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THE FATTY ACID ALKANOLAMIDE REACTION

BY

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A THESIS
SUBMITTED TO THE FACULTY OF
THE DEPARTMENT OF CHEMICAL ENGINEERING
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ABSTRACT

An alkanolamide detergent can be formed by the condensation of coconut fatty acid and diethanolamine. A great many factors can influence this formation. It is shown that varying the molar ratio of amine to acid, results in a change in the fraction of acid converted. It similarly affects the molar quantities of amine converted. Molar conversion of acid or amine is dependent upon the type of atmosphere in the reaction: more acid is converted when a nitrogen sweep is employed than when it is not used. However, proportionately more amine is converted, when a nitrogen sweep is not used. When two mols of water are distilled, there is evidence of a second reaction occurring. This phenomenon is not observed when just one mol of water is removed. The most probable second reaction involves the formation of cyclic ethers.

APPROVAL OF THESIS

FOR

DEPARTMENT OF CHEMICAL ENGINEERING

NEWARK COLLEGE OF ENGINEERING

BY

FACULTY COMMITTEE

APPROVED: _____

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NEWARK, NEW JERSEY

JUNE, 1956

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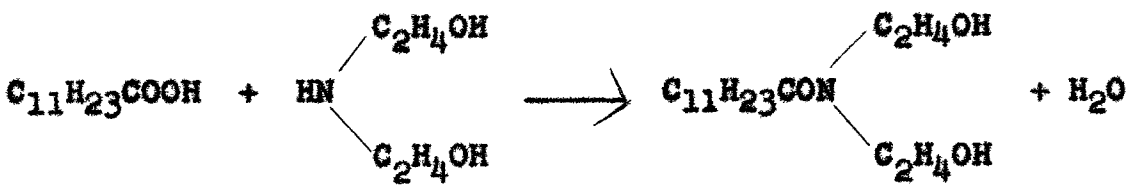
THEORY

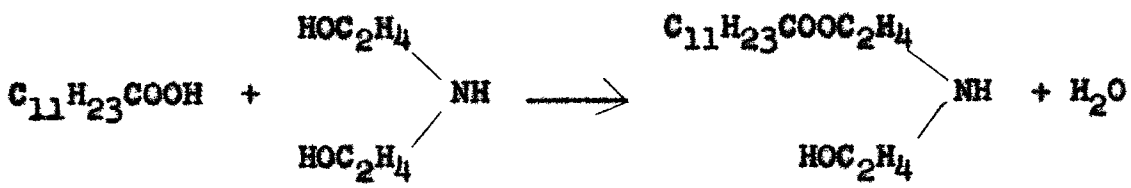
The condensation reaction between fatty acids and secondary alkanolamines are known to produce water soluble detergents. Essentially, the reaction between coconut fatty acid and diethanolamine produces a non-ionic detergent.

Wolf Kritchevsky [1] held that the chemistry of the process was such that the lauric acid (I) and diethanolamine (II) condensed to form an amide (III) called a fatty alkanolamide as follows:

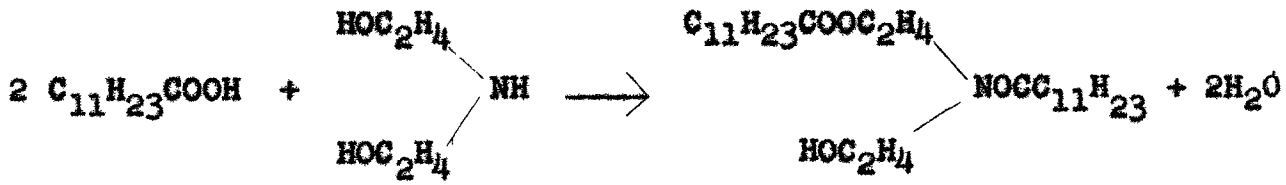


(I) (II) (III)

It was also assumed that although the amide formation predominated, it was also possible to form ester amines (IV) and ester amides (V) [2].



(I) (II) (IV)



(I) (II) (V)

Commercially, alkanolamide detergents are made by the condensation of equimolar ratios of fatty acids with amines by the removal of one mol of water. The resultant alkanolamide are not as soluble as are those in which a one molar excess of amine is used, nor do they act as foam builders and foam stabilizers. Another method of making these detergents, is to react an equimolar amount of acid with amine and distill off one mol of water. After cooling, another mol of amine is added. It is interesting to note that a simple physical addition of the second mol of amine is not in itself enough to produce a detergent with the most desirable characteristics. The batch must again be heated to 150°C without distilling off additional water. This heating cycle apparantly catalyzes the formation of a complex structure which has not been elucidated. Depending upon the type and amount of fatty acid and alkanolamine used, optimum results occur when one mol of water has been removed by distillation. These results involve:

a. an optimum of viscosity

b. an optimum foam building

c. an optimum foam stabilizing

d. minimum color

e. optimum solubility

Essentially the purpose of this study was to investigate the nature of the postulated complex structure; what optimum conditions existed when just one mol of water was removed and what effect several variables would have on the reaction. The factors which were investigated were as follows:

Series I. Variations of the molar ratio of amine to acid, wherein the amine ratio was varied over the range of 1.5 mols to 2.5 mols.

Series II. Effect of an inert atmosphere on the reaction, wherein a nitrogen sweep was employed.

Series III. Variation of time of reaction. Comparison of slowest run with most rapid experiment.

Series IV. Variation of molar ratio of lauric acid to enhance the coconut fatty acid. These experiments were performed over the range of 25% to 100% lauric acid in admixture with double distilled coconut fatty acid.

Series V. Variations of the amount of water removed as a function of complex formation.

With the exception of the Kritchevsky patents, there is no published material on the chemistry of the reaction aside from unsupported works of conjecture. Therefore, the work in this investigation is primarily an original compilation of experimental data and conclusions. The amount of investigation necessary to completely elucidate this subject is beyond the scope of this introductory study. It is recommended that future workers pressure certain plases of this subject, which were touched upon only lightly in the present work.

EXPERIMENTAL

Series I

In this series, seven experiments were performed wherein the molar ratio of amine to acid was varied in increments of 0.2 of a mol over the range of 1.5 to 2.5 mols of diethanolamine to one mol of coconut fatty acid. In each case the equipment used was identical. The only variable was the change in amine ratio. The experimental data of this series is shown in Table 1.

Experiment G-1. Into a 1-liter, 3-neck, round bottom flask equipped with a glass agitator, thermometer, coil condenser, and Barrett distilling receiver were placed 217.0 grams (1.0 mol) of previously melted, double-distilled coconut fatty acid (commercial grade obtained from E. F. Drew Company with a combining weight of 217). To this were added 157.5 grams (1.5 mols) of 95% commercial grade diethanolamine (obtained from Union Carbide and Carbon with a combining weight of 105). The reaction mixture was heated by means of a Bunsen burner. The heating cycle was such that the time lapse from start of heat until the first drop of water formed at 154°C pot temperature was fifty minutes. From the start of the water formation until one mol of water was distilled into the Barrett apparatus, the time lapse was sixty-five minutes. The range of temperature was from 64°C at the start (due to heated coconut fatty acid) to 174°C

pot temperature at the end. The reaction was cooled, the agitator stopped and the contents transferred to a bottle.

Experiment G-2 was conducted in similar apparatus, the only change being that 178.5 grams (1.7 mols) of diethanolamine were reacted with 217.0 grams (1.0 mol) of coconut fatty acid. The time cycle was thirty minutes.

Experiment G-3 was performed in the same manner. However, to 217.0 grams (1.0 mol) of coconut fatty acid were added 199.5 grams (1.9 mols) of diethanolamine. The time cycle was forty minutes.

Experiment G-4 was modified so that 210 grams (2.0 mols) of diethanolamine were reacted with 217.0 grams (1.0 mol) of coconut fatty acid. The time cycle for this experiment was twenty-six minutes.

Experiment G-5 was modified so that 220.5 grams (2.1 mols) of diethanolamine were reacted with 217.0 grams (1.0 mol) of coconut fatty acid. This experiment took thirty minutes to complete its cycle.

Experiment G-6 was performed in similar equipment, the only change being that 241.5 grams (2.3 mols) of diethanolamine were reacted with 217.0 grams (1.0 mol) of coconut fatty acid. The time cycle for this experiment was twenty-five minutes.

Experiment G-7 was prepared in the same manner. The modification here was that 262.5 grams (2.5 mols) of diethanolamine were reacted with 217.0 grams (1.0 mol) of coconut fatty acid. The time cycle was twenty minutes.

Series II

Two experiments were performed in Series II. The equipment used was, with the exception of the nitrogen addition tube, similar to that of Series I. The conditions of the reaction were such that in one experiment a nitrogen sweep was employed throughout the reaction. In the other experiment, nitrogen was used only at the end of the reaction. The experimental data for this series is found in Table 5.

Experiment G-8. In apparatus similar to that of Series I were placed 217.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine. At the start of heating, Linde water-pumped nitrogen was bubbled in beneath the surface of the reaction from a cylinder at such a rate that the entire reaction chamber was swept completely. The nitrogen was permitted to bubble in throughout the reaction cycle and until the mixture was cool enough to be transferred. The final pot temperature rose to 188°C until one mol of water was removed.

Experiment G-9 was conducted in the same manner as was G-8, with the exception that nitrogen was used as a sweep

only after one mol of water was taken off and until the reaction was cool to the hand.

Series III

Experiment G-4 was used as a control in this series to determine what effect a longer reaction time would have on the alkanolamide formation. Experiment G-10 took two hours to complete its cycle. Experiment G-4 took twenty-six minutes. Experimental data on this series can be found in Table 9.

Experiment G-10 was performed in equipment similar to that used in the other series. To the flask were added 217.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine. The heat was applied slowly. Forty-five minutes elapsed before there was evidence of water formation. The time lapse for one mol of water to distill over was seventy-five minutes.

Series IV

The four experiments in this series were all performed in equipment identical with that of the other series. The variable changed in this series was the lauric acid content of the fatty acid. This acid was used to determine what enhanced effect it would have on the final product. The lauric acid ratio to coconut fatty acid was varied in increments of 0.25 mol per experiment over the range of 0 to 100%.

Experiment G-4 was again used as a control in this series as the compound with no added lauric acid. Experimental data for this series is found in Table 13.

Experiment G-11 was conducted in equipment similar to that of the other series. Into the flask were placed 162.8 grams (0.75 mol) of coconut fatty acid and 52.8 grams (0.25 mol) of 96% lauric acid (containing 2% palmitic acid and 2% myristic acid from El Durado Oil Company). To this mixture were added 210.0 grams (2.0 mols) of diethanolamine. Heat was applied and one mol of water was distilled off in fifty minutes.

Experiment G-12 used 103.5 grams (0.50 mol) of coconut fatty acid and 105.5 grams (0.50 mol) of lauric acid. To this were added 210.0 grams (2.0 mols) of diethanolamine. The time cycle for this experiment was thirty minutes.

Experiment G-13 used 54.3 grams (0.25 mol) of coconut fatty acid and 158.2 grams (0.75 mol) of lauric acid, to which were added 210.0 grams (2.0 mols) of diethanolamine. The reaction time for this experiment was thirty minutes.

Experiment G-14 required that 211.0 grams (1.0 mol) of lauric acid be reacted with 210.0 grams (2.0 mols) of diethanolamine. No coconut fatty acid was used at all. The time for this reaction was twenty-five minutes.

Series V

Using the equipment as previously described, the only modification in this series of seven experiments was the variation of the amount of water removed by distillation. Water was removed from each reaction in increments of 0.2 mol per reaction over the range of 0.6 to 2.0 mols. Experimental data for this series is shown on Table 17.

Experiment G-15 was conducted in the equipment as previously described. In this reaction 214.0 grams (1.0 mol) of coconut fatty acid was reacted with 210.0 grams (2.0 mols) of diethanolamine. Heat was applied and 0.6 mol or 10.8 cc of water were removed by distillation in sixty minutes.

Experiment G-16 was essentially the same wherein 214.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine were heated together with the removal of 0.8 mol or 14.4 cc of water. The time cycle for this reaction was twenty minutes.

Experiment G-17 was performed using 214.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine. 1.2 mols or 21.6 cc of water were distilled in thirty-five minutes.

Experiment G-18 used 214.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine.

The reactants were heated and 1.4 mols or 25.2 cc of water were removed by distillation in twenty-five minutes.

Experiment G-19 was prepared by reacting 214.0 grams (1.0 mol) of coconut fatty acid with 210.0 grams (2.0 mols) of diethanolamine. The heating cycle took forty minutes wherein 1.6 mols or 28.8 cc of water were distilled.

Experiment G-20 was conducted whereby 214.0 grams (1.0 mol) of coconut fatty acid were added to 210.0 grams (2.0 mols) of diethanolamine. 1.8 mols or 32.4 cc of water were distilled in thirty minutes.

Experiment G-21 was performed by adding 214.0 grams (1.0 mol) of coconut fatty acid and 210.0 grams (2.0 mols) of diethanolamine together and heating until 2.0 mols or 36 cc of water were removed. This reaction took thirty minutes.

ANALYTICAL

Determination of Free Acidity

(a) Determination of acid number. A sample of the product alkanolamide of Experiment G-1 (used in this case as an example) was accurately weighed into a tared 125 ml Erlenmeyer flask on an analytical balance. The sample was dissolved in 25 ml of 95% neutral ethanol and warmed slightly to effect solution. Four drops of 1% alcoholic phenolphthalain solution was added and the sample titrated with 0.1 N sodium hydroxide until the clear solution showed a pink end point.

The acid number of the sample was calculated by use of the expression:

$$\frac{\text{ml} \times N \times (\text{MgKOH/Meq})}{\text{gms. sample}} = \text{acid number}$$

expressed in the usual terms of mgm of KOH per gram of acid where

ml = ml of sodium hydroxide used

N = normality of sodium hydroxide

(b) Calculation of percent free acid and percent conversion of acid. The acid number of 100% coconut fatty acid was calculated as follows:

One equivalent weight of fatty acid requires 56,100 mgm of KOH for complete neutralization. One gram of the acid

would therefore require

$$\frac{56,100}{217} \text{ or } 258.53 \text{ mgm KOH} = \text{acid number}$$

where 217 is the equivalent weight of coconut fatty acid. Therefore:

$$\frac{\text{acid number found}}{\text{acid number for 100\% Coco FA}} \times 100 = \% \text{ free acidity}$$

From the experimental data (TABLE I) it is shown that in Experiment G-1, 217.0 grams of coconut fatty acid was condensed with 157.5 grams of diethanolamine with the subsequent removal of one mol of water. The final weight of the product was 356.5 grams. By dividing the weight of acid initially present was calculated thus:

$$\frac{\text{weight of acid used}}{\text{final weight of product}} \times 100 = \% \text{ acid initially present}$$

In order to calculate the percent of acid which was converted, the following expression was used:

$$\frac{\% \text{ initial acid} - \% \text{ free acid}}{\% \text{ initial acid}} \times 100 = \% \text{ acid converted}$$

(c) Sample calculation: Experiment G-1. A 2.0322 gram sample from Experiment G-1 required 6.20 ml of a 0.1048 normal sodium hydroxide solution for complete neutralization. The acid number of this sample was therefore:

$$\frac{6.20 \times 0.1048 \times 0.561}{2.0322} = 17.94 \text{ mgm KOH/gm acid}$$

Hence, the percent of free acid in the product was:

$$\frac{17.94}{258.53} \times 100 \text{ or } 6.94\% \text{ free acidity}$$

The percent of acid present initially was:

$$\frac{217.0}{356.5} \times 100 \text{ or } 60.9\% \text{ initial acid}$$

The percent of conversion of acid was calculated thus:

$$\frac{60.9 - 6.94}{60.9} \times 100 \text{ or } 88.7\% \text{ conversion}$$

The analytical data for the determination of the free acidity and percent conversion of all the experiments are shown in TABLES 2, 6, 10, 14, and 18.

Determination of Free Alkalinity

(a) Determination of free alkalinity. A sample of the resulting alkanolamide of Experiment G-1 (used in this case as an example) was accurately weighed into a tared 125 ml Erlenmeyer flask on an analytical balance. The sample was dissolved in 25 ml of 99% neutral isopropanol and warmed slightly to effect solution. A mixed indicator consisting of 3 drops of alcoholic 0.1% methyl orange and 1 drop of 0.4% bromphenolblue was added and the sample titrated with 0.5 N aqueous hydrochloric acid until the blue solution changed to a faint amber end point.

The weight percent of free amine as diethanolamine or free alkalinity was calculated using the expression

$$\frac{\text{ml} \times \text{N} \times \text{Meq DEA}}{\text{gm. sample}} \times 100 = \text{free alkalinity}$$

where

ml = ml of 0.5 N hydrochloric acid used

N = normality of hydrochloric acid

Meq DEA = milliequivalent weight of diethanolamine

(b) Calculation of percent amine converted to amide. From the experimental data (TABLE I) it is seen that in Experiment G-1, 157.5 grams of diethanolamine was reacted with 217.0 grams of coconut fatty acid with the subsequent removal of one mol of water. The final weight of the product was therefore 356.5 grams. By dividing the weight

of amine used initially by the final weight of the product, the percent of amine present initially was calculated thus:

$$\frac{\text{weight of amine used}}{\text{final weight of product}} \times 100 = \% \text{ amine present initially}$$

Hence, to calculate the percent of amine which was converted to amide, the following expression was used:

$$\frac{\% \text{ initial amine} - \% \text{ free amine}}{\% \text{ initial amine}} \times 100 = \% \text{ amine converted}$$

(c) Sample calculation. A 2.1051 gram sample from Experiment G-1 required 6.0 ml of a 0.5336 N hydrochloric acid solution for complete neutralization. The free alkalinity in this product, therefore, was

$$\frac{6.0 \times 0.5336 \times 0.105}{2.0151} \times 100 \text{ or } 15.7\% \text{ free alkalinity}$$

The percent of amine used initially was calculated as

$$\frac{157.5}{356.5} \times 100 \text{ or } 44.2\% \text{ initial amine}$$

The percent of conversion of amine to amide was calculated thus:

$$\frac{44.2 - 15.7}{44.2} \times 100 \text{ or } 64.5\% \text{ converted to amide}$$

The analytical data for the determination of the free alkalinity and the percent conversion of all the experiments are shown in TABLES 3, 7, 11, 15, and 19.

CALCULATION OF MATERIAL BALANCES

Since in each experiment exactly one mol of coconut fatty acid was taken, it is obvious that the percent acid converted corresponds to the fractional mols of acid converted (X).

Similarly, fractional molar conversion of amine is calculated by multiplying mols of amine taken by percent amine converted (Y).

Assuming that equimolar amounts of acid and amine react, the value of (Y) can also be assigned to the mols of acid converted to amide (Y'). The difference then, between (X) and (Y') may be considered as that mol fraction of acid which is presumably converted to ester (Z). Hence the expression

$$(X) - (Y') = Z$$

where:

X = mols of acid converted

Y' = mols of acid converted to amide

Z = mols of acid converted to "ester".

Using the values found in Exp. G-1, the percent of acid converted was found to be 88.7% or 0.887 mols. The percent of amine converted was 64.5% or 0.645 x 1.5 mols of amine charged; or 0.968 mols. The mols of acid converted to amide was therefore 0.968 mols.

Substituting for the values of (X) and (Y') one arrives at (Z).

Hence:

$$(0.887) - (0.968) = -0.081$$

which is the value of mols of acid converted to ester. The negative sign for the value of the ester indicates that since more than one mol of amine reacted per mol of acid, there is no evidence for ester formation. The numerical value of Z can be attributed to analytical uncertainty.

The data for the material balances of all experiments is shown in Tables 4, 8, 12, 16, and 20.

DISCUSSION

Series I.

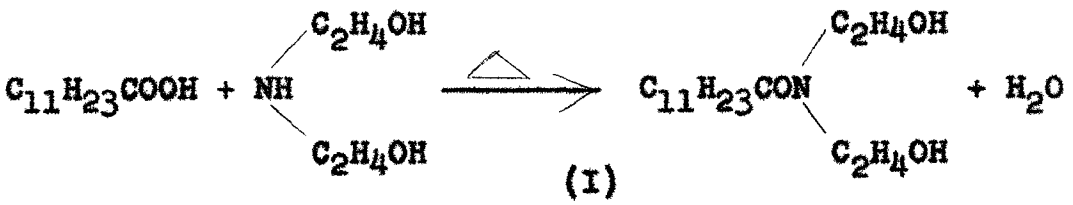
A comparative study of the experiments in this series requires an examination of the variable change in molar ratio of amine to acid. The five factors entering into this examination are:

a. free acidity
b. acid conversion
c. amine conversion
d. molar conversion of amine per mol amine changed.
e. molar conversion of amine per mol acid changed

An examination of Figure 2 Page 53 shows a decrease in free acid as the mol ratio of amine to acid is increased to 1.9. From that point, the acid value rises and finally tapers off as the amine ratio approaches 2.5 mols. Figure 4 page 55 indicates that acid conversion reaches a maximum of 91 per cent at an amine ratio of 1.9 mols and a minimum conversion of 86.5 per cent at 2.05 mols.

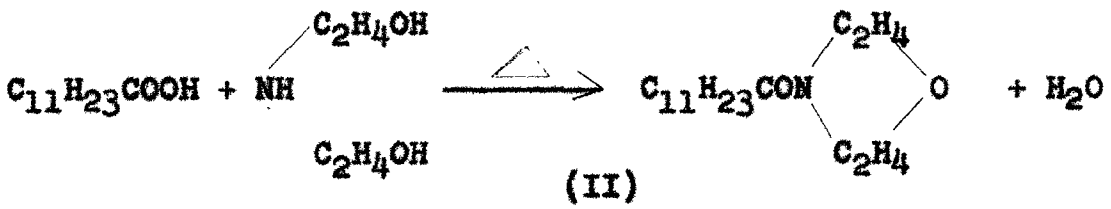
Why there should be more free acid when there is excess amine (at 2.0 mols) may be explained by the fact that there is only 91 per cent acid conversion when there is less free acid. Knowing that only one mol of water

was removed in each case, it can be assumed that 91 per cent of the water came from the primary reaction:



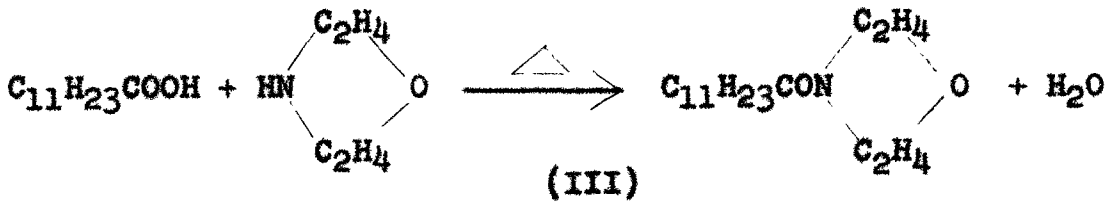
(I)

The other 9 per cent of water could come from the postulated reaction:



(II)

or:



(III)

wherein a cyclic ether is formed by heating with the coconut fatty acid as the catalyst. The cyclic ether III can be called a p-isoxazine ring or more exactly, morpholine. Definate evidence for the possibility of this ether formation is given by Hampton and Pollard [3] wherein they state that morpholine is made by heating two mols of diethanolamine in the presence of a mineral acid until the water is removed. This is strictly analogous to what has been done in these experiments; in that an excess of amine has been

Heated in the presence of an acid and water has been removed. Hence it can be stated with some assurance that morpholine is formed in the alkolanide reaction to some extent. This would explain the fact that if 0.91 mols of the water came from the acid and thereby obtaining a 91 per cent conversion, 9 per cent of the acid would be unconverted and would exhibit free acidity. The postulation would appear to hold true because of the fact that there is less acid conversion as the mol ratio of amine is increased.

It follows, therefore, that where there is maximum acid conversion, the amine conversion should approach theory. It is clearly seen in Figure 5, Page 55that at the point of highest acid conversion, the amine conversion stands at one mol. It is further shown (Figure 6, Page 56) that the mol fraction of amine converted per mol amine charged is converted at a constant rate to the point of maximum acid conversion (1.9 mols amine/mol acid). The rate then drops off as the amine ratio is increased. Figure 7, Page 56 indicates the fact that the fraction of mol amine converted per mol of acid converted is at a maximum within the range of 1.9 to 2.1 mols amine per mol acid.

Series II.

The effect of an inert atmosphere upon the reaction is shown in Figure 8, Page 57 . From this evidence it can be stated that a nitrogen sweep throughout the reaction cycle is beneficial. Graph A shows that the mols of acid

converted approaches theory when nitrogen is used throughout. The partial use of nitrogen more closely approximates theory than does experiment G-4 where no nitrogen was used at all. Similarly, when nitrogen was employed throughout the cycle as in experiment G-8, the mols of amine converted more closely approaches theory than does G-4. Whereas G-9 again lags slightly behind G-8. In Graph C, it is shown that the mol fraction of amine to acid converted is nearer to theory with the full use of nitrogen than when it is used partially or not at all.

Referring again to Figure 2, Page 531t can be seen that the full use of nitrogen in the reaction effects a drop in free acidity of approximately 2.5 per cent; whereas the partial sweep lowers the free acidity approximately 1.5 per cent. It may be assumed, therefore, that if a full nitrogen sweep were employed when the mol ratio of amine to acid was at 1.9 (experiment G-3), the free acidity would have been appreciably lowered. The reason for the lower free acid value may be postulated on the premise that the nitrogen sweep excludes oxygen from the reaction chamber thereby reducing the chance that oxidation of the reactants could inhibit more complete conversion. It was observed that the color of the product in undergoing reaction was lighter when nitrogen was used. However, when the nitrogen was removed, the solution darkened in color. This darkening in color may be attributed to the

oxidation effect.

Moreover, when nitrogen is used throughout the reaction, it may be that it more fully sweeps the water and with it at a lower pot temperature, thereby lessening the possibility of ether formation and increasing the efficiency by which the water is removed from the acid. Hence more complete acid conversion and decreased free acidity.

Series III.

A comparative analysis of this series points out two significant facts. First, from an examination of Figure 11, Page 60, it is obvious that if the reaction is conducted over a two-hour period, as in experiment G-10 the mols of acid converted closely approaches theory. Similarly, the amine conversion also approximates theory, as does the mol fraction of amine to acid converted. Secondly, Figure 2, Page 53, indicates that the free acidity of the two-hour reaction is one per cent lower than that of experiment G-4 whose time cycle was twenty-six minutes.

When the reaction is prolonged over a two-hour period, the average pot temperature is approximately 25°C less than that of the fast reaction. This is shown in Figure 17, Page 64. As previously noted the lower the pot temperature, the less chance there is for ether formation, hence there is increased acid conversion and lower free acidity. This has been shown in Figures 11, Page 60, 17 Page 64, 2 Page 53.

Series IV.

An examination of Figures 12 and 13, Page 61, shows that the addition of lauric acid to the coconut fatty acid content within the range of 25 to 50 per cent lauric, gives a product wherein the amine conversion is almost theoretical. In a similar manner, it can be observed, from Figures 14 and 15, Page 62, that the acid conversion is at a maximum within that range. The mol fraction of amine to acid converted approximates theory within the 25 to 50 per cent range. Because of the fact that the main constituent of coconut fatty acid is lauric acid, it is obvious that increasing the lauric content, would have an enhanced effect on the product of the reaction. Figure 2, Page 63, shows that within the range, the free acidity is minimized and by increasing the lauric acid content, the free acid of the product is also increased proportionally.

Series V.

This series of experiments definately shows that by increasing the molar quantities of water distilled after one mol has been removed, further evidence is obtained for postulating the formation of the ether as a second reaction. An examination of Figure 16, Page 63, indicates that when the factors of acid and amine conversion and final pot temperature are plotted against mols of water distilled, there is a definate linear relationship established between the point where one mol of water has been

removed at that point, the slopes of the lines indicate that the initial reaction is occurring, namely the formation of the alkanolamide. Beyond that point, the slopes of the lines change abruptly. This would be indicative of second reaction or ether formation. Support for this evidence would stem from the fact that it is possible to remove more than one mol of water from the reaction. The second mol is found, therefore, when ring closure is effected to produce the ether amide. This ether is shown on infrared Curve C, Page 68. The instrument used was a Perkin-Elmer model 21, double beam, infrared recording spectrophotometer; using a Neinst Glower emitter. The samples were run as a smear in the sodium chloride cells using air as the reference beam. Curve C shows an adsorption at 1155 cm^{-1}. This is indicative of ether amides [4]. This adsorption at 1155 cm^{-1} is also observed on Curve D, Page 69. It is not noticed in Curves A or B, Pages 66 & 67 These curves refer to samples in which only one mol of water has been removed. Hence there would be a minimum amount of ether formation which cannot be shown on an infrared graph. Curves C and D, Pages 68 & 69 have adsorptions in the range of 1120 to 1125 cm^{-1} which are indicative of free amine. This is naturally due to the excess molar ratio of amine to acid.

Figure 17, Page 64 shows clearly the formation of ether amide when final pot temperature is a function of quantities of water removed.

SUMMARY

The factors which affect the fatty alkanolamide reaction are:

(a) The use of a nitrogen sweep converts more acid and amine to amide thereby lowering the free acid content.

(b) Evidence has been given that there is a possibility of forming either morpholine or cyclic ether amide when more than one mol of water is removed.

(c) Prolonging the reaction time over a two-hour period reduces the pot temperature by approximately 25°C. It has been shown that a lower reaction temperature results in decreased ether formation, and decreased free acidity thereby increasing the percent of amide formation.

(d) A molar ratio of 1.9 to 2.0 mols of amine to 1.0 mols of acid was observed to result in highest amide formation.

(e) Addition of between 25 to 50 per cent lauric acid to the coconut acid leads to higher acid and amine conversion to amide.

Series 1

VARIATION OF MOLAR RATIO OF REACTANTS

Table 1. Experimental Data

Table 2. Determination of Free Acidity

Table 3. Determination of Free Alkalinity

Table 4. Material Balance

Table of Experimental Data

for Series 1

Variation Mol Ratio of Reactants

Run No.	Mols Coco FA	Grams Coco FA	Mols Amine	Grams Amine	Mols Laurie	Grams Lauric	Mols H_2O Distilled	Total Reax Time Min.	Atmosphere of Reax	Final Pot Temp °C
G-1	1.0	217.0	1.5	157.5	-	-	1.0	110	O_2	174
G-2	1.0	217.0	1.7	178.5	-	-	1.0	30	O_2	182
G-3	1.0	217.0	1.9	199.5	-	-	1.0	50	O_2	190
G-4	1.0	217.0	2.0	210.0	-	-	1.0	26	O_2	192
G-5	1.0	217.0	2.1	220.5	-	-	1.0	30	O_2	189
G-6	1.0	217.0	2.3	241.5	-	-	1.0	25	O_2	202
G-7	1.0	217.0	2.5	262.5	-	-	1.0	20	O_2	202

Table 1

Table of Analytical Data

for Series 1

Variation Mol Ratio of Reactants

Determination of Free Acidity

Run No.	% Acid Based on Wt. of Prod.	Wt. of Sample Taken	Titration cc	Normality of NaOH	Acid No. of 100% Coco FA	Acid No. Found	% Free Acidity in Product	% Acid Charged Converted
G-1	60.9	2.0322	6.20	0.1045	258.53	17.94	6.94	88.7
G-2	57.5	2.0165	5.70	0.1045	258.53	16.62	6.43	88.9
G-3	54.5	2.0320	4.45	0.1045	258.53	12.88	4.98	90.8
G-4	53.1	2.0048	6.14	0.1045	258.53	18.01	6.96	86.9
G-5	51.7	2.0289	6.20	0.1045	258.53	17.97	6.95	86.7
G-6	49.3	2.0427	5.05	0.1045	258.53	13.82	5.34	89.2
G-7	47.0	2.0462	4.65	0.1045	258.53	13.36	5.17	89.0

Table 2

Table of Analytical Data

for Series 1

Variation Mol Ratio of Reactants

Determination of Free Alkalinity

Run No.	% Amine on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of HCl	Meq of Acid Used	Grams of free Amine	Wt. of Free Amine (DEA)	% Amine Converted to Amide
G-1	44.2	2.1051	6.00	0.5336	3.2018	0.337	15.7	64.5
G-2	47.3	2.0784	7.55	0.5336	4.0287	0.423	20.4	56.9
G-3	50.1	2.0192	8.55	0.5336	4.5623	0.479	23.7	52.7
G-4	51.3	2.0262	8.20	0.5336	4.3760	0.460	22.7	55.8
G-5	52.6	2.1136	10.90	0.5336	5.8160	0.611	28.9	45.1
G-6	54.8	2.0284	11.40	0.5336	6.0830	0.639	31.5	42.5
G-7	56.9	2.0059	12.65	0.5336	7.500	0.788	39.3	30.9

Table 3

Table of Analytical Data

for Series 1

Variation of Mol Ratio of Reactants

Material Balance

Run No.	Gram Wt. of Reactants	Gram Wt. of Final Prod.	Gram Wt. of H_2O Dist.	Mols Acid Converted	Mols Amine Converted	Mols Acid Conv. to Amide	Mols Acid Conv. to Ester
G-1	374.5	356.5	18.0	0.887	0.968	0.968	-0.081
G-2	395.5	377.5	18.0	0.889	0.967	0.967	-0.078
G-3	416.5	398.5	18.0	0.908	1.001	1.001	-0.093
G-4	427.0	409.0	18.0	0.869	1.116	1.116	-0.247
G-5	437.5	419.5	18.0	0.867	0.947	0.947	-0.080
G-6	458.5	440.5	18.0	0.892	0.9775	0.9775	-0.086
G-7	479.5	461.5	18.0	0.890	0.773	0.773	+0.117

Table 4

Series 2

EFFECT OF INERT ATMOSPHERE ON REACTION

Table 5. Experimental Data

Table 6. Determination of Free Acidity

Table 7. Determination of Free Alkalinity

Table 8. Material Balance

Table of Experimental Data

For Series 2

Effect of Inert Atmosphere

Run No.	Mols Coco FA	Grams Coco FA	Mols Amine	Grams Amine	Mols Lauric	Grams Lauric	Mols H_2O Distilled	Total Reax Time Min.	Atmosphere of Reax	Final Pot Temp. °C
G-4	1.0	217.0	2.0	210.0	-	-	1.0	26	O_2	192
G-8	1.0	217.0	2.0	210.0	-	-	1.0	40	N_2	188
G-9	1.0	217.0	2.0	210.0	-	-	1.0	35	N_2 (partial)	188

Table 5

Table of Analytical Data

For Series 2 Effect of Inert Atmosphere

Determination of Free Acidity

Run No.	% Acid Based on wt. of Product	wt. of Sample taken	Titration cc	Normality of NaOH	Acid No. of 100% Coco FA	Acid No. found	% Free Acidity in Product	% Acid Charged Converted
G-4	53.1	2.0048	6.14	0.1045	258.53	18.01	6.96	86.9
G-8	53.1	2.0529	4.01	0.1045	258.53	11.48	4.44	91.6
G-9	53.1	2.0592	5.10	0.1045	258.53	14.56	5.63	89.4

Table 6

Table of Analytical Data

For Series 2 Effect of Inert Atmosphere

Determination of Free Alkalinity

Run No.	% Amine on wt. of Product	wt. of sample taken	Titration cc	Normality of HCl	Meg. of Acid Used	Grams of Free Amine	wt. % free Amine (DEA)	% Amine Converted to Amide
G-4	51.3	2.0262	8.20	0.5336	4.3760	0.460	22.7	55.8
G-8	51.3	2.0421	9.60	0.5336	5.123	0.5379	26.3	48.7
G-9	51.3	2.0024	9.50	0.5336	5.069	0.5320	26.6	48.1

Table 7

Table For Analytical Data

For Series 2 Effect of Inert Atmosphere

Material Balances

Run No.	Gram wt. of Reactants	Gram wt. of Final Prod.	Gram wt. of H_2O Dist.	Mols Acid Converted	Mols Amine Converted	Mols Acid Converted To Amide	Mols Acid Converted To Ester
G-4	427.0	409.0	18.0	0.869	1.116	1.116	-0.247
G-8	427.0	409.0	18.0	0.916	0.975	0.975	-0.059
G-9	427.0	409.0	18.0	0.894	0.962	0.962	-0.068

Table 8

Series 3

VARIATION OF TIME OF REACTION

Table 9. Experimental Data

Table 10. Determination of Free Acidity

Table 11. Determination of Free Alkalinity

Table 12. Material Balance

Table of Experimental Data

for Series 3

Variation of Time of Reaction

Run No.	Mols Coco FA	Grams Coco FA	Mols Amine	Grams Amine	Mols Lauric	Grams Lauric	Mols H_2O Distilled	Total Reax Time Min.	Atmosphere of Reax	Final Pot Temp °C
G-4	1.0	217.0	2.0	210.0	-	-	1.0	26	O_2	192
G-10	1.0	217.0	2.0	210.0	-	-	1.0	120	O_2	170

Table 9

Table of Analytical Data

for Series 3

Variation of Time

Determination of Free Acidity

Run No.	% Acid Based on Wt. of Prod.	Wt. of Sample Taken	Titration cc	Normality of NaOH	Acid No. of 100% Coco FA	Acid No. Found	% Free Acidity in Product	% Acid Charged Converted
G-4	53.1	2.0048	6.14	0.1045	258.53	18.01	6.96	86.9
G-10	53.1	2.0549	5.50	0.1045	258.53	15.74	6.09	88.7

Table 10

Table of Analytical Data

for Series 3

Variation of Time

Determination of Free Alkalinity

Run No.	% Amine on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of HCl	Meq of Acid Used	Grams of free Amine	Wt. of Free Amine (DEA)	% Amine Converted to Amide
G-4	51.3	2.0262	8.20	0.5336	4.3760	0.460	22.7	55.8
G-10	51.3	2.0394	9.25	0.5336	4.936	0.518	25.4	50.5

Table 11

Table of Analytical Data

for Series 3

Variation of Time

Material Balance

Run No.	Gram Wt. of Reactants	Gram Wt. of Final Prod.	Gram Wt. of H_2O Dist.	Mols Acid Converted	Mols Amine Converted	Mols Acid Conv. to Amide	Mols Acid Conv. to Ester
G-4	427.0	409.0	18.0	0.869	1.116	1.116	-0.247
G-10	427.0	409.0	18.0	0.887	1.010	1.010	-0.123

Table 12

Series 4

VARIATION OF MOLAR RATIO OF LAURIC ACID

Table 13. Experimental Data

Table 14. Determination of Free Acidity

Table 15. Determination of Free Alkalinity

Table 16. Material Balance

Table of Experimental Data

For Series 4

Variation of Molar Ratio of Lauric Acid

Run No.	Mols Coco FA	Grams Coco FA	Mols Amine	Grams Amine	Mols Lauric	Grams Lauric	Mols H_2O Distilled	Total Reax Time Min.	Atmosphere of Reax	Final Pot Temp °C
G-4	1.00	217.0	2.0	210.0	0.0	0.0	1.0	26	O_2	192
G-11	0.75	162.8	2.0	210.0	0.25	52.8	1.0	50	O_2	182
G-12	0.50	103.5	2.0	210.0	0.50	105.5	1.0	30	O_2	189
G-13	0.25	54.3	2.0	210.0	0.75	158.2	1.0	25	O_2	185
G-14	0.00	0.0	2.0	210.0	1.00	211.0	1.0	25	O_2	191

Table 13

Table of Analytical Data

For Series 4 Variation of Molar Ratio of Lauric Acid

Determination of Free Acidity

Run No.	% Acid Based on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of NaOH	Acid No. of 100% Coco FA	Acid No. Found	% Free Acidity in Product	% Acid Charged Converted
G-4	53.1	2.0048	6.14	0.1045	258.53	18.01	6.96	86.9
G-11	52.9	2.0260	5.49	0.1045	258.53	15.93	6.16	88.4
G-12	52.1	2.0567	6.11	0.1045	258.53	17.47	6.76	87.0
G-13	52.5	2.0230	6.71	0.1045	258.53	19.50	7.54	85.6
G-14	52.4	2.0494	6.70	0.1045	258.53	19.22	7.44	85.8

Table 14

Table of Analytical Data

For Series 4 Variation of Molar Ratio of Lauric Acid

Determination of Free Alkalinity

Run No.	% Amine on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of HCl	Meq. of Acid Used	Grams of Free Amine	Wt. % Free Amine (DEA)	% Amine Converted to Amide
G-4	51.3	2.0262	8.20	0.5336	4.3760	0.460	22.7	55.8
G-11	51.3	2.0366	9.20	0.5336	4.909	0.515	25.3	50.7
G-12	52.4	2.0136	9.35	0.5336	4.989	0.524	26.0	50.4
G-13	52.4	2.0203	9.20	0.5336	4.909	0.515	25.5	51.3
G-14	52.4	2.0185	9.12	0.5336	4.866	0.511	25.3	51.7

Table 15

Table for Analytical Data

For Series 4 Variation of Molar Ratio of Lauric Acid

Material Balance

Run No.	Gram Wt. of Reactants	Gram Wt. of Final Prod.	Gram Wt. of H_2O Dist.	Mols Acid Converted	Mols Amine Converted	Mols Acid Converted to Amide	Mols Acid Converted to Ester
G-4	427.0	409.0	18.0	0.869	1.116	1.116	-0.247
G-11	425.6	407.6	18.0	0.884	1.009	1.009	-0.125
G-12	419.0	401.0	18.0	0.870	0.972	0.972	-0.102
G-13	422.5	404.5	18.0	0.856	1.005	1.005	-0.149
G-14	421.0	403.0	18.0	0.858	1.008	1.008	-0.150

Table 16

Series 5

VARIATION OF AMOUNT OF WATER REMOVED

Table 17. Experimental Data

Table 18. Determination of Free Acidity

Table 19. Determination of Free Alkalinity

Table 20. Material Balance

Table of Experimental Data

For Series 5

Variation of Amount of Water Removed

Run No.	Mols Coco FA	Grams Coco FA	Mols Amine	Grams Amine	Mols Lauric	Grams Lauric	Mols H_2O Distilled	Total Reax Time Min.	Atmosphere of Reax	Final Pot Temp °C
G-15	1.0	217.0	2.0	210.0	-	-	0.6	60	O_2	186
G-16	1.0	217.0	2.0	210.0	-	-	0.8	20	O_2	190
G-17	1.0	217.0	2.0	210.0	-	-	1.2	35	O_2	192
G-18	1.0	217.0	2.0	210.0	-	-	1.4	25	O_2	210
G-19	1.0	214.0	2.0	210.0	-	-	1.6	40	O_2	231
G-20	1.0	214.0	2.0	210.0	-	-	1.8	30	O_2	233
G-21	1.0	214.0	2.0	210.0	-	-	2.0	30	O_2	246

Table 17

Table of Analytical Data

For Series 5 Variation of Amount of Water Removed

Determination of Free Acidity

Run No.	% Acid Based on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of NaOH	Acid No. of 100% Coco FA	Acid No. Found	% Free Acidity in Product	% Acid Charged Converted
G-15	52.1	2.0043	11.75	0.1045	258.53	34.47	13.30	74.5
G-16	52.6	2.0254	8.45	0.1045	258.53	24.53	9.49	81.1
G-17	53.5	2.0871	3.50	0.1045	258.53	9.86	3.81	92.9
G-18	54.0	2.0197	2.65	0.1045	258.53	7.71	2.98	94.5
G-19	54.1	2.0273	1.50	0.1045	258.53	4.35	1.68	96.9
G-20	54.6	2.0000	2.22	0.1045	258.53	6.53	2.50	95.4
G-21	55.2	2.0157	2.38	0.1045	258.53	6.94	2.68	95.1

Table 18

Table of Analytical Data

For Series 5 Variation of Amount of Water Removed

Determination of Free Alkalinity

Run No.	% Amine on Wt. of Product	Wt. of Sample Taken	Titration cc	Normality of HCl	Meq. of Acid Used	Grams of Free Amine	Wt. % Free Amine (DEA)	% Amine Converted to Amide
G-15	50.5	2.0650	11.42	0.5336	6.094	0.639	31.0	38.6
G-16	50.9	2.0030	9.91	0.5336	5.288	0.555	27.7	45.6
G-17	51.8	2.0134	8.89	0.5336	4.744	0.498	24.7	52.3
G-18	52.3	2.0222	8.60	0.5336	4.589	0.482	23.8	54.5
G-19	53.1	2.0612	8.20	0.5336	4.376	0.4595	22.3	58.0
G-20	53.6	2.0193	8.31	0.5336	4.434	0.4660	23.1	56.9

Table 19

Table of Analytical Data

For Series 5 Variation of Amount of Water Removed

Material Balance

Run No.	Gram Wt. of Reactants	Gram Wt. of Final Prod.	Gram Wt. of H_2O Dist.	Mols Acid Converted	Mols Amine Converted	Mols Acid Converted to Amide	Mols Acid Converted to Ester
G-15	427.0	416.2	10.8	0.745	0.772	0.772	-0.027
G-16	427.0	412.6	14.4	0.811	0.912	0.912	-0.101
G-17	427.0	405.4	21.6	0.929	1.046	1.046	-0.117
G-18	427.0	401.8	25.2	0.945	1.090	1.090	-0.145
G-19	424.0	395.2	28.8	0.969	1.160	1.160	-0.191
G-20	424.0	391.6	32.4	0.954	1.138	1.138	-0.184
G-21	424.0	388.0	36.0	0.951	1.198	1.198	-0.247

Table 20

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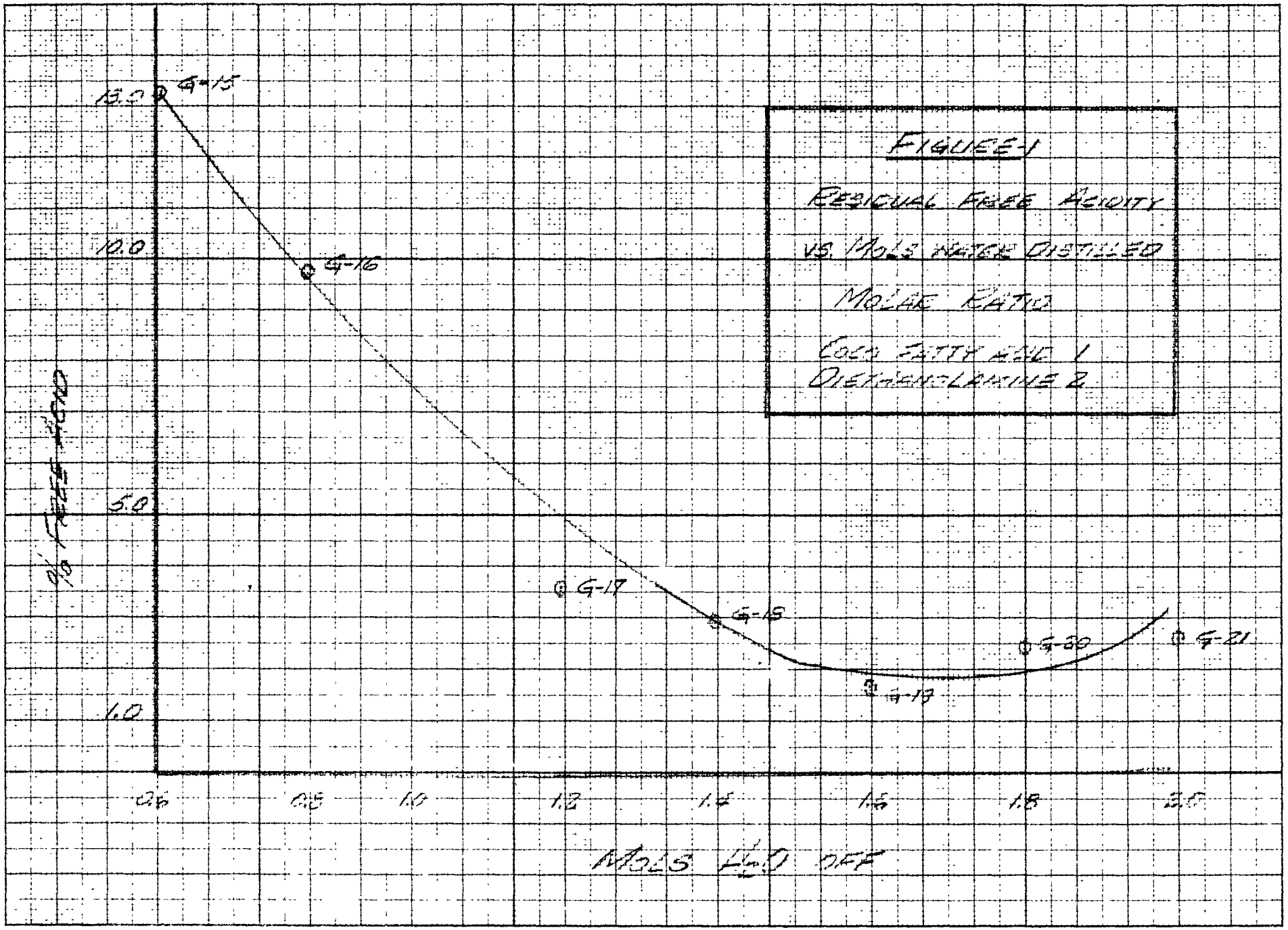

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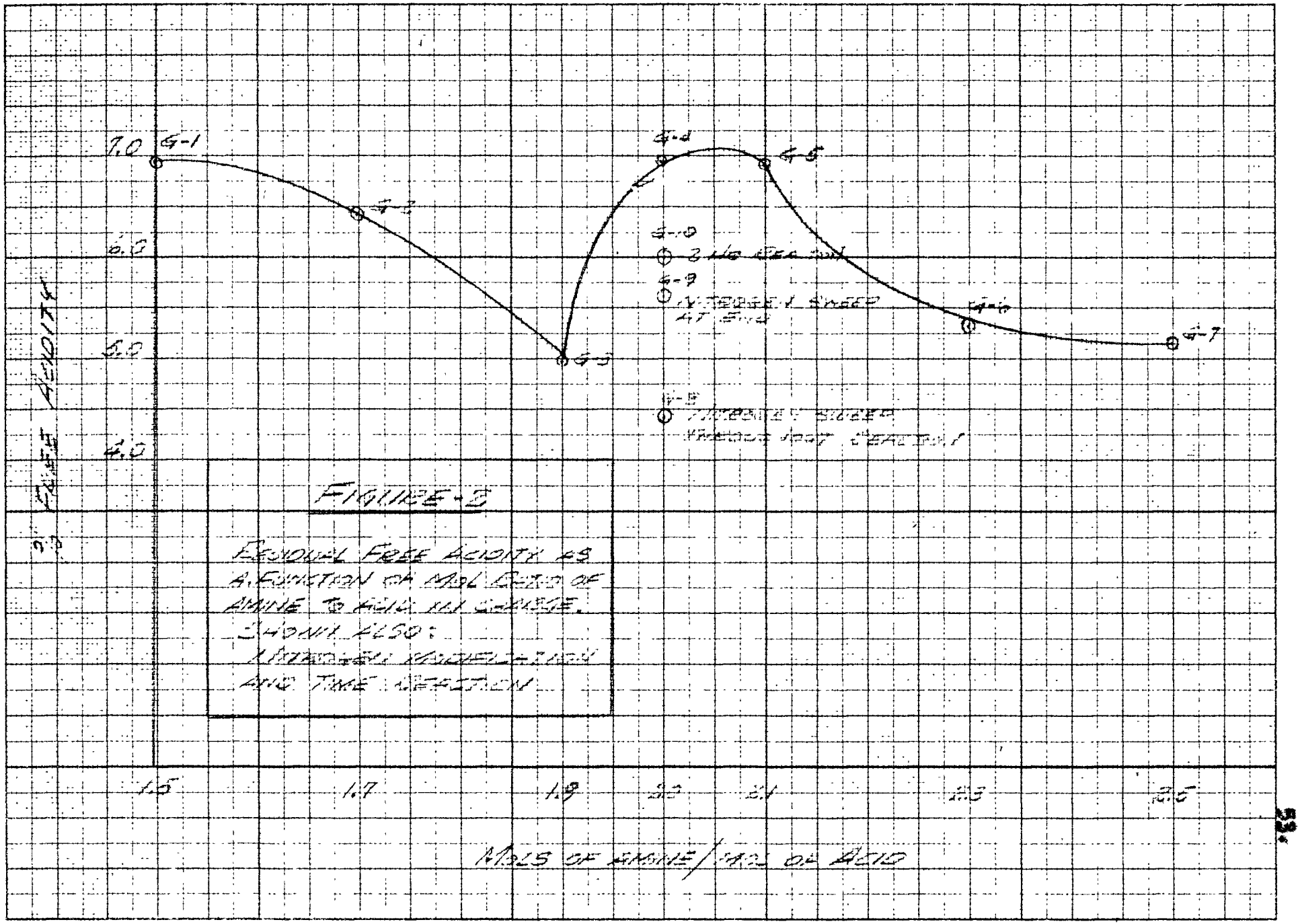
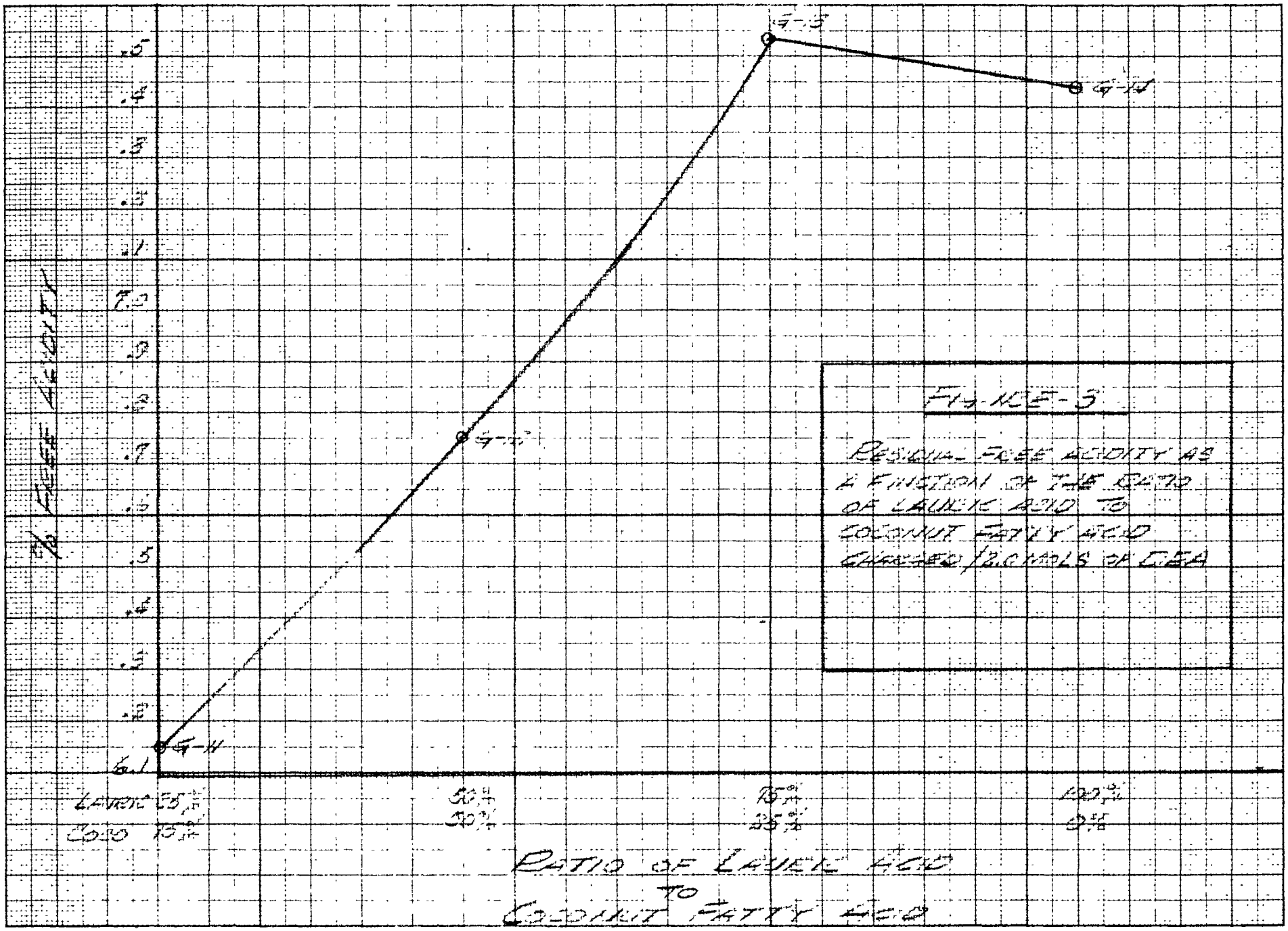


FIGURE-2

RESIDUAL FREE ACIDITY AS A FUNCTION OF MOL RATIO OF AMINE TO ACID IN CHARGE.
SHOWN ALSO:
NITROGEN MODIFICATION AND TIME REACTION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FLOW

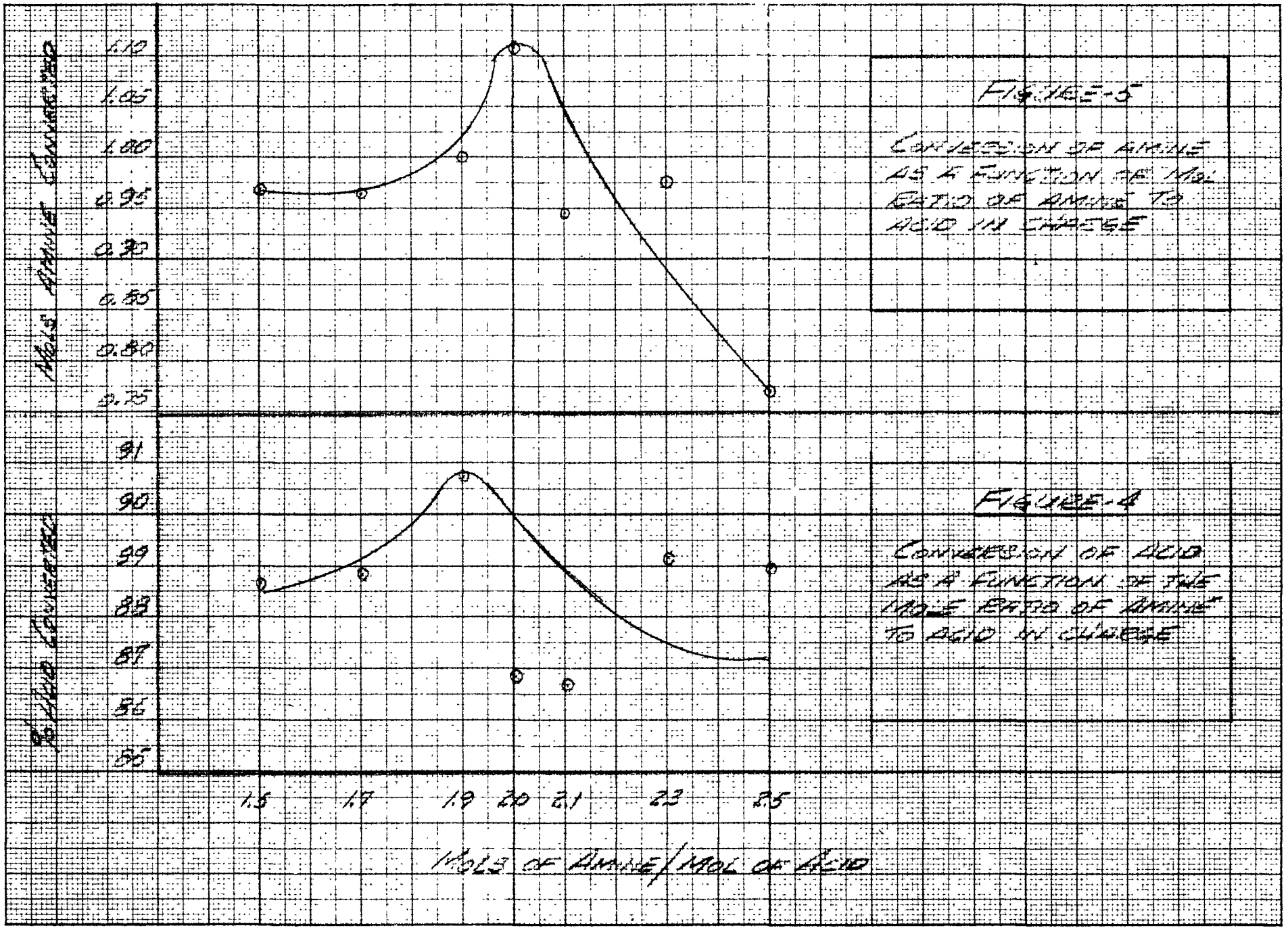
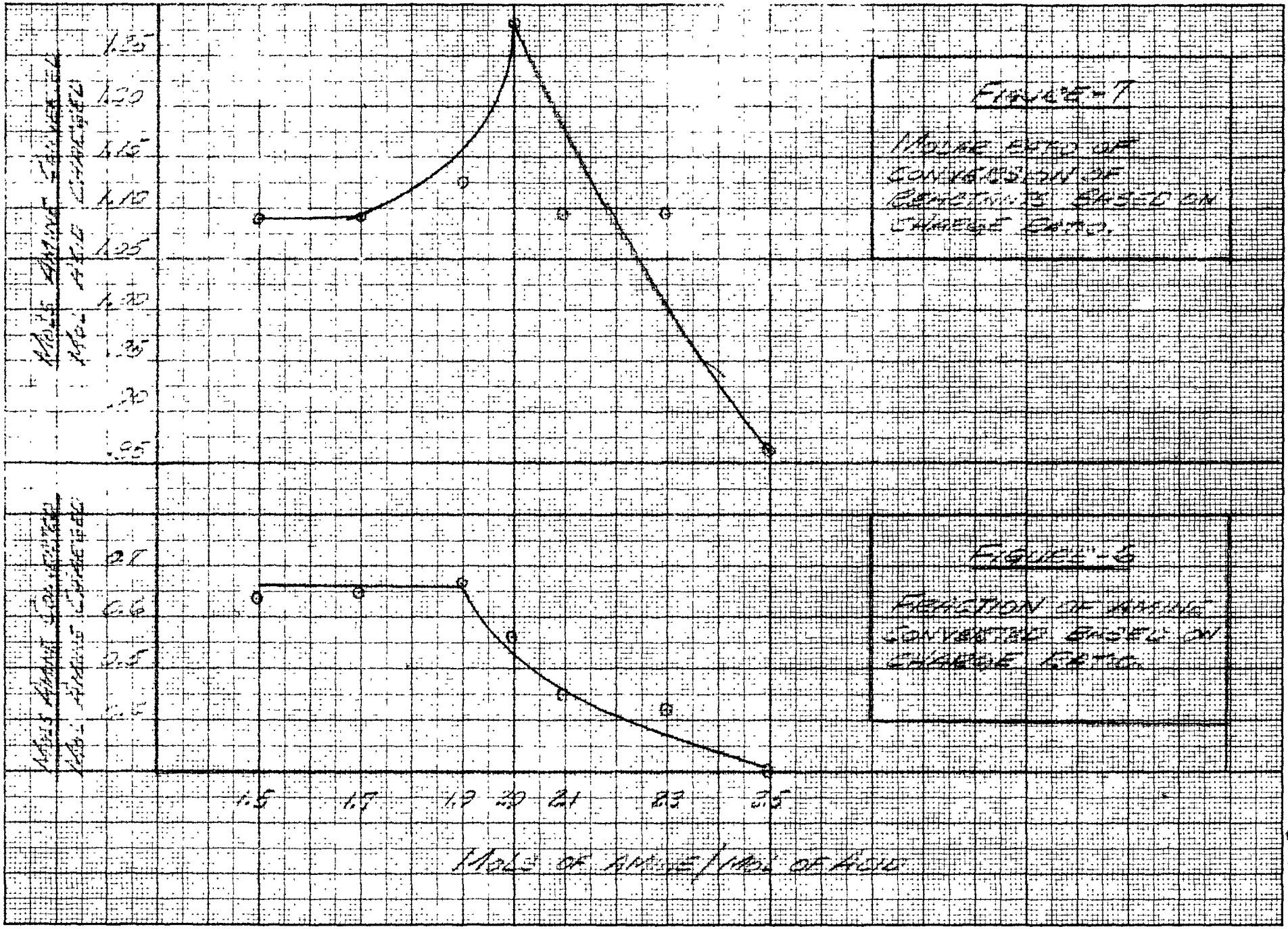


FIGURE-3

CONVERSION OF AMINE AS A FUNCTION OF MOL. RATIO OF AMINE TO ACID IN CHARGE

FIGURE-4

CONVERSION OF ACID AS A FUNCTION OF THE MOLE RATIO OF AMINE TO ACID IN CHARGE



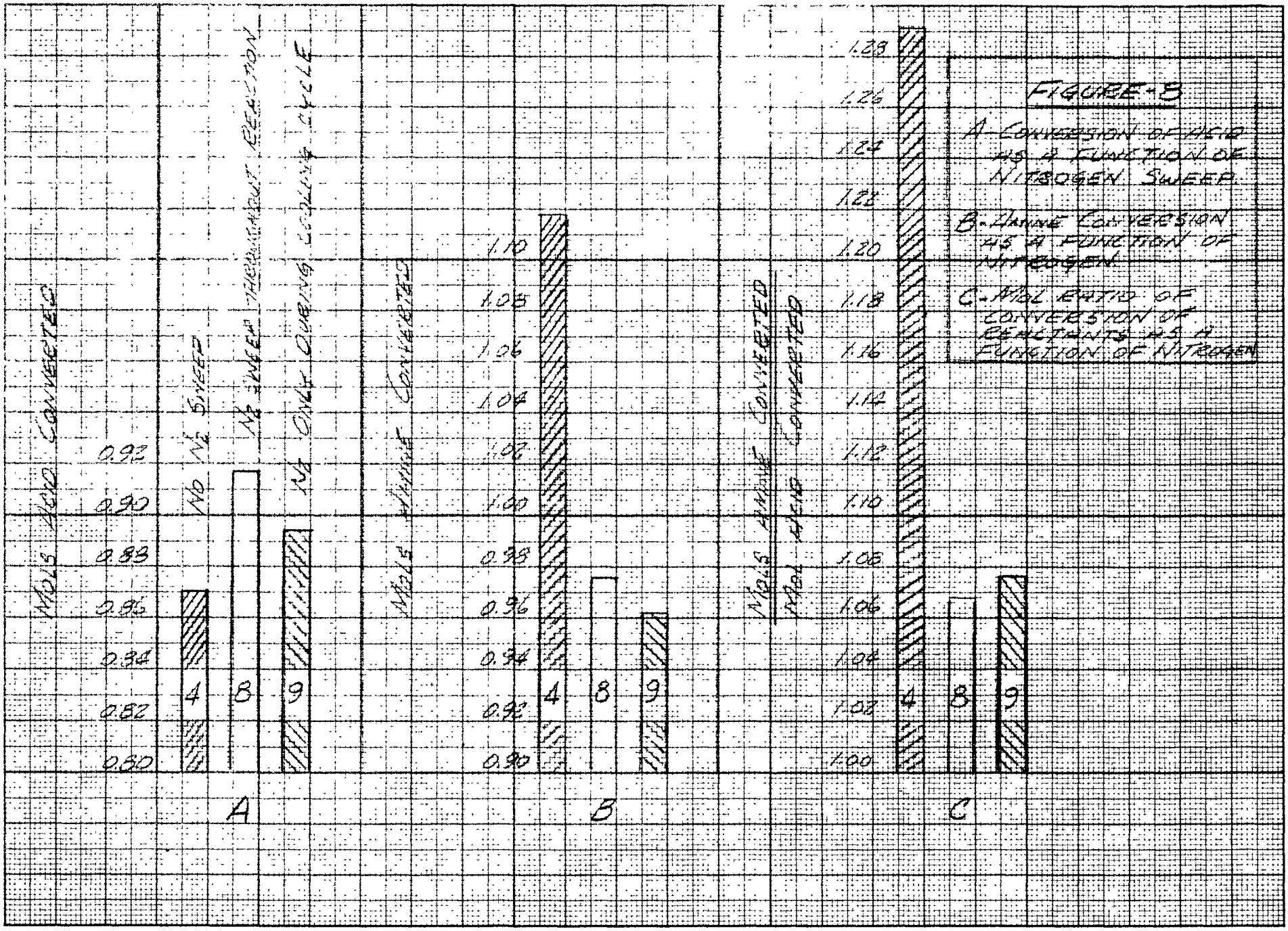


FIGURE-5

A-CONVERSION OF AMINE AS A FUNCTION OF NITROGEN SWEEP.

B-AMINE CONVERSION AS A FUNCTION OF NITROGEN.

C-MOL EQUIV OF CONVERSION OF REACTANTS AS A FUNCTION OF NITROGEN

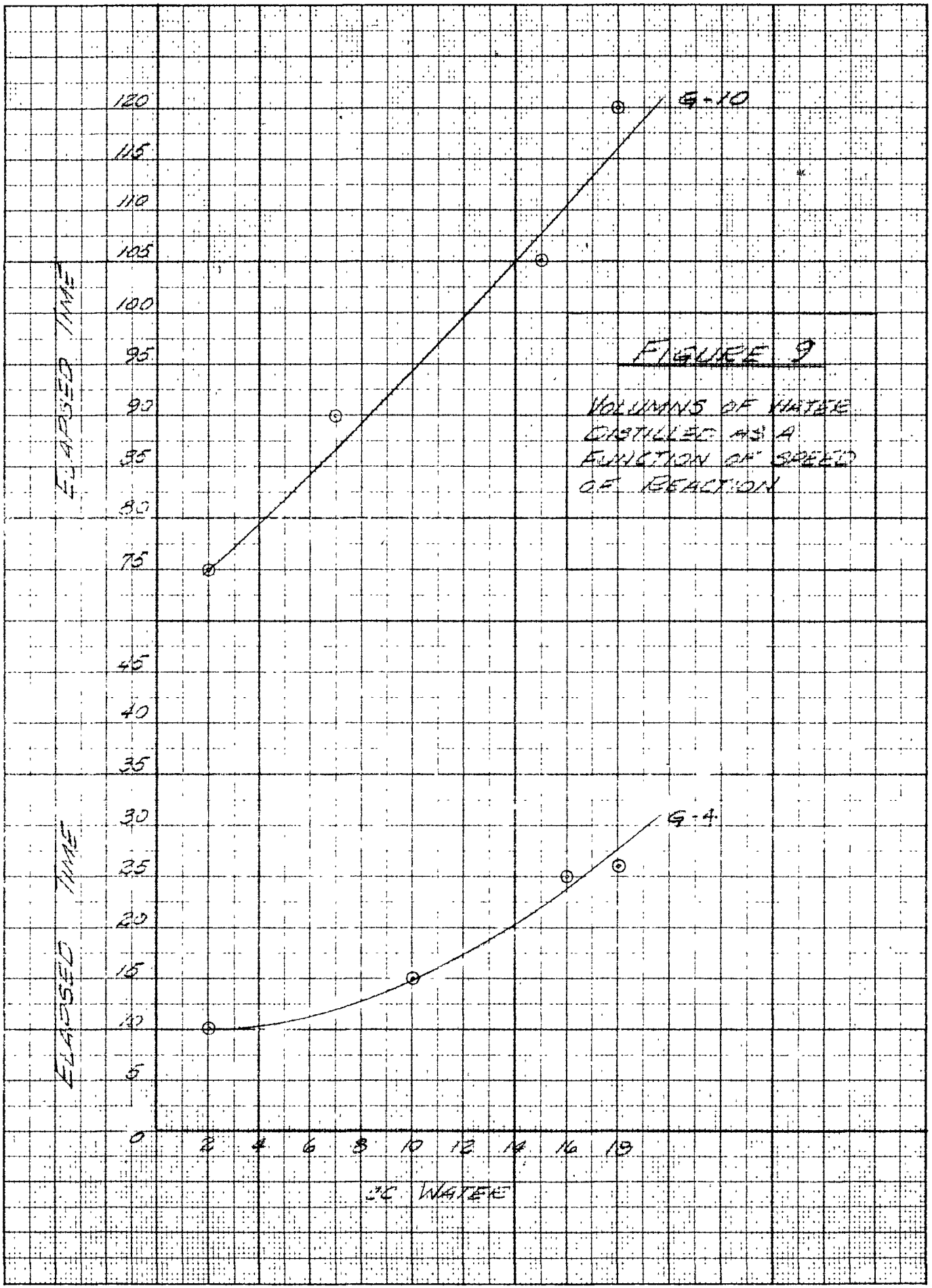


FIGURE 9

VOLUMES OF WATER CAPILLED AS A FUNCTION OF SPEED OF REACTION

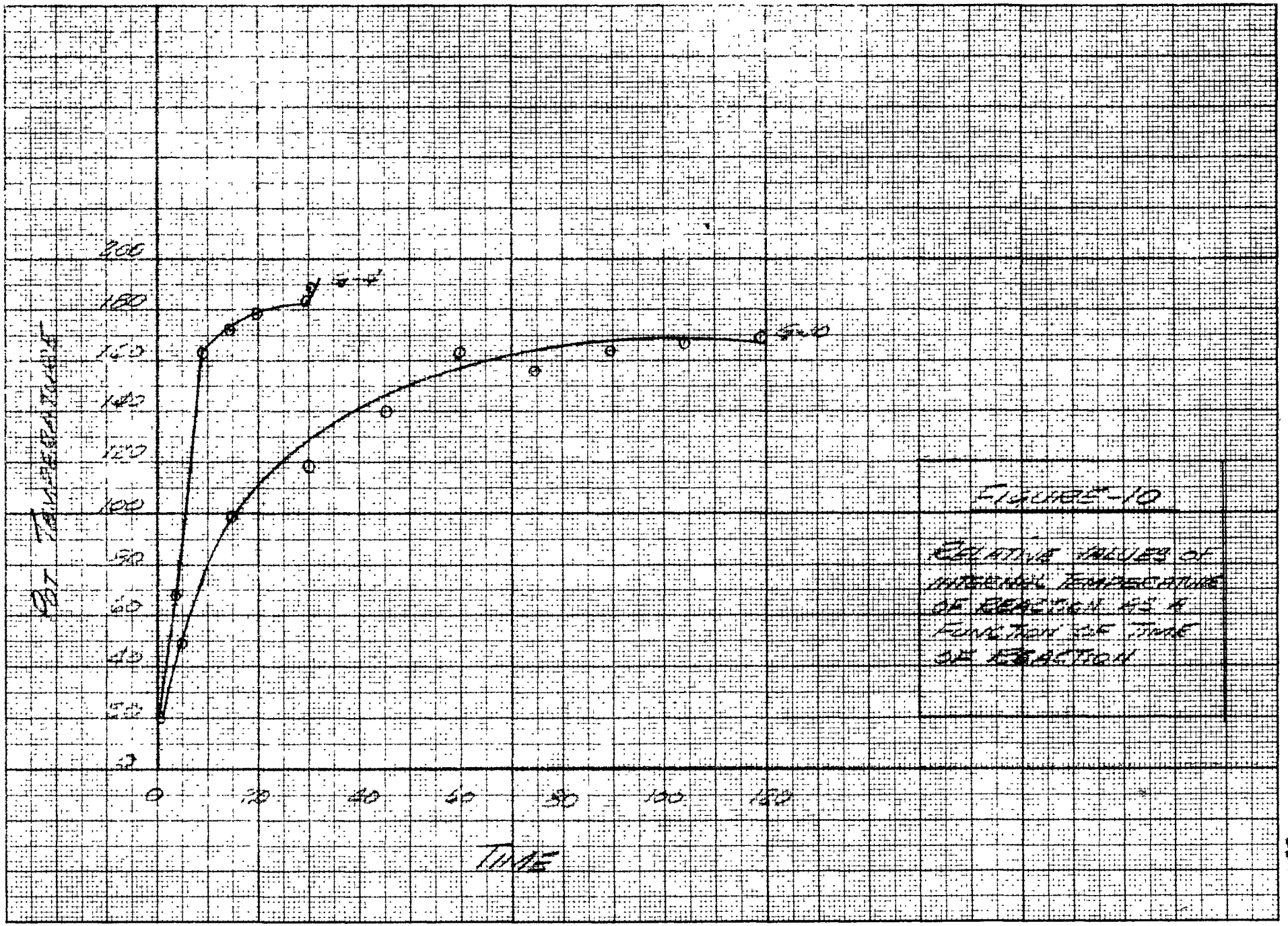
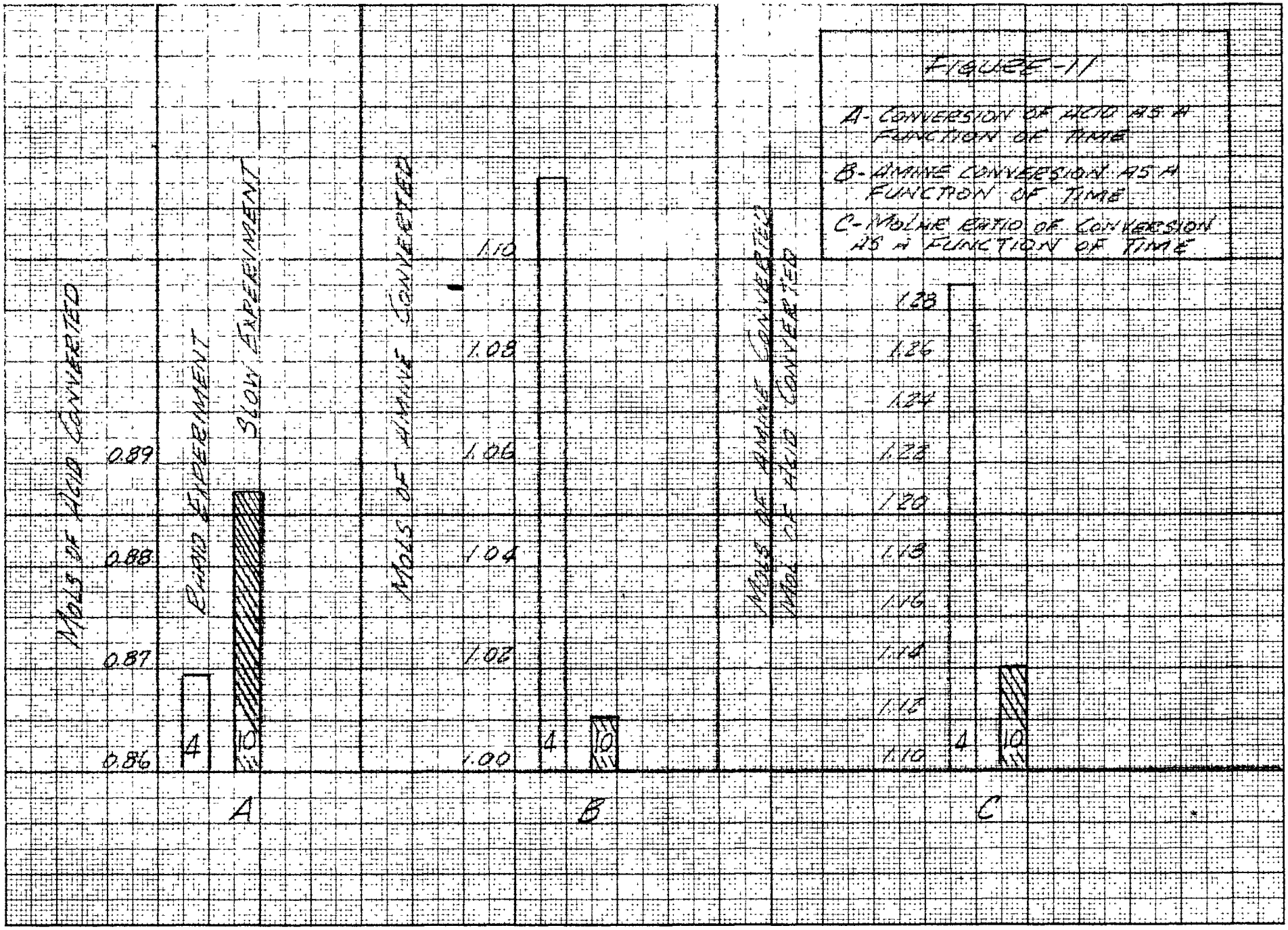
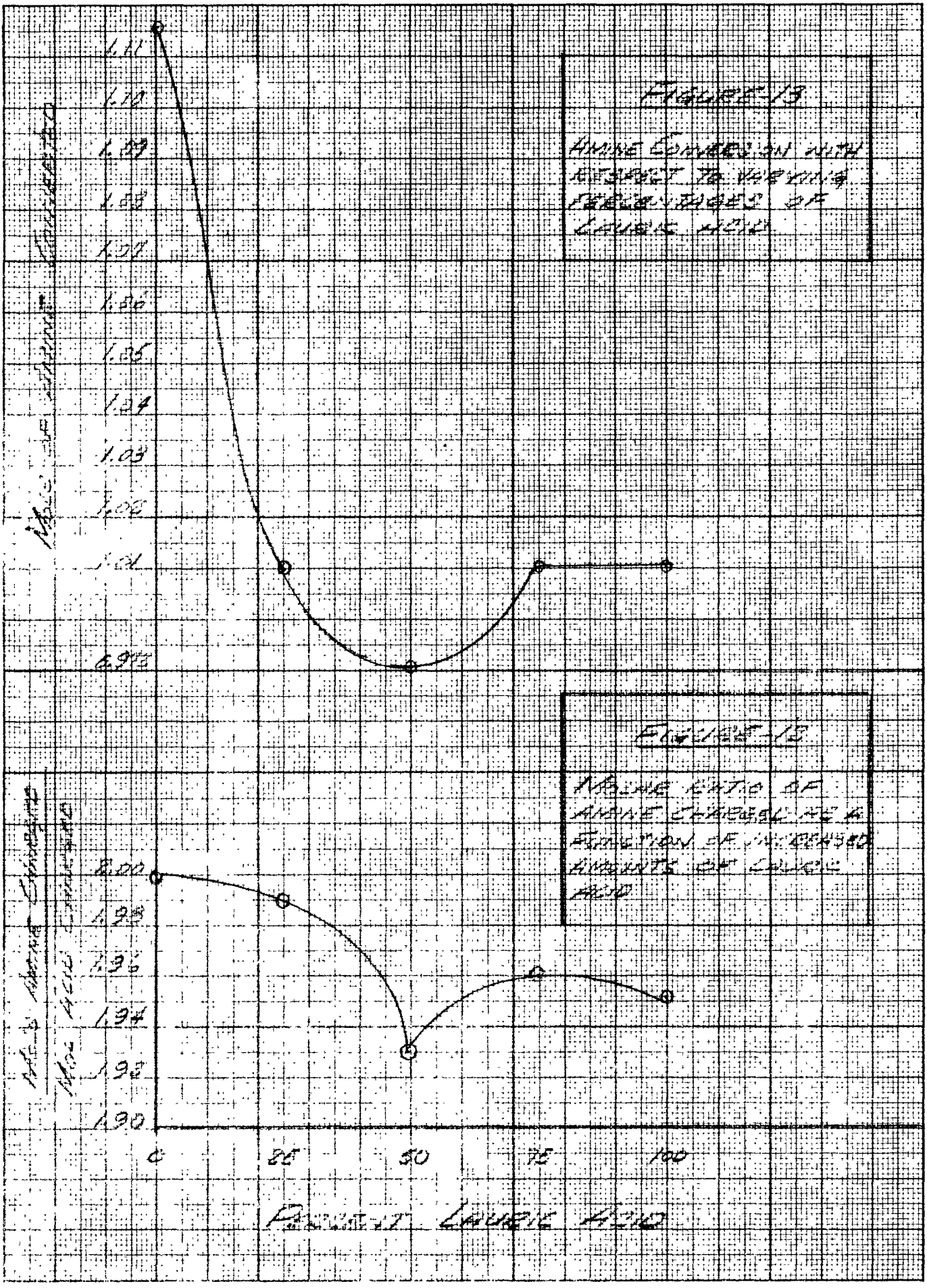


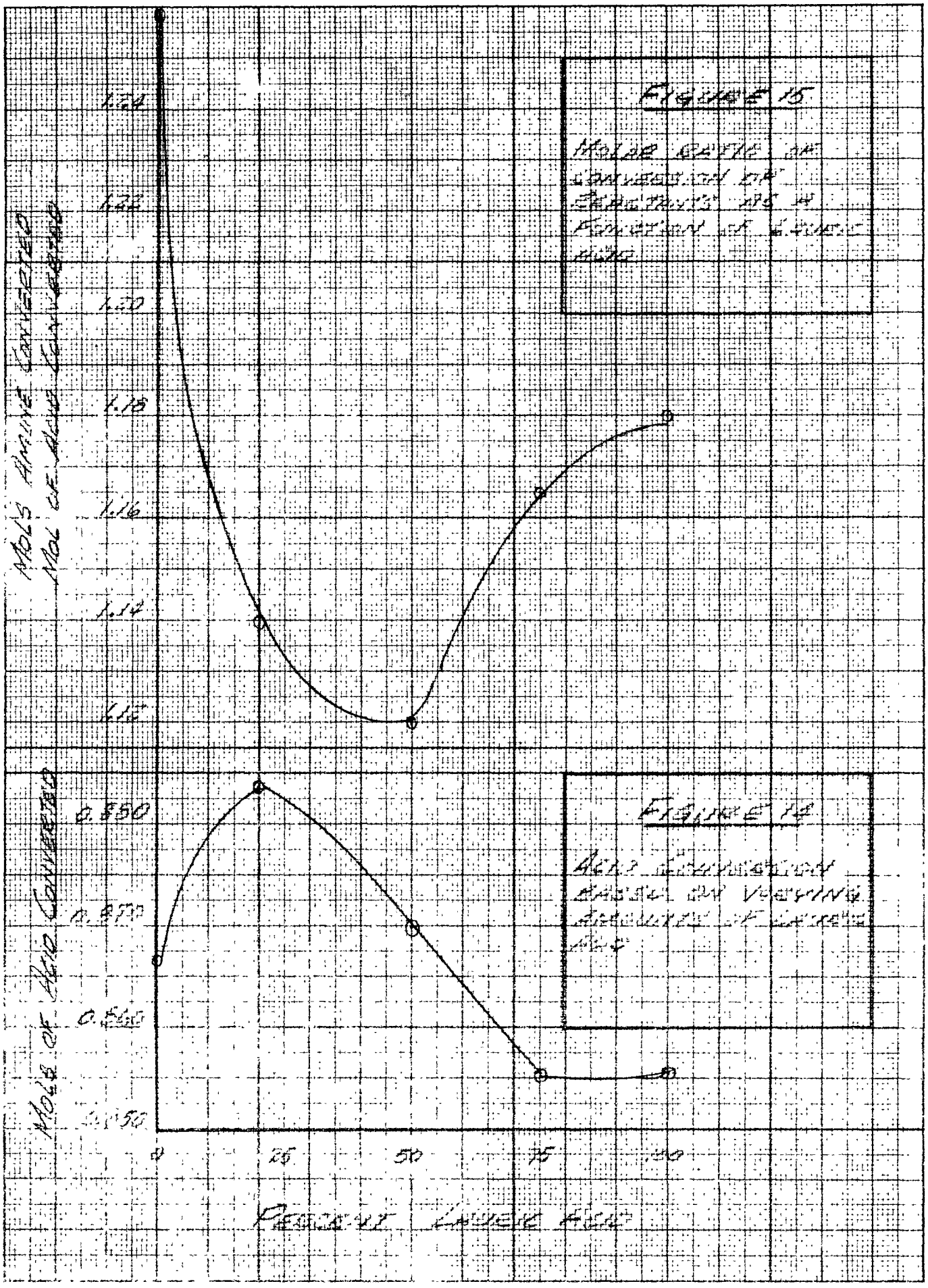
FIGURE-10

EXPERIMENTAL VALUES OF INTERNAL TEMPERATURE OF ELECTRICAL WIRE AS A FUNCTION OF TIME OF HEATING

MILLIMETER PRINTED IN U S A




1.11
1.10
1.09
1.08
1.07
1.06
1.05
1.04
1.03
1.02
1.01
0.99
2.00
1.98
1.97
1.96
1.95
1.94
1.93
1.90
0
25
50
75
100


Moles Amine Converted
Mol of Amine Converted
1.24
1.22
1.20
1.18
1.16
1.14
1.12
Moles of Acid Converted
0.550
0.500
0.450
0.400
0
25
50
75
100

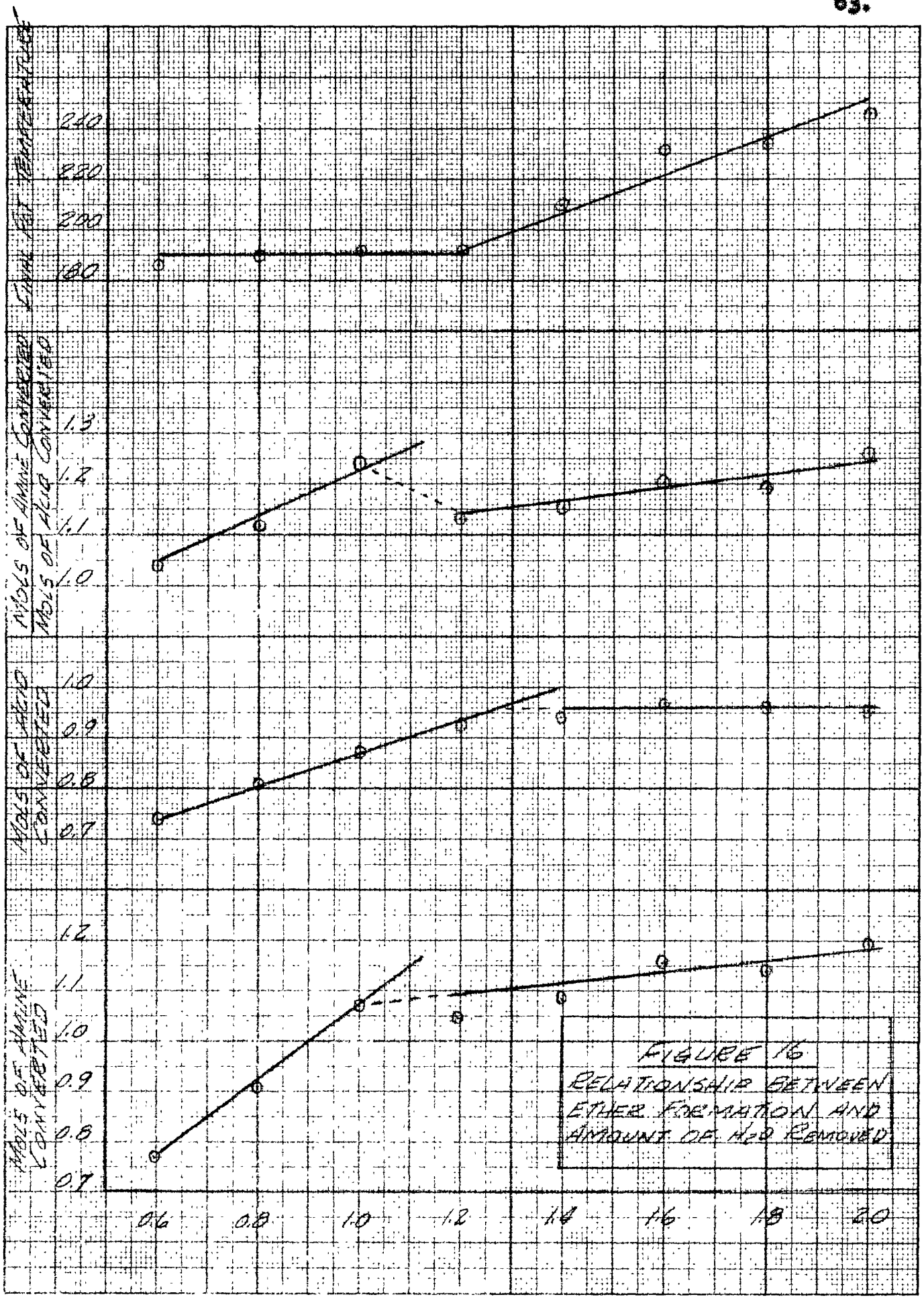


FIGURE 16
RELATIONSHIP BETWEEN
ETHER FORMATION AND
AMOUNT OF H_2O REMOVED

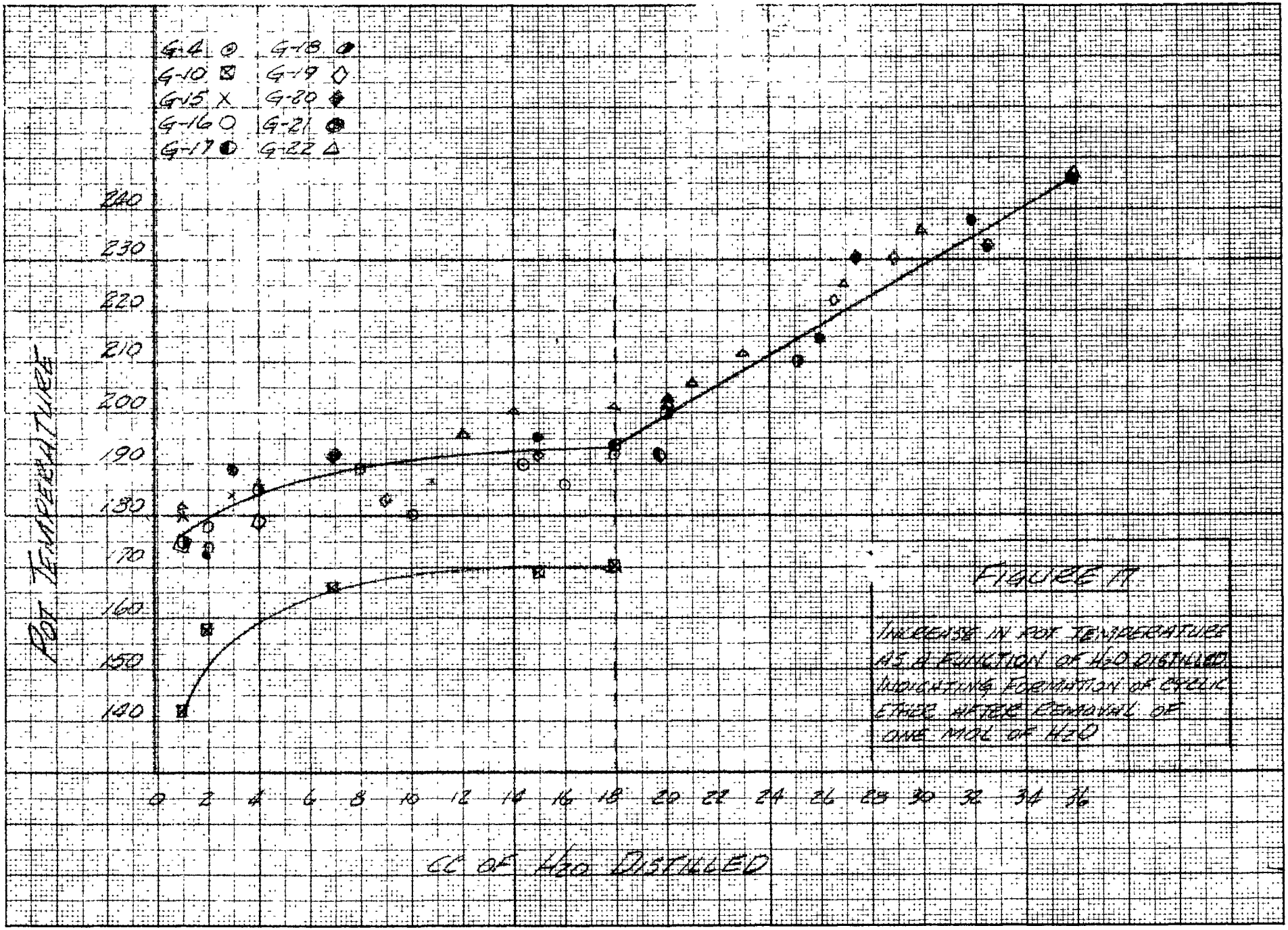


FIGURE IV

INCREASE IN POT TEMPERATURES AS A FUNCTION OF H_2O OBTAINED INDICATING FORMATION OF CYCLIC ETHER WITH REMOVAL OF ONE MOL OF H_2O

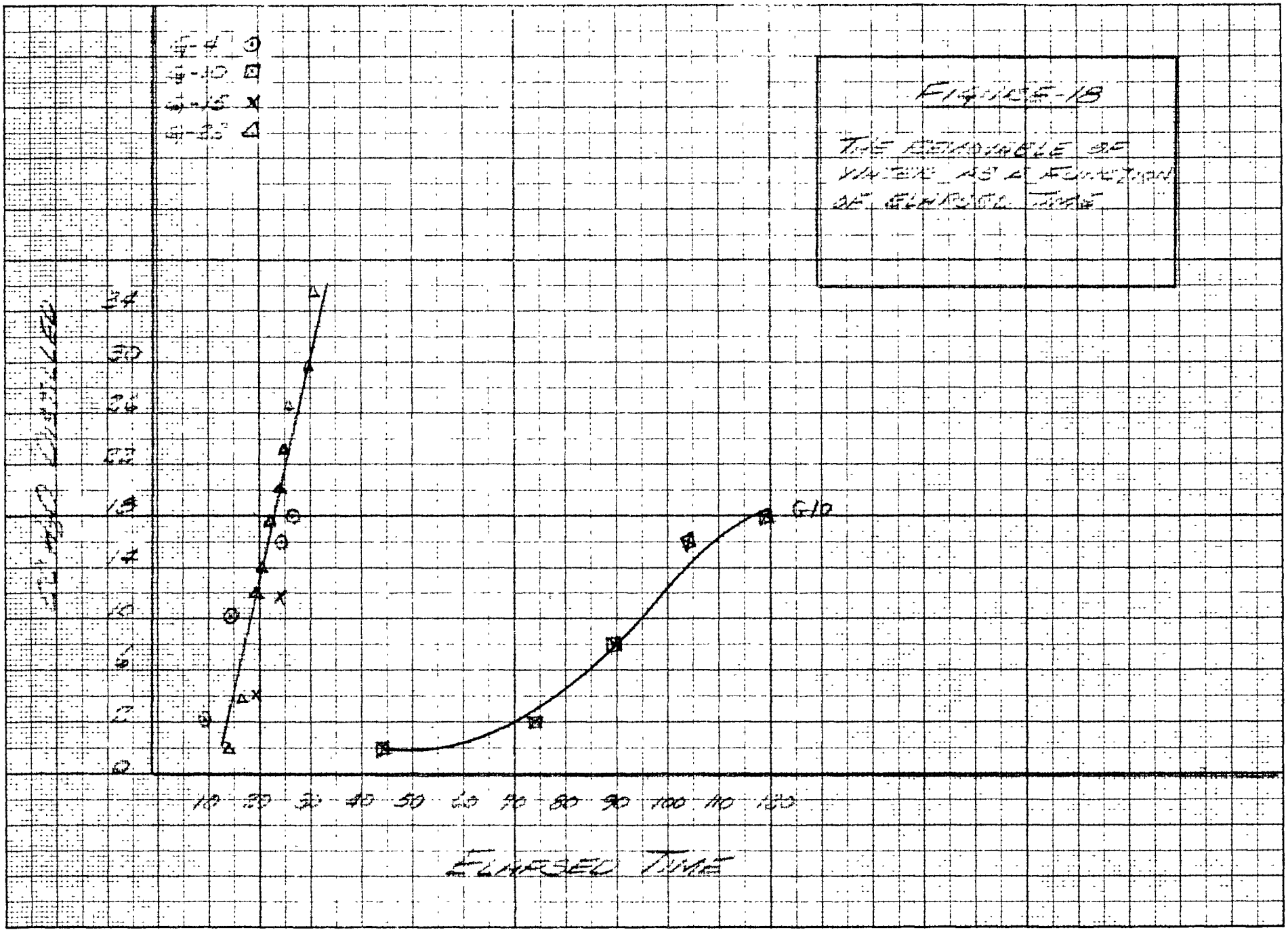
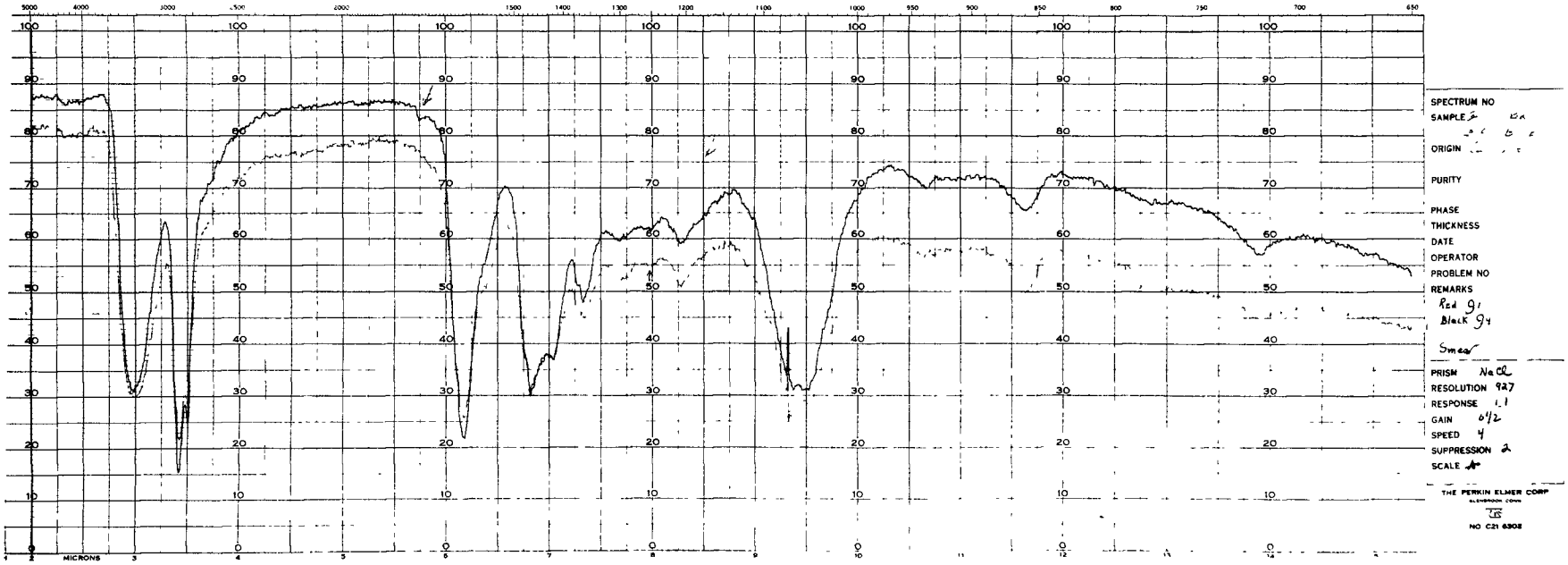
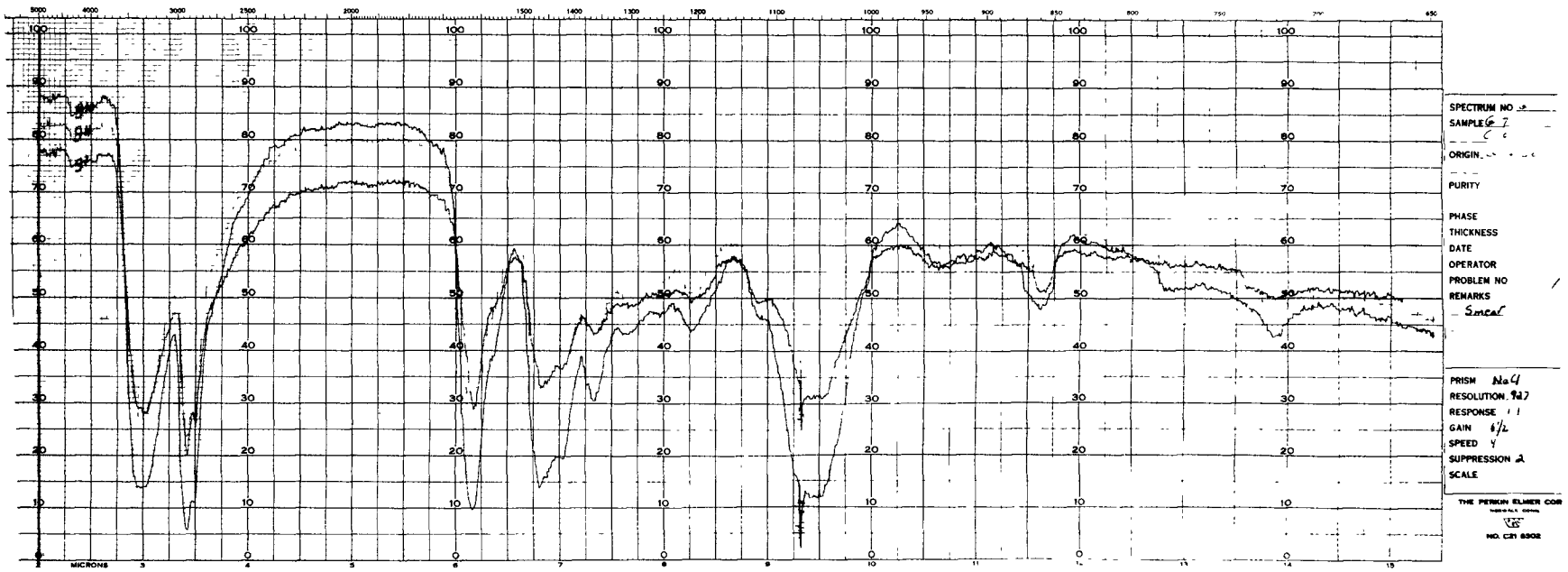


FIGURE-18

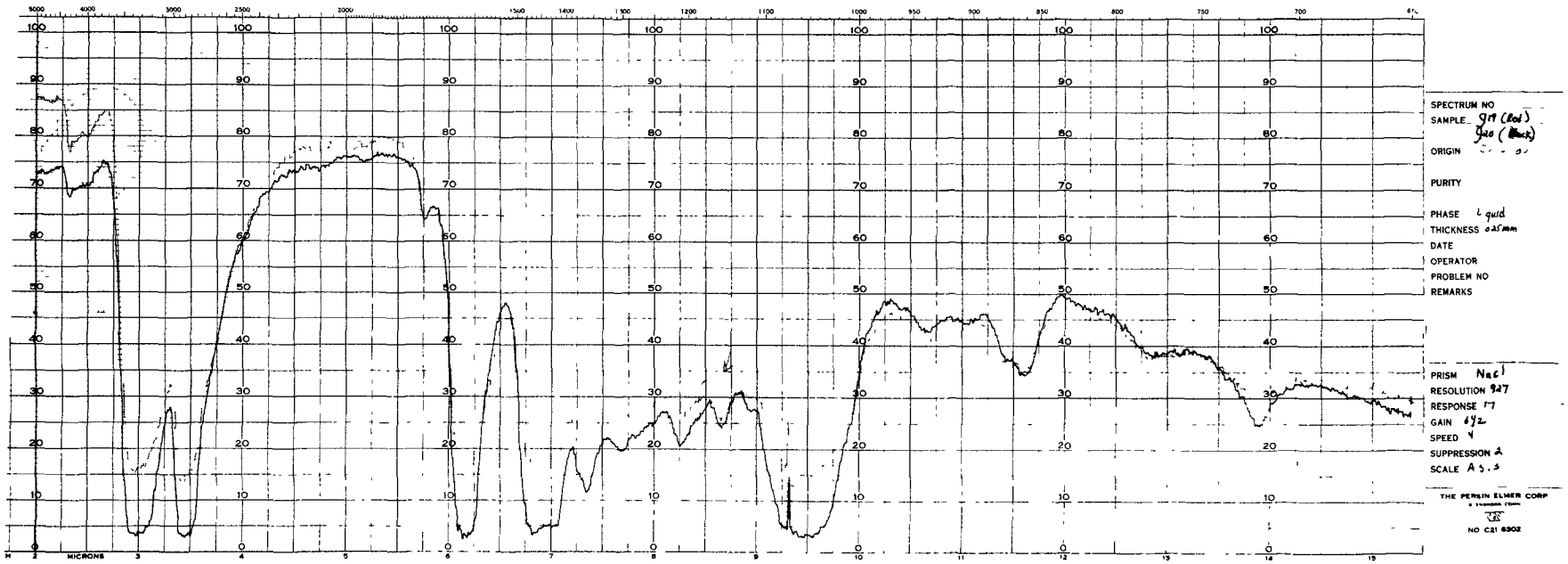
THE REMOVABLE OF WATER AS A FUNCTION OF ELAPSED TIME


MICRONS
SPECTRUM NO
SAMPLE
ORIGIN
PURITY
PHASE
THICKNESS
DATE
OPERATOR
PROBLEM NO
REMARKS
Red 91
Black 94
Smear
PRISM NaCl
RESOLUTION 927
RESPONSE 1.1
GAIN 0½
SPEED 4
SUPPRESSION 2
SCALE
THE PERKIN ELMER CORP
NO C21 6302

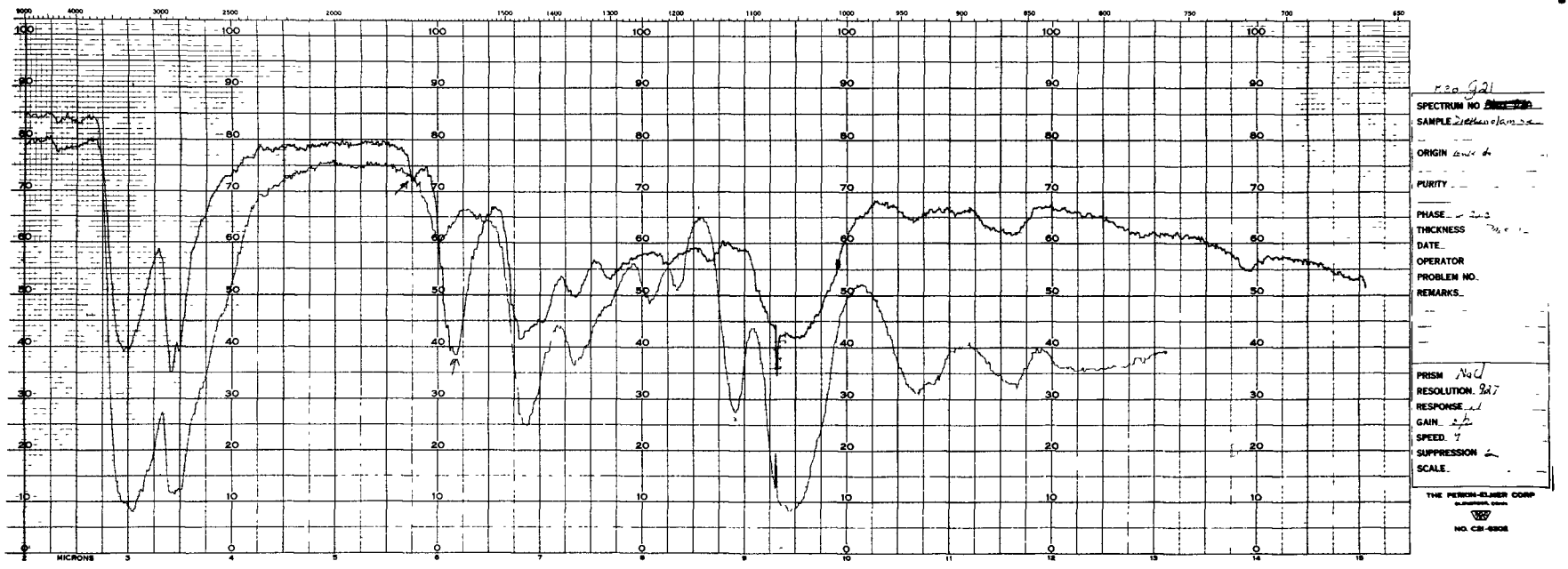
Curve A


SPECTRUM NO
SAMPLE 67
ORIGIN
PURITY
PHASE
THICKNESS
DATE
OPERATOR
PROBLEM NO
REMARKS
Smear
PRISM NaCl
RESOLUTION 9d7
RESPONSE 1
GAIN 6½
SPEED 4
SUPPRESSION 2
SCALE
THE PERKIN ELMER COR
NO. 021 6302
MICRONS

Curve B


SPECTRUM NO
SAMPLE 919 (Red)
920 (Black)
ORIGIN
PURITY
PHASE Liquid
THICKNESS 0.25mm
DATE
OPERATOR
PROBLEM NO
REMARKS
PRISM NaCl
RESOLUTION 927
RESPONSE 1.7
GAIN 6½
SPEED 4
SUPPRESSION 2
SCALE A 5.5
THE PERKIN ELMER CORP
NO C21 6302
MICRONS

Curve C


MICRONS
SPECTRUM NO
SAMPLE
ORIGIN
PURITY
PHASE
THICKNESS
DATE
OPERATOR
PROBLEM NO.
REMARKS
PRISM
RESOLUTION
RESPONSE
GAIN
SPEED
SUPPRESSION
SCALE
THE PERKIN-ELMER CORP
NO. CSI-6808

Curve D

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(3) Hampton, B. L. and Pollard, C. B.; Journal of American Chemical Society, Volume 58, 1936, Page 2338

(4) Bellamy; Infra Red Spectra of Complex Molecules Wiley, 1954