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Continuous interfacial polycondensation of nylon 6-6

Chandrakant A. Patel
New Jersey Institute of Technology

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CONTINUOUS INTERFACIAL POLYCONDENSATION

OF NYLON 6-6

BY

CHANDRAKANT A. PATEL

A THESIS

PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

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ABSTRACT:

Nylon 6-6 has been prepared by continuous interfacial polycondensation from hexamethylene diamine and adipyl chloride in a stirred system. The effect of monomer concentration and stirring on the intrinsic viscosity of polyamide 6-6 solution in formic acid (90%) was determined.

As stirring rate and monomer ratio increased, the intrinsic viscosity and polymer yield went through a maximum. Diffusion coefficient (D_{AB}) for hexamethylene diamine was calculated and from that J_D and mass transfer coefficient $/D_{AB}$ were calculated. A relation between Reynolds number and J_D factor was established by correlation curve.

APPROVAL OF THESIS

CONTINUOUS INTERFACIAL POLYCONDENSATION

OF NYLON 6-6

BY

CHANDRAKANT A. PATEL

FOR

DEPARTMENT OF POLYMER ENGINEERING

NEWARK COLLEGE OF ENGINEERING

BY

FACULTY COMMITTEE

APPROVED: _____

_____

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NEWARK, NEW JERSEY

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INTRODUCTION:

In 1928 W. H. Carothers worked with polyamides. Nylon 6-6 was first prepared in 1935 from adipic acid and hexamethylene diamine. Commercial production of Nylon began in 1939. (1).

T   The nylon numbering system is an abbreviated description of material used in the preparation. For example, nylon 6-6 means that diamine contained six carbon atoms and the dibasic acid contained six carbon atoms.

The diversity in type of polyamid resins is remarkably large. It results from wide choice of starting materials, variation in degree of reaction, modification through other chemical reactions.

There are several methods to prepare polyamids. Of these methods, the following two are important.

1. Condensation reaction

2. Interfacial polymerization

Condensation reaction takes place under high temperature and reduced pressure. Also, it requires special types of equipment which are expensive. In interfacial polycondensation, reaction occurs at the interface of two immiscible liquids at room temperature. Equipment for this kind of reaction is simple and less expensive.

Nylon 6-6 is a white translucent and crystalline material with an approximate melting point of 265° C. and having 1.09 to 1.15 gms/cc density. Both as a plastic and as a fiber, it has high mechanical strength and temperature and chemical resistance. Its largest application is in the manufacture of fibers, films, and molded plastics.

It is the purpose of this paper to present a novel and relatively simple method to study intrinsic viscosity yield of nylon 6-6.

BACKGROUND AND THEORY:

Basically, polymers have been divided into two main groups on the basis of a comparison of the structure of the repeating unit of the polymer with the structure of the monomer from which the polymer was derived.

(A) Addition Polymer:

An addition polymer is a polymer in which the molecular formula of repeating unit is identical with that of the monomer. Addition polymerization involves chain reactions in which the chain carrier may be an ion or a reactive substance with one unpaired electron, called a free radical.

$$CH_2 = \underset{X}{CH} \longrightarrow CH_2 - \underset{X}{CH} - CH_2 - \underset{X}{CH} -$$
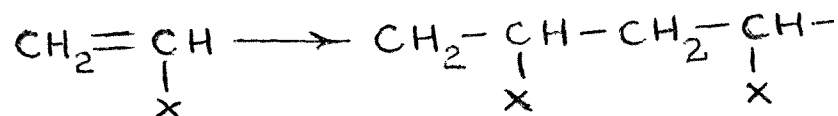
ADDITION POLYMER

(B) Condensation Polymer:

In condensation polymerization, the polymer may be formed from monomers bearing two or more reactive groups of such a character that they may condense intermolecularly with elimination of a by-product, often water.

Polymers may also be classified by their structural configuration, as per example linear, branched or cross linked polymers.

In a linear polymer, the structural units are connected to one another in linear sequence.

—A— A— A— A— A— A—

LINEAR POLYMER

The branched polymers have side chains growing off at various points.

BRANCHED POLYMER
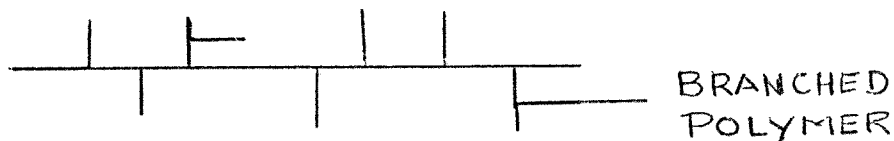
Crosslinked polymers are linked with small structural units.

CROSSLINKED POLYMERS
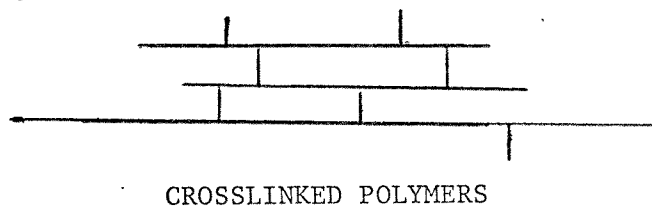
The extent of condensation polymerization may be controlled at any intermediate point, and the degree of polymerization is a measure of molecular weight, (2). Addition polymerization utilizes free radical or ion-forming types of catalysts, and the extent of addition polymerization is a measure of the percentage of monomer conversion.

The effect of heat at high temperature on the stability of natural and synthetic polymers has been investigated. Study of thermal behavior of polymers, particularly on their thermal degradation, is of prime importance from the scientific point of view. Practically all of the work done on nylon 6-6 involving the effect has been accomplished at temperatures above the melting point. At these high temperatures, polymers undergo both physical and chemical changes. Thermal oxidation, degradation, branching, crosslinking and further polymerization may cause the polymers to crosslink and degrade, or they may lead to acidic products which may cause further degradation.

The effects of heat on nylon 6-6 at temperatures above the melting point were investigated by Achhammer (4), and Liquori(1). In their reports, nylon 6-6 at high temperatures undergoes decomposition, cross-linking, and the degradation.

Achhammer (4) pyrolysed a polyamide sample at 400° C. in vacuum, and gaseous products were CO, CO_2, Hydrocarbons', and cyclopentanone. The mechanism is described as follows:

$$R-NH-CO-(CH_2)_4-COOH \longrightarrow R-NH_2 + CO_2 + \text{Cyclopentanone}$$

The fission in the $-CO.(CH_2).CO-$ units might decompose to hydrocarbons, CO_2, cyclopentanone, and CO. However, Liquori (1) could not find cyclopentanone or keto-diacids from pyrolysis of nylon 6-6 at $275^{\circ}C$. Hill (28) found that the free amino end groups in nylon 6-6 react and have a tendency to form secondary amine link. The secondary amino group is believed to act as a point for cross-linking and gel formation.

$$-CO.NH.(CH_2)_6.NH_2 + NH_2(CH_2)_6.NH.CO \longrightarrow$$

$$-CO.NH.(CH_2)_6.NH.(CH_2)_6.NH.CO + NH_3$$
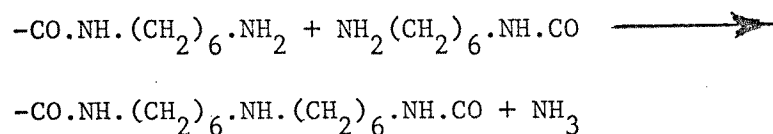
Crosslink can only be formed in presence of few reactive sites which are sufficient to create dimensional network. The crosslinked product is no longer soluble in formic acid.

Straus and Wall (29) suggested that most possible scission would occur at the two linkages shown by dotted lines:
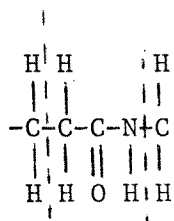
were bonding energy of normal C-C is 80 kcal and that of normal C-N

bond is 68 kcal.

The following summary can be made regarding the types of reactions

taking place when a polyamide is heated: polycondensation, crosslinking,

and degradation mechanism will be summarized in general form.

1. Polycondensation

(A) A reaction between two $-NH_2$ groups:

$$R.NH.(CH_2)_6NH_2 + NH_2.(CH_2)_6NH.CO.R' \rightleftharpoons$$

$$R.NH.(CH_2)_6N.(CH_2)_6.NH.CO.R' + NH_3$$
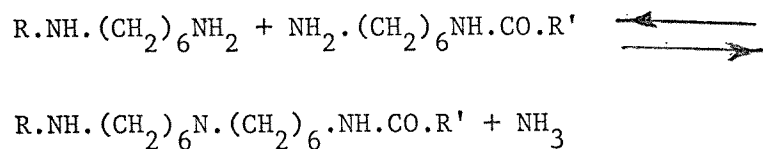
(B) A reaction between the amino group and -OCl end group.

$$R.CO.NH.(CH_2)_6.NH_2 + ClCO.(CH_2)_4CO.NH.R' \rightleftharpoons$$

$$R.CO.NH.(CH_2)_6.NH.CO.(CH_2)_4.CO.NH.R' + HCl$$
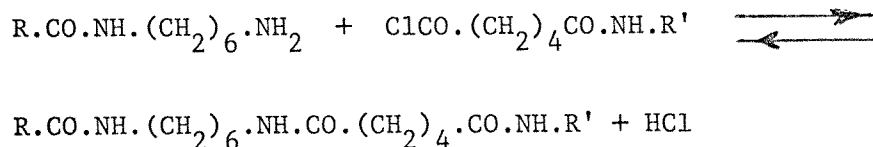
2. Crosslinking

(A) An active site from the -OCl group may cause branching

$$C-O-Cl + H_2N \qquad \overset{|}{\underset{|}{C}}-NH + HCl$$

(B) An active site from secondary amino group may cause branching

$$\overset{|}{\underset{|}{N}}-H + -ClCO \qquad N-\overset{\|}{\underset{O}{C}} + HCl$$
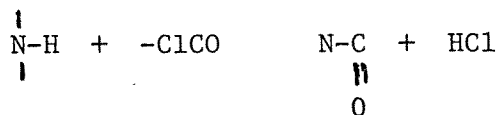
3. Degradation

(A) Scission in the NH.CH - linkage

$$-NH.(CH_2)_5.CH_2 \overset{|}{\underset{|}{.}} NH.CO.(CH_2)_4.CO \longrightarrow$$

$$-NH(CH_2)_5 CH_3 + NH_2.\underset{O}{C}.(CH_2)_4.CO-$$
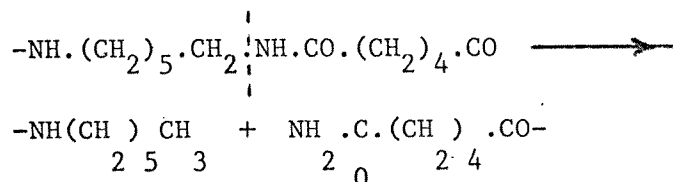
INTERFACIAL POLYMERIZATION:

In interfacial polycondensation, the formation of the polymer takes place at the interface of two non-miscible liquids and takes place largely by diffusion mechanism. (17, 18).

The polymer is formed at the interface, i.e., on the contact surface of two phases, the system is stirred to ensure better contact. Reaction is rapid at room temperature. The reaction between adiphl chloride and hexamethylene diamine takes place in the following manner:

$$ClCO(CH_2)_4COCl + NH_2(CH_2)_6NH_2 \longrightarrow k'$$

$$ClCO(CH_2)_4\underset{Cl}{-CO}\ldots\ldots\underset{H}{NH}-(CH_2)_6NH_2 \longrightarrow k''$$

$$ClCO(CH_2)_4CONH(CH_2)_6NH_2 + HCl$$
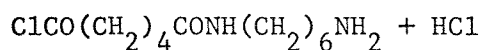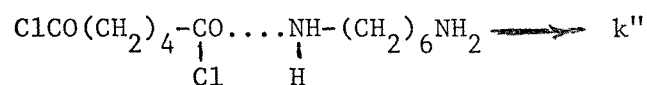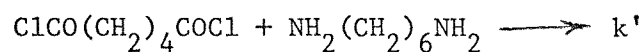
The acid halides react with diamines by way of nucleophilic substitution, through the stage of protonized amid, which readily gives up the proton in the presence of a base. Either the amine group of the monomer or the end group of the polymer acts as proton acceptor. Water is also a proton acceptor.

In the interfacial polycondensation method, starting reagents are dissolved separately in two immiscible liquids. One of these liquids is water and the other is organic phase. Aqueous phase serves as a proton carrier and removes by product (acid) from the polymerization zone.

Due to specific features of chemical reactions at liquid liquid interface the course of reaction is probably affected to a substantial extent by the following factors: (19).

1) Chemical equilibrium at the interface may differ from the equilibrium in the bulk.

2) The reacting molecules at the interface may be oriented to different extents, up to and including formation of an oriented film.

3) The rate of diffusion across the interface is slower than in the bulk.

4) The presence of potential difference at the interface.

Effect of Principle Factors on Interfacial Polycondensation

This section will deal with the effect of the temperature, time duration, nature of solvent, reactant flow rate, stirring rate and monomer ratio on the course of interfacial polycondensation.

TEMPERATURE:

Interfacial polycondensation may be affected if the rate of reaction is much faster than the rate of diffusion. In interfacial polycondensation, the effect of temperature on the molecular weight and yield of polymer obtained is of complex nature. The molecular weight and yield of polymer urea decreases with increasing temperature. This is caused by the relative acceleration in the rate of hydrolysis of acid chloride, connected with the increase in the hydrolysis rate constant and mutual stability of liquid at higher temperature. Korshak and his co-workers (20) found increase in temperature did not improve yield of nylon 6-6 in benzene water system. In fact, by cooling or controlling the temperature yield is increased. (21).

RATE OF REACTION:

The rate of reaction in formation of polymer may vary, depending on the type of monomer employed. If the reaction conducted under static condition, i.e., without mixing the process rate slows down considerably, since the polymer film formed inhibits the diffusion of polymers toward one another (20).

In homogeneous solution systems, the rate of reaction of aliphatic chloride with primary diamines is of the order of 10^2 to 10^6 1/mole-sec. The rate of reaction is faster than the rate of mixing. The rate of reaction would be much slower in non-polar solvents. It is essential for interfacial polycondensation polymerization, that rate of reaction should be faster, so that polymerization completes before the polymer becomes immobilized in the precipitated state.

EFFECT OF STIRRING AND MONOMER CONCENTRATION

Stirring is a critical variable in the preparation of polymer by the interfacial polycondensation process. A polymer film would be formed at the interface of two immiscible liquids. By agitation, polymer film would be torn away and a fresh interface would be available which would form new polymer film. Korshak found that yield of nylon 6-6 increases as the stirring rate increases from 1000 to 7500 R.P.M. He also found that specific viscosity increases as the R.P.M. increases. (20).

The effect of stirring is to increase the availability of the diamine. To keep the reactants balanced at the polymerization site and thus to attain highest viscosity, the acid chloride must be more concentrated, or diamine more diluted, as the stirring rate increases.

A number of researchers confirmed the fact that peaks in the viscosity of polymer arise from variation of the reactant concentration, but do not confirm the shift in position of the peaks with changes in solvent phase, or rate of stirring.

Mogat (29) prepared polyamide 6-10 by a batch process in a stirred system. He used 0.022 molar hexamethylene diamine, 0.022 molar sebacoyl chloride and 0.044 molar sodium hydroxide. The product obtained in this method had an inherent viscosity of 1.76 dl/gm. He concluded that 12% excess of either of the reactants would not increase inherent viscosity.

The effect of the monomer ratio on the molecular weight of polymer may be explained in terms of diffusional mechanism of interfacial poly-condensation. It is known that molecular weight or degree of polymerization P, of polycondensed polymer is connected with the initial monomer ratio in the reaction zone by following equation (25).

$$P = \frac{1 + q}{q - 1}$$

where $q = N_A/N_B$

N_A and N_B is molar ratio of monomer A and B.

The diamine can be used as acid acceptor in an interfacial polyconden-sation reaction. The small amount of diamine might work better. Earckson (30) found that if monomer concentration of diamine is more than 1:1, the yield and inherent viscosity of polymer decreases.

REACTANT FLOW RATE:

In interfacial polycondensation reactions it is important to observe the flow rate of the reactants, R. C. Kispert (9) indicated that eddy diffusion is responsible for an increase in mass transfer of the diamine in continuous cascade system. The effect of improved mass transfer can be explained by considering the treatment of uncatalysed liquid-liquid heterogeous reactions by Hougen and Watson (31).

In stirring system

$$r_b = \frac{1}{1/k_b} + (R_{Bb} X_{Bb} V_b / k_{AL} A_v) \ (R_{Aa} R_{Bb} X_{Aa} X_{Bb} k_a)$$

k_b = Specific reaction rate inorganic phase.

$X_{Aa} \ X_{Bb}$ = Mole fraction of diamine (aqueous phase) and dibasic chloride (organic phase) respectively.

k_a = Distribution equilibrium constant of diamine.

$R_{Aa} R_{Bb}$ = Activity coefficient of diamine and dibasic chloride.

V_b = Volume of organic phase.

A_v = Interfacial area per unit volume.

k_{AL} = Mass transfer coefficient of diamine.

pH OF AQUEOUS PHASE:

The yield and molecular weight of polymer formed depend to a large extent on the pH value of the water phase. In interfacial polycondensation polymerization which involves acid chlorides, HCl acceptors (mostly alkali) are introduced in aqueous phase.

The rate of chain propagation in polycondensations of diamine with chlorides of dicarboxylic acids is generally written as follows: (32)

$$v_g = k_1 C_{dA} C_{xa}$$

Where k_1 = Rate constant of chain propagation reaction.

C_{dA} = Concentration of diamine.

C_{xa} = Concenrration of the acid chloride.

Chain break off during interfacial polycondensations involving diamines and chlorides of dicarboxylic acid may be produced by the reaction of the terminal acid chloride group with water or alkali.

$$-ROCl = H_2O \qquad RCOOH + HCl$$

or by the reaction of a terminal amino group with acid:

$$-RNH_2 + HCl \qquad -RNH_3^* + Cl^-$$

Even though the reaction is accompanied by the evolution of HCl, the molecular weight of the product can be increased by adding a certain amount of acid to the aqueous phase. (33). Molecular weight may also increase in an acid medium due to increase in the value of k_1, i.e. due to catalysis of the main reaction by protons.

EXPERIMENTAL PROCEDURE:

A flow chart of the experimental apparatus is shown in figure 1. Solutions of adipyl chloride and hexamethylene diamine were stored in constant head reservoirs. From the reservoirs the liquids went to polyvinyl duplex pumps. This duplex pump regulates the flow rates of the reactants which have a maximum flow rate capacity of 12.4 gallons/ hour. The flow from the pump was then split through the use of a 1/4" polyvinyl chloride tube (D). One stream (the bypass) passed through a 1/4" polyvinyl needle valve (E) and returned to reservoirs. The other stream passed through a 1/4" polyvinyl check valve (F) and continued to the 2"x4"x4" Durallin block (G), which was used to support the reactor and keep it in a horizontal position.

The reactor used in the experiment has dimensions as shown in figure 2. It was attached to the Durallin block (C) by means of flange (F) and a gasket arrangment. A stainless steel paddle with a pitch of 45 and diameter of 3/8" was used as the agitator (E) for the reactor. The stirrer was attached to a 3/32" diameter shaft and extended 1/2" in to reacting zone. The agitator was driven by a variable speed motor (A) which had shaft extentions on both sides of the motor body. One side was attached to the stirrer through the use of bearing blocks and couplings and the other end to a Smith hand tachometer to measure the R.P.M. During the experiment speed was maintained between 2000 and 7000 R.P.M. A 2½" long U-shaped glass tube (H) was attached to the reactor as shown in Figure 2.

Before the reactants enter, the reactor pump is primed for one hour

to minimize the fluctuation in flow rate. After setting desired R.P.M.,

the flow rate of both the reactants was set to 70 cc/min. After the form-

ation of the polymer in the reactor, product was collected into a 4-litre

beaker containing a dilute solution of hydrochloric acid which serves as

a terminator. This solution was stirred at a constant speed.

After stirring for five minutes, the mixture was filtered and washed

thoroughly with distilled water. Then the polymer was oven-dried at 60°C

for 24 hours.

Viscosity Measurement

To determine the instrinsic viscosity, the following apparatus was

used: Ubbelohde Viscometer, a stop watch reading to 0.01 second accuracy,

thermoset bath, graduated pipets, rubber suction bulb, medium-sintered

glass crucible and 90% formic acid.

In 10 ml formic acid .1 gm of polymer powder was dissolved at 40°C.

Then the solution was filtered through a medium-sintered glass crucible.

The ubbelohode viscometer was used to determine the efflux time for the

polymer solutions. Viscometers used during the experiment were cleaned

thoroughly with cleansing solution and then washed with water. After dry-

ing the viscometer 3cc. of filtrate was transferred to the viscometer.

Then the viscometer was suspended in a constant temperature bath which was

set to 40°C. About 45 minutes were required to reach thermal equilibrium.

Using the suction bulb on top of the capillary tube, the solution was

raised to about 1" above the upper line on the capillary tube. After re-

moving the suction bulb, efflux time of the solution was measured with the

stop watch. During the period of this measurement of time, the pump was
turned off to prevent vibration. Three readings, showing no trends, were
averaged for the solution time. 1 ml of formic acid was then added to
this solution to obtain another concentration. After thorough mixing and
allowing time for thermal equilibrium, efflux time was measured. Diluting
the original solution was done five times. The measurement of efflux time
for formic acid was carried out similarly.

Since the solutions examined were sufficiently diluted, the relative
viscosity was obtained from the ratio of two corresponding times.

$$\frac{n}{n_o} = \frac{T}{T_o}$$

Where n and n_o are viscosity of the Polymer solution and solvent
respectively.

T = Efflux time for solution

T_o = Efflux time for solvent.

Using linear graph paper each reduced specific viscosity was plotted
against concentration, then the straight line connecting these points
was extrapolated to zero concentration. Intrinsic viscosity is found
from the reduced viscosity at zero concentration.

RESULTS:

Tables 1 to 4 represent the data collected for relative viscosity, specific viscosity, reduced specific viscosity and intrinsic viscosity as a function of different stirring rate and monomer concentration.

Table 5 gives data of polymer yield per minute as a function of different stirring rates and monomer ratio.

Table 6 represents the calculated value of Reynolds number as a function of relative viscosity.

Table 7 contains the data of calculated values of mass transfer coefficient and j_D factor.

Figures 1 to 24 represent the plot of reduced specific viscosity vs. concentration of polymer solutions.

Figures 25 to 28 show the plot of polymer yield per minute vs. Reynolds number and different R.P.M.

Figures 29 to 32 show the plot of intrinsic viscosity vs. Reynolds number and different stirring rates.

Figure 33 shows the plot of intrinsic viscosity vs. monomer ratios.

Figure 34 shows the plot of Log j_D vs. Log R_e.

DISCUSSION:

The data of relative viscosity (nR_e), specific viscosity (n_{sp}),

reduced specific viscosity ($n_{Re-sp.}$) and intrinsic viscosity are given

in Tables 1 to 4 and Figures 1 to 24.

$$n_{Re} = \frac{\text{Efflux time of solution}}{\text{Efflux time of solvent}}$$

$$n_{SP} = n_{Re} - 1$$

$$n_{Re.sp} = \frac{n_{sp.}}{\text{Concentration of polymer}}$$

As from the figures, linear plots of reduced specific viscosity versus

concentration of polymer were obtained. By extrapolating it to zero con-

centration of polymer solution, intrinsic viscosity of polymer was deter-

mined. This intrinsic viscosity is the measure of the degree of polymeri-

zation of polymer.

POLYMER YIELD:

In Figures 25 to 28 plots of yield (gms/minute) versus stirring speed

(R.P.M.), and Reynolds number (R_e) have been drawn. Table 5 presents the

data of yield.

The yield was calculated as follows: The two monomers Adipyl chloride

(70 c.c./minute) and Hexamethylene diamine (70 c.c./minute) were fed into

the reactor, and the stirring was at constant R.P.M. Reaction takes place

in the reactor. The polymer was then collected in a beaker for ten minutes.

Hydrochloric acid was used as terminator. Then polymer was washed with

water and oven-dried at 60°C. By weighing out polymer yield was calculated

as per gms/minutes.

The yield of nylon 6-6 increases at a constant reactant ratio and then decreases or levels off, as the R.P.M. increases. Hexamethylene diamine is the controlling factor. As the R.P.M. increases, mass transport of hexamethylene diamine improves in the reaction. That is why yield of the polymer increases. The decrease in the yield is explained by following fact. Due to the improved mass transport of hexamethylene diamine over Adipyl chloride, the proportion of reactant becomes imbalanced, which leads to the lower yield. Decrease in yield may be due to the choking the reactor. As the R.P.M. increases the reaction becomes faster and forms polymer more rapidly, filling the reactor. Presence of this polymer in the reactor hinders incoming reactant, which leads to the lower yield.

The polymer yield increases as the reactant ratio decreases (adipyl chloride/hexamethylene diamine, i.e. decreasing the concentration of adipyl chloride) at constant stirring speed. Morgan and Kispert (9, 10, 11) had reported that yield of the polymer increases as the reactant ratio increases. But in this case it behaves the other way. This may be explained as follows:

1. The diamine molecules are used by acid (by product) in the presence of more molecules of adipyl chloride.

2. Polymerization zone has more molecules of adipyl chloride than hexamethylene diamine, which imbalances the reactant and leads to the lower yield.

In Figures 25 and 26 the plots show that as we have lower concentration of hexamethylene diamine the yield has decreased noticably, even if we have higher concentration of adipyl chloride.

Intrinsic Viscosity as a Function of
Stirring Rate and Reactant Ratio

Intrinsic viscosity of polymer has been calculated for different

reactant ratios at different stirring rates. From that, Reynolds num-

ber (R_e) was calculated by the following formula:

$$R_e = (d^2/v) \ N$$

where d = diameter of the stirrer units

$v = \mu/\rho$ kinematic viscosity (cm^2/sec.)

μ = viscosity of slurry (cp)

ρ = density of slurry (gm/cc)

N = the stirring rate R.P.M.

The calculations are shown in sample form. Figures 26 and 27 show

plots of intrinsic viscosity versus R.P.M. Figures 30 and 31 show

the plots of intrinsic viscosity versus Reynolds number (R_e).

Figures 26, 27 and 30, 31 show that intrinsic viscosity increases

at constant R.P.M. or at constant Reynolds number as the reactant ratio

decreases. One can observe that at a reactant ratio of 0.4 we have high-

est intrinsic viscosity.

Sokolv (12) has reported that intrinsic viscosity decreases as the

reactant ratio increases (in gas - liquid interfacial polycondensation).

Morgan (10) has also reported that intrinsic viscosity in liquid-liquid

interfacial polycondensation decreases as the reactant ratio increases,

keeping the stirring rate constant.

From Figures 26, 27 and 30, 31 one can see that the intrinsic viscosity of the polymer first increases and then decreases at constant reactant ratio as R.P.M. and Reynolds numbers are decreasing.

These resultant changes are because of the change in hydrodynamic conditions which affects the mass transfer. Intrinsic viscosity decreases in the transition zone and increases in the turbulent zone. Intrinsic viscosity also decreases in the laminar zone. The resultant changes in hydrodynamic condition affect the mass transfer and ultimately the molecular weight of the polymer. Because of the above theory, intrinsic viscosity increases in the turbulent zone and decreases in laminar and transition zones.

MASS TRANSFER IN CONTINUOUS POLYMERIZATION

The reaction apparently takes place at the interface between a solution of adipyl chloride and hexamethylene diamine in water which is an acid acceptor. Carbon tetrachloride is used as organic solvent for adipyl chloride. The reaction rate in interfacial polycondensation system is in the order of 10^2 to 10^6 1/mole-sec (10, 11, 13). The mass transfer of diamine is very important. It has been pointed out by many scientists (10, 11, 13, 14).

Mass transfer coefficient (K) and j_D factor have been computed by using following formulae.

$$k = (91/6) \ (1/m_i n \ c_i) \ (dci/dt) \qquad (15)$$

$$m_i = 35 \qquad (15)$$

$$m_{in} \ c_{o \ in}/c_{i \ in} \qquad (15)$$

$$c_i = \text{concentration of hexamethylene diamine}$$

j_D factor (Dimensionless group)

$$j_D = (K/cN) \quad (\quad D_{AB})^{0.66} \tag{16}$$

K   Mass transfer coefficient

c   Concentration of diamine (molar)

N   RPM

     Viscosity of the slurry (cp)

     Density of the slurry (gm/cc)

D_{AB} Diffusion coefficient of diamine

The calculations for mass transfer coefficient (K) and j_D factor are given in examples. The unit for mass transfer coefficient is cm/hr. The data for mass transfer coefficient are given in Table 7. A plot of Log J_D versus Log R_e is shown figure 34.

CONCLUSION AND RECOMMENDATIONS:

1. Intrinsic viscosity at constant reactant ratio first increases as the stirring rate increases. It has been found that at reactant ratio of 0.4 intrinsic viscosity first increases, then decreases, and again increases. This behavior may be explained by the following fact; as the stirring rate increases, mass transfer of the diamine to organic phase increases. This leads to the imbalance of the reactant which results in the formation of lower molecular weight polymer.

At lower reactant ratio of 0.4 diamine and adipyl chloride goes under faster rate of reaction and forms longer chains of polymer.

2. Intrinsic viscosity at constant R.P.M. increases as the reactant ratio (Adipyl chloride/Hexamethylene diamine) decreases. Kispert (18) has stated that intrinsic viscosity reaches the maximum point when there is balance of reactants. It can be seen from the results that at the reactant ratio of 0.4, intrinsic viscosity is at the peak.

3. Polymer yield increases as the R.P.M. increases and decreases or levels off. As the R.P.M. increases mass transfer of diamine also improves, which imbalance the reactants, and that is why yield is decreased.

RECOMMENDATIONS:

From the results of these experiments the following recommendations are made for future work.

1. For better yield and higher intrinsic viscosity of polymer monomer concentration ratio (Adipyl Chloride/Hexamethylene diamine) should of narrow.

2. Purity of monomer:

Reactant should be in a pure form. Solvent should be free from moisture and alcohol. If moisture gets into dibasic chloride from the solvent, it decomposes dibasic chloride and forms diacid and mono acid mono-chloride. The diacid may take away acid acceptor or diamine as salts. Mono chloride acts as a chain terminator.

3. Select the narrow range of R.P.M. which will give better prediction of actual trend of the viscosity and polymer yield curve.

4. Keep concentration of one monomer constant and change the concentration of another monomer for wider range.

5. Following modifications can be made to the equipment for better results.

   (a). Diameter of the reactor tube can be increased.

   (b). Longer reactor tube can be used.

   (c). More than one stirrer can be used.

BIBLIOGRAPHY:

1. Liquori, A.M., and Carelli, V., J. Polymer Sci., 1o, 510 (1953)

2. Carothers, W.H., Trans Faraday Soc., 32, 39-49 (1936)

3. Groggins, P.H., "Unit Processes in Organic Synthesis", McGraw
   Hill Book Co., Inc., New York, 1958, p 866, p 905.

4. Achhammer, B.G., Reinhart, F.W. and Kline, G.M., J. Appl Chem.
   (London) 1, 301 (1951).

5. Liquori, A.M., and Carelli, V., J. Polymer Sci., 10, 510 (1953)

6. Boulton, J., and Jackson, D.C., J. Soc Dyes Colourists, 59 21 (1943)

7. Hassenlstrom, T. Coles., H.W., Balmer, C.E., Textile Research J.
   22 742 (1952)

8. Taylor, G.B., J. Ame. Chem Soc., 69 635 (1947)

9. Kispert, R.C., and Griskey, R.G., J. Appl. Polymer Sci., 9 2337 (1965)

10. Morgan, P.W., "Condensation Polymers" Inter Science Publishers, New York,
    1962 p. 1-113

11. Morgan, P.W., SPE J., 15, 485 (1959)

12. Sokolov, L.B., and Kudim, T.V., Vysokomecullyrane Soedineniya 2(5),
    699 (1960)

13. Kispert, R.C., Foote, S.R., Griskey, R.G., J. Appl Polymer Sci.,
    Vol 12, p 137 (1968)

14. Kispert, R.C., Griskey, R.G., J. App. Polymer Sci., 9, 2337 (1965)

15. Handols, A.E., and Barton, T., Mass Heat Transfer From Drops in
    Liquid-Liquid Extraction., A.I.Ch.E. Journal, Vol 3, 127 (1957)

16. Sherwood, T.T. and Pigford, R.L., "Absorption and Extraction",
    McGraw Hill Book Co., Inc., New York (1952) p 63-80.

17. Grant, G.H., and Hinshelwood, C.N., J. Chem. Soc., 1351 (1953)

18. Hall, H.K., J. Am., Chem. Soc., 77, 5993 (1955)

19. Davies, J., Catalysis,, A Study of Heterogeneous Processes (Russian Translation).

20. Frunze, T.M., Korshak, V.V., Alieski, P.A., Vysokomolekualiya Soedin, 1795 (1959).

21. Wittbecker, E.L. and Morgan, P.W., J. Polymer Sci., 40, 289 (1959)

22. Fainburg, E.Z., and N.V. Mikhalove., Vysokomolekulya Soedinia 2(7), 1039 (1960).

23. Akutin, M.S., and Rodivilova, L.A., J. Polymer Sci., 52, 287 (1961).

24. Korshak, V.V., Frunz, T.M., Kurashav, V.V., Vysokomolekulya Soedinea, 2, 633 (1961).

25. Frunze, T.M., V.V. Kurashev, and L.V. Kozlov., Uspekhi Khimill 30(5), 606 (1961)

26. Magat, E.E., U.S. Patent #2831834 (4-22-58)

27. Jeurissen L., Plasica., 13(7), 520-23 (1960).

28. Hill, R., Chem. Ind. (London), 1983 (1954).

29. Straus, S., and Wall, L.A., J. Research Natl. Bur. Standards 63A 269 (1959).

30. Eareckson, W.M., Unpublished work.

31. Hougan, O.A., and Watson, K.M., "Chemical Process Principles, Part III" John Willy and Sons, New York, 1947, pp 1053-63.

32. Jeruissen, L., Plastica., 13(7)., 520-23.

33. Fedotova, O.Ya., I.P. Losove and N.M. Kozyreva., Vysokomoleklyarne Soedineniya., 5(3)., 363, (1963).

FLOW CHART OF THE APPARATUS   Fig 1
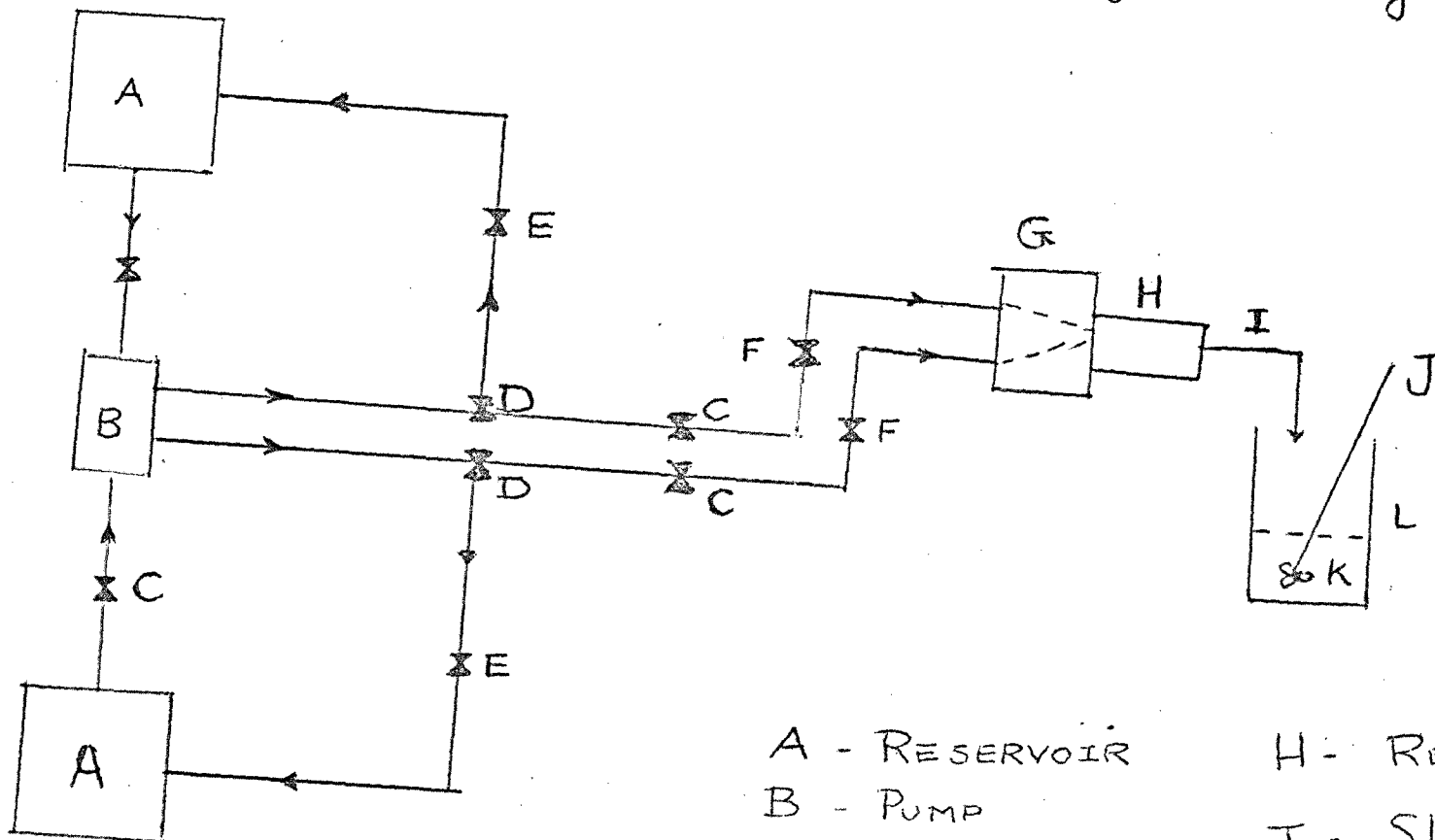
A - Reservoir   H - Reactor
B - Pump   I - Slurry
C - Two Way Valve   J - Stirrer
D - Neddle „   K - HCl Solut
E - Fitting   - ion
F - Ball   L - Beaker
G - Supporter

Fig 2

b

a

c

d

e

g

f

h

6"

2.5"

DIAGRAM OF EXPERIMENTAL REACTOR

TABLE I

Temperature 40°C

Solvent   Formic acid

Hexamethylene Diamine   0.025 molar

Adipyl Chloride   0.01 molar

Serial Number	R.P.M.	Efflux time of solvent Seconds	Concentration of Poymer gm/dl	Efflux time of solution Seconds	Relative Viscosity	Specific Viscosity	Reduced specific viscosity dl/gm	Intrinsic viscosity dl/gm
1	2000	132.14	1.00	187.13	1.4276	0.4276	0.4276	0.295
			0.75	173.16	1.3103	0.3103	0.4138	
			0.60	163.85	1.2404	0.2404	0.4001	
			0.50	154.75	1.1898	0.1898	0.3796	
			0.4285	152.17	1.1522	0.1522	0.3575	
2	3000	133.05	1.00	189.72	1.4258	0.4258	0.4258	0.267
			0.75	173.30	1.3025	0.3025	0.4025	
			0.60	163.5	1.2298	0.2298	0.3833	
			0.50	158.38	1.1903	0.1903	0.3806	
			0.4285	152.95	1.1499	0.1499	0.3501	
3	4000	121.70	1.00	177.00	1.4651	0.4651	0.4651	0.378
			0.75	163.5	1.3444	0.3444	0.4409	
			0.60	154.4	1.2587	0.2587	0.4310	
			0.50	152.81	1.2119	0.2119	0.4238	
			0.4285	144.00	1.1832	0.1832	0.4083	

TABLE 1 (CONTINUED)

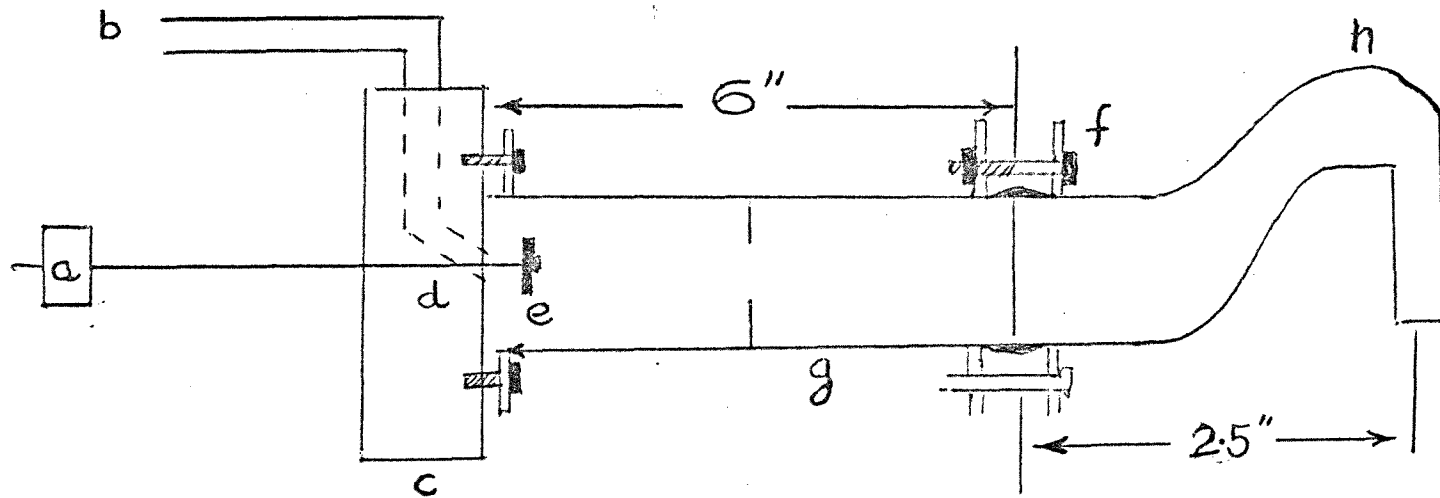| Serial Number	R.P.M.	Efflux Time of solvent Seconds	Cocentration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific viscosity dl/gm	Intrinsic viscosity dl/gm
4	5000	133.05	1.00	199.00	1.4910	0.4910	0.4910	0.330
			0.75	178.01	1.3408	0.3408	0.4544	
			0.60	168.25	1.2586	0.2586	0.4326	
			0.50	160.05	1.2100	0.2100	0.4200	
			0.4285	158.08	1.1724	0.1724	0.4023	
5	6000	132.14	1.00	201.67	1.5262	0.5262	0.5262	0.461
			0.75	183.48	1.3885	0.3885	0.5180	
			0.60	172.06	1.3012	0.3012	0.5020	
			0.50	164.60	1.2460	0.2460	0.4920	
			0.4285	159.85	1.2099	0.2099	0.4900	
6	7000	132.4	1.00	215.53	1.6310	0.6310	0.6310	0.561
			0.75	193.50	1.4651	0.4651	0.4651	
			0.60	180.05	1.3626	0.3626	0.6043	
			0.50	172.05	1.3021	0.3021	0.6000	
			0.4285	166.10	1.2532	0.2532	0.5910	

TABLE 2

Temperature 40°C
Solvent Formic Acid

Hexamethylene Diamine 0.025 molar
Adipyl Chloride 0.030 molar

Serial Number	R.P.M.	Efflux Time of Solvent Seconds	Concentration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific viscosity dl/gm	Intrinsic viscosity dl/gm
1	2000	121.70	1.00	162.30	1.3327	0.3327	0.3327	0.201
			0.75	147.95	1.2242	0.2242	0.2949	
			0.60	141.73	1.1654	0.1654	0.2756	
			0.50	137.82	1.1322	0.1322	0.2644	
			0.4285	132.90	1.0928	0.09278	0.2476	
2	3000	133.05	1.00	159.35	1.1937	0.1937	0.1937	0.182
			0.75	151.10	1.1358	0.1358	0.1810	
			0.60	146.90	1.0410	0.1041	0.1735	
			0.50	144.10	1.0831	0.0831	0.1662	
			0.4285	143.00	1.0751	0.0751	0.1655	
3	4000	132.14	1.00	154.86	1.1720	0.1720	0.1720	0.135
			0.75	143.39	1.1230	0.1230	0.1640	
			0.60	144.42	1.0903	0.0903	0.1505	
			0.50	141.95	1.0743	0.0743	0.1485	
			0.4285	140.60	1.0643	0.0643	0.1405	

TABLE 2 (CONTINUED)

Serial Number	R.P.M.	Efflux Time of Solvent Seconds	Concentration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific Viscosity dl/gm	Intrinsic Viscosity dl/gm
4	5000	121.70	1.00	153.70	1.2629	0.2629	0.2629	0.181
			0.75	143.95	1.1737	0.1737	0.2360	
			0.60	139.95	1.1504	0.1504	0.2306	
			0.50	134.69	1.1075	0.1075	0.2150	
			0.4285	133.03	1.0994	0.0994	0.2036	
5	6000	133.05	1.00	173.80	1.3063	0.3063	0.3063	0.225
			0.75	162.90	1.2243	0.2243	0.2992	
			0.60	155.01	1.1640	0.1640	0.2733	
			0.50	154.00	1.1318	0.1318	0.2626	
			0.4285	147.50	1.1114	0.1114	0.2600	
6	7000	133.05	1.00	170.05	1.2781	0.2781	0.2781	0.231
			0.75	159.30	1.1978	0.1978	0.2637	
			0.60	153.80	1.1536	0.1536	0.2560	
			0.50	149.80	1.1259	0.1259	0.2518	
			0.4285	147.10	1.1058	0.1058	0.2470	

TABLE 3

Temperature $40^{\circ}C$ Hexamethylene Diamine 0.025 molar
Solvent   Formic Acid   Adipyl Chloride   0.060 molar

Serial Number	R.P.M.	Efflux Time of Solvent Seconds	Concen- tration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific Viscosity dl/gm	Intrinsic Viscosity dl/gm
1	2000	121.70	1.00	155.05	1.2750	0.2750	0.2750	0.2350
			0.75	146.6	1.2014	0.2014	0.2685	
			0.60	142.15	1.1568	0.1568	0.2375	
			0.50	135.62	1.1145	0.1145	0.2290	
			0.4285	133.00	1.0944	0.0944	0.2225	
2	3000	132.14	1.00	172.14	1.3006	0.3006	0.3006	0.2480
			0.75	169.93	1.2177	0.2177	0.2890	
			0.60	154.28	1.1672	0.1672	0.2787	
			0.50	150.33	1.1370	0.1370	0.2741	
			0.4285	140.51	1.0621	0.0621	0.2680	
3	4000	121.70	1.00	143.45	1.1787	0.1787	0.1787	0.1460
			0.75	137.5	1.1306	0.1306	0.1742	
			0.60	134.00	1.1019	0.1019	0.1683	
			0.50	131.80	1.0722	0.0722	0.1643	
			0.4285	130.07	1.0685	0.0685	0.1605	

TABLE 3   (CONTINUED)

Serial Number	R.P.M.	Efflux Time for Solvent Seconds	Concentration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific Viscosity dl/gm	Intrinsic Viscosity dl/gm
4	5000	121.70	1.00	147.20	1.2095	0.2095	0.2095	0.152
			0.75	139.25	1.1445	0.1445	0.1930	
			0.60	136.00	1.1116	0.1116	0.1860	
			0.50	133.11	1.0950	0.0950	0.1812	
			0.4285	130.9	1.0760	0.0760	0.1775	
5	6000	133.05	1.00	164.45	1.2359	0.2359	0.2359	0.195
			0.75	155.60	1.1696	0.1696	0.2261	
			0.60	150.45	1.1308	0.1308	0.2180	
			0.50	147.30	1.1077	0.1077	0.2153	
			0.2485	146.40	1.1012	0.1012	0.2128	
6	6000	132.14	1.00	168.75	1.2763	0.2763	0.2763	0.257
			0.75	160.20	1.2048	0.2043	0.2730	
			0.60	149.05	1.1380	0.1380	0.2700	
			0.50	147.00	1.1225	0.1225	0.2650	
			0.4285	145.00	1.1141	0.1141	0.2600	

TABLE 4

Temperature 40° C
Solvent   Formic Acid

Hexamethylene Diamine   0.01 molar
Adipyl Chloride   0.042 molar

Serial Number	R.P.M.	Efflux Time of Solvent Seconds	Concentration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific Viscosity dl/gm	Intrinsic Viscosity dl/gm
1	2000	133.05	1.00	162.46	1.2170	0.2170	0.2170	0.111
			0.75	151.80	1.1410	0.1410	0.1880	
			0.60	146.78	1.1032	0.1032	0.1720	
			0.50	143.90	1.0818	0.0818	0.1635	
			0.4285	141.90	1.0668	0.0668	0.1560	
2	3000	132.14	1.00	155.20	1.1743	0.1743	0.1743	0.09
			0.75	147.25	1.1144	0.1144	0.1525	
			0.60	143.25	1.0840	0.0840	0.1400	
			0.50	140.86	1.0660	0.0660	0.1320	
			0.4285	139.35	1.0543	0.0543	0.1275	
3	4000	132.14	1.00	159.14	1.2085	0.2085	0.2085	0.109
			0.75	150.37	1.1380	0.1380	0.1840	
			0.60	145.50	1.1005	0.1005	0.1675	
			0.50	142.55	1.0788	0.0788	0.1575	
			0.4285	140.75	1.0653	0.0653	0.1524	

TABLE 4   (CONTINUED)

Serial Number	R.P.M.	Efflux Time of Solvent Seconds	Concen- tration of Polymer gm/dl	Efflux Time of Solution Seconds	Relative Viscosity	Specific Viscosity	Reduced Specific Viscosity dl/gm	Intrinsic Viscosity dl/gm
4	5000	121.70	1.00	148.53	1.2205	0.2205	0.2205	0.120
			0.75	139.18	1.1435	0.1435	0.1900	
			0.60	134.65	1.1065	0.1065	0.1775	
			0.50	131.90	1.0840	0.0840	0.1680	
			0.4285	130.20	1.0697	0.0626	0.1626	
5	6000	133.05	1.00	163.58	1.2295	0.2295	0.2295	0.126
			0.75	152.75	1.1482	0.1482	0.1976	
			0.60	147.80	1.1108	0.1108	0.1847	
			0.50	144.50	1.0860	0.0860	0.1720	
			0.4285	142.40	1.0703	0.0703	0.1640	
6	7000	121.70	1.00	149.10	1.2250	0.2250	0.2250	0.121
			0.75	139.95	1.1500	0.1500	0.2000	
			0.60	135.00	1.1102	0.1102	0.1836	
			0.50	132.09	1.08545	0.0854	0.1709	
			0.4285	130.06	1.0736	0.0736	0.1642	

TABLE 5

Serial Number	Concentration of Hexamethylene Diamine Molar	Concentration of Adipyl Chloride Molar	Ratio of Monomer Solution	Flow Rate of Hexamethylene Diamine cc/min.	Flow Rate of Adipyl Chloride cc/min.	R.P.M.	Polymer yield gm/min.
1.	0.025	0.010	0.40	70.0	70.0	2000	0.0314
						3000	0.0319
						4000	0.03995
						5000	0.0412
						6000	0.0401
						7000	0.0375
2.	0.025	0.030	1.2	70.0	70.0	2000	0.0202
						3000	0.0187
						4000	0.0162
						5000	0.0258
						6000	0.0172
						7000	0.0132
3.	0.025	0.060	2.4	70.0	70.0	2000	0.0147
						3000	0.01245
						4000	0.01999
						5000	0.01800
						6000	0.01642
						7000	0.01379
4.	0.01	0.0425	4.25	70.0	70.0	2000	0.01360
						3000	0.01592
						4000	0.01764
						5000	0.01700
						6000	0.01431
						7000	0.01236

TABLE   6

Viscosity of Solvent   0.8418 cp.
Density of the Slury   0.79862 gm/cc

Diameter of the stirrer 0.9525 cms.
$D^2 - 0.90725$

R.P.M.	Cocentration of Hexamethylelene Diamine molar	Cocentration of Adipyl Chloride molar	Relative Viscosity of 1 % Polymer Solution	Viscosity cp.	Kinematic Viscosity cm cm/sec	Intinsic Viscosity dl/gm	Reynolds Number
2000	0.025	0.010	1.4276	1.2017	0.0151	0.290	1205.80
	0.025	0.030	1.3327	1.1219	0.0141	0.201	1291.52
	0.025	0.060	1.2750	1.0733	0.0134	0.235	1350.13
	0.010	0.0425	1.2170	1.0691	0.0133	0.110	1355.44
3000	0.025	0.010	1.4257	1.2002	0.0153	0.267	1781.86
	0.025	0.030	1.1937	1.0090	0.0126	0.182	2163.20
	0.025	0.060	1.3006	------	------	------	2198.86
	0.010	0.0425	1.1743	0.9885	0.0124	0.090	2198.86
4000	0.025	0.010	1.4651	1.2333	0.0155	0.378	2344.61
	0.025	0.030	1.1720	0.9868	0.0124	0.135	2937.51
	0.025	0.060	1.1701	0.9850	0.0123	0.146	2942.27
	0.010	0.0425	1.2085	------	------	-----	-------
5000	0.025	0.010	1.4910	1.2551	0.0157	0.330	2886.39
	0.025	0.030	1.2629	1.0631	0.0133	0.181	3407.64
	0.025	0.060	1.2095	1.0182	0.0128	0.152	3558.12
	0.010	0.0425	1.2205	------	------	-----	-------
6000	0.025	0.010	1.5262	1.2848	0.0161	0.460	3383.78
	0.025	0.030	1.3063	1.0996	0.0138	0.225	3953.45
	0.025	0.060	1.2359	1.0403	0.0130	0.195	4178.95
	0.010	0.0425	1.2295	1.0350	0.0129	0.126	4200.00
7000	0.025	0.010	1.6310	1.3730	0.0172	0.561	3543.00
	0.025	0.030	1.2781	1.0759	0.0135	0.231	4714.63
	0.025	0.060	1.2763	1.0744	0.0134	0.257	4720.00
	0.010	0.0425	1.2250	1.0312	0.0129	0.121	4918.49

R.P.M.	Concentration of Hexamethylene Diamine Molar	Concentration of Adipyl Chloride Molar	Yield per Minute	dci/dt	K	K/D_{AB}	K/C	Reynolds Number	J_D
2000	0.025	0.010	0.0314	.0000814	.00001343	6.957	.0462	1205.80	0.0013
	0.025	0.030	0.0222	.0000622	.00010960	5.283	.035	1291.52	0.00099
	0.025	0.060	0.0147	.0000412	.0000675	3.499	.233	1350.13	0.0066
	0.010	0.0425	0.0136	.0000381	.0001561	8.088	.1343	1355.44	0.0038
3000	0.025	0.010	0.0319	.00008932	.0001464	7.585	.0539	1781.86	0.00095
	0.025	0.030	0.0187	.0000524	.00008589	4.450	.0296	2163.20	0.00056
	0.025	0.060	0.0124	.00003458	.00005668	2.937	.01951	-------	0.00029
	0.010	0.0425	0.01592	.00004458	.0001827	9.464	.1607	2198.86	0.00030
4000	0.025	0.010	0.03995	.0001188	.0001948	10.09	.06705	2344.61	0.00095
	0.025	0.030	0.0162	.00004536	.00007435	3.851	.01565	2937.51	0.------
	0.025	0.060	0.0208	.00005824	.00009537	4.946	.02000	2942.27	0.00028
	0.010	0.0425	0.01764	.00004939	.0002024	10.485	.17416	-------	0.00250
5000	0.025	0.010	0.0412	.0001154	.0001890	9.796	.06506	2886.39	0.00074
	0.025	0.030	0.0258	.00007224	.0001182	6.135	.04669	3407.64	0.00053
	0.025	0.060	0.0180	.0000504	.00008263	4.280	.02844	3558.12	0.00032
	0.010	0.0425	0.0170	.0000476	.0001905	10.05	.1678	-------	0.00190
6000	0.025	0.010	0.04101	.0001128	.0001849	9.579	.06506	3383.78	0.00061
	0.025	0.030	0.0172	.00004816	.00008154	4.285	.02807	3953.45	0.00029
	0.025	0.060	0.01642	.00004598	.00007537	3.961	.02594	4178.95	0.00250
	0.010	0.0425	0.01431	.00004007	.00001642	8.506	.14128	4200.00	0.01300
7000	0.025	0.010	0.0375	.0001050	.0001721	8.920	.05920	3543.0	0.00048
	0.025	0.030	0.0132	.00003696	.00006059	3.139	.02857	4714.63	0.00023
	0.025	0.060	0.01379	.00003718	.00006095	3.157	.02098	4720.00	0.00017
	0.010	0.0425	0.01236	.00003461	.00014182	7.347	.1220	4918.49	0.00098

Fig. 1

R.P.M. 2000

Monomer Ratio 0.4

REDUCED SPECIFIC VISCOSITY

5

4

3

2

0.5

1.0

CONCENTRATION

Fig. 2

RPM 3000

Monomer Ratio 0.4

Fig.3

RPM 4000
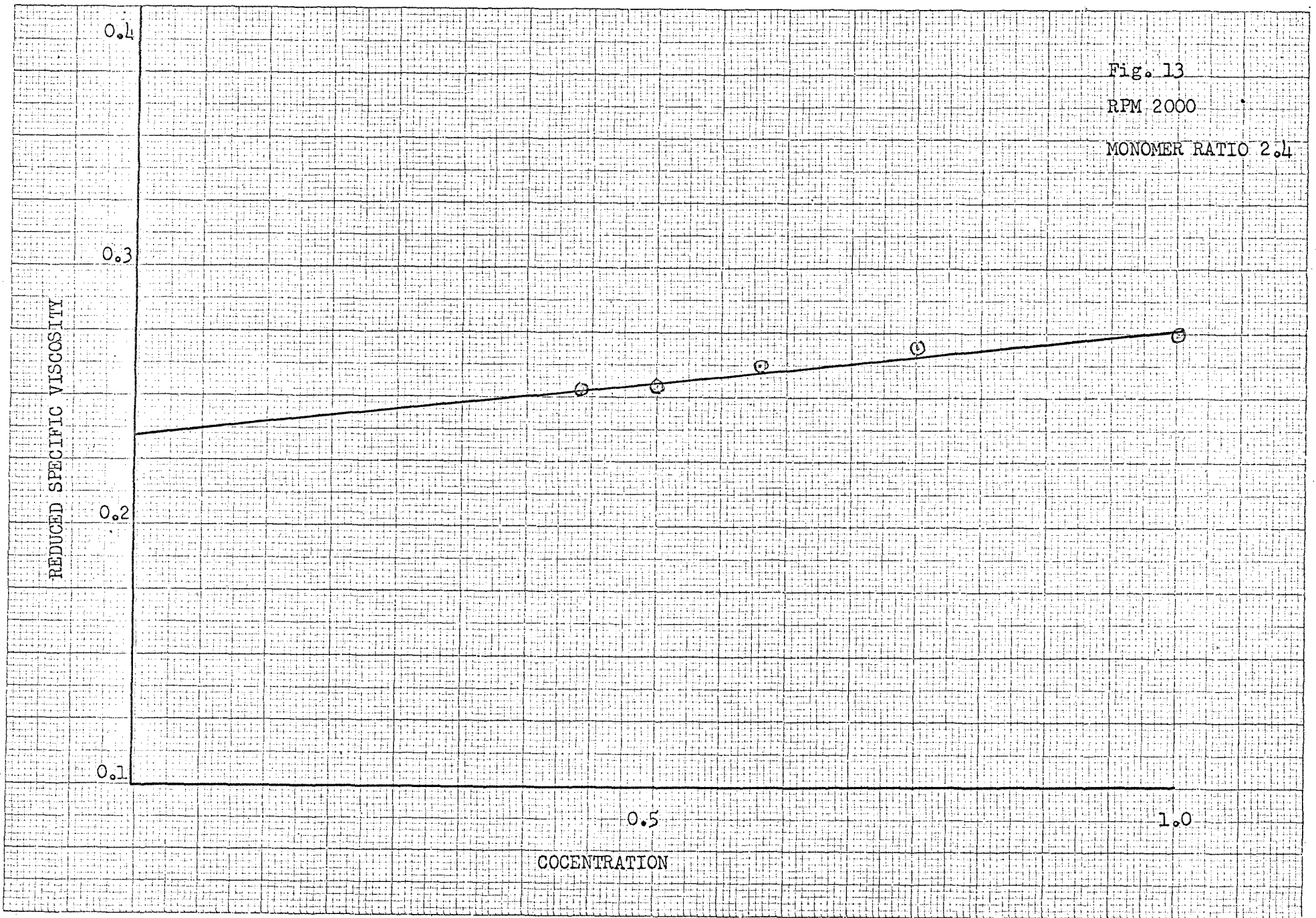
Monomer Ratio 0.4

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

Squares to the Inch

Fig. 4

R.P.M. 5000

Monomer Ratio 0.4

Fig. 5

R.P.M.   6000

Monomer Ratio   .4

Fig. 7

RPM 2000

Monomer Ratio 1.2

Fig. 6

R.P.M. 7000

Monomer Ratio 0.4

Fig. 8

RPM 3000

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

Fig. 9

RPM 4000

REDUCED SPECIFIC VISCOSITY

0.2

0.1

0.0

0.50

1.0

COCENTRATION

Fig. 10

RPM 5000

REDUCED SPECIFIC VISCOSITY

COCENTRATION

RPN 6000

Fig. 11

0.1 0.2 0.3 0.4

0.5

1.0

Fig. 12

RPM 7000

squares to the Inch

REDUCED SPECIFIC VISCOSITY

COCENTRATION

Fig. 13
RPM 2000
MONOMER RATIO 2.4

REDUCED SPECIFIC VISCOSITY

COCENTRATION

0.4

0.3

0.2

0.1

0.5

1.0

Fig.14

RPM 3000

REDUCED SPECIFIC VISCOSITY

0.3

0.2

0.1

0.5

1.0

COCENTRATION

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

Fig. 15

R.P.M 4000

REDUCED SPECIFIC VISCOSITY

COCENTRATION

Fig. 16
R.P.M. 5000

Fig. 17

RPM   6000

MONOMER RATIO 1.2

REDUCED SPECIFI VISCOSITY
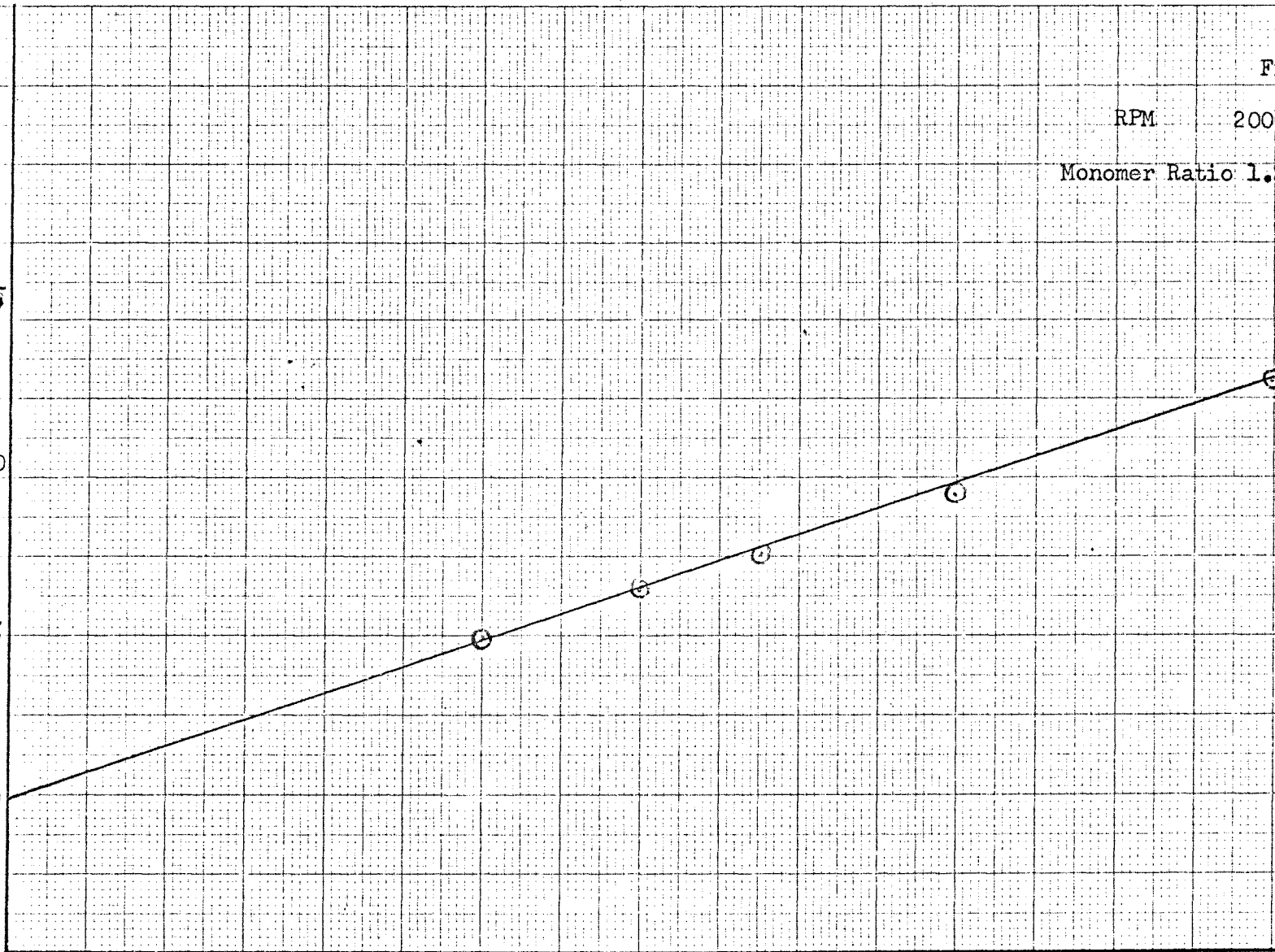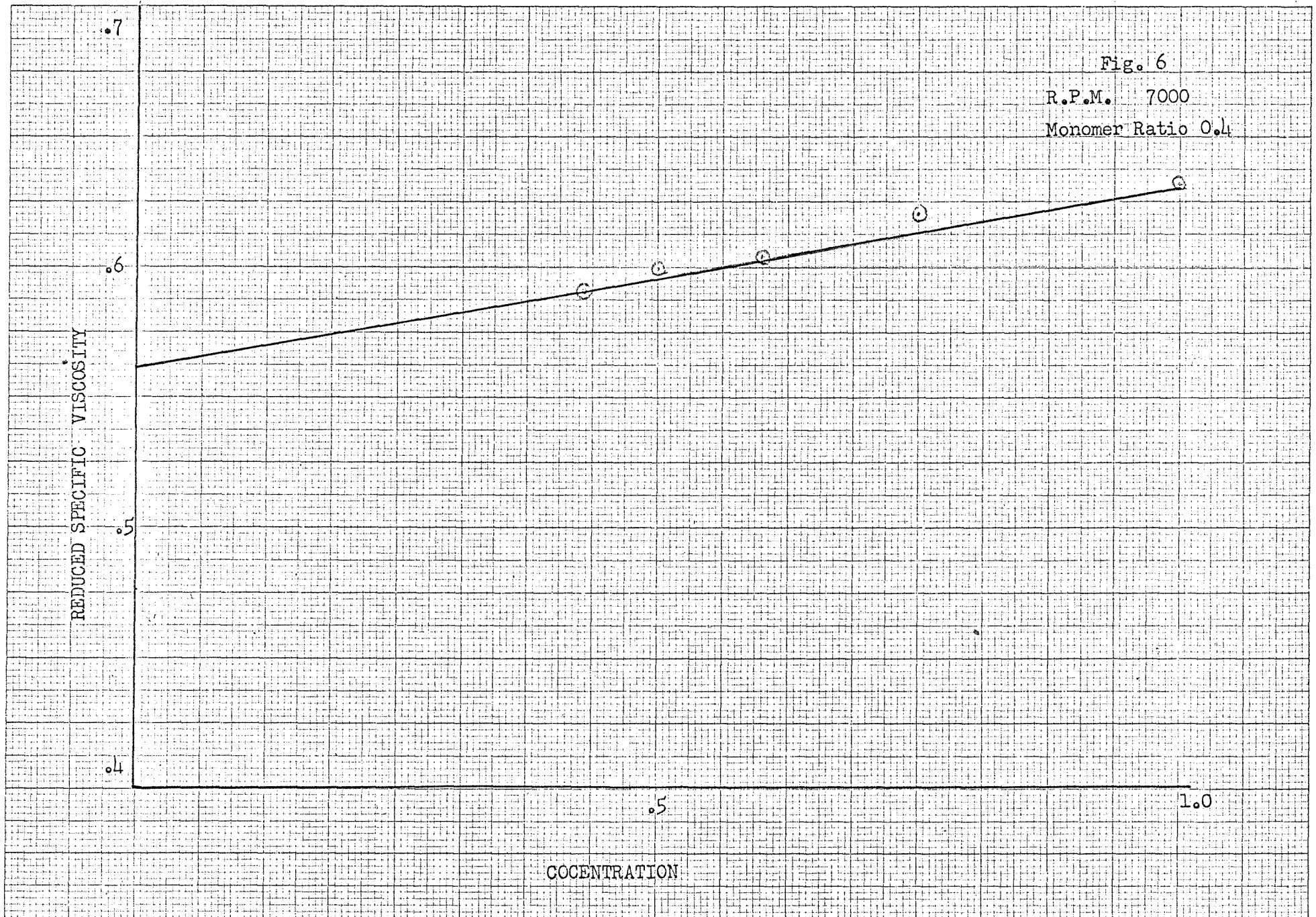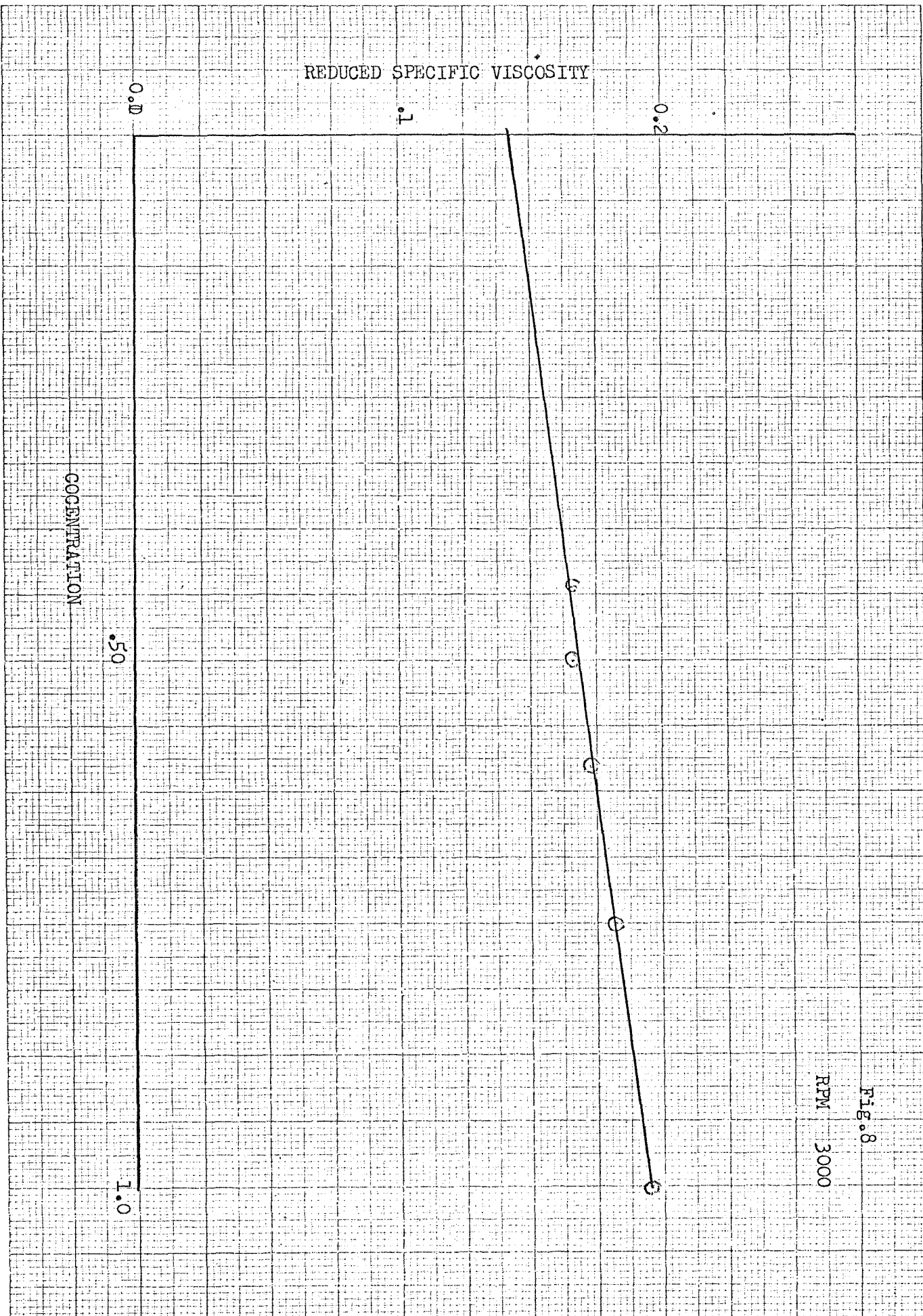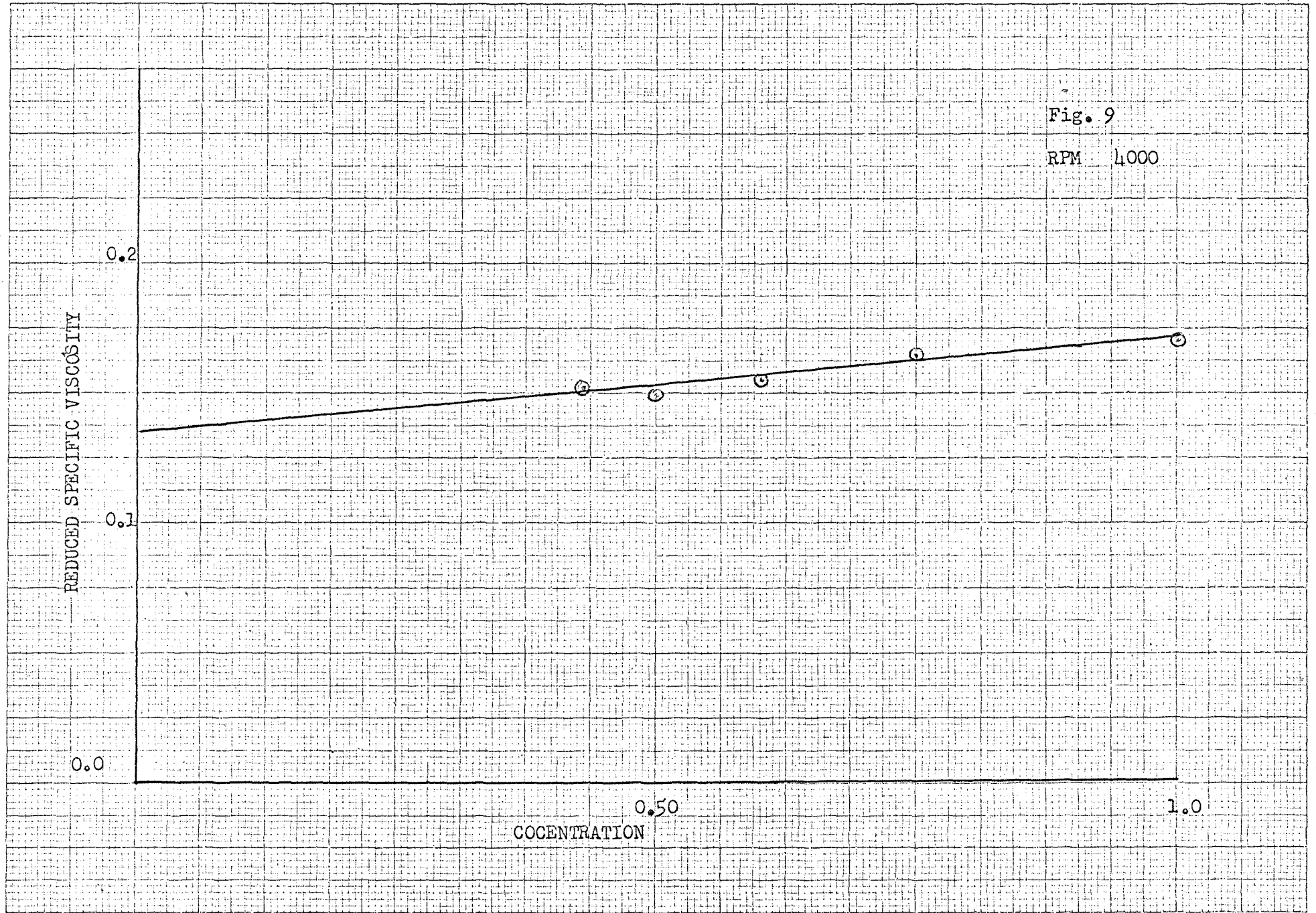
0.3

0.2

0.1

0.5

1.0

COCENTRATION

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

Fig. 18

R.P.M. 7000

FIG. 19

MONOMER RATIO 2.4

RPM 2000

REDUCED SPECIFIC VISCOSITY

COCENTRATION

Fig. 20

RPM 3000

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

Fig. 21

RPM 4000

Squares to the Inch

REDUCED SPECIFIC VISCOSITY

0.2

0.1

0.0

0.5

1.0

CONCENTRATION

Fig. 22

RPM 5000

REDUCED SPECIFIC VISCOSITY

CONCENTRATION

0.2

0.1

0.0

0.5

1.0

REDUCED SPECIFIC VISCOSITY

0.0

0.1

0.2

CONCENTRATION

0.5
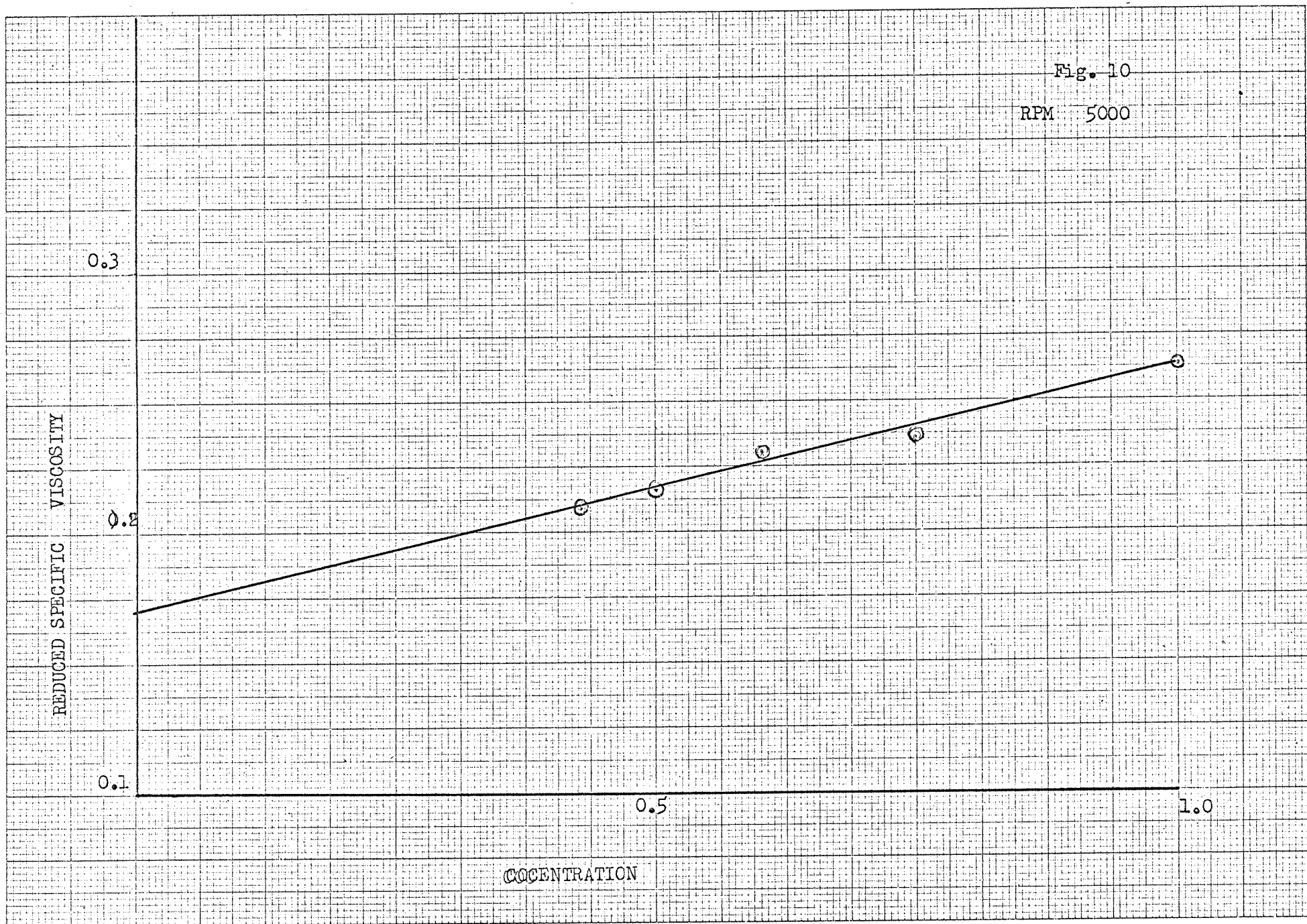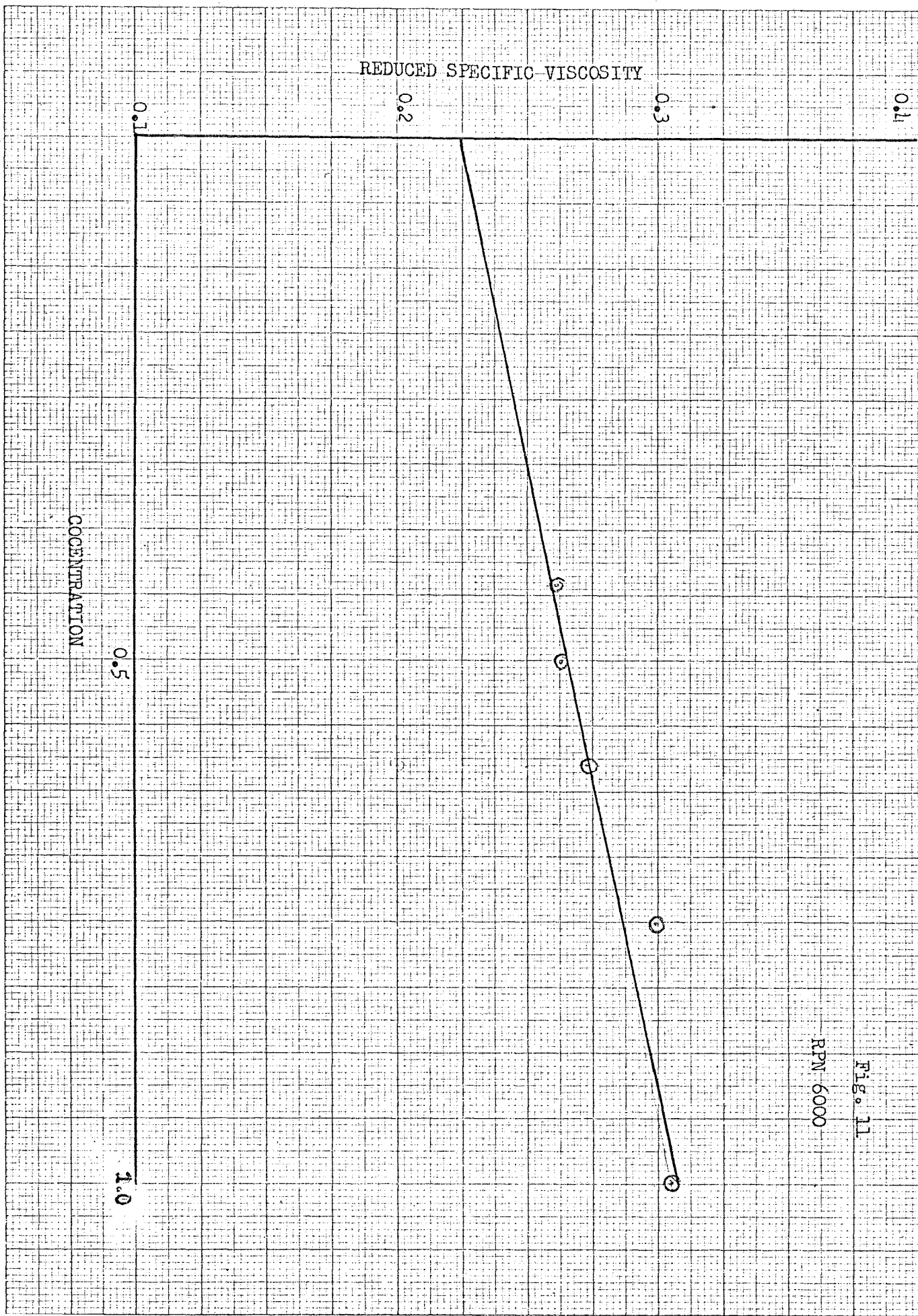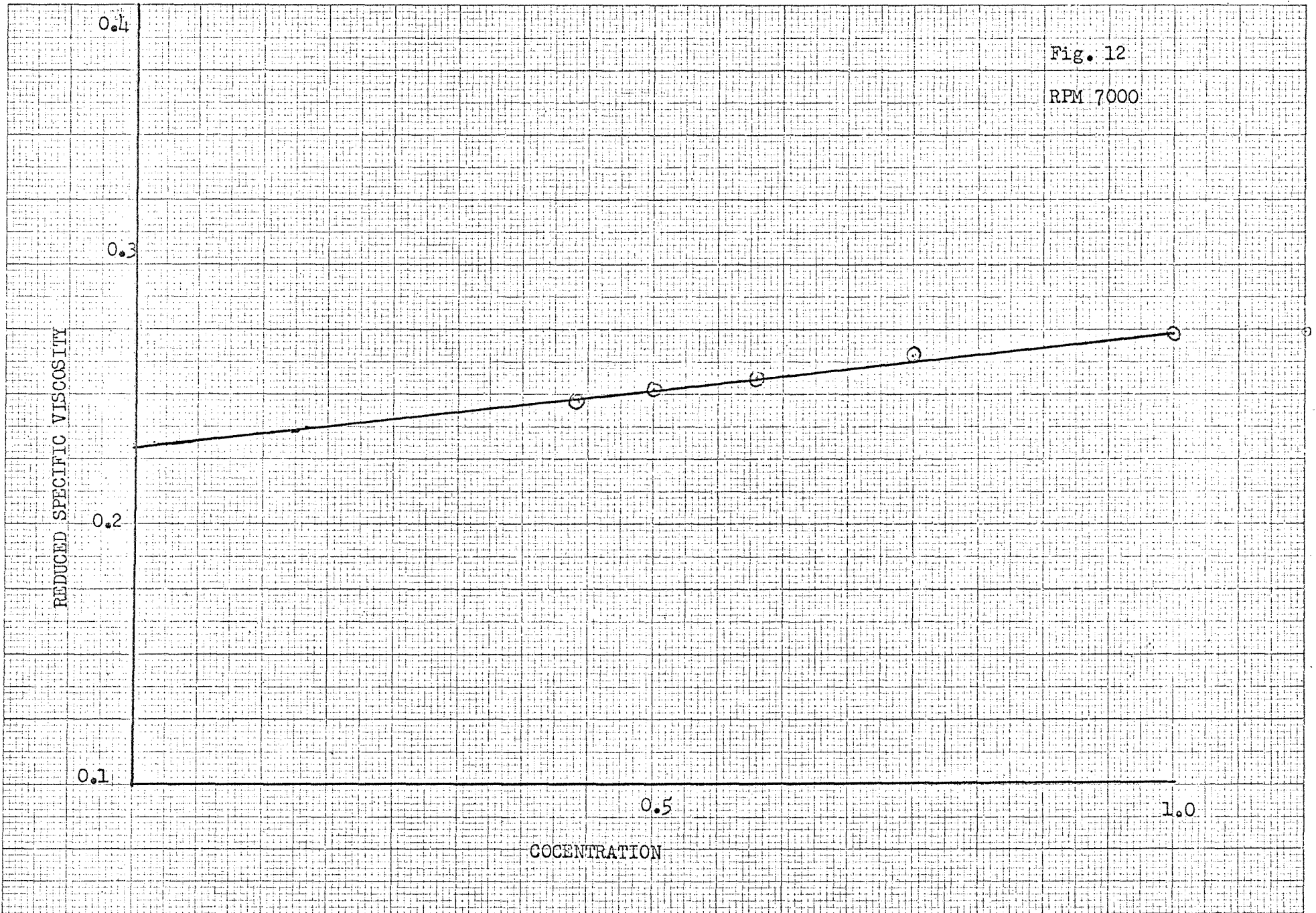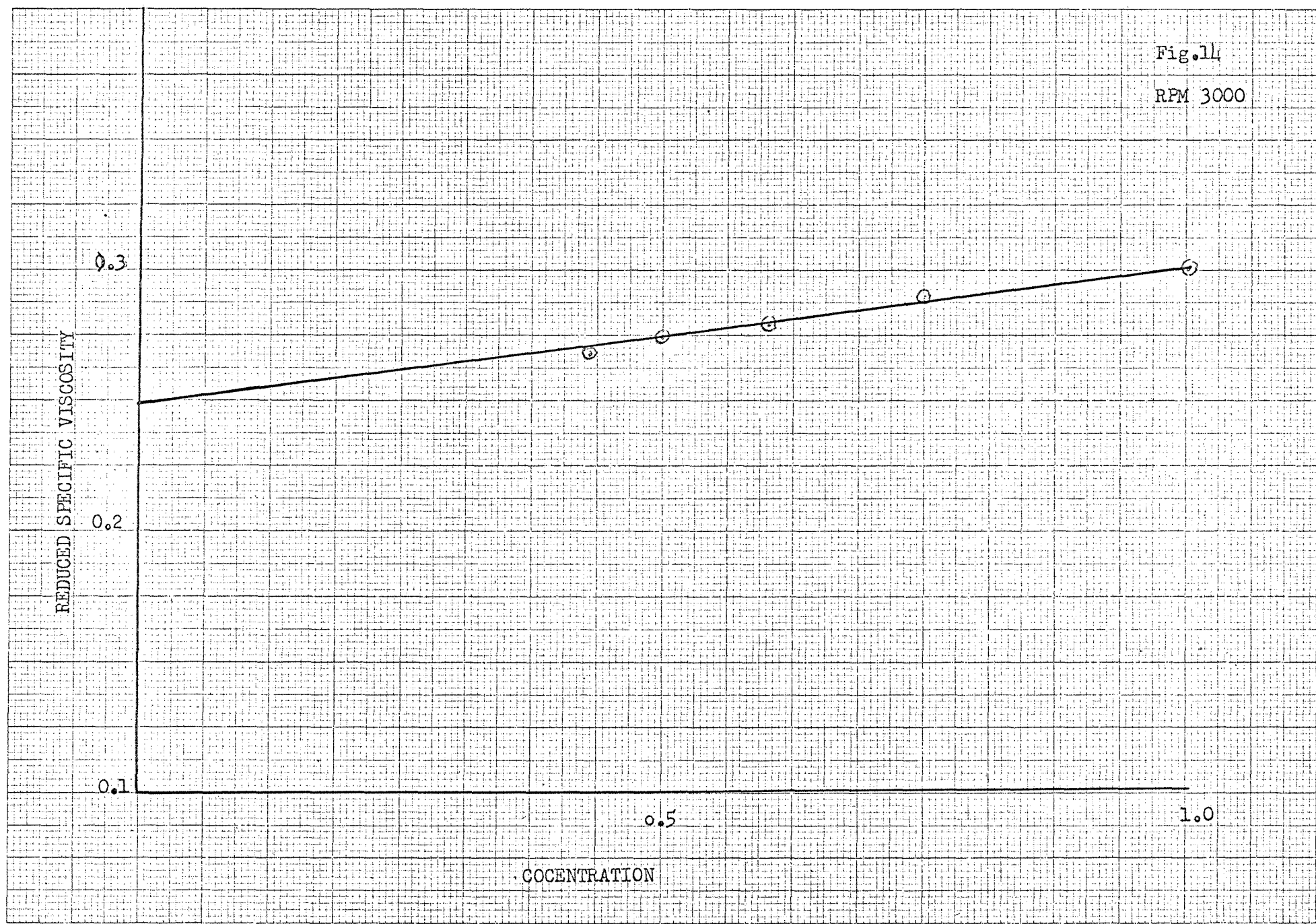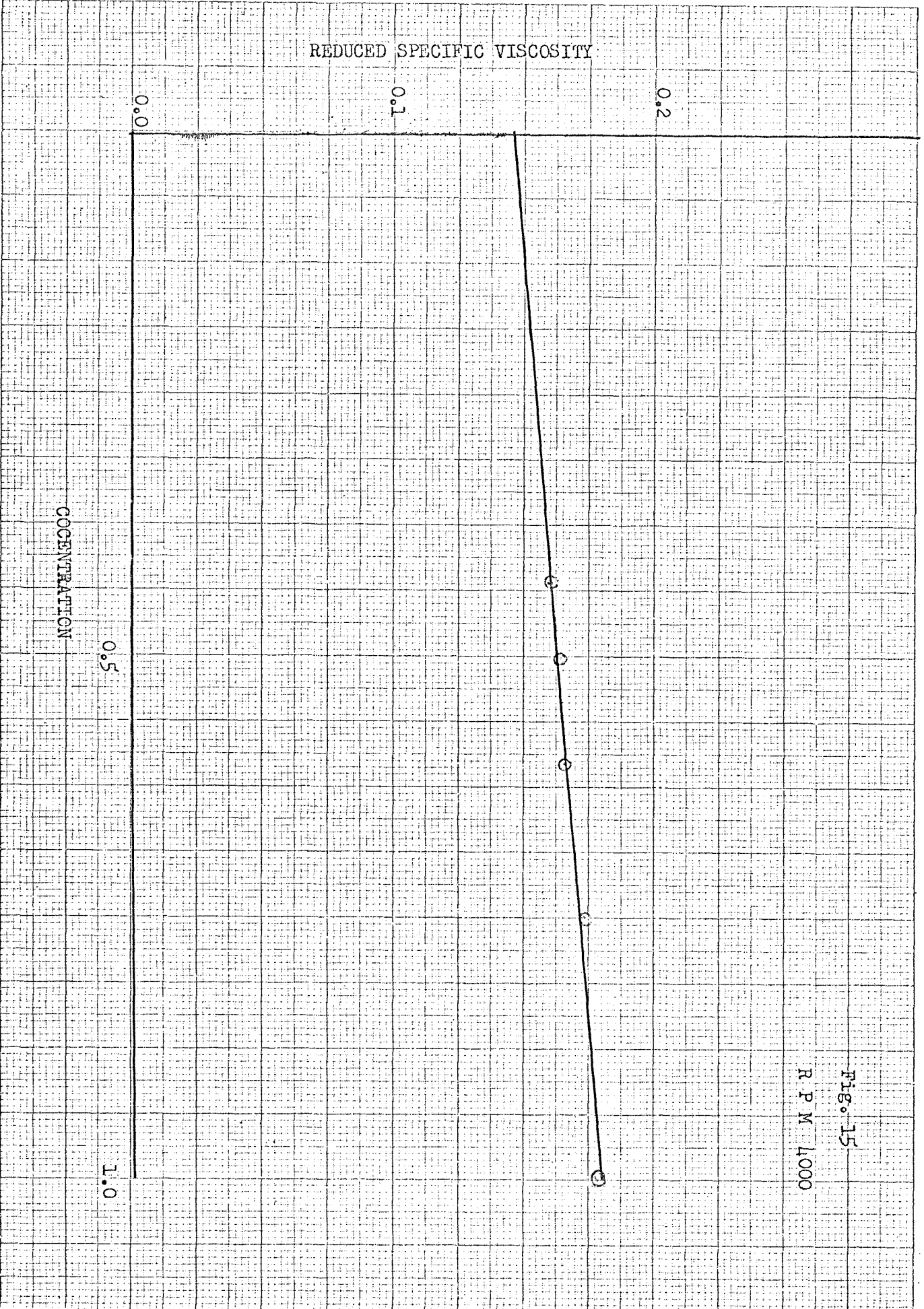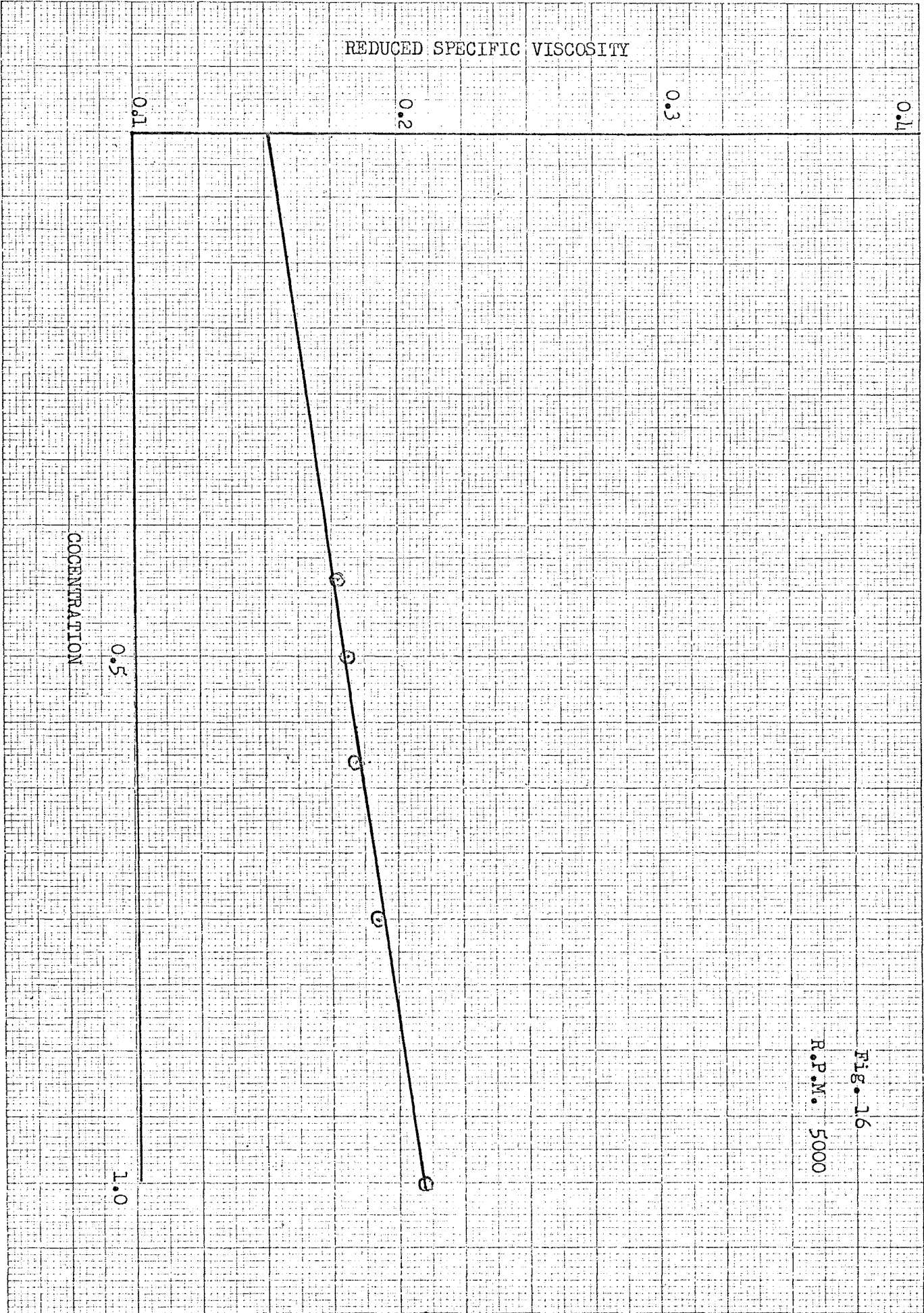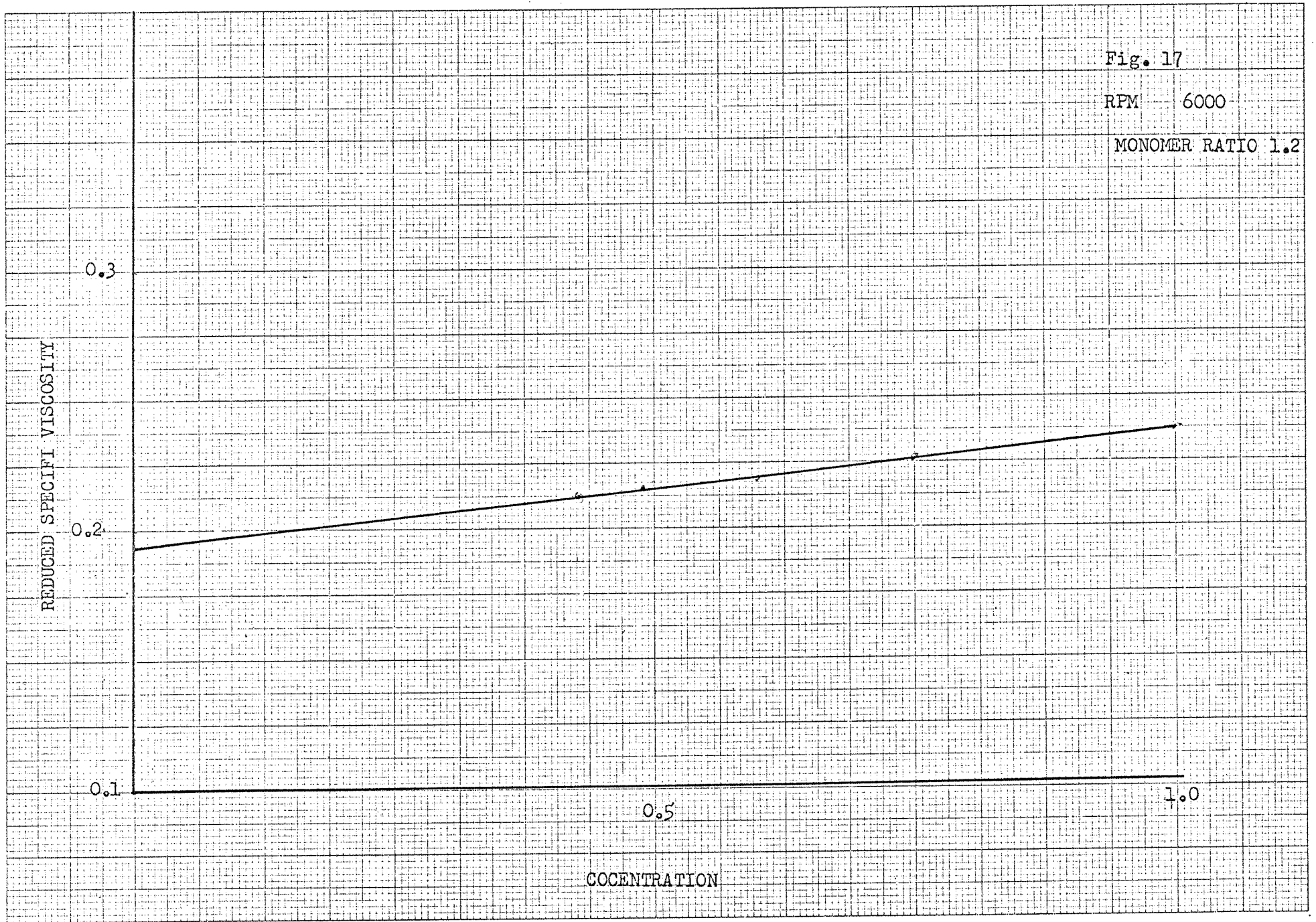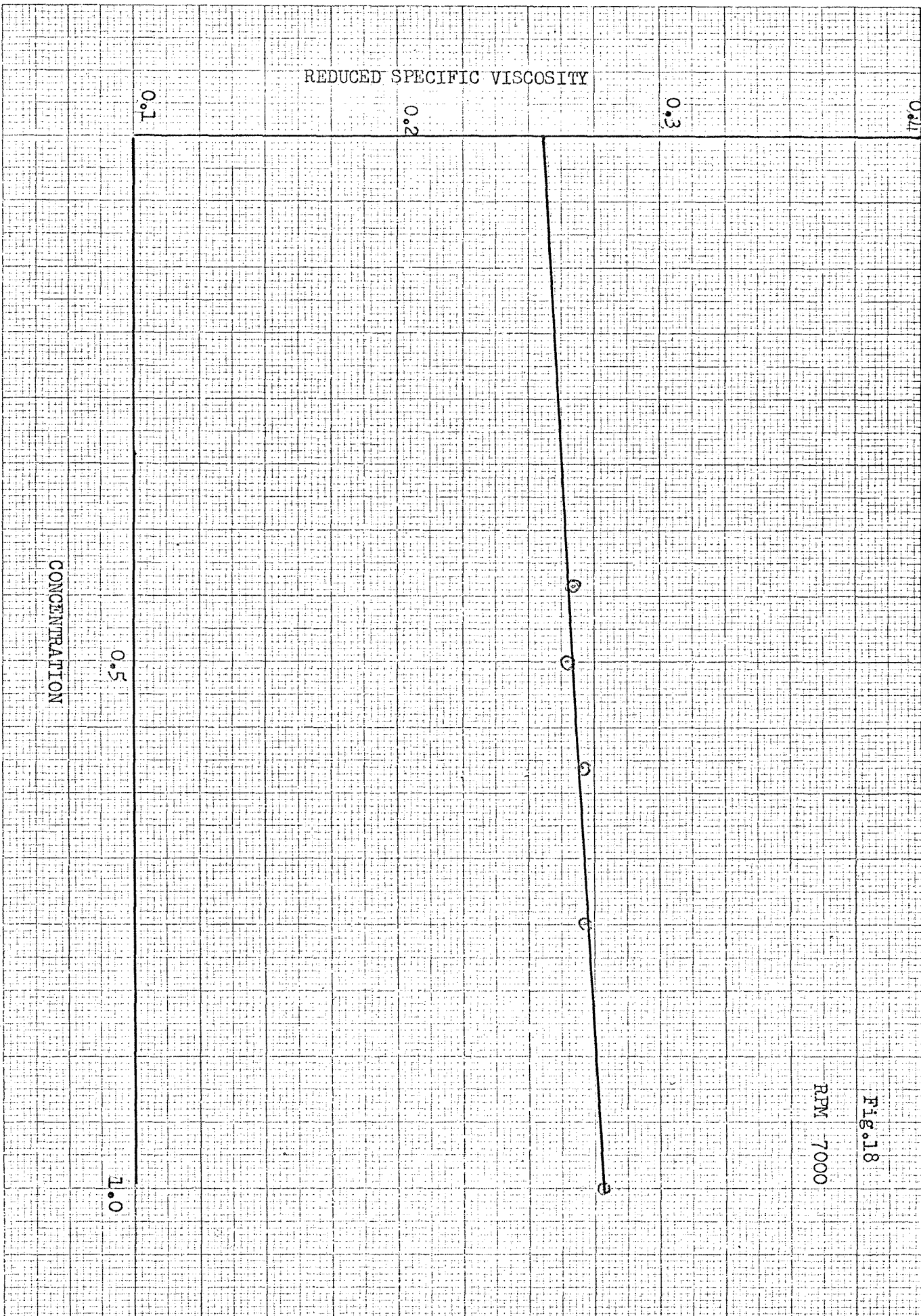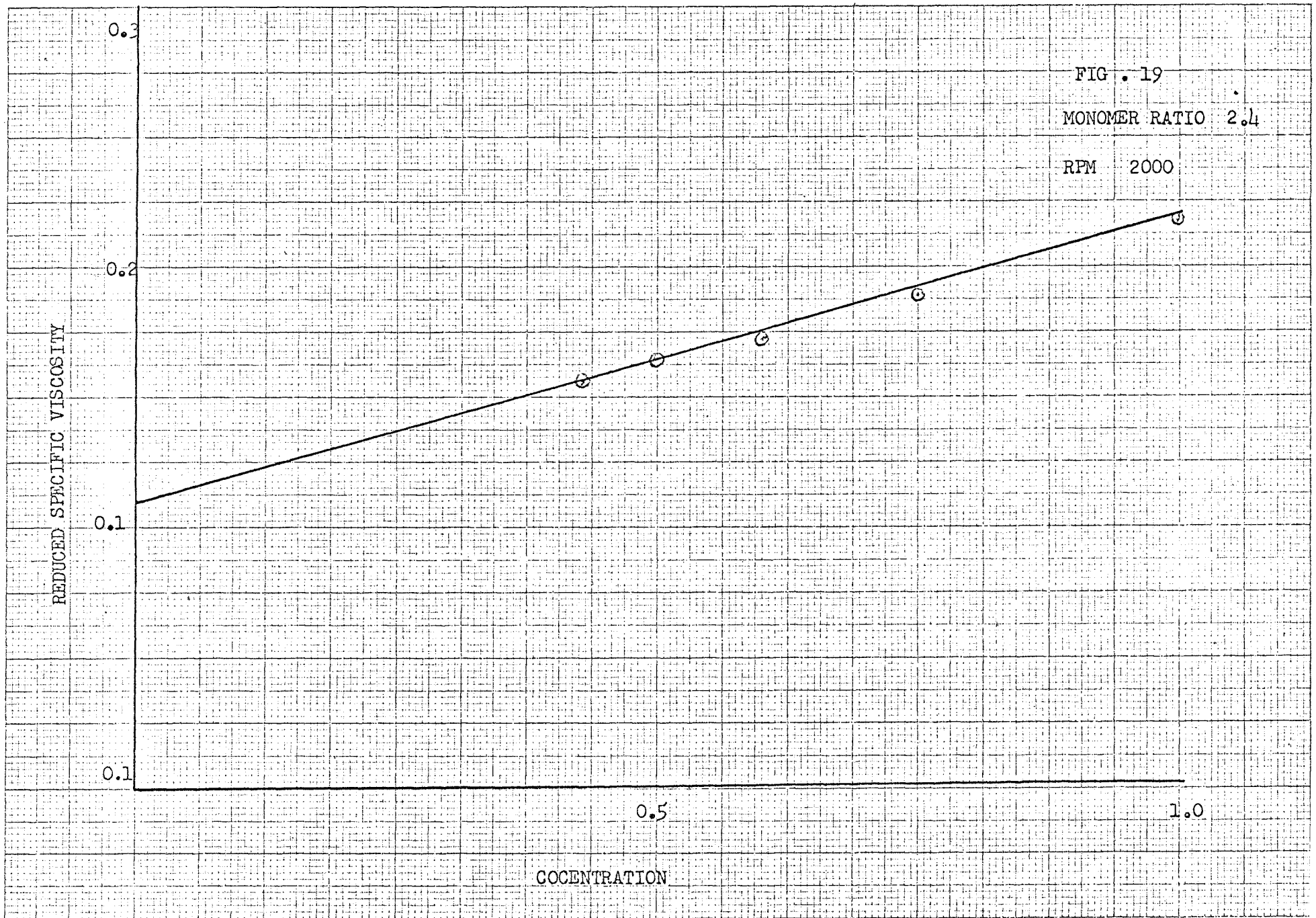
1.0

0.0
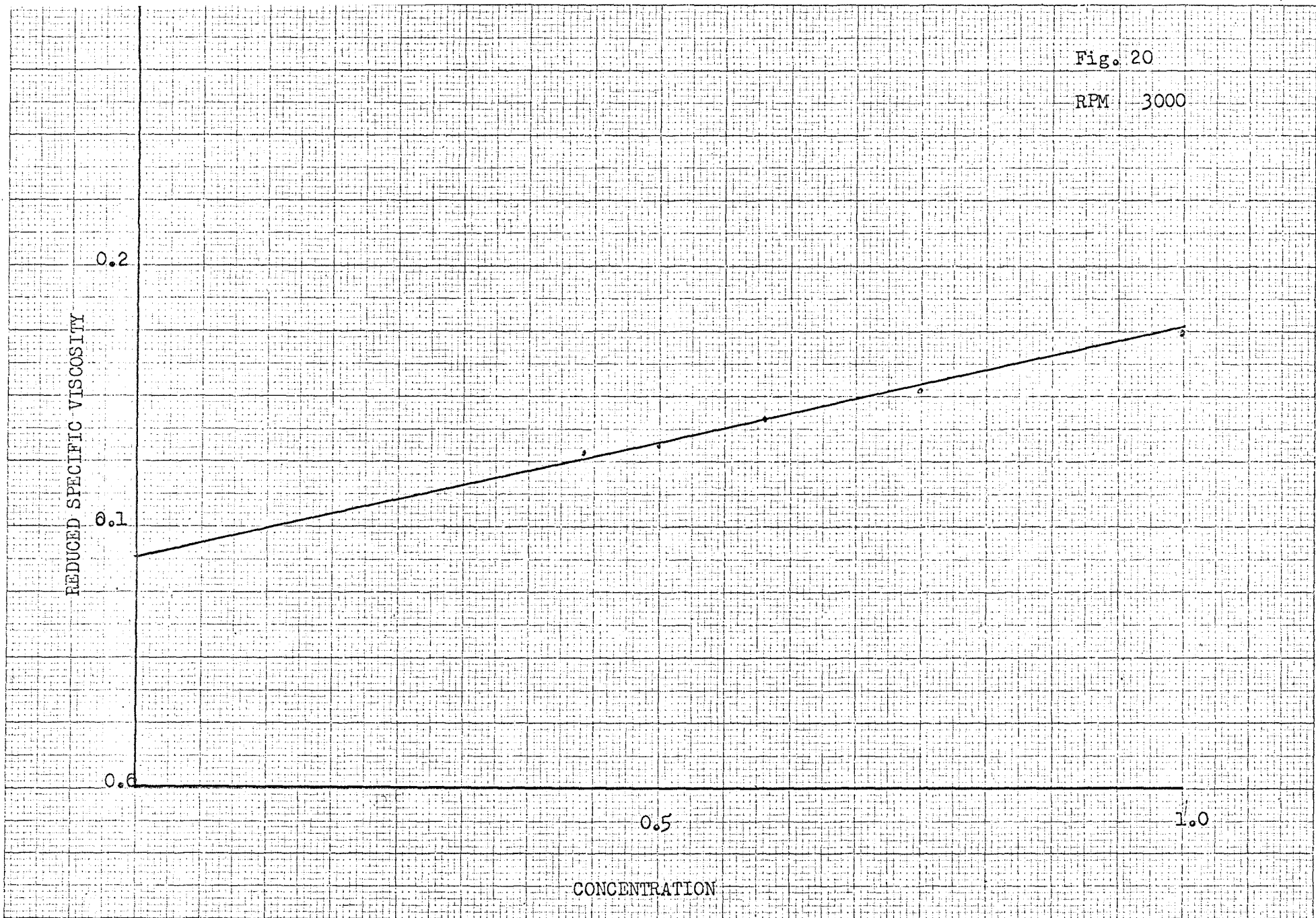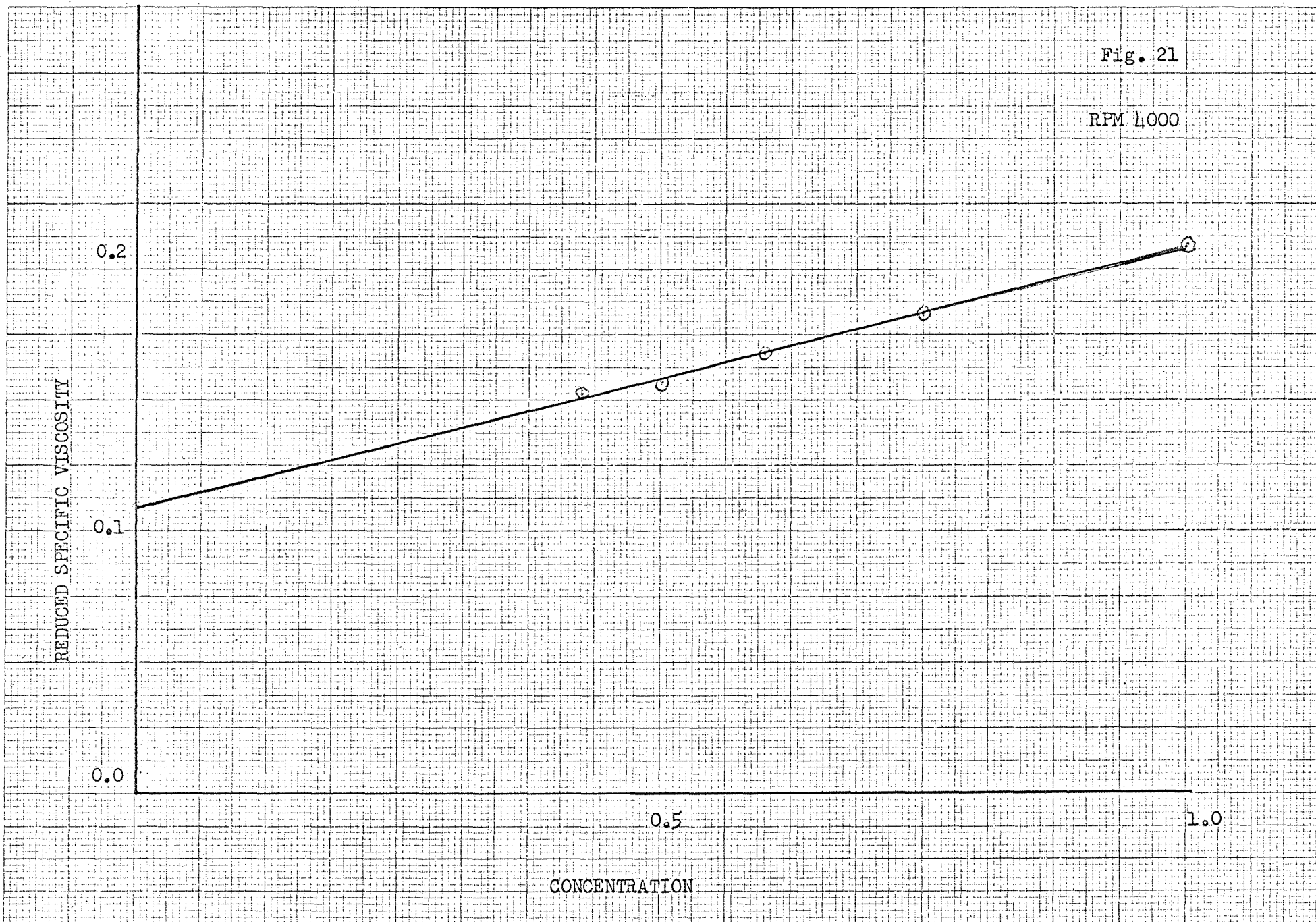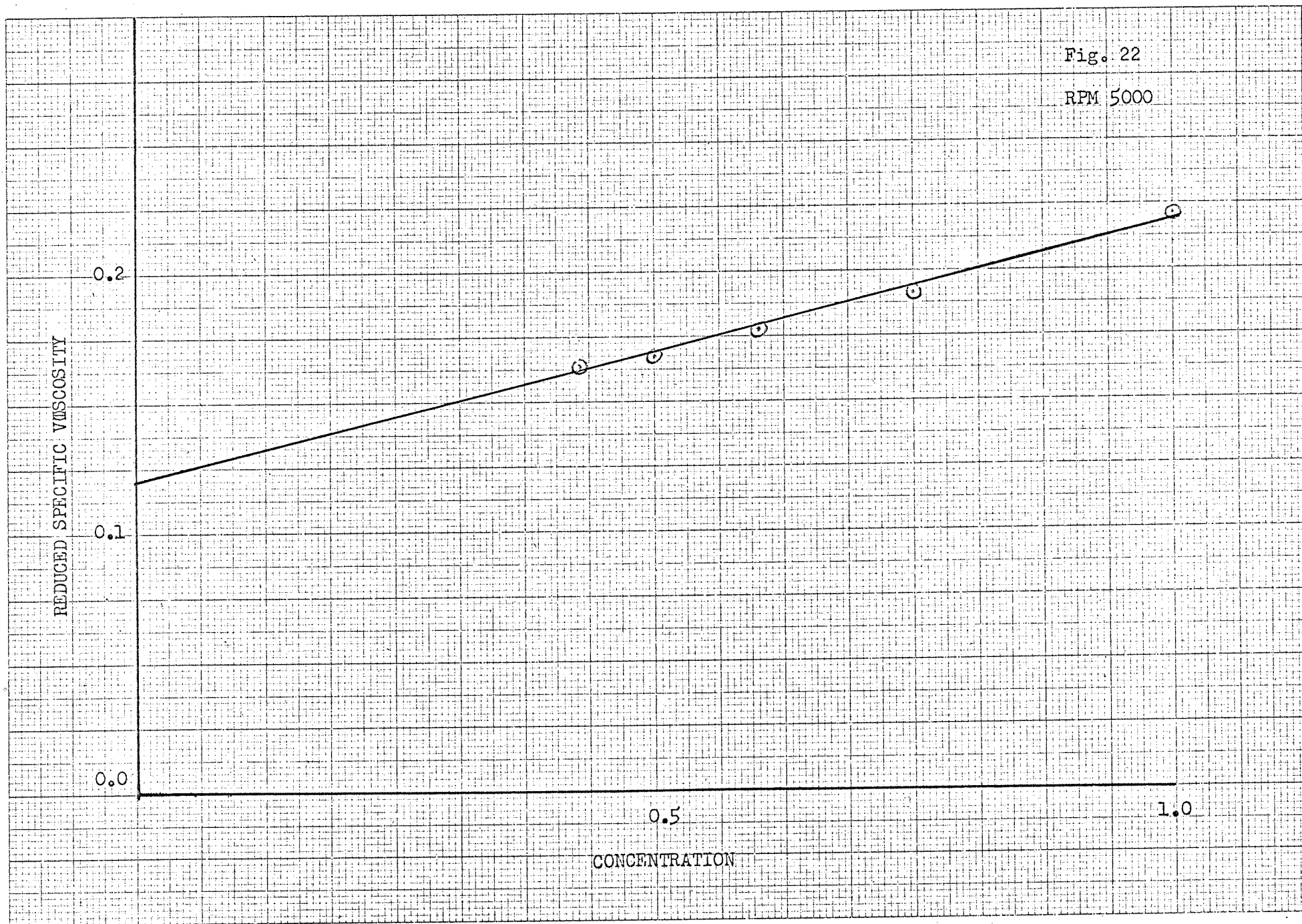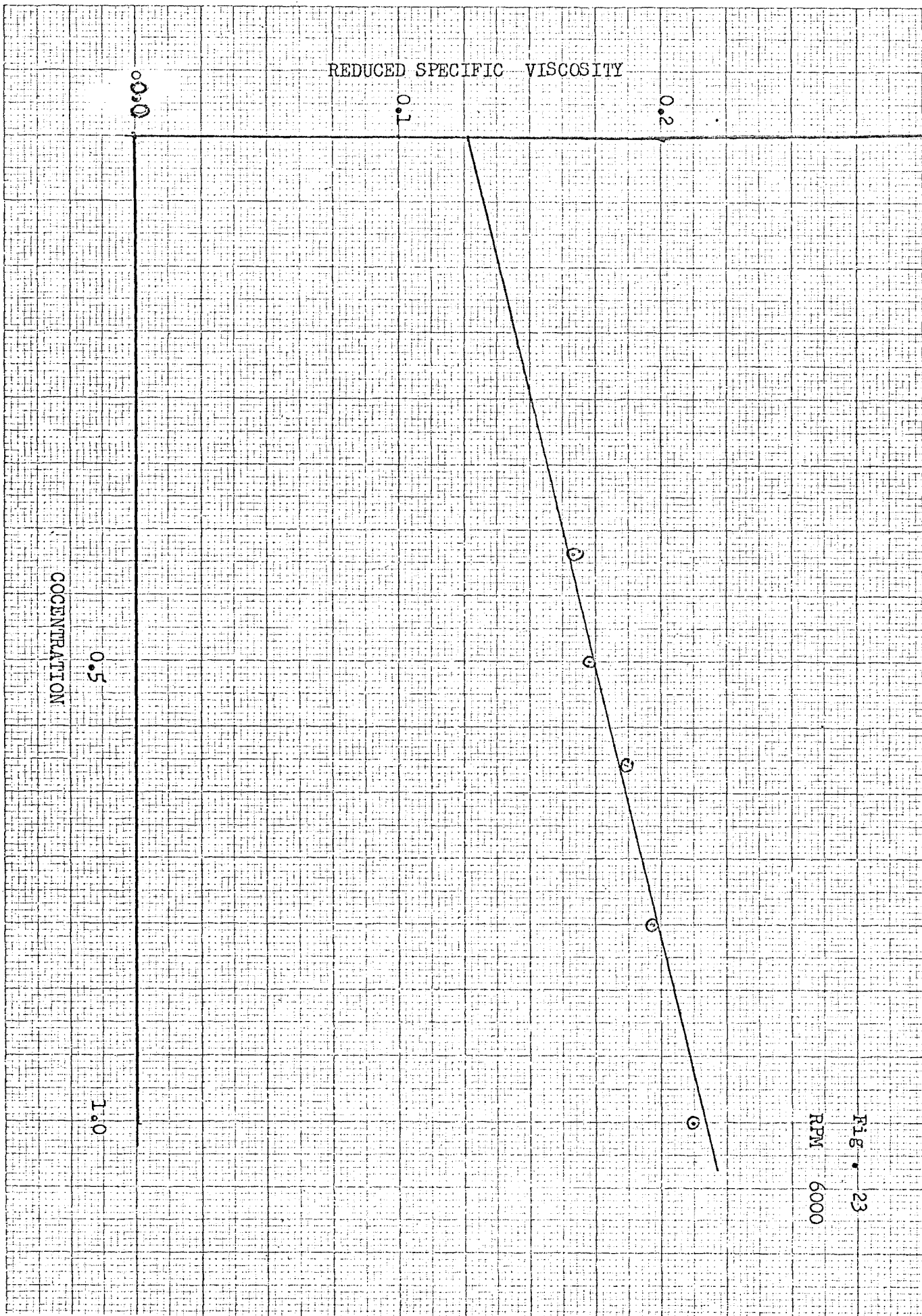
Fig. 23

RPM 6000

Fig. 24

RPM 7000

REDUCED SPECIFI VISCOSITY

0.2

0.1

0.0

0. 0.5 1.0

Fig. 25

Fig. 27

Fig. 28

MONOMER RATIO

⊙   2.4
·   4.25

Fig. 29

MONOMER RATIO

Fig. 30

MONOMER RATIO

2.4

4.25

Fig. 31

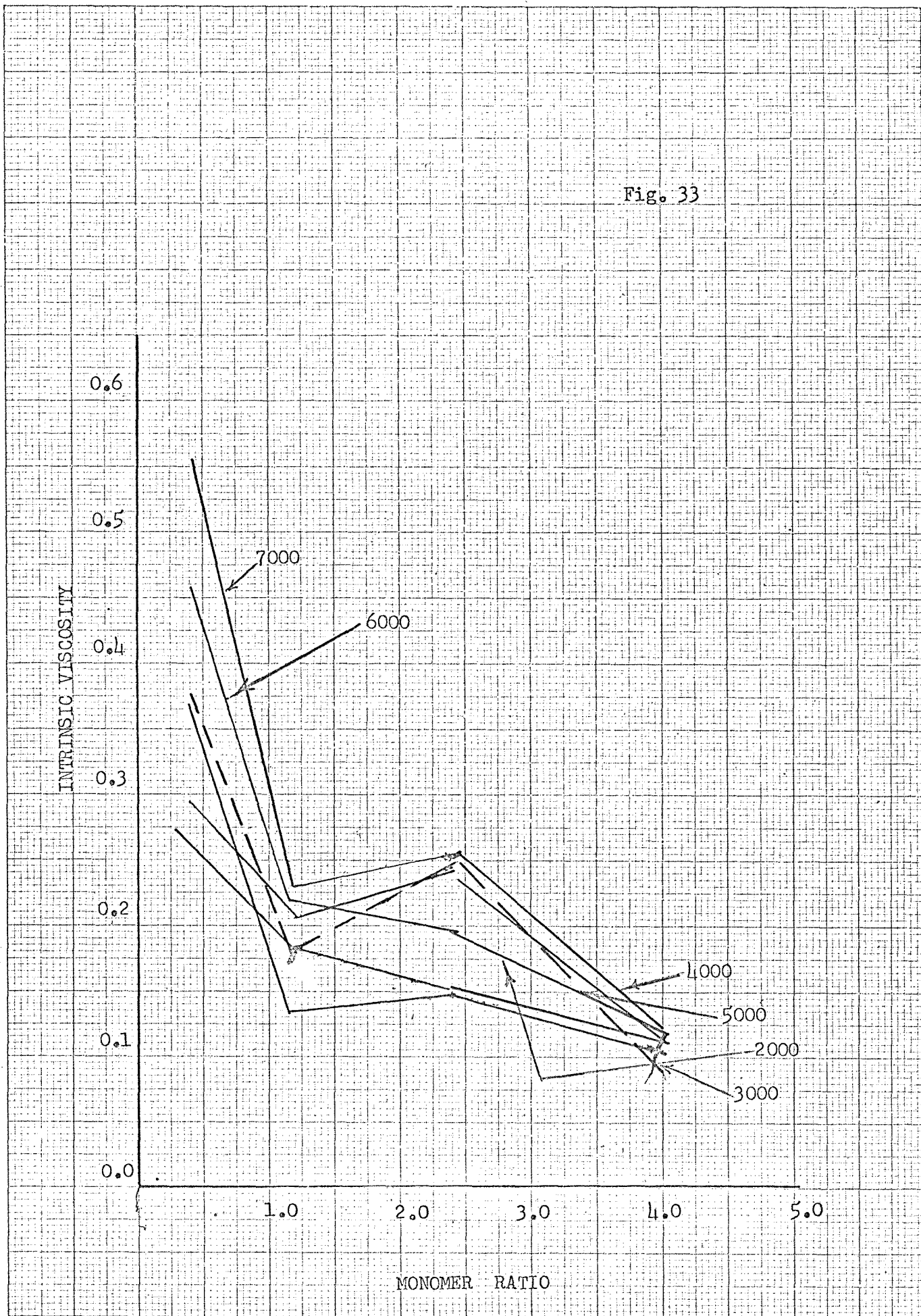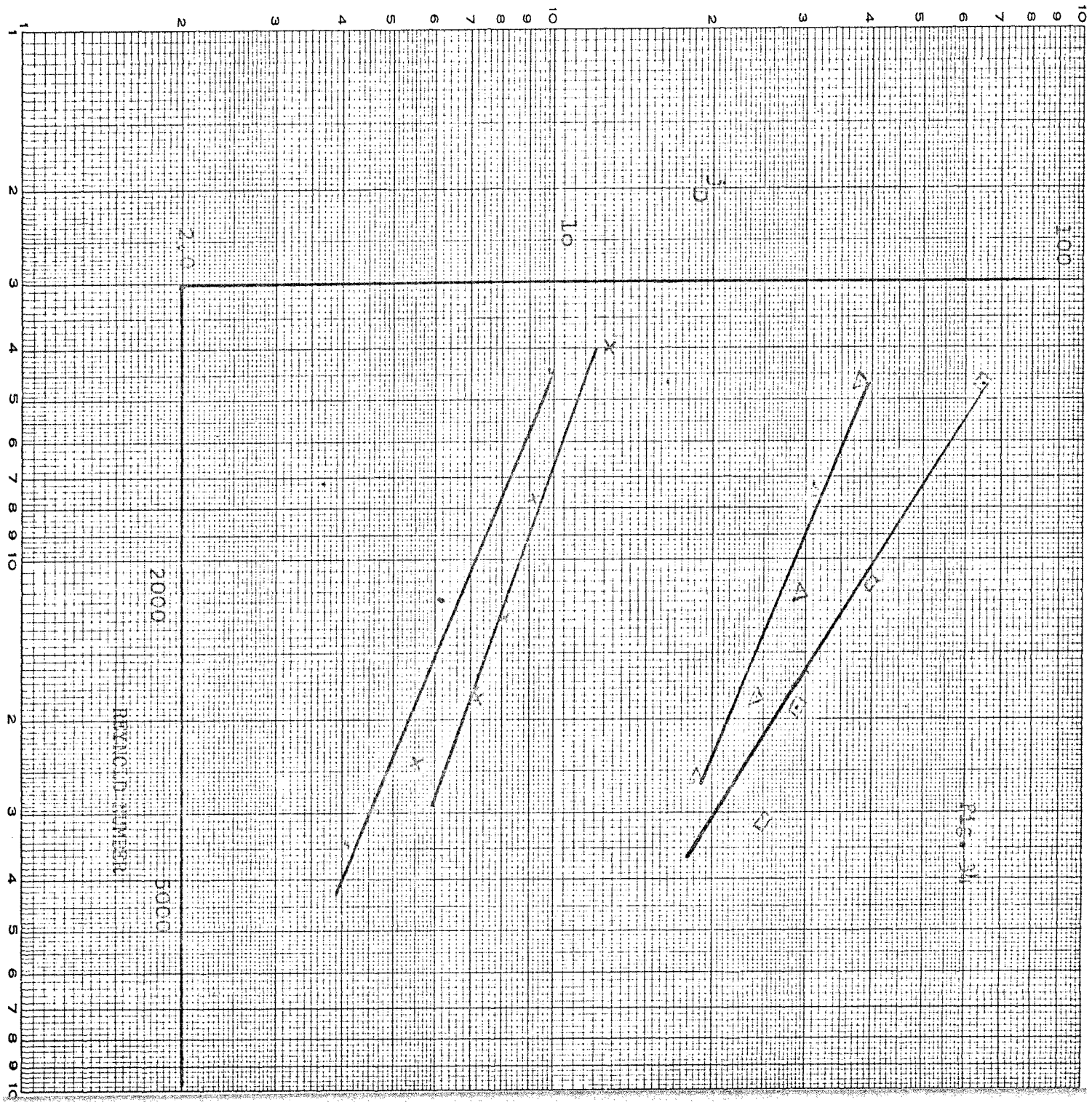MOMMER RATIO

0.4

1.2

REYNOLD NUMBER

Fig. 32
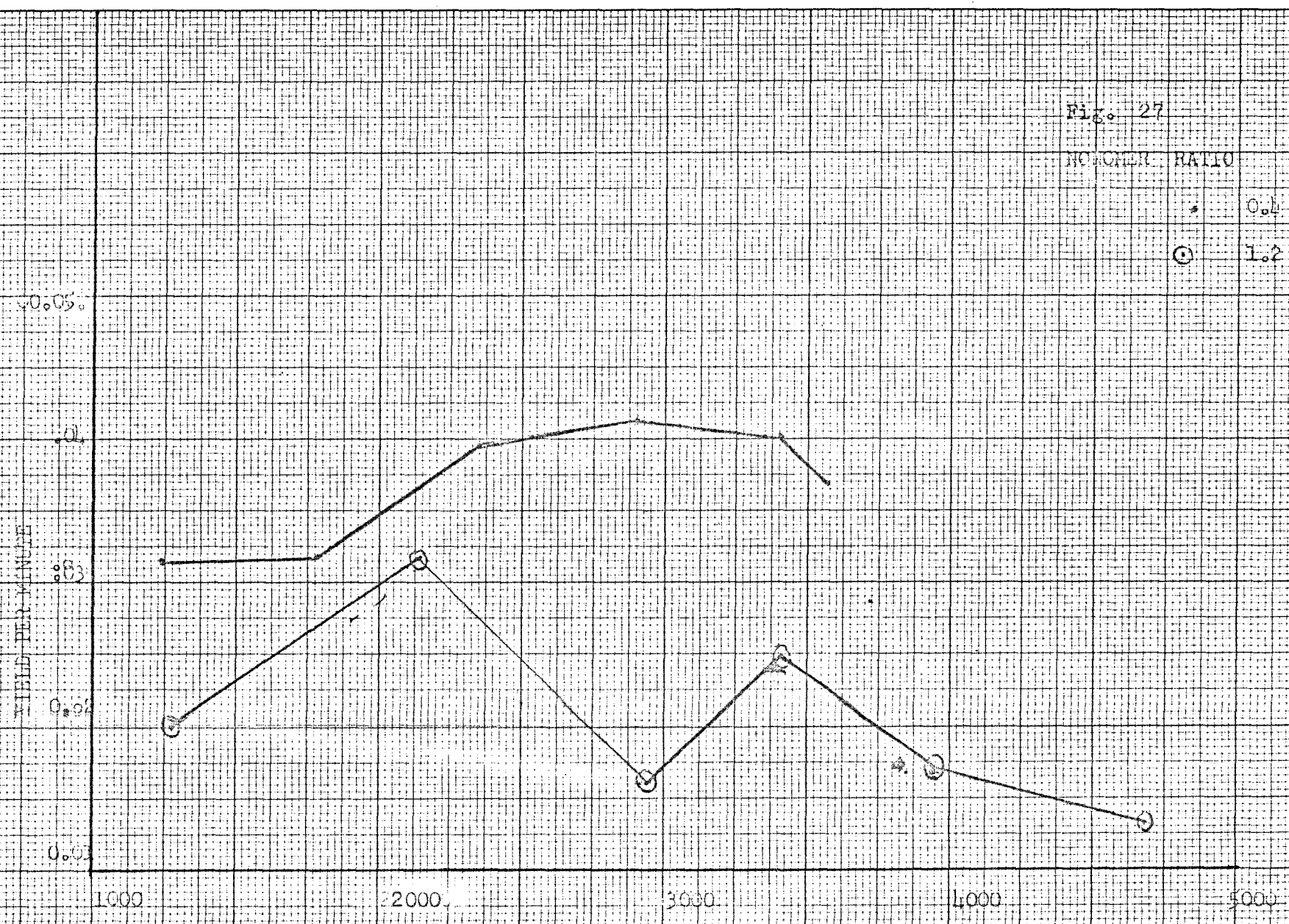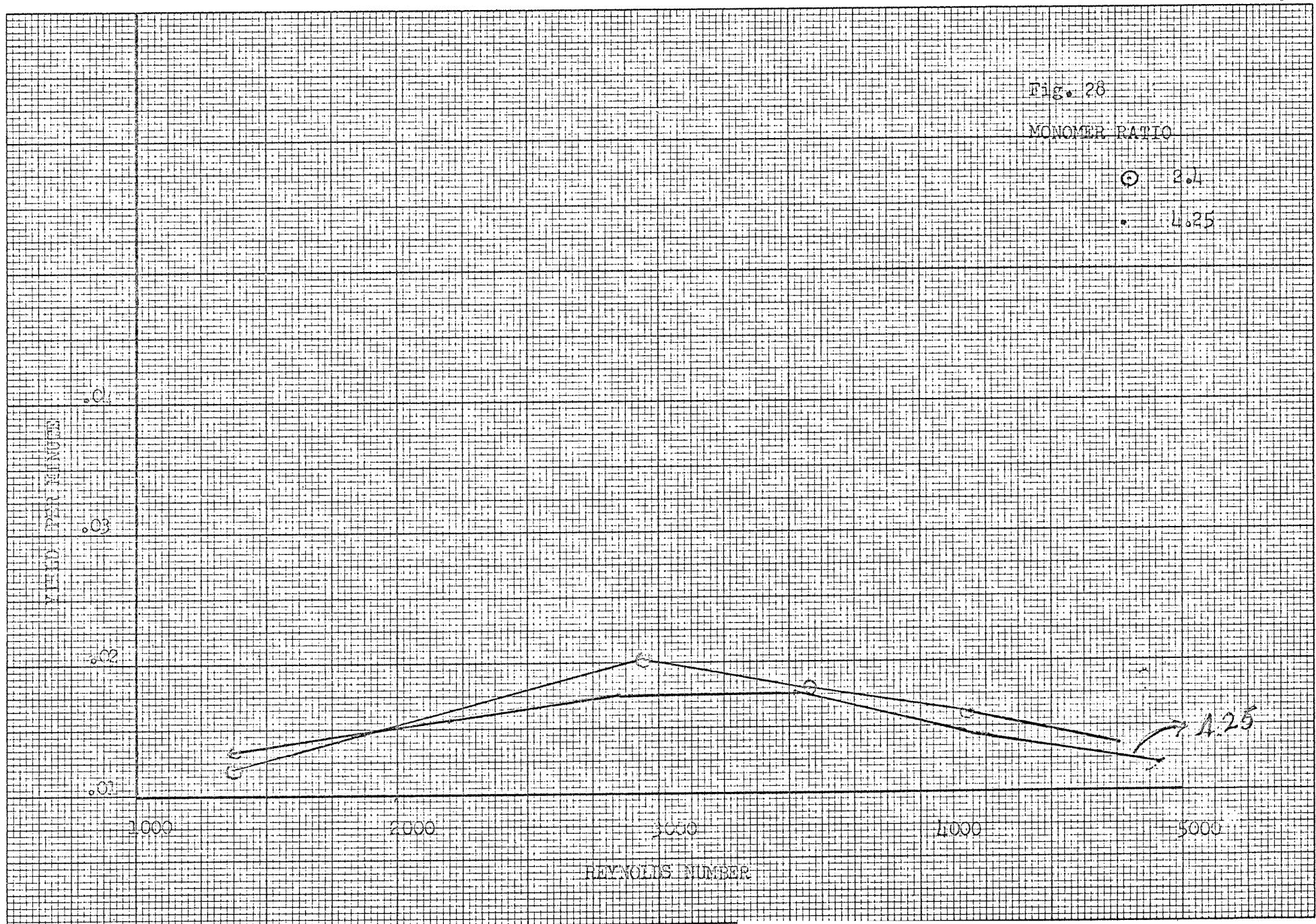
INTRINSIC VISCOSITY

REYNOLDS NUMBER

MONOMER/RATIO

Fig. 33

SAMPLE CALCULATION:

REYNOLDS NUMBER:

$$R_e = (d^2/v) \; N$$

d = Diameter of Stirrer 0.9595

v = Kinamatic Viscosity

= Relative viscosity of polymer x Viscosity

of solvent/ Density of Slurry

$$= \frac{1.12194 \; x \; .8414}{.79862} = \frac{.0140485}{.79862} = .013387$$

N = R.P.M. = 2000

$$R_e = 1291.59$$

Calculation For J_D Factor:

$$J_D = (K/CN) \; (D_{AB})^{.66}$$

K= mass transfer coefficient cm/hr = .0001343

C=Concentration of hexamethylene diamine = .002905

N= R.P.M. = 2000

= viscosity cp. = 1.2017

= density = .79862

$$J_D = \frac{(.0001343)}{(.002905 \; x \; 2000)} \quad X \quad (\underline{6}.957)$$
$$= .0013$$