# ION EXCHANGE TECHNOLOGY IN THE SODIUM AND FERRIC ION-DOWEX 50 SYSTEMS

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# ABSTRACT

This investigation was directed toward the extension of data on the kinetics of monovalent - trivalent cation exchange and to establish data on the relationship between temperature and total capacity of an ion exchange resin. In order to compare the present study to a previous investigation by Klosowski (2), the capacity of the resin used for ferric and chromic ions was also measured.

In the kinetic study, the ferric ion-hydrogen ion exchange using Dowex 50 X-8 resin was studied in an agitated vessel. A two-bladed marine type propellor was used and the agitator speed was varied from 400 to 800 rpm. The exchange rates were measured for seven different particle size ranges from -16 mesh to +200 mesh.

The results of the kinetic study shows that within the range of variables studied, the exchange rate is film diffusional controlled for this system. The exchange rate varied directly with the agitator speed and inversely with the particle size range. The equation proposed by Reichenberg (6) for the calculation of film thickness gave results which agree with the experimental data for particle sizes from -16 to +60 mesh but failed for particle sizes below 70 mesh.

At 400, 600, and 800 rpm the average film thicknesses calculated for the ferric ion Dowex 50 system are within 6%, 1%, and exactly equal to the corresponding values of the chromic ion Dowex 50 system. Since the diffusion coefficients for the ferric and chromic ion systems are $0.57 \times 10^{-5}$ and $0.642 \times 10^{-5}$ $cm^2/sec.$ respectively, the agreement on the values of the film thicknesses are as expected. In view of this agreement, the experimental procedure used in this thesis with a modified form of the Reichenberg Equation is proposed as a method for the determination of diffusion coefficients. In applying this method one would determine the rate of ion exchange at 600 rpm for a screened Dowex 50 resin fraction and then substitute the data taken in the equation shown below.

$$D = 0.0051 \frac{r}{(Co)} \frac{d\phi}{dt}$$

where D - Diffusion coefficient

r - Average particle radius

Co - Initial concentration in the bulk solution

$d\phi/dt$ - Exchange rate per unit volume

This method would greatly simplify the determination of these coefficients but would be subject to the assumptions given in the section on Calculations. The principal assumptions are that the exchange process is film diffusional controlled and that the process is carried out in dilute aqueous solutions in view of possible viscosity changes.

The results of the kinetic study can also be successfully applied to increase the efficiency of various methods which have been proposed for continuous ion exchange. In the mixer settler method proposed by Hiester (13), the present study indicates that optimum mixer conditions could be selected for any system based on the relationship between film thickness and agitator speed. From this relationship it would be possible to establish an optimum agitator speed. Once this speed was established, the exchange rate could be calculated using the Reichenberg Equation and the film thickness given in this study.

In the capacity vs temperature phase of this investigation, the amount of sodium ion hydrogen ion exchange was measured at temperatures from 0° to 100°C. using Dowex 50 X-8 and X-2 resin. The effect of regeneration temperature was also tested.

No change in the total resin capacity was found by variation in the loading temperature for either Dowex 50 X-8 or X-2 ion exhange resins. Some change in total capacity was effected, however, by variation in the regeneration temperature from ambient to 65°C.

The capacity of Dowex 50 X-8 ion exchange resin as determined for ferric ion and chromic ion was 13.2 and 9.89 meq. per gram of dry resin respectively. Manufacturer's specification for this resin for monovalent ions is 5.0 ± 0.3 meq. per dry gram. No data on the capacities for di and trivalent ions are available.

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# INTRODUCTION

With the increased application of ion exchange in the chemical industry, the need for additional basic data applicable to the design of continuous operations has become increasingly important. The mechanics of continuous processing have been studied by many authors and a commercial unit has been erected by the Chemical Separations Corporation (1). In general, however, most of the proposed modes of operation are still in the experimental stages and additional experimental data will be required before they can be successfully applied in industry.

The aim of this study was to extend the data on the kinetics of monovalent-trivalent exchange and to ascertain the effects of temperature on the total capacity of an ion exchange resin. The ionic system chosen for the kinetic study was ferric ion-hydrogen ion exchange using Dowex 50 X-8 resin. The results of this study were then compared with existing data on chromic ion-hydrogen ion exchange (2).

In the capacity portion of this investigation the sodium ion-hydrogen ion exchange was studied at various temperatures to ascertain if a relqtionship between capacity and temperature existed. Peremyslova et al (3) have proposed that such an effect exists.

# THEORY

Synthetic ion exchange resins are high molecular weight polyacids which are insoluble in most aqueous and nonaqueous media. The resin structure can be pictured as containing large polar exchange groups which are held together by a three dimensional hydrocarbon network. In the case of cation resins, sulfonic, phosphonous, phosphonic, carboxylic, and phenolic groups are used as exchanging groups while primary, secondary, and tertiary amine groups and quaternary ammonium groups are used for anion exchangers. Boyd, et al (4) have shown that the exchange groups are dispersed throughout each particle which lends some support for the proposed structure.

Physically ion exchange resins behave like hygroscopic gels, swelling or shrinking reversibly with absorption or desorption of moisture. The amount of swelling varies inversly with the degree of cross-linkage. As the resin swells, the particle approaches the density of water and the volumetric capacity decreases. Pepper et al (5) investigating cross linkage, have suggested that the water content of a resin may be the prime factor effecting its behavior. Modern theories proposed by Gregor regarding osmatic and elastic forces of the cross-linked network support this view.

Ion exchange processes can be regarded as metathetical chemical reactions between a dissolved compound and the resinous exchanger. The exchanger consists of an insoluble or non-exchanging ion and a soluble or exchanging ion of the opposite polarity. A stoichiometric equation for cation exchange may be written as follows:

$$H^{+} + XR = X^{+} + MR$$

Ion exchange reactions are governed by mass action and therefore can be reversed. The three basic types of equilibrium which exist are: favorable or predominantly in the direction written, unfavorable or predominantly in the reverse direction, and linear or exactly equal.

In the consideration of the mass transfer between the exchanging ions in the liquid and solid phases, three rate controlling steps might be conceived. These steps are as follows: (1) Liquid phase diffusion to the surface of the exchanger particles, (2) Internal diffusion in the exchanger particles, (3) Chemical reaction of exchange at active sites. The step or steps contributing the major resistance would be considered rate controlling.

Boyd et al (4) established that, depending on conditions such as concentration levels, particle size, and liquid turbulence, the rate of ion exchange may be controlled either by particle or film diffusion for a specific ionic system. Some generalized limits based on concentration levels were determined for the controlling diffusional processes. Particle diffusion was found rate controlling at electrolyte concentration of 0.1M or greater and film diffusion controlling at levels less than 0.003M.

Reichenberg (6) supported these data and proposed an equation to determine the controlling diffusional mechanism. This equation was used by Klosowski et al (2) in the study of the chromium hydrogen exchange but failed for particle sizes less than 80 mesh. The equation was also used to tabulate the data taken in the course of this study.

The effect of temperature on ion exchange has not been extensively investigated. Peremyslova et al (3) have indicated that as the temperature of exchange increases the apparent and equilibrium exchange capacities increase correspondingly. The effect of temperature on the total capacity of an exchange resin, however, was not defined. It is possible that at increased temperatures resin micropores might increase in diameter allowing the exchanging ions to reach otherwise hindered exchanging sites.

## EXPERIMENTAL

### Resin

The resin used in this study was Dowex 50 produced by the Dow Chemical Co., Midland, Michigan. The resin is a styrene type nuclear sulfonic acid which is cross-linked with various amounts of divinylbenzene. Both the 2% and 8% (standard) cross-linked resins were used in the course of this study. The resin was received from the manufacturer with specifications as follows:

Dowex 50 X-8

1. 20-50 mesh (H form)
   Total capacity 5.0 ± 0.3 meq./dry gm.
2. 50-100 mesh (H form)
   Total capacity 5.2 ± 0.3 meq./dry gm.
3. 100-200 mesh (H form)
   Total capacity 5.2 ± 0.3 meq./dry gm.
4. 200-400 mesh (H form)
   Total capacity 5.2 ± 0.3 meq./dry gm.

Dowex 50 X-2

1. 20-50 mesh (H form)
   Total capacity 5.2 ± 0.3 meq./dry gm.

In addition to the above resins, samples of Amberlite Resins IRC-120 and IRA-400, produced by Rohm & Haas Co., Philadelphia, Pa., were also obtained. These resins were used in the deionization of all water used in the course of the experimental work. These resins were received from the manufacturer with specifications as follows:

IRC-120

16-50 mesh (H form)
Total capacity 4.85 ± 0.2 meq./dry gm.

IRA-400

16-50 mesh (OH form)
Total capacity 5.75 ± 0.2 meq./dry gm.

## Deionization of Water

Samples of IRC-120 and IRA-400 were regenerated separately in a column-wise operation with an appropriate solution of acid or caustic. Following regeneration, the resins were washed and discharged from the columns. A mixed bed deionization column containing equal volumes of IRC-120 and IRA-400 resin was then set up for deionizing all process and wash water used. The mixed bed technique was employed because it was deemed more efficient than two single bed operations. After deionization, the water was stored in a polyethylene container. The resistance of the water was checked periodically from both the column and container at 10,000,000 ohms specific resistance.

## Resin Preparation

Rate Study. The Dowex 50 X-8 resin used in this study was fractionated into the following eleven cuts:

-16 +20
-20 +30
-30 +40
-40 +50
-50 +60
-60 +70
-70 +80
-80 +100
-100 +150
-150 +200
-200 +325

The fractionation operation was carried out using a set of U. S. Standard Laboratory Screens. To insure that the resin had swelled to its maximum size before screening, the resin was soaked for one week.

The screening operation was carried out by placing the resin to be fractionated on a -16 +20 mesh screen. A stream of tap water was then directed on the screen in a manner such that the resin was moved gently across the screen. A soft paint brush was used to aid the action of the water. The resin passing through the screen was collected in a large white evaporating dish. During the course of the screening the wire became plugged with resin. At this point the resin retained on the screen was discharged and held. The screen was then cleaned with a stiff brush to dislodge resin and the fractionation operation resumed. The screening was continued until no more resin passed through the screen as indicated by the absence of resin in the white collection dish.

The fractionation operation was very slow and became extremely time-consuming in the finer mesh sizes. The screens used were new and were made of stainless steel. On this basis it is believed that the cuts obtained were quite accurate. A microscopic measurement of a few of the various cuts confirmed this conclusion.

Following the screening operation the individual resin cuts were regenerated with a ten-fold excess of a five percent nitric acid solution. The resin cuts were then washed with deionized water until the cut pH reached 5.0 as measured by a Beckman pH Meter. The resin was then vacuum dried for 48 hours at 100°C and 29 inches of vacuum.

The dried resin was subdivided into 1.0000 gram samples which were stored in small bottles. Before the resin was used it was placed in a desicator containing a saturated sodium acetate solution which controlled the relative humidity at 94%. After three days at these conditions, 30 ml of deionized water was added to the sample and it was allowed to stand for an additional three days. At this point the resin was considered fully swollen and ready for use.

<u>Capacity Study</u>. The Dowex 50 X-8 and X-2 resin used in this study was fractionated into a -20 +50 mesh cut in the same manner as outlined in the Rate Study. Following the fractionation,

the resin was transferred to a column for either room temperature or hot regeneration. The resin was subject to two complete regeneration and loading cycles, as outlined in their respective sections, before final regeneration. This technique was employed to insure complete activation before use (7).

Room Temperature Regeneration. The resin to be regenerated was charged to the column shown on Figure No. 1. The resin was then loaded with a 5% sodium hydroxide solution until a titration of the column effluent with standard hydrochloric acid equaled the column feed. At this point the column was washed with deionized water until pH dropped to 7.0. The column was then regenerated with a 5% hydrochloric acid solution until a titration of column effluent with standard sodium hydroxide equaled the column feed. The column was then washed to pH 5.0 with deionized water. The loading cycle was then repeated.

The final regeneration cycle was carried out as outlined above, but the acid flow to the column was continued for one hour after inlet and exit titration values became equal. The resin was washed with deionized water to pH 5.0. The resin was then discharged from the column. The drying, subdivision, and rewetting of the resin was carried out in this same manner, as outlined in the Rate Study. Samples of Dowex 50 X-8 and X-2 were prepared in this manner.

Hot Regeneration (65°C). The resin to be regenerated was charged to the column shown on Figure No. 2. A 5% solution of sodium hydroxide was then charged to tanks A and B. Heat was applied to both the column and tank B. The temperature of the column wall as measured by thermocouple A was adjusted to 65°C by manual adjustment of powerstat A. The temperature in tank B was measured with a glass thermometer and controlled with a Thermo Cap Relay at 70°C. When both the column and tank reached the desired temperature, stopcocks A and B were opened. Flow rate was controlled with stopcock B. Additional caustic was added when required. Approximately four hours were required for the column to reach 65°C. Loading and regeneration times and amounts were gauged from this point. The column was operated continuously until resin had been processed through two complete loading and regeneration cycles.

In the initial attempts at hot regeneration, some difficulties occurred in maintaining the resin bed at a constant temperature during regeneration and loading. This problem was solved after several column revisions. The equipment shown in Figure No. 2 worked satisfactorily.

## Solutions

Ferric Nitrate. Reagent grade ferric nitrate containing $9H_2O$ was used as a source of ferric ions. The solution was made up in a volumetric flask so that 5 ml of this solution contained 5.0 meq. of ferric ion.

Potassium Biphthalate. Reagent grade potassium biphthalate which had been dried at 100°C for twenty-four hours was used as a primary solution standard. The solution was made up by adding a dried sample weighed on an analytical balance to a volumetric flask.

Sodium Hydroxide. Reagent grade sodium hydroxide was used to make all caustic solutions used. The solutions were standardized against the potassium biphthalate solution.

Chromic Nitrate. Reagent grade chromic nitrate containing $9H_2O$ was used as a source of chromic ions. The solution was made up in a volumetric flask so that 5 ml of this solution contained 5.0 meq. of ferric ion.

## Equipment

Rate Study. The apparatus used in this study is shown on Figure No. 3. It consisted of the following components: 1000 ml beaker; Beckman pH Meter, Model N with electrodes; stirrer motor, and two-blade marine-type propellor. The beaker was emptied and cleaned after each run. These operations were carried out by marking the position of all pieces and then removing the beaker from the system. After cleaning, all the equipment was returned to its original position. In this manner it was possible to maintain constant positioning between runs.

The agitator speed was set at the beginning of each run with the electronic control on the stirrer motor. The speed was checked periodically throughout the run using a direct reading tachometer. The position of the propellor and shaft was determined experimentally to minimize vortexing at all speeds. A speed of 400 rpm was found necessary to maintain all size resin particles in suspension. At speeds in excess of 800 rpm vortexing became excessive and liquid was lost over the side.

Capacity Study. The apparatus used in this study is shown on Figure No. 4. It consisted of the following components: 1000 ml, three-necked flask with special opening to insert electrodes; Beckman pH Meter, Model N with electrodes; stirrer motor; Tru Bore stirrer with flat blade; mercury thermometer; reflex condenser; Glasco Heating Mantel; powerstat; and Thermo Cap Relay. The entire assembly was held rigidly in position throughout a complete run. All equipment was marked so that it could be returned to its original position after cleaning and re-assembly.

The agitator speed was set at the beginning of each run with the electronic control on the stirrer motor. The speed was checked with a direct reading tachometer.

The bulk temperature of the batch was controlled to ±0.5°C by the Thermo Cap Relay.

## Procedure

Rate Study. The pH meter was standardized with pH 4.0 buffer. The equipment was then set in the predetermined positions and clamped securely. A 900 ml charge of deionized water was added to the beaker and the temperature checked. Agitator was then started and speed adjusted to the desired setting. Five ml of ferric nitrate solution was propelled from stock solution and added to the beaker. The pH was then recorded.

A 1.0000 gram sample of resin treated as described previously was then added and a stopwatch started. A 15 ml wash of deionized water was used to transfer resin remaining in the bottle to the beaker. This operation was carried out as rapidly as possible. The time required to change 0.10 pH unit was recorded. The run was continued until the desired pH range had been covered.

At the end of a run, the pH meter was again checked against a buffer. If any meter drift was determined, the run was rejected.

Capacity Study. The pH meter was standardized with pH 4.0 buffer. The equipment was then set in the pre-determined positions and clamped securely. A 500 ml charge of deionized water was added to the flask and temperature adjusted to desired point with Thermo Cap Relay. Agitation was then adjusted to the desired setting. The pH was then determined and recorded.

A 1.000 gram sample of resin treated as described previously was then added. A standardized sodium hydroxide solution was then added until pH was raised back to the initial value. Amount of caustic required was recorded.

The solution was then checked every eight hours and any additional caustic required was added. At the end of twenty-four hours the run was considered complete. The pH meter was again checked against a buffer. If a meter drift greater than 0.1 pH unit was detected, the run was rejected.

## CALCULATIONS

### Rate Study

The data taken during this phase of the investigation are given in Table No. 1. The criterion used for the acceptance or rejection of a run was the drift of the pH meter during the course of the run. If this drift was under 0.05 pH units the run was accepted. Several runs were rejected on this basis. Duplicate runs under the same test conditions gave excellent experimental checks.

The experimental data were analyzed using the kinetic treatment of Reichenberg who uses the equation shown below to define a differential exchange rate for liquid diffusion in the case of a single resin particle.

(1) $dQ/dt = 4\pi r^2 D/\Delta r \; (Co - C)$

where

$dQ/dt$ - Exchange rate meq./sec

$r$ - Average particle radius cm

$D$ - Diffusion coefficient $cm^2/sec$

$\Delta r$ - Effective film thickness microns

$Co$ - Concentration in the liquid immediately in contact with the particle surface. meq./ml

$C$ - Concentration in the bulk solution or at the outside of the film. meq./ml

The following assumptions were made in order to justify the substitution of the quantity $4\pi r^2 D/\Delta r$ for the mass transfer coefficient generally used in rate equations:

1. All resin particles are spherical in shape.

2. The film thickness is small compared to the radius of a particle and thus the effective surface area available for diffusion may be assumed equal to the surface area of the spherical particle.

3. The size change of a resin particle due to conversion from one ionic form to another may be neglected.

4. The film thickness is constant with respect to the direction of diffusion, and the ionic hold-up in the film is small compared to total capacity of the resin particle.

5. The exchange rate is film diffusional controlled and therefore, a) concentration gradients inside the resin particle are negligible; b) instantaneous steady state exchange is achieved; c) rate of diffusion in either direction is equal on an equivalent basis.

6. Concentration gradients in the bulk solution are negligible.

7. Diffusion coefficient does not vary within the concentration range utilized.

In order to utilize this equation to calculate the film thickness, it is first necessary to convert the equation from a single resin particle basis to a unit volume of resin basis. This may be done by defining a quantity $\phi$ as the meq. exchanged per ml of swollen resin.

$$(2) \quad \phi = \frac{Q}{4/3\pi r^3}$$

Now, differentiating Eq. (2) and substituting in Eq. (1), the following expression results:

$$(3) \quad d\phi/dt = \frac{3D}{r\Delta r}\left[Co-C\right]$$

Now, considering the case of monovalent-trivalent ion exchange and assuming the ratio of activity coefficients is unity, the following expression results:

$$(4) \quad \frac{q/Q}{(1-q/Q)^3} = K\,\frac{C/Co}{(1-C/Co)^3}$$

If this expression is solved for C considering only the cases of low value of q/Q and substituted in Eq. (3), the following expression results:

$$(5) \quad d\phi/dt = \frac{3D}{r\Delta r}\left[Co\right]$$

The film thickness can now be calculated by substitution in Eq. (5). The $d\phi/dt$ data taken from Figures No. 5 to 11 are shown on Table No. 4. The particle size radius used was taken from the data shown on Table No. 3. The diffusion coefficient for ferric ion was calculated from information given by Mellor (8) and the International Critical Tables (9). The results of the film thickness calculations are shown on Table No. 5.

If, as proposed, the film thickness is constant for all particle sizes at a fixed speed, equation (1) reduces to the following expression for fixed concentrations, temperatures, and agitator speeds:

(6) $dQ/dt = 4\pi r^2 \text{(Constant)}$

or $dQ/dt \cdot A = \text{Constant}$

This expression can be used to check the proposed film thickness theory using experimental data.

Capacity Study

The data taken during this phase of the investigation are given in Tables No. 10, 11, and 12. The criterion used for acceptance of a run was the drift of the pH meter during the course of the run. All runs where this drift was greater than 0.10 pH units were rejected.

The data in Tables No. 10 and 11 were analyzed by calculating the standard deviation as outlined by Moroney (10). The standard deviation for the data shown on Table No. 10 and 11 were 0.07 and 0.05 meq. per gram dry resin respectively. Applying this deviation to the data, it can be seen that no variation of resin capacity occurred with temperature.

## DISCUSSION

### Rate Study

The data shown on Table No. 4 gives the rate of exchange per unit volume of resin at various agitator speeds for the seven particle size ranges studied. The data was obtained from the rate information plotted on Figures Nos. 5 to 11. These data show an increased rate of exchange for both increased agitator speed at a fixed particle size range and decreased particle size at a fixed agitator speed. It can be concluded that the rate of ion exchange for the operating conditions and ionic system studied varied directly with the agitator speed and inversely with the particle size range. Since these are the conditions which define a film diffusional controlled process, it follows that the exchange studied was film diffusional controlled. This finding is in agreement with the results of Mar and David (11) who found that the ferric ion-Dowex 50 system was film diffusional controlled in fixed-bed operations for total solution molarities up to 0.5M.

### Film Thickness

Using the exchange rate information shown on Table No. 4, the film thickness was calculated by substitution of the appropriate quantities in equation No. 5. The results of these calculations are shown on Table No. 5. The film thickness was constant at a constant agitator speed for resin sizes from -16 to +60 mesh and decreased at resin sizes below 70 mesh. These results are in fair agreement with the data of Klosowski et al (2) for the chromic ion-Dowex 50 system. In this study the decreasing film thickness occurred for resin sizes below 80 mesh. No theoretical reason based on agitation theory or Stokes Law analysis can be advanced to explain the phenomenon. It is believed, however, that the break point is due to the invalidation of the assumption that film thickness is small compared with particle radius. A plot of the ratio of the film thickness vs particle radius is shown on Figure No. 12. From this plot it can be seen that at about 60 mesh the slope increases sharply with particle size.

## Film Thickness The Same For All Particle Sizes

In the preceding sections it has been shown that at particle sizes above 70 mesh the film thickness was constant for a fixed agitator speed. No conclusion could be reached on the film thickness for particle sizes less than 70 mesh.

It has been proposed by Klosowski et al (2) that the film thickness is completely independent of particle size and solely a function of agitator speed. If true, then the equation

$$dQ/dt \cdot A = \text{Constant}$$

must be valid.

The values of dQ/dt are shown on Table No. 6 and were obtained from Figures Nos. 14 to 20. These data are subject to some experimental error due to the linear equilibrium between ferric and hydrogen exchange and the pH range investigated. In this range, differences of 0.10 pH units represent large changes in hydrogen ion concentration. This caused a wide separation of data points which made the determination of the initial slope difficult. The values of A and subsequently the value of dQ/dt•A were calculated on three different bases.

In the first method, the area normal to the direction of diffusion was calculated based on a particle radius including the film thickness. These data are shown on Table No. 7 and indicate a direct relationship of exchange rate with particle size.

In the second method, the exchange area was calculated neglecting the addition of the film thickness to the particle radius. These data are shown on Table No. 8 and indicate an inverse relationship of exchange rate with particle size.

In the third method, the exchange area was calculated including one-half of the film thickness in addition to the particle radius. These data are shown on Table No. 9 and indicate that the rate is constant.

Based on this information, it might be stated that the film thickness is independent of particle size and constant at a fixed agitator speed. It should be pointed out, however, that the film thickness is an effective, not an absolute value. It is probable that the actual film thickness does vary with particle size since the mass to surface area ratio is changing with decreasing particle size. With this change, the forces acting on particles of different sizes at a fixed agitator speed will also vary. The number of particles, however, will also increase sharply with decreasing particle size for a fixed amount of resin. This will increase the number of particle collisions shearing off additional segments of film. An entirely different particle flow pattern is also set up as the number of particles increases. The net effect of these forces must cancel out and lead to the conclusion that the film thickness is independent of particle size.

### Effect of Agitator Speed On Film Thickness

It can be seen from Figure No. 13 that as the agitator speed increases the film thickness decreases for a fixed particle size. In the region studied, the film thickness seems to be reaching a limiting value where increased speed will shear off little or no additional film. This effect is also indicated by the data of Reichenberg (6) who found no significant effect on the rate of exchange over the range 500 to 2500 rpm. This would indicate no significant change in film thickness was effected over the range studied.

This effect would be expected in an unbaffled agitated system. The structure of the resin particles is that of a porous gel whose shape varies from irregular to spherical. High shearing forces would be required to overcome the forces of adhesion and cohesion which secure the film to the particles. In order to effect further reduction in film thickness, it would be necessary to carry out the exchange in a fully baffled vessel with turbine type agitator.

## Comparison Of Calculated Film Thickness With Existing Data

The average film thickness found for the first three resin cuts shown on Table No. 5 are listed below with the corresponding values determined by Klosowski et al (2) for the chromic ion-Dowex 50 system.

| Agitator Speed rpm | Film Thickness (microns) Ferric Ion-Dowex 50 | Film Thickness (microns) Chromic Ion - Dowex 50 |
|---|---|---|
| 400 | 18.2 | 19.4 |
| 600 | 15.3 | 15.5 |
| 800 | 13.3 | 13.3 |

These data are in excellent agreement. In view of this agreement, the possibilities of employing this technique for the rapid determination of diffusion coefficients are excellent.

The experimental procedure is relatively simple and the laboratory equipment required is generally readily available in most laboratories.

In the proposed mode of measurement there are two limitations. The first is that the system to be studied must be film diffusional controlled, and second, the pH of an aqueous solution should be greater than 3.0. Since most exchange processes are film diffusional controlled at low concentrations, the first limitation would only apply to the concentrated level to be studied. The second limitation is not firm and could be varied depending on the degree of accuracy required. Since the rate of exchange would be measured by a change in pH, the closer the measurement is made to 7.0 the greater the accuracy. This is due to the logarithmic relationship between pH and hydrogen ion concentration.

In applying this method, one would determine the rate of ion exchange at 600 rpm for a screened resin fraction and then substitute the data taken in the equation shown below.

$$D = 0.00051 \frac{r}{(Co)} (d\phi/dt)$$

Continuous Ion Exchange

The two modes of continuous ion exchange considered most feasible have been analyzed in view of the data taken during this investigation. Several design modifications have been suggested based on these analyses.

Hiester et al (13) have developed a mixer settler type operation in which the liquid stream moves countercurrent to the resin which remains in a fixed reactor throughout the operation. Although this operation is not truly continuous, it can be instrumental to give completely automatic operation. In a film diffusional controlled process, the retention time in each mixer can be minimized by increasing agitator speed to reduce film thickness. Since the film thickness approaches a minimum value with increased agitation, an optimum value can be set on this variable. In the settler portion of the cycle the particle size of the resin will determine the settling rate. Since the film thickness is dependent solely on agitator rate, the rate of exchange per unit area will remain fixed for all particle sizes. A particle size for operation can then be selected based on settling velocity as determined by Stokes Law.

An improvement in this operation could be effected by installation of baffles in the mixers. This would increase shear on the resin particles decreasing film thickness.

Higgins et al (14) have designed a moving bed ion exchange column in which resin is charged and discharged from the column by a hydraulic pulse. This system operates basically like a fixed bed exchange except the resin is slowly moved throughout the column.

An improvement could be effected in the column by adding pulse periods in which no resin was transferred. If the pulses imparted to the column were low amplitude high frequency type, the advantage of agitation could be added to the column. This would aid in reducing film thickness and increase rate for film diffusional controlled processes.

## Capacity Study

In general, the capacity of an ion exchange resin is given in terms of the number of millequivalents of ions exchanged. This definition, however, is not complete since there are two possible types of resin capacity. These are the equilibrium capacity and the total capacity. Articles by Streicher et al (12) and Peremyslova et al (3) report variations in resin capacity with increased temperatures but are not specific as to the type of capacity. The data taken in this study are in terms of the total capacity and are an attempt to define a relationship between capacity and temperature.

The data shown in Tables Nos. 10 and 11 give the total capacity of Dowex 50 X-8 and X-2 ion exchange resin for the sodium hydrogen exchange at various temperatures. The resin used in this part of the study was regenerated at room temperature. A statistical analysis of these data shows no variation in the total resin capacity takes place as the loading temperature is varied.

Since the resin used in this study was regenerated at room temperature, it was believed that some of the active sites which could be reached during loading at high temperatures were not regenerated at the lower temperature. On this basis the regeneration was carried out at the higher temperatures (65°C) to insure the activation of all possible exchange sites. The data shown on Table No. 12 gives the results of a loading study using the hot regenerated resin. These data show no increase in capacity with increased loading temperatures. The average capacity of the hot regenerated resin was 5.14 meq. per dry gram compared to 4.90 meq. per dry gram obtained using room temperature regenerated resin. This would seem to indicate that a slight increase in resin capacity can be obtained using resin which has been regenerated at higher temperatures.

It is interesting to note that the data of Peremyslova et al (3) when analyzed shows a 100% increase in the equilibrium capacity for the calcium-hydrogen exchange with a 60°C change in the loading temperature. The rate of exchange based on equilibrium capacities, however, was the same for both temperatures.

## Equilibrium Capacity of Dowex 50 X-8 For Ferric and Chromic Ions

The equilibrium capacity of Dowex 50 X-8 ion exchange resin was 13.2 and 9.89 meq. per dry gram for ferric ion and chromic ion respectively. These capacities were determined using one gram samples of resin which were allowed to equilibriate in 250 ml of 0.95N solutions of the respective ions for 48 hours. At the concentration levels studied, it is believed that the equilibrium capacities and total capacities of the resin for each ion were equal. Manufacturer's specification for this resin for monovalent ions is 5.0 ± 0.3 meq. per dry gram. No data on the capacities for di and trivalent ions are available.

# SUMMARY

This study was directed toward the extension of the data on the kinetics of monovalent - trivalent cation exchange and the establishment of data on the relationship between temperature and total capacity of an ion exchange resin.

The kinetic study showed the exchange of ferric ion and hydrogen ion to be film diffusional controlled at a concentration of 0.00527N. The film thickness was found to vary directly with agitator speed in the range 400 to 800 rpm and seemed to be reaching a minimum thickness for the agitation range studied. This concept seems real since it is difficult to increase shear on a particle in an unbaffled vessel by increasing agitator speed. In order to effect further reductions in film thickness, it would be necessary to carry out the exchange in a baffled reactor with turbine type agitator.

The equation proposed by Reichenberg (6) to calculate film thickness checked experimental data for particle sizes from -16 to +60 mesh but failed for particle sizes below 70 mesh.

The film thickness was shown to be constant for all particle sizes at a fixed agitator speed. It should be noted,

however, that the film thickness is an effective not an absolute value. It is probable that the absolute value does vary since the mass to surface area ratio is changing with decreasing particle size. The effective value, however, remains constant to the increased number of particles per unit weight which cause increased collisions between particles and alter the particle flow pattern.

A comparison of the film thickness calculated at 400, 600, and 800 rpm with existing data shows the values to be within 6%, 1%, and exactly equal to corresponding values. Since the diffusion coefficient for the ferric and chromic ion systems are $0.57 \times 10^{-5}$ and $0.642 \times 10^{-5}$ $cm^2/sec$. respectively, the agreement on the values of the film thickness is as expected. In view of this agreement, the experimental method used in this study with a modified form of the Reichenberg Equation is proposed to calculate diffusion coefficients. In applying this method, one would determine the rate of ion exchange at 600 rpm for a screened Dowex 50 resin fraction and then substitute the data taken in the equation shown below.

$$D = 0.0051 \frac{r}{(Co)} \frac{d\phi}{dt}$$

where D - Diffusion Coefficient

r - Average particle radius

Co - Initial concentration in the bulk solution

$d\phi/dt$ - Exchange rate per unit volume

The above method should aid in the rapid determination of diffusion coefficients at low concentrations in aqueous solutions. In these cases the processes are generally film diffusional controlled and no viscosity changes take place.

In applying the results of this study to continuous ion exchange, it is seen that optimum agitator speeds can be selected for the mixer settler system proposed by Hiester (13). An optimum resin particle size can also be selected based on rate information and Stokes Law analysis.

No change in resin capacity was found by variation in loading temperature from 0°C to 100°C. Some change in capacity, however, was noted between resin regenerated at 65°C over resin regenerated at 25°C.

The capacity of Dowex 50 X-8 ion exchange resin as determined for ferric ion and chromic ion was 13.2 and 9.89 meq. per gram of dry resin respectively. These capacities are of the equilibrium type and were determined using solutions with initial concentrations of 0.95 meq. per milliliter. At this concentration level equilibrium and total capacities should be approximately equal. No data on trivalent capacity of this resin is available but monovalent capacity is 5.0±0.3 meq. per dry gram.

## SUGGESTIONS

Based on the results of this investigation, experimental work on the following subjects is recommended:

1. The proposed method for the determination of diffusion coefficients should be tested on systems with known coefficients to further test its validity. If valid, this procedure could be used to determine many of the diffusion coefficients currently unavailable in the literature.

2. A kinetic study of anion exchange should be carried out to determine if a similar approach to anion diffusion coefficients is applicable.

3. The range of particle sizes studied should be expanded to mesh sizes below 200 mesh to ascertain if film thickness remains constant at a fixed agitator speed. This information would be basic to fluid mixing and leaching theory.

4. In the range of film diffusion the effects of higher agitation speeds and baffling on the present system should be determined to ascertain there the relationship with the film thickness.

5. The relationship between regeneration temperature and ion exchange capacity should be extended to higher regeneration temperatures.

6. The effect of temperature on the rate of exchange should be studied.

# NOMENCLATURE

| | |
|---|---|
| $A$ | Area normal to diffusion - $cm^2$ |
| $A_1$ | Area normal to diffusion based upon the effective particle size at the periphery of the film - $cm^2$ |
| $A_2$ | Area normal to diffusion based upon the effective particle size at the mid-point of the film - $cm^2$ |
| $A_3$ | Area normal to diffusion based upon the effective particle size neglecting the presence of the film - $cm^2$ |
| $C$ | Concentration in the bulk solution or at the outside of the film - meq./ml. |
| $Co$ | Concentration in the liquid immediately in contact with the particle surface - meq./ml. |
| $d_s$ | Average screen particle diameter - cm |
| $D$ | Diffusion coefficient - $cm^2$/sec. |
| $N$ | Number of particles calculated per gm. of dry resin |
| $Q$ | Amount of exchange - meq. |
| $r$ | Average particle radius determined by screen analysis - cm |

| | |
|---|---|
| $\Delta r$ | Effective film thickness - microns |
| $t$ | Time - sec. |
| $X$ | Percent exchange |
| $\phi$ | Amount of exchange per unit volume - meq./ml. |

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## TABLE 1

## AVERAGE TIME - pH DATA

$Fe^{+++}$ - 0.00527N

| | Speed - rpm | 400 | 600 | 800 |
|---|---|---|---|---|
| | pH | | Time - Sec. | |
| -16 +20 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 147 | 138 | 131 |
| | 2.60 | 406 | 364 | 309 |
| | 2.50 | 797 | 665 | 556 |
| | 2.40 | 1408 | 1144 | 944 |
| | 2.30 | 2296 | 1974 | 1616 |
| | 2.20 | 4074 | 3600 | 2987 |
| -30 +40 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 102 | 80 | 70 |
| | 2.60 | 224 | 185 | 160 |
| | 2.50 | 390 | 320 | 285 |
| | 2.40 | 659 | 515 | 485 |
| | 2.30 | 1145 | 905 | 850 |
| | 2.20 | 2170 | 1820 | 1750 |
| -50 +60 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 50 | 40 | 35 |
| | 2.60 | 110 | 96 | 85 |
| | 2.50 | 205 | 175 | 156 |
| | 2.40 | 335 | 290 | 257 |
| | 2.30 | 600 | 480 | 435 |
| | 2.20 | 1400 | 900 | 830 |
| -70 +80 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 32 | 27 | 22 |
| | 2.60 | 69 | 60 | 50 |
| | 2.50 | 126 | 107 | 92 |
| | 2.40 | 210 | 175 | 151 |
| | 2.30 | 380 | 305 | 280 |
| | 2.20 | 957 | 655 | 585 |
| -80 +100 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 22 | 20 | 18 |
| | 2.60 | 49 | 45 | 41 |
| | 2.50 | 100 | 80 | 76 |
| | 2.40 | 158 | 145 | 127 |
| | 2.30 | 285 | 245 | 223 |
| | 2.20 | 684 | 532 | 455 |

## TABLE 1 (Cont'd.)

| | Speed - rpm | 400 | 600 | 800 |
|---|---|---|---|---|
| | pH | | Time - Sec. | |
| -150 +200 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 14 | 12 | 11 |
| | 2.60 | 31 | 27 | 25 |
| | 2.50 | 56 | 49 | 45 |
| | 2.40 | 94 | 80 | 72 |
| | 2.30 | 170 | 140 | 125 |
| | 2.20 | 410 | 346 | 295 |
| -200 +325 | 2.80 | 0 | 0 | 0 |
| | 2.70 | 10 | 7 | 6 |
| | 2.60 | 22 | 17 | 15 |
| | 2.50 | 39 | 33 | 31 |
| | 2.40 | 75 | 70 | 53 |
| | 2.30 | 160 | 140 | 103 |

## TABLE 2

## EXTENT OF REACTION AS A FUNCTION OF pH

| pH | (H)x$10^3$ Moles or gm.eq.Lit. | Total meq. $H^+$ per 950 ml Solution | Total Change in meq. $H^+(Fe^{+++})$ $\theta$ | X $\theta/5.11$ | $\phi = \frac{\text{cap.-meq.}}{\text{ml Wet Resin}}$ X·2.55 |
|---|---|---|---|---|---|
| 2.80 | 1.585 | 1.505 | 0 | 0 | 0 |
| 2.70 | 1.995 | 1.895 | 0.390 | 0.0764 | 0.1950 |
| 2.60 | 2.51 | 2.38 | 0.865 | 0.1693 | 0.433 |
| 2.50 | 3.16 | 3.00 | 1.495 | 0.2925 | 0.747 |
| 2.40 | 3.98 | 3.78 | 2.275 | 0.445 | 1.137 |
| 2.30 | 5.01 | 4.76 | 3.255 | 0.637 | 1.628 |
| 2.20 | 6.31 | 6.00 | 4.495 | 0.879 | 2.245 |

## TABLE 3

## PARTICLE SIZE DATA

| Fraction | Avg. Particle Diameter (ds) (Screen) cm. | Avg. Particle Radius (r) (Screen) cm. | Wet Volume per gm. Dry Resin (Experimental) cm$^3$ | Calculated No.(N) of Particles per gm. Dry Resin (2.00 ml Wet Resin) |
|---|---|---|---|---|
| -16 +20 | 0.102 | 0.051 | 2.00 | 3,610 |
| -30 +40 | 0.0505 | 0.0252 | 2.00 | 29,900 |
| -50 +60 | 0.0274 | 0.0137 | 2.00 | 186,100 |
| -70 +80 | 0.0194 | 0.0097 | 2.00 | 525,000 |
| -80 +100 | 0.0163 | 0.0081 | 2.00 | 901,000 |
| -150 +200 | 0.00889 | 0.00444 | 2.00 | 5,450,000 |

## TABLE 4

## INITIAL VALUES OF $d\phi/dt$

$(d\phi/dt)$ initial - meq./ml. Swollen Resin Sec.

| | Speed - rpm | | |
|---|---|---|---|
| Fraction | 400 | 600 | 800 |
| -16 +20 | 0.00100 | 0.001175 | 0.00136 |
| -30 +40 | 0.001900 | 0.002360 | 0.002680 |
| -50 +60 | 0.003650 | 0.004330 | 0.004900 |
| -70 +80 | 0.005850 | 0.006950 | 0.008150 |
| -80 +100 | 0.007500 | 0.008550 | 0.010500 |
| -150 +200 | 0.012100 | 0.014300 | 0.016300 |
| -200 +325 | 0.014800 | 0.018800 | 0.021600 |

## TABLE 5

## CALCULATED FILM THICKNESSES

| | Film Thickness - Microns | | |
|---|---|---|---|
| | Speed - rpm | | |
| Fraction | 400 | 600 | 800 |
| -16 +20 | 17.70 | 15.05 | 13.00 |
| -30 +40 | 18.80 | 15.15 | 13.35 |
| -50 +60 | 18.00 | 15.20 | 13.40 |
| -70 +80 | 15.90 | 13.38 | 11.41 |
| -80 +100 | 14.85 | 13.01 | 10.60 |
| -150 +200 | 13.40 | 10.55 | 9.16 |

## TABLE 6

## EXCHANGE RATES

| | dQ/dt - meq. Fe exchanged/Sec. | | |
|---|---|---|---|
| | Speed - rpm | | |
| Fraction | 400 | 600 | 800 |
| -16 +20 | 0.00186 | 0.00224 | 0.00274 |
| -30 +40 | 0.00380 | 0.00464 | 0.00530 |
| -50 +60 | 0.00820 | 0.00950 | 0.01050 |
| -70 +80 | 0.01170 | 0.01380 | 0.01500 |
| -80 +100 | 0.01540 | 0.01650 | 0.0190 |
| -150 +200 | 0.02780 | 0.02990 | 0.03340 |

## TABLE 7

### $A_1$ Total Effective Area at Periphery of Film ($cm^2$)

| | Speed - rpm | | |
|---|---|---|---|
| Fraction | 400 | 600 | 800 |
| -16 +20 | 127.36 | 124.96 | 122.59 |
| -30 +40 | 274.79 | 258.73 | 256.42 |
| -50 +60 | 561.56 | 540.04 | 525.92 |
| -70 +80 | 841.99 | 805.14 | 776.16 |
| -80 +100 | 1053.81 | 1010.58 | 957.82 |
| -150 +200 | 2302.72 | 2070.67 | 1959.26 |

### EXCHANGE RATES PER UNIT AREA

| | $10^5 \times dQ/dt/A_1$ (meq. Fe/Sec. $cm^2$) | | |
|---|---|---|---|
| -16 +20 | 1.460 | 1.795 | 2.23 |
| -30 +40 | 1.543 | 1.790 | 2.07 |
| -50 +60 | 1.462 | 1.760 | 2.00 |
| -70 +80 | 1.389 | 1.713 | 1.930 |
| -80 +100 | 1.460 | 1.632 | 1.98 |
| -150 +200 | 1.208 | 1.443 | 1.705 |

## TABLE 8

### $A_3$ Total Effective Area at Periphery of Film (cm$^2$)

| | Speed - rpm | | |
|---|---|---|---|
| Fraction | 400 | 600 | 800 |
| -16 +20 | 117.92 | 117.92 | 117.92 |
| -30 +40 | 238.16 | 238.16 | 238.16 |
| -50 +60 | 438.85 | 438.85 | 438.85 |
| -70 +80 | 619.43 | 619.43 | 619.43 |
| -80 +100 | 751.66 | 751.66 | 751.66 |
| -150 +200 | 1352.47 | 1352.47 | 1352.47 |

### EXCHANGE RATES PER UNIT AREA

| | $10^5$ x dQ/dt/$A_3$ (meq. Fe/Sec. cm$^2$) | | |
|---|---|---|---|
| -16 +20 | 1.578 | 1.903 | 2.33 |
| -30 +40 | 1.595 | 1.948 | 2.23 |
| -50 +60 | 1.870 | 2.17 | 2.40 |
| -70 +80 | 1.890 | 2.23 | 2.43 |
| -80 +100 | 2.250 | 2.20 | 2.53 |
| -150 +200 | 2.055 | 2.21 | 2.47 |

## TABLE 9

### $A_2$ Total Effective Area at Film Midpoint (cm$^2$)

| | | Speed - rpm | |
|---|---|---|---|
| Fraction | 400 | 600 | 800 |
| -16 +20 | 122.13 | 121.42 | 120.95 |
| -30 +40 | 256.81 | 253.86 | 251.92 |
| -50 +60 | 498.24 | 488.06 | 481.33 |
| -70 +80 | 726.99 | 706.37 | 692.78 |
| -80 +100 | 896.37 | 876.34 | 856.33 |
| -150 +200 | 1780.44 | 1694.23 | 1646.89 |

### EXCHANGE RATES PER UNIT AREA

| | $10^5$ x dQ/dt/$A_2$ (meq. Fe/Sec. cm$^2$ | | |
|---|---|---|---|
| -16 +20 | 1.523 | 1.845 | 2.27 |
| -30 +40 | 1.480 | 1.832 | 2.11 |
| -50 +60 | 1.648 | 1.943 | 2.18 |
| -70 +80 | 1.612 | 1.950 | 2.17 |
| -80 +100 | 1.718 | 1.885 | 2.22 |
| -150 +200 | 1.563 | 1.765 | 2.03 |

# TABLE 10

## CAPACITY OF DOWEX 50 X-8 AT VARIOUS TEMP.

(Regenerated at room temperature)

| Loading Temperature °C | Capacity Meq./Gm. dry resin |
|---|---|
| 0 | 5.01 |
| 2 | 4.86 |
| 25 | 4.77 |
| 35 | 4.89 |
| 35 | 4.89 |
| 35 | 4.94 |
| 40 | 4.90 |
| 54 | 4.81 |
| 54 | 5.05 |
| 72 | 4.83 |
| 94 | 4.99 |

## TABLE 11

## CAPACITY OF DOWEX 50 X-2 AT VARIOUS TEMP.

(Regenerated at room temperature)

| Loading Temperature °C | Capacity Meq./Gm. dry resin |
|---|---|
| 1.5 | 4.91 |
| 20 | 4.91 |
| 35 | 4.87 |
| 35 | 5.04 |
| 52 | 4.86 |
| 61 | 4.94 |
| 75 | 5.06 |
| 100 | 5.00 |

## TABLE 12

## CAPACITY OF DOWEX 50 X-8 AT VARIOUS TEMP.

(Regenerated at 65°C)

| Loading Temperature °C | Capacity Meq./Gm. dry resin |
|---|---|
| 2 | 5.21 |
| 35 | 5.10 |
| 78 | 5.11 |

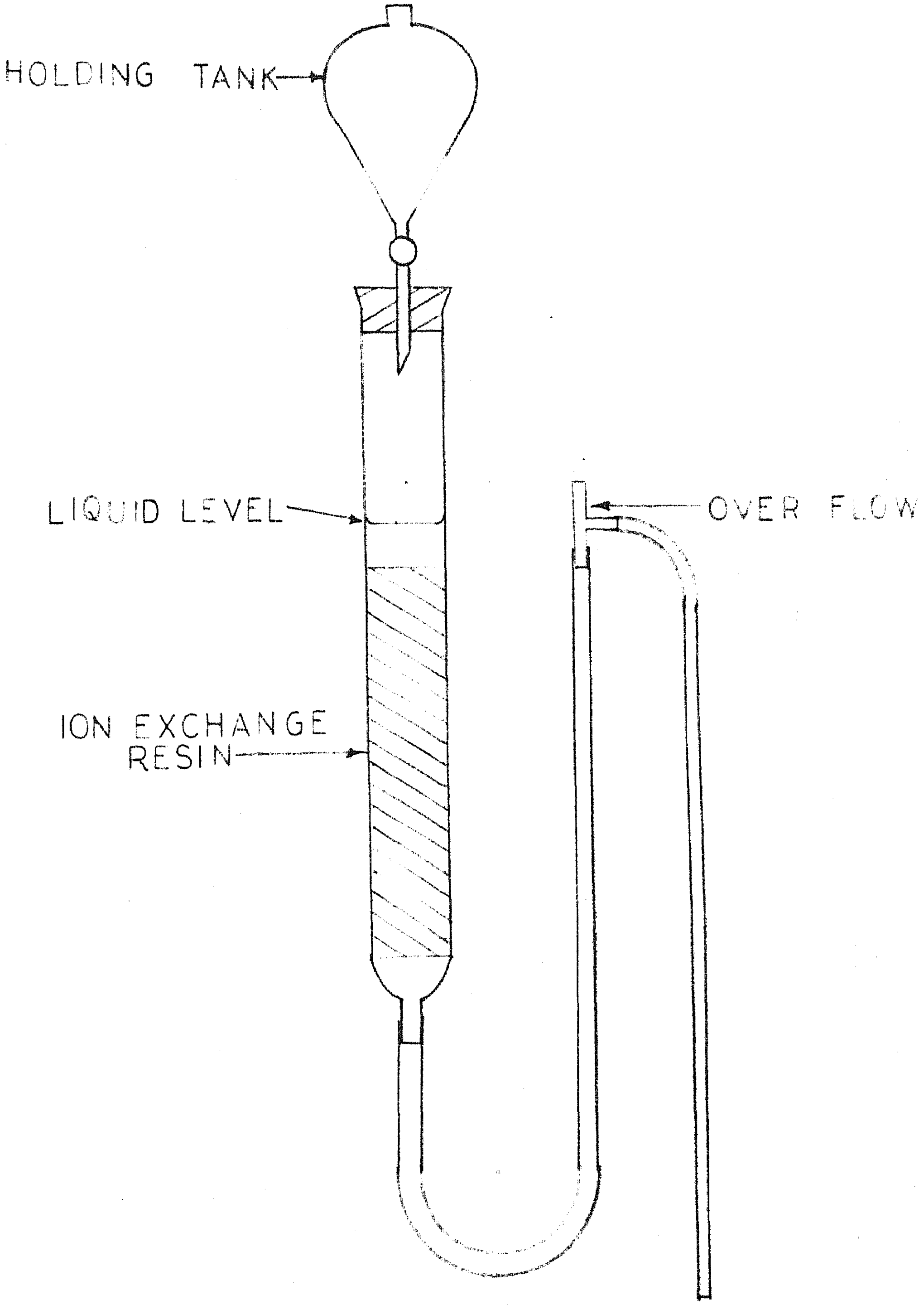

FIGURE NO.1

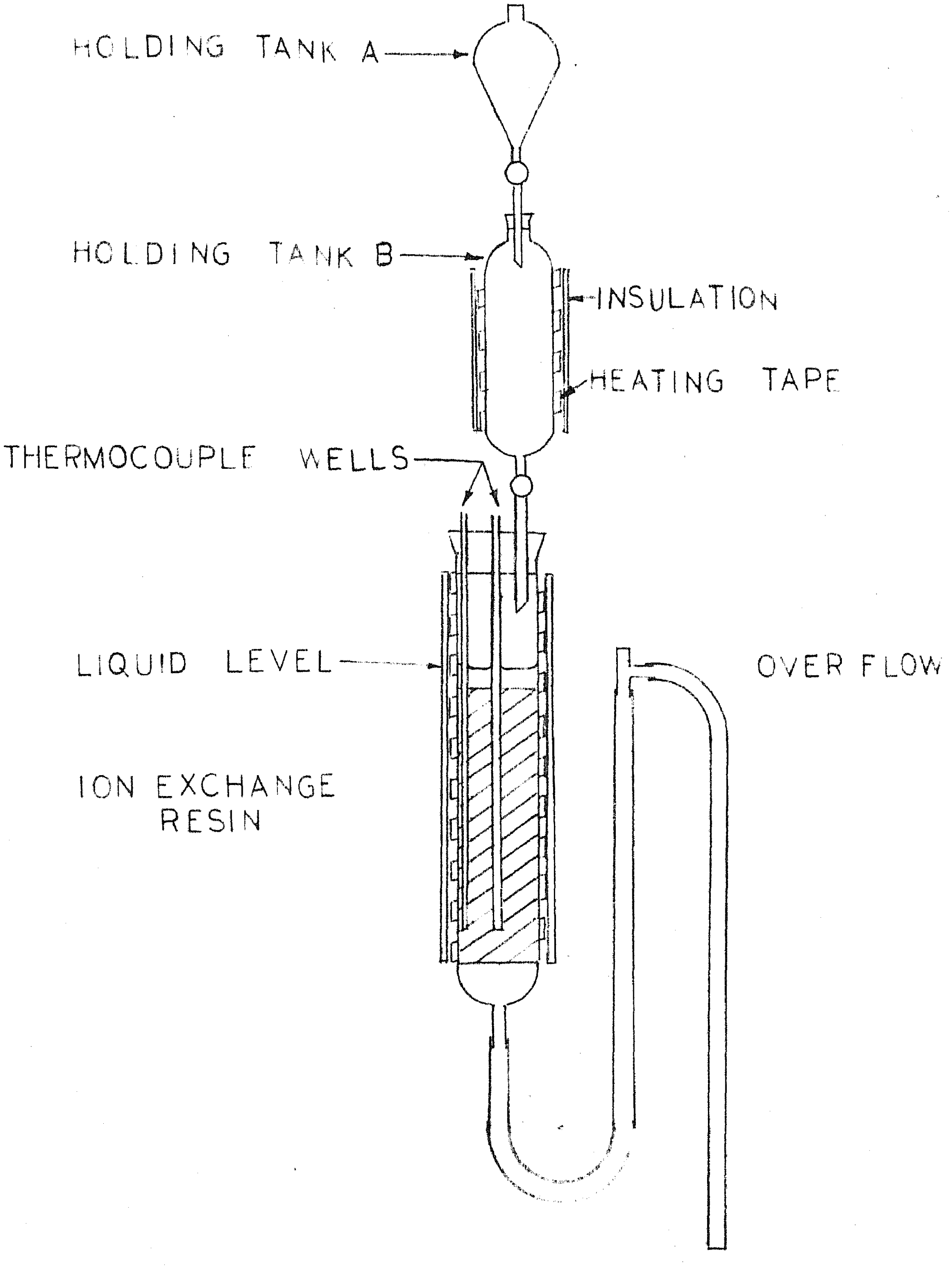
HOLDING TANK A
HOLDING TANK B
INSULATION
HEATING TAPE
THERMOCOUPLE WELLS
LIQUID LEVEL
OVER FLOW
ION EXCHANGE RESIN

FIGURE NO. 2

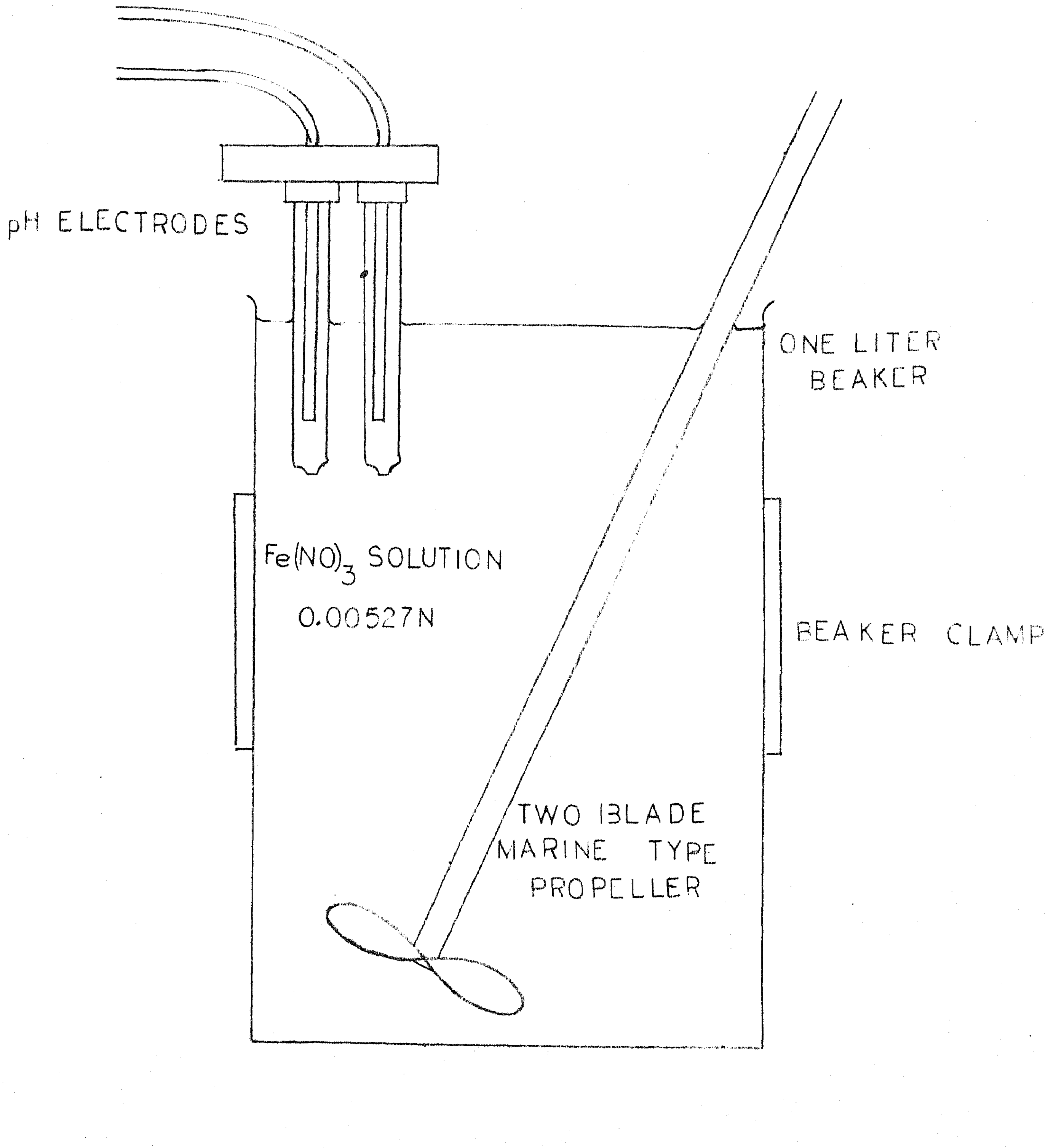
pH ELECTRODES
ONE LITER BEAKER
$Fe(NO)_3$ SOLUTION
0.00527N
BEAKER CLAMP
TWO BLADE MARINE TYPE PROPELLER

FIGURE NO. 3

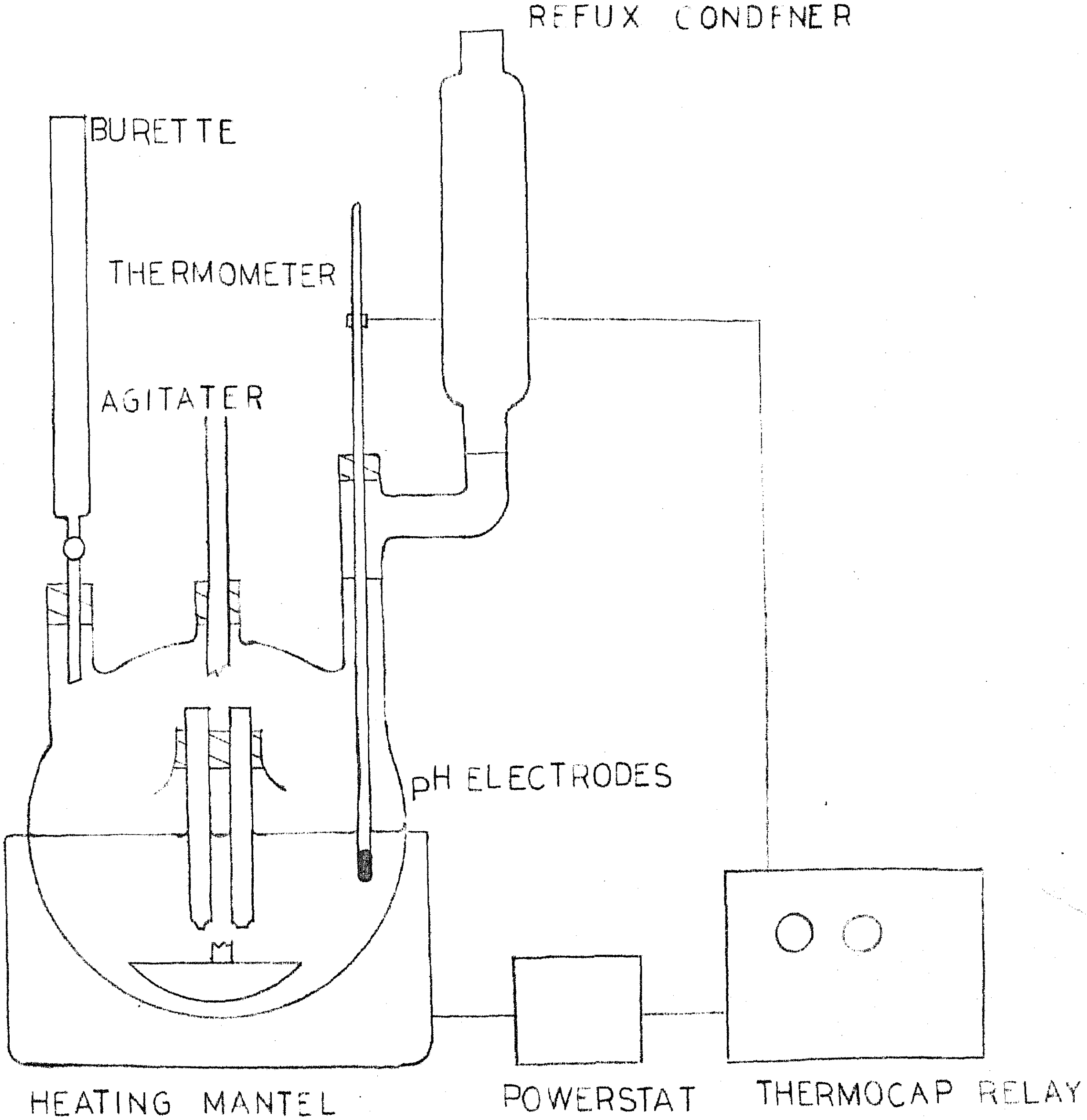
REFUX CONDENER
BURETTE
THERMOMETER
AGITATER
pH ELECTRODES
HEATING MANTEL
POWERSTAT
THERMOCAP RELAY

FIGURE NO.4

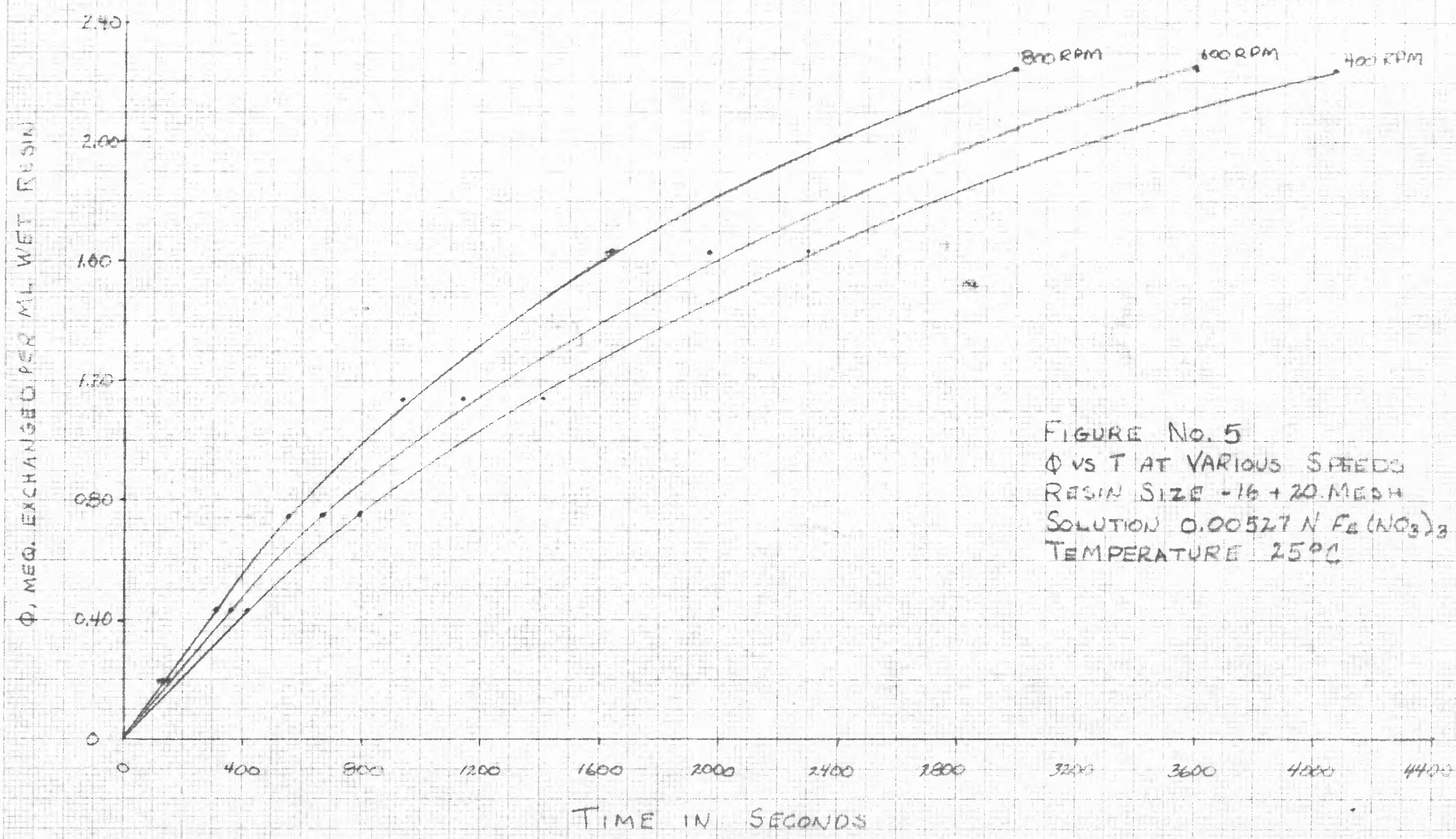

FIGURE No. 5
Φ VS T AT VARIOUS SPEEDS
RESIN SIZE -16 + 20 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25°C

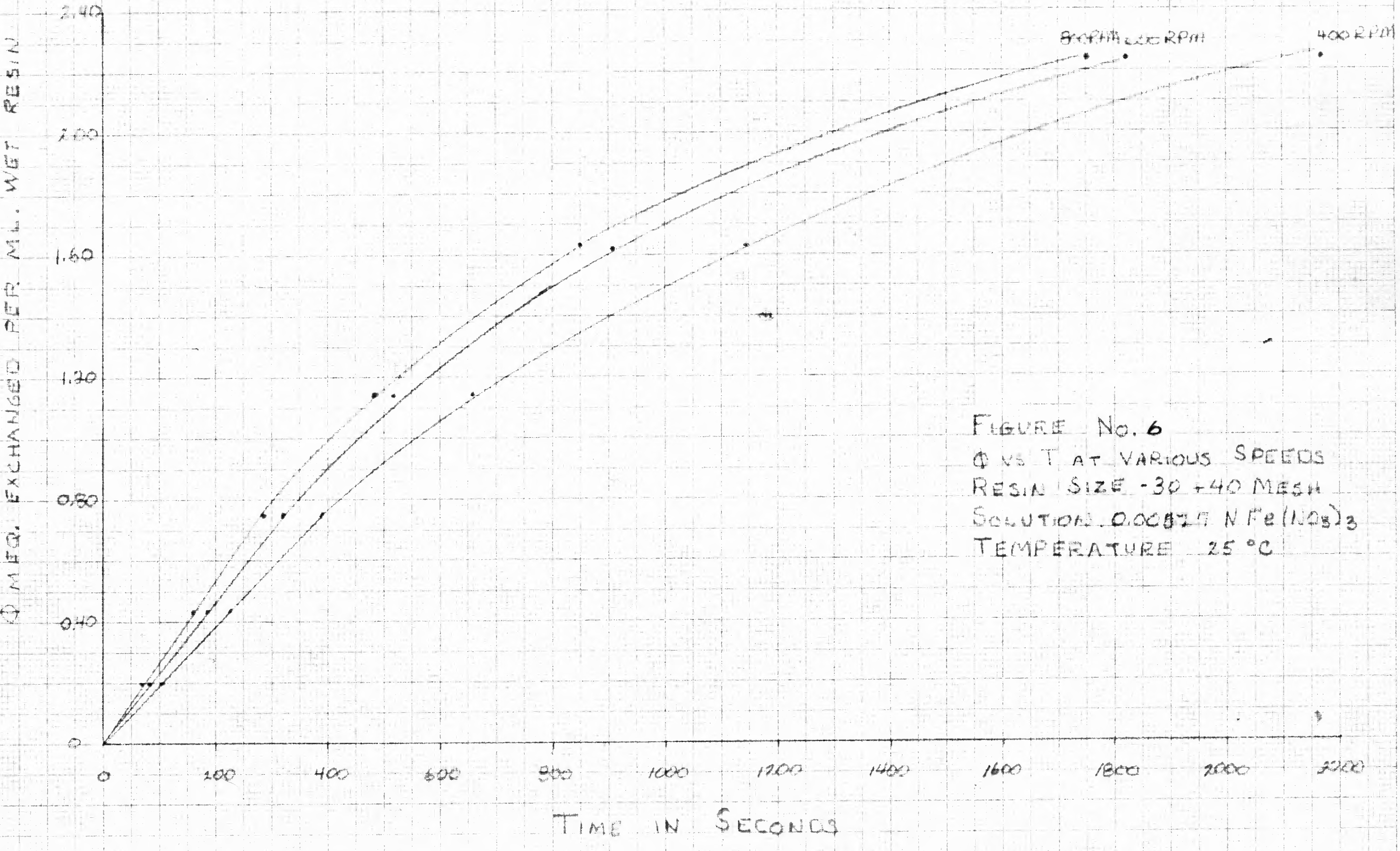

FIGURE No. 6

Φ VS T AT VARIOUS SPEEDS

RESIN SIZE -30 +40 MESH

SOLUTION 0.00827 N $Fe(NO_3)_3$

TEMPERATURE 25 °C

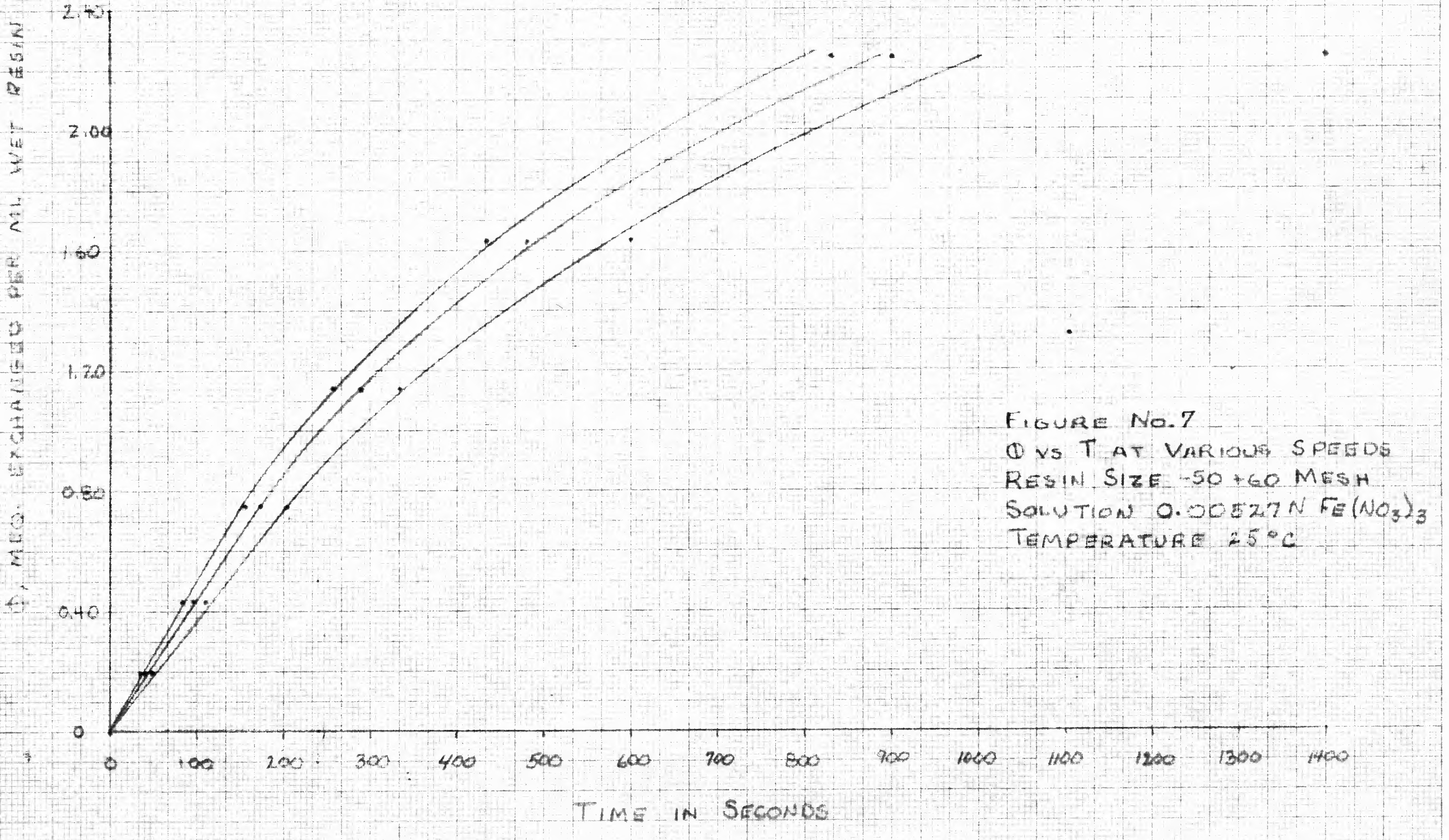

Figure No. 7
Θ vs T at Various Speeds
Resin Size -50 +60 Mesh
Solution 0.00527 N $Fe(NO_3)_3$
Temperature 25°C

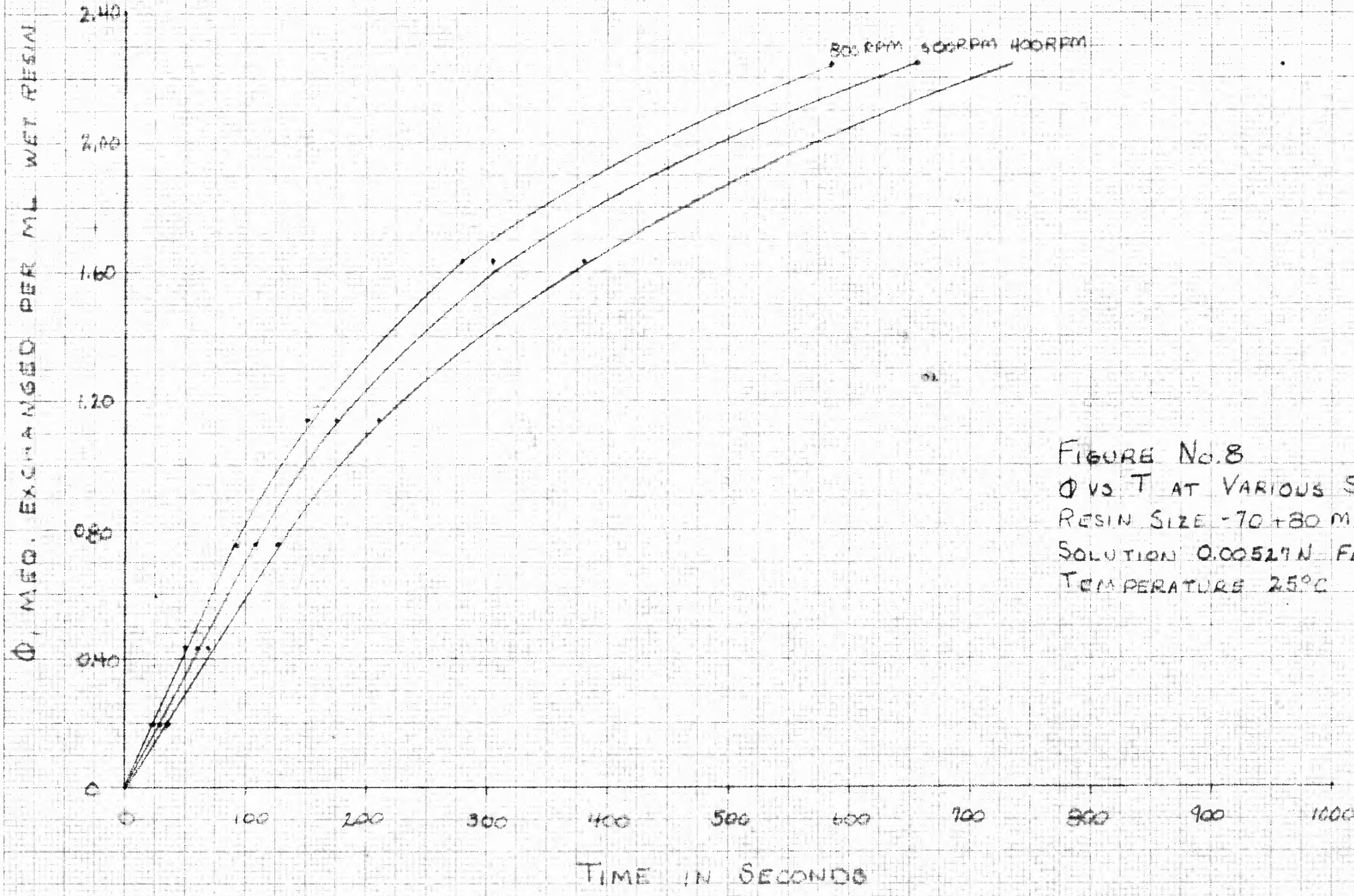

FIGURE No. 8
$\Phi$ VS T AT VARIOUS SPEEDS
RESIN SIZE -70 +80 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25°C

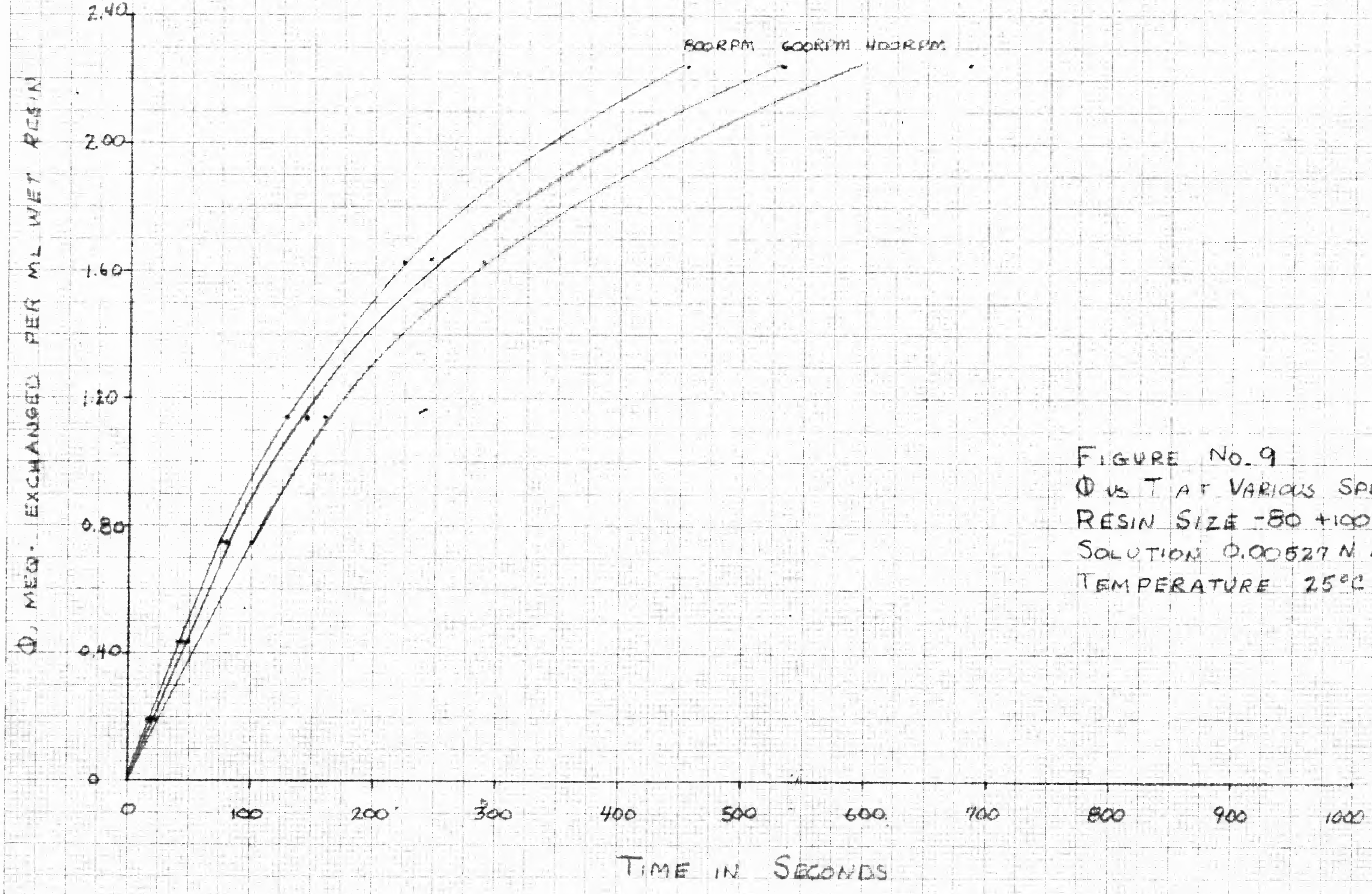

FIGURE NO. 9
Φ vs T AT VARIOUS SPEEDS
RESIN SIZE -80 +100 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25°C

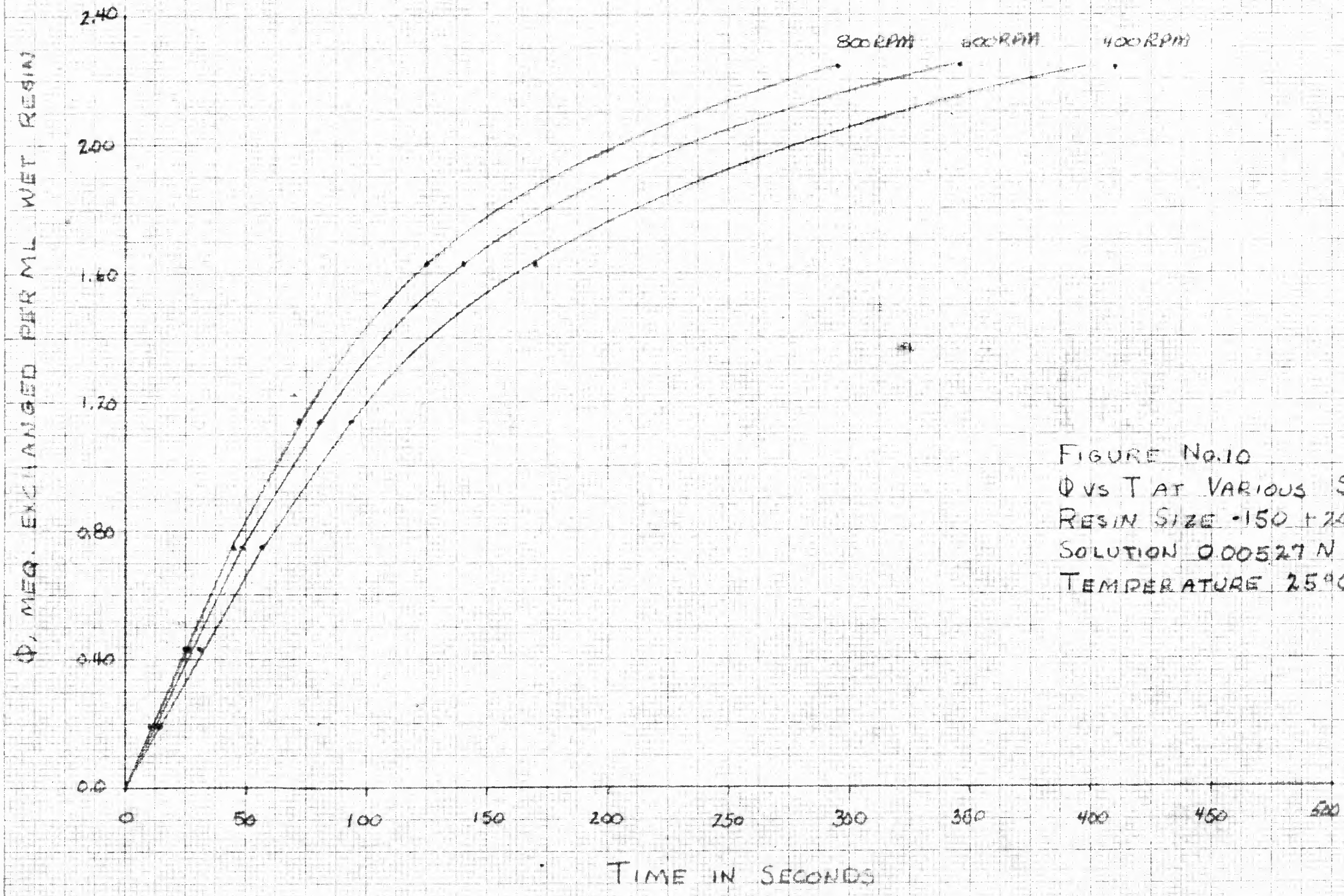

FIGURE No.10
Φ VS T AT VARIOUS SPEEDS
RESIN SIZE -150 +200 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25°C

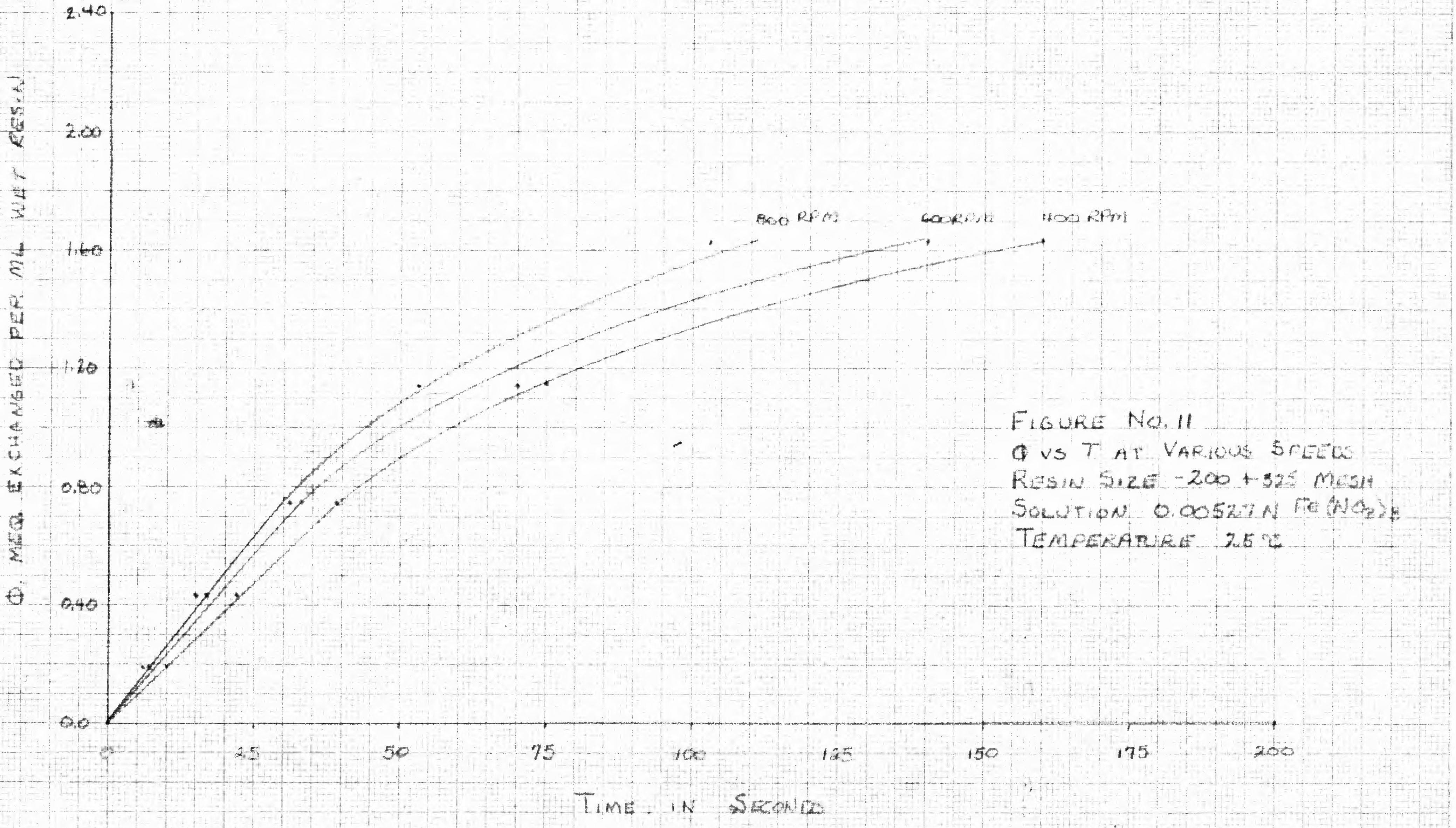

FIGURE NO. 11
Θ VS T AT VARIOUS SPEEDS
RESIN SIZE -200 +325 MESH
SOLUTION 0.00527 N $Fe(NO_2)_3$
TEMPERATURE 25 °C

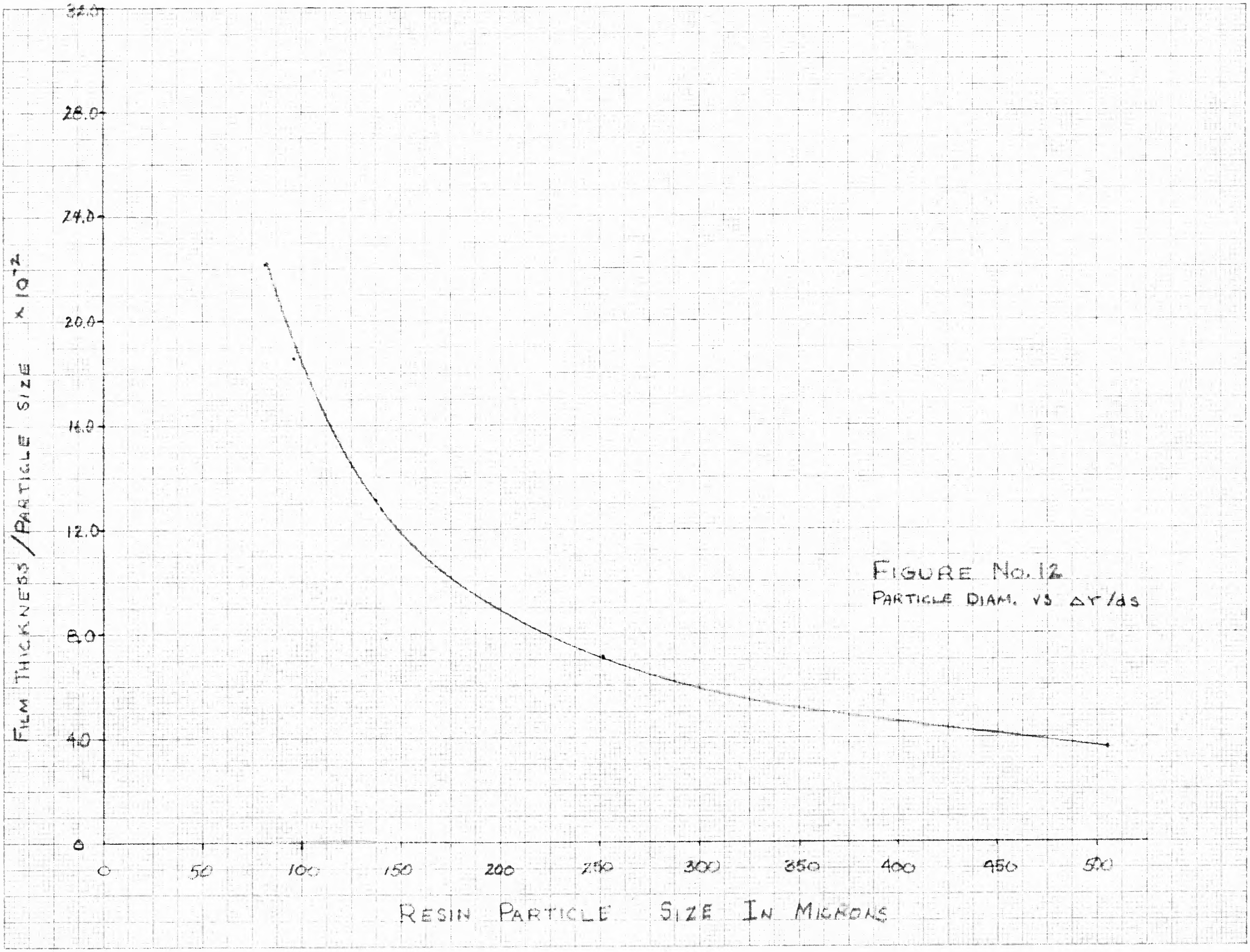

FIGURE No. 12
PARTICLE DIAM. VS $\Delta Y/ds$

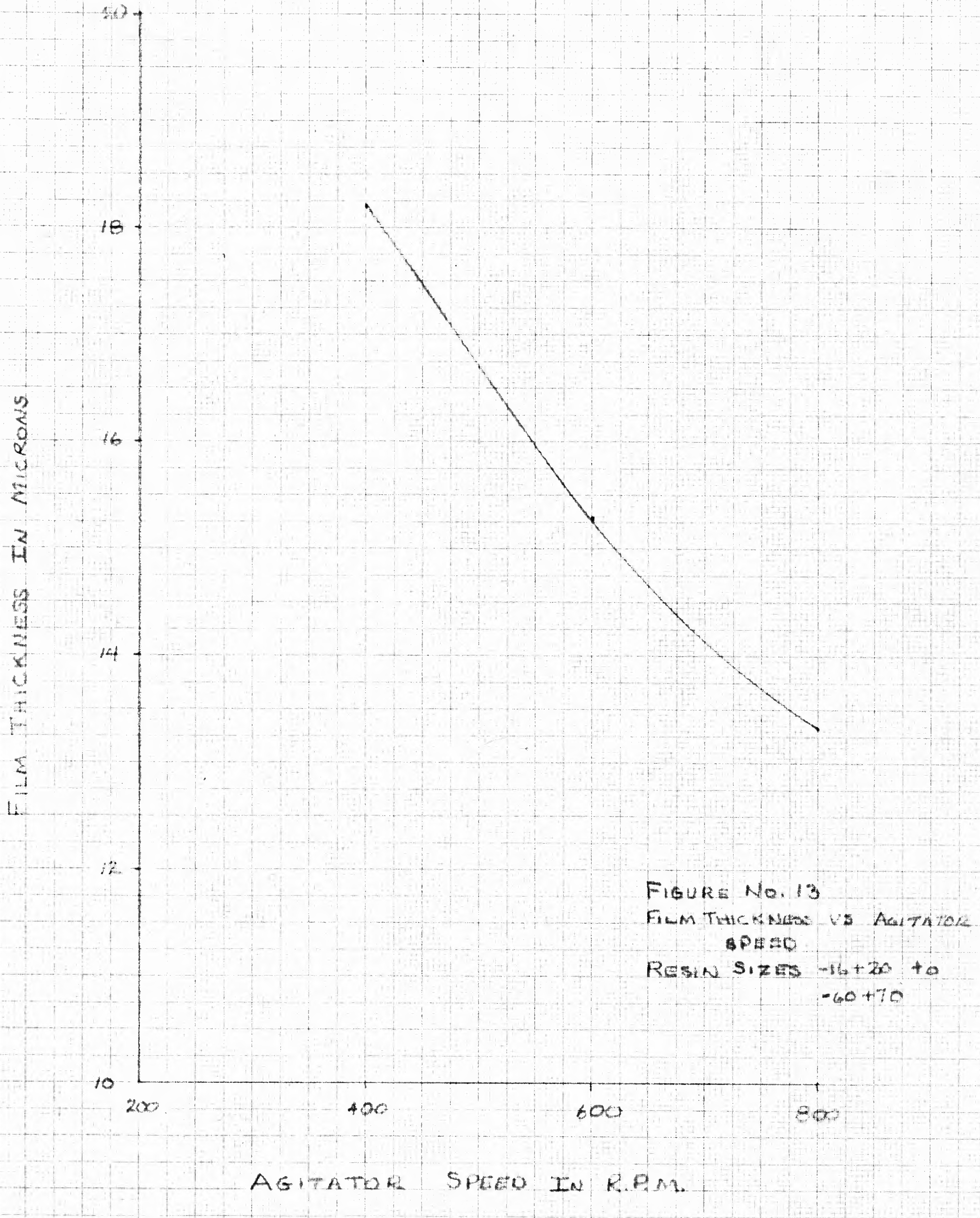

FIGURE NO. 13
FILM THICKNESS VS AGITATOR SPEED
RESIN SIZES -16+20 to -60+70

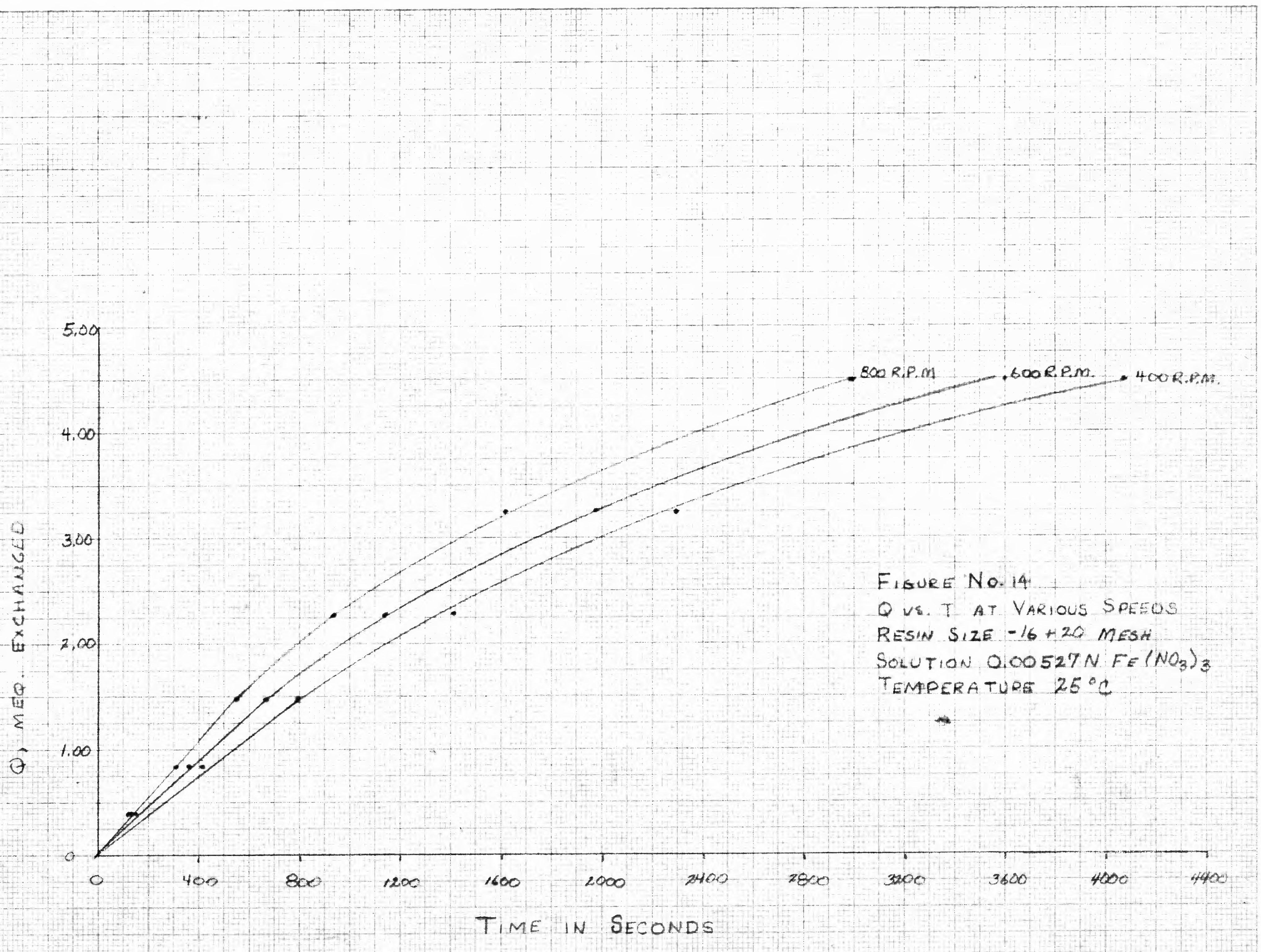

FIGURE NO. 14
Q VS. T AT VARIOUS SPEEDS
RESIN SIZE -16 +20 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25 °C

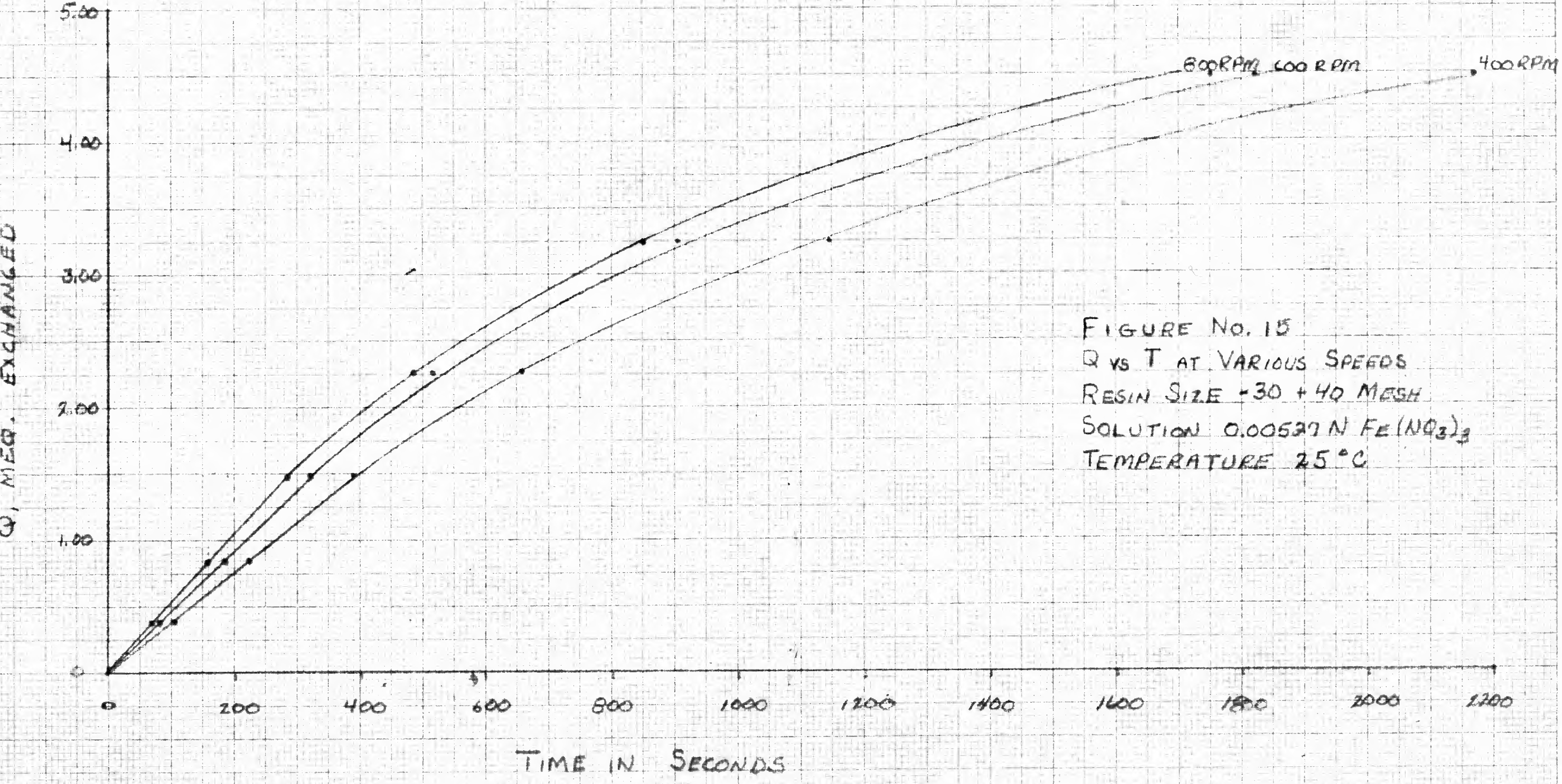

FIGURE NO. 15

Q VS T AT VARIOUS SPEEDS

RESIN SIZE -30 + 40 MESH

SOLUTION 0.00527 N $Fe(NO_3)_3$

TEMPERATURE 25 °C

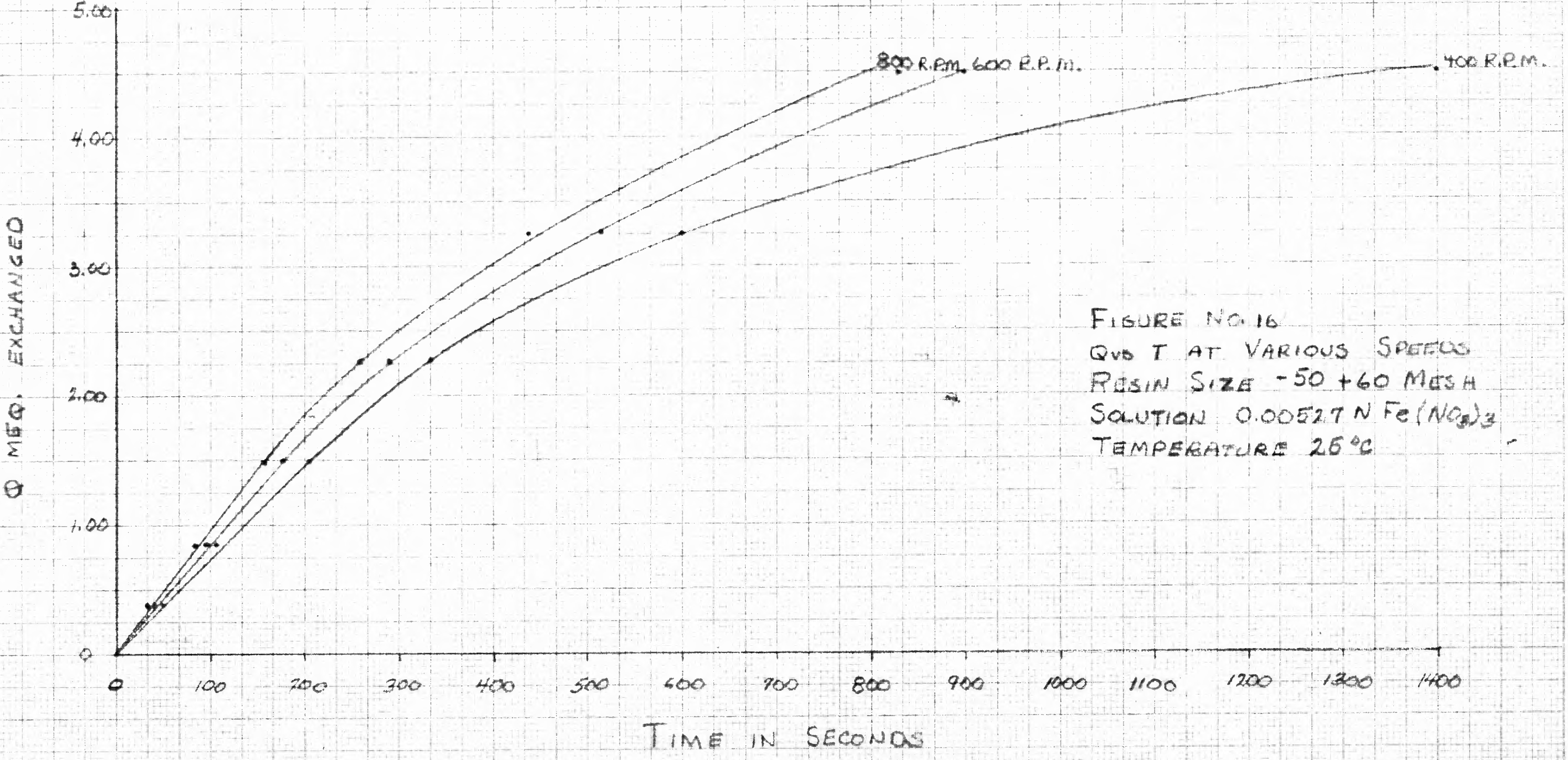

FIGURE NO. 16
Q VS T AT VARIOUS SPEEDS
RESIN SIZE -50 +60 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25 °C

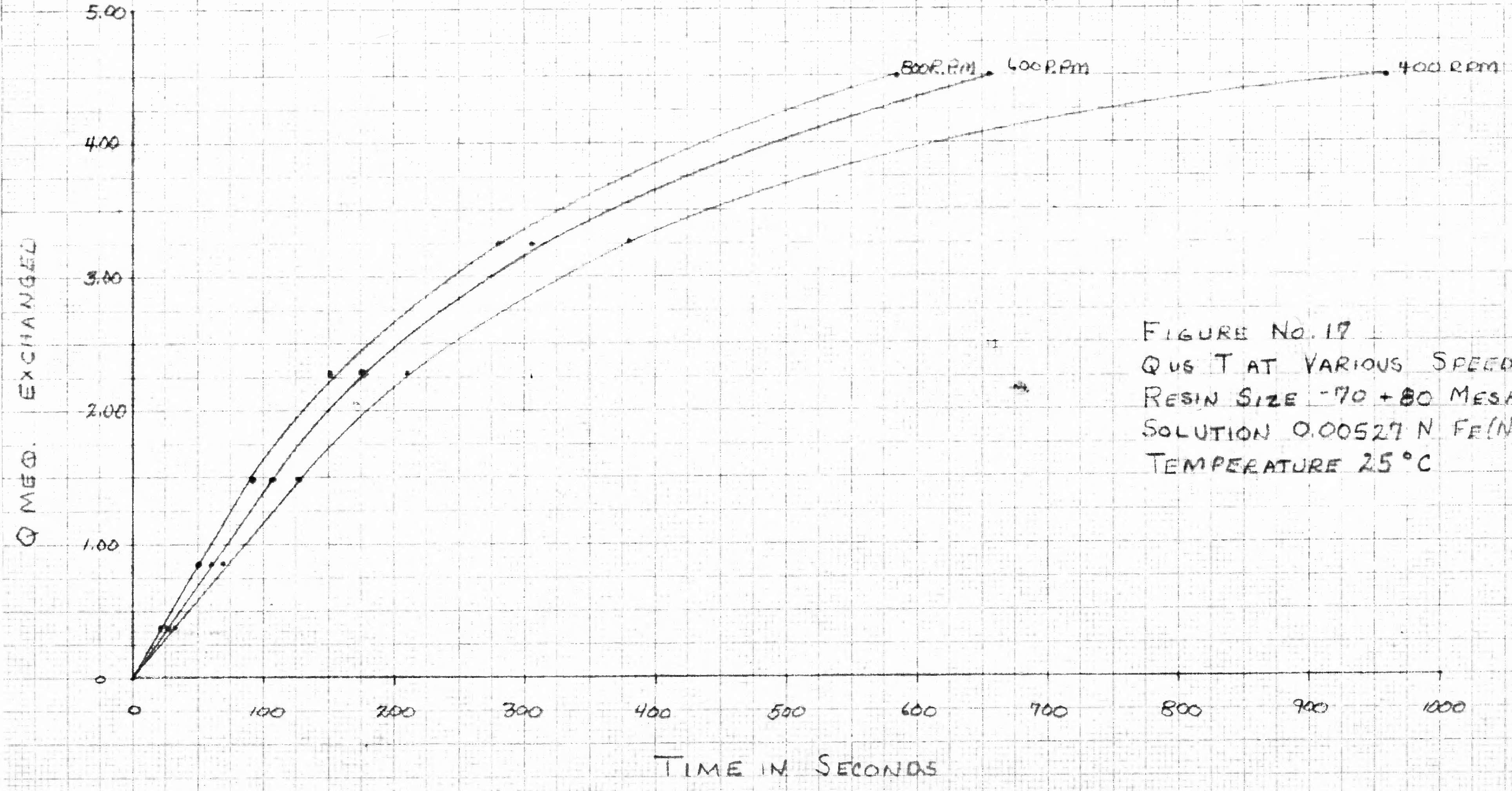

FIGURE NO. 17
Q vs T AT VARIOUS SPEEDS
RESIN SIZE -70 +80 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25 °C

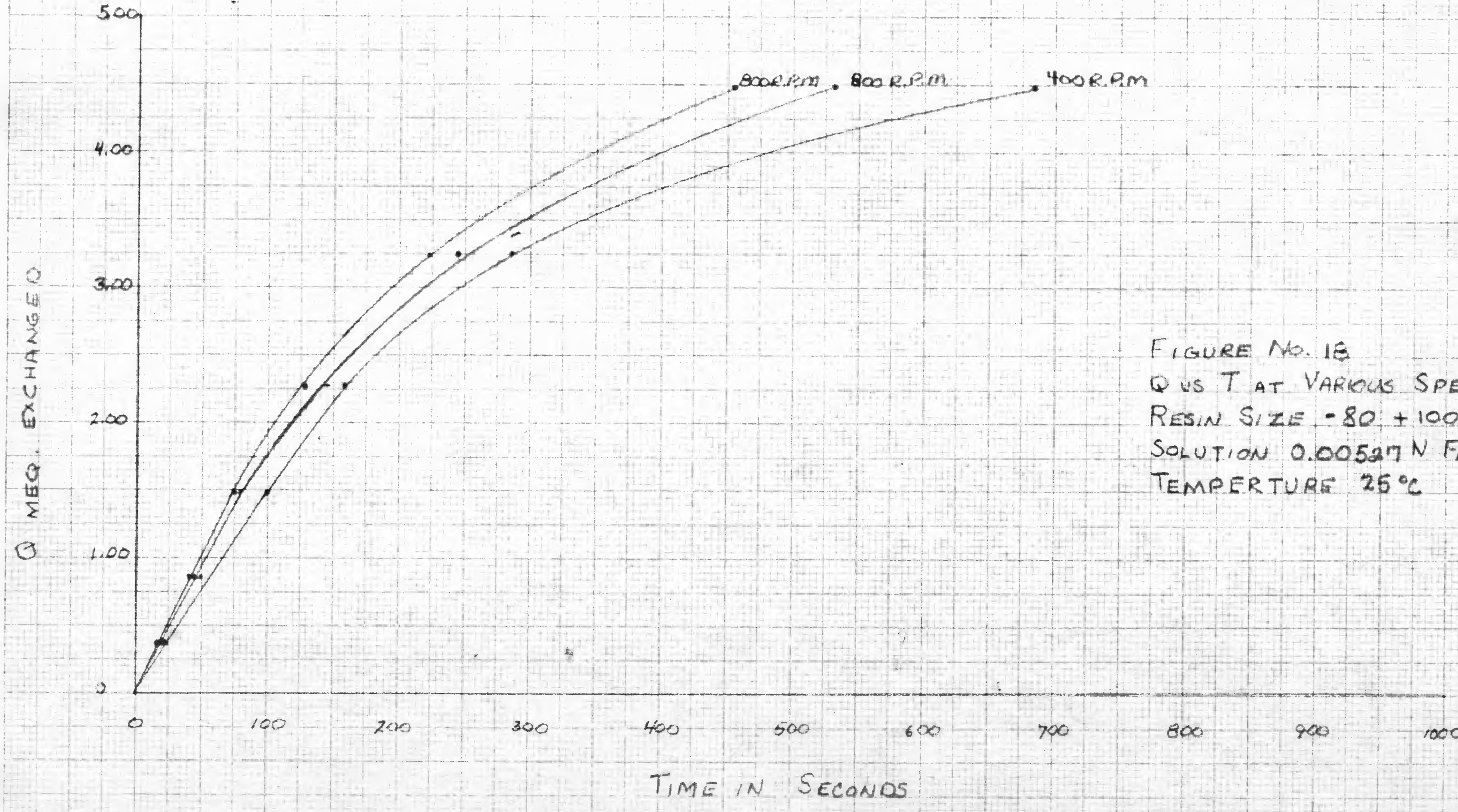

FIGURE No. 18
Q VS T AT VARIOUS SPEEDS
RESIN SIZE -80 +100 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERTURE 25 °C

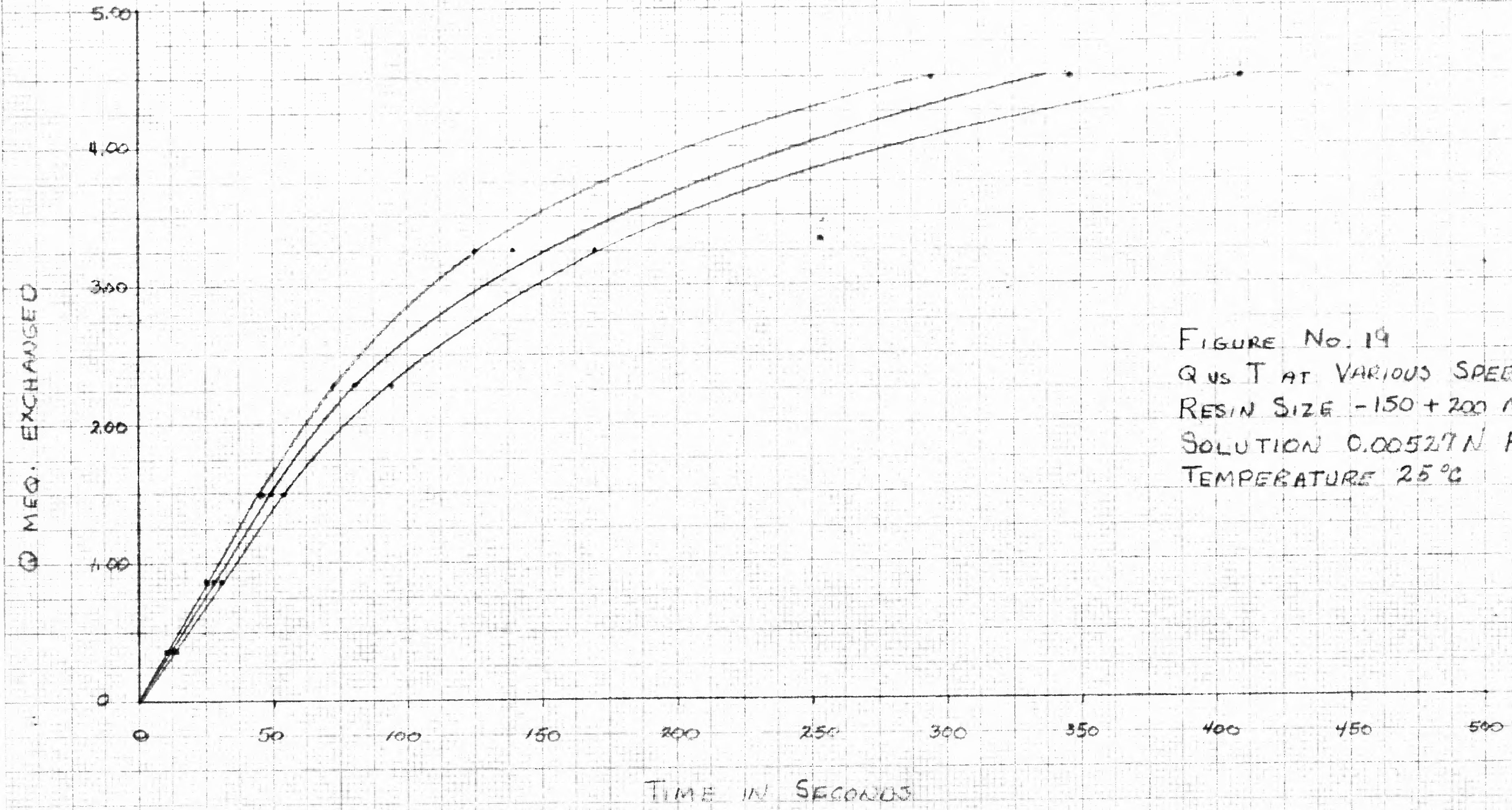

FIGURE NO. 19
Q vs T AT VARIOUS SPEEDS
RESIN SIZE -150 + 200 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25 °C

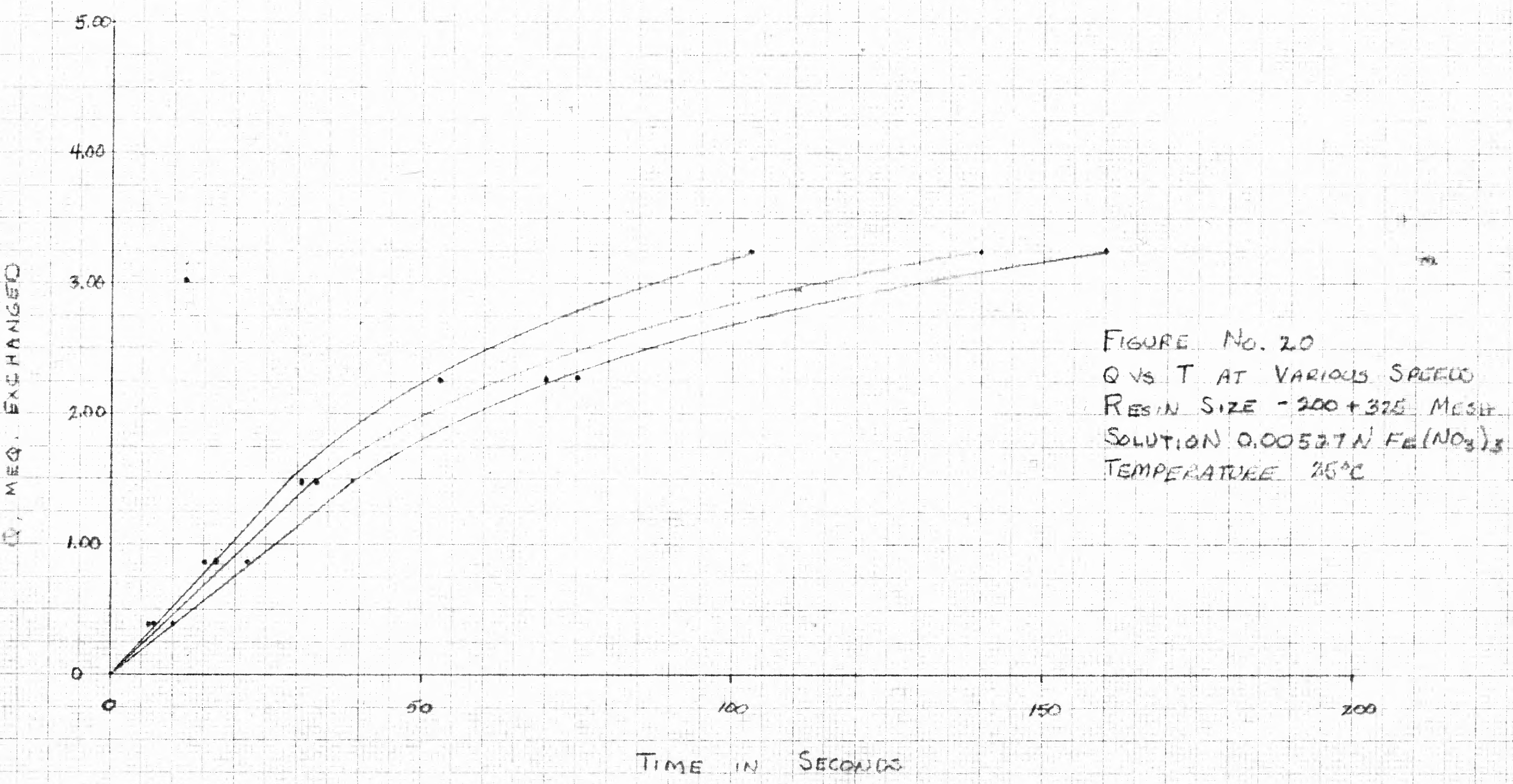

FIGURE NO. 20
Q VS T AT VARIOUS SPEEDS
RESIN SIZE -200 +325 MESH
SOLUTION 0.00527 N $Fe(NO_3)_3$
TEMPERATURE 25°C