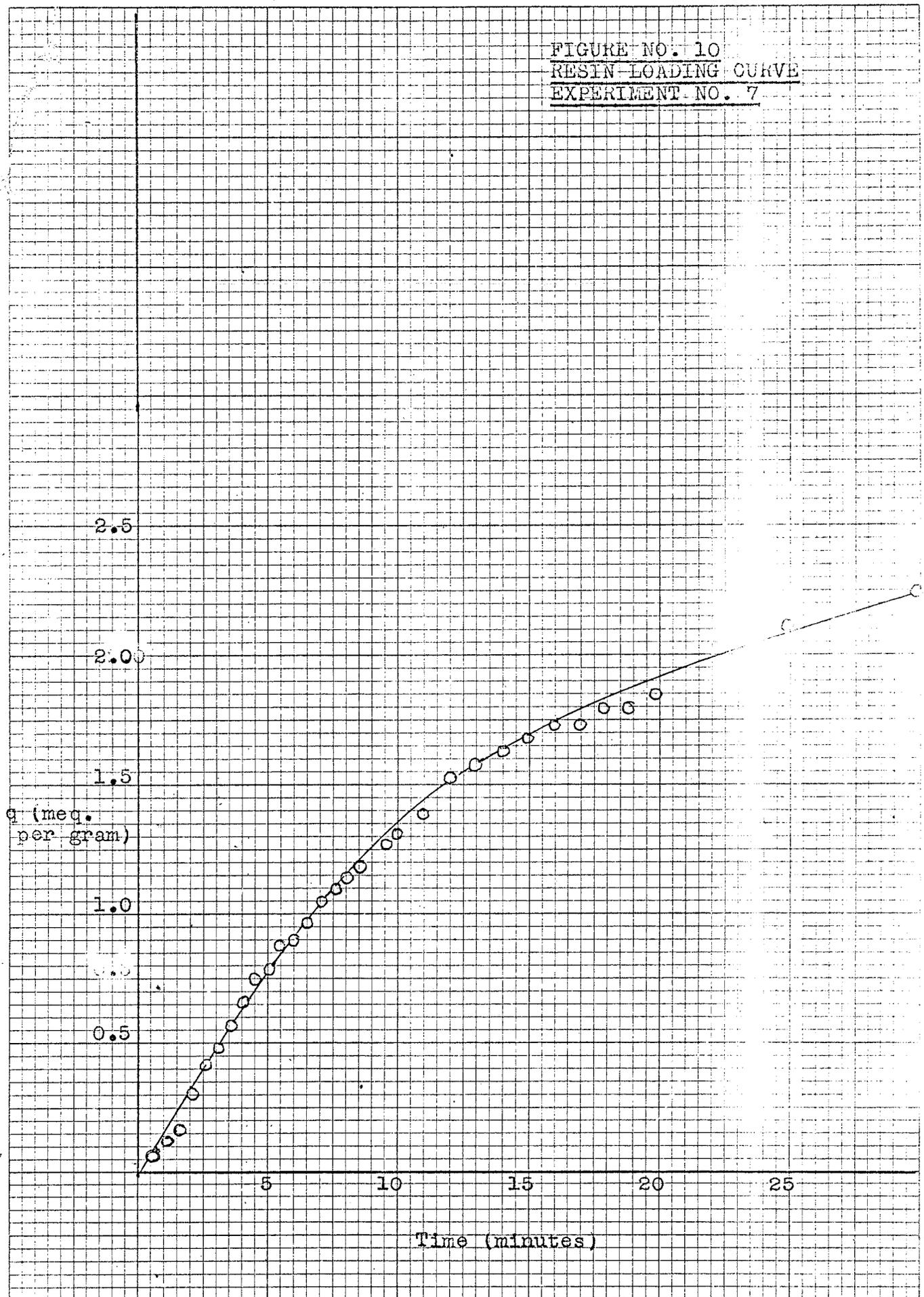


FIGURE NO. 11
RESIN LOADING CURVE
EXPERIMENT NO. 8

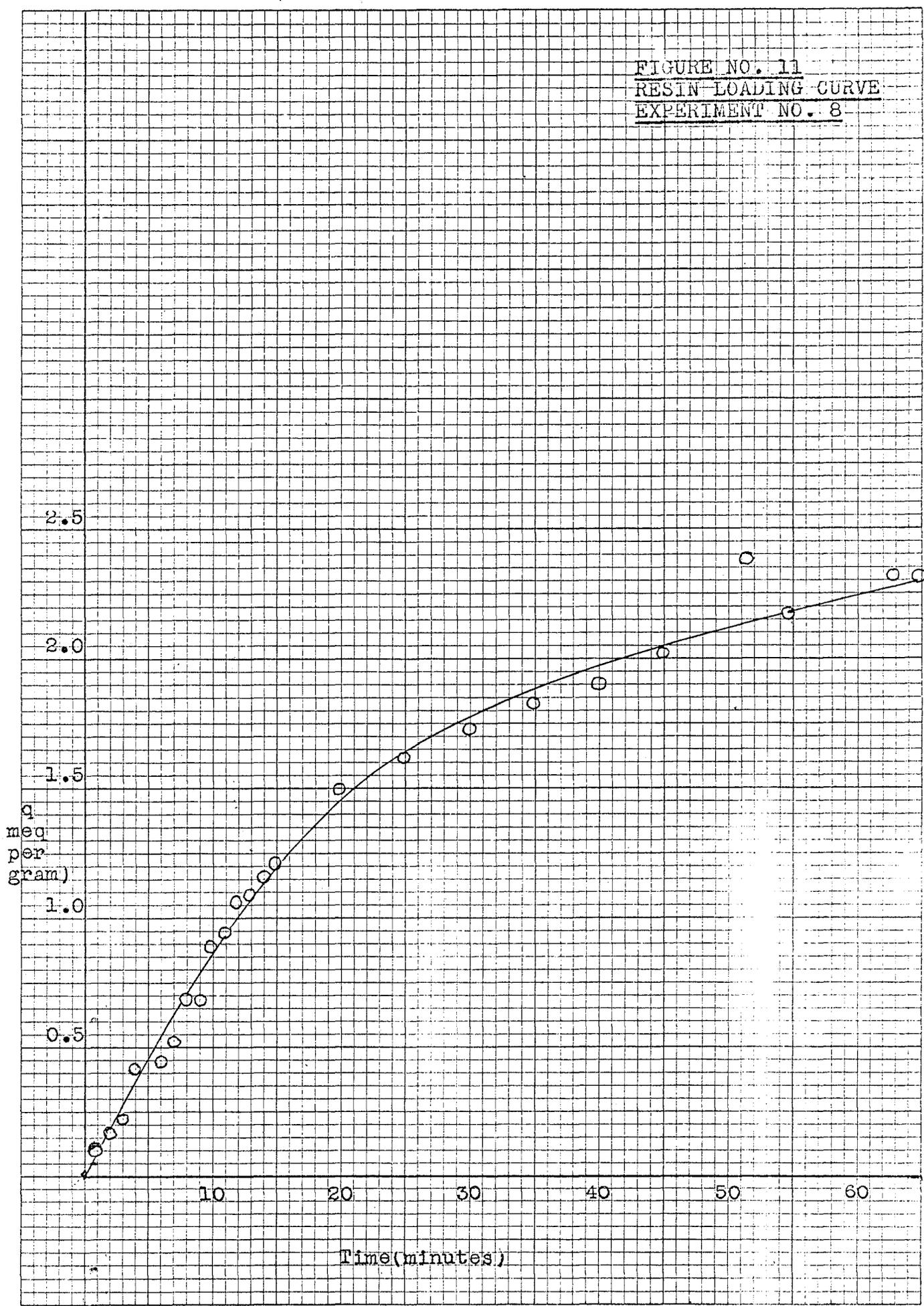


FIGURE NO. 12
RESIN LOADING CURVE
EXPERIMENT 10
BATCH RUN
46.6 GRAMS RESIN
1241 ML. SOLUTION

CONCENTRATION OF FERRIC IONS IN RESIN IN MEQ PER GRAM

0.05
0.10
0.15
0.20
0.25
0.30
0.35

2 4 6 8 10 12 14 16

TIME IN MINUTES

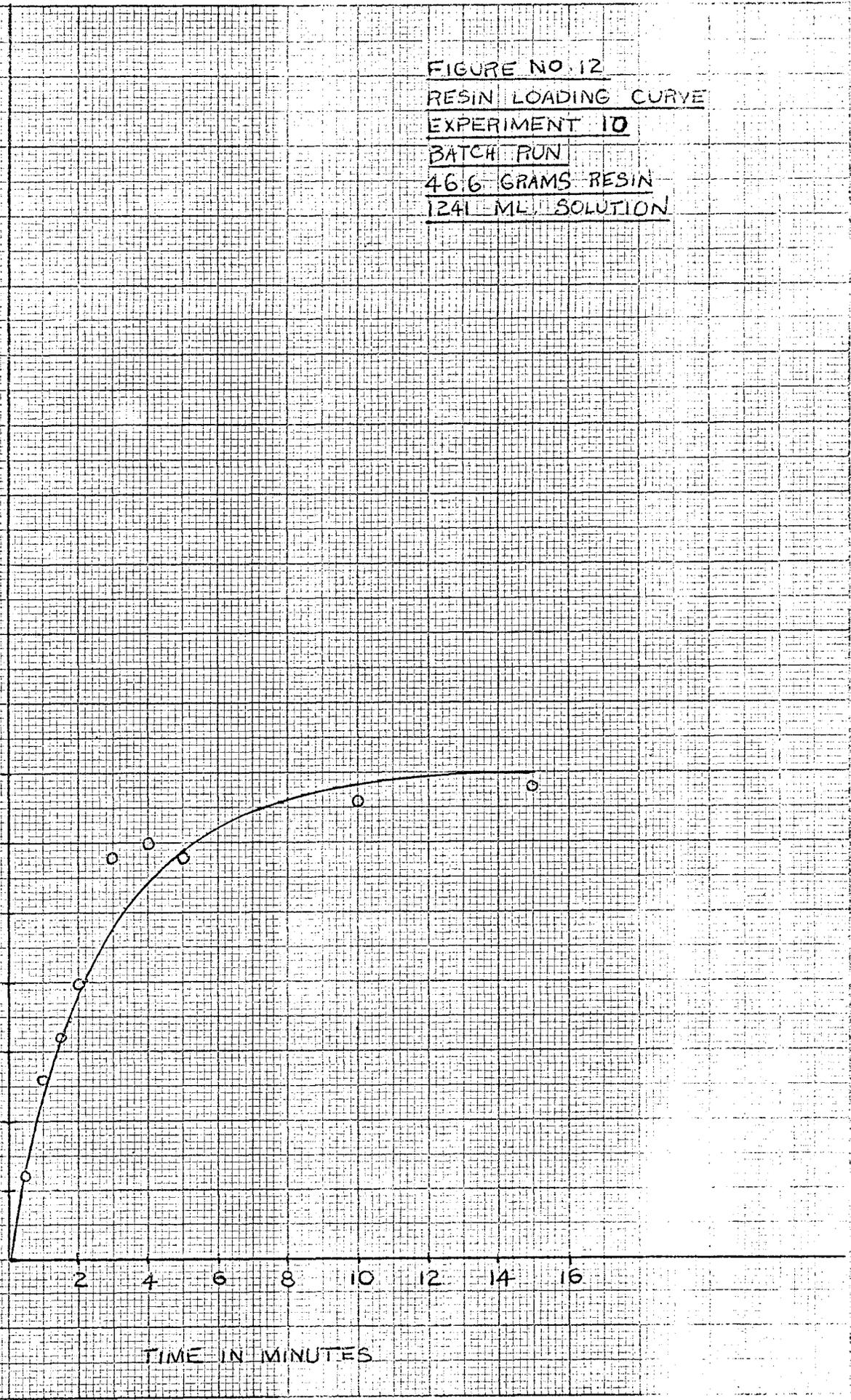


FIGURE NO. 13
RESIN LOADING CURVE
EXPERIMENT. 1
COLUMN RUN
0.59 G. RESIN
10 ML/MIN FLOW RATE

CONCENTRATION OF Fe⁺⁺⁺ IN RESIN IN MEQ PER GRAM (g)

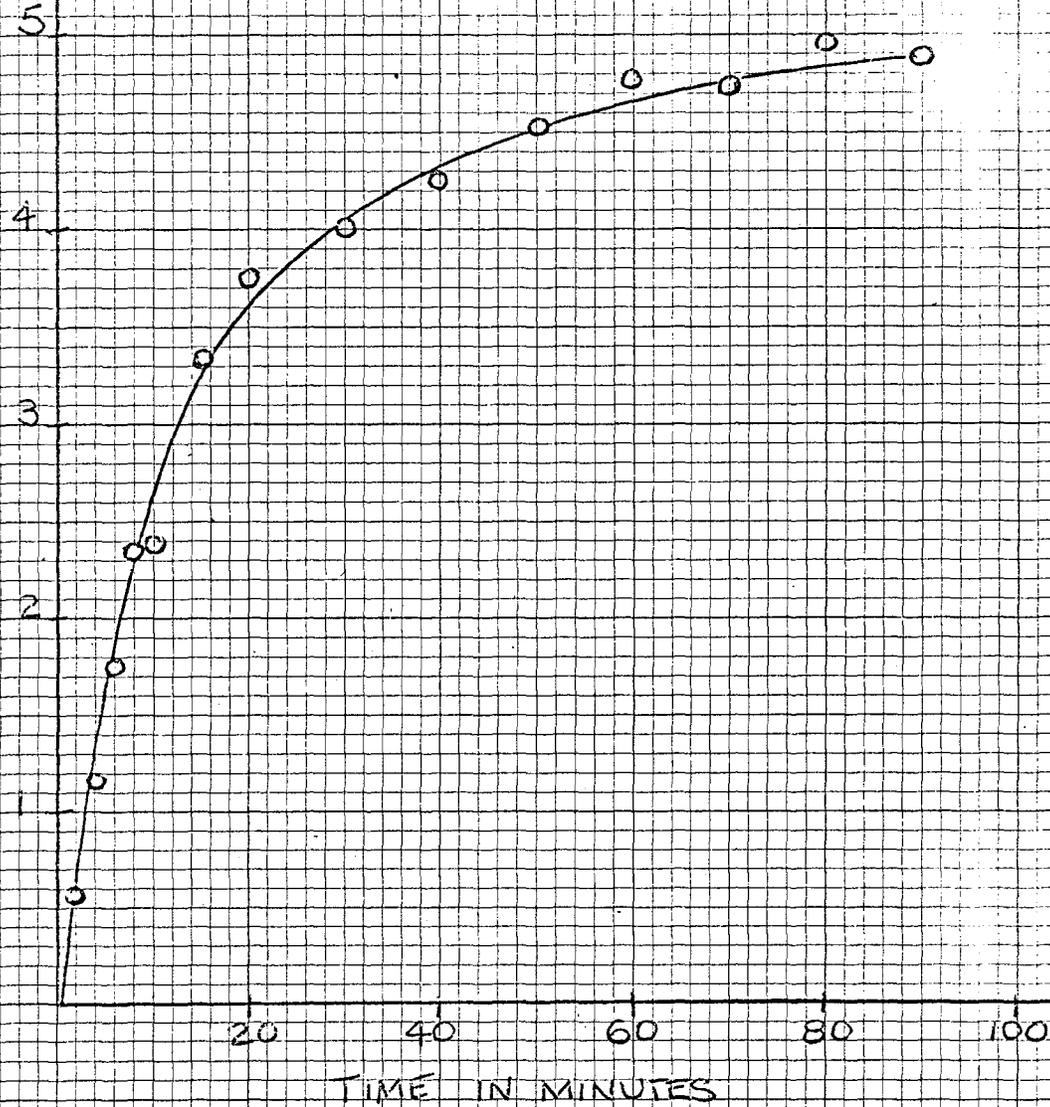


FIGURE NO. 14
RESIN LOADING CURVE
EXPERIMENT NO. 12

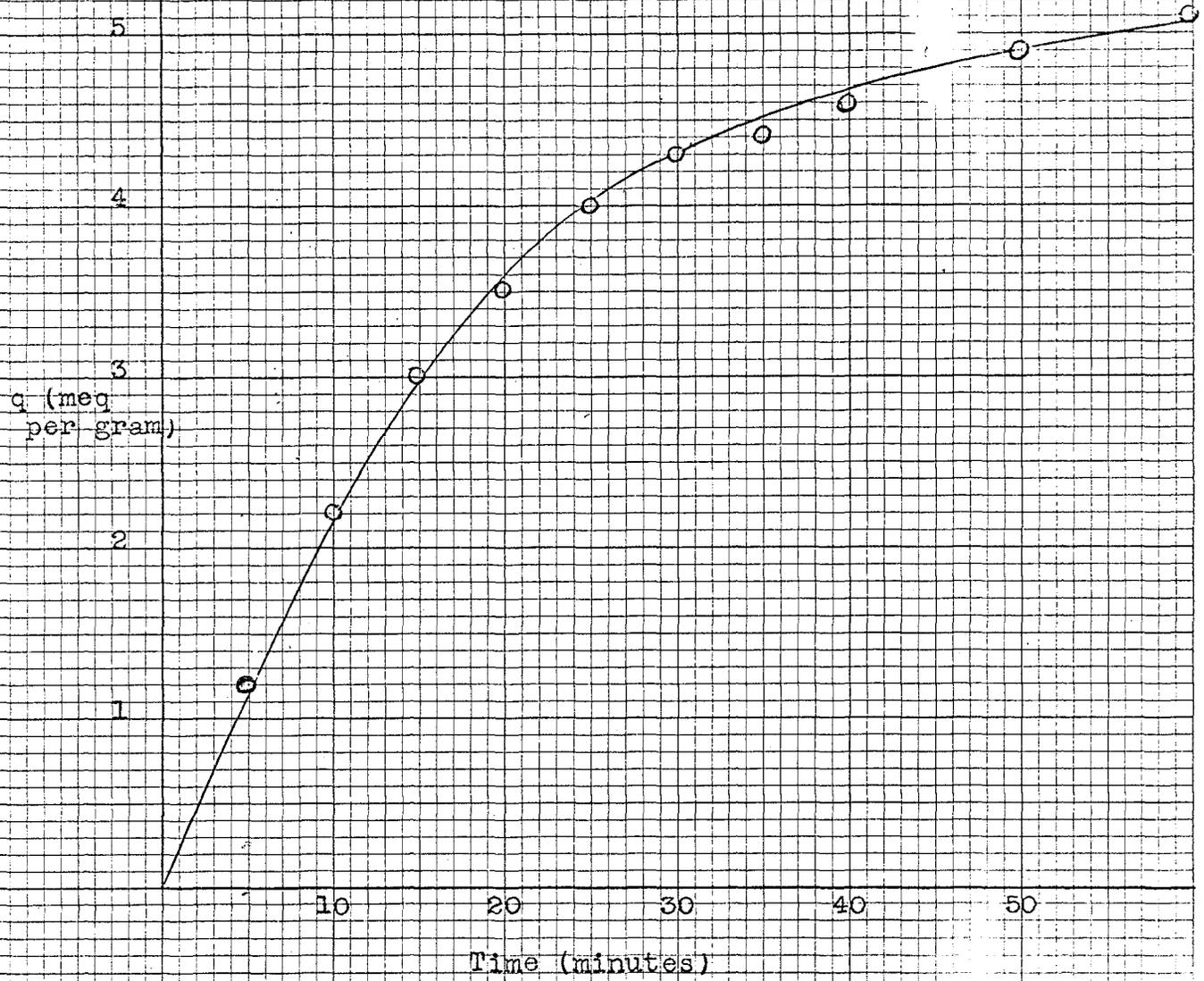


FIGURE NO. 15
EXPERIMENT # 13
RESIN LOADING CURVE
COLUMN RUN
0.60 GRAMS RESIN
100 ML./MIN FEED RATE

CONCENTRATION OF Fe^{+++} IN RESIN (%) IN MEQ PER GRAM

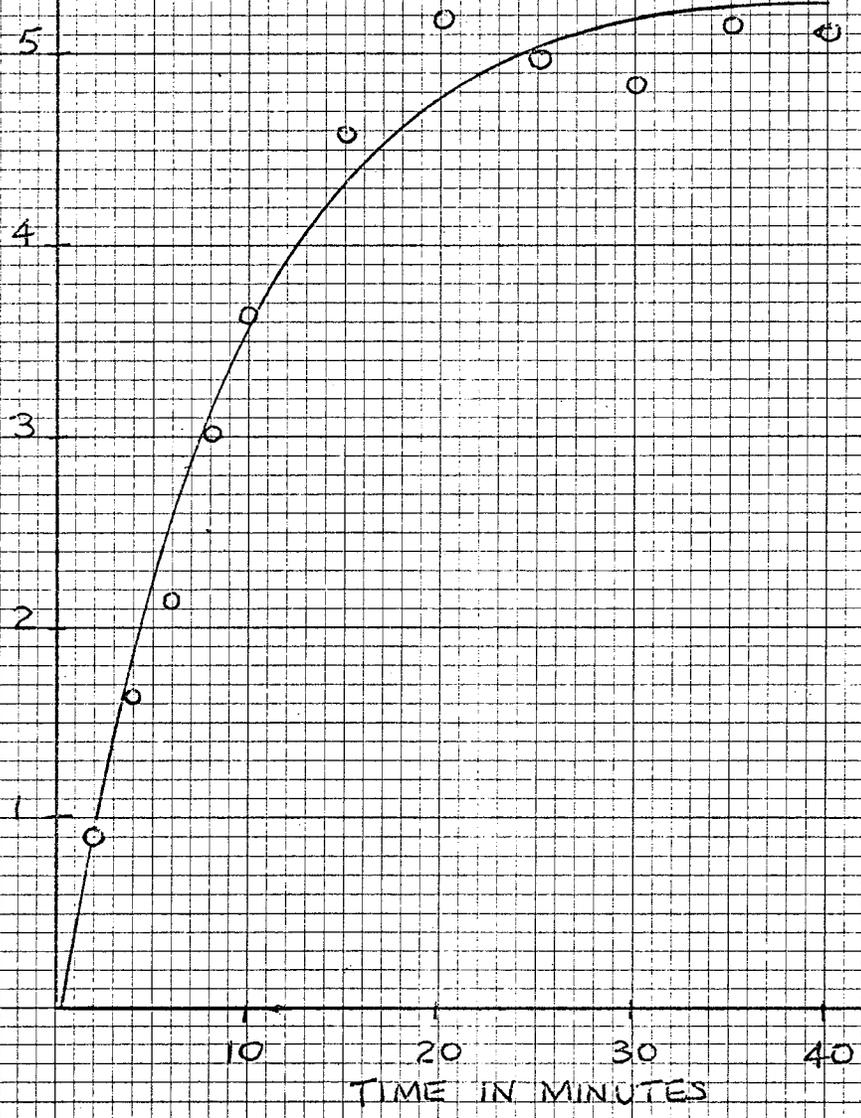


FIGURE NO. 16
RESIN LOADING CURVE -
EXPERIMENT 331A CCLT RUN
2.95 GRAMS RESIN
100 ML/MIN FEED RATE

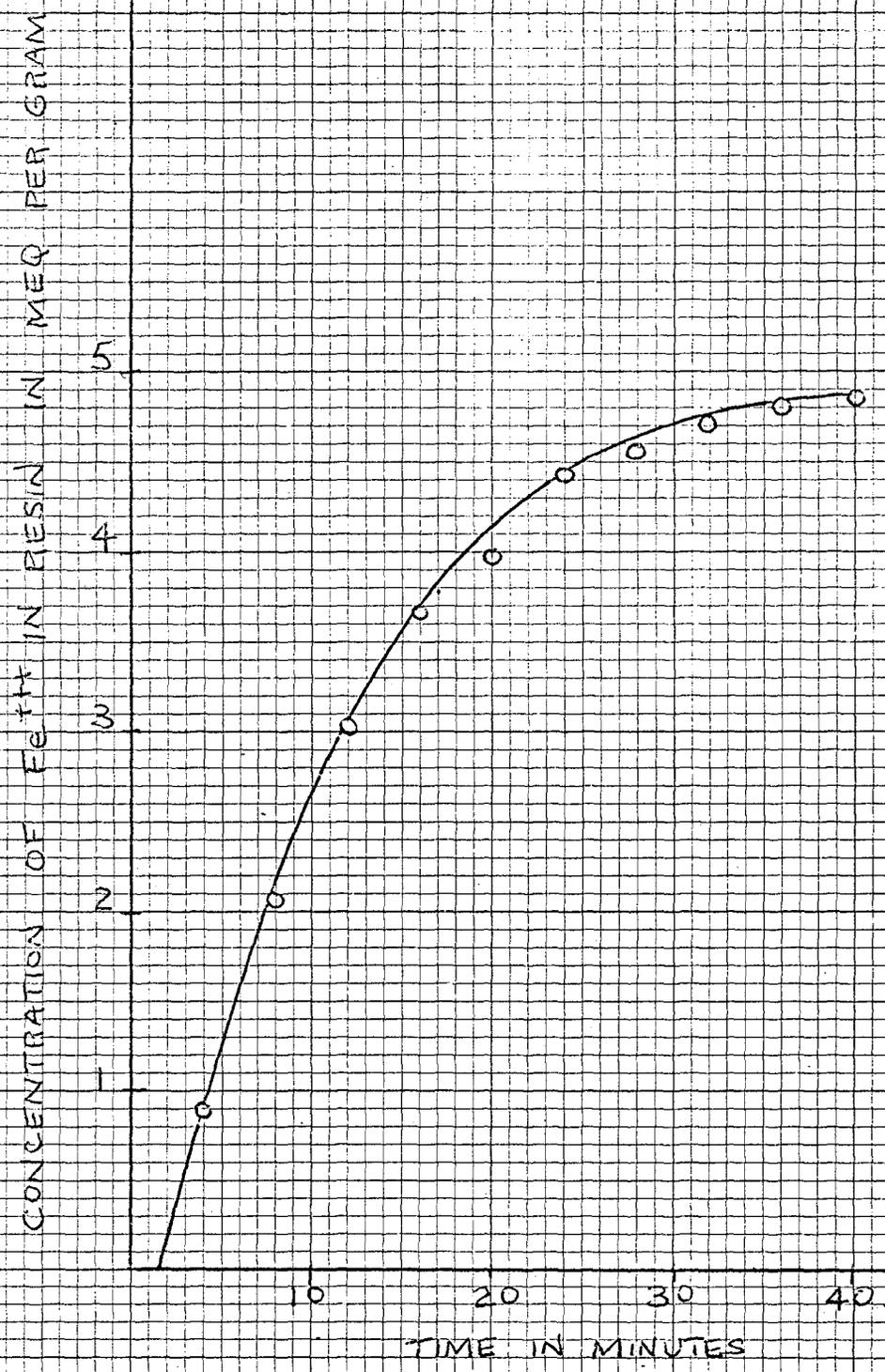


FIGURE NO. 17
RESIN LOADING CURVE
EXPERIMENT NO. 15

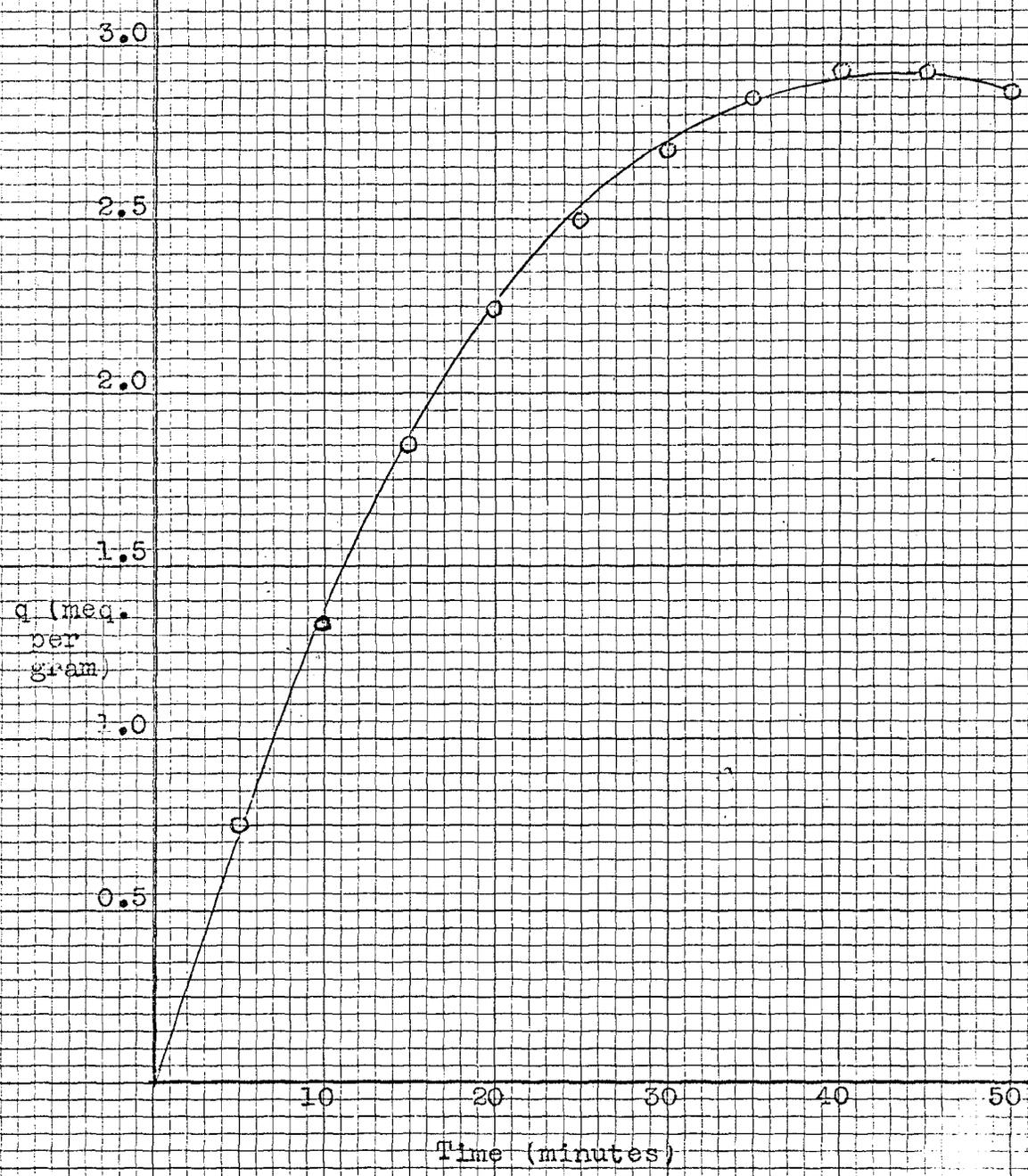


FIGURE NO. 18
RESIN LOADING CURVE
EXPERIMENT NO. 116
COLUMN RUN
5.90 GRAMS RESIN
100 ML/MIN FEED RATE

CONCENTRATION OF RESIN (MEQ Fe⁺⁺ PER GRAM)

5

4

3

2

1

10

20

30

40

50

TIME IN MINUTES

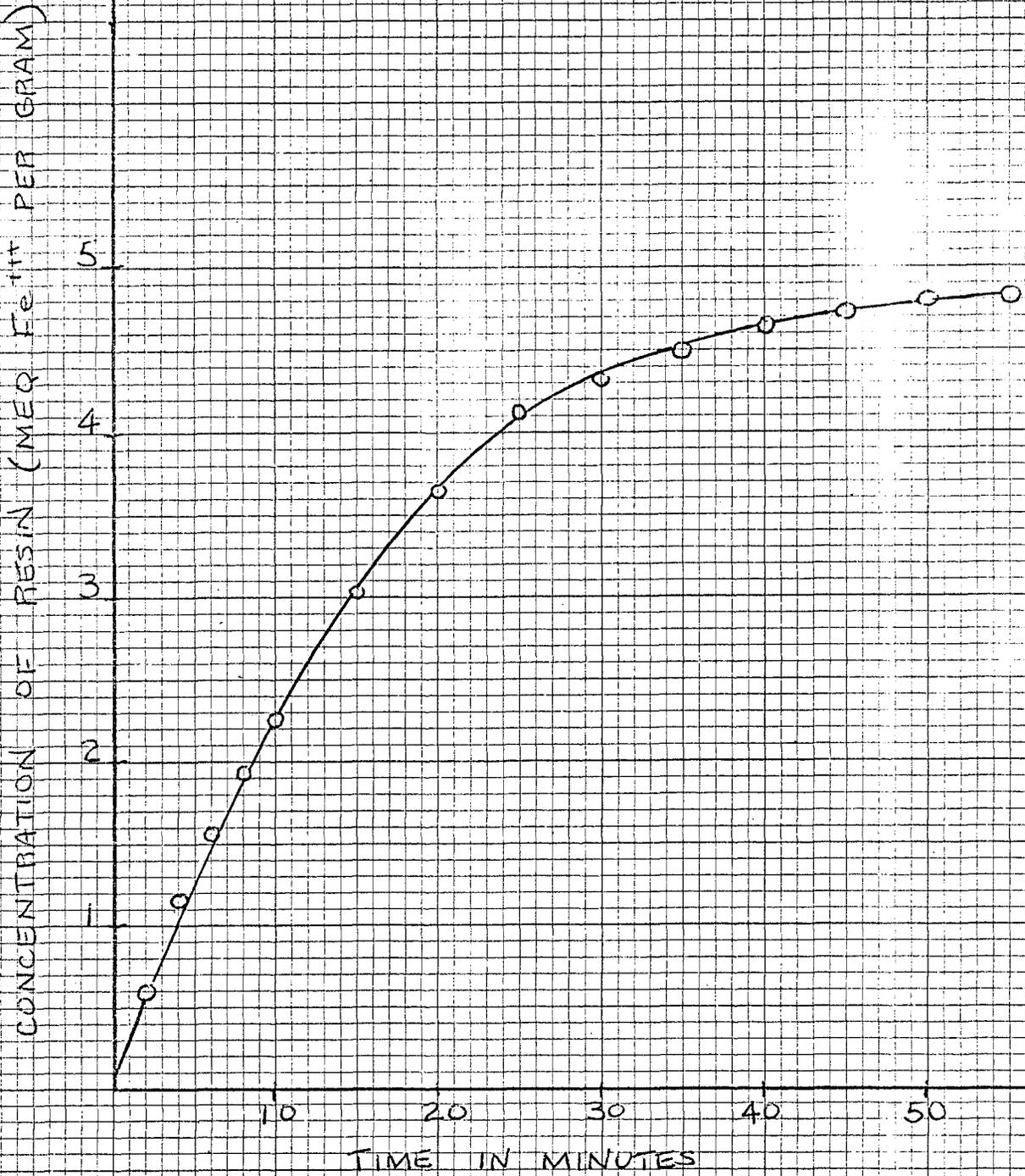
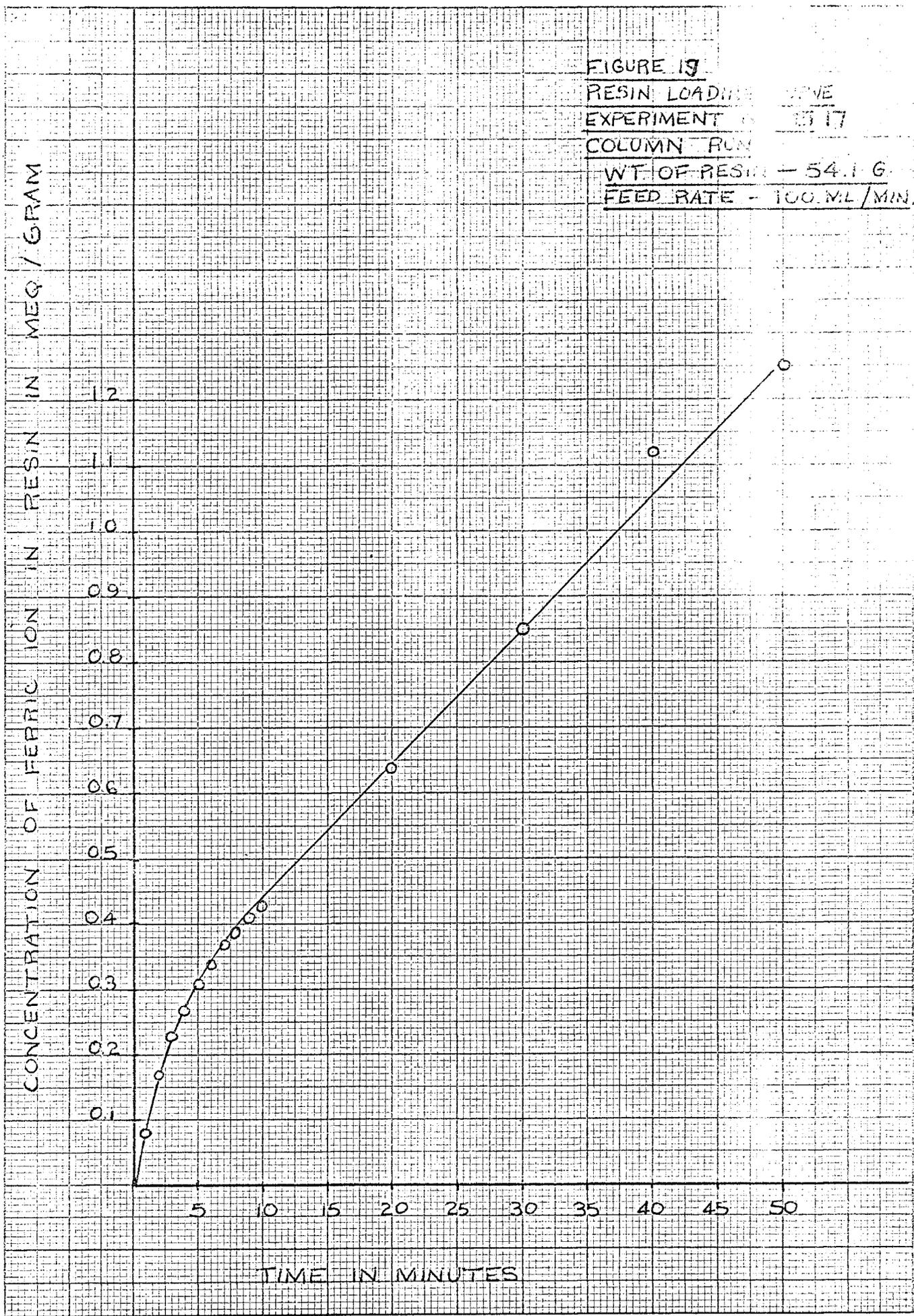


FIGURE 19
RESIN LOADING CURVE
EXPERIMENT NO. 1517
COLUMN RUN
WT. OF RESIN - 54.1 G.
FEED RATE - 100. ML/MIN.



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