FUEL RESEARCH INSTITUTE OF SOUTH AFRICA

TECHNICAL MEMORANDUM NO. 23/1958

A CAPILLARY TYPE VISCOMETER FOR TESTING INDUSTRIAL SUSPENSIONS

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INTRODUCTION:

Having modified the Stormer Viscometer to make it suitable for studying unstable heavy medium suspensions[1,2,3] as used in practice, and having found a suitable technique[4] to evaluate the results obtained in terms of absolute viscosity units, it appeared desirable to have available an alternative method of determining the viscosity of such suspensions so as to substantiate the conclusions reached.

Of the various alternatives a capillary viscometer appeared to hold out most promise. Designs already suggested have shortcomings when applied to unstable suspensions but it was hoped to obtain satisfactory operation by applying some of the techniques used with the modified Stormer viscometer.

A comparatively simple apparatus has been developed and experiments conducted in it suggested that it might have certain advantages over the modified Stormer Viscometer, for example

(1) It is simpler to operate and even inexperienced assistants should be able to produce satisfactory experimental results within a short time.

(2) This/.....

(2) This apparatus offers better possibilities than the Stormer viscosimeter of obtaining viscosity results in the laminar flow region.

(3) It provides better possibilities than the Stormer viscosimeter of studying flow characteristics at low shearing rates. (This region is of particular importance when considering the separation in coal washing, of the smaller near gravity particles in the feed).

(4) The technique for evaluating the results obtained in terms of absolute viscosity units appeared much simpler than that required for evaluating results obtained with the Stormer viscosimeter.

The main disadvantage of the apparatus is, however, that it requires quite a large volume of sample. This is not a serious disadvantage when the apparatus is used for the study or control of heavy medium suspensions used e.g. in coal washing practice.

DESCRIPTION OF THE APPARATUS:

The new viscometer is shown schematically in Figure 1. It consists essentially of:-

(a) a two litre bottle (A) for taking the sample to be studied.

(b) a set of capillaries (or tubes) of suitable diameter (B)

(c) a specially designed receiving vessel (C) consisting of a lower, a central and an upper bulb.

(d) a manometer (D) for measuring the applied pressure.

(e) a pressure regulator (E).

Compressed air/.....

Compressed air from a suitable source is admitted to the bottle (A) via the pressure regulator (E). The applied pressure is determined by the density of the liquid in E and by the depth of immersion of the bubbler tube (F). The liquid or suspension is forced from A up the capillary (B) by the applied pressure and the effluent is collected in the collecting vessel (C). The time taken to fill the central bulb of the collecting vessel is determined, timing marks being provided for this purpose.

A sample container of relatively large diameter should be selected to ensure that the variation in depth of immersion of the capillary (B) is negligible during an experiment.

CALIBRATION PROCEDURE:

The apparatus was calibrated using true liquids of known viscosity and density, and the results reported in Table 1. For this purpose Glycerine solutions were used. A fixed amount (one litre) of the liquid to be tested was placed in A and the latter then placed in a thermostatically controlled water bath until the liquid had attained a temperature of $25^{o}C$.

Pressure was then applied to the sample container and the time, t, to fill the central bulb of the collecting vessel was determined. (The lower bulb was provided to ensure that steady flow had been obtained before timing started). Results are given in Table 1.

This procedure was repeated for a number of applied pressures and a graph of $\frac{1}{t}$ against total applied pressure (P_T) was plotted. (Under laminar flow conditions,

this graph/.....

this graph will be a straight line and the initial pressure due to the height of liquid in the capillary can be determined readily by extrapolation).

The effective pressure, P, causing the flow was determined by deducting the initial pressure from the total applied pressure.

If the flow is laminar, results may be evaluated using the formula

$$\eta = \frac{Pt}{A} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where A is a constant for a given capillary.

Thus A can be found by conducting calibration experiments with liquids of known viscosity and subsequently this A value can be used to calculate the viscosity of an unknown liquid.

Before applying equation (1) to the calibration experiments, it was necessary to determine whether the flow was laminar and this was done by plotting curves of the resistance co-efficient against the Reynold's number.

The Reynold's number for a liquid flowing through a tube is given by the equation

$$Re = V.D.\rho./\eta \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where V = mean velocity of flow in the tube

D = diameter of the tube = 2r

ρ = density of the liquid

and η = its viscosity.

The velocity of flow $V = \frac{Q1}{t \pi r^2}$ .....(3)

where Q_1 = quantity flowing in time t.

Substituting/.....

Substituting the value of V given by (3) in equation (2) this equation becomes

$$Re = \frac{Q_1}{t\pi r^2} \times \frac{2r\rho}{\eta}$$

$$= \frac{2Q_1}{\pi} \times \frac{\rho}{rt\eta}$$

$$= Ca \cdot \frac{\rho}{rt\eta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (4)$$

The resistance co-efficient for a liquid flowing through a tube is given by the general equation (Fanning)

$$Q = \frac{Pr}{2L} \cdot \frac{1}{\rho V^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

where P = the effective Pressure, causing flow
and L = the length of the capillary (or tube).

Substituting for V from equation (3)

$$Q = \frac{P}{2L} \times \frac{t^2\pi^2r^4}{\rho Q_1^2}$$

$$= \frac{\pi^2}{2LQ_1^2} \times \frac{r^5t^2P}{\rho}$$

$$= Cb \cdot \frac{r^5t^2P}{\rho} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (6)$$

when the flow is laminar

$$Q \propto \frac{1}{Re^n} \qquad (\text{with } n = 1)$$

The values of Re and Q were calculated from the experimental results using equations 4 and 6 respectively, and Q was plotted against Re on log. paper. This Q - Re relationship was found to be a straight line with slope, n=1, which indicated that the flowwas laminar.

TEST ON/.....

TEST ON SUSPENSIONS:

Initial experiments were confined to stable suspensions which, according to results obtained from a study in the Stormer viscosimeter had Bingham characteristics.

It appeared possible that experiments conducted in the Capillary tube apparatus would be conducted under substantially lower shearing rates than those conducted in the Stormer viscosimeter. If the viscosity is a function of the rate of shear, viscosity values obtained by the two methods might therefore differ.

In order to obtain stable suspensions for the first series of experiments a narrow specific gravity fraction (1.3 - 1.4, averaging 1.356) was obtained from a coal sample and ground to minus 200 mesh B.S.S. This coal was then suspended in zinc chloride solution having a density of 1.356 grams/cc.

With such suspensions the effect of the solids concentration in the suspension and of the bore of the capillary tube were studied.

The results of these experiments are reported in Table 2 and curves of $\frac{1}{t}$ against total applied pressure are shown in Figure 2. The curves of viscosity of suspension as determined by applying equation 1, are plotted against the capillary diameter in Figure 3.

The viscosity of these suspensions was also determined in the Stormer Viscosimeter and the results obtained are reported in Table 3. These experimental results were evaluated in this case in accordance with a

procedure outlined/.....

procedure outlined in a previous paper[4], and it was found that all the suspensions had Bingham Characteristics. The corresponding differential viscosity values are reported in Table 3, and also in Table 2 for purposes of comparison.

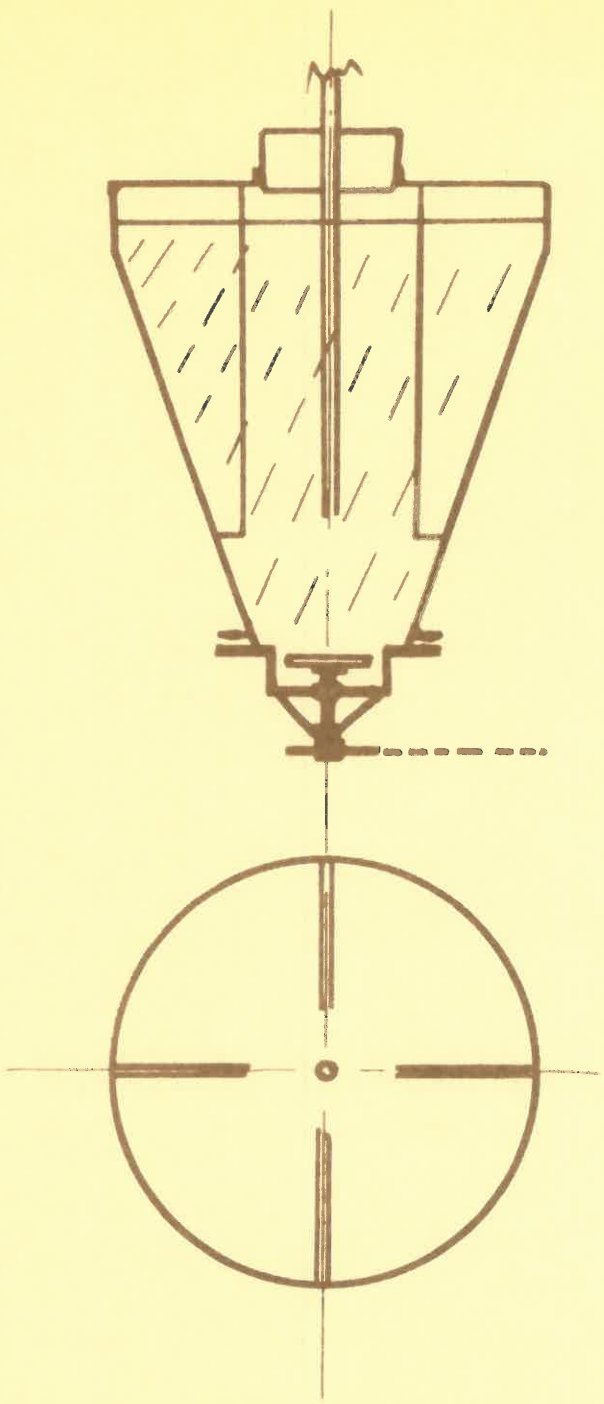
A study of Table 2 and Figure 3 reveals that the viscosity of a suspension as determined by the capillary method, at first increased with increasing diameter of capillary tube but reached a constant value after some critical diameter had been exceeded. When using a suspension having a solids concentration of 34.1 per cent by volume, the critical diameter was of the order of 5.6 mm. which represented a value of the ratio

$$\frac{\text{capillary diameter}}{\text{diameter of largest particle.}}$$ of about 70.

It is probable that the viscosity results are unreliable if the value of this ratio is smaller than the critical. Although sufficient results are not yet available to form definite conclusions, there is some indication that the critical value of this ratio is also a function of the solids concentration.

Provided the diameter of the capillary exceeds the critical value, the viscosity values obtained by this method agree very well with those obtained in the Stormer viscosimeter. The agreement is particularly remarkable if it is borne in mind that the capillary experiments were conducted under laminar flow conditions while the Stormer experiments were conducted under semi-turbulent conditions.

UNSTABLE SUSPENSIONS/.....



THE MODIFIED CONTAINER

FIGURE. 4.

UNSTABLE SUSPENSIONS:

In order to investigate the potentialities of the capillary method for testing unstable suspensions, the sample container was slightly modified and provided with an agitation arrangement similar to that developed for the Stormer viscosimeter. The impeller was redesigned to give a horizontal circulation instead of a vertical circulation. This modification is shown in Figure 4.

It was again necessary to investigate to what extent this modification would influence the test results.

Experiments were carried out at first with the impeller stationary and then in motion. The results obtained are reported in Table 4.

It was found that the value of the constant A increased with increasing impeller speed. This resulted in an apparent increase in the viscosity value. This apparent increase in viscosity was found to be approximately 2 per cent at 250 revolutions per minute.

There was also a slight increase in the value of the total applied pressure, which could probably be ascribed to the variation in liquid level, due to agitation.

The instrument was calibrated at 250 r.p.m. in the manner already described.

A suspension of Baum refuse ground to minus 200 mesh, suspended in water was now studied in both the modified capillary apparatus and in the Stormer viscosimeter. The results of these experiments are reported in Table 5.

It will be/.....

It will be observed that the viscosity values obtained by the two methods agree reasonably well.

In order to investigate the influence of solids concentration and capillary diameter on viscosity it was decided to extend this study by carrying out a number of experiments on different suspensions.

The results obtained were now evaluated in accordance with the procedure previously outlined.

It was found however, that the results could not be correlated with the same accuracy as previously obtained with a stable suspension.

It was therefore concluded that the procedure adopted for evaluating the results (by applying equation 1) was not entirely valid.

A literature survey revealed that the flow of non-Newtonian fluids through capillary tubes is much more complicated than originally anticipated.

Although Bingham[5] applied equation (1) directly to his capillary flow experiments in the form

$$\phi = k\,(P-P_o) \qquad \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

Buckingham[6] states that this equation is only a very rough approximation and does not fit the observations well. The correct procedure according to Buckingham is to introduce it into the appropriate differential equation and to intigrate in the usual way.

Under these conditions he obtained the following modified Poiseiulle formula in which the mobility φ is given by

$$\frac{1}{\varphi} = \frac{\pi r^4}{8l\phi}\left(P - \frac{4}{3}p + \frac{p^4}{3P^3}\right) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

where p is the pressure corresponding to the yield value i.e. $So = \frac{pr}{2l}$.

Over the/.....

Over the region of the tube in which $\frac{pr}{2l}$ is less than So, there is no shear and consequently the material moves as a solid plug. When P>p the last term of equation (8) may be neglected and since shearing stress at the tube wass $S = \frac{Pr}{2l}$;

$$\frac{1}{\eta} = \frac{\pi r^3}{4\phi}\left(S - \frac{4}{3}So\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

This curve is of the general form of the Bingham type but the extrapolated intercept is 4/3 So, So being the true intercept.

The results previously obtained were now evaluated along these lines and although the correlation was improved, it was still far from satisfactory.

The findings suggested that a rather more detailed study of the flow characteristics in capillary tubes would have to be undertaken in order to interpret experimental results better. Pressure of other work made such a study impossible at the time and therefore the work was stopped at this stage.

PRETORIA (Sgd) A.M. FOURIE.

4th November, 1958.

/JAZ.

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TABLE 1.

RESULTS OF TESTS ON TRUE LIQUIDS (CAPILLARY METHOD)
IMPELLER STATIONARY.

Length of Capillary (cm)	Capillary Diameter (m.m.)	Effective Pressure P	Time for 78cc (t) (secs)	Viscosity (η) (Poises)	Constant A
37.95	1.45	17.3	1425	0.156	283,000
		28.7	857		
		35.5	708.4		
		41.4	620.1		
37.95	3.06	4.05	307.1	0.156	13,900
		10.7	115.2		
		15.9	77.3		
		21.7	56.7		
		27.8	44.5		
		34.6	35.9		
		41.9	29.4		
37.95	4.7	3.7	108.5	0.156	2,590
		8.6	46.9		
		13.7	29.5		
		19.2	21.05		
		24.8	16.4		
37.95	4.7	3.8	275.7	0.404	2,600
		8.6	122.3		
		13.6	77.3		
		17.5	59.7		
		21.4	48.2		
37.95	6.96	1.9	42.3	0.156	510
		3.9	20.0		
		6.2	12.85		
IMPELLER IN MOTION (250 r.p.m.)					
51.4	6.04	1.5	85.5	0.196	66.3
		3.3	38.85		
		5.6	23.05		
		7.1	18.3		

TABLE 2/.....

TABLE 2.

RESULTS OF TESTS ON SUSPENSIONS (CAPILLARY METHOD)

Length of Capillary (cm)	Capillary Diameter (m.m.)	Total applied Pressure (P_T)	Time for 78 ccs (t) (secs)	Differential Viscosity (ηd)	Stormer Result (η d)	Concentration % Solids by Volume.
37.95	1.45	54.0 59.0 64.9	1459 1176 956	0.19	0.366	34.1
37.95	3.06	37.1 41.6 46.1 50.3 55.6 60.3 64.2	370.4 207.2 145.0 112.5 85.2 72.1 62.35	0.26	0.366	34.1
37.95	4.7	56.1 59.0 61.7 65.2	222.2 127.8 93.3 68.0	0.35	0.366	34.1
37.95	6.96	54.7 56.4 58.5 60.8	65.1 41.85 28.5 21.5	0.38	0.366	34.1
37.95	4.7	31.5 33.1 35.0 37.0 39.1	96.2 57.2 36.6 27.0 20.9	0.136	0.153	30.0
37.95	4.7	38.5 44.0 50.6 59.7	301.3 150.5 89.8 57.8	1.002	1.432	40.0
37.95	6.96	34.8 38.0 41.2 47.5	177.1 83.2 53.0 28.5	1.44	1.432	40.0

TABLE 3/.....

TABLE 3.

RESULTS OF TESTS ON STABLE SUSPENSIONS (STORMER METHOD)

Coal- Zinc chloride Suspension (% Solids by volume)	Effective driving weight F_{TS}	$\frac{1}{T_a}$	$d\frac{1}{t}\cdot\frac{P=Rho}{t}$ -8 x 10	Re	Differential Viscosity	Q	Viscous & Turbulent resistance Fa + Fb obtained from curve	Yield Value F_o	Viscous resistance $Fa=\eta\frac{A}{T}$	F_{LS} $(F_a + F_o)$	Values obtained from reconstructed curves	
											Yield Value Fo	Differential Viscosity (η d)
	8.2	0.018	723	0.1645	0.148	13,200	5.8	2.4	5.0	7.4		
	9.45	0.021	802	0.187	0.152	11,700	7.0	2.45	6.0	8.45		
	10.9	0.025	918	0.222	0.153	10,050	8.5	2.4	7.15	9.55		
	10.95	.003	1050	0.264	0.154	8,550	10.4	2.35	8.6			
	14.7	0.035	1178	0.305	0.156	7,550	12.5	2.2	10.2	12.4	2.5	0.153
	16.75	0.04	1315	0.357	0.152	6,550	14.2	2.55	11.4	13.95		
	18.8	0.045	1407	0.392	0.156	6,050	16.6	2.2	13.1	15.3		
30.0	21.0	0.05	1532	0.44	0.154	5,500	18.65	2.35	14.4	16.75		
	23.05	0.018	286	0.056	0.436	34,500	15.15	7.9	14.7	22.6		
	25.6	0.021	333	0.0657	0.434	29,850	17,85	7.75	17.0	24.75		
	29.05	0.025	397	0.0787	0.431	25,300	21,45	7.6	20.15	27.75		
	33.3	0.03	476	0.097	0.42	21,150	25.8	7.5	23.55	31.05		
	37.5	0.035	532	0.1115	0.426	18,800	31.2	6.3	27.9	34.2		
	41.8	0.04	610	0.133	0.408	16,100	34.9	6.9	30.5	37.4	10.4	0.366
	46.4	0.045	660	0.147	0.415	14,680	40.3	6.1	34.9	41.0		
34.1	51.05	0.05	729	0.166	0.408	13,050	44.2	6.85	38.1	44.95		
	74.4	0.018	867	0.017	1.436	111,500	48.9	25.5	48.3	73.8		
	83.4	0.021	1012	0.0193	1.476	98,000	58.6	24.8	57.9	82.7		
	94.4	0.025	1204	0.0235	1.443	81,200	68.9	25.5	67.4	92.9		
	108.6	0.03	1446	0.028	1.454	69,000	84.2	24.4	81.5	105.9		
	122.7	0.035	1686	0.0327	1.451	59,000	98.1	24.6	94.9	119.5	26.1	1.432
	136.5	0.04	1927	0.0375	1.446	52,300	113.5	23.0	108.1	131.1		
40.0	151.1	0.045	2167	0.0425	1.436	45,650	125.3	25.8	120.8	146.6		

TABLE 4.

RESULTS OBTAINED WITH CAPILLARY INSTRUMENT ON TRUE LIQUIDS.

Length of Capillary (cm)	Capillary Diameter (m.m.)	Total Applied Pressure P_T	Time for 78 cc. (t) (secs.)	Viscosity (η) (Poises)	Constant A
IMPELLER STATIONARY.					
51.4	4.7	25.9	217.8	0.177	1959
		28.0	91.6		
		30.5	55.7		
		32.9	40.3		
		36.7	27.9		
		39.9	22.15		
IMPELLER SPEED (250 r.p.m.)					
51.4	4.7	28.1	99.8	0.177	2,000
		30.8	58.7		
		32.9	43.2		
		36.8	28.75		
		40.1	23.05		
IMPELLER SPEED (350 r.p.m.)					
51.4	4.7	28.3	123.0	0.177	2,161
		31.8	56.5		
		36.4	33.35		
		41.3	23.8		

TABLE 5/.....

TABLE 5.

RESULTS OF TESTS ON AN UNSTABLE SUSPENSION.

1. WITH STORMER VISCOSIMETER

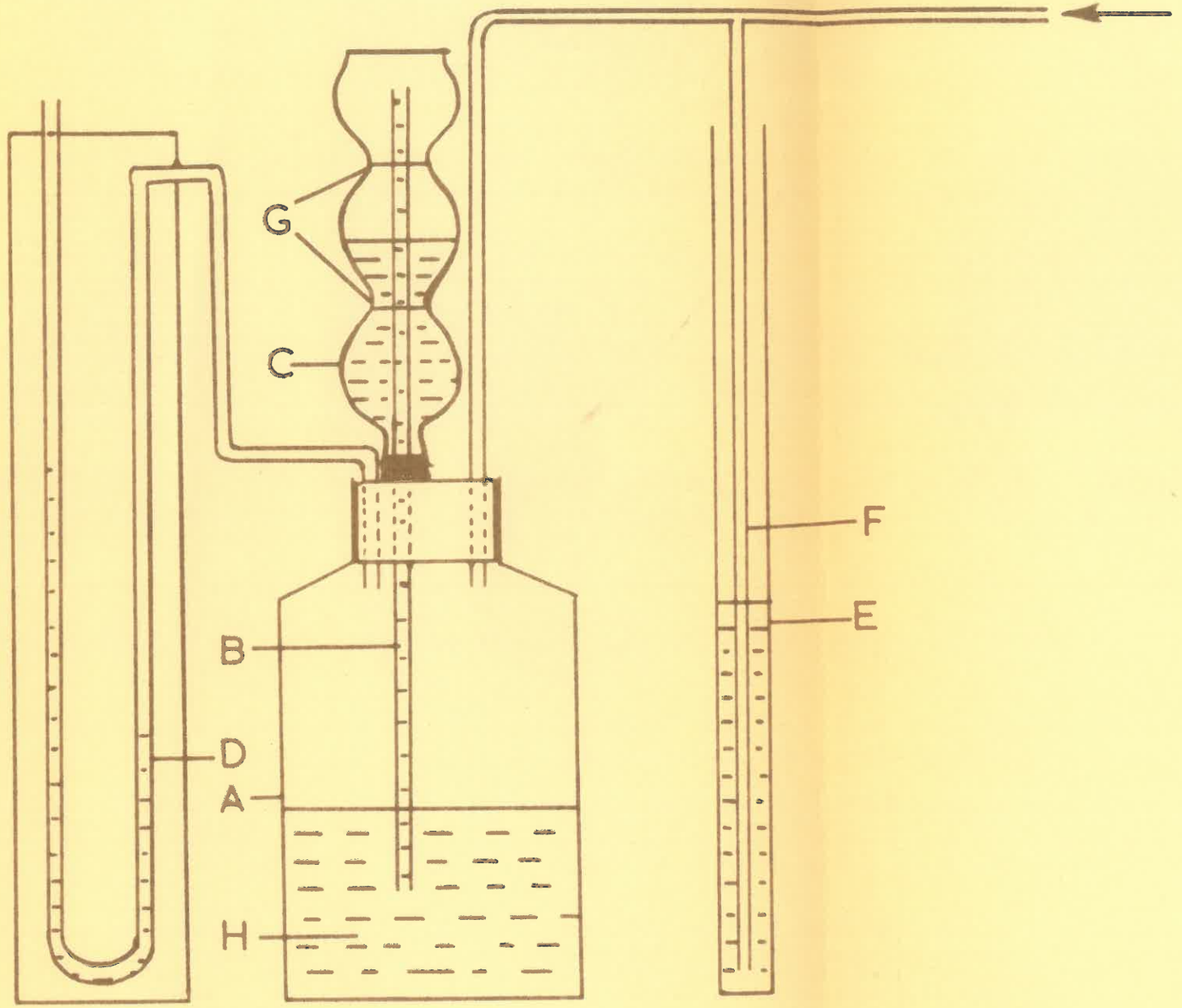
Suspension	Effective Driving Weight F_{rs}	$\frac{1}{T_a}$	$d\frac{1}{t} \cdot \frac{p}{t}$ x 10^{-8}	Re	Differential viscosity	ø	Viscous and Turbulent resistance $F_a + F_b$ obtained from curve.	Yield Value F_o	Viscous Resistance $F_a = \frac{\eta A}{T}$	F_{LS} (Fa+Fo)	Value obtained from reconstructed curve.	
											Yield Value Fo	Differential Viscosity(η d)
Baum Refuse suspended in water. Suspension S.G.1.27	11.2	0.018	686	0.1535	0.148	14,050	5.8	5.4	5.0	10.4		
	12.5	0.021	808	0.1885	0.142	11,600	6.5	6.0	5.6	11.6		
	13.9	0.025	934	0.227	0.14	9,850	7.8	6.1	6.5	12.6		
	15.5	0.03	1104	0.2815	0.135	8,100	9.3	6.2	7.6	13.8		
	17.3	0.035	1209	0.316	0.1[illegible]	7,300	11.3	6.0	9.2	15.2	6.4	0.135
	19.1	0.04	1355	0.372	0.1[illegible]	6,300	12.8	6.3	10.2	16.5		
	21.1	0.045	1465	0.414	0.138	5,820	15.0	6.1	11.6	17.7		
	23.0	0.05	1568	0.457	0.139	5,430	17.2	5.8	13.0	18.8		

2. WITH CAPILLARY VISCOMETER.

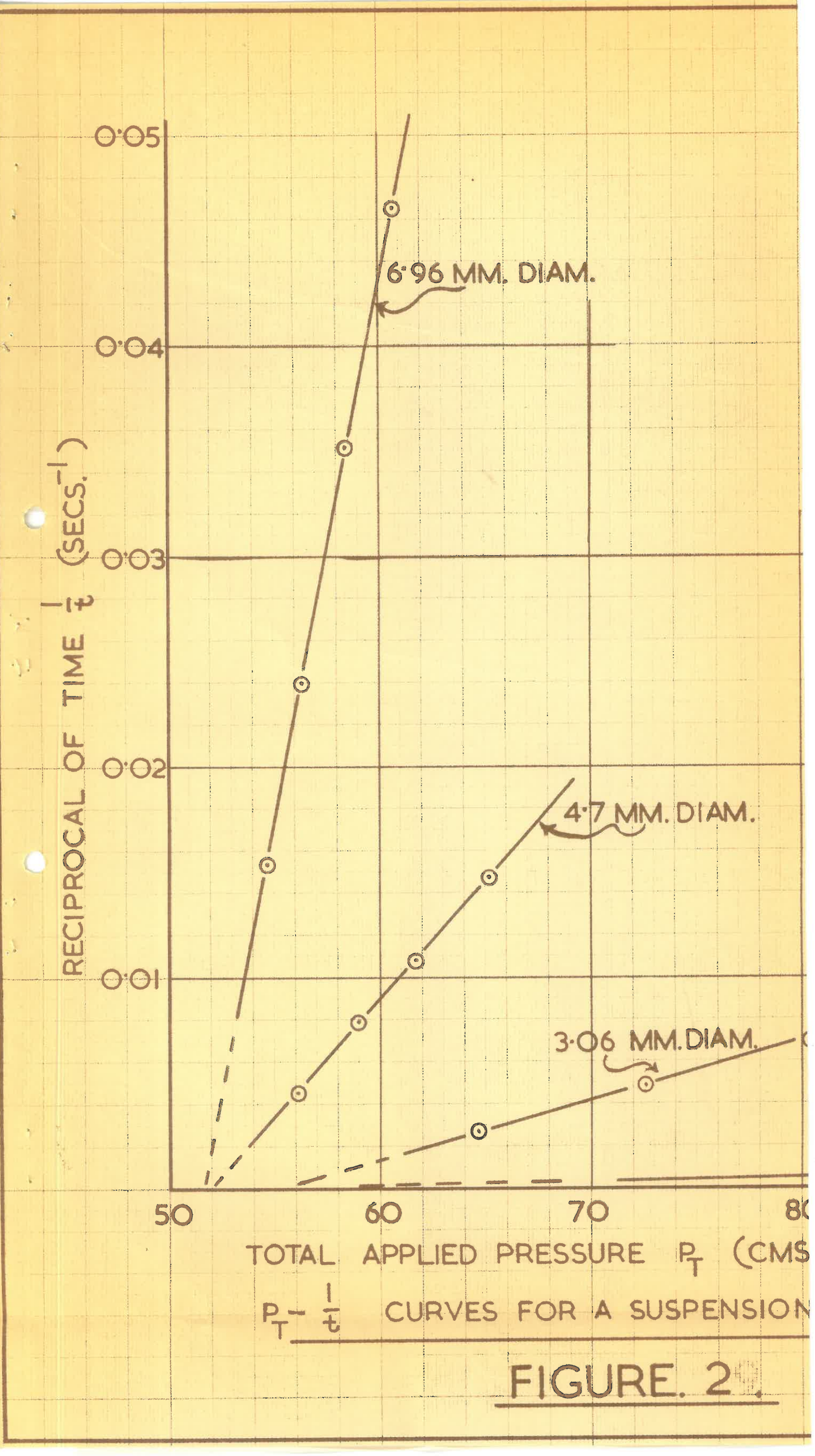
Suspension	Length of Capillary (cm.)	Capillary Diameter (m.m.)	Total Applied P_T	Time for 78 cc.(t) (secs)	$\frac{1}{t}$ $^{-1}$ (secs.)	Constant A	Different-Viscosity (Poise) (η d)	Stormer Result (η d)	Impeller Speed (r.p.m.)	
Baum Refuse Suspended in Water. Suspension s.g.1.27	51.4	6.04	0.6	136.4	0.0073	66.3	0.133	0.135	250	
			2.7	31.15	0.0321					
			6.4	14.5	0.069					

FIGURE. 1.

OVERFLOW VISCOMETER

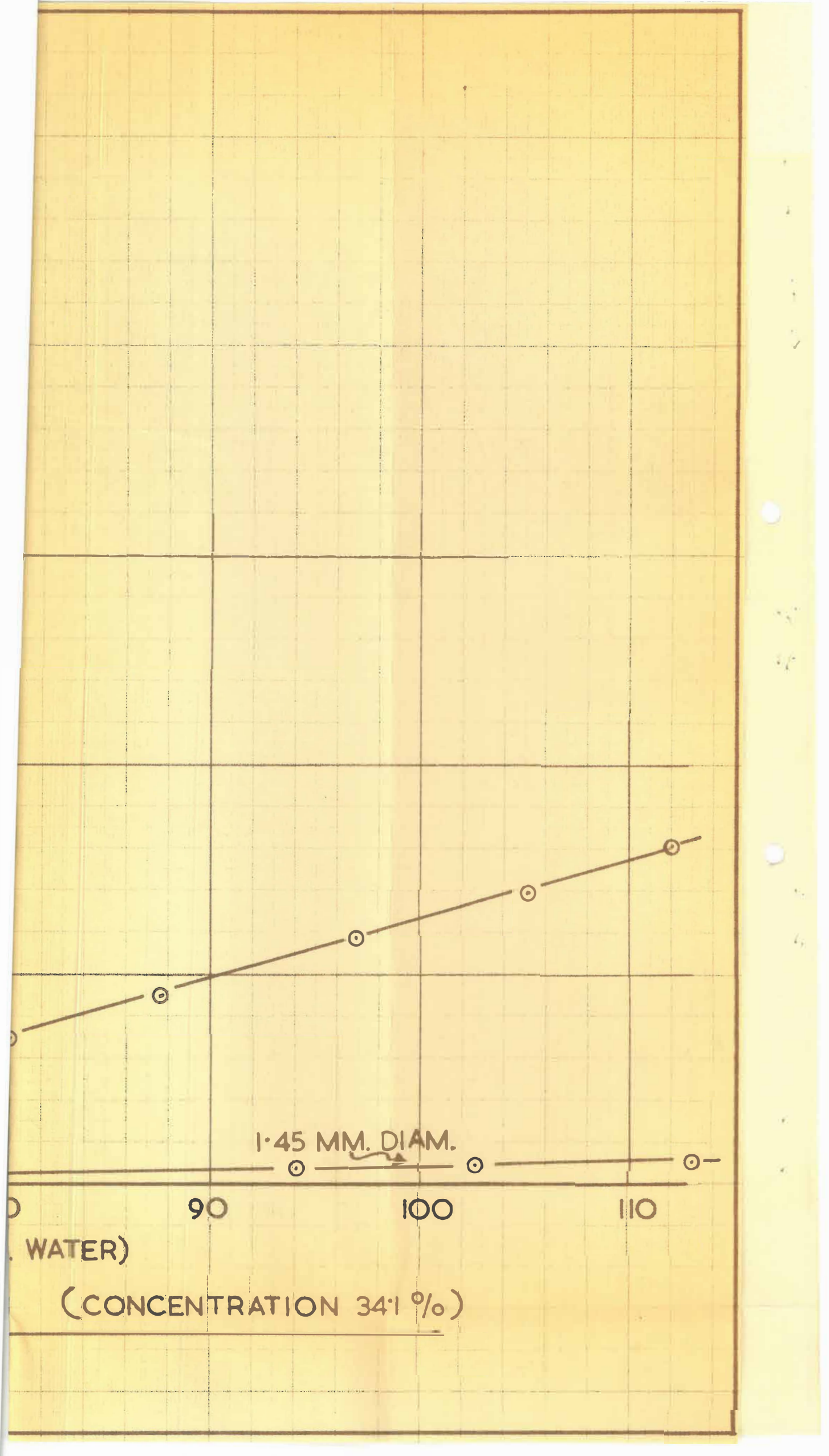


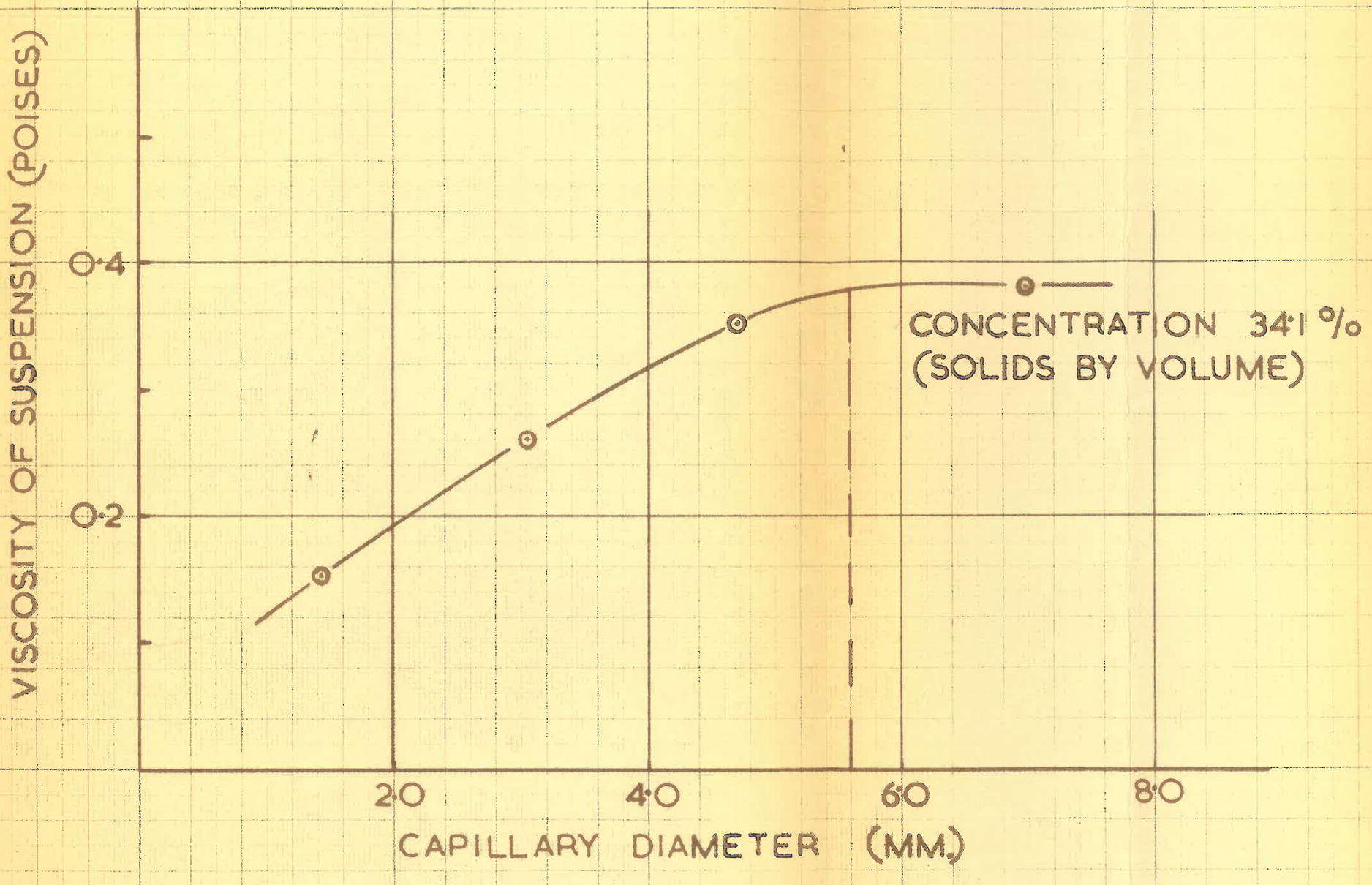
A. SAMPLE COTAINER
B. CAPILLARY
C. COLLECTING VESSEL
D. MANOMETER
E. PRESSURE REGULATOR
F. BUBBLER TUBE
G. TIMING MARKS
H. TEST FLUID



$P_T - \frac{1}{t}$ CURVES FOR A SUSPENSION

FIGURE. 2.


1·45 MM. DIAM.
90
100
110
WATER)
(CONCENTRATION 34·1 %)



DECREASE IN VISCOSITY BELOW A CRITICAL CAPILLARY DIAMETER

FIGURE. 3.