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EXPLOSIVES RESEARCH & DEVELOPMENT ESTABLISHMENT

REPORT No. 10/R/49

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Viscous and Elastic Properties of Concentrated Solutions of High Polymers

I. A Suitable Thixoviscometer and Thermostat

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INV. 90

G. Stainsby and A. G. Ward

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WDGS – INTELLIGENCE REPORT

COUNTRY Gt. Brit. JLA/mjs
I. D. NO. 643234

SUBJECT: Instrument for Measuring Viscosity
FROM: ARMYAT-London REFERENCES:
EVALUATION: A-1 DATE OF INFORMATION: November 1949 DATE OF REPORT: 21 February 1950
INCL. 1 PREPARED BY: J.L.Atkins, Research Analyst SOURCE: Ministry of Supply

SUMMARY OF SID REPORT: The U.S. Naval and Air Attaches, London, have each received copies of the inclosure.

G. Stainsby and A.C.Ward in Explosives Research and Development Establishment report 10/R/49 discuss a suitable thixoviscometer and thermostat for measuring the viscous and elastic properties of binders which have been used in experiments upon the plastic propellant. Studies upon the flow properties of the finished plastic have been undertaken by a plastic-meter, while those of the constituent binder are being investigated by the thixoviscometer described in this report. Between them these two instruments cover the complete range from a pure liquid of viscosity of 7 poises to a complex plastic suspension with a viscosity well over 100,000 poises.

COMMENT: The Ministry of Supply request that copy No 33 be transmitted direct to R&D Board.

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P. H. DRAPER, Jr.
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MINISTRY OF SUPPLY.

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT.

E.R.D.E. REPORT NO. 10/R/49.

Viscous and Elastic Properties of Concentrated Solutions of High Polymers.

I. A Suitable Thixoviscometer and Thermostat.

by

G. Stainsby and A.G. Ward.

This report contains no information of overseas origin.

Approved by C.H. Johnson
A/C.S./E.R.D.E.

Waltham Abbey,
Essex.

Note: The investigation described in this Report was carried out when the Section concerned was an integral part of the Armament Research Department, Fort Halstead, Nr. Sevenoaks, Kent. The work and the Section are now under the direction of the Chief Superintendent, Explosives Research and Development Establishment, Waltham Abbey, Essex.

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SUMMARY.

1. Object of Investigation.

To design a suitable instrument for measuring the viscous and elastic properties of binders which have been used in experiments upon the plastic propellant, this being the first stage in a thorough study of the fundamental rheological properties of those binders.

2. Scope of Investigation.

(i) Design of an absolute thixoviscometer which can be used

- (a) as a Couette-type instrument, using constant rate of shear
- (b) as a Searle-type instrument using constant shearing stress, and
- (c) for measurement of relaxation-times at constant strain.

(ii) Design of an air-thermostat, suitable for general use to cover the temperature range -20°C. to +100°C. with close temperature-control.

(iii) Calibration of the thixoviscometer for uses (a) and (b) by means of suitable fluids of known viscosity.

3. Conclusions.

An instrument has been designed to fulfil conditions (a), (b) and (c) above which, when used in the new air-thermostat, is capable of determining absolute viscosities over the range 10 poises to 100,000 poises with an accuracy of 1-2%.

An air-thermostat has been designed which easily covers the range -20°C. to +100°C. with a fluctuation of not more than 0.1°C. provided the external room temperature is reasonably steady. The power consumed to operate the thermostat is rather large.

4. Suggested further developments.

The inner and outer cylinders should be re-made with greater precision, if possible, so that the accuracy of the instrument should be increased to better than ± 1%.

A well should be available below the thermostat so that when used as a Searle-viscometer the present restriction of one and a half revolutions of the outer cylinder is removed.

/Introduction.

INTRODUCTION.

The viscous and elastic properties of the binders used in the plastic propellant affect, in complex ways, the manufacture and storage of the finished plastic. Studies upon the flow properties of the finished plastic have been undertaken by a plastometer (P.R. Freeman, A.R.D. Explosives Report 228/46), whilst those of the constituent binder are being investigated by the thixoviscometer described in this report. Between them, these two instruments cover the complete range from a pure liquid of viscosity of 7 poises to a complex plastic suspension with a viscosity well over 100,000 poises.

The thixoviscometer has been designed so that the instrument possesses three main modes of operation:

1. Viscosity determination at constant rate of shear (Couette type).

2. Viscosity and elasticity determinations at constant shear (Searle type).

3. Measurements of relaxation times at constant strain.

In principle it is a rotating-cylinder viscometer.

Two types of cylinder are used :-

(a) cone-ended.

(b) flat-ended.

The former gives absolute viscosities since, following the theoretical treatment of Mooney (1934) the angles of the cones are made so that the mean shearing stress over the conical surface is numerically equal to the arithmetic mean shearing stress over the cylindrical surfaces. This principle has been used in the conicylindrical viscometer of Lee and Warren (1940) which also is suitable for highly viscous liquids (10^3 - 10^8 poises) and the design of which was used as a starting point for the present instrument.

The flat-ended cylinders are capable of giving absolute viscosities only after the magnitude of the "end-effect" has been ascertained as a correction to be supplied to the result. One method of determining the correction is by means of duplicate measurements with the conicylinders.

The annulus, into which the material is filled by weighing, is only 5/32 inch, the radius of the outer cylinder being 1.000 inch. With such a small annulus the difference in the shearing stresses at the inner and outer cylinders is very small, i.e. the shearing stress is approximately constant across the annulus, and for most materials the mean shearing-stress may be used.

DESCRIPTION OF THE THIXOVISCOMETER.

The cylinders are of brass, except the tips of the conicylinders, which are of hardened steel. When in position the axes of the cylinders are coincidant and vertical, this being achieved by the relative position of the axis of the main frame and the axis of the housing of the vertical spindle (Figures 1 and 2). The main frame fixes the axis of the inner cylinder, the housing that of the outer cylinder. The housing is bolted to the base of the main frame and held in the correct position by two locating pins.

/The

The vertical spindle is held axially in the housing by two MS8 ball races (Hoffmann), the races being completely enclosed and 6 inches apart. The upper end of the spindle is a horizontal circular table to which, in a unique position, the cup forming the lower end of the outer cylinder can be fixed by four bolts. Into this cup the main (cylindrical) portion of the outer cylinder is screwed. Around the lower edge of the cup a 6 inch brass protractor, marked in degrees, is held by two set-screws at right-angles to one another. The reference-line for this protractor is attached to the top of the housing.

Between the MS8 ball races there is a slightly grooved pulley fixed to the spindle. A couple is applied to this pulley by two pieces of oiled-silk fishing line acting tangentially. The lines are attached to weights after passing over two smaller pulleys, the housings for which are bolted to machined surfaces on the main frame in suitable positions. The axes of the smaller pulleys are horizontal and run in self-aligning ball journals (Hoffmann US1). (The pulleys and weights are used in the method employing constant shearing stress).

The lower end of the vertical spindle projects just below the main frame and can be attached to a connecting rod from a motor, the rod being held by a tapered pin. The motor employed is of the H-gear pattern (Type 14-9 VM; 1/2 H.P.). A 100 to 1 reducing gear is attached to the output so that a continuous range of speeds from 1 r.p.m. to 9 r.p.m. can be applied to the outer cylinder via the vertical spindle. This is used in the method involving constant rate of shear.

The upper ends of the inner cylinders are held axially by steel locating bushes which, in turn, are held in position by brass sleeves held in the cross-members of the main frame. The sleeves are machined so that the bushes are push fits and the upper rims of the bushes rest on the machined horizontal surfaces of the sleeves.

For the method employing a constant shearing stress (Fig.1) the inner cylinder is fixed in position by clamping the locating bush, and the outer cylinder is rotated by the couple applied to the vertical spindle. The speed of rotation of the outer cylinder is measured as it increases to a constant value by timing known consecutive angular intervals of the protractor. The immersed length of the inner flat-ended cylinder can be varied by the vertical movement of the locating bush in the sleeve. The immersed length of the conicylinder can be varied only by adding to or removing material in the annulus.

For the method using constant rate of shear (Fig.2) the inner cylinder is suspended by a torsion wire. The outer cylinder is rotated at constant speed by the motor and the viscous drag of the material acts on the inner cylinder in the direction of rotation. This torque is opposed by the torsion-wire, an equilibrium position of the inner cylinder resulting when the two are equal. Here two locating bushes and sleeves are used, one on the uppermost cross-member and the other on the lower cross-member. The upper locating bush locates the upper end of the torsion wire. The lower bush is hollow and locates a ball race (L.S.7. Hoffmann) which in turn locates a spindle.

The upper end of the spindle locates the lower end of the torsion wire. A small mirror is used to determine the equilibrium

/twist

twist of the wire, the mirror being fixed to a machined surface close to the axis of the spindle. This surface is near the lower end of the spindle, i.e. just above the inner cylinder, which is located axially by the spindle.

The race is not a push fit into the hollowed locating bush but the lower part of the bush is sprung and surrounded by a spring-steel ring of adjustable diameter. The upper part of the bush is clamped in the main frame by a set-screw. A view of the suspension of the inner cylinder for the method of constant rate of shear is shown.

The torsion wire is soldered at each end into a keyed brass cylinder. This is a push fit into a hollow cylinder threaded on the exterior. A third cylinder, hollow and threaded on the interior, screws down over the other hollow cylinder. The exterior cylinder has a wide hole in the base through which the torsion wire passes.

The hollow cylinder for the lower end of the torsion wire is the uppermost part of the spindle supporting the inner cylinder. At the upper end of the wire the hollow cylinder is the lower end of a long solid keyed cylinder which is a push fit into the locating bush. The upper end of this cylinder is threaded and bears two threaded discs which serve to move the flat-ended inner cylinder in a vertical direction so adjusting the immersed length. With the inner conicylinder they form a useful way of ensuring that the cones just bear on one another and that the fine torsion wires are just taut.

To measure relaxation-times at constant strain the arrangement of the instrument just described has two modifications. Firstly the outer cylinder is clamped. This is done by engaging a spring-loaded spindle horizontally into a cylindrical hole in the main vertical spindle. The loaded spindle is housed in the front of the main housing. By disengaging the spindle from the hole, into which it is an easy sliding fit, the outer cylinder can be rapidly released without imparting to it circular motion. This is used in the method of obtaining viscosities and elasticities at constant shearing stress.

The second modification is in the torsion head at the upper end of the wire (Fig.2). The locating spindle is not keyed in this instance, nor is it as long. The upper end carries a short thread, by which a brass cylindrical head is attached. A double spring washer fits between the locating spindle and the locating bush, a little way above the clamping mechanism for the torsion wire. The brass head is screwed down so as to compress this spring: thus further movement of the brass cylinder in either direction is followed by the upper end of the torsion wire. The outer lower edge of the head is marked every 5° and a reference mark is attached to the top of the main frame. Between the double spring washer and the torsion-clamp a flat is machined on the locating spindle to carry a mirror. The length of the brass cylinder is such that it just does not touch the top of the locating bush.

A peg projects radially from near the lower edge of the head. Around the locating bush a brass ring is screwed to the top of the main frame. The ring is provided with small tapped holes, each at an angular distance of 10°. Into these holes small threaded pegs may be fitted. These serve to restrict to known values the movement of the upper end of the torsion-wire.

/The

The operations, which require two observers, are as follows:-

With the outer cylinder clamped the torsion-head is quickly rotated through a known angle using the pegs as steps. The lower end of the torsion-wire begins to follow this movement. As soon as any initial elastic effects are over the upper end of the torsion wire is relaxed so as to keep the lower end at some chosen deflection. The relaxation of the upper end is followed using the graduated head for large movements and the mirror for small movements. One observer chooses the strain and performs the relaxation: the other records the relaxation with time. Clearly only very thin torsion wires may be used to impart large strains to the material. Figures 4 and 5 show the general assembly for each of the two methods of measuring viscosity.

Modifications incorporated in the present Thixoviscometer.

The original design of the thixoviscometer has been improved in three main ways. Only the improvements have been included in the description so far, but it is of value to record the failures of the original design.

The most significant improvement is in the manner in which rotation of the outer cylinder is achived for measurements at constant rate of shear. Originally the cylinder was driven by a belt from a geared motor. The belt passed round a grooved-pulley on the vertical spindle between the MS8 ball-races. The motor was outside the thermostat. This system was not satisfactory because of (i) lack of real constancy of speed

(ii) vibrations imparted to the mirror on the inner cylinder

(iii) narrow choice of speeds.

The present design, using a 3 phase squirrel-cage motor coupled directly to the vertical spindle has overcome objections (i) and (ii). The third objection has been satisfactorily met since any speed in the range one to nine r.p.m. can be obtained from the motor. This range is that most suitable for the expected uses of the instrument.

The second improvement is in the pulleys over which the lines carrying the beam and load pass. Previously the bearing of each pulley was a line-bearing formed by the insertion of a solid cone into a hollow cylinder. The modified bearing is formed by small self-sligning ball-races. The two assemblies are shown in Figure 6. The system used originally was unsatisfactory in that the magnitude of the friction was large and that the dependence upon load was marked, e.g. with no load, 22 grms.

with a load of 1000g, friction (dynamic) = 50 grms.

The bearing was lubricated by Allen's Clock Oil. The friction of the present system is lower and is less dependent upon load (fig.7).

The third modification to the original design was an addition - the LS7 aligning bearing for the upper-end of the inner cylinder. This is essential for measurements using the flat-ended cylinders at constant rates of shear, and is a great help in preventing distortion of the thinner torsion wires whilst assembling the instrument.

/Finally

Finally, a hole has been drilled diametrically through the inner flat-ended cylinder about 1 cm. from the upper edge. Through the hole a thin steel rod is inserted and rests on the outer-cylinder. This makes it possible to change the torsion-wires as the temperature is varied for one filling of the annulus. Previously it was almost impossible for the inner flat-ended cylinder to be held whilst the wires were being changed, unless the material under investigation possessed a viscosity of about 100,000 poises at the temperature employed.

The Air Thermostat.

Viscosity changes rapidly with temperature. Therefore viscosity measurements require close temperature control. The thixoviscometer is a large instrument not capable of immersion in a liquid during use, and so the employment of air thermostat becomes necessary. Other workers have surrounded the rotating cylinders by coils through which various fluids at constant temperature could be passed, but the use of an air thermostat gives greater flexibility in operation and avoids doubtful effects due to conduction along the various metal parts. The control with the present air thermostat is to ± 0.1°C., a figure not easily attainable by enclosing the cylinders in constant temperature coils.

Essentially the thermostat consists of a large wooden box, (Figure 8,) almost square in horizontal section. The internal volume is 30 cubic feet. The inner walls are of 1/4" sheet stippled board (compressed filled asbestos) and are separated from the outer match-boarding by 7" of slag wool. There is a wooden false roof and back, the latter ending about 1 foot from the floor. Into the false roof a circular hole has been cut, 13" in diameter to contain the 12" fan used to circulate the air inside the thermostat. Circulating air passes through this hole, along the false roof, down the false back and thence into the main volume of the thermostat. The fan is driven by a three phase motor (Keith Blackman Ltd. Type TE) at a constant speed of 1100 r.p.m. The motor is housed on the top of the thermostat and the connecting spindle is of such a design that no air can enter or leave at that point.

The front of the thermostat is mainly taken up by one door, which also is lagged by 7" of slag wool and which closes into the main frame by two "steps" of soft rubber. In the centre of the door there is a window (two square feet of 3/8" plate glass). Spaced around the door in suitable positions are five plugs. These are wooden blocks which fit into square holes in the door. When in position good seals are made by narrow pressure-tubing gaskets. Each plug has two fasteners, one each side, so arranged that when the plugs are fitted the rubber is compressed. When a plug is taken out there is a 6" square hole through which manipulation of instruments etc., inside the thermostat can be performed.

The floor of the thermostat is covered by a 1/4" steel plate, to which instruments can be bolted. Where connections are required to the exterior through the floor, holes have been drilled through and then suitable sized paxolin tubing inserted and held in position vertically by a small circular bakelite disc at each end, the discs being inset into the inner and outer floors. When such holes are not in use they are filled at each end by putty.

/The

The thermostat stands on four legs of 6" steel tubing, each leg being 27" long. Each leg is enclosed at either end by a 6" square 1/8" steel plate. These plates are screwed into the matchboarding by coach-screws.

For use at temperatures at least 5°C. higher than room temperature heat is supplied by 9--500 ohm resistance mats, each measuring 12" x 10". These are connected in parallel across the output of a Variac transformer - Zenith Type 100L, capable of carrying up to 8 amps. The mats are on the front of the false back, being suitable insulated from it. To maintain a temperature of, say, 70°C. the current through these mats is so adjusted that when a steady state has been reached the temperature is 68°-69°C. The actual control of temperature is then made by a toluene-mercury regulator (similar to Townsen and Mercer, Ref.S.75) operating the control mats through a relay (Sunvic Type F102-4, lag about 3 seconds). These are two 500 ohm mats (as above) placed just below the fan so that they are in the path of the complete air-circulation. The regulator stands about half-way between the false-back and the door. With the main heating of 9 mats it is possible to get well over 110°C. and so boil the toluene out of the regulator (B.Pt. toluene 111°C. 760 mm.Hg.) For higher temperatures than 90°C., therefore, it would be convenient to use a similar regulator filled entirely with mercury. Because of the low specific heat of air it is preferable to use a regulator with a larger bulb than the S.75 regulator recommended for use in water-baths. In parallel with the control mats there is a small neon lamp. The bulb is fixed above the main electrical instrument panel which is fixed by a bracket so as to protrude as an extension of the front of the thermostat. The purpose of the neon bulb is to aid in arranging equal time-intervals of heating and cooling about the mean temperature; it also indicates any abnormal behaviour due, for example, to dirty mercury in the regulator.

For temperatures between 15°C. and 30°C. water-cooling is used. During the winter the upper limit for such practice becomes 25°C. The cooling coils are at present made of "compo" tubing (mainly lead) but finned copper would be preferable. They are situated between the back and the regulator i.e. just below the false back. It would be more desirable to have these coils suspended below the control mats but in the present thermostat that space is mainly needed to manipulate parts of the thixoviscometer. Cold water is circulated at full mains pressure through the coils. Leakage of air is prevented by stuffing the holes where the pipes enter the thermostat. This cooling is nearly balancedby the main heaters, the final balance being made as before.

For temperature below 15°C. the principle is the same as that described immediately above except that a refrigerator is used in place of water cooling. The machine, supplied by Messrs. J. & E. Hall Ltd., is powered by a 2 H.P. Crompton-Parkinson 3-phase motor. A double-belt drives the two-cylinder compressor. Methyl chloride is the refrigerant. After passing through a separator, to remove most of the oil, the gas passes through a water-cooled condenser, and then through an expansion-valve into the coils. The unit is situated on the floor at the side of the thermostat, occupying a space about 2'3" x 3'. The coils (1" piping finned) are situated between the false and real backs and are provided with a drip-tray below them, since it is inevitable that some condensation occurs upon them. The refrigerator has no regulator in the usual sense, i.e. it is always run continuously. There are automatic cut-outs attached so that the electricity supply to the motor is stopped if the cooling-water stops or if the gas pressure

/inside

inside the circuit becomes greater than some chosen value.

With continuous running the refrigerator will reduce the temperature inside the thermostat to -20°C. from $+20^{\circ}$C. in about 2 hours, assuming an external air-temperature of about $+20^{\circ}$C. During an 8 hour day, the temperature inside will fall to -32°C. Thus the lower temperature limit of -30°C is readily attainable. To control the temperature the main-heaters are used as a rough control and the control mats give a final control of $\pm$ 0.1°C. over the whole of the inside of the thermostat, the refrigerator being in continuous operation.

The nine main heater mats do not develop sufficient power to balance the cooling due to the refrigerator if a temperature higher than 0°C. is required. Therefore two subsidiary heaters each of 1000 watts have been installed just below the false-back. Using these in addition to the main heater mats it has been found quite easy to maintain $+10^{\circ}$C. to 0.1°C. provided

(a) the mains voltage does not fluctuate widely

(b) the lab. temperature does not show wide variation during the day.

If these effects are present it is necessary to follow the changes about every hour by a small adjustment of the current through the main heaters.

In all instances rapidity of attainment of low temperature is facilitated by covering the window with a large lagged plug made to fit the space. For taking measurements it is necessary to prevent ice-formation on the window. This has been satisfactorily achieved over hourly periods by smearing the window with a solution of Aerosol O.T. in glycerol. Inside the thermostat it has been found convenient to place trays of calcium chloride to keep the humidity at a low value.

Some considerable time (up to one day) is needed for the thermostat to become steady when the refrigerator is used. This is probably due in the main to the fact that the efficiency of the machine is automatically controlled. A copper tube containing some liquid methyl chloride is attached to the tube leading back from the refrigerator coils to the compressor. This virtually records the temperature of the refrigerator coils by the vapour pressure of the methyl chloride. The vapour pressure controls the gap in the expansion valve, thus controlling the efficiency of the machine. Low temperatures tend to increase the efficiency of the machine whilst high temperatures tend to decrease it. Consequently when attaining a steady $+10^{\circ}$C. it is necessary to allow time for the efficiency of the refrigerator to become steady.

This mode of operation is very wasteful of electrical energy but the more usual method of having the refrigerator controlled by a thermo-regulator is certainly not practicable for the accuracy desired here since in such instances the amplitude of hunting may amount to 4°C. The possibility of pumping brine from a controlled refrigerator around coils in the thermostat was considered but was found to be more expensive in outlay and the necessary apparatus required much more laboratory space.

To operate the thixoviscometer at low temperatures a plug has to be withdrawn since high viscosities are mainly measured at

/such

such temperatures and the method of constant shearing stress is the more suitable for high viscosities. This entails operating the release mechanism for each measurement. Rubber gloves with long sleeves can be attached around the inner edge of the plughole so that, on removing the plug the operator does not introduce warm moist air.

It is clear that this type of thermostat could be used for containing many other types of large apparatus for measurement of properties which are very dependent upon temperature. In thermostat design it has been found that the best results are obtained when the inside of the box is roughly cubical in shape; the fan is most conveniently mounted in the roof of the chamber, with suitable ducting to ensure complete circulation of the air.

EVALUATION OF ABSOLUTE VISCOSITIES.

I. Using the conicylinders.

(a) With the method of constant shearing-stress.

Let L (cm) = mean immersed length of inner and outer cylinders.

R_1 (cm) = radius of inner cylinder (2.145 cm)

R_2 (cm) = radius of outer cylinder (2.540 cm)

G (dyne-cm) = effective couple applied

Ω (radians/sec) = constant ang.vel. for a couple of G dyne-cm.

R_o (cm) = effective mean radius = $\sqrt{2R_1^2 R_2^2/(R_1^2 + R_2^2)}$ = 2.317 cm.

θ_o (degrees) = mean half apex angle of cone ends = $34^\circ\ 30'$

η (poises) = viscosity

then, following Mooney and Ewart (1934) and Warren and Lee (1940)

$$\eta = \frac{G}{4\pi\Omega L\left(1 + \frac{R_o}{3L\sin\theta_o}\right)}\left\{\frac{1}{R_1^2} - \frac{1}{R_2^2}\right\} \text{ poises}$$

The equivalent mean shearing stress S,

$$S = \frac{G}{2\pi R_o^2 L\left(1 + \frac{R_o}{3L\sin\theta_o}\right)} \text{ dynes/sq.cm.}$$

and the mean rate of shear = $\Omega\,\dfrac{R_2^2 + R_1^2}{R_2^2 - R_1^2}$ radians/sec.

For Newtonian liquids the last two equations apply exactly, but for non-Newtonian liquids they apply only to a second degree of approximation.

The effective mean length $L = \ell_1 + \dfrac{R_2 - R_1}{2}\;\dfrac{1 - \cos\theta_o}{\sin\theta_o}$

if ℓ_1 (cm) = immersed length of inner cylinder (Figure 9).

/The

The couple $G = (W + w - W^1)$ ga dyne -cm,

where W (grms) = load added to beam

w (grms) = weight of beam

W^1 (grms) = frictional force (grms) for a load $(W + w)$ grms.

$g = 981 \text{ cm/sec}^2$

a (cm) = radius of torque pulley + diam. of line = 5.10 cm.

[illegible] is obtained by plotting the reading of the protractor against time from the moment of release.

l_1 is obtained from the total length of the inner cylinder (16.78 cm) from a series of measurements upon the height not immersed - a dip-stick graduated in mm. is used.

W^1 is measured directly for the pulleys and ball races together, as follows :-

One pulley-housing is removed and clamped so that the tensions in the lines are in opposition instead of forming a couple. The beam is removed. Then, with equal weights attached to each line, extra weights are attached to one line until those weights drop at a constant speed i.e. dynamic friction is measured. A graph of total friction against total load is shown in Fig.7.

A special viscometer oil, Ex.183 (Messrs. Griffin & Tatlock), recommended by the National Physical Laboratory has been used at 25°C. in the instrument. The value obtained for the viscosity using the conicylinders was 970 ± 10 poises at 25.0°C. Measurements upon a sample of the material have been made at the N.P.L. and a viscosity of 1004 poises at 25.0°C. was reported.

(b) <u>The method of constant rate of shear</u>.

Let G^1 (dyne-cm) = effective couple in this instance

then $G^1 = \frac{\pi n r^4 \phi}{2x}$ dyne-cm

if n (dynes/sq.cm) = rigidity modulus of torsion wire

r (cm) = radius of torsion wire

x (cm) = length of torsion wire

ϕ (radians) = angle turned through by bottom of torsion wire when a steady value has been reached for a constant speed of rotation of the outer cylinder of radians per second.

The rigidity modulus, n , is measured separately for each wire by means of the S.H.M. of an inertia disc supported by the wire. There are four wires and four discs. The dimensions of the discs are arranged so that, with a minimum period for one swing of one second, each wire can conveniently be used with at least two discs. Using the formula

$$n = \frac{8\pi \times M b^2}{2 r^4 T^2}$$

in which M (gms) = mass of inertia disc

b (cm) = radius of inertia disc

T (secs) = mean time for one complete swing,

part of Table 1 has been compiled.

It is thought probable that the accuracy in the determination of the rigidity modulus is low due to the soldering of the wires into the keyed brass cylinders affecting the heat treatment of the wires.

Table 1. Characteristics of the wires.

Wire number	Diameter (cms)	10^{-11} n (dynes/sq.cm)	* Max. scale rdg. (cms).	* Viscosity range(poises)
1A	0.1022	7.48 ± 0.07	>35	8-40
2	0.1670	8.04 ± 0.05	31	40-260
3	0.2360	8.09 ± 0.07	21	180-750
4	0.3165	7.93 ± 0.02	16	590-1800

* Scale at 54.8 cms. from mirror.

The maximum scale reading is limited in practice to 35 cm. by the window of the thermostat.

ϕ is measured by the usual mirror and scale method. A small correction is applied for the lateral displacement of the reflected beam by the plate glass of the thermostat, the incident beam being perpendicular to the plate glass (Figure 10). All measurements have been made at a single convenient distance, measured directly from mirror to scale in the first place by removing the window. This position is fixed relative to the front of the thermostat and so can be found easily and accurately. The graph of corrected angle in radians and scale reading in cms. for that mirror-scale distance (54.8 cms) is shown in Figure 11. For scale readings of less than 7 cm. the formula :-

$$2\phi = \text{scale reading}/54.8,$$

which is accurate to at least 1 in 200, is used instead of the graph.

It is essential that the wires must not be strained beyond the elastic limit for steel. This imposes a maximum scale reading, at the given distance, for each wire. In calculating this for each wire a value for the maximum shearing stress of 5 tons per sq. inch ($5.0 \times 1.545 \times 10^8$ dynes/sq.cm) was used. This allows a safety factor of at least two. The maximum scale distances in cm. are shown in Table 1. If each maximum distance is the steady deflection given when the motor speed is highest, values of the maximum viscosity measurable by each wire can be calculated, assuming a convenient practical immersed length. The minimum viscosity measurable by each wire is determined by the accuracy

/with

with which scale readings may be measured. A convenient limit is 5 cm. for the highest speed (i.e. 0.60 c.m. for the lowest speed for a Newtonian liquid). This sets a lower viscosity limit for each wire. These limits are also shown in Table 1.

In every instance the speed of rotation of the outer cylinder is measured by using the protractor.

The friction of the aligning ball race does not enter into any measurement provided the zero is taken after the motor has been running. Then the friction is retained by the wire as a small deflection: only differences between this deflection and some other deflection are measured, these differences being due solely to viscous couples. This treatment assumes constant friction at a given temperature but does not depend upon the magnitude of the friction.

30-poise Castor Oil, a Newtonian liquid, measured with the conicylinders at 25.0°C. gave the following values :-

Wire 1A: viscosity 40.1 poises

" 2: " 39.6 "

A sample was sent to the N.P.L. for calibration and the figure given was 41.9 poises at 25.0°C.

The special viscometer oil EX183 has been investigated at 60.0°C using wire 2. The value obtained for the viscosity is 54.6 poises. Calibration of a sample at the N.P.L. at 60°C. yielded a figure of 53.8 poises.

II. Using the Flat-ended cylinders.

In this instance a set of measurements at one immersed length will not give an absolute viscosity. Absolute values may be obtained after applying a suitable "end correction" which may be determined either from the conicylinders or by altering the immersed length and keeping the "dead-space" constant. It is to be expected that the "end correction" will decrease as the dead-space increases.

Castor-oil has been measured at 25°C. using a torsion-wire. The dead-space was maintained constant and the change in immersed length calculated by adding a known weight of castor oil of known density. Two additions were made. After the second addition the depth not immersed was measured with the dip-stick. The original depth could not be measured with accuracy and was calculated from the final depth and the two changes in length. Table 2 shows the viscosities calculated assuming no end correction in column 4.

Table 2. Castor Oil at 25°C. Wire 1A. Flat-ended cylinder.

Density of oil at 25°C.	Weight added (grammes)	Change in length (cms)	Uncorrected Viscosities	Length immersed (cms)	Slope($\frac{\Phi}{\eta}$) (secs)
0.994	-	-	43.2	7.17	0.177
	23.83	4.11	41.6	11.28	0.265
	16.95	2.92	41.0	14.20	0.331.

/Since

Since $$\eta = \frac{n r^4 \phi}{8 \times \Omega \cdot L_f}\left(\frac{1}{R_1^2} - \frac{1}{R_2^2}\right)$$

if L_f = corrected immersed length of flat-ended cylinder

$= \ell + e$

where ℓ = measured immersed length

e = end-correction

$\ell + e = \text{constant} \times \frac{\phi}{\Omega}$, provided the dead-space does not change.

A plot of ℓ and $\frac{\phi}{\Omega}$ is shown in Figure 12. From this, the value of the end correction is seen to be 0.55 cm. For this determination the inner cylinder was held always so that the base was 2.54 cm. above the base of the outer cylinder, i.e. a dead-space of 2.54 cm.

The mean value of the viscosity of castor oil at 25°C. using the conicylinder is 39.85 poises. Using this value, effective immersed length can be calculated for the flat-ended cylinder for each of the three determinations. The values obtained are subtracted from the measured values, the difference representing the end-correction. The end-corrections obtained by such procedure are :

End correction	Measured immersed length
0.45 cm.	14.20 cm.
0.53 "	11.28 "
0.62 "	7.17 "

The mean value 0.52 cm., calculated by weighting the first two readings twice in relation to the latter reading since the accuracy of the latter is only 50% of that for the former, is in good agreement with the graphical value of 0.55 cm. It is not possible to measure any length to more than ± 0.02 cm.: two lengths have to be used to obtain the immersed depth. The flat-ended cylinders have also been used to measure the viscosity of oil EX183 at 25°C. using the method of constant shearing stress. In this instance both the immersed length and the dead space were varied. The viscosity for each set of measurements is shown in Table 3. Assuming the value of the viscosity to be 970 poises (the value obtained by the conicylinders), the end-correction for each dead-space has been calculated. Table 3 shows that, within a total error of 0.1 cm. in the measurement of the immersed and effective lengths, there appears to be no significant decrease in the end-correction with increasing dead-space.

/Table 3.

Table 3. End-correction using weights and oil Ex.183 at 25°C.

Dead space (cm)	Immersed length (cm)	Viscosity (poises)	End-correction (mm)
5.37	10.1 ± 0.05	1047 ± 20	8
4.31	11.8 ± 0.05	1020 ± 10	6.0
3.31	12.82 ± 0.02	1024 ± 10	7.1
2.54	14.08 ± 0.02	1010 ± 10	5.4
		Mean	6.6

(The values previously obtained at 2.54 cm. dead space were 5.5 mm. and 5.2 mm. for the end-correction).

Determination of the Shear Modulus of Elasticity.

This is described in detail in E.R.D.E. Report 11/R/49. Briefly, the method consists of allowing a constant speed of rotation to be attained by the outer cylinder (inner cylinder fixed) using a constant couple applied tangentially by weights via the pulleys and then suddenly removing the couple whilst observing the total back-deflection on the protractor. A mean shear modulus for the material across the annular space may be calculated and the values obtained (10^4 dynes per sq. cm.) are in fair agreement with those reported by Ferry (1942), for solutions of polystyrene in xylene, using a method of transverse vibrations.

Measurement of Relaxation under constant strain.

This, too, is reported in detail in E.R.D.E. Report 11/R/49. Essentially the method involves clamping the outer cylinder and supporting the inner cylinder by a torsion wire, the top of which is attached to a graduated disc. Thus known couples can be transmitted to the inner cylinder. After a rapid deflection of the torsion-head through a known angle the inner cylinder begins to follow, its motion being retarded by the viscous drag of the material in the annulus. As soon as any initial elastic effects are over a certain deflection of the inner cylinder from the rest position is chosen and the torsion head manually turned so as to release the stress in the wire to keep the inner cylinder at the given deflection. This deflection represents a constant strain in the material and the progress of the relaxation with time is recorded. Two observers are necessary.

As yet only one complete experiment has been performed in this manner. A 35% solution (by weight) of polystyrene in polymeths was used at 20°C., and the results indicate that the use of a single relaxation time may be permitted for that system.

Suggested further developments.

The accuracy of the instrument could be improved if the cylinders were re-made to a precision of 0.001 inch. The rigidity moduli of the torsion wires have a large experimental error which may be due to the soldered brass nipples. Possibly a slight modification in design could be made to cause an improvement in the final accuracy.

/The

The present apparatus is restricted in scope for measurements at constant shearing stress since the available space below the thermostat will allow only 1.5 revolutions of the outer cylinder to be measurable. With very viscous materials, especially if they are visco-elastic, the constant speed of rotation is not reached until the first revolution is nearly complete for certain loads. Thus it is desirable that more space should be available below the thermostat and it is suggested that a well be made. The entrance to the well might conveniently be covered by an iron grille when the motor is to be used.*

Acknowledgments.

The authors are indebted to Mr. Marriott and the members of the staffs of the Drawing Offices and Workshops of C.S.P.D.E. for their assistance in the design and making of the instrument; also to the N.P.L. for their determinations of the absolute viscosities of the calibration oils.

Literature.

Ferry. J.A.C.S. (1942) 64, 1323 - 1336.

Lee and Warren. Journal of Scientific Instruments. March 1940. p.63.

Mooney and Ewart. Physics (1934) 5, 350

* The apparatus has now (1949) been moved to Waltham Abbey and a well covered by movable steel plates has been provided; it is very useful.

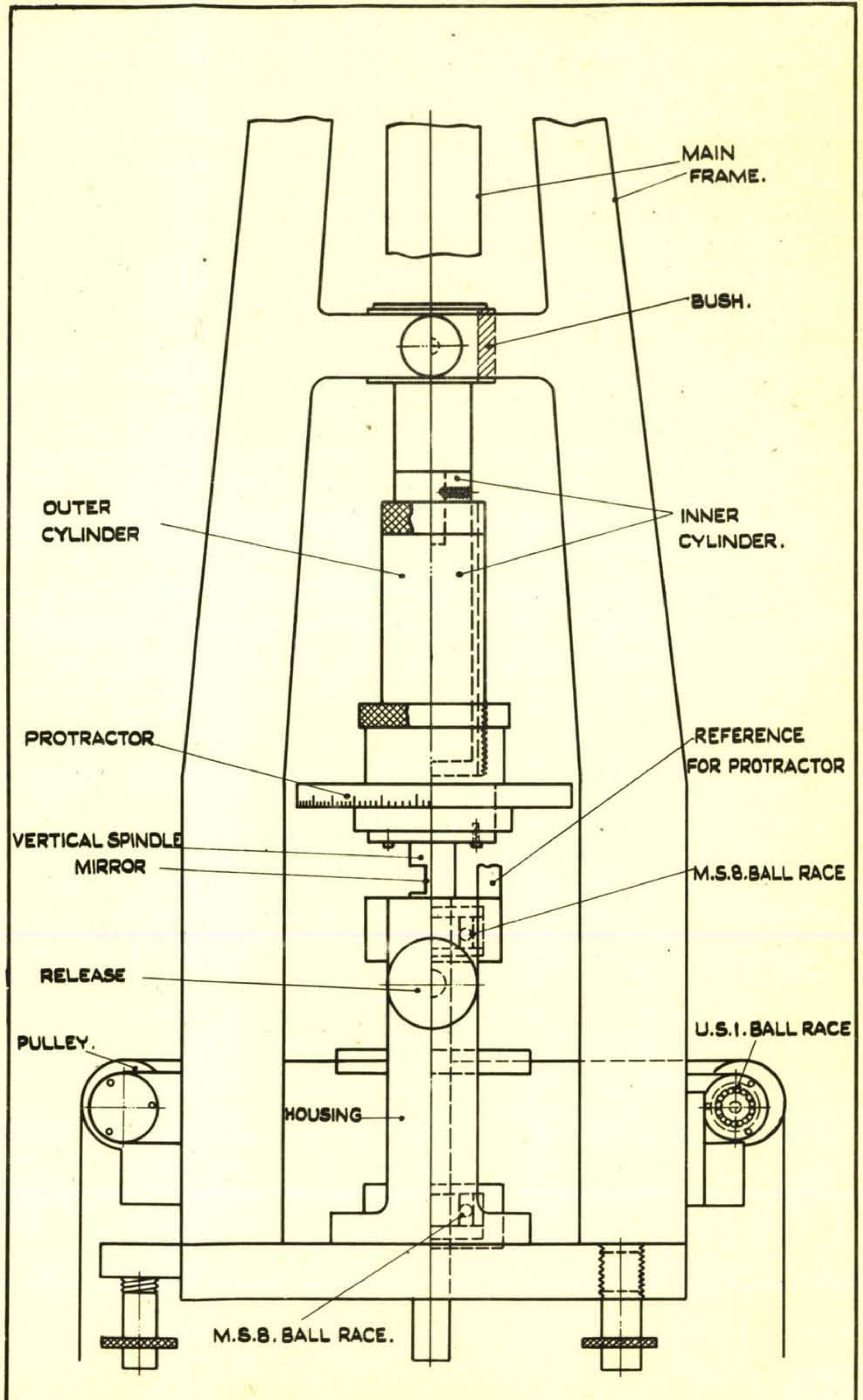


Fig. 1. Diagram of Thixoviscometer, Showing Method of Constant Shearing-stress.

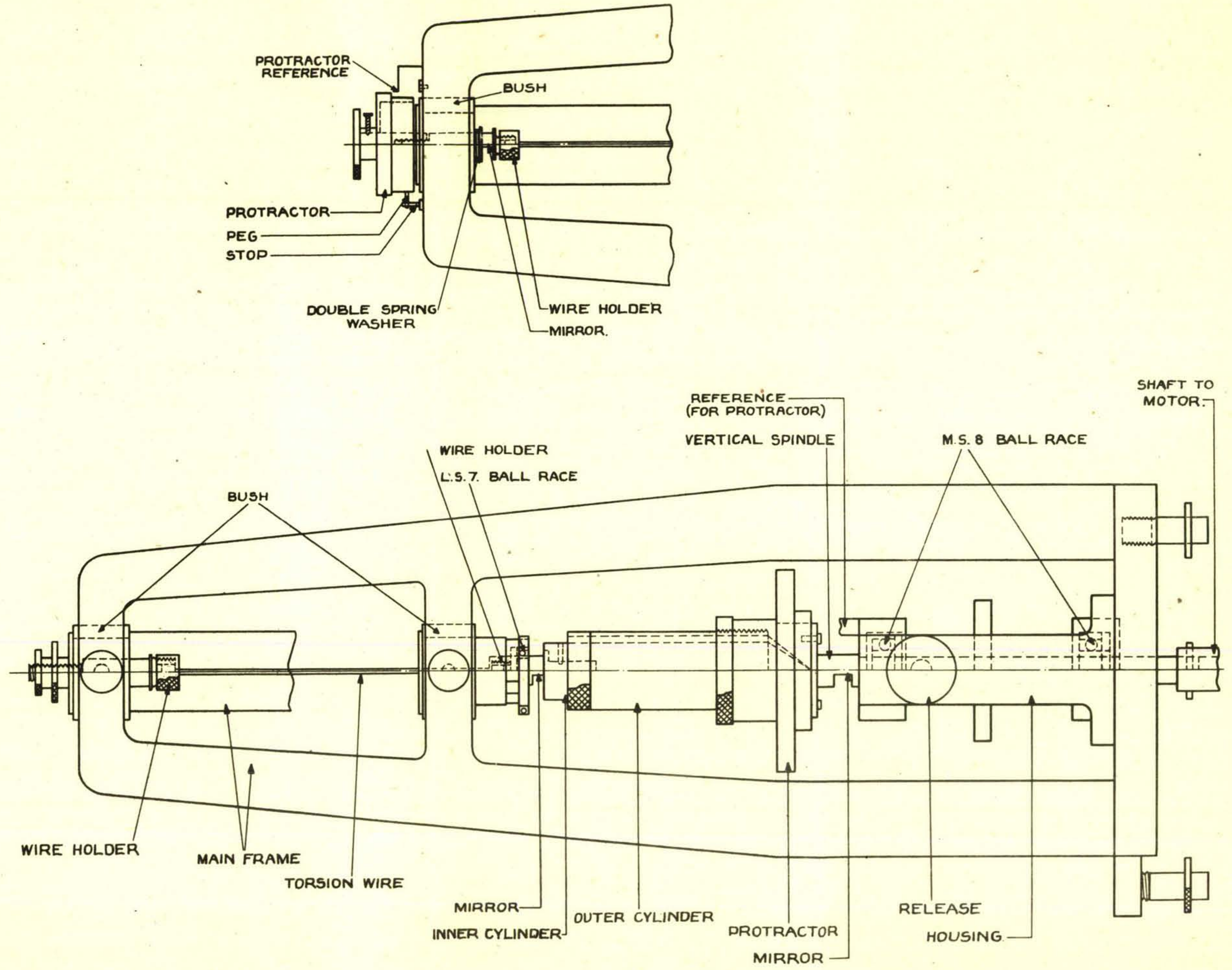


FIG. 2 DIAGRAMS OF THIXOVISCOMETER SHOWING THE ASSEMBLY FOR THE METHOD OF CONSTANT RATE OF SHEAR AND THE MODIFICATIONS FOR MEASUREMENTS OF RELAXATION TIMES AT CONSTANT STRAIN.

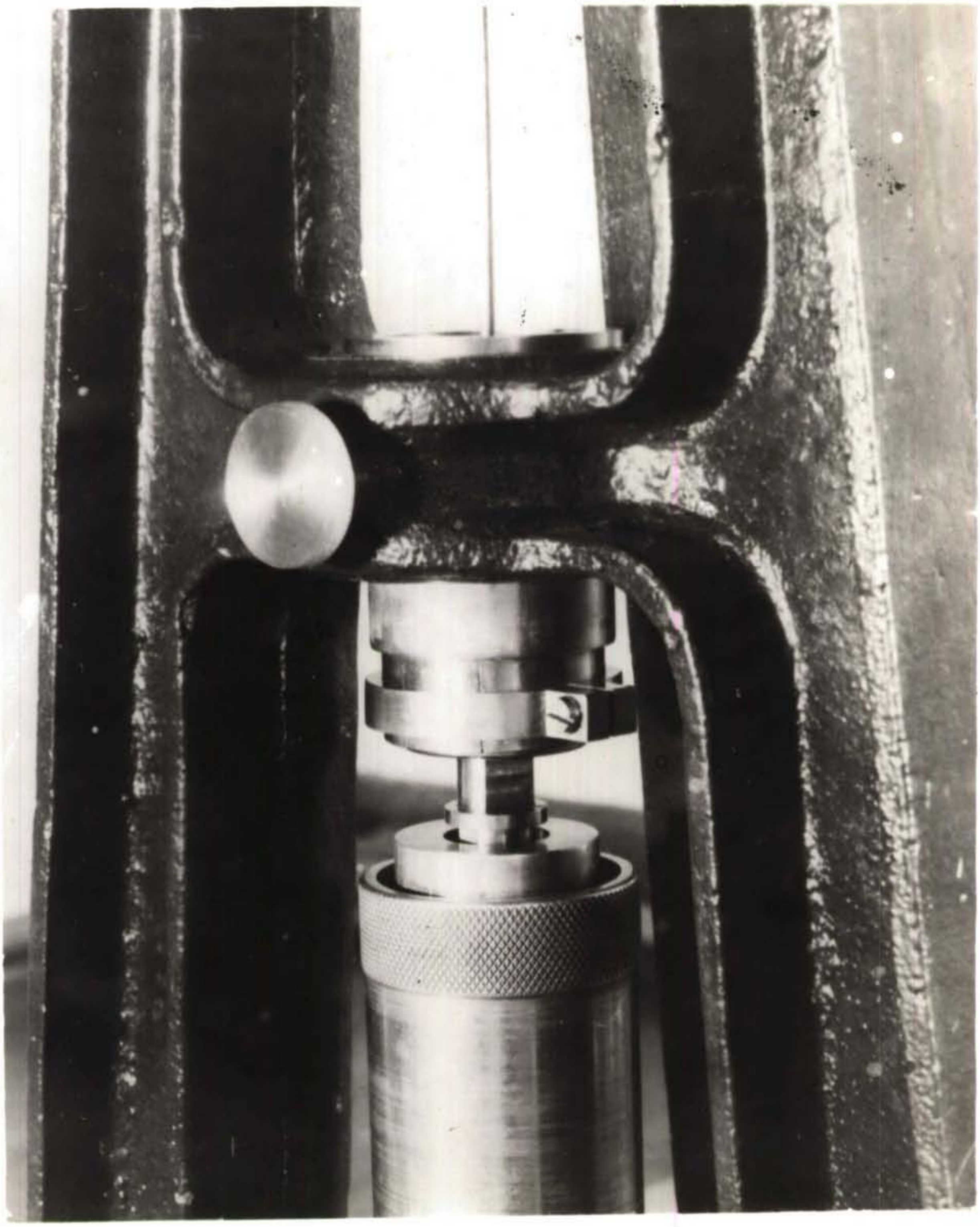


FIG. 3. LOCATION OF THE INNER CYLINDER FOR MEASUREMENTS AT CONSTANT RATE OF SHEAR.

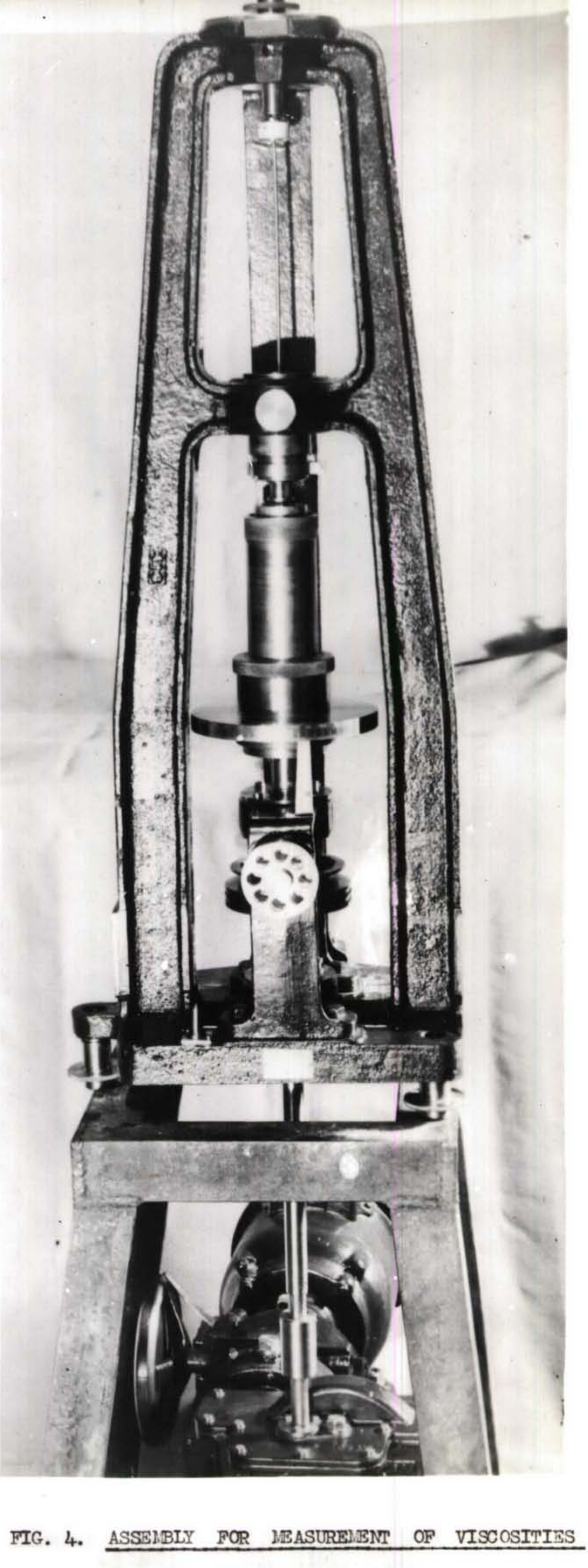


FIG. 4. ASSEMBLY FOR MEASUREMENT OF VISCOSITIES AT CONSTANT RATE OF SHEAR.

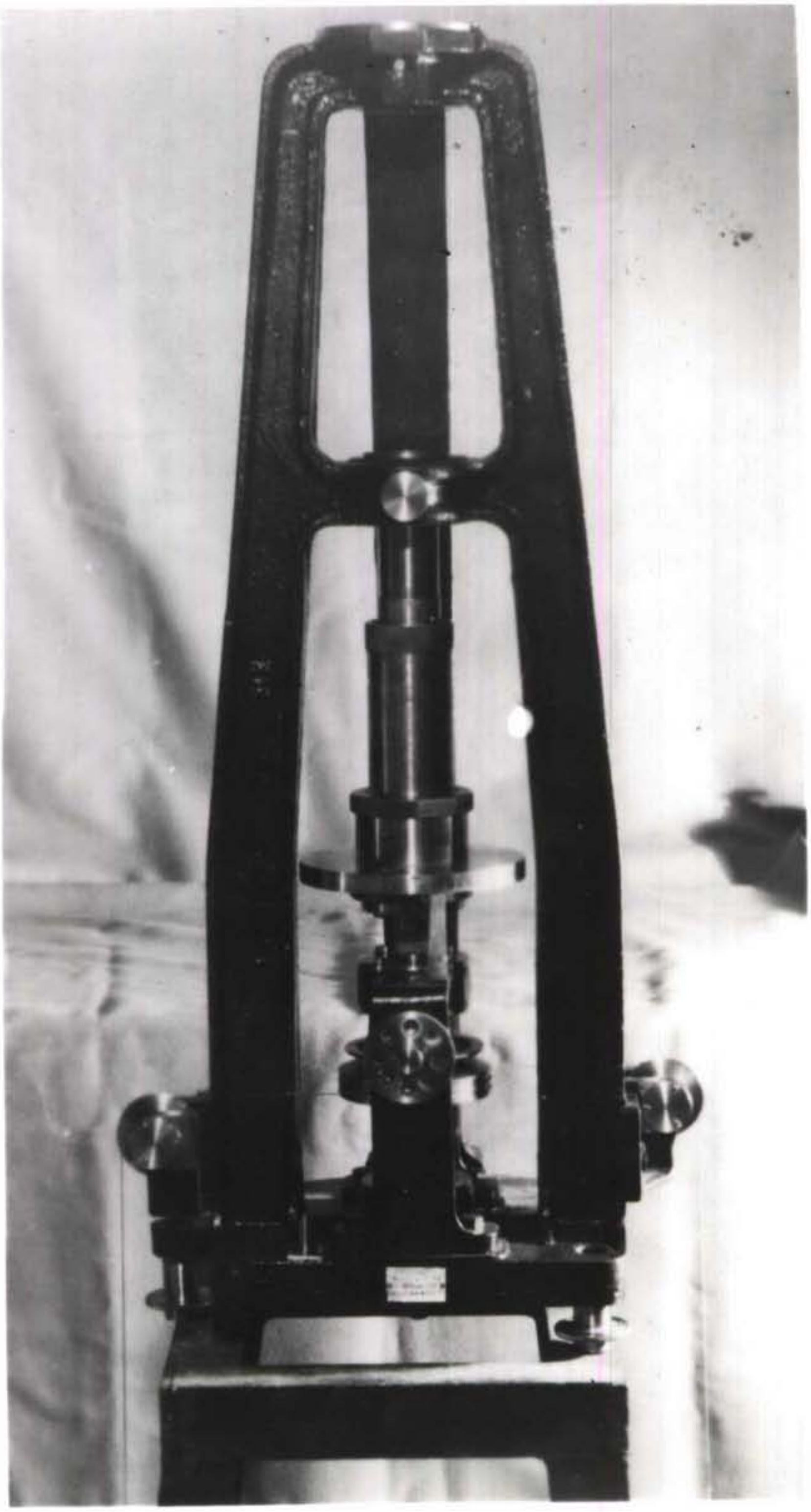
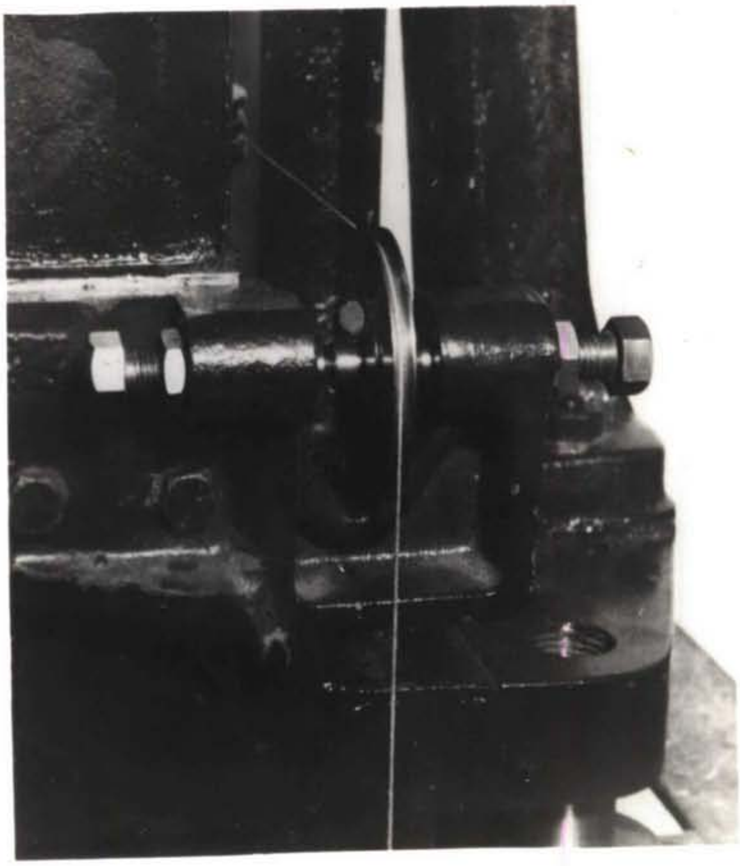
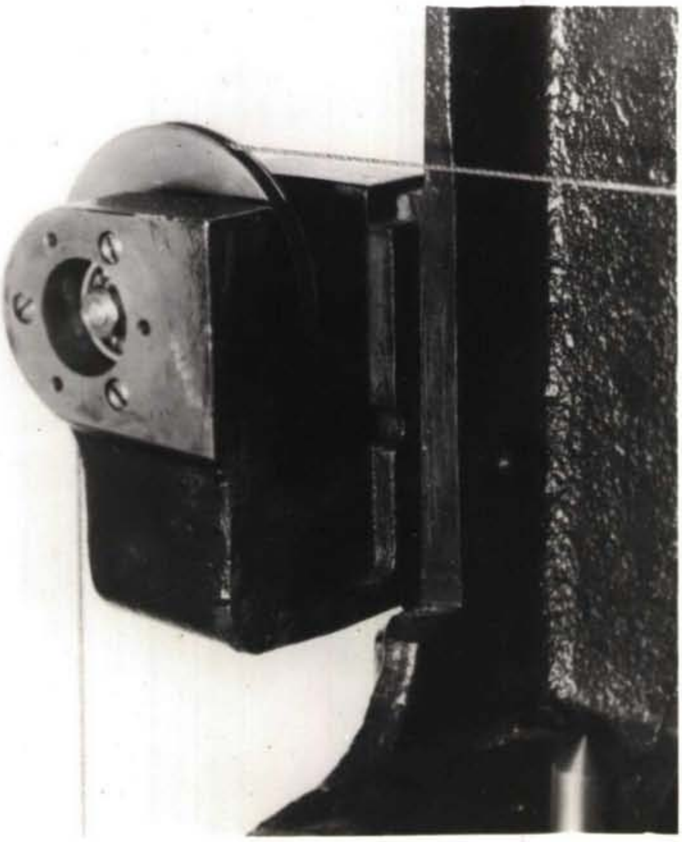


FIG. 5. ASSEMBLY FOR MEASUREMENT OF VISCOSITY AND ELASTICITY AT CONSTANT SHEARING - STRESS



THE LINE - BEARING.



THE US1 - RACE BEARING.

FIG. 6. PULLEY BEARINGS

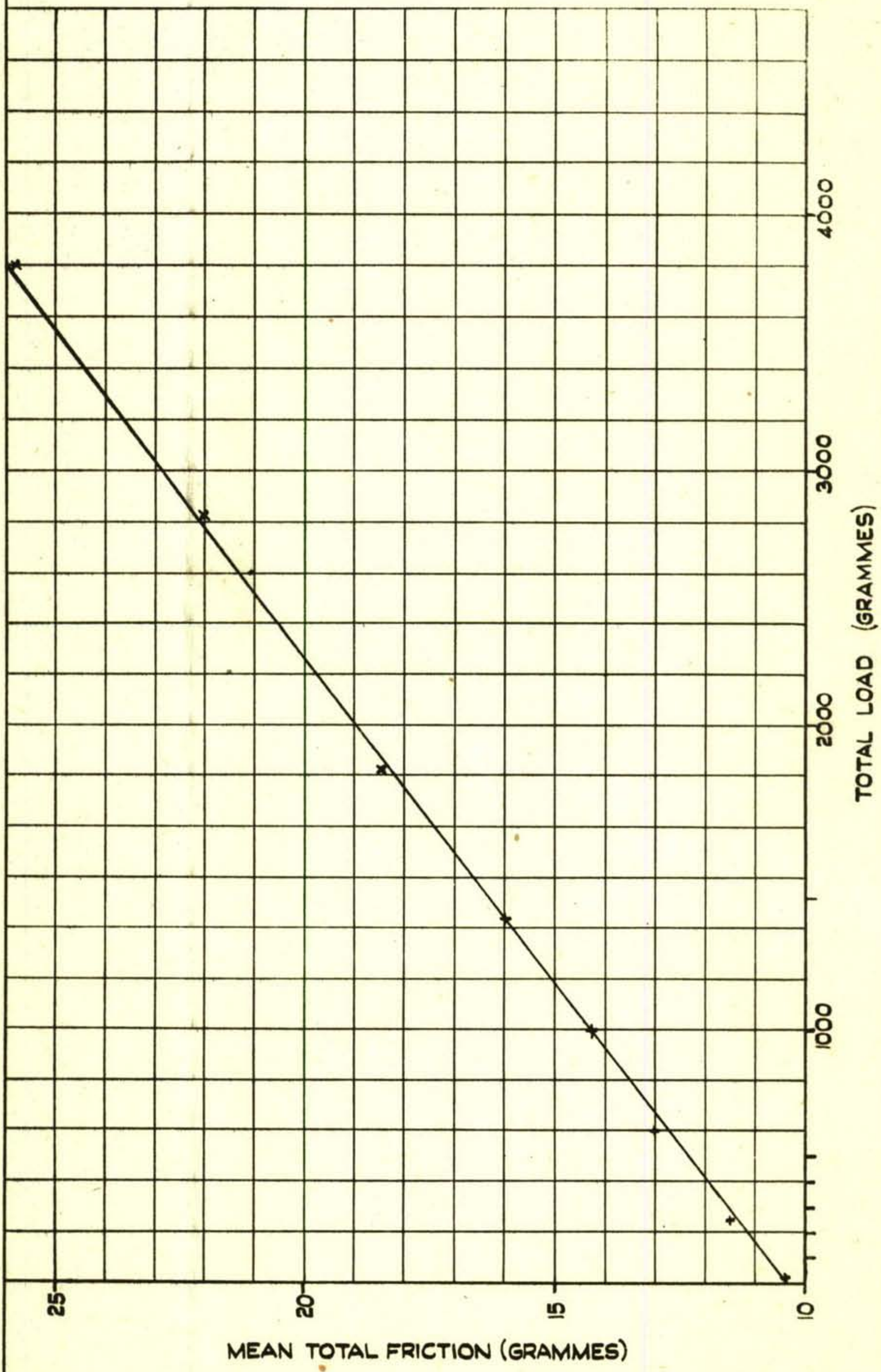
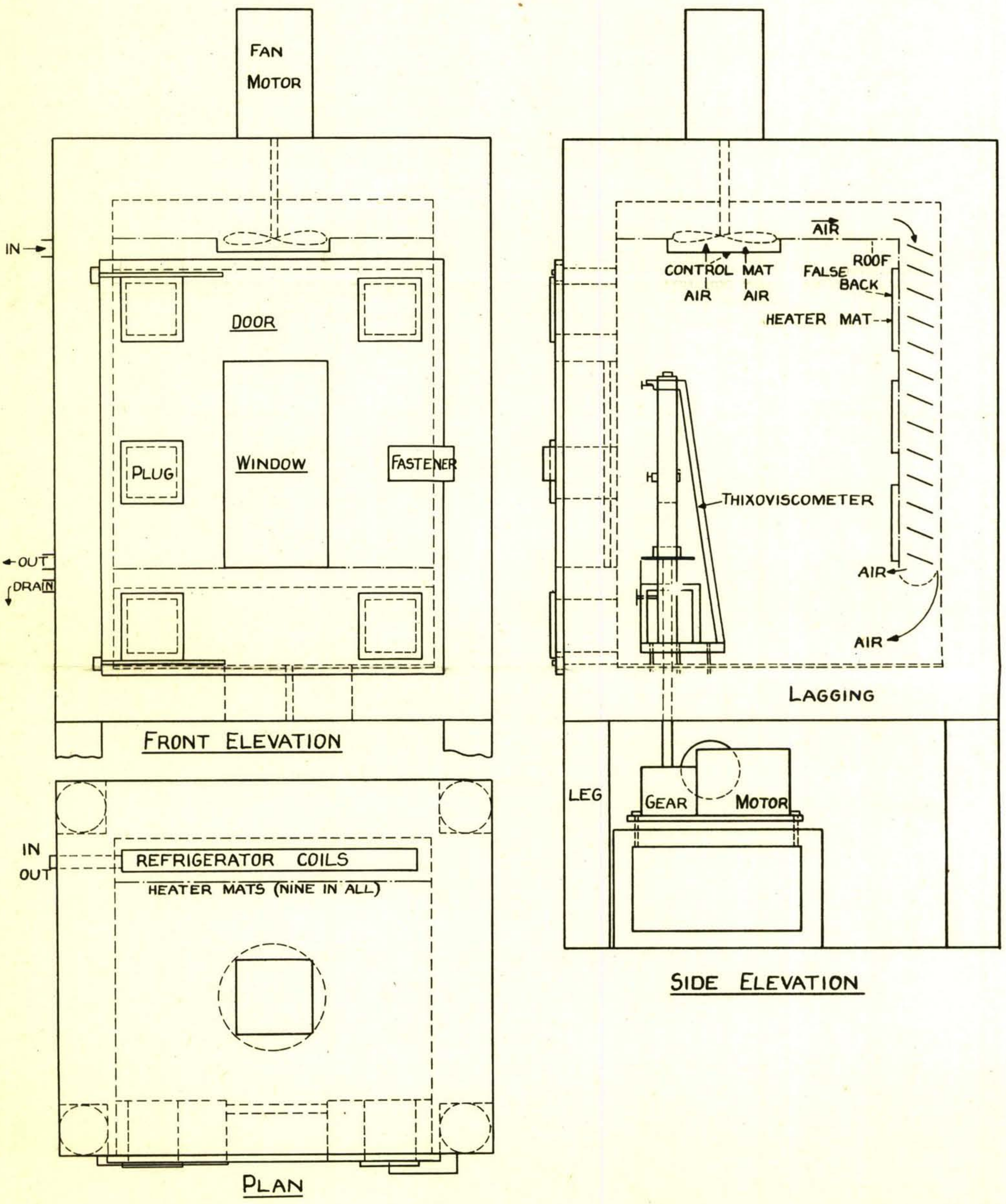


FIG. 7. VARIATION OF TOTAL FRICTION WITH TOTAL LOAD FOR THE THIXOVISCOMETER.

FIG. 8 DIAGRAMS OF AIR THERMOSTAT, RANGE - 30°C TO + 100°C, CONTROL ±0·1°C



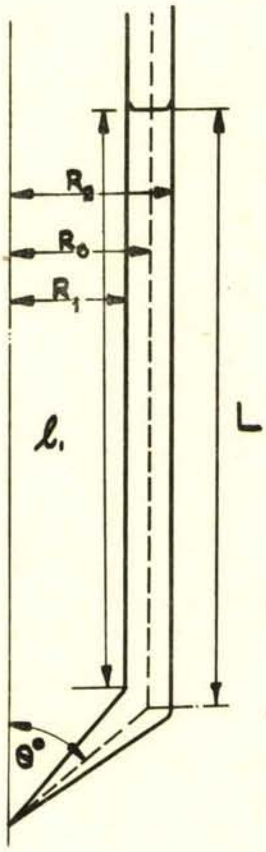
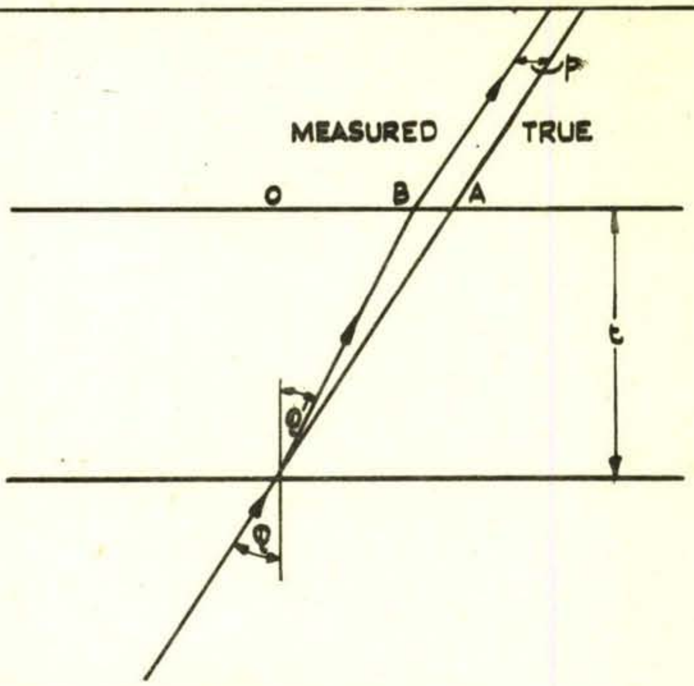


FIG.9. SECTION THROUGH CONICYLINDERS.



$$p = \text{DISPLACEMENT} = OA - OB$$
$$= t(\tan\phi - \tan\phi')$$

$$\text{BUT, } \sin\phi' = \frac{1}{\mu}\sin\phi$$
$$\cos\phi' = \frac{\sqrt{\mu^2 - \sin^2\phi}}{\mu}$$

$$\therefore\ p = t\left(\tan\phi - \frac{\sin\phi}{\sqrt{\mu^2 - \sin^2\phi}}\right)$$

BY MEASUREMENT, $\mu = 1{\cdot}570$

$t = 0{\cdot}80$ CMS.

$$\tan\phi = \frac{\text{SCALE READING(CMS)}}{54{\cdot}8\text{(CMS)}}$$

HENCE p AS A CORRECTION TO MEASURED SCALE READING

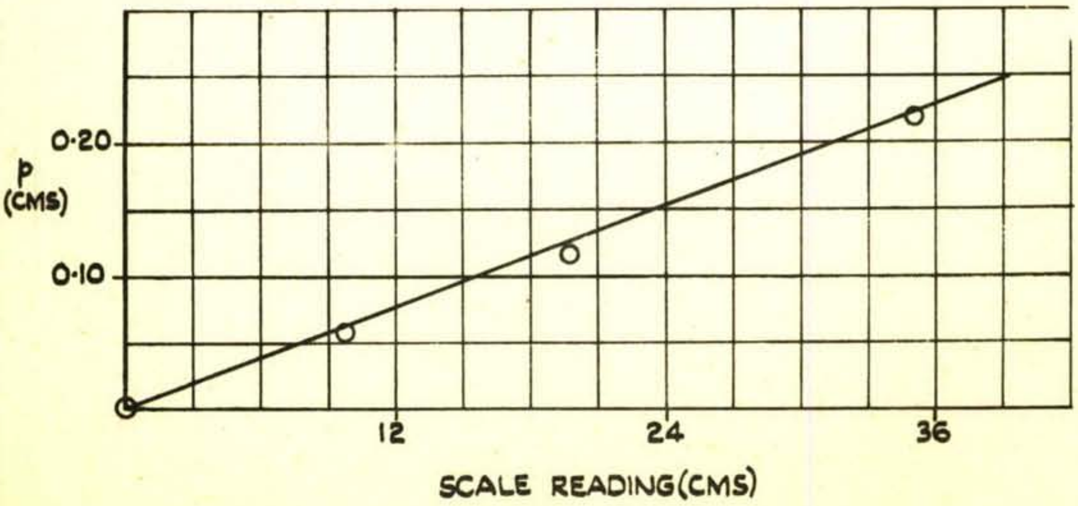


FIG. 10. THE CORRECTION TO THE SCALE READING DUE TO THE THERMOSTAT WINDOW.

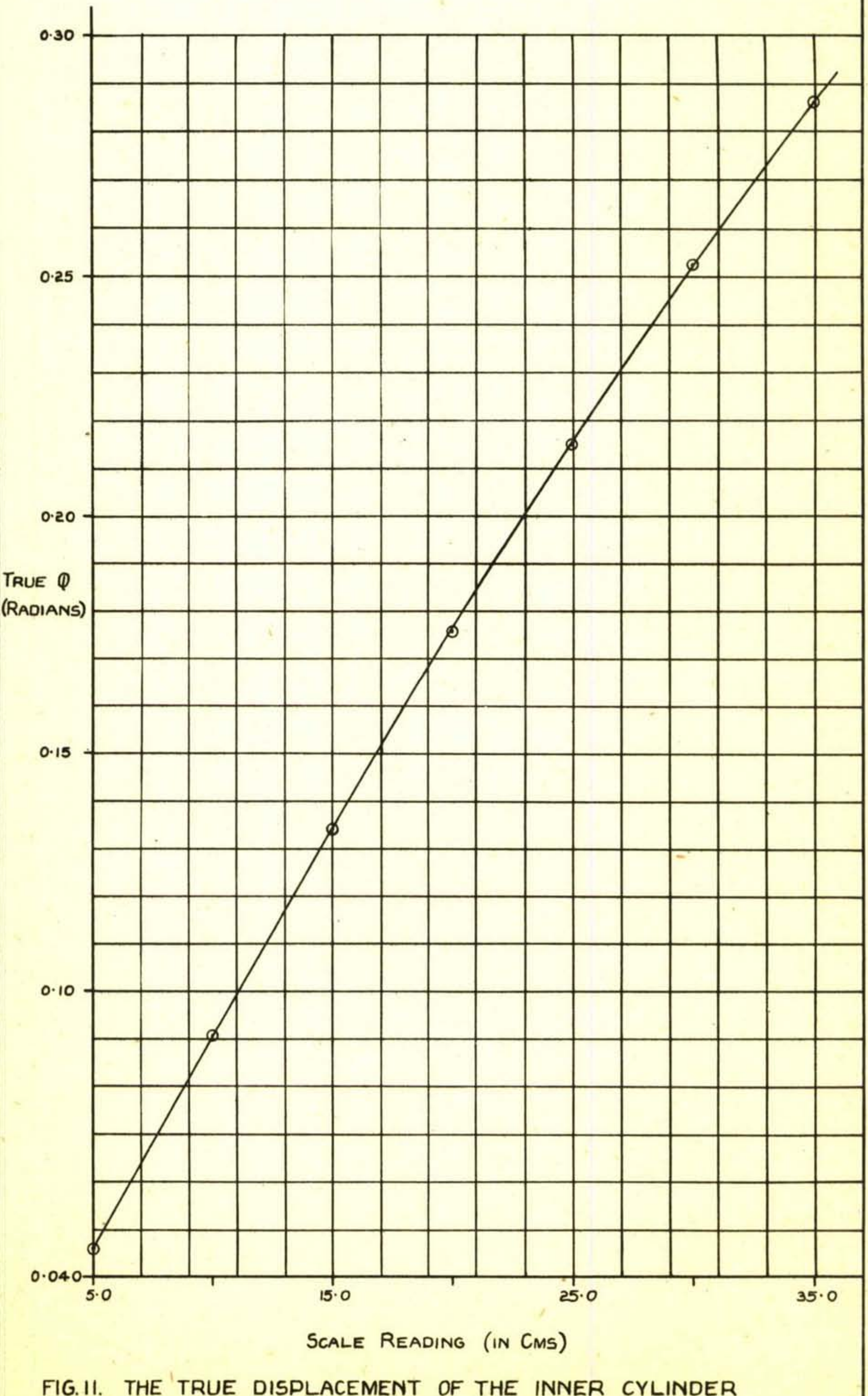


FIG. 11. THE TRUE DISPLACEMENT OF THE INNER CYLINDER WHEN THE MIRROR AND SCALE ARE 54·8 CMS APART.

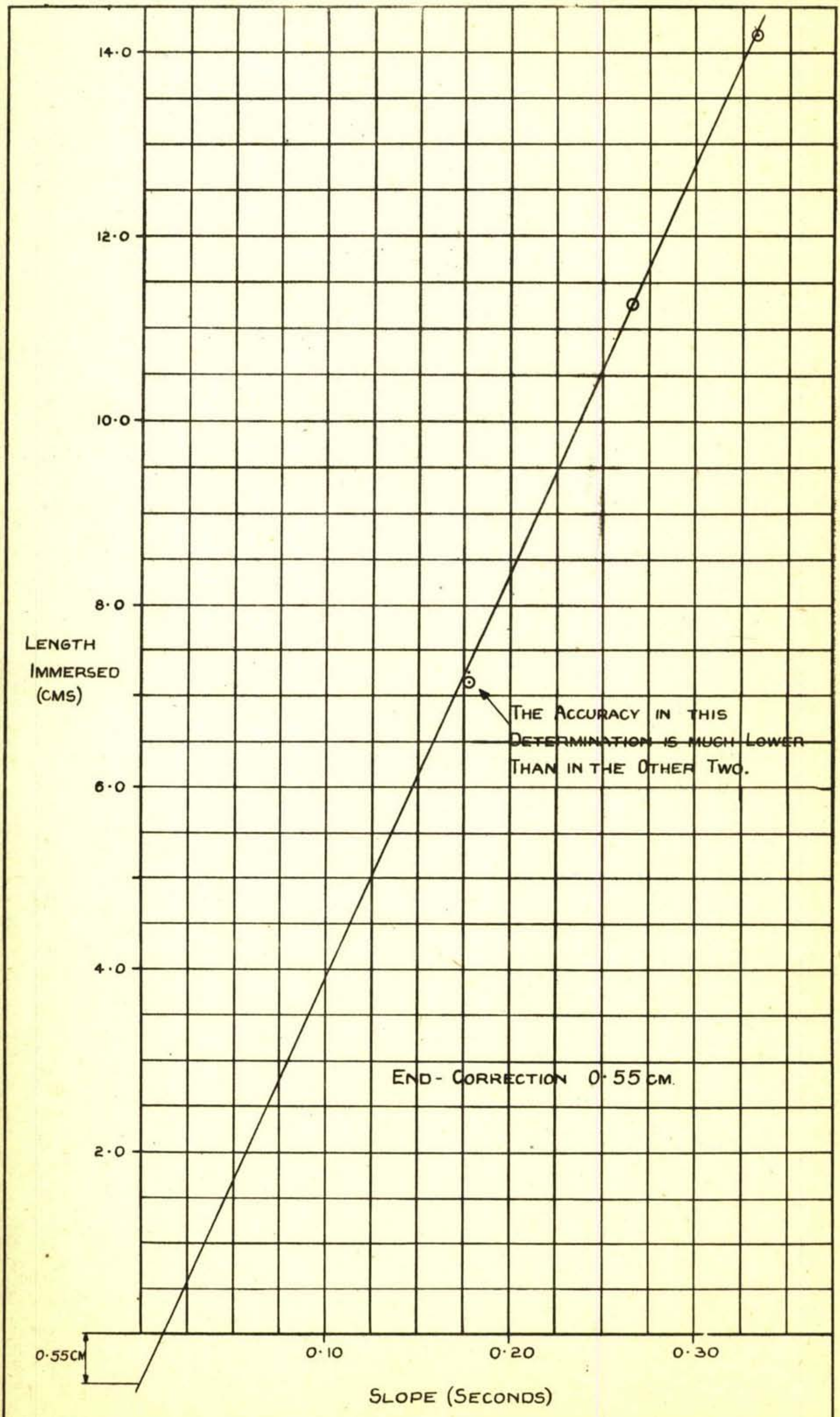


FIG. 12. TO OBTAIN THE END-CORRECTION USING CASTOR-OIL AT 25° C AND THE THINNEST WIRE.