DETERMINATION OF VISCOSITY OF STEAM

BY

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SUMMARY OF THESIS

Title: Determination of Viscosity of Steam

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This thesis forms a record of the experimental work carried out by the author on the measurement of the coefficient of viscosity of steam at atmospheric pressure, in the temperature range 110°C to 1071°C. Two apparatus were designed and developed, both of which were transpiration capillary type viscometers enabling absolute determinations to be made. The design of the apparatus was such that the capillaries were removable, the main advantage of this feature being that the capillary bores could be checked before and after use and that capillaries of different lengths and bores could be introduced.

The first apparatus, which was a single capillary glass viscometer, was developed as a prototype for the second apparatus. Two different capillaries having approximately the same length and nominal bores of 1.0 mm and 1.5 mm were used over the temperature range 110°C to 460°C.

The second apparatus, which was made of quartz, utilised two capillaries in series. Six different sets of capillaries were used, the nominal bores of which were 1.0 mm, 1.3 mm and 1.7 mm; and 1.5 mm, 1.0 mm and 2.0 mm. The latter three sets had a different ratio of the lengths of the capillaries in a set to that of the former three. The apparatus was used over the temperature range 110°C to 1071°C.

The overall estimated accuracy of the determinations is thought to be about ± 1.0 % to 1.25 % or better. Polynomials have been computed using the results of the present work and a fifth degree polynomial of the

form of

$$\eta = a + bt + ct^2 + dt^3 + et^4 + ft^5$$

selected, as the best fit, to represent the viscosity of steam at atmospheric pressure. The standard deviation of the determinations from this equation is ± 1.2 %. A Sutherland equation of the form of

$$\eta = \frac{K\,T^{3/2}}{(C + T)}$$

has been fitted graphically to the data.

The results of this present work have been compared with the results of other workers in this field and agreement is good.

CONTENTS

CONTENTS (Contd.)

CONTENTS (Contd.)

PREFACE

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The results of this present work have been compared with the results of other workers in this field and agree quite favourably.

LIST OF SYMBOLS

Unless otherwise defined in the text, the symbols used in this thesis have the following meanings.

A and a	constants
B and b	constants
C	Sutherland constant
c	constant
D	constant
d	inside diameter of capillary
E and e	constants
F and f	constants
g	acceleration due to gravity
K	constant
l	length of capillary
m	end correction constant
P	absolute pressure
Δp	pressure drop
Q	mass flow rate
r	radius
Re	Reynolds number
t	time or temperature °C
T	temperature °K
V	mean specific volume of the fluid in a capillary
v	velocity
η	coefficient of viscosity
ε	slip correction factor
ρ	density
L	mean free path
ω	angular velocity

Suffices (1) and (2) refer to the downstream and upstream capillaries respectively

LIST OF FIGURES

LIST OF FIGURES (Contd.)

LIST OF FIGURES (Contd.)

Photographs

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Chapter I

Introduction

The physical properties that characterise the resistance to flow of simple (Newtonian) fluids, be they liquid or gas, is the viscosity. A concept of viscosity is the idea that the internal friction between the molecules of the fluid causes adjacent parts in the fluid to move when either of these parts is caused to move. In actual fact this effect is produced by a change of momentum from one layer in the fluid to the adjacent layer, producing a velocity gradient within a fluid moving between finite boundaries. The resistance of the fluid to develop a velocity gradient is the essential feature of viscosity and forms the quantitative assessment of the viscosity.

The viscosity is one of the properties of a fluid termed the "transport properties", the others being the diffusion and the thermal conductivity, all of which are physically similar in that they involve the transport of some physical property through the fluid.

In a simple fluid, the resisting force produced by the fluid flow is proportional to the velocity gradient and it may be defined by the equation

$$F_{xy} = \eta \left(\frac{\delta v_x}{\delta y} \right) , \ldots\ldots\ldots\ldots \quad 1.1$$

where F_{xy} is the shearing stress between adjacent parallel layers in a fluid having a dynamic viscosity η and a velocity gradient of $\delta v_x/\delta y$ normal to the flow axis.

This definition, which was first propounded by Newton, is incomplete since it only applies to steady, laminar flow and does not apply for low rates of shear.

The equation of motion for a homogeneous fluid can be represented by

$$\rho \frac{Dv}{Dt} = -\nabla p - \nabla.\tau + \rho.g \quad \ldots \ldots\ldots\ldots\ldots \; 1.2$$

where the normal stress for Newtonian fluids can be represented by

$$\begin{aligned} \tau_{xx} &= -2\eta \frac{v_x}{x} + \left[2\eta/3 - k \right] (\nabla.v) \\ \tau_{xy} = \tau_{yx} &= -\eta \left[\frac{\partial v_x}{\partial y} + \frac{\partial v_y}{\partial x} \right] \quad \ldots\ldots\ldots\ldots \; 1.3 \\ \tau_{zx} = \tau_{xz} &= -\eta \left[\frac{\partial v_z}{\partial x} + \frac{\partial v_x}{\partial z} \right] \end{aligned}$$

where η is the dynamic or first viscosity and k is the 2nd coefficient of viscosity or bulk viscosity. From equations (1.2) and (1.3), the equation for the x direction can be written as

$$\rho \frac{Dv_x}{Dt} = \rho.g - \frac{\partial p}{\partial x} + \frac{\partial}{\partial x} \left\{ 2\eta \frac{\partial v_x}{\partial x} - \left(2\eta/3 - k \right) (\nabla.v) \right\} + \frac{\partial}{\partial y} \left\{ \eta \left(\frac{\partial v_x}{\partial y} + \frac{\partial v_y}{\partial x} \right) \right\} + \frac{\partial}{\partial z} \left\{ \eta \left(\frac{\partial v_z}{\partial x} + \frac{\partial v_x}{\partial z} \right) \right\}$$

and similarly for the y and z directions. 1.4

Therefore the three basic hydrodynamic equations may be written,

$$\begin{aligned} \rho \frac{Dv_x}{Dt} &= \rho.g - \frac{\partial p}{\partial x} + \eta \nabla^2 v_x + \left(\frac{2}{3}\eta - k \right) \frac{\partial}{\partial x} (\nabla.v) \\ \rho \frac{Dv_y}{Dt} &= \rho.g + \frac{\partial p}{\partial y} + \eta \nabla^2 v_y + \left(\frac{2}{3}\eta - k \right) \frac{\partial}{\partial y} (\nabla.v) \qquad 1.5 \\ \rho \frac{Dv_z}{Dt} &= \rho.g + \frac{\partial p}{\partial z} + \eta \nabla^2 v_z + \left(\frac{2}{3}\eta - k \right) \frac{\partial}{\partial z} (\nabla.v) \end{aligned}$$

These equations along with the equation of state $p = p(\rho)$, the density dependence of viscosity $\eta = \eta(\rho)$ and the boundary and initial conditions, determine completely the pressure, density and velocity components in a flowing isothermal fluid.

These equations in their complete form are seldom used to set up flow problems. It is more usual to use restricted forms of the equations of motion to suit particular requirements.

The bulk viscosity 'k' is identically zero for low density monotomic gases and probably not too important in dense gases and liquids. For constant density ρ and viscosity η, the flow equation becomes

$$\rho\, D.v = -\nabla.p + \eta\nabla^2.v + \rho\, g \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1.6)$$

and for continuity $\nabla.v = 0$.

This is the well known Navier-Stokes equation, initially developed by Navier (1) in 1822 using molecular arguments and by Stokes (2) based on Newton's hypothesis.

In terms of cylindrical co-ordinates, which are the most convenient forms to use for the majority of problems

$$\rho \left\{ \frac{\partial v_r}{\partial t} + v_r.\frac{\partial v_r}{\partial r} + \frac{v_\theta}{r}\frac{\partial v_r}{\partial \theta} - \frac{v_\theta^2}{r} + v_z\frac{\partial v_r}{\partial z} \right.$$

$$= -\frac{\partial p}{\partial r} + \eta \left\{ \frac{\partial}{\partial r}\left(\frac{1}{r}\frac{\partial}{\partial r}(rv_r) \right. + \frac{1}{r^2}\frac{\partial^2 v_r}{\partial \theta^2} - \frac{2}{r^2}\frac{\partial v_\theta}{\partial \theta} + \frac{\partial^2 v_r}{\partial z^2} \right\} + \rho.g_r$$

$$\rho \ [\frac{\partial v_\theta}{\partial t} + v_r . \frac{\partial v_\theta}{\partial r} + \frac{v_\theta}{r}\frac{\partial v_\theta}{\partial \theta} + \frac{v_r}{r} v_\theta + v_z \frac{\partial v_\theta}{\partial v_z}] =$$

$$= -\frac{1}{r}\frac{\partial p}{\partial \theta} + \eta \ [\frac{1}{r} \frac{\partial}{\partial r}(r v_\theta) + \frac{1}{r^2}\frac{\partial^2 v_\theta}{\partial \theta^2} + \frac{2}{r^2} \frac{\partial v_r}{\partial \theta} + \frac{\partial^2 v_\theta}{\partial z^2}]$$

$$\rho \ [\frac{\partial v_z}{\partial t} + v_r . \frac{\partial v_z}{\partial r} + \frac{v_\theta}{r}\frac{\partial v_z}{\partial \theta} + v_z \frac{\partial v_z}{\partial z}]$$

$$= -\frac{\partial p}{\partial z} + \eta \ [\frac{1}{r}\frac{\partial}{\partial r} (r \frac{\partial v_z}{\partial r}) + \frac{1}{r^2}\frac{\partial^2 v_z}{\partial \theta^2} + \frac{\partial^2 v_z}{\partial z^2}] + \rho . g_z$$

.... 1.7

Kinetic Theory for Dilute Gases

A brief resume of the relevant kinetic theory is given in this section. It is not the author's intention to describe in detail the developments in the theory of viscosity, but merely to point out the more important relevant data based on the kinetic theory of low density gases. During the past century the kinetic theory of the transport phenomena of dilute gases has been developed in some considerable detail. A historical summary is given by Chapman and Cowing (3), and a thorough account of the molecular theory of liquids and gases is given by Hirschfelder, Curtiss and Bird (4).

Maxwell (5) in 1859 was the first of several workers to concern himself with the mechanism of viscosity and its connection with the kinetic theory. The general solution derived was

$$\eta = \frac{\rho \ v \ L}{3} \quad \text{.................................... 1.8}$$

where L denotes the mean free path of the molecules and v their mean velocity. The assumptions made are those of the ideal case of a gas whose molecules are perfectly elastic spheres and molecular forces are neglected. Maxwell's expression for the viscosity predicted a dependence on the square root of the absolute temperature ($\propto T^{1/2}$) which showed it was not a function of the density.

For forceless spherical molecules with a Maxwellian velocity distribution Tait obtained an expression,

$$\eta = 0.064 \frac{m (v^2)^{1/2}}{(2a)^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 1.9$$

where m is the molecular mass, 'a' is the molecular radius and $(v^2)^{1/2}$ the root mean square velocity.

The simple Maxwell equation fails in practice. In actual fact, it has been found experimentally that η does not vary as the square root of the absolute temperature, but to a higher power.

In the absence of definite knowledge, it is quite often assumed that the force between two molecules is proportional to r^{-s}. It can be proved by methods of similitude or by dimensional analysis that

$$\eta = T^n \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 1.10$$

where, $n = \frac{1}{2} + \left\{\frac{2}{s - 1}\right\}$

and provided that $s > 1$, then n will be greater than 1/2. Hence, another way of expressing this equation is

$$\eta_T = \eta_o \left\{ T/T_o \right\}^n , \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 1.11$$

where η_T and η_o are the viscosities at temperatures T and To. This has been found to be tolerably accurate over small temperature ranges for some gases. This is not so significant, since any curve whatever, can be fitted to some power of the independent variable, as accurately as desired over a sufficiently short range of values.

In 1881 Boltzmann (6) derived an integro-differential equation for the distribution function, in terms of inter-molecular collisions, now known as the Boltzmann equation. This equation forms essentially, the basis of the modern kinetic theory of dilute gases. Although Boltzmann and Maxwell obtained approximate solutions to the Boltzmann equation, they could not obtain general solutions. Later, Chapman (7) and Enskog (8) obtained, almost simultaneously, using different methods, a general solution for the equation.

The basic theory has been further developed, using slightly different assumptions, by Sutherland (9), Stockmeyer (10) and Lennard-Jones (11), to mention the more prominent workers.

The previous basic theory assumed that the molecules attract or repel with a force proportional to r^{-s}. Sutherland (9) retained the assumption of hard spheres but added a weak attraction between them, falling off rather rapidly with distance. He decided that

$$\sigma = \sigma_{\infty} \left[1 + \frac{C}{T} \right] \text{ .... } 1.12$$

where σ and σ_{∞} are the molecular diameters at temperatures T and respectively and C is a constant. From the above, he obtained the well known equation,

$$\eta = \frac{k\,T^{1/2}}{\left\{ 1 + \frac{C}{T} \right\}} \text{ .... } 1.13$$

Lennard-Jones (9), taking both softness and the mutual attraction of the molecules into account, arrived at the equation

$$\eta = \frac{k\ T^{3/2}}{\left[C + T^{\nu-3/\nu-1} \right]} = \frac{k\ T^{3/2}}{\left[C + T^{a} \right]} \quad \ldots\ldots\ldots \ 1.14$$

where C is the Sutherland constant and 'a' and ν are constants.

For single non-polar gases, an intermolecular potential of the Lennard-Jones type has been used by several workers

$$E\ (r) = 4\ \epsilon\ \left[\left(\frac{ro}{r} \right)^{12} - \left(\frac{ro}{r} \right)^{6} \right] \quad \ldots\ldots\ldots \ 1.15$$

where r is the separation between the molecules, ϵ is the maximum energy of attraction and ro is the low velocity collision diameter (i.e. the separation for which the energy of interaction is zero). The expression assumes non-polar gases to be composed of spherical molecules which attract according to the 6th power of their separation and repel according to the 12th.

For non-polar gases the parameter ϵ and ro may be determined from a knowledge of the viscosity or second virial coefficients at two temperatures.

Hirschfelder, Bird and Spotz (12) using the same potential model for non-polar gases expressed the first approximation of the viscosity as

$$\eta = \frac{266.93 \times 10^{-7}\ (MT)^{1/2}}{(r_o)^2 \cdot \Omega\,(2,2)} \cdot f_\eta \quad \ldots\ldots\ldots\ldots \ 1.16$$

where M is the molecular weight, T is the temperature in °K, r_o is the low energy collision diameter and f_η and $\Omega\,(2,2)$ are functions of $\frac{KT}{\epsilon}$ to be worked out for each potential, and K is the Boltzmann constant. Their findings showed that the temperature dependence of the viscosity cannot be represented by the Sutherland equation over a large range of temperature. They inferred that Sutherland's equation should be re-written as

$$\eta = \frac{K^1 T^{3/2}}{\left[1 + \frac{KT}{\epsilon}\right]} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 1.17$$

where K is the Boltzmann constant and K^1 is another constant.

Stockmayer (10) showed that for polar gases the energy of interaction between a pair of molecules can be expressed by

$$E(r) = 4\epsilon\left[\left(\frac{ro}{r}\right)^{12} - \left(\frac{ro}{r}\right)^{6}\right] - \left[\frac{\mu^2}{r^3}\right]\zeta \ \ldots\ldots 1.18$$

where $\zeta = 2\cos\theta_1\cos\theta_2 - \sin\theta_1\sin\theta_2\cos\phi_1$

μ is the dipose moment of the molecule and ζ is an orientation factor in which θ_1 and θ_2 are the angles of inclination of the two dipole axes to the intermolecular axis and ϕ is the azimuthal angle between them. Since ζ is quite complex, a value of 2 is assumed, i.e. the dipoles are in an attractive end-on position and maintain their minimum attractive energy value of $-\ \mu^2 / r^3$ throughout their encounter. The equation can therefore be expressed in the form

$$E\,r = 4\epsilon\left[\left(\frac{r_o}{r}\right)^{12} - \left(\frac{r_o}{r}\right)^{6} - \delta\left(\frac{r_o}{r}\right)^{3}\right] \ \ldots\ldots 1.19$$

where δ is a pure number called, after Rowlinson (13), the "reduced dipole energy" and is defined by the relation $\delta = \mu^2 / 2\epsilon r_o^3$.

Using the same treatment as given by Hirschfelder, Bird and Spotz, the viscosity is found to be given by

$$\eta = \frac{266.93 \times 10^{-7}\,(M\,T)^{1/2}}{(r_o)^2\,\Omega\,(2,2)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 1.20$$

The majority of theoretical calculations have been made with the Lennard-Jones 12 - 6 potential or the modified Buckingham exp - 6 potential,

$$E\ (r) = \frac{\epsilon}{(1 - \frac{6}{\alpha})} \quad \frac{6}{\alpha} \exp. \quad \left[\alpha (1 - \frac{r}{r_m}) \right] - \left[\frac{r_m}{r} \right]^6 \quad \ldots .1.21$$

when $r > r$ max

and $E\ (r) = \infty$ when $r < r$ max.

and ϵ is the minimum potential energy, r_m is the separation at the position of minimum potential energy, α is a measure of the steepness of the repulsive potential and r_{max} is the maximum in the first of the above two equations.

To summarize the above resume, equations (1.16) and (1.12) have been used with some success for both polar and non-polar gases. However, the author cannot observe any immediate advantage in using these equations to fit the expected viscosity results. To quote Whalley (14), who investigated a number of gases, "the Sutherland equation provides a better fit over a wide temperature range than the exact Sutherland (12:6) or Exp. 6 potentials". Whalley also pointed out that several workers had shown that a (12:6) potential is not good enough for the viscosity of many gases at the higher temperatures. Kihara and Kotani (15) showed that for N_2 and CH_4 the Sutherland potential is better than the 12:6) potential, the approximate equation being even better.

Krieger (16) using an equation of the form of (1.20), and using the viscosity results of Braune and Linke (17), Smith (18), Shugajew (19), Timrot (20) and Keyes (21) computed values for the viscosity of steam up to 3000°K. The values are about 2 % higher, at 800°C, than the experimental results obtained by Shifrin, but do show a reasonable agreement. Krieger's work was based on "end-on" collision and did not take into account angular momentum effects.

Keyes (21) used a modified Sutherland equation of the form

$$\eta = \frac{a_o T^{1/2}}{1 + \left(\frac{a \tau}{10^{a_1 \tau}} \right)} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 1.22$$

where $\tau = T^{-1}$, a and a_1 are constants, to correlate successfully the viscosities of a number of simple gases over a wide temperature range. At higher temperatures equation (1.22) reduces to the basic Sutherland equation.

It has been argued that the value of Sutherland constant 'C' varies with temperature; Bonilla, Wang and Weiner (22) fitted curves of the form

$$\eta = \frac{T^{3/2}}{a + b\,T} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 1.23$$

for the temperature range 200°C to 1200°C and

$$\eta = \frac{T^{3/2}}{a_{1/T} + b_1 T} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 1.24$$

for the temperature range 1030°C to 1300°C, where a, a_1, b and b_1 are constants.

It is therefore expected that equations of the Sutherland or modified Sutherland type and a polynomial in T will be fitted in the first instance to the results expected from this present work. If it is thought to be necessary or worthwhile, equations of the form (1.16) and (1.20) might be investigated.

Chapter II

Literature Survey

Only work relevant to the viscosity of steam at atmospheric pressure is to be discussed here and the author wishes to refer the reader to excellent literature surveys for steam and water given in Ph.D. theses by Kjelland-Fosterud (23), Whitelaw (24) and Ray (25), also a paper by Liley (26).

In 1924 Smith (18) obtained results for superheated vapour at pressures well below atmospheric and in the temperature range 100°C to 260°C. The apparatus used was based on the O.A. Rankine (27) design. No allowance was made for the effect of flow and the associated pressure drop correction, at the entrance to and exit from the capillary; therefore, the results may be expected to be up to 0.5 % high. The experimental accuracy appears to be not better than ± 1 %, making an overall accuracy of not better than ± 2 %. It can be seen from the deviation plot, (figure 2.1), that the results vary from 2.75 % to 1 % above those of Shifrin (28) over Smith's temperature range, which is within the latter's accuracy band.

In 1924 Trautz and Weizel (29) also obtained results for superheated steam at atmospheric pressure over the temperature range 100°C to 400°C. In the viscometer the fluid was driven through a capillary under a decreasing mercury head, the time to sweep out a fixed volume being measured. A preliminary calibration was made to obtain absolute results. It is difficult to estimate the accuracy of these results but with reference to the deviation plot, figure (2.1), it can be seen that these results deviate from those of Shifrin

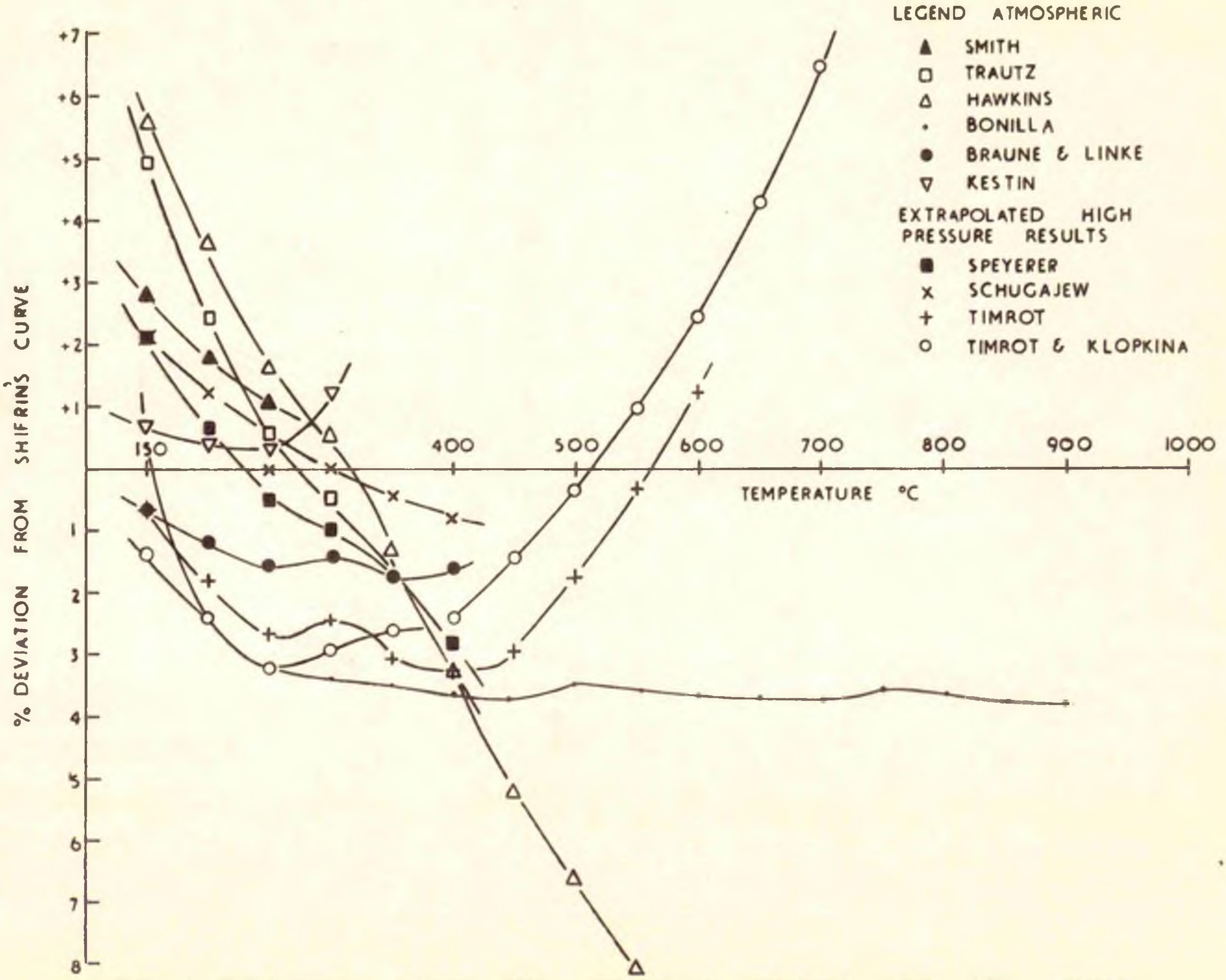


FIG 1.1 DEVIATION PLOT FOR RELEVANT RESULTS FOR 1AT CURVE

by + 5 % to -3 %.

Speyerer (30) in 1925, made the first investigation of the pressure dependence of the viscosity of steam. His apparatus was an open circuit capillary type viscometer. The capillary being made of brass and divided into three sections. The sections were connected by sleeves having annular slots in them. Pressure tappings were made at the annular slots. This arrangement was made to avoid the need to use end corrections; the sections, over which the pressure drop was measured, being assessed to have fully developed Poiseuille flow within it. Speyerer assumed that the slots would have no effect on the flow, an assumption which is open to considerable doubt. He claims an accuracy on the results over the ranges 100°C to 350°C and 1 to 10 atmospheres of 4 % for the reduced pressure results. However the experimental scatter is up to 10 % from the mean and the results therefore do not appear to be reliable. These discrepancies may be due to fluctuations in pressure and flow and/or errors in pressure measurement due to poor location of the Steam/water interface.

Braune and Linke (17) obtained values for the viscosity of steam in the range 20°C to 400°C at pressures below atmospheric using an oscillating disc viscometer. The results were of a secondary nature, the apparatus being calibrated with air at 17°C. The accuracy of the results may be considered to be of the order of 2 %. The pressures used were in the range 14 mm to 210 mm of mercury, the pressure effect being negligibly small.

Later in 1934 Shugajew (19) used a Rankine (27) type viscometer to measure the viscosity of steam in the ranges 1 to 93 atmospheres and 100°C to 400°C. The capillary used was 27 cm long and was calibrated with air; there was no correction for compressibility or slip. There was a large scatter of results at the higher temperatures which may have been caused by the use of tin as the displacing medium. The accuracy claimed by the author is ± 1 % at 100°C, ± 3 % at 350°C and ± 6.5 % at 400°C. Because of the large scatter Shugajew concluded that the pressure dependence of the viscosity could not be determined from his experiments. Further results obtained from the apparatus by Schugajew and Sorokin (31) must also be concluded to be unreliable at the higher temperatures, since, as before, the scatter was too large. The deviation plot shows that the results vary between + 2 % to - 1 % from those of Shifrin (28).

Hawkins, Selberg and Potter (32) in 1935 obtained results in the range 1 - 230 atmospheres and 320°F to 1009°F. The viscometer used was of the Lavaczeck (33) type, using falling bodies of various shapes. These results were superseded in 1939 (34) by additional results obtained by the same authors using a capillary viscometer. The discrepancies in the 1935 results were attributed to high Reynolds' numbers and incorrect assumptions in the development of the theory. There has been no attempt to correct the initial results although Shugajew suggested that recalculation of the 1935 results could be carried out by introducing an end correction. The approach does not appear to be sufficiently rigorous. The 1939 results covered the ranges 5 to 120 atmospheres and 180°C to 550°C. The capillary used was coiled, being 103.2 ft. long and 0.09294 in bore. These sizes were used to make the end effects negligible and make it possible to measure the pressure drop accurately. The system was a secondary open circuit type in which the steam was supplied from a boiler and collected as condensate in the usual way, the capillary being calibrated using water and nitrogen. The scatter on the results was as much as ± 10 % at the highest pressures and consequently do not appear to be reliable. Any discrepancies may be due to instability in temperature, pressure and Reynolds numbers. There was no correction made for thermal expansion and there may also be errors in the assumptions made for coiled capillary theory. The higher pressure results, extrapolated to atmospheric pressure, vary as much as + 6 % to - 8 % when compared with Shifrin's equation, see fig.

In 1940 Timrot (20) published results for steam in the ranges of 93 to 352 atmospheres and 19°C to 601°C. The apparatus used was a modified Rankine viscometer using a platinum capillary 38.45 cm long and 0.04224 cm. bore. The pressure drop was obtained using a ring balance manometer containing mercury. The ring balance also served as a driving mechanism. No indication was made of the method used to determine the bore of the tube, which was given to rather a high accuracy. The accuracy of the results was quoted as 2 % up to 250°C and 3 % at higher temperatures. The higher pressure results, extrapolated to atmospheric pressure, are plotted on a deviation graph, fig. (2.1) and show a deviation from Shifrin's equation of - 3 % to + 1 %. Vukalovich (35) used Timrot's work as a basis for his viscosity tables. Later in 1954 Timrot and Klopkina (36) published results for steam in the ranges 17° to 700°C and 28 to 600 atmospheres, using essentially the same apparatus. The estimated accuracy given was ± 3 %. The extrapolated results for the one atmosphere curve are shown on the deviation plot and the differences can be seen to be approximately - 3 % to + 6.5 %, when compared with Shifrin's equation.

Two papers were published by Bonilla on his results for the viscosity of steam at atmospheric pressure up to very high temperatures. The first, by Bonilla, Brooks and Walker (37) in 1954 covered the temperature range 450° to 1200°C. Although the accuracy was not stated the mean deviation of each point from the average of each group of readings was 0.65 %. An equation was fitted to the results of the form

$$\eta = \frac{T^{3/2}}{a + bT}$$ where a and b are constants,

yielding,

$$\eta = \frac{T^{3/2}}{46.623 + 0.040989T}, \quad T \text{ in } {}^0\text{K and } \eta \text{ in } \mu\text{poise.}$$

The experimental information was sparse and therefore a critical analysis is not possible. However, their results for nitrogen and argon agree closely with those of Vasilesco (38) and this is to their credit since Vasilesco's work is considered to be very good. The apparatus was an efflux type, having a coiled capillary of platinum approximately 0.03" bore and 30.4" long in a 1.125" diameter helix. The system was calibrated with air. White's (39) correction for coiling and corrections for slipping and end effects were applied. The second paper, by Bonilla, Wang and Weiner (22), using the same type of apparatus but covering the range 0 to 1500°C was published in 1954. The mean deviation from the average for each group was 0.2 % to 0.3 %, the results agreeing to within 1 % to their previous equation. From the deviation plot it can be seen that up to 900°C the discrepancy of these results to those of Shifrin is approximately - 3.6 %. A new capillary was used for this apparatus, made of 90 % platinum, 10 % iridium alloy, the bore of which was .025" and the length 28.5". Shifrin is of the opinion that the difference between their results is due to an incorrect assumption in the spiral capillary theory used by Bonilla. Only more work in this particular field can help to assess who is correct.

In 1959 Shifrin (28) published results for steam at atmospheric pressure and temperatures in the range 149°C to 866°C. The apparatus employed was a capillary viscometer in which two capillaries were used in series. The capillaries were made of glass for the lower temperatures and quartz for the higher temperatures. The system was a standard type in which the steam generated in a boiler was passed via a superheater to the capillaries, the condensate being collected and weighed on leaving the viscometer. Two sets of capillaries were used, the glass capillaries had bore of 0.874 mm.

and 0.874 mm. and lengths of 38.7 mm. and 20.8 mm., and the quartz capillaries had bores of 1.438 mm. and 1.465 mm., the corresponding lengths were 35.9 mm. and 19.5 mm. The pressure drops were obtained using a heated mercury-in-glass 'U' tube manometer. Corrections were made for the thermal expansion only at the higher temperatures for each material and amounted to a maximum of about 0.14 %. The effect of slip at the walls was neglected, although using Knudsen's (40) assumption based on the mean free path of a gas, the correction could amount to as much as 0.28 % at 860°C. The accuracy was given as between 1.5 % and 2.0 %. There is insufficient information available for a truly critical assessment of the validity of the stated accuracy. The standard deviation based on his straight line equation

$\eta = (80.4 + 0.407\ t\ ).\ 10^{-6}$ poise, is ± 1.3 % which is greater than the assessed accuracy. However the accuracy appears to be rather ambitious, since the main boiler pressure controller was connected to the upstream limb of the manometer, this would produce hunting and therefore instability. There is also a lack of data concerning the capillary calibration; it is very doubtful if two capillaries of exactly the same size could be obtained. A further point to note is that the entrance and exit of each capillary was smoothed, which would invalidate the assumption that the end corrections are the same for both tubes.

The most recent values for the viscosity of steam have been obtained by Kestin and Richardson (41) in 1962. Using an oscillating disc viscometer they obtain values at pressures slightly in excess of atmospheric to pressures near the saturation value in the temperature range of 149° to 275°C. The apparatus was calibrated using nitrogen. The standard deviation of the results from those made previously by Kestin and Wang (42) was 0.81 μpoise, the deviations being positive and increasing with temperature. The assessed accuracy is ± 0.4 % which seems to be justified. The results are plotted on the deviation plot and can be seen to deviate from those of Shifrin by between + 0.7 % to + 1.2 % up to 300°C.

The following works have not been considered in detail for the atmospheric work since it is not possible to extrapolate the results to atmospheric pressure with any reasonable degree of accuracy. However they are briefly mentioned.

In 1958 Kjelland-Fosterud (23) (43) used another type of modified Rankine viscometer to measure the viscosity of steam in the ranges 200 to 100 atmospheres and 370° to 680°C. The apparatus differed from that of Timrot in that the driving force was produced by a mercury pellet falling in a vertical drop tube. The accuracy was originally assessed to be 2 %, but this is rather ambitious and should be more like 4 %.

Whitelaw (24), (44), (45), following Kjelland-Fosterud, modified the apparatus. In 1960 he published results for the ranges 200 to 800 atmospheres and 370°C to 650°C; the assessed accuracy of the work was ± 1.5 %. This again on closer examination appears to be too high and is now thought to be more like ± 3 %.

Further development of the above apparatus has been made by Ray (25). The ranges which he has covered are 300 to 700 atmospheres and 380°C to 700°C with an improved accuracy of ± 1.3 %. In the above work the apparatus was a secondary type using a fluid of known viscosity to calibrate the capillary, air being the fluid initially used and nitrogen the fluid used in the later work.

Schmidt and Mayinger (46) in 1961 published results for steam in the ranges up to 700°C and 800 atmospheres with an assessed accuracy of ± 1.5 %. The apparatus was a single capillary efflux type. Two capillary sizes were used, 0.32 mm. and 0.51 mm. internal diameter, both of which had a nominal length of 500 mm. In this apparatus the capillaries were quite long and had to be calibrated with a fluid of known viscosity and therefore the apparatus was really of the secondary type.

An apparatus employing flow through an annular space was used by Jackson (47), (48), (49) in 1958 and 1963 to obtain results for steam in the ranges 1.5 to 700 atmospheres and 224°C to 601°C, the assessed accuracy being uncertain. However his results disagree with the existing data by as much as 15 % and are therefore open to considerable doubt.

Chapter III

Types of Viscometer

In general viscometers may be classified as belonging to either one or other of two main groups. In group (1) (moving fluid) may be put those instruments in which the fluid is essentially flowing and in group (2) (moving body) those in which the fluid is essentially at rest and a given body moves in the fluid. The following subdivisions of these groups illustrate the two groupings:

Group (1), moving fluid, (a) capillary flow
(b) radial flow
(c) flow through annular gap,
Group (2), moving body, (d) falling bodies
(e) rotating bodies
(f) oscillating bodies.

In the group (1) type of viscometer, the fluid is forced through a fixed aperture and the viscous resistance measured. In the group (2) type of viscometer, the body, which is usually some geometrically well defined shape, translates, rotates or oscillates, within or without the working fluid, and the viscous drag is deduced. Although a great deal of elaboration has been brought into the design of viscometers, the basic principles are the same.

Only a brief discussion of the types of viscometer, suitable for use with Newtonian fluids, is given in this chapter, but, if required, more comprehensive data can be obtained from texts such as Barr's (50) Monograph of Viscometry, Viscometry by Merrington (51), Viscosity and its Measurement by Dinsdale and More (52) and Viscosity and Flow measurement by Van Wazer, Lyons, Kim and Colwell (53).

The systems used can be further sub-divided into open circuit and closed circuit systems. In the open circuit system the fluid is continuously supplied from a source and passed through the viscometer, to be collected on leaving the viscometer. This type of system can be either primary or secondary, depending on whether the capillary has been calibrated by direct measurement or by passing a gas of known viscosity through it. On the other hand, in the closed circuit system the circuit is continuous and not open to the surroundings at any point. This system can also be primary or secondary, but usually the latter is used. The fluid is forced through the viscometer and may be permitted to flow in a continuous cycle. This system is very useful for measuring the viscosities of small quantities of a fluid.

(1) Moving Fluid Viscometers

(a) Capillary Type

There have been a number of variations of the simple capillary viscometer, in which the evaluation of the viscosity is based on the Poiseuille equation. The first successful arrangement was that of A. O. Rankine (27).

In the basic system the fluid flows through a small bore tube and the pressure drop across the tube measured. This is one of the simplest and most widely used viscometers. There are several arrangements, (one using two capillaries in parallel or in series), but the basic principles are the same. The theory is very well developed and appears to be one of the most reliable and a viscometer, using this principle, can be either of the open or closed circuit type.

The theory for the capillary viscometer will be discussed in greater detail in chapter (V).

The viscosity of the fluid to be measured is determined from the equation,

$$\eta = \frac{\pi\, d^4\, \Delta P\, g\, \rho \left\{1 + 8\varepsilon/d\right\}}{128\, Q\, l} - \frac{m\, Q}{8\pi\, l} \qquad \ldots\ldots\ldots\ldots\ 3.1$$

where Q is the mass flow rate, 'm' is the end correction, $\left\{1 + 8\varepsilon/d\right\}$ is the correction for slip at the walls of the tube.

Rankine's viscometer was a closed circuit secondary type in which the fluid was forced through a capillary under the force of gravity. The apparatus, which could be inverted to reverse the flow, was used to measure the viscosity of Ar., He., Kr., Ne., and Ye., in the temperature range 10°C to 100°C at atmospheric pressure. The measurements were made relative to that of air. His data are rather scanty and in themselves relatively unimportant, yet his system is the basis of a large number of later successful viscometers.

Trautz and co-workers (29) (54) have published a large number of papers on the viscosity of H_2, Ar, CO_2, CO, C_2H_6, C_2H_4, He, HCl, CH_4, Ne, N_2, O_2, C_3H_8, SO_2, X_e and H_2O at atmospheric pressure and temperatures from 90°K to 1100°K. In the apparatus used mercury falls from one reservoir to a second one, which in turn is connected to a capillary. The mercury, in falling from one reservoir to the other, displaces the fluid through the capillary into the atmosphere. The pressure head and hence the pressure drop across the capillary continually decreases with time.

The same type of apparatus, as that used by Trautz, has been used by Michels and Gibson (55) and also by Lazarre and Vedar (56), (57), both being capable of use at pressures.

Rose and Brown (58) produced another variation by mounting the capillary between two bellows which were calibrated. The capillary being initially displaced and then allowed to resume its original position. This action made the fluid flow from one of the bellows to the other. It was used at pressures up to 700 atmospheres.

A further variation was that, used by Hanks (59) and later by Lemaire (60) and Flynn (61), in which a servo-controlled piston was used to force the fluid through the capillary. The arrangement was such that as the piston moved along the cylinder it forced the fluid through the system and received the discharging fluid at the other end of the cylinder.

Dolphin (62) had the same arrangement, as that used by Hanks, for pumping the fluid into the viscometer; however, he used two capillaries in parallel instead of a single one. The method used was to pass the fluid in turn through each of the capillaries with the same mass flow rate. The capillaries were not of the same length but had the same diameter, but from the two resulting equations the unknown end effects could be eliminated.

For obvious reasons it is desirable that a long tube with a large bore be used in the capillary type viscometer, the main drawbacks being the difficulty of obtaining a good temperature distribution along the tube and the difficulty of obtaining a long tube with a uniform bore along its entire length. Experiments have attempted to overcome the first difficulty by coiling the capillary into a toroid. However, this obviously necessitates a correction to the flow equation due to the centrifugal effect. Hawkins (32), (34), found that for his coiled capillary the viscosity became dependent upon the value of the Reynolds number when it was greater than 180. This necessitates the calibration of the viscometer for values of Re greater than 200, the apparatus then ceasing to be a primary instrument. Since the main object of using a coiled instrument is to obtain primary results this system loses its value as a viscometer.

Kenney (63) also used a coiled capillary tube apparatus to measure the viscosity of exhaust gases; however the procedure was slightly different. The fluid was allowed to escape from a charged bylinder to another reservoir. The rates of the pressure rise in the reservoir and of the pressure fall in the cylinder were noted and could be used to calculate the mass flow rate and hence the viscosity of the fluid being measured. The apparatus was initially calibrated with air and was therefore of the secondary type.

Bonilla, Brooks and Walker (37) used another variation in which a long capillary was coiled into a helical coil. This apparatus was used up to temperatures in excess of 1400°C and appears to have been one of the more successful coiled capillary apparatus.

Shifrin (28) used an apparatus in which two capillaries in series were used. The capillaries had the same diameter but were of different lengths. Since the mass flow would be the same in both the tubes, the end corrections could be eliminated. In essence the concept was the same as that used by Dolphin.

Speyerer (30) used an open circuit system in which a brass capillary was split into three sections, the sections being connected by sleeves which had provisions for pressure tappings to be made at them. The idea was that the pressure drop across the central section, where fully developed flow had been established, could be measured. This system removed the need for end corrections and therefore made use of the same principle as that adopted by Shifrin and Dolphin. It has since been found that the slots can cause disturbance of the flow in the region of the connections. For this reason this type of viscometer is not recommended.

Several experimenters have used a modified Rankine viscometer for determining the viscosity of steam at high pressure. Schugajew (19) used this type, mercury being used as the driving fluid for low temperatures and tin for high temperatures. The system was almost identical to that of Rankine, the whole viscometer being put into the furnace.

Comings, Mayland and Elgy (64), and later Carr (65) used a Rankine viscometer to measure the viscosities of several gases and gas mixtures in the temperature range 70° to 250°F at pressures up to 10000 p.s.i.a. Pyrex glass capillary tubes were used in this apparatus.

Timrot (20) used a modified Rankine viscometer in which the pressure drop across the capillary was produced by a ring balance containing mercury. When the ring balance, which was supported by knife edges, was displaced, the mercury returned to its equilibrium position and forced the steam through the capillary. It is not certain whether the apparatus was of the primary or secondary type, there being insufficient data available.

More recently Kjelland-Fosterud (23), Whitelaw (24) and Ray (25) have used another version of the Rankine viscometer. Like Timrot's the apparatus has the mercury piston outside the furnace and the capillary inside. The apparatus uses a straight drop tube unlike Timrot's semi-circular type. This was a secondary system and was calibrated with air for the first experiments made by Kjelland-Fosterud, the latter workers using nitrogen as the more reliable calibrating fluid.

(b) <u>Radial Disc Viscometer</u>

The fluid passes between two circular parallel plates which are at a given distance apart, and the viscous pressure drop between the entrance and the exit is measured. This method has not been used very much although the theory has been relatively well developed. There has been very little empirical work carried out with this apparatus. Gümbel (66) appears to have been the first person seriously to propose this type of arrangement to measure the viscosity of a fluid. However, little thought had been given to the matter until Liley (26) in 1963 discussed the use of this instrument.

The equation of flow may be found to be,

$$Q = \frac{\pi . t^3 . \Delta P . \rho}{6.\eta \log_e(R_2/R_1)} \quad . \quad \left[1 + 6\eta/t\epsilon \right] \quad \ldots\ldots\ldots \; 3.2$$

where ρ is the mean density, t is the separation distance between the discs, R_1 and R_2 are the inner and outer radii af the discs and $\left[1 + 6\eta/t\epsilon \right]$ is the slip correction, the slip factor being η/ϵ.

Liley, in his note, made some preliminary investigations and has some suggestions for the dimensions of a proposed apparatus.

The fact that the viscometer consists of two discs makes it possible for the inner surfaces to be produced with a good surface finish. Thus the inherent difficulties encountered in the capillary instrument due to coniality and ellipticity of the bore along the tube length are avoided. The distance between the discs is required to a lesser degree of accuracy than is the bore for the capillary instrument, since it is raised to the third power, one less than for the capillary apparatus. Also a further point to note is that the geometry of the system makes it possible to determine the separation distance accurately with modern gauging techniques.

While the above advantages apply equally to the oscillating disc viscometer, the equation for flow is simpler than that required for the oscillating disc and the capillary viscometers. However, there is the uncertainty of the edge effects. There are also the manufacturing difficulties, which are greater than those for the simple capillary apparatus. Although the gap could be altered to suit given temperature and pressure conditions, any slight temperature gradient across the discs could produce very marked effects on the results.

(e) Annular Gap Viscometers

There appears to have been very little experimental or for that matter theoretical work done on this type of viscometer. In this type of instrument the fluid flows through an annular gap between two concentric cylinders. Jackson (67) in 1949 did some experiments with steam up to high pressures and moderately high temperatures. His apparatus was a primary type using a working equation of the form,

$$\eta = \frac{\pi . \Delta P.}{8.Q.l.} \left\{ (b^4 - a^4) - \frac{(b^2 - a^2)^2}{\log_e . b/a} \right\} \ldots\ldots\ldots 3.3$$

which can be found in Lamb's (68) treatise.

This equation can alternatively be written,

$$\eta = \frac{\pi . \Delta P. R^4 . Vs.}{8.Q.l.} \left\{ (1 - K^4) - \frac{(1 - k^2)^2}{\log_e 1/k} \right\} \ldots\ldots 3.4$$

where $k = R_1/R_2$.

His results are up to 17 % above existing data and are therefore suspect. In 1961 Isshiki (69) commenced work on a similar secondary apparatus about which little is known. However, Jackson does note that further study of the apparatus is desirable.

There are many variations of the three basic types of viscometer described above, but the majority are based on these three systems.

MOVING BODY VISCOMETERS

(d) Falling and Rolling Sphere Type

This method relates the viscous drag to the velocity of fall of a sphere in the fluid to be measured. Stokes (2) developed the following law for the particular case of a sphere moving with a uniform velocity in an infinite fluid,

$$F = 6\pi . r . \eta . v. \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.5$$

where F is the viscous resistance, 'r' is the radius of the sphere and v the uniform velocity.

It may quite easily be shown that

$$\eta = \frac{2.g.r^2. \quad [\sigma - \rho]}{9.v.} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.6$$

where σ and ρ are the densities for the body and the fluid respectively. In practice however it is not possible to work with a fluid of infinite extent and the introduction of finite boundaries involves the use of corrections to Stokes' equation. Among the earliest attempts to allow for the wall and end effects was that of Ladenberg (70) in 1907, who deduced a correction of the form

$$\eta_t = \frac{\eta_{measured}}{[1 + 2.4\ r/R] \ . \ [1 + 3.3\ r/h]} \ \ldots\ldots\ldots 3.7$$

where R is the radius of the containing cylinder, and 'h' is the height of the containing cylinder, $[1 + 2.4\ r/R]$ is the correction for the effect of the wall and $[1 + 3.3\ r/h]$ is the correction for the finite depth of the fluid.

In 1936 Bacon (71) discussed the validity of the various corrections and referred to an equation put forward by Faxen (72), which it is suggested gives fair agreement with capillary results. Faxen's equation is $\eta_t = \eta_{measured} \cdot \left\{ 1 - 2.104\ r/R + 2.09(r/R)^3 - 0.95(r/R)^5 \right\}$ 3.8

However, the basic law assumes that the motion of the sphere relative to that of the fluid is slow. Rayleigh (73) showed that Re must be less than one, and subsequent experimenters have shown that it is desirable to have Re << 1. This is very difficult to achieve in low viscosity fluids with sufficient accuracy to justify the use of this method for absolute determinations. This method can be used for relative measurements as shown by the above equation, but it is unrealistic for use with water or steam.

Hawkins (74) experimented unsuccessfully with different shapes of falling body in an attempt to retard the fall of the body and maintain Reynolds numbers below 2000. Sage and Lacey (75), Schroer and Becker (76), Smith and Brown (77) and several others have attempted to measure the viscosity of gases by this method. Their results are considered to have a discrepancy of more than 5 %. On the other hand, this method has been applied to liquids with a greater degree of success by Fritz and Weber (78).

(e) <u>Oscillating Bodies</u>

Of the moving body viscometers those utilising oscillating bodies have been the most widely used for gases. The oscillating body viscometer is perhaps the most simple, mechanically, of the viscometers. If a body of known moment of inertia is suspended on a wire and allowed to perform small oscillations in a given fluid, the equation of motion may be represented as,

$$I.D^2\phi = -F.D\phi - G.\phi \qquad \ldots\ldots\ldots\ldots\ldots\ 3.9$$

where I is the moment of inertia of the system and D is the usual form of operator d/ dt, F is the viscous force resisting oscillations and is proportional to the angular velocity D ϕ and the returning couple produced in the suspension.

$$\text{or} \quad \left\{ I.D^2 + F.D + G \right\}\phi = 0 \qquad \ldots\ldots\ldots\ldots\ldots\ 3.10$$

the solution of which is $\phi = A.\ e^{-\delta t/T}\cos 2\pi t/T$,

where ϕ is the angular displacement in time 't', T is the period of oscillations, δ the log. dec. of the amplitude for one complete oscillation. This principle has been applied to several simple geometric shapes and configurations, such as cylinders, spheres, discs and pendulums.

Cylinder

Although a thorough treatment of the theory of hollow, or solid cylinders performing small torsional oscillations has been given by Kestin and Persen (79) using an exact solution of the Navier-Stokes equation, little experimental work has been carried out using this method. This is mainly because no satisfactory end correction has been deduced; also it is difficult to eliminate the secondary flow in the annular space between the inner cylinder and the outer one. It is therefore not envisaged that this type of instrument will be used until a considerable amount of development has been made.

Sphere

The sphere, which is suspended on a wire, may be used in a combination of the following ways:- (1) solid sphere in an infinite fluid, (2) a solid sphere in a concentric shell, the fluid being in the intervening space, (3) a hollow sphere, filled with a fluid, in an infinite fluid (the fluid within and without may be the same or different), (4) the same as (3) only using a boundary of a concentric sphere or (5) the fluid inside the sphere and a vacuum outside.

It is obvious that the major problem is in producing spheres of suitable accuracy.

Investigation into the theory connected with spheres has been carried out by Kestin and Persen (79) and later by Kestin and Wang (80), who concluded that it may be possible to produce an oscillating sphere viscometer for high viscosity fluids provided the spheres could be manufactured with sufficient accuracy. The existing theory being inapplicable when the damping, and therefore the viscosity, is small.

Mossynski (81), (82), successfully used a solid stainless steel sphere with had a 1" nominal diameter, to measure the viscosity of water at temperatures up to 186°C and pressures up to 341 atmospheres. The measured values agree quite closely with results from other experimenters, thus showing that this system can be used with some precision.

Oscillating Disc Viscometers

There are several variations in the system which may be used with an oscillating disc; (1) the disc can be made to oscillate in an infinite fluid, (2) the disc can be made to oscillate equidistant between two identical fixed discs, the space between being filled with the fluid to be measured, (3) the system can be the same as that for (2) only having finite boundaries (i.e. the disc oscillating within a concentric cylinder). The latter arrangement is the most practicable.

Recent developments in the study of the hydrodynamics of an oscillating disc by Kestin and Persen (7(), Kestin and Leidenfrost (83) (84) and Newell (85) indicate that this type of apparatus may be quite suitable for accurate absolute determinations of the viscosities of fluids having densities up to 0.03 mg/cm^3., the exact limit depending on the gas to be used and the geometry of the system. Kestin and Leidenfrost (83) (84) and later Kestin and Whitelaw (86) (87) have used an oscillating disc to measure the viscosities of a large number of gases in the range '1' to 130 atmospheres and up to 270°C. The work of Kestin and his co-workers and that of Iwasaki (88) constitute the only high pressure work on simple gases using an oscillating disc viscometer.

Other important work has been published by Johnston and McCloskey (89), Johnston and Grilly (90) and Van Itterbeek and co-workers (91-95), all the work having been carried out at low temperatures and atmospheric pressure.

Naldrett and Maas (96) pointed out the suitability of the oscillating disc viscometer for use in the critical region.

The first approximate analytical solution for a disc, oscillating in an infinite fluid, was obtained by Meyer (97). His solution was based on the assumption that the viscous fluid forms a cylinder of which the disc is a part and that the effect of the surrounding fluid is neglected. Meyer's equation may be written,

$$\delta = \frac{\left[\pi/2\right]^{3/2} r^{4}. \quad \left[\rho\eta T\right]^{1/2}}{I} + \frac{\pi r^{3}\eta T}{I} \quad \ldots\ldots\ldots\ldots\ 3.11$$

where T is the period of oscillation, I is the moment of inertia of the system, r is the radius of the disc and $\frac{\pi r^{3}\eta T}{I}$ represents the effect of the edge.

The equation has been developed further by Verschaffett (98) and Kobayaski (99) who corrected for the effect of the moment of inertia of the fluid itself. Further theoretical improvements were made by Macwood (100) and Hollis and Hallet (101), who used an equation in the form $\eta = k\ \eta_{\text{observed}}$ where k is a correction factor for the edge of the disc, both authors assuming that the square of the log. dec. was very much less than unity.

Theories have been advanced by Kestin and Persen (79), Mariens and Van Paemel (102), who have produced corrections for wire damping, side drag and mirror rod drag. However, this does not make this method suitable for absolute determinations having a high degree of accuracy.

However, recently, Appiettia and Newell (103) (104) (85) have analysed the problems for both thin and thick discs. The solution of the derived equation being achieved by successive approximations. The experimental work of Kestin and Pilarczyk (105) and Kestin and Wang (106) is claimed to substantiate the theories of Newell with an estimated error of 1%. Unfortunately the geometrical limitations controlling this theory tend to limit the range of viscosity for which the theory is valid.

Oscillations between two fixed plates have been studied and used quite frequently as a basis for viscometer design. A solution was obtained by Maxwell (5) to evaluate the viscosity of air, a more refined theory being later produced by Macwood (100). For small separation between the plates the equation for the viscosity can be expressed as,

$$\eta = \frac{2.I.}{\pi.R^4.T.} \left\{ \left[T/T_o \right] \frac{\delta}{2\pi} - \left[T/T_o \right] \frac{\delta_o}{2.\pi} \right\} \left[I - E \right]$$

$$\text{where } E = \frac{8\ d \left[\pi\ \rho/T\ \eta_1 \right]^{1/2}}{\left[4.d.\ \left[\frac{\eta\ \rho}{T\ \eta_1} \right]^{1/2} - a \right]} \qquad \ldots\ldots\ldots\ldots\ldots\ 3.12$$

For large separations,

$$\eta = \frac{4.I.}{\pi.R^4} \sqrt{\frac{\pi}{T.\rho}} \left[\left\{ \left(\frac{T}{T_o}\right)^2 \frac{\delta}{2\pi} - \left(\frac{T}{T_o}\right) \frac{\delta_o}{2\pi} \right] \left(1 - \frac{\delta}{4\pi} \right) \right]^2 \times (1 - E)$$

$$\text{where } E = \frac{\left\{ 2 \sqrt{\frac{T\ \eta}{\pi\ \rho}} - 4\ d \right\}}{\left\{ 2 \sqrt{\frac{T\ \eta}{\pi\ \rho}} + \left(\frac{R}{2} - 2\ d \right) \right\}} \qquad \ldots\ldots\ldots\ldots\ 3.13$$

and where for both the expressions it is assumed that $\delta^2 \ll 1$ and $\delta_o \ll 1$ and that x is the separation between the fixed and moving plates. E is the edge correction, T is the period of oscillation of the system, T_o is the period of oscillation of the system in a vacuum, δ and δ_o the log dec in the fluid and in a vacuum, and R is the radius.

The equation for small separation has been proved by Kestin and Pilarczyk (105) to be unreliable. This may be due to the original assumption that the angular velocity of the fluid up to the edge of the disc is constant. For Macwood's equations, for the theoretical error to be less than 1 % the following conditions must hold,

for small separations $$x \Big/ \sqrt{\left\{\frac{\pi . \rho}{T}\right\}} < 0.3$$

.... 3.14

for large separations $$x \Big/ \sqrt{\left\{\frac{\pi . \rho}{T}\right\}} < 4.0$$

Other typical instruments used at a pressure of one atmosphere are those of Vogel (107), Sutherland and Maass (108), Van Itterbeek and Keesom (91) and Johnston and McCleskey (89), all working to quite low temperatures.

The working formulae used by Kestin and Whitelaw (109) (110) in their most recent work is rather complex, the edge correction C may be written

$$C = \frac{\left({}^{2}/_{\theta} \right) \left(\frac{\Delta}{\theta} - \Delta_{0} \right)}{\left(\pi \rho R^{4} \, {}^{f}/_{I}\right) \left(H_{1}K_{2} + H_{2}K_{1} + \left(\frac{2d}{R} \right) \left\{ H_{1} + \frac{3\delta}{2R\theta} \right\} \right)} \quad \ldots\ldots 3.15$$

where the following symbols may be defined,

$$H_{1,2} = \left\{ 1 \pm \frac{3}{2}\Delta - \frac{3}{8}\Delta^{2} \right\} \left(2\theta^{3} \right)^{-1/2} \quad \ldots\ldots\ldots\ldots 3.16$$

$$K_{1} = \frac{\sin . Y}{\cosh X - \cos Y} , \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.17$$

$$K_{2} = \frac{\sinh X}{\cosh X - \cos Y} , \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.18$$

$$X, Y, = (2\theta)^{-1/2} \left\{ 1 \pm 1/2\,\Delta + 1/8\,\Delta^{2} \right\} \left(\beta_{1} + \beta_{2} \right) \quad \ldots 3.19$$

$$_{1,2} = b_{1,2,} / \delta \qquad \delta = \frac{\left\{ \eta T_{0} \right\}^{1/2}}{\left\{ 2\pi \rho \right\}} \qquad \theta = \left(T/T_{0} \right) \quad \ldots\ldots 3.20$$

and $\Delta = \left\{ 1/2\pi m \right\} . \operatorname{Log}_e \dfrac{\alpha_{\eta}}{\alpha_{(\eta + m)}}$ 3.21

From the above equations it can be seen that the solution is quite complex. The authors plotted C against L / δ and smoothed graphically and then transformed into a table with intervals that permitted linear interpolation. The table was then fed into an electronic computer for the evaluation of the actual experimental results. Experimental work was carried out on argon, carbon dioxide, helium, neon and nitrogen in the ranges 25 to 270°C and 1 to 150 atmospheres. The measurements were relative to nitrogen at 22°C and have a claimed accuracy of 0.5 %.

Rotating Cylinder Viscometers.

This method in principle is one of the most accurate ways of obtaining absolute viscosity measurements, and has frequently been used to determine standard values by relative methods. The main arrangement for the viscometer is to have two concentric cylinders immersed in the fluid to be measured. When one of the cylinders is made to rotate, the viscous drag exerted on the other cylinder by the fluid between them may be measured and used to determine the viscosity of the fluid. There are two main modes of operation for this system which are: (1) to rotate the outer cylinder and measure the angular deflection of the inner cylinder, which is suspended on a wire, and (2) to rotate the inner cylinder and observe the angular deflection of the outer cylinder. For low speeds, the angular deflection of the stationary cylinder is directly proportional to the viscosity. However, correction must be made for the effects of the ends of the cylinder, which may be minimised by the use of guard cylinders, the guard cylinders, being cylinders of the same diameter as the inner cylinder placed co-axially at the ends of the inner cylinder.

Maragules (111) derived the theory for the case of a cylinder of infinite length and radius R, rotating with a constant angular velocity ω inside a fixed concentric cylinder of radius 'r' and length 'h'. The retarding couple was estimated to be represented by the equation

$$G = 4\pi.\eta.h.\omega \left\{ (R^2 - r^2) / (R^2 r^2) \right\} \qquad \ldots\ldots\ldots\ldots 3.22$$

This expression was based on the assumption that both the cylinders were of infinite length, and does not take into account the finite length of the outer cylinder. This effect has been partly overcome by the use of guard cylinders mentioned above. It was also assumed that the flow of the fluid was laminar and entirely circumferential between the cylinders and that there was no slip. With these assumptions, the theory is equally valid for both systems of rotation. Most of the rotating viscometers which have been used for gases were closely based on the apparatus produced by Gilchrist (112) in which the outer cylinder is rotated. Kellstrom (113) used a rotating cylinder viscometer for measurements up to pressures of 30 atmospheres, which apparently is the only rotating cylinder instrument to have been used at pressures above 1 atmosphere. Bearden (114) used an apparatus in which the inner cylinder rotated, to determine the absolute viscosity of air at 20°C and 1 atmosphere. Bearden was of the opinion that only the rotating cylinder and capillary viscometers were capable of highly accurate absolute measurements. More recently Reamer, Bokelet and Sage (115) have obtained results for the viscosity of n-pentane in the ranges 0 to 500°F and up to 2500 p.s.i.a. Their apparatus was a rather complex arrangement of the basic form of rotating cylinder viscometer with quite a few refinements, but a high accuracy was not claimed.

Rotating Sphere Viscometers

The sphere is theoretically perhaps the most ideal shape for the rotational method. The arrangements are basically the same as those given for the oscillating sphere in this chapter. Brillouin (116) showed that for the case when a fluid is enclosed between two spheres, one concentrically inside the other, the viscous drag produced on the inner one of radius 'r' by the outer one of radius R, rotating with an angular velocity ω, may be expressed by the equation,

$$G = 8\pi.\eta.\,\omega\left[(Rr)^3 / (R^3 - r^3)\right] \quad \ldots\ldots\ldots\ldots \; 3.23$$

However, in spite of the theoretical completeness of this system, the practical difficulties seem to be insurmountable.

Zemplen (117) did do some work with a rotating sphere, the viscosity results from which appear to be higher than the accepted values.

Rotating Disc Viscometers

The general arrangements for the rotating disc are basically the same as those for the oscillating disc, given previously in this chapter. The theory relevant to the rotating disc is quite difficult to predict with any measure of accuracy. It is possible to use this type of apparatus in a secondary capacity for high viscosity fluids, calibrating with a fluid of known viscosity. However, the theory for the rotating disc has not really been fully investigated for the instrument to be used for absolute determinations.

In the ideal case of an infinite disc, rotating in an infinite fluid, it can be shown that the retarding couple is given by,

$$G = \frac{32.\eta.r^3.\omega}{3} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 3.24$$

where ω and r are the angular velocity and the radius respectively of the disc.

In the case, where the disc rotates within given boundaries, e.g. when two similar discs are placed on both sides of the disc and equi-distant from it, the retarding couple is given by

$$G = \pi \eta \frac{r^4}{t} \omega \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 3.25$$

where 2t is the distance of separation of the two outer discs.

Both of these equations are hypothetical, since the behaviour of the fluid at the edges is not taken into account. Merch et al. (118) did do some experiments with a rotating disc, but there are no records of any other serious attempts to do so.

OTHER METHODS.

There are many other varied methods of determining the viscosity of Newtonian and Non-Newtonian fluids, more especially the latter, none of which have been used to any great extent or accuracy. The author refers the reader to the references given at the beginning of this chapter for information regarding other methods.

Conclusion Regarding the Most Suitable Method for Use with Steam Measurement

As a conclusion to this chapter on viscometers, the author wishes to compare the relative merits of the types of viscometers discussed.

The rolling or falling ball viscometer cannot be used with any good accuracy for absolute determinations, but the range of viscosities for which it can be used is large. The theory for radial flow viscometers is incomplete and this type of viscometer has not been proved experimentally. For annular gap type transpiratory viscometers the theory is relatively well developed; however, the experimental results are not conclusive. Of the rotating body viscometers the rotating cylinder method appears to be the most potentially capable of accurate absolute measurements. Unfortunately, the rotating cylinder is restricted to low pressure and temperature measurements for the present. The oscillating sphere method is also potentially capable of accurate absolute measurements; however, there are almost insurmountable practical difficulties to overcome. An exact theory has been established for the oscillating disc, but with present instruments the theory can only be rigorously applied to low pressure work. There are also serious practical difficulties when it is used for high temperature measurements. The theory for a capillary flow viscometer is considered to be the most reliable of all the viscometers. This type has the best potential, the range of viscosities, temperatures and pressures being almost unlimited.

Chapter IV

Selection of Viscometer

At the commencement of the project described in this thesis, work had already been started at Glasgow University on the determination of the viscosity of water and steam up to high pressures and temperatures. A modified Rankine viscometer (43) was already in use and a rotating cylinder viscometer (24) (43) was in the design stage. There was also research in progress, using oscillating disc viscometers, both at Brown University (119) and the National Engineering Laboratory, Scotland (120). The author therefore considered it unwise to pursue any duplication of these projects, at least until the results from these apparatus were known, unless there were some definite advantage for this project. From the discussion regarding viscometers in the previous chapter, it can be seen that with the present knowledge of the theory the field can be considerably narrowed down, if a viscometer is required which will give absolute determinations of a reasonable accuracy. It was finally decided that some type of capillary viscometer would be the most suitable instrument, for the measurement of the viscosity of steam at atmospheric pressure, up to high temperatures; since the theory is well developed and the simplicity of the design makes it possible to use this apparatus over a wide range of temperatures.

As stated previously there are three main arrangements for a capillary viscometer,

(1) single capillary,

(2) two capillaries in parallel,

(3) Two capillaries in series.

(1) Single Capillary System

The single capillary system has a drawback in the fact that the end correction factor 'm', in the basic Poiseuille equation,

$$\eta = \frac{\pi \, d^4 \, g \, \Delta P}{128 \, Q \, l \, V} - \frac{m \, Q}{8 \, \pi \, l}$$

is not known to any accuracy, not better than ± 4 % (see chapter (V)). The effect of this uncertainty in the value of 'm' on the value of η, is dependent on the magnitude of the end correction in comparison to η. The importance of the value of 'm', can be considerably reduced by designing the system so that Q, the mass flow rate, is small compared with 'l', the length of the capillary. This is not really practicable in absolute systems, since the mass collected in a given time must not be too small. It is, of course, possible to have a small mass flow and collect volumetrically, but this involves both temperature and pressure difficulties where water vapour is concerned.

A further way of getting round this problem is to find the value of 'm', for each condition. This can be done by varying the mass flow at each isotherm and obtaining 'm' graphically, and has been used by some experimenters. However some workers are of the opinion that 'm' might vary with Reynolds number and/or temperature, as well as entry conditions. This method would therefore be unsuitable, if this is the case.

There is the further alternative, where pressure tappings could be made through small slits in the capillary, within a central, fully developed flow length of the tube. Thus avoiding the need for 'm'. As stated in the previous chapter, this has been found to be unsatisfactory, since the slits disturb the fluid flow in their vicinity. Also, small disturbances in the pressure lines can cause very unstable flow conditions in the capillary. There is also the added difficulty of obtaining a tube with uniform bore over a long length and maintaining uniform temperature conditions along this tube, which is what would be required.

Finally, an alternative method would be to have flared ends to the capillary. With suitable diameter profiles along the tube, at the entrance and exit, the end correction could be made zero. Although it is quite possible to manufacture [x] such capillaries, calibration difficulties would arise. The author is therefore of the opinion that a flared capillary system is only suitable for a secondary apparatus, where a calibrating fluid could be used.

[x] see paper by Caw and Wylie (121)

(2) <u>Two Capillaries in Parallel</u>

This system has the advantage that the need for the value of 'm' may be eliminated by passing the fluid through each capillary in turn. There are two main difficulties with this system, (1) the difficulty of establishing Reynolds numbers, in the tubes, which are very nearly equal at a given temperature, and (2), the practical problem of having an arrangement that will divert the fluid down each tube in turn, and will operate at the high fluid temperatures expected.

(3) <u>Two Capillaries in Series</u>/

(3) Two Capillaries in Series

As with the parallel system, the end corrections can be eliminated by equating the results, for each capillary, at a given temperature. The main disadvantage of this system is in measuring small differential pressures, requiring high stability and accuracy.

Of the two systems (2) and (3), it was felt that (2) had more practical drawbacks for high temperature work than (3). Since there is the difficulty of the "change-over" arrangement and the problem of obtaining stable flow conditions after a "change-over", this is a very real problem when dealing with steam. The series system incorporates the basic advantages of the parallel system without the practical difficulties.

Although Shifrin (28) has used a series capillary apparatus, very little is known about it. It was considered that it would be interesting to develop a series capillary apparatus, since it would have the advantages, given above, over the single capillary apparatus plus the fact that, if any inherent difficulties arose, the system could be run as two single capillaries, thus duplicating the results at a given temperature. It was also thought that there may be some possibility of checking the value of 'm' by the series method.

With the above reasoning in mind, it was decided to embark on the design of a series capillary viscometer, to measure the viscosity of steam at atmospheric pressure.

Chapter V

Theory for the Capillary Viscometer

In 1846, Poiseuille (122) put forward his empirical equation, based on experiments of flow through small tubes. At almost the same time Hagen (123) produced quite independently the same equation, which was,

$$V = K.\frac{\Delta P.r^4}{l.}$$ 5.1

where K is a constant, for a volumetric flow rate of V, through a tube of length 'l' and radius 'r', when the pressure drop across the tube is ΔP.

Later in 1856, Wiedman (124) showed that the constant K was equal to $\pi/(8\eta)$. That is, the equation for the volume flow rate is,

$$V = \frac{\pi.\Delta P.r^4}{8\ \eta.l.}$$ ....referred to as the Hagen-Poiseuille equation. 5.2

The Hagen-Poiseuille equation was alter derived in a more rigorous manner, the derivation assuming that (a) the flow is parallel to the axis of the tube and (b) that the flow is steady, (c) there is no slip at the walls of the tube, (d) the fluid is incompressible and (e) the viscosity is proportional to the radial velocity gradient.

Reynolds showed that condition (a) above can be achieved if the flow is laminar, which is when the Reynolds number ($Re = \frac{\rho\ v\ d}{\eta}$) is less than about 2000. Conditions (b), (c) and (d) are not completely valid for gases and (e) is dependent on whether the fluid is Newtonian.

The Hagen-Poiseuille equation can be arrived at by using the Navier-Stokes equation. Using the cylindrical co-ordinate form of the Navier-Stokes equation and applying it to the particular case, where, (1) the axis of the tube is the z axis, (2) the tangential and radial components of the velocity are zero, (3) the pressure components parallel to the x axis depends on z alone, and (4) the pressure is constant in every cross section,

$$0 = -\frac{dp}{dz} + \eta \left\{ \frac{1}{r} \cdot \frac{\partial \left(r \frac{\partial v_z}{r} \right)}{\partial r} \right\} \quad \ldots\ldots\ldots\ldots \quad 5.3$$

$$\text{or} \quad \frac{dp}{dz} = \eta \left\{ \frac{d^2 v_z}{dr^2} + \frac{1}{r} \frac{dv_z}{r} \right\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.4$$

Applying boundary conditions to this equation, that is, velocity v is zero when r is R,

the solution is

$$v_r = -\frac{1}{4\eta} \frac{dp}{dz} \left(R^2 - r^2 \right) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.5$$

and when, $r = 0$ and the velocity v_r is v_m

then

$$v_m = -\frac{1}{4\eta} \cdot \frac{dp}{dz} \cdot R^2 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.6$$

It can be seen that the velocity profile across the tube is parabolic, and therefore the mean velocity $\bar{v}$ is equal to half the maximum velocity

$$V = \frac{1}{8\eta} \cdot R^2 \cdot \left\{ -\frac{dp}{dx} \right\} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.7$$

but $V = \pi R^2 V$,

$$\text{hence,} \quad V = \frac{\pi R^4}{8.\eta} \left\{ -\frac{dp}{dx} \right\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.8$$

which again is the Hagen-Poiseuille equation.

This simple equation is inadequate for accurate work in the above form since conditions (b), (c) and (d) are not really satisfied.

A correction for (c), the slip at the walls of the tube, can be made by increasing the radius of the tube by a factor $\left\{ 1 + \frac{4\varepsilon}{r} \right\}$, where r is the radius of the tube, and ε is a constant, which is thought to be a function of the mean free path of the molecules of the fluid in the tube. In effect, slip may be assumed to be the slipping of the fluid at the boundaries.

In 1875, Kundt and Warburg (125) performed a series of experiments upon the damping of a vibrating disc surrounded by a gas. They found that at low pressures the damping decreased. This effect they ascribed to a slipping of the gas over the walls of the tube. Their interpretation of the phenomenon has been confirmed by later work, both experimentally and theoretically. In 1879, Maxwell developed a theory for the value of slip. Although not perfect, it is the best that has been put forward. He utilised for the purpose the results of an elaborate analysis that he had previously made of the stresses in a working gas. His reasoning could be put into a very simple form (126) (127). He obtained that,

$$\varepsilon = 2e . \frac{2 - f}{f} . \left\{ \pi/2 \right\}^{1/2} . \frac{\eta}{p} . \left\{ RT \right\}^{1/2} \quad \ldots\ldots \; 5.9$$

or $$\varepsilon = 2e . \left\{ \left\{ 2 - f \right\} / f \right\} L \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 5.10$$

where T is the absolute temperature, R the gas for a gram, p the pressure, L the mean free path, e a constant lying between 0.491 and 0.499 (so that 2e approximately = unity), and f is the fraction of the tangential moment of the molecules given to the solid boundary.

The value of f is uncertain and could even exceed unity, indicating that the molecules are reflected, on the average, with a partial reversal of their tangential velocities. Assuming that the molecules are reflected diffusely according to the same cosine law that holds for the diffuse reflection of light, or for the effusion of molecules from a hole, (therefore being distributed as if they came from a Maxwellian gas at rest relative to the wall), $f = 1$, and therefore $\varepsilon = 2cL$. Hence, ε is very nearly equal to L.

Direct measurements for the value of slip have been made by Timiriazeff (128) using the method of two coaxial cylinders with the fluid between them. Stacy (129) and Van Dyke (130) have also used the coaxial cylinder method, but have employed a more accurate formula for their work.

Knudsen (40) did some experiments with flow in a tube to find the coefficient of slip. He obtained the formula for the mean free path as

$$L = \frac{8}{3}\eta \left[2/(\pi \rho p) \right]^{1/2} = \frac{2.128\ \eta}{(\rho p)^{1/2}} \qquad \text{5.11}$$

He carried out a substantial number of experiments with several different fluids on solids and concluded that the slip correction coefficient could be represented by the equation,

$$\varepsilon = c.\frac{8}{3}\eta \left[2/p\rho\pi \right]^{1/2} \qquad \text{5.12}$$

He found empirically that the average value of the constant 'c' was 0.81.

During the last half century a number of different formulae have been put forward for the mean free path L. It is therefore very difficult to say which is the best equation to use for calculating the slip correction. Obviously it would be better to obtain the values experimentally with the apparatus to be used for the viscosity measurements; unfortunately this would produce serious difficulties in the design. In the worst case the correction to the viscosity would be of the order of 0.5 %, and even an uncertainty of 20 % in the mean free path value would only mean an uncertainty in the viscosity of 0.1 %.

Assuming a Maxwell-Boltzmann velocity distribution, the mean free path can be established to be,

$$L = \frac{2.02.\ \eta}{(p\rho)^{1/2}} \quad \text{or} \quad L = 4039\ \eta \frac{(T)^{1/2}}{p} \quad \ldots\ldots\ldots \quad 5.13$$

where L is the average collision distance.

These equations were derived by simple kinetic theory, with molecules devoid of intermolecular attraction, in which case the free path of a molecule is a straight line between collisions. In actual fact the path is thought to be curved, increasing the collisions and therefore decreasing the length of the path between collisions. Sutherland (9) used the assumption of hard spheres but added a weak attraction between them, falling off rather rapidly with distance. Such an attraction acts in two ways to produce a shortening of the mean free path. He deduced that the mean free path in a homogeneous gas was of the form,

$$L = \frac{Lo}{(1 + C/T)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.14$$

where C is the Sutherland constant, T is the absolute temperature and Lo is the mean free path for the simple molecule. Unless qualified in some way the term mean free path is ambiguous. In common practice it means either L or Lo.

Summing up, the value of the slip correction can be expressed in a number of ways. Here are a few of them. Assuming F = 1 and 2s = 1, then the slip correction is

$$\left[8 \varepsilon / d \right] = \frac{8 \times 2.02 \eta}{d \, (p\rho)^{1/2}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.15$$

$$\text{or} \quad = \frac{8 \times 4039 \, \eta \, (T)^{1/2}}{d \, p} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.16$$

$$\text{or} \quad = \frac{8 \times 2.02 \, \eta}{(p\rho)^{1/2} \, (1 + C/T)} = \frac{8 \times 2.02 \, \eta}{d \left[1 + 1150/T \right] (p\rho)^{1/2}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.17$$

taking C from Shifrin's work to be 1150°C.

From Knudsen's work,

$$8 \varepsilon / d = \frac{0.81 \times 8 \times 2.128 \, \eta}{d \, (p\rho)^{1/2}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.18$$

The above four equations for the mean free path (not the slip) are plotted against temperature, see figure (5.1). The equation which was finally used by the writer was Knudsen's, since his equation is based on experimental work.

The viscosity equation with the slip correction is therefore,

$$\eta = \frac{\pi r^4 \Delta P}{8 \, l \, Q} \left[1 + 8eL/d \right] \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.19$$

Poiseuille's equation was altered to allow for compressible flow conditions, discussed by Erk (131). The equation which is

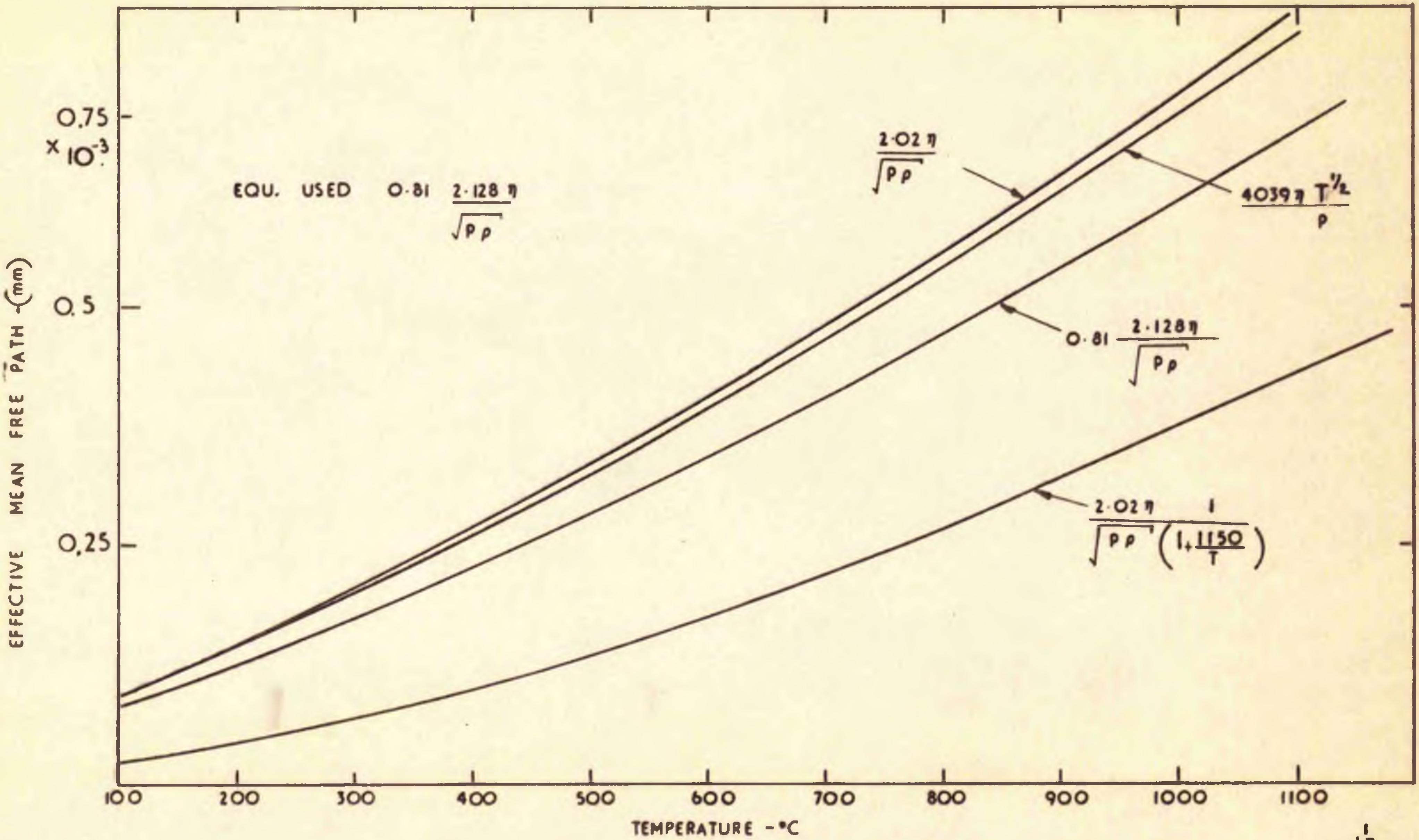


FIG 5.1 MEAN FREE PATH ∼ TEMPERATURE, BASED ON SHIFRIN'S VISCOSITY DATA.

based on the assumption of isothermal conditions, constant mass flow and ideal gas behaviour, may be written in the form

$$\eta = \frac{\pi r^4 \Delta P}{8 l V} \left\{ 1 - \frac{\Delta P}{2P_1} \right\} \left\{ 1 + \frac{4\xi}{r} \right\} \quad \ldots\ldots\ldots\ldots\ldots\ 5.20$$

where P_1 is the upstream pressure and V the volume flow rate.

For simple capillary flow conditions the above equation is still inadequate, since it neglects the acceleration of the flow at the entrance to the tube. At the entrance to the tube the velocity profile may be assumed to be uniform, at some distance along the tube this profile changes to parabolic. It therefore follows that there must be some drop in pressure due to the fact that the fluid will have gained some kinetic energy. This, in itself, would not affect the equation for the viscosity, if the velocity profile could be fully converted to a uniformly distributed profile at the exit and therefore recover this energy. This cannot be fully achieved and allowances must be made for this loss of head. The kinetic energy given to the fluid per unit time is,

$$= \int_0^R \frac{1}{2} v^2 \left(2\pi r \rho v \right) \delta r. \quad = \frac{\rho V^3}{\pi^2 R^4} \quad \ldots\ldots\ldots\ldots\ldots\ 5.21$$

The effective pressure drop in the transition length then becomes

$$= \frac{\rho . V^2}{\pi^2 . R^4}$$

This loss of pressure head does not take into account the actual viscous friction in this transition length. In order to allow for this, the end correction is assumed to be of the form, $\frac{m.Q^2}{\pi R^4}$, where 'm' is a constant. The length of the tube over which this transition takes place is called the transition length. Several theoretical and experimental determinations of this length and the value of the constant have been made over the last half century or so. The transition length usually defined in the form $\left\{\frac{x}{d}\right\}_{99\%} = \beta$ Re, where x is the distance from the entrance where the flow is 99% parabolic, d is the diameter of the tube and β is a constant.

Boussinesq (132), Schiller (133) (134) (135) and Atkinson and Goldstein (136) have all obtained values for the constants β and 'm', using approximate forms of the Navier-Stokes equation. Boussinesq, assuming a rounded entrance, derived a value of 'm' = 1.12. Many derivations have since failed to produce a rigorous value of 'm' for square cut ends. The values obtained for the constant 'm' vary from 1 - 1.205, from both experimental and theoretical work. Experimental results have reduced the scatter of the value of 'm' to 1.08 to 1.21; however, it can be seen that 'm' cannot be very well defined. Some experimenters believe that the constant varies with both temperature and Reynolds number. Unfortunately there are not enough results of sufficient accuracy available to prove or disprove this theory.

A further correction, first suggested by Couette (137), is the correction to allow for the viscous forces between the converging and diverging streamlines outside the entrance and exit of a capillary. This correction is usually achieved by increasing the length of the capillary by amount 'nr', to (L + nr), where 'n' is a constant of the order of unity. In 1895, Knibbs (138) derived a graphical method of evaluating m and n, and used it to analyse Poiseuille's data and deduced that n was zero. Barr (30) described several other attempts to find n, concluding that the best value was n = 0.57. This value has been used by several authors. On the other hand, Swindells, Coe and Godfrey (139) found that 'n' was probably less than 0.57 and perhaps even zero. It also seems evident that 'n' should be included in the constant 'm'.

For some considerable time the magnitude of the end corrections have been discussed. Much of the earlier work has been discussed by Barr (50). The main complication in establishing 'm' appears to be in disassociating the two constants 'm' and 'n'. Since the equation of flow for a capillary viscometer can be expressed as,

$$\eta = \frac{\pi d^4 g \Delta P \rho}{128 (L + nr)} - \frac{m Q}{8\pi (L + nr)} \text{, neglecting slip.....5.22}$$

defining $\eta_p = \frac{\pi d^4 g \Delta P \rho}{128}$ and $X = \frac{Q}{8\pi}$

equation (5.22) can be expressed as,

$$\eta_p = \eta \left[1 + nr/\lambda \right] + mX \text{ ....5.23}$$

It is obvious that unless η is accurately known any variation in the expected form of the equation may either be due to 'n' or 'm'. Therefore, none of the constants can be evaluated without a knowledge of the other constants. However, equation (5.23) may be plotted, and if n = 0, may be used in the form $\eta_p = -\eta + mX$, where 'm' will be the slope and η the intercept. As Re approaches zero the curve becomes horizontal, at least for rounded entrances.

Dorsey (140) investigated the measurements that Bond (141) made with capillary tubes with square cut ends and found that 'm' = 0 and 'n' = 1.146 when Re < 10, and 'm' = 1 and 'n' 0.57 when Re > 10 with no apparent transition region. However, it was pointed out by Barr that Bond's data for Re > 10 are unreliable. Using capillaries with tapering ends, Tuomikoski (142), Peter and Wagner (143) and Cannon, Manning and Bell (144) have observed that the end correction constant 'm' is less for tapering ends than it is for rounded entries, when Re is low, (n = 0). Swindells, Hardy and Cottington (145) observed a similar effect for square cut ends: their observations are not what might be expected, but may have been due to the fact that in some cases they "fire-polished" the ends of the tubes. It is believed that 'm' can be made as small as required by suitably flaring the bore at the ends of the tubes. However, there is no definite proof of the truth of this statement. There is also some speculation that 'm' is constant down to quite low values of Re, for tubes with square cut ends, while this appears to be reasonable, there is no evidence to bear this out.

Riemann (146) found that 'm' had a constant value of 1.124 ± .006, in the range 200 to 1010, and stated that he did not support the results found by Bond, that 'm' decreases as Re increases, when Re is greater than 700.

Flynn (61) and Lemmir (60) obtained values for the constant 'm', using several different gases. Their results indicated a value of 'm' equal to 1.175 ± 0.014 , using Reynolds numbers up to 1600. The authors did note that in some cases it was difficult to ascertain the value of 'm' and in some cases got values between -1.62 and 2.19.

Reynolds found that the upper limit for the Reynolds number, for which laminar flow will prevail in tubes is 2000. Nissan (147), however, claimed that deviations from laminar flow conditions can occur when Re > 1000, which is open to doubt, since he neglected the K.E. corrections.

To sum up, due to the lack of evidence, 'n' may be assumed to be zero. The value of 'm' is uncertain and may vary considerably with the type of ends (between 1.08 to 1.21). At present the variation of 'm' with density or Re appears to be small, although 'n' and 'm' have not been examined separately. However, Cannon, Manning and Bell (144) did find that $m = 0.037 (Re)^{0.5}$, but with square cut ended capillaries. It is possible, and perhaps most reliable, to obtain the end correction constant for individual capillaries using the equation $\eta_p = \eta + mK$. That is, the value of 'm' may be deduced by varying the mass flow rate through a given capillary, whilst keeping the temperature constant.

To the present date, Langharr's (148) results for the transition length, assuming round entry, must be considered to be the most accurate. He found that the centre line velocity would reach 99 % of its maximum value according to the equation

$$\left\{\frac{x}{d}\right\}_{99\,\%} = 0.0575\ Re. \qquad \text{, where x is the development length, and d is the diameter of the tube} \ldots\ldots 3.24$$

The experimental determinations of Nikuradse (149), Shapiro and Smith (150) and Reshotko (151) tend to strengthen the results obtained by Langharr (148). Theoretical investigations have been made by Boussinesq (132), Schiller (133), Atkinson and Goldstein (136), Tatsumi (152), Langharr (148), Shapiro (153), and Siegal and Kline (154). Experimental determinations have been made by Nikuradse (149), Shapiro and Smith (150), Shapiro and Kline (155), and Reshotko (151).

As stated before, Boussinesq employed an approximate form of the Navier-Stokes equation to obtain a solution for the development length. For the purely laminar zone he predicted,

$$\left\{\frac{x}{d}\right\} = 0.065 \text{ Re} \quad \ldots\ldots\ldots\ldots \quad 5.25$$

for a bell-mouthed entry.

Schiller assumed that the velocity profile in the transition length was composed of a straight line segment, terminated by parabolic arcs, and by applying the momentum equation to the entry cross section and the Bernouille equation to the central frictionless core of the fluid, he obtained,

$$\left\{\frac{x}{d}\right\} = 0.029 \text{ Re} \quad \ldots\ldots\ldots\ldots \quad 5.26$$

The main objection to his reasoning was his assumption of a central core. Nikuradse, did some experimental work that showed that up to approximately $\left\{\frac{x}{d}\right\} = 0.01$ Re the assumption of the frictionless core was justified. When $x/(d \text{ Re})$ is larger this central core does not exist.

Atkinson and Goldstein also used the approximate form of the Navier-Stokes equation, improving on the work of Boussinesq by employing a series solution based on the Blasius boundary layer equation.

The solution which retains the largest number of terms in the Navier-Stokes equation is that of Langharr.

Kline, Shapiro and Siegal presented three theories for the treatment of the transition length, and like Tatsumi are similar in approach to Schiller.

The results of Atkinson and Goldstein indicate that Langhaar's results are approximately 2 % high. A great deal of the work on the transition length has been done using high Reynolds numbers, that is when $[x/(dRe)]$ is very small, and has not been discussed here. For any calculations for the transition length, the equation attributed to Langhaar will be used. That is,

$$[x/(dRe)] = 0.0575 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 5.27$$

Two more corrections can be applied to the basic equation. These are, corrections for conicality and ellipticity of the bore of the capillary tube. If the included angle of the cone is small and the bore uniformly conical, for small flow rates Knibbs (138) proved that a correction for r^4 can be made in the form,

$\dfrac{3\,r_1^3\,r_2^3}{(r_1^2 + r_1r_2 + r_2^2)}$, where r_1 and r_2 are the 5.28

radii at the ends of the tube.

Knibbs actually preferred to express the conicality by $e = \dfrac{(r_1 - r_2)}{(r_1 + r_2)}$ in which case, if $2r = r_1 + r_2$, the simplified equation for r^4 is,

$$r^4 \; [\, 1 + (10/3)e^2 + (37/9)e^4 + \ldots\ldots\ldots \,] \ldots\ldots\ldots\ldots \; 5.29$$

However, in the majority of cases r is found by a volume method and not by direct measurement of the end diameters, in which case the correction is

$$\frac{r^4\,(1 - e^2)}{(1 + e^3/3)} = r^4 \; [\, 1 - 4e^2 + 20e^4/3 \ldots\ldots\ldots \,] \ldots\ldots \; 5.30$$

Hemmy (156) raised objections to the total neglect of the radial velocities and introduced a factor of ($1 - 2e$) by taking into account the variation of the kinetic energy per unit volume, along the tube.

It has been assumed that the capillary bore is perfectly circular, however, it is quite likely that the bore may be elliptic. It is difficult to assess the ellipticity of the bore of a tube and the effect it has on the flow equation. Starting with the definition of viscosity, it can be proved that a correction for r^4 in the Poiseuille equation can be made in the form, $(2b^3c^3) / (b^2 + c^2)$, where b and c are the semi-axes of the ellipse. Knibbs pointed out that if the ellipticity is defined by $\in = (b - c) / (b + c)$, then for small flow rates, using a bore of mean radius $r = \frac{1}{2} (b + c)$, r^4 may be corrected by multiplying by $(1 - 4 \in^2 + 7 \in^4 + \ldots)$...5.31

Therefore, allowing for both conicality and ellipticity, the fourth power of the radius, if obtained by a volume method, must be replaced by

$$r^4 (1 - 2 \in^2 - 4e^2 \ldots) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 5.32$$

If the conicality and ellipticity are small, the above corrections are very small and may be neglected. The main difficulty is in the actual physical measurement of the conicality and ellipticity of the bores of the tubes with any accuracy. Two methods used to check the ellipticity are (1) to X-ray the tube filled with mercury, radiographs being taken with the tube rotated into several positions and (2) to cut the tube into small sections and actually measure the axes. The accuracy of the former method is open to some doubt, since the rays have to come from what might be considered to be a point source, producing a pen-umbra. The latter method is not always convenient. Further comments on measuring the bores of tubes are given in appendix (5).

Two final corrections which do not really concern the work with steam, but are thought to be worth mentioning are, (1) the inconsistency of the value P/ρ for the fluid along the tube, and (2) when the flow is not truly isothermal.

Trautz and Weizel (29) derived a correction for the case when the gas is not ideal, that is, inconsistency of the ratio P/ρ along the capillary. Using Van der Waals equation of state, neglecting higher order terms, the correction may be made by multiplying ΔP by $\left(1 + \frac{C \, \Delta P}{2} \right)$, where $C = (b/RT) - (a /(RT)^2)$, a and b being the Van der Waals constants. This correction is usually very small and therefore neglected.

When the gas flow is not really isothermal, according to Bond (141) a correction factor for this effect should be taken into account. This correction amounts to $\left[1 + (\gamma - 1)(V/c)^2\right]$ where 'γ' is the ratio of the specific heats, and c is the velocity of sound. Like the previous correction this is small and therefore usually neglected.

Therefore, in conclusion, the fully developed equation for a single capillary, assuming compressible flow, is

$$\eta = \frac{\pi d^4 \, \Delta P}{128 \, l \, V_1} \left\{ 1 - \frac{\Delta P}{2Pl} \right\} \left\{ 1 + \frac{8\xi}{d} \right\} \left\{ 1 + 3\Delta t \alpha \right\} - \frac{\rho_1 V_1 \left\{ m + \log e \, P_1/P_2 \right\}}{8 \pi l (1 + \alpha \Delta t)} \quad \text{.......... } 5.33$$

where is the coefficient of linear expansion for the material of the capillary tube and suffices 1 and 2 refer to upstream and downstream conditions and ΔP is the pressure drop across the tube.

The corresponding flow equation, applicable when steam is the fluid being measured, is,

$$\eta = \frac{\pi d^4 \Delta P g}{128 l Q V_s} \left[1 + 8\varepsilon/d \right] \left[1 + 3\alpha\Delta t \right] - \frac{m Q}{8\pi l (1 + \alpha\Delta t)} \quad \ldots 5.34$$

where, V_s is the specific volume of steam at the mean pressure in the capillary.

The present work deals with a viscometer in which the two capillaries are in series. The equations for the two capillaries may be expressed as,

$$\eta = \frac{\pi d_1^4 \Delta P_1 g (1 + 8 \varepsilon/d_1) \left[1 + 3\Delta t\alpha\right]}{128 Q Vs_1} - \frac{m_1 Q}{(1 + \Delta t \alpha) 8\pi l}$$

$$\eta = \frac{\pi d_2^4 \Delta P_2 g (1 + 8 \varepsilon/d_2) \left[1 + 3\Delta t\alpha\right]}{128 Q Vs_2} - \frac{m_2 Q}{(1 + \Delta t \alpha) 8\pi l} \quad \ldots . 5.35$$

where suffices 1 and 2 refer to capillaries 1 and 2 respectively.

Making the assumption that the end corrections m_1 and m_2 are very nearly the same and equal to m, and treating the equations simultaneously to eliminate m, we get,

$$\eta = \frac{\pi g d_1^4 \left[1 + 8\varepsilon/d \right] (1 + 3\Delta t\alpha)}{128 Q (l_1 - l_2) f} \left\{ \frac{\Delta P_1}{Vs_1} - \frac{\Delta P_2}{Vs_2} (d_2/d_1)^4 K \right\} \quad ..5.36$$

where K is the ratio $(1 + 8 \varepsilon/d_2) / (1 + 8 \varepsilon/d_1)$, which is small and therefore neglected in the actual working equation, f is the buoyancy correction applied to Q and d is the mean value of d_1 and

d_2, if $d_1 = d_2$. The buoyancy correction for the weighings is

$$f = \frac{\left\{1 - \rho_a/\rho_{vt}\right\}}{\left\{1 - \rho_a/\rho_w\right\}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.37$$

where ρ_a, ρ_{vt} and ρ_w are the densities of air, balance weights and water respectively, Vs_1 and Vs_2 are the specific volumes at the mean pressures in their respective tubes. If Pa is the atmospheric pressure and $\Delta P'$ the pressure drop between the exit plenum chamber and the condenser, the mean pressures in each tube will be

$$\left.\begin{aligned} Pm_1 &= Pa + \Delta P' + \frac{\Delta P_1}{2} \\ \text{and} \quad Pm_2 &= Pa + \Delta P' + \Delta P_1 + \frac{\Delta P_2}{2} \end{aligned}\right\} \quad \ldots\ldots\ldots\ldots\ldots \quad 5.38$$

.

Chapter VI

Design and Development of the Apparatus

Since the primary aim of this project was to obtain the viscosity of steam at atmospheric pressure over the temperature range 100°C to 1100°C, the initial step was to decide on the type of material to be used for the viscometer. There are quite a number of materials available for use up to, and above 1100°C, the temperature ceiling which was set. Unfortunately, only a few materials are suitable for the intended work. The short list was: metals, the stainless steel group and the noble metals (mainly platinum and its alloys), ceramics and quartz.

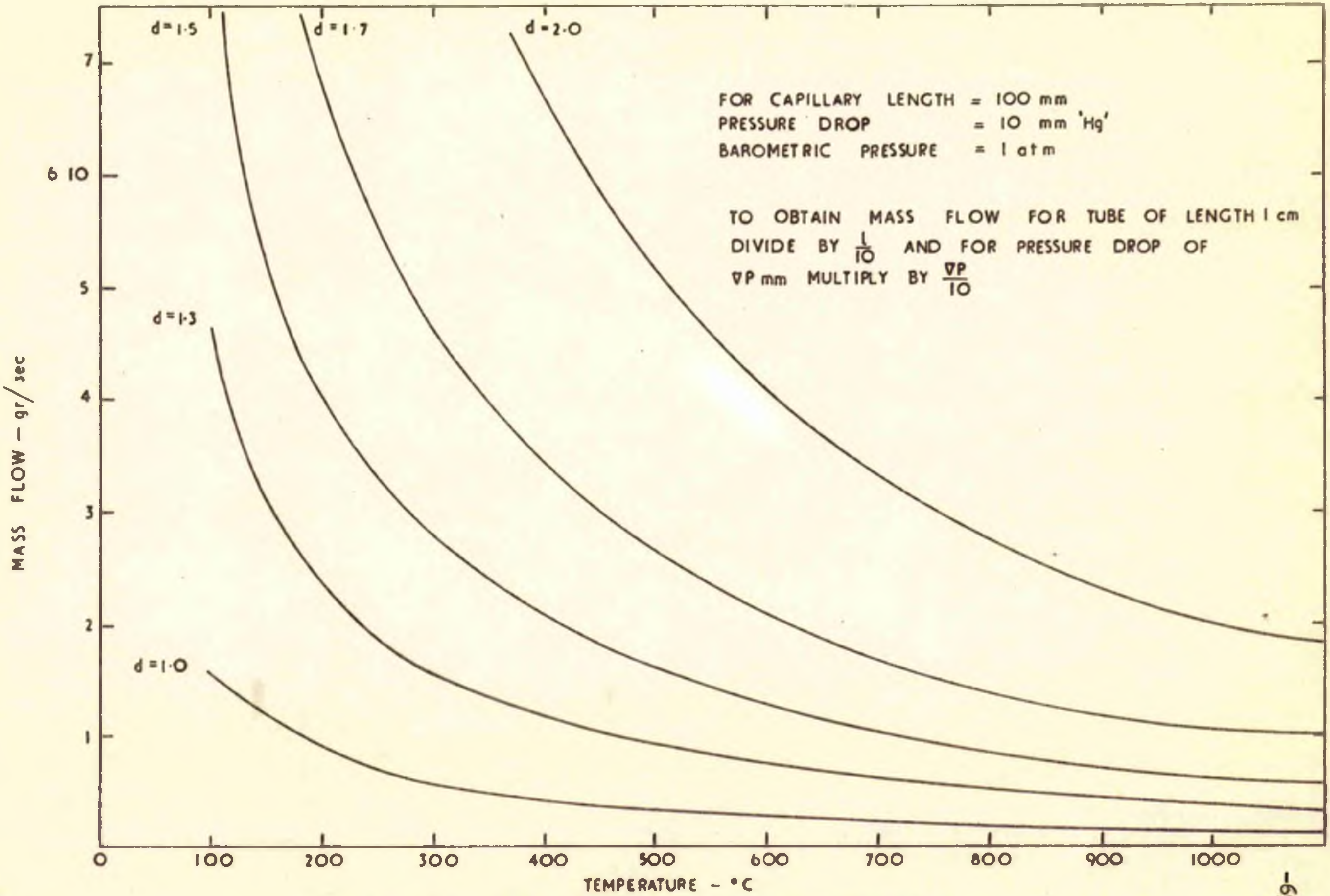
At this point it would be necessary to note that, since the system is intended to be a primary open circuit one, the diameter of the capillary is required to a high degree of accuracy. It was envisaged that the capillary diameters would be of the order of 1 mm to 2 mm. There are several standard methods of measuring the bores of capillary tubes, see appendix (5), the majority requiring transparent media for the capillaries. This, together with the temperature limit, was the criterion for deciding on the material or materials to be used.

There are only a few nickel-chromium, chromium or oxidation resistant steels which are suitable for very high temperature work, the majority having high oxidation rates at temperatures above 800°C. The platinum alloys would be suitable with regard to the temperature consideration, but there would be difficulty in determining the bores accurately and the cost would be very high. After some discussion with the manufacturers, it was concluded that ceramics could not be produced to any suitable tolerance. This left quartz, which is transparent and can stand being repeatedly heated up to about 1060°C without any devitrification (melting at about the same temperature as platinum, between 1700°C and 1800°C). Quartz also appears to be highly resistant to chemical attack and is inert to steam and air at high temperatures and atmospheric pressure. The main disadvantage is its fragile nature. Nevertheless it was decided that the viscometer should be made of quartz.

One of the initial considerations of the system was that the capillaries should be removable, after use, in order that they could be re-calibrated. The need for removal of the capillaries could be overcome by the use of a check fluid; a stable gas which could be passed through the apparatus, before and after use, thus checking the stability of the system. (This should not be confused with a calibrating fluid). However, a further reason for having removable capillaries is that the diameters and the length ratios could be varied. Although a given diameter size of capillary could be used over a wide temperature range, there are both practical and theoretical difficulties which arise when it is used over too wide a temperature range. The development length x in the entrance section of the capillaries has been assessed as being dependent on the bore of the capillary and the Reynolds number of the fluid flow, i.e. $x = 0.0575 \text{ Re } d$ see chapter (V). It is therefore obvious that this is one of the criteria determining the minimum temperature for which a capillary of given bore, having a given pressure drop across it, can be used. The upper temperature limit is dependent on the practical consideration of collecting a small mass of condensate, for a given pressure drop across the capillary, apart from the obvious limiting properties of the material of the capillary.

There appeared to be two main ways of making it possible to remove the capillaries, (1) to have standard ground conical joints and (2) to weld the tubes to the rest of the system. Arrangement (1) has two main disadvantages, the likelihood of leaks at the joints and a diffusion of the materials at the joints, which in effect tends to produce a weld at elevated temperatures. Arrangement (2) has the advantage that there would be no leaks; however, it would be necessary to weld the capillary in position, with the likelihood of distortion of the capillary bore and the breaking of the joint to remove the capillary. For practical reasons, method (1) was initially investigated. In the first place, standard conical glass joints of the B.5 and B.10 were tested at temperatures up to 500°C for leakage with different internal pressures, see appendix (1). The maximum leakage rate, with a 5 cm mercury (gauge) internal pressure, was about 0.63g/sec, which is very small, even considering that with two capillaries four joints will be used. After this, several B.5 and B.10 standard conical quartz joints were subjected to temperatures up to 1400°C. Up to about 1200°C the joints could be parted, with some difficulty, by soaking them in alcohol or penetrating oil. However, if heated to above 1200°C, they could not be parted.

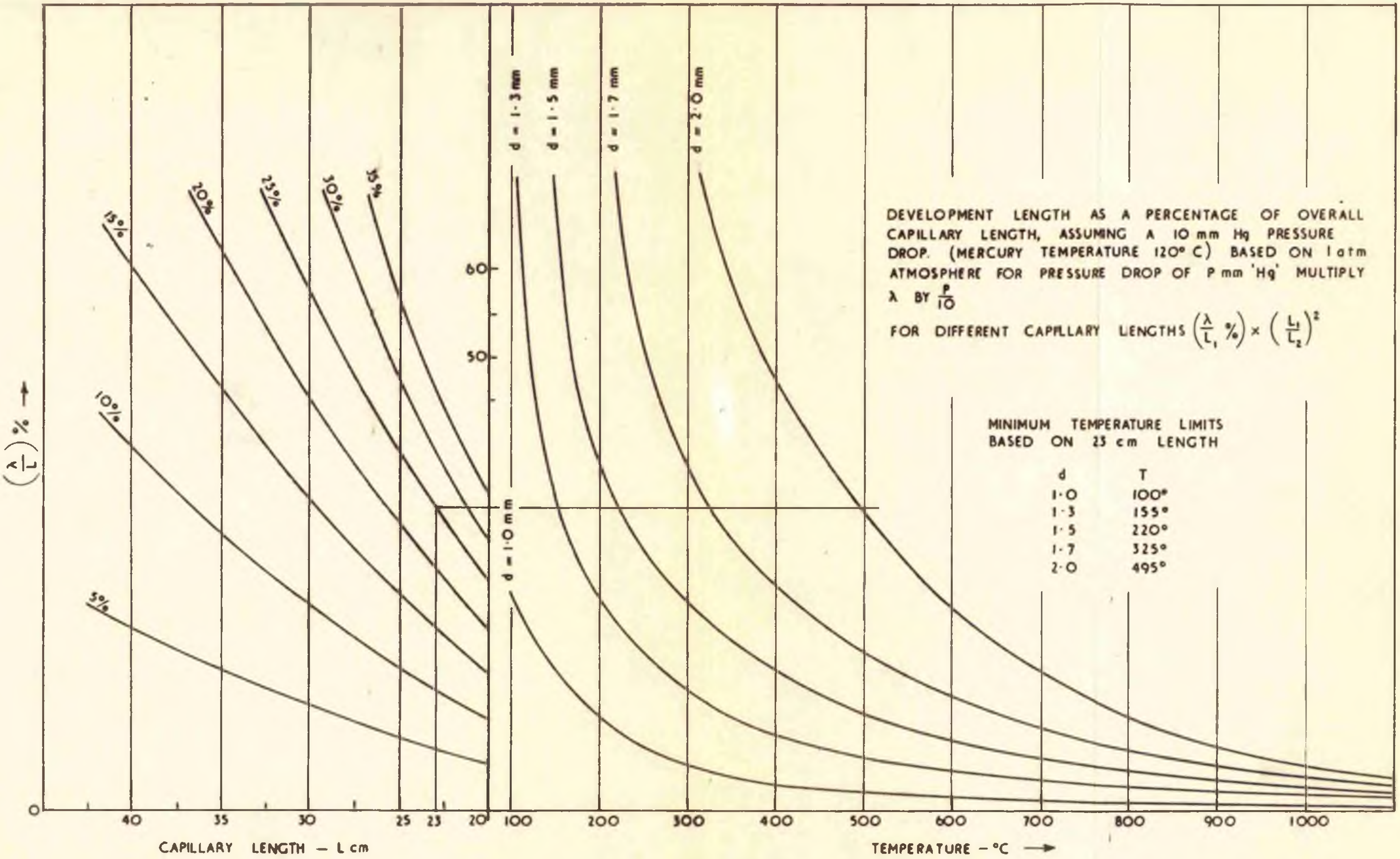
The results of the joint tests being reasonable, it was then necessary to decide on the lengths and the bores of the capillaries. The three main factors influencing the bores and lengths are as follows:



FOR CAPILLARY LENGTH = 100 mm
PRESSURE DROP = 10 mm 'Hg'
BAROMETRIC PRESSURE = 1 atm

TO OBTAIN MASS FLOW FOR TUBE OF LENGTH 1 cm DIVIDE BY $\frac{1}{10}$ AND FOR PRESSURE DROP OF ∇P mm MULTIPLY BY $\frac{\nabla P}{10}$

FIG 6.1 MASS FLOW AGAINST TEMPERATURE



DEVELOPMENT LENGTH AS A PERCENTAGE OF OVERALL CAPILLARY LENGTH, ASSUMING A 10 mm Hg PRESSURE DROP. (MERCURY TEMPERATURE 120° C) BASED ON 1 atm ATMOSPHERE FOR PRESSURE DROP OF P mm 'Hg' MULTIPLY λ BY $\frac{P}{10}$

FOR DIFFERENT CAPILLARY LENGTHS $\left(\frac{\lambda}{L_1}\%\right) \times \left(\frac{L_1}{L_2}\right)^2$

MINIMUM TEMPERATURE LIMITS
BASED ON 23 cm LENGTH

d	T
1·0	100°
1·3	155°
1·5	220°
1·7	325°
2·0	495°

(1) The Reynolds Number. It has been well established that for stable laminar flow conditions to prevail in the tube, the Reynolds number should be less than 2000, although laminar conditions can exist at higher Reynolds numbers, the conditions being then inherently unstable.

(2) The Length of the Entrance Transition Length. This obviously should not be greater than the length of the capillary tube or else there would not be any Poiseuille flow in the tube.

(3) The practical difficulty of collecting small quanities of water or steam. The method used to collect exhausting fluid governs the minimum mass flow that can be tolerated.

To investigate conditions (1) and (3) curves of mass flow against temperature for various diameters with a given pressure drop were plotted, (see figure 6.1), using existing viscosity data. To investigate condition (2), curves of development length against temperature for given diameters with a constant pressure drop were plotted, see figure (6.2). From these curves estimations were made for the diameters and the length ratios for the capillaries. It was decided that where possible the development length should not exceed 25 % of the overall tube length, although in actual fact experiments were carried out with the development length over 50% of the overall length.

At this juncture, it was decided to make a simple single capillary viscometer out of glass, in order that the auxiliary equipment could be developed and decided upon. The main auxiliary equipment being a manometer, a collection system, a boiler, a superheater and pressure stabiliser, furnaces and control equipment.

Auxiliary Equipment and Design of the Single Capillary Apparatus

Boiler The boiler was the first item to be developed and at first sight this would appear to be a simple task. However, after several tests it was found to be difficult to maintain steady ebullition when using de-mineralised water. When a simple externally heated boiler was used to boil super-pure water, the water adjacent to the wall of the vessel superheated prior to vapourising and the resulting sudden ebullition produced large pressure fluctuations. A simple rig was set up, to develop a suitable boiler, in which the steam from a spherical flask passed through a suitable capillary. The pressure at the boiler was measured using a simple mercury-in-glass U tube manometer. In an attempt to reduce the pressure fluctuations obtained when using an externally heated boiler, glass beads, very small glass capillary tubes and stainless steel wire gauze were in turn put into the bottom of the boiler. The pressure fluctuations did decreased with each modification, but were not reduced sufficiently.

A nichrome element was then wound on to a 2 cm diameter glass tube (see figure (6.3)). The heater was fully immersed in the water and used as a heating element. This proved to be very successful, the pressure fluctuations being reduced to about 1 mm of mercury. This type of heating element was used for the single and the series capillary systems.

A standard cyclone type of boiler was adopted (see figure (6.4)) in which the heater is in a side arm, the steam entering the main body tangentially, producing a vortex in the main body. The boiler for the single capillary system was specially made up. Its

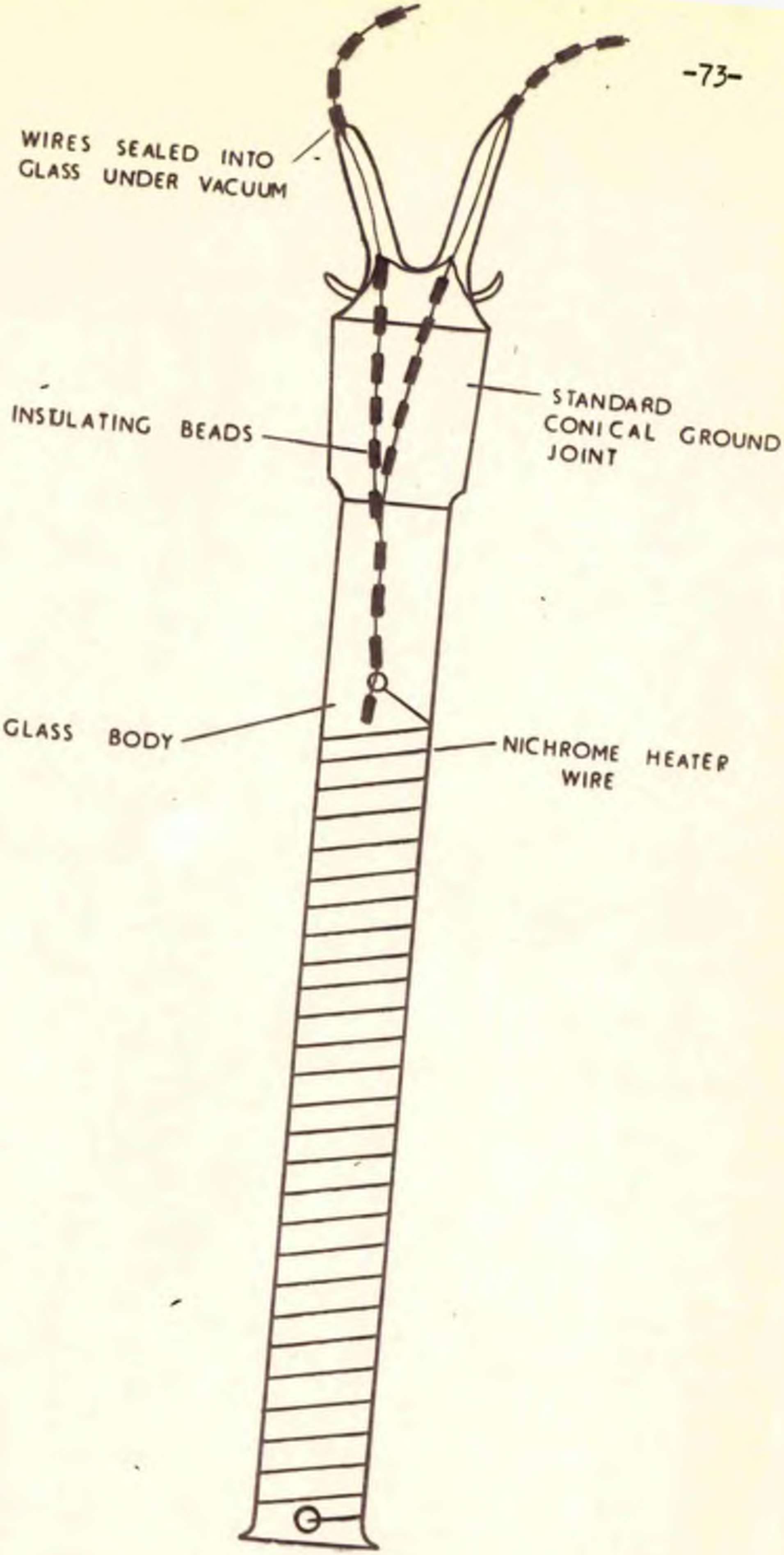


FIG 6·3 CONTROL HEATER USED FOR BOILERS

capacity was about one litre and it had only one 1 Kw heater of the type described above. The boiler for the series capillary system was bought from Messrs. Quickfit and Quartz and had a water capacity of one and a half litres with three one-kilowatt heaters. One of the heaters was made in the way previously described and used as a control heater. The heaters supplied with the boiler were encased in glass and proved to be useless as control heaters; however, they were used to boil the water initially. The control heater then took over when the water commenced to boil.

A further refinement was attempted with the first boiler. It was assumed that if high boiling rates were used, the boiling stability would improve. With this in mind, it was decided to have a loop circuit, with a condenser in this circuit and to tap off small quantities of steam for the viscometer. The loop circuit with the condenser in it can be seen in figure (6.4). This was not a success, since only slight variations in the condenser cooling water inlet temperature and/or flow rate produced alarming pressure fluctuations.

After the greater part of the experimental work had been completed, it was found that Collins and Keyes (157) had successfully used a heating element which floated on the surface of the water in the boiler.

A mercury-filled control manometer was connected to the boiler, and the manometer was connected to a Sunvic electronic relay which 'cut in' and 'out' a rheostat which was in series with the control heater. A variac (variable transformer) was used for course adjustment of the control heater power, the control manometer varying the heater power by 1 % to 2 %. Even with this control system the pressure of the

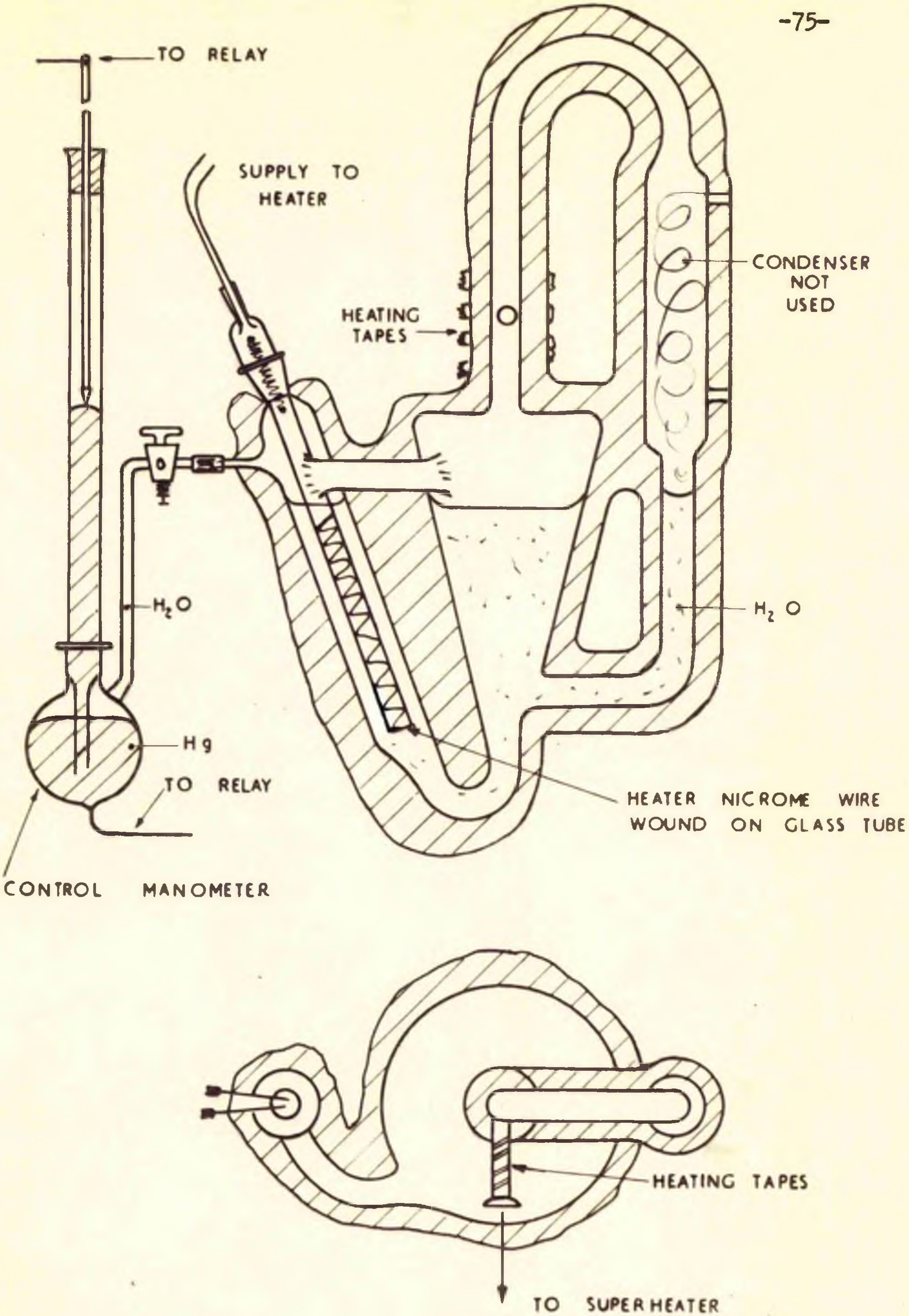


FIG 6. 4 BOILER FOR INITIAL APPARATUS

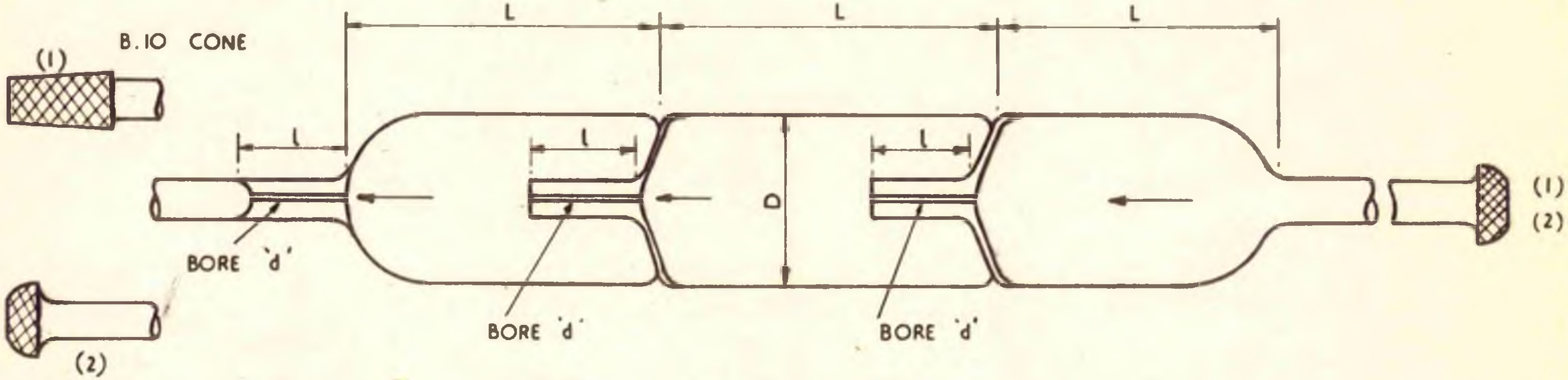
steam issuing from the boiler varied by as much as 0.5 mm of mercury.

Superheater and Pressure Stabiliser

Initially a large spiral glass tube was connected to the boiler for use as a superheater. This functioned satisfactorily but the pressure fluctuation from the boiler was only slightly damped out. It was therefore decided that either the pressure control at the boiler should be improved, or some type of throttling device should be used prior to the viscometer, to damp out any fluctuations. Novak (158) had some success with a damping arrangement which he used in a gas chromatography apparatus. A superheater (see figure (6.5)), based on his personal suggestions, was produced, initially in glass, later in quartz. This device proved successful in reducing the pressure fluctuations at the exit from the superheater down to 0.005 mm mercury. A standard electrical tube furnace was designed for the superheater. The superheater furnace was supplied from a 250 volt Sorensen voltage regulator, the voltage being varied using a Variac.

Viscometer

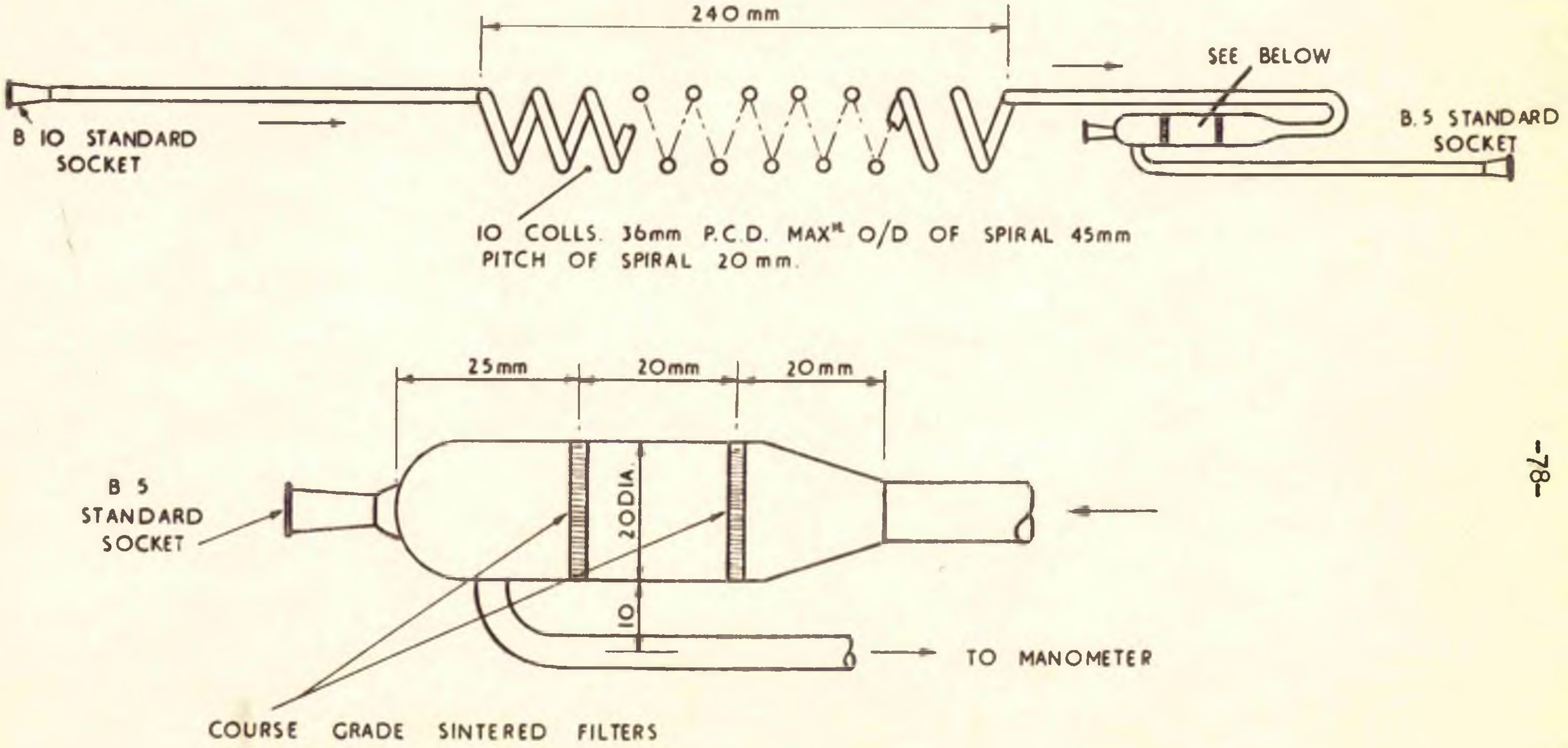
The viscometer is as shown in figures (6.6) (6.7) and (6.8). The lead in tube was a long spiral so that the steam could attain temperature stability prior to the capillary. An entrance plenum chamber is positioned at the end of the stabilising coil. The object of this plenum chamber is to ensure that the velocity of the steam at the entrance to the capillary is small in magnitude and uniform across the chamber. Some plenum chamber design experiments were carried out (see appendix (2)). From these tests, it was found that a 2 cm bore entrance plenum chamber, with two course grade sintered glass filters,



	JOINT	L	D	l	d
FOR SINGLE CAPILLARY SYSTEM	(1)	75 mm	$1\frac{1}{2}''$	30 mm	0·4 mm
FOR SERIES CAPILLARY SYSTEM	(2)	130 mm	$1\frac{3}{4}''$	25 mm	0·35 mm

$$\left(\frac{P_o}{P_i}\right)^{\frac{1}{n}} = \left\{ \frac{\left(\Delta P_i + \Delta P_o + 2P_o\right) r_g^4}{16 . f \eta l V} \right\} \text{— SEE APPENDIX.}$$

FIG 6. 5 PRESSURE STABILIZER AND SUPERHEATER



STABILISING COIL AND ENTRANCE PLENUM CHAMBER FOR SINGLE CAPILLARY APPARATUS
FIG 6.6

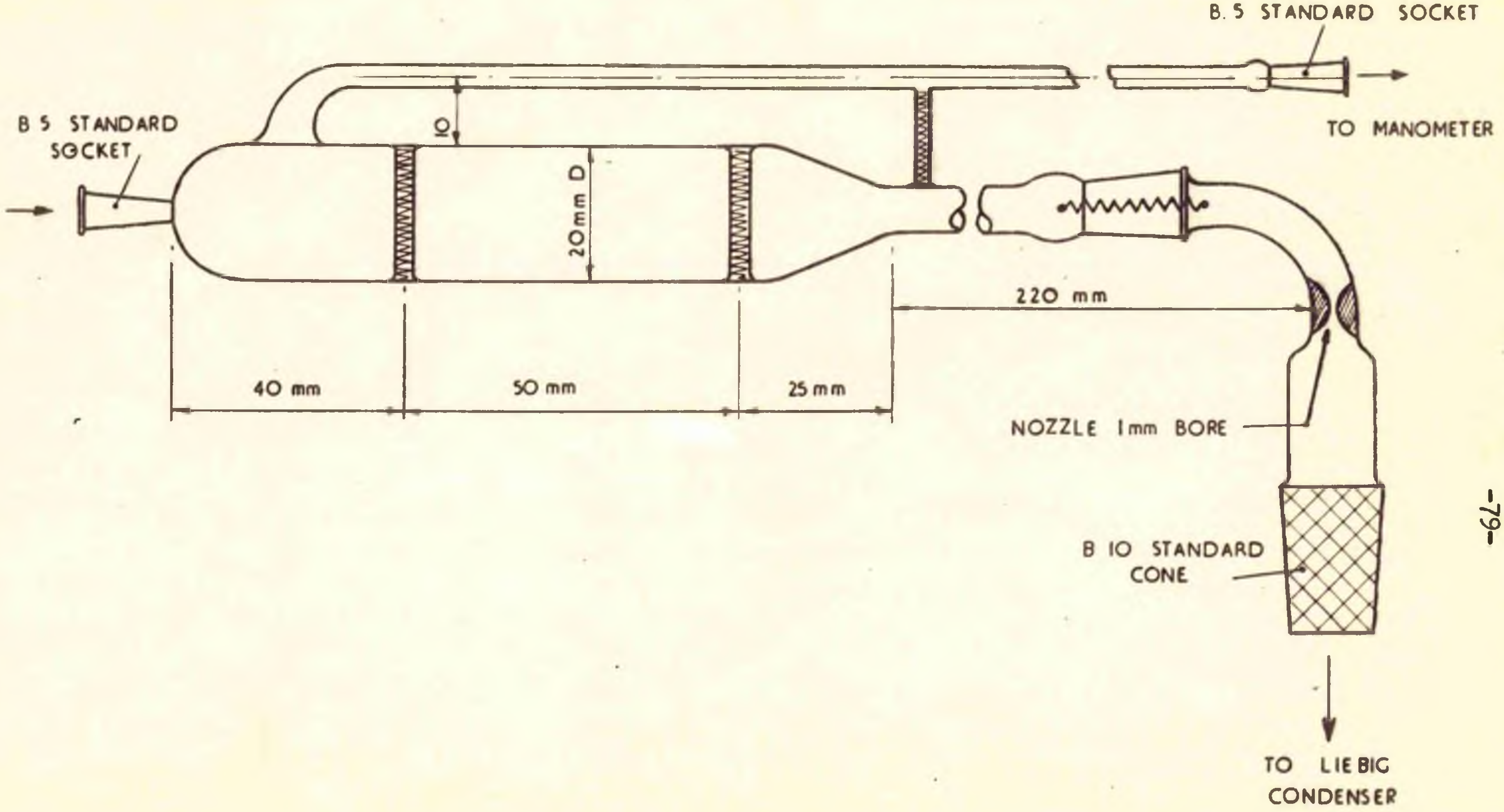


FIG 6.7 EXIT PLENUM CHAMBER FOR SINGLE CAPILLARY SYSTEM

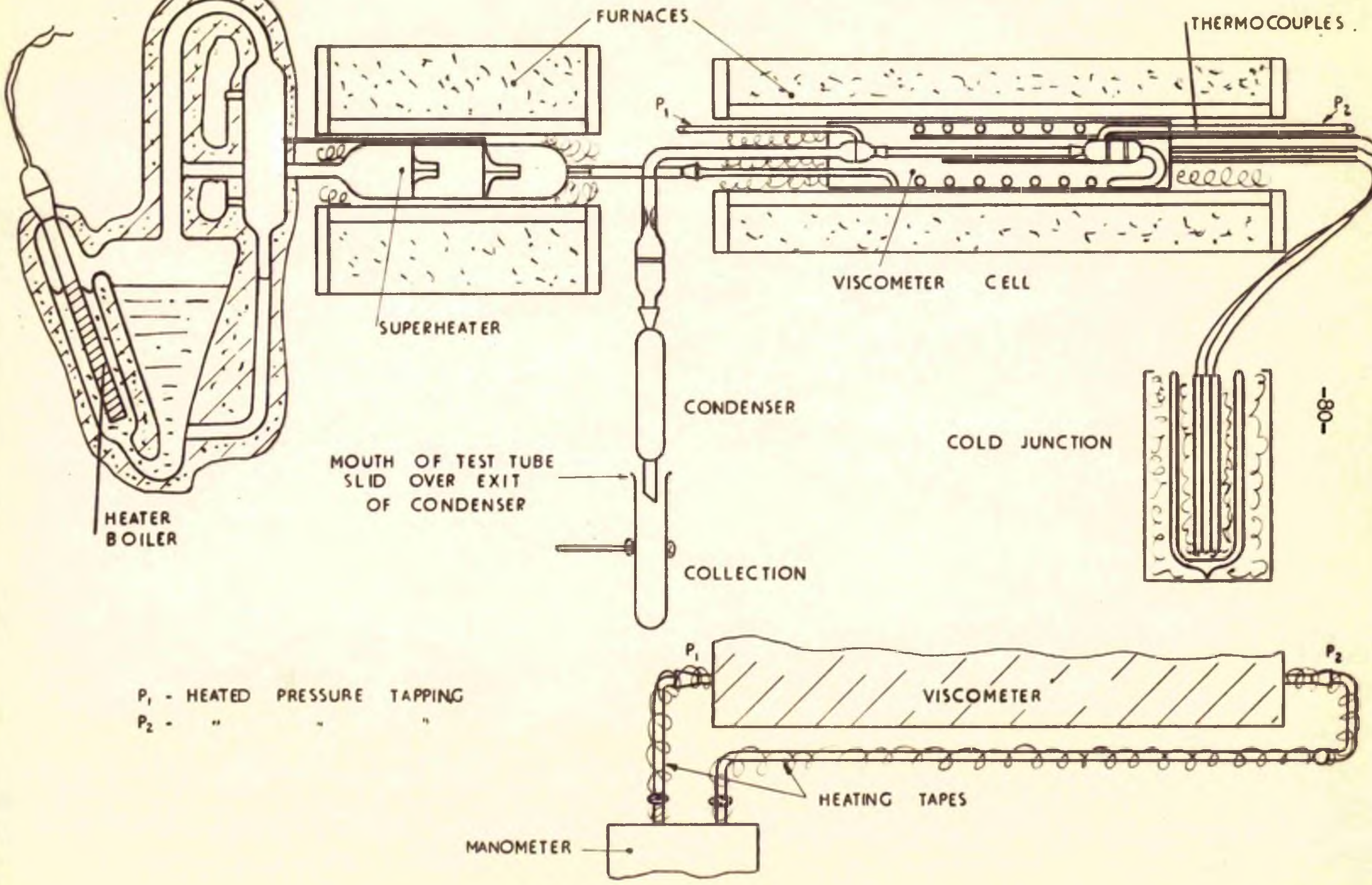
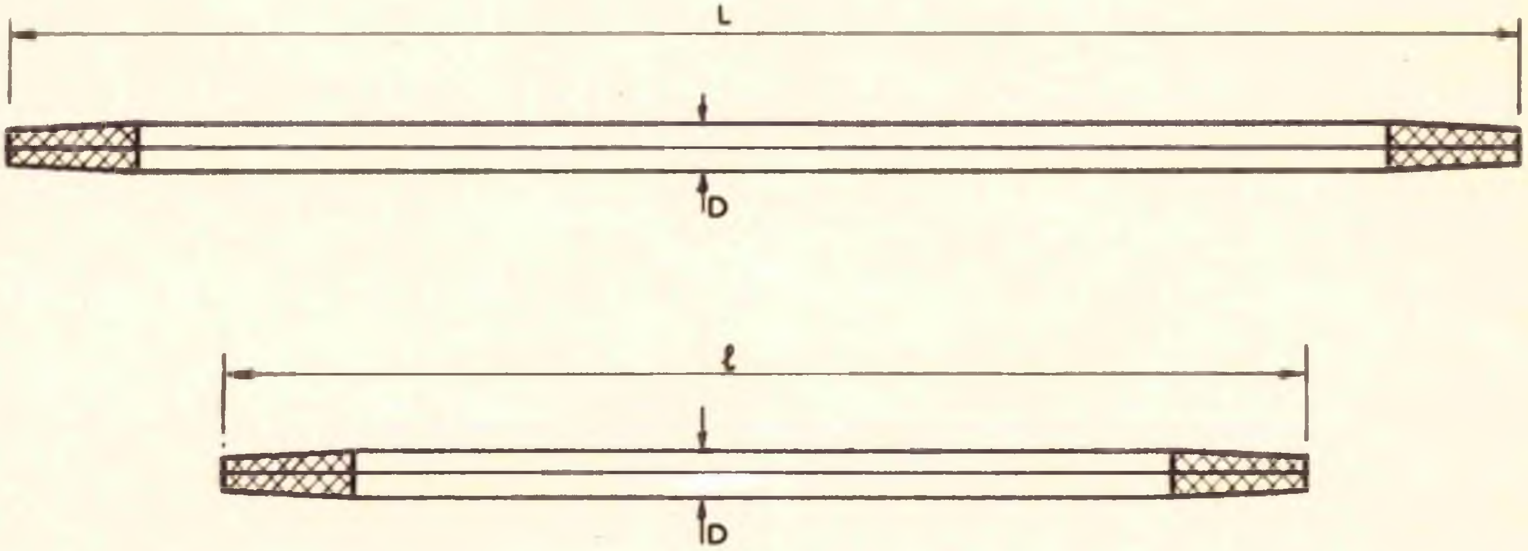

FURNACES
THERMOCOUPLES .
P₁
P₂
SUPERHEATER
VISCOMETER CELL
CONDENSER
COLD JUNCTION
MOUTH OF TEST TUBE SLID OVER EXIT OF CONDENSER
HEATER
BOILER
COLLECTION
P₁ - HEATED PRESSURE TAPPING
P₂ - " " "
P₁
VISCOMETER
P₂
HEATING TAPES
MANOMETER

FIG 6. 8

2 cm apart in it, would be the minimum requirements to ensure that there was no "coring" of the flow. Pressure tappings are taken from the entrance and exit plenum chambers, as near to the capillary ends as possible. The downstream tapping is taken out at the further end of the furnace and runs outside the furnace for its full length before reaching the manometer. All the tubes in the system which were outside the furnaces were heated with heating tapes, which are supplied through a Variac from a 250 volt stabilised voltage supply. The long pressure tapping outside the furnace caused a considerable amount of trouble, since, if any point along it dropped below the saturation temperature, condensation took place at this point. Any subsequent pressure flucutations, however small, moved this droplet into a hot section, producing instant ebullition and an accompanying pressure rise. The system then reacted like a feed-back oscillator at resonance, the amplitude of the fluctuations increasing until the mercury in the manometer was ejected.

Capillary

The initial capillary sizes selected were, length 30 cm, and two diameters 1.0 mm and 1.5 mm. The ends of the capillaries were ground to suit B.5. standard conical sockets, as this was the most suitable cone size for the outside diameter of the tube (see figure (6.9)). The first capillaries had a recess ground at the end of the cone as shown in figure (6.9). The recess was made to reduce the length of the cone, but it was found that the recess was a point of weakness and was therefore not included in later cones. A large number of capillaries were initially roughly calibrated and



① ENDS GROUND TO B. S STANDARD CONE SPECIFICATIONS.

② BOTH TUBES ARE CUT FROM THE SAME CAPILLARY TUBE TO INSURE THAT THE BORES ARE AS NEAR AS POSSIBLE EQUAL.

③ MINIMUM OUTSIDE DIAMETERS 'D' ARE 5mm.

ORIGINAL END GRINDING THE RECESS WEAKENED THE CAPILLARY RESULTING IN SEVERAL FRACTURES. ALL THE TUBES USED HAD THE ABOVE TYPE OF END CONE.

FIG 6. 9

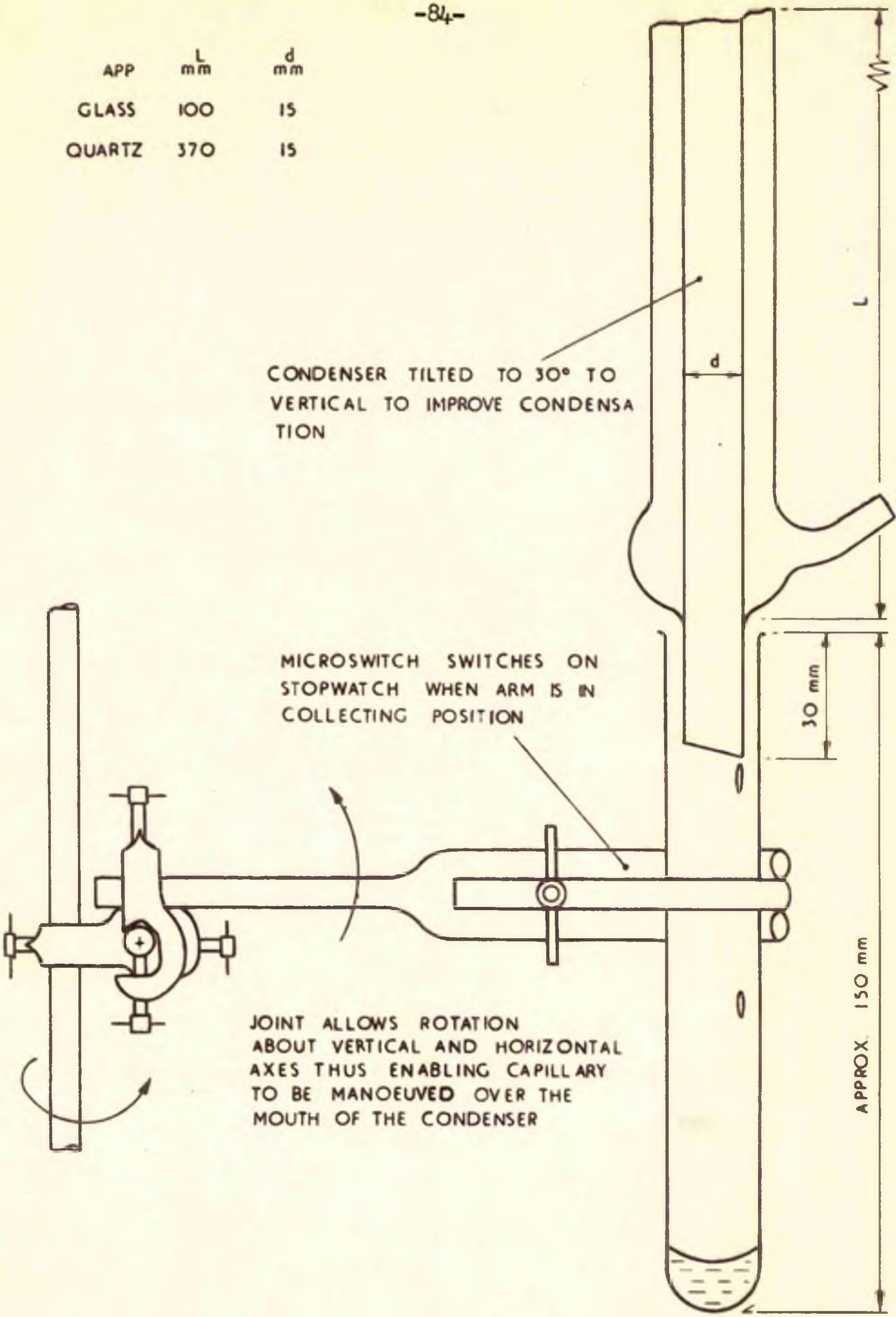
a short list made up of the better ones. After a second calibration the final capillaries were selected. For method of calibration see appendix (5).

Determination of Mass Flow

There appeared to be four main ways to determine the mass flow rate, (1) collecting the steam in a heated cylinder, (2) collecting the condensate, (3) putting a flow meter in either the supply or exhaust line, or (4) checking the rate of loss of weight of the boiler. Methods (1) and (3) have similar difficulties, in that they both require to be calibrated and that they both need excellent temperature stability. It is true that method (1), if perfected, could give continuous readings and would therefore be an excellent method. However, it was thought that without a considerable amount of development, measurements of sufficient accuracy could not be made with this type of instrument. Method (3) has the drawback that the meter needs to be calibrated and the author is not aware of any flow meter that has been produced to measure mass flow rates of the order of 0.01 to 0.001 gr/sec, with an accuracy of better than 0.1 %. Method (4) introduces many practical difficulties, i.e. having a very sensitive balance at the boiler which would not be affected by the heater leads and the main steam tube; also the tubing from the boiler would have to be completely leak-proof. It was therefore decided that method (2) was potentially the most likely to give results of sufficient accuracy.

Several types of condenser were tried for condensing the exhaust steam, the type which proved to be the most successful being the Liebig condenser. The condenser was hung at an angle of about 30°

APP	L mm	d mm
GLASS	100	15
QUARTZ	370	15



CONDENSER TILTED TO 30° TO VERTICAL TO IMPROVE CONDENSATION

MICROSWITCH SWITCHES ON STOPWATCH WHEN ARM IS IN COLLECTING POSITION

JOINT ALLOWS ROTATION ABOUT VERTICAL AND HORIZONTAL AXES THUS ENABLING CAPILLARY TO BE MANOEUVED OVER THE MOUTH OF THE CONDENSER

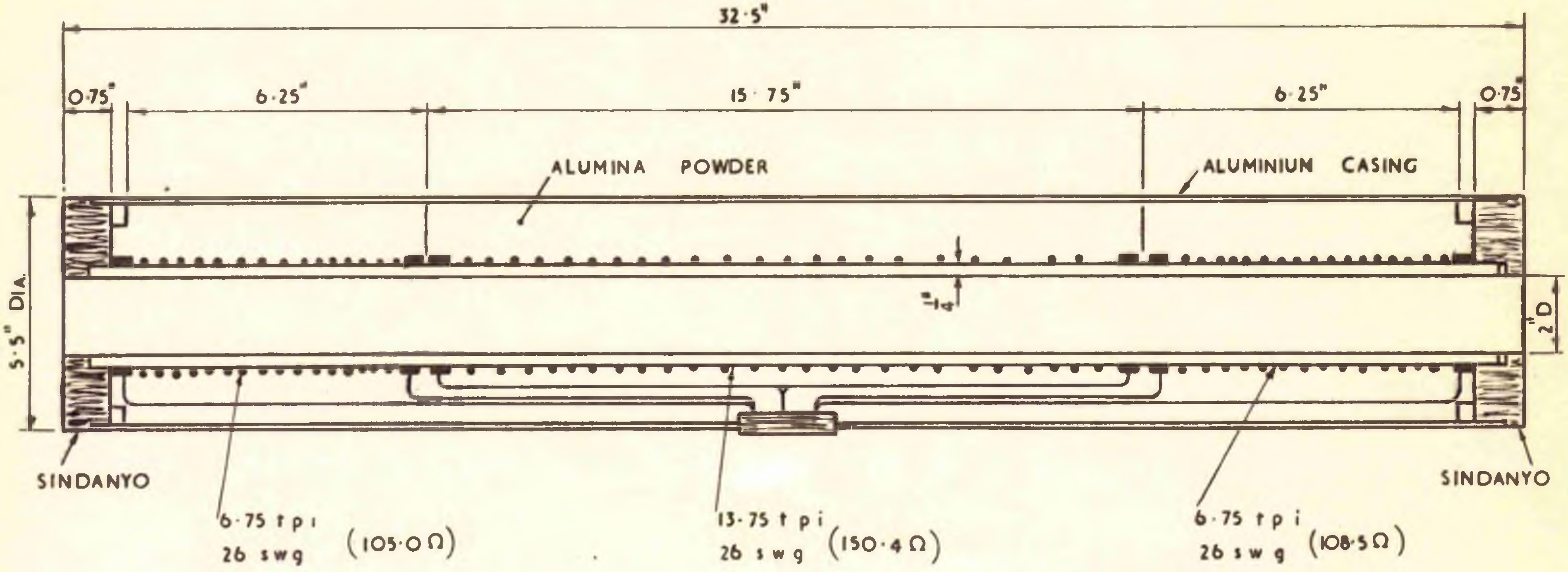
FIG 6. 10 CONDENSATE COLLECTION ARRANGEMENT to the vertical, as this stopped any supercooled vapour from leaving the condenser, but did not stop the water droplets from running down the condenser at regular intervals (see figure 6.10).

The condensate was collected in pre-weighed test tubes. Trial tests were made to check the evaporation rate from the condensate. It was found that the evaporation rate was negligibly small when the test tube was about 6 inches deep. Initially tests were carried out to check the best type of condensate receiver. The receptacles were varied in an attempt to smooth out the variable size of the droplets falling down the condenser. Unfortunately the results were not very successful and so the simple test tube was used as a receiver (see figure (6.10)). Under normal conditions, it was noticed that the time between droplets was remarkably constant for a given set of conditions, but small vibrations could very much upset the time between droplets. A later modification was to put a small piece of asbestos rope about half-way down the condenser and a small piece of quartz wool at the exit from the condenser. In both cases the materials did not fully fill the bore of the condenser but were only at the walls. The effect was only a mild damping, and tended to even out the frequency of the droplets. The condensate collection time was made by a Camerer Cuss relay-operated stopwatch, the system being tripped by a micro-switch situated under the condenser. For the calibration of the timing system see appendix (3). A 1 mm bore nozzle followed by a coarse sintered filter was put in the exit line prior to the condenser, in both apparatus. The object of the nozzle and sintered filter was to stop any diffusion of air back into the viscometer.

The main viscometer furnace for the single capillary system was designed on the standard tubular principle with three main windings (see figure (6.11)). The electrical power supply was from a Sorensen voltage regulator via three Variacs (see circuit diagram figure (6.26)).

Thermocouples

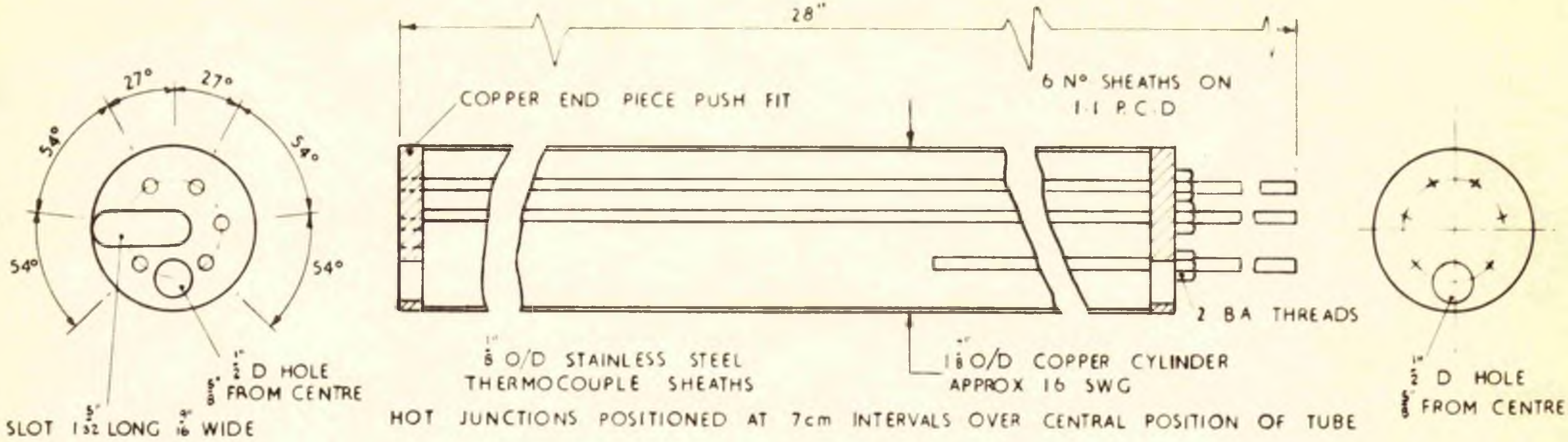
A thin wall copper tube was produced for a dual purpose. The tube housed the stabilising spiral and the capillary tube concentrically within it and also acted as a conductor to even out the temperature along the viscometer (see figure (6.12)). The tube also has six stainless steel thermocouple sheaths in one end of it, the position of these sheaths being such that they are on a P.C.D. between the outer stabilising spiral and the capillary. The hot junctions being at 7 cm intervals along the viscometer, the two extreme end ones are therefore 2.5 cm beyond the ends of the capillary. Initially the thermocouples were made of chromel-alumel wire, which were calibrated against a calibrated N.P.L. platinum-rhodium thermocouple. These thermocouples were scrapped because they proved unstable. Uncalibrated thermocouples made of platinum-platinum 10 % rhodium wire were finally used, a sample thermocouple being calibrated against a N.P.L. standard thermocouple. The calibration was found to be within the tolerance given by the manufacturers of the wire, Messrs. Johnson Mathey Ltd. The equipment used in conjunction with the thermocouples was a Tinsley Auto Vernier potentiometer, the smallest subdivision being 1 µV, a selector switch immersed in paraffin, a standard cell having a N.P.L. certificate and a Tinsley galvanometer.



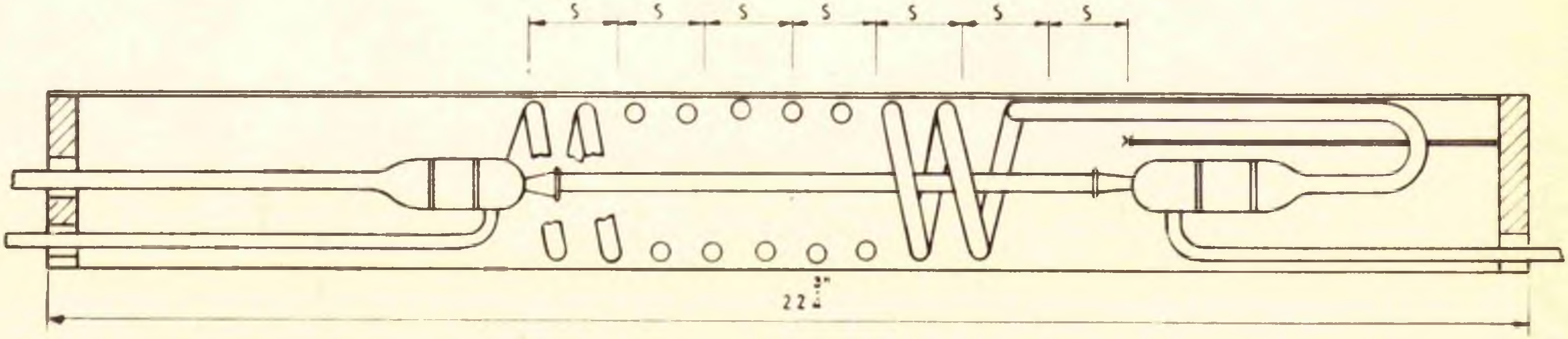
WIRE — BRIGHTRAY 'H' MAXIMUM OPERATING TEMPERATURE 1150 °C

FURNACE TUBE — 'H' MATERIAL - POROUS - CONTAINING 80% ALUMINA - FOR USE UP TO 1700 °C
-GOOD THERMAL SHOCK

FIG 6. 11 MAIN FURNACE FOR SINGLE CAPILLARY VISCOMETER



RADIATION SHIELD AND THERMOCOUPLE SUPPORT



DISTANCE BETWEEN THERMOCOUPLE POSITIONS 'S' = 2 3/4"

FIG 6.12 SINGLE CAPILLARY VISCOMETER ASSEMBLY

Manometer

A simplified form of the flow equation for the series capillary system, since the design is mainly for the series capillary system, can be written as,

$$\eta = K \left\{ \Delta P_1/Vs_1 - k\, \Delta P_2/Vs_2 \right\} ,$$

and assuming that $Vs_1 = Vs_2$, which is not strictly true, the equation becomes,

$$\eta = K \left\{ \Delta P_1 - k\, \Delta P_2 \right\} .$$

If $d_1 = d_2$, which is near enough for this purpose, then the equation may be further modified into

$$\eta = K \left\{ \Delta P_1 - k\, \Delta P_2 \right\}$$, since k will approximately equal unity.

Therefore,

$$\delta\eta/\eta = \sqrt{\frac{(\delta(\Delta P_1))^2}{(\Delta P_1 - \Delta P_2)^2} + \frac{(\delta(\Delta P_2))^2}{(\Delta P_1 - \Delta P_2)^2}}$$

It is therefore apparent that the error in the viscosity due to errors in the pressure measurement is not the straightforward sum of the two errors in the individual pressure drops, but a more complex effect. On account of this effect, the accuracy of the pressure measurement is one of the most important factors in the overall accuracy of the viscosity values.

A further point of which account had to be taken when deciding on the type of manometer to use, is that the fluid in which the pressure drop is to be measured, is steam. Consequently, the manometer must be heated or have some type of diaphragm to transfer the pressure or there must be a steam/water interface somewhere in the system. A brief resume of the types of manometer that were considered is given in the table on the following page. It was decided that a manometer which required, either a water/steam interface or a diaphragm in the pressure lines to transmit the pressure would not be suitable for the required purpose. A diaphragm reduces the sensitivity and in some cases reduces the accuracy, and a water/steam interface is a most unreliable method of transmitting the pressure, the latter method being especially unreliable because any slight flustuations in the pressure might move the water into the hot section and produce violent boiling with an associated pressure rise. It was therefore decided that a fluid-in-glass, U tube type of manometer would be used.

After some research into the matter, it was decided that of the types of manometers listed in the table, the mercury-in-glass instrument suited the required purpose best. The manometer tubes may either be inclined or vertical. The main reason for using inclined tubes is to increase the length of the mercury columns and therefore improve the sensitivity, but the angle of inclination must be measured accurately. There is also the added difficulty of reading the elongated menisci. The practical difficulties of alignment and measuring the angle of inclination of the columns outweigh the advantage of precision of the inclined column method. It was therefore decided that a vertical column instrument would be used. Although there are quite a few fluids which have a specific gravity that is much less than mercury, few are as adaptable. The majority of manometer fluids would require that either a diaphragm or a water/steam interface be used.

There appear to be four main methods of measuring the height

Type	Sensitivity	Accuracy	Remarks
Bourdon	1/500	1/300	Requires a diaphragm or a steam/water interface. Cannot be readily heated. Sensitivity and accuracy refer to instruments made by Messrs. Wallace and Tiernan.
Ring Balance	± 0.1 % f.s.d.	0.5 % f.s.d.	As above, sensitivity and accuracy refer to instruments made by Hagan Ring Balance Co. Ltd.
Micromanometer	± 3 %		As above, produced by the Infra Red Co.
Electrical Manometer	10^{-6} ins Hg.	10^{-6} ins Hg.	As above, see reference number (159)
Spoon or Spiral Gauges	Can be high but appears to be dependent on the auxiliary equipment.		Can be heated but requires calibration, see references (160) (161)
Electrolytic Manometer			Requires a diaphragm, see reference (162).
Betz Manometer	0.02 in H_2O	0.01 in H_2O	Would be required to be filled with Hg or, if water, would need a diaphragm, all reducing accuracy.
Diaphragm operating Strain Gauges	Dependent on the calibration.		Could be heated.
Fluid in Glass or Metal U tube Manometer	Dependent on equipment - could be high.		Could be heated, depending on the fluid used in the instrument

f.s.d. = full scale deflection

of a mercury column: (1) using a telescope with a cathetometer, (2) using probes which are connected to an electrical indicating circuit (3) using a capacitance method to indicate the surface level, and (4) using an inductive method to indicate the surface level.

Several tests were carried out using mild steel probes to detect mercury surfaces. An electronic relay was used in the electrical indicating circuit, since, the current required through the probe was of the order of micro-amps, and therefore too small to produce any appreciable oxidation of the mercury surface or the probe points. It was found that the sensitivity rapidly decreased with time and only slow pressure fluctuations could be picked up. The author did not try using an oscilloscope in the indicating circuit, although it is reckoned that such a method improves the accuracy. It was felt that the probe method was not sensitive enough to pressure fluctuations. Hanks (59) and later Flynn (61) and Lemaire (60) used a probe manometer. They reckoned that their accuracies were of the order of 0.0001 inch with a sensitivity of slightly less than this value, a fact which the author finds very hard to believe.

The capacitance method has constructional drawbacks, but would appear to be potentially the most accurate of the four methods, since the accuracy would almost entirely depend upon the micrometer heads operating the probes, the capacitance between the probe and the mercury surface being kept constant, that is, using a null method of indicating the surface. It should be appreciated that this method has the drawbacks that, the electrical circuit would require repeated calibration checks and that the capacitance of the arrangement could be easily upset, by slight alterations in the system in close proximity to the probes. These are not insurmountable problems, but should be borne in mind.

The inductive method is to have coils outside the manometer tubes, using a null method to indicate the surface of the mercury, a micrometer, attached to the coils, doing the actual measuring. This method, although it does not require leak-proof sealing of probes, does have all the drawbacks of the capacitance method.

The most straightforward method of measuring the height of the mercury columns is to use an optical system. There are a large number of variations of the basic system; however, the most straightforward is to view the mercury surface through a telescope attached to a graduated scale. The overall accuracy of this system is dependent on the accuracy of the measuring instrument (cathetometer) and the precision to which the mercury surface can be observed. Obviously the overall accuracy in all the above methods is also dependent upon the temperature stability inside and outside the manometer box.

Initially a three limb manometer was made. The main body of the manometer was made of mile steel and had three 2 cm diameter precision bore glass tubes for the columns. The tubes were sealed in at their top and bottom by glands using P.T.F.E. latern rings. It was found that although mild steel is quite inert to mercury, the mercury was contaminated at the working temperature used, and had to be replaced quite frequently; also at the working temperatures used, the P.T.F.E. packing tended to extrude and break the glass tubes. For these reasons this manometer was scrapped and replaced by an all glass one, (see figure 6.13). This manometer was placed in a Sindanyo box which has heating elements down two opposite sides and plate glass at its front and back (see figure 6.14). A thermometer recorded the mercury temperature and a contact thermometer situate in the manometer box controlled an electronic relay which cut in and out a rheostat in series with the manometer heaters. The power was supplied from a 110 volt "Sola" voltage stabiliser and was varied by the controller by about 40 % of the maximum power. This kept the temperature in the manometer box to within 1°C over a half hour period.

The centre column was viewed through a fixed telescope and therefore kept at a constant level. The outer columns were observed with a single cathetometer. The constant level in the central colum was achieved with the help of a "make up" cylinder, (see figure 6.15). The cylinder, which was connected to the manometer by a tube, had a coarse and a fine adjustment piston which could supply or remove mercury from the system and thereby keep the level of the centre column constant. The three columns were initially open to the atmosphere and the centre column adjusted to the position indicated by the fixed telescope. The two outer columns were then read and the readings used as zeros for subsequent pressure readings, no pressure readings being taken unless the centre column was properly "zeroed". The approximate relative positions of the two telescopes to the manometer are given in the figure (6.16).

Sighting of the mercury surfaces produced one of the biggest problems (see appendix (6)), since incorrect illumination of the mercury surface could produce an apparent meniscus. This was partly

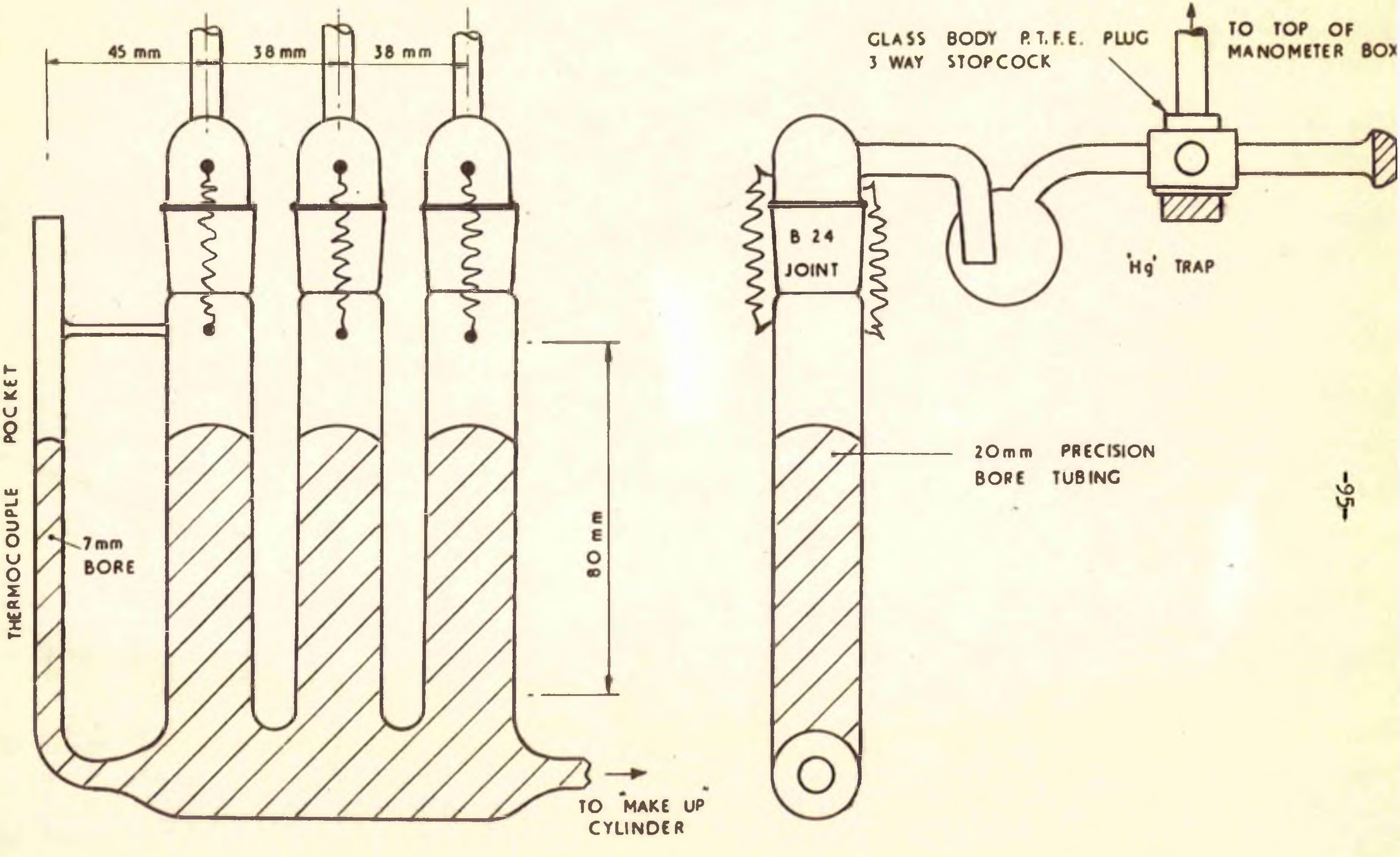
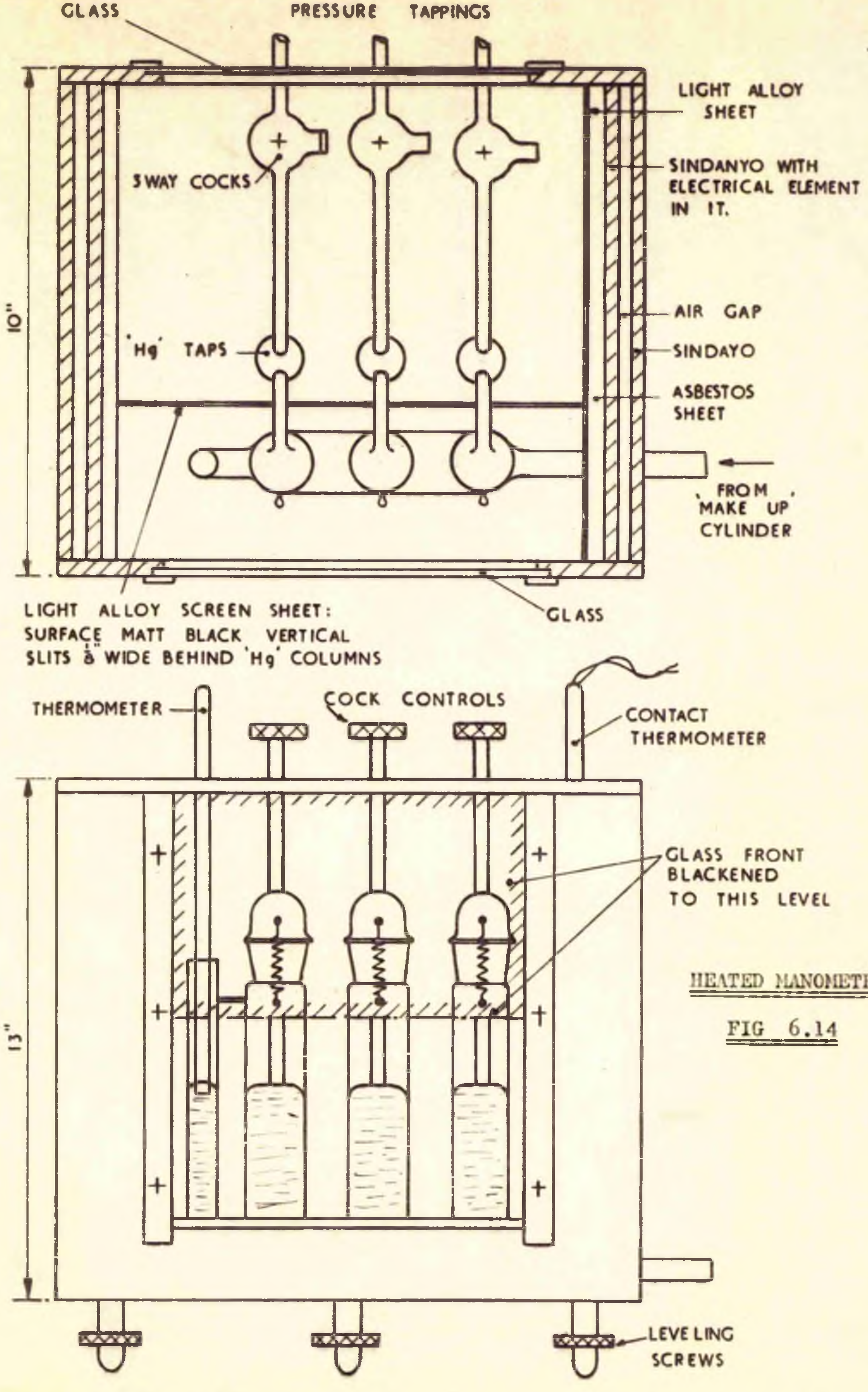

45 mm
38 mm
38 mm
THERMOCOUPLE POCKET
7mm BORE
80 mm
TO 'MAKE UP' CYLINDER
GLASS BODY P.T.F.E. PLUG
3 WAY STOPCOCK
TO TOP OF MANOMETER BOX
B 24 JOINT
'Hg' TRAP
20mm PRECISION BORE TUBING

FIG 6.13



HEATED MANOMETER

FIG 6.14

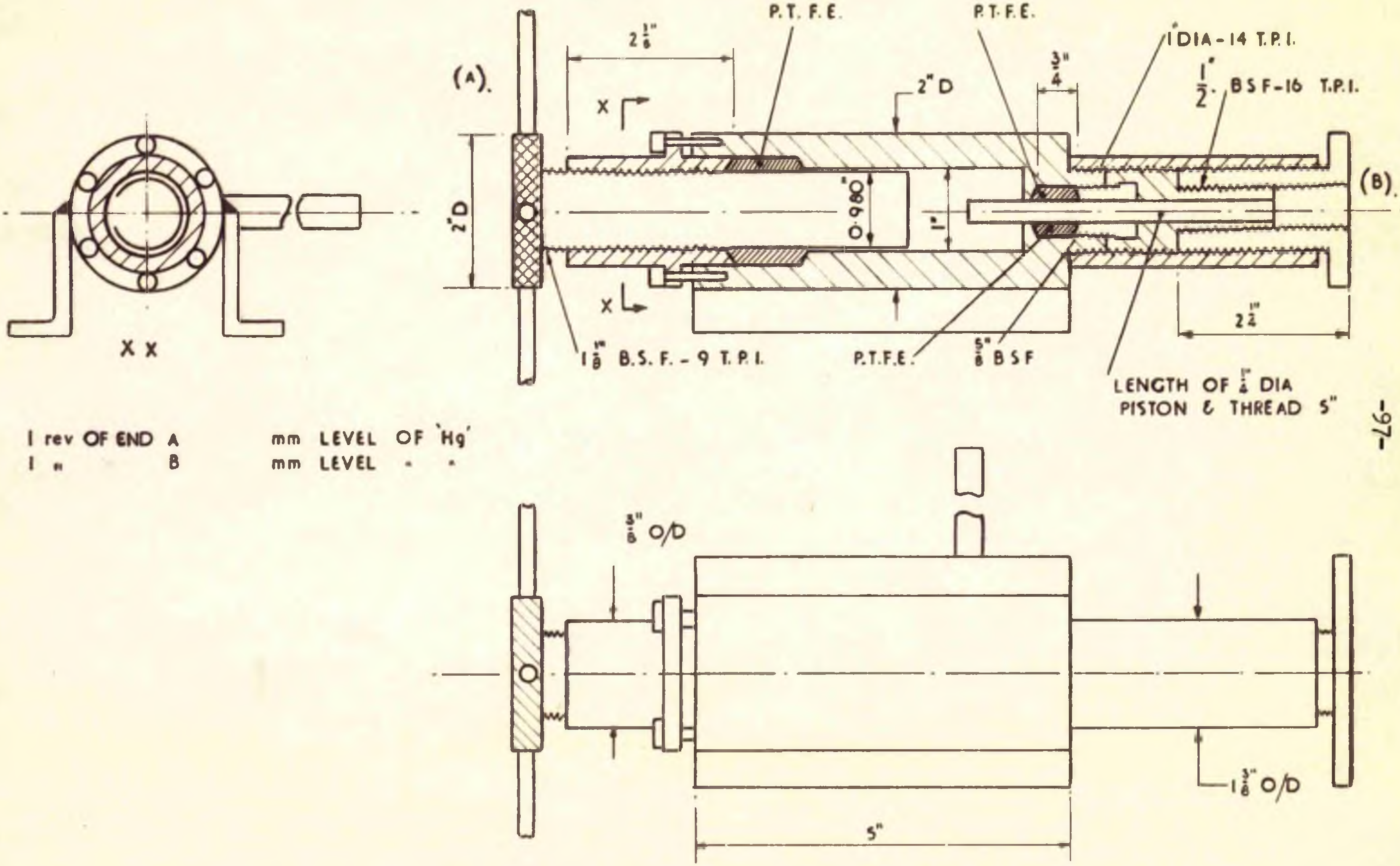


FIG 6.15 MAKE UP PISTON AND CYLINDER ASSEMBLY

overcome by putting a light alloy sheet, with three 1/8" slits in it, inside the manometer box, just behind the actual manometer. The slits being centrally positioned behind each of the columns. With illumination from the rear the top of the mercury meniscus appeared as a dark silhouette, only when the meniscus appeared in this way was a reading taken. A further improvement was made by blacking part of the front window to stop too much light getting in from the front and reflecting from the mercury surface.

Initially an electrically powered fan was fitted inside the top of the manometer box in an attempt to improve the temperature distribution in the box. This was later discarded since it tended to vibrate the mercury menisci and had little effect on the temperature distribution.

Three way "Excelo" flat type glass valves were originally fitted to each limb of the manometer. Unfortunately they needed frequent attention, such as greasing and tightening up, and on some occasions they leaked in certain positions. This type of valve was discarded and three way cocks produced by Messrs. Fisons Ltd. fitted in their place. These cocks have a glass body with a knurled P.T.F.E. conical plug. Although the P.T.F.E. plug could tolerate the temperatures experienced in the manometer box, they did tend to pull through the body after about three or four days use, when they were replaced. It was also necessary to tighten them up at the beginning of each day's experiments.

There are many refinements that can be applied to a manometer, but it was felt that any further refinements to the manometer were

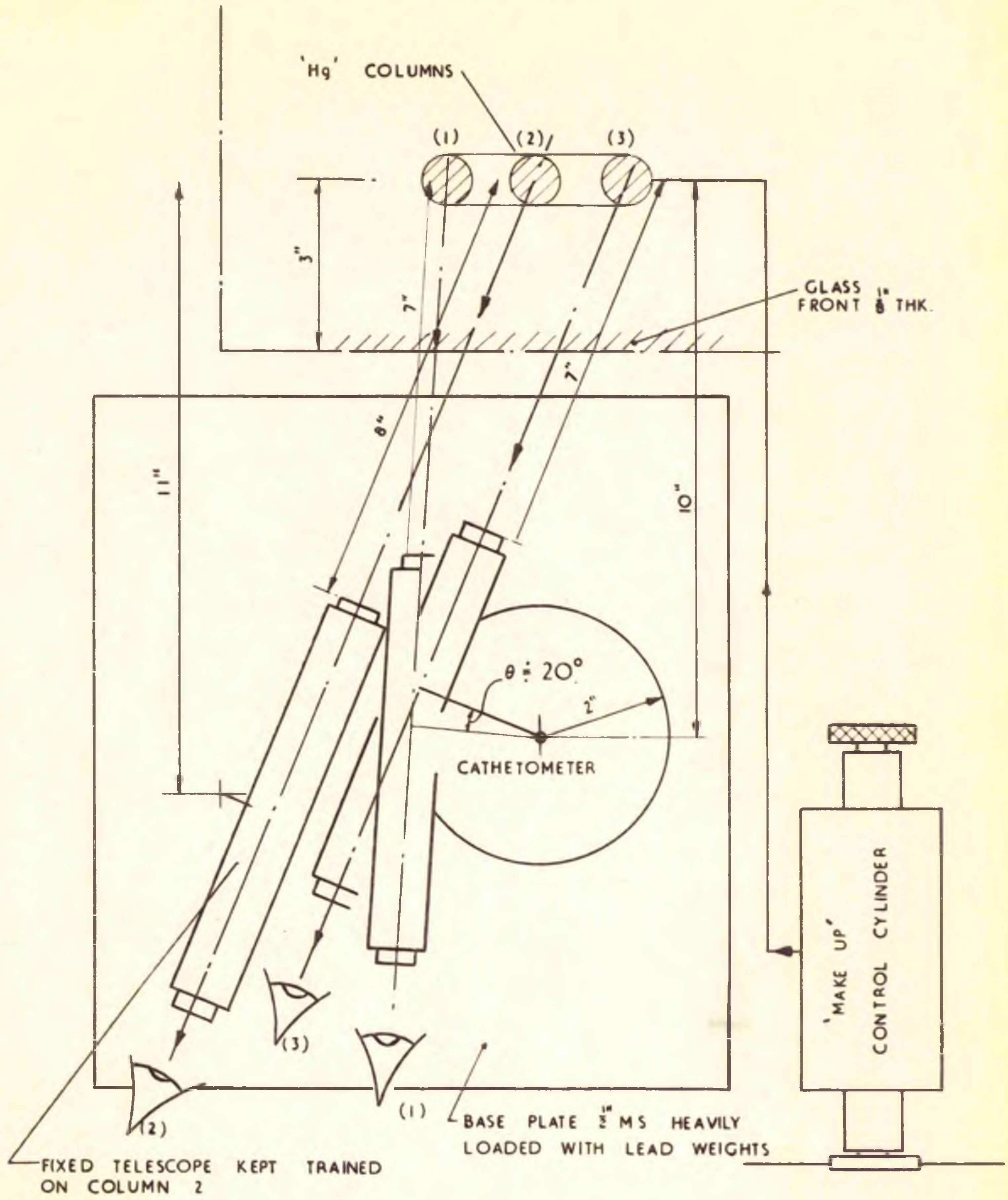


FIG 6.16 POSITIONS OF TELESCOPES W.R.T. 'Hg' COLUMNS

unnecessary under the circumstances.

On assembly of the single capillary viscometer, all the vacant spaces in the superheater furnace and the main furnace were filled with glass wool.

The Series Capillary Viscometer

Although results had been obtained from the single capillary system, its main object was to develop the subsidiary equipment and check a system using ground cone and spherical joints. Having obtained results for the viscosity of steam, at atmospheric pressure up to 450°C, that were within the tolerance limits recommended by the Munich International Steam Meeting of 1962 (163), i.e. ± 1 % between the temperature limits of 100°C and 300°C and ± 3 % between the temperature limits of 300°C and 900°C, it was decided to go ahead with the series capillary system.

The majority of the auxiliary control and measuring equipment was the same for both the apparatus. As stated previously, the boiler was larger, having a capacity of one and a half litres, a two kilowatt starting heater and a one kilowatt variable power control heater, (see figure (6.3.)).

The superheater and pressure stabiliser is larger and made of quartz, (for design see appendix (4) and figure (6.5)).

The Liebig condenser is also larger, since it must be able to deal with steam issuing from the viscometer at nearly 1100°C, (see figure (6.10)) for condenser and condensate collection arrangement).

Thermocouples

Seven new thermocouples made of platinum-platinum-10% rhodium wire were produced, all the wire being taken from the same batches of materials. One of these thermocouples was calibrated against a N.P.L. calibrated thermocouple, the calibration agreeing very well with the values issued by the manufacturers of the wire. The arrangement of the thermocouples in the apparatus can be seen in figures (6.17) and (6.24). Six of the couples were arranged in the apparatus so that their hot junctions were at equal intervals over the total length of the capillaries. That is, the distance between the hot junctions was about 8 cm. The capillaries were positioned along the central axis of the viscometer, on a diameter about two centimetres less than that of the P.C.D. of the capillaries. The seventh thermocouple was located in the centre of the group of thermocouples and could be moved axially along the group, being used as a check thermocouple. The electrical instruments and circuitry were the same as those used for the single capillary arrangement.

Furnaces.

Two new furnaces were constructed, one for the superheater and one for the viscometer, both of which were able to go to temperatures in excess of 1200°C. In both cases the furnaces were designed with reference to papers by Lambitz (164) (165), on the design of furnaces. Reference was also made to design data supplied by Morgan Refractories and Wiggin Electrical Resistance Materials.

Superheater Furnace

Overall size..............9.5"   9.5"   24"

Outer casing ....light alloy sheeting.

Furnace tube..............alumina 2" I/D and 22" long

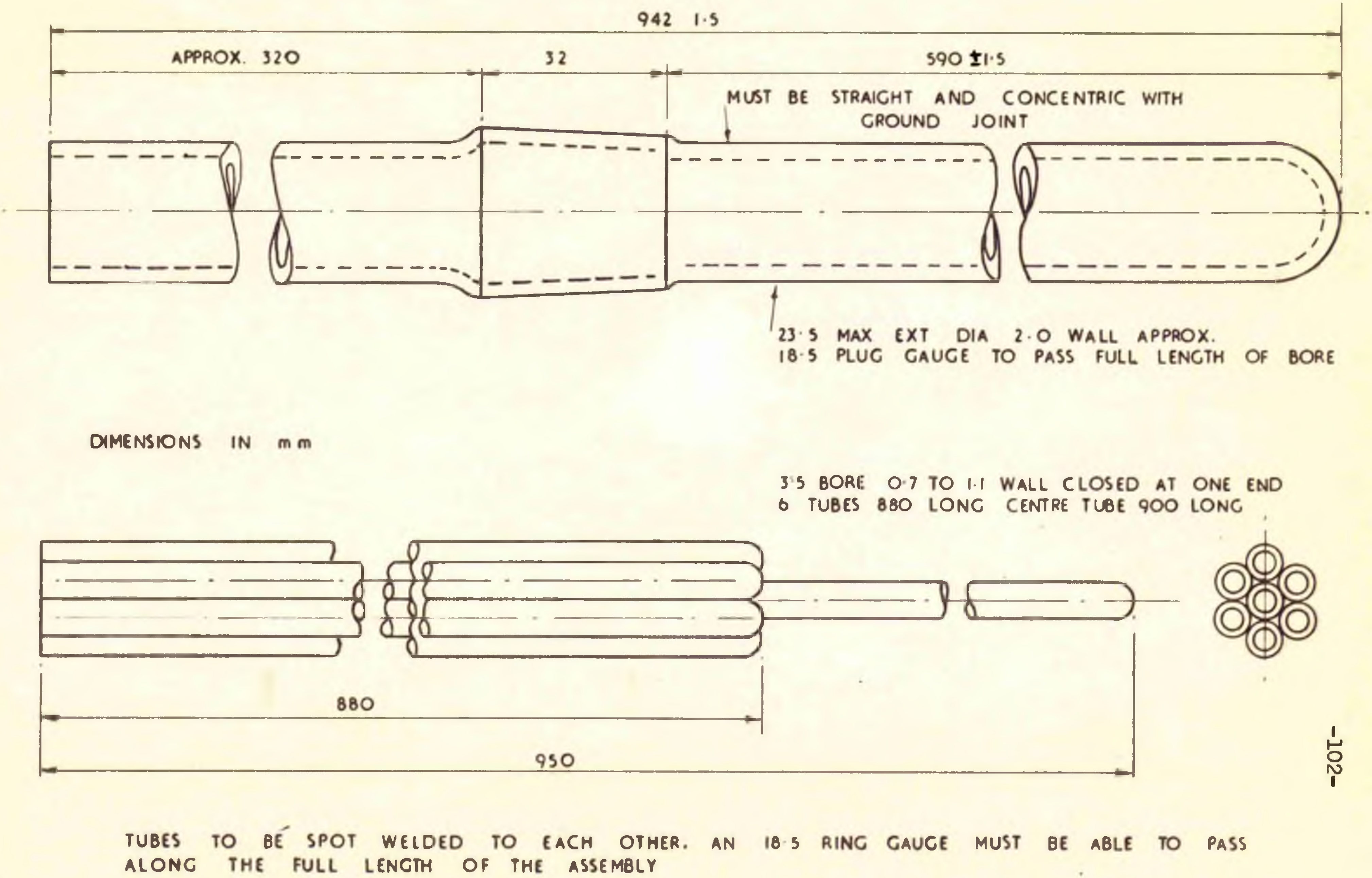


FIG 6. 17 THERMOCOUPLE TUBE

MATERIAL TRANSLUCENT SILICA

Superheater Furnace (contd)

Insulation............. 9"   9"   4.5" M.I.28 Morgan insulating bricks.

Electrical windings.... 1..32.3 ohm. resistance...18 s.w.g. Wiggin
Brightray H wire...wound at 10 t.p.i.

Main Furnace (see figure (6.18))

Overall size........... 9.5"   9.5"   49"

Ends................... Sindanyo

Furnace tube .... Dense alumina..4" bore by 0.5" thick by 44" long.

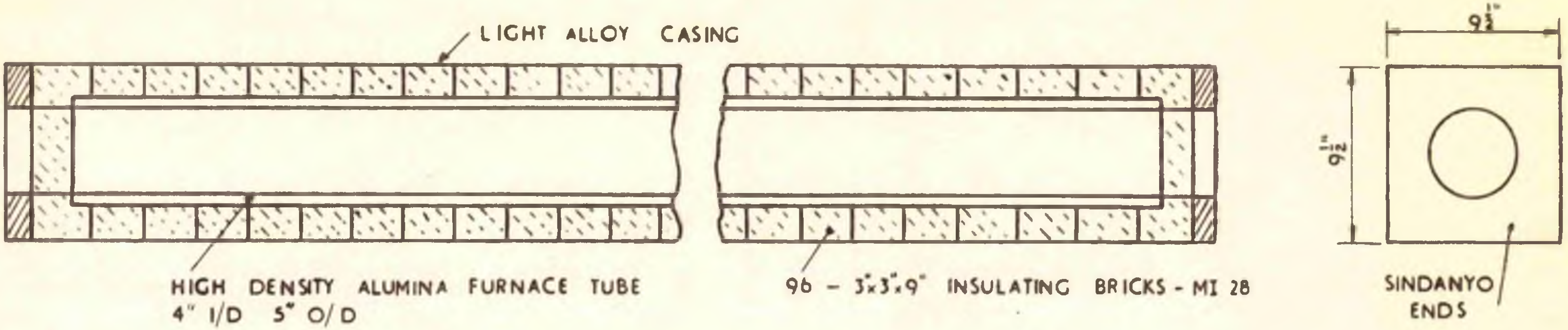
Insulation .... 9"   9"   4.5" Morgan M.I.28 insulating bricks.

Electrical windings.... one continuous winding tapped at three points to make four actual windings, the central windings being supplied from the same Variac, i.e. making them two windings in parallel. The end windings having resistances of 42.09 ohms and 39.65 ohms with a pitch of 10 t.p.i. The central windings having resistances of 48.64 ohms and 47.66 ohms with a pitch of 8 t.p.i. All the windings using 18 s.w.g. Wiggin Brightray H wire.

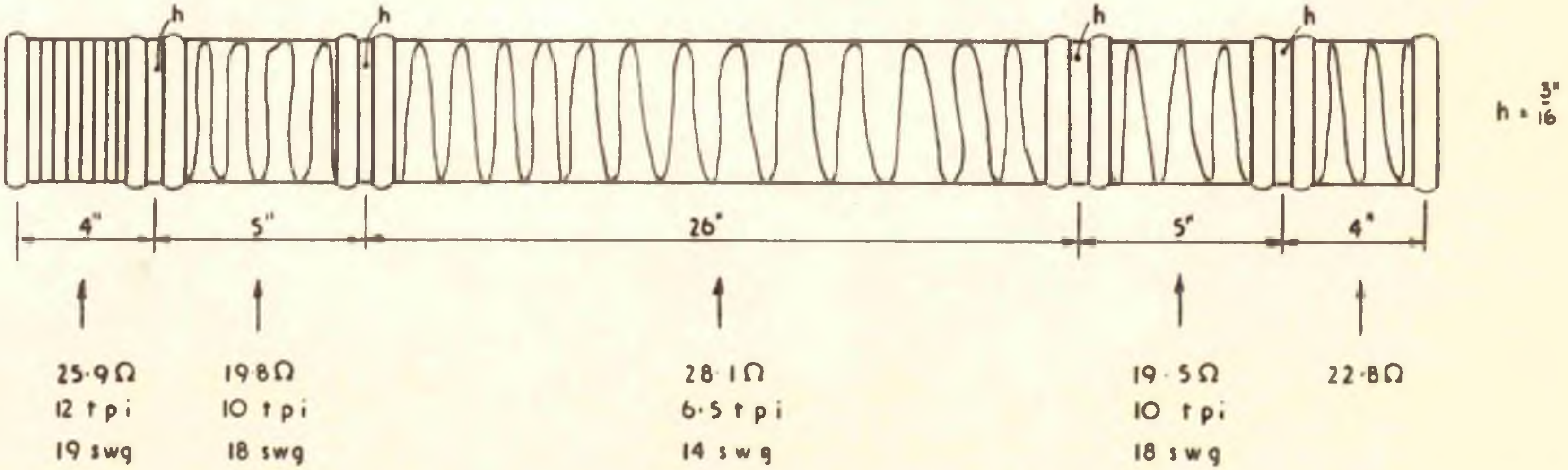
All the electrical power used was voltage stabilised and adjusted by Variacs.

The electrical windings were wound on to the furnace tube which had been previously coated with a layer of Fibrefrax paper, after which they were coated in a thick layer of Fibrefrax cement.

Initially, the main furnace had five windings, as shown in figure (6.18), as it was thought that five windings would be necessary to obtain a uniform temperature distribution over the central 400 mm. In actual fact, it was found that five variables were really too many to control, since there were a large number of power variations for



MAIN FURNACE ARRANGEMENT



AFTER INITIAL 'BURN OUT' GAP 'h' WAS INCREASED TO 3/8". THIS GAP WAS STILL INSUFFICIENT

FIG 6. 18 MAIN FURNACE WINDING FOR SERIES CAPILLARY SYSTEM the four end heaters, which could produce the same temperature distribution in the centre section of the furnace. However, after nearing the end of the first set of runs with a given set of capillaries, the end of the central winding and the end of the intermediate winding shorted together. The temperature of the furnace was about 920°C. when this failure occurred. Fortunately the electrical circuits were fully fused and little damage resulted. At first sight, it was thought that the end coil of one of the windings had slipped and shorted the windings together, as there was a deposit of metallic slag at the failure. The gaps between the windings were then cleaned up and the furnace once again heated up to 920°C. The same type of failure once again occurred. In an attempt to clear the matter up, the gap between the windings was increased, (see figure (6.18)); the initial gap was about 3/16", the final gap was about 3/8". The furnace was once again heated up, but failed at about 960°C this time. At this point it was obvious that this was not a simple shorting failure. The last failure caused considerable damage; one coil was burned out and there was actually a hole completely through the furnace tube, which was thought to have a melting temperature of about 1800°C. (It might be noted that the furnace tube had a wall thickness of 1/2").

It was finally assumed that the oxide film on either the stainless steel retaining clips or the actual windings had diffused into the alumina furnace tube, at the high temperatures being used. The resulting compound being a low melting point slag, which allowed the windings to short together with low potential differences being used. The maximum potential differences between the ends of the windings was about 60 volts, which is not excessively large.

If temperatures of up to at least 1060°C were going to be used, it was obvious that this winding arrangement would have to be scrapped. It was replaced by a single continuous winding (see figure (6.19)). Electrical tappings were made at three points along the winding and also at the extreme ends. The actual power supply being at the centre tapping and the extreme end tappings, the two intermediate tappings were neutrals. This in effect made three windings, the two centre ones being in parallel, and therefore reduced the number of variables for controlling the temperature distribution. The main point about this winding arrangement was that there would be no large voltage drops between any two adjacent coils, the maximum being about 5 volts.

Series Capillary Tube Viscometer

The sizes of the capillaries had first to be decided upon. Using the mass flow and development length curves, given in figures (6.1) and (6.2), it was decided to make the length ratio of the capillaries as 25 cm to 35 cm. The range of diameters that appeared to cover the temperature range best was 1.0 mm, 1.3 mm and 1.7 mm. In actual fact it was found that the length ratio, given above, tended to reduce the accuracy of the experimental work, see chapter (VII). The later work used diameters of 1.0 mm, 1.5 mm and 2.0 mm, with a length ratio of 25 cm to 37.5 cm.

The main dimensions of the viscometer are given on figures (6.20) to (6.24). The drawings and photographs (see pages 257 to 267) are self explanatory. For diagrams of the assembled apparatus

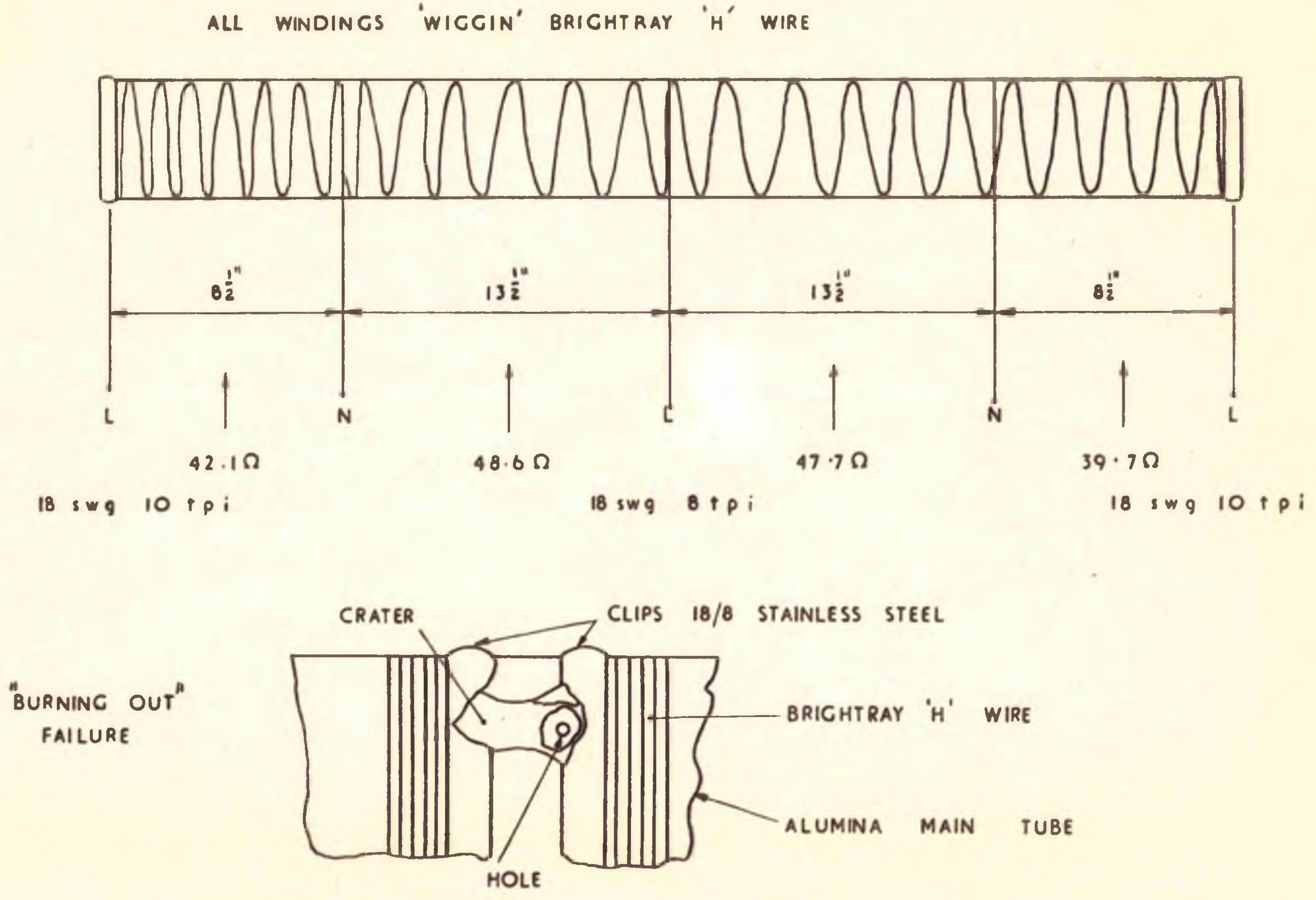


FIG 6. 19 REVISED FURNACE WINDINGS

see figure (6.25). The steam from the boiler is passed via the superheater and pressure stabiliser, (figure 6.5), to the actual viscometer; it then flows via the temperature stabilising coil, figure (6.23) (which has about 10 ft. of tubing in it), to the upstream plenum chamber, figure (6.23), after which it flows through the upstream capillary (which is the shorter capillary) to the intermediate pressure chamber (figure 6.21). The intermediate chamber is a closed annular ring, allowing the thermocouple sheath to pass through its centre (figure (6.21)). The steam then passes to the down-stream chamber and then, via a nozzle and a sintered filter, to the Liebig condenser (figure (6.10)), there being pressure tappings from the entrance plenum chamber, the intermediate and exit chambers. All the pressure lines pass out of one end of the furnace to the manometer. In each pressure line there are two standard spherical joints and one B.10. standard cone joint. Any tubing outside of the furnaces was heated with heating tapes which were supplied from a stabilised voltage source. After the viscometer cell and the superheater had been put into their respective furnaces the spaces at the ends of the furnaces were filled with quartz wool.

The superheater, stabilising coil, intermediate chamber, capillaries and any connecting tubing are made of clear quartz. The cylindrical body tube, end caps and the thermocouple sheaths are made of fused silica.

There are the obvious considerations that the thermocouples should be placed as near as possible to the capillaries and that the temperature distribution along the capillaries should be uniform, but the major part of the design was arranged in its present form after consultation with the manufacturers, Thermal Syndicate Co. Ltd., of Wallsend on Tyne, Northumberland.

For electrical circuit diagrams for the single capillary system and the series capillary system see figures (6.26) and (6.27) respectively.

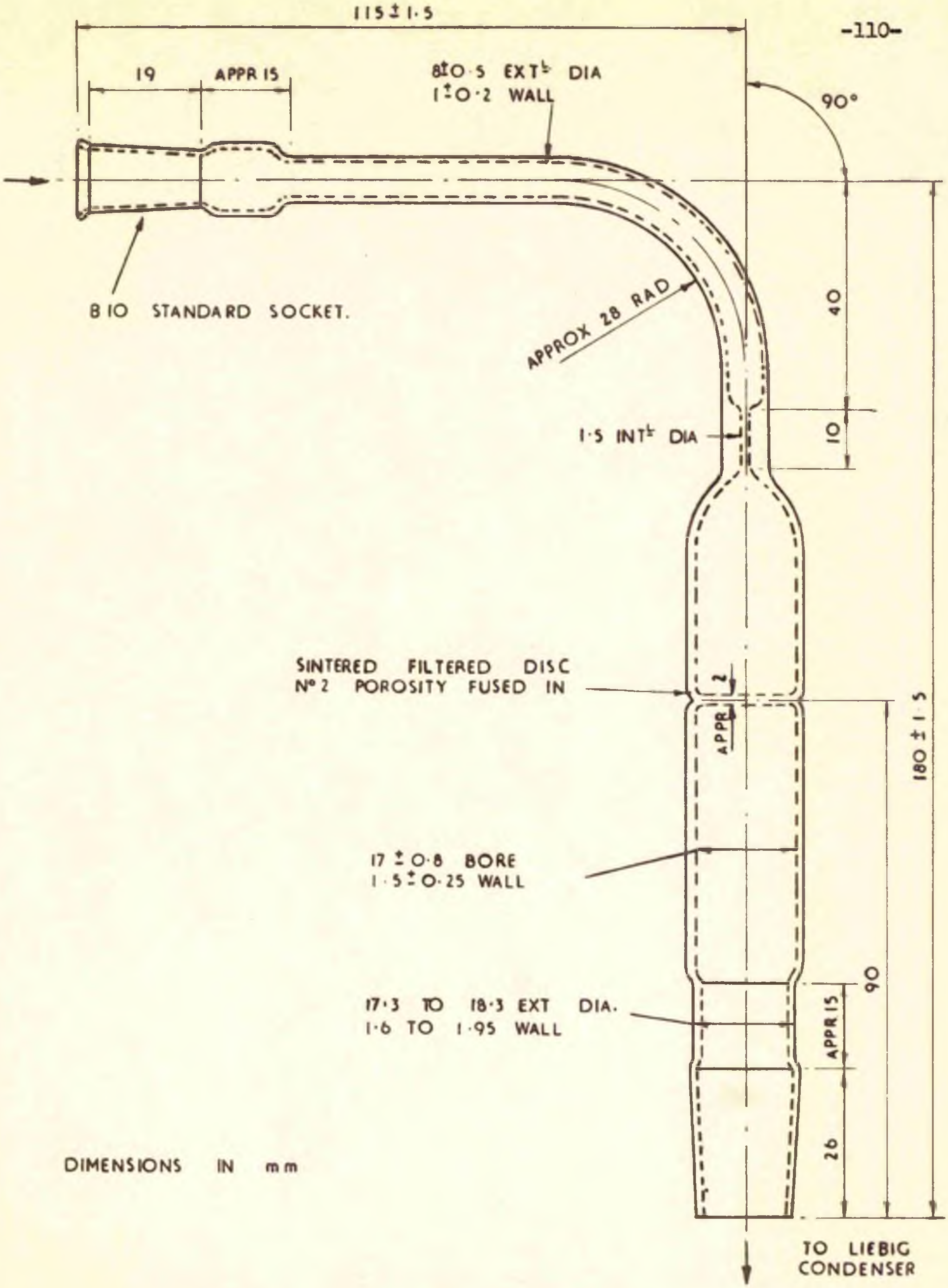


FIG 6. 20 EXIT ELBOW

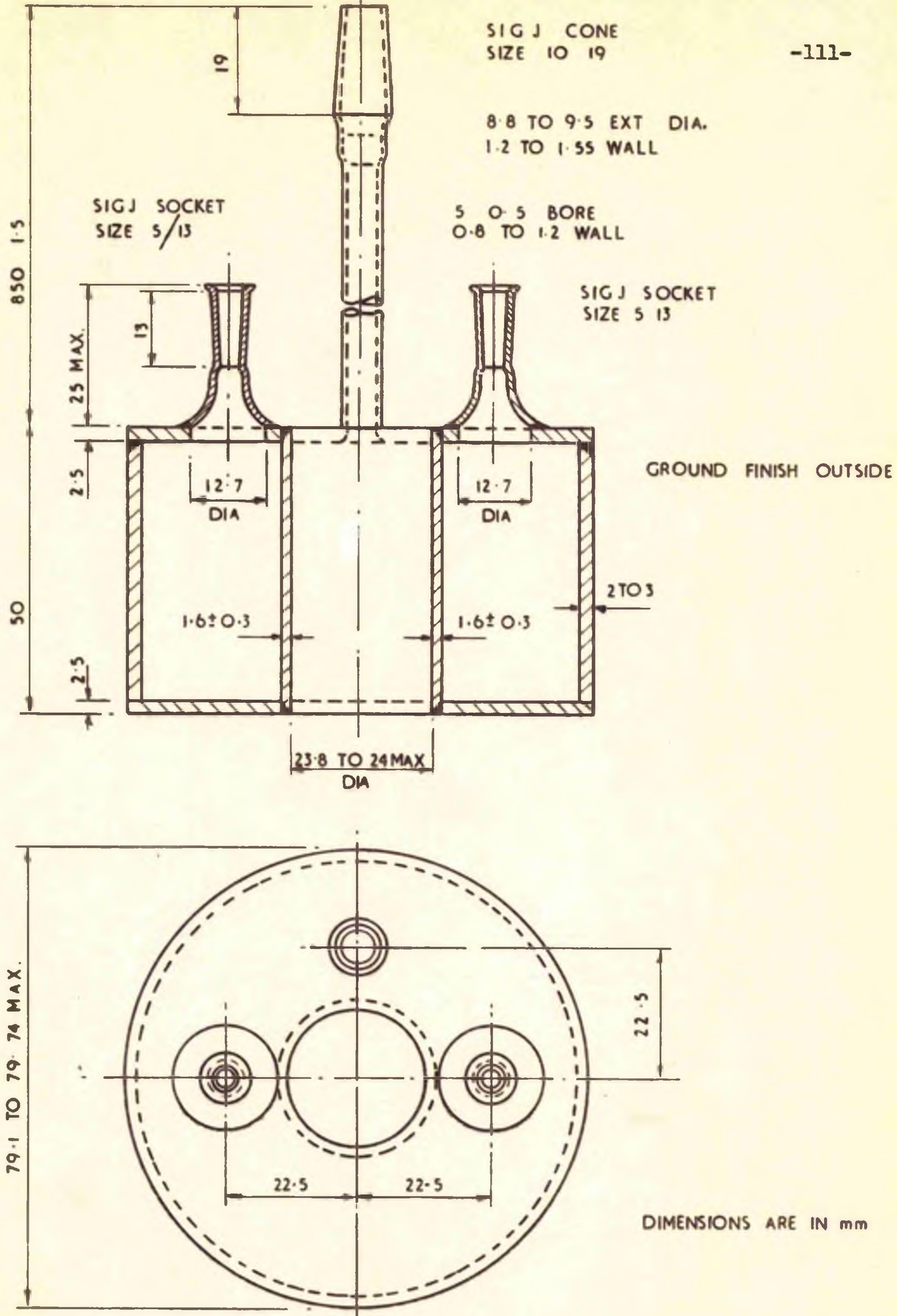
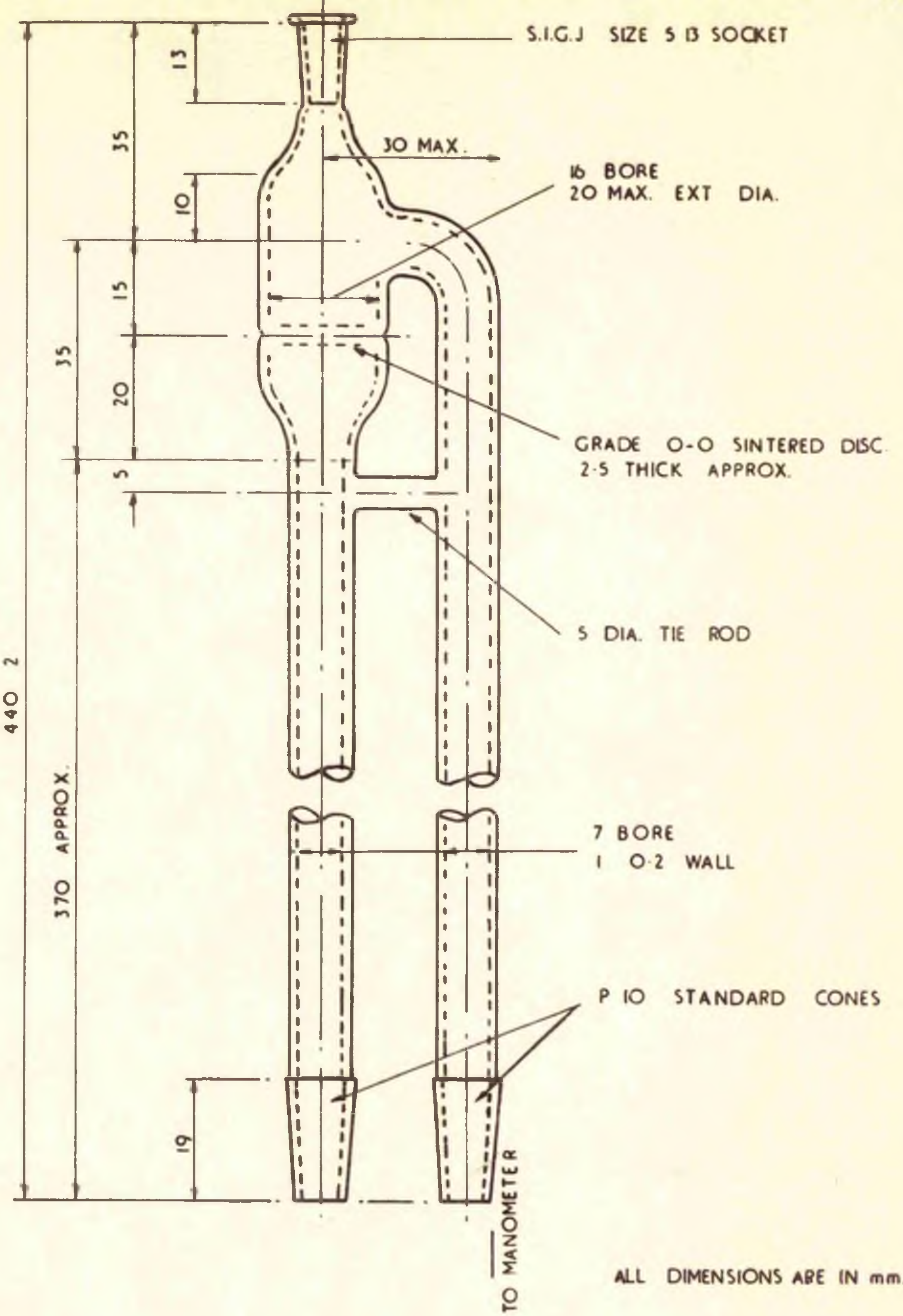


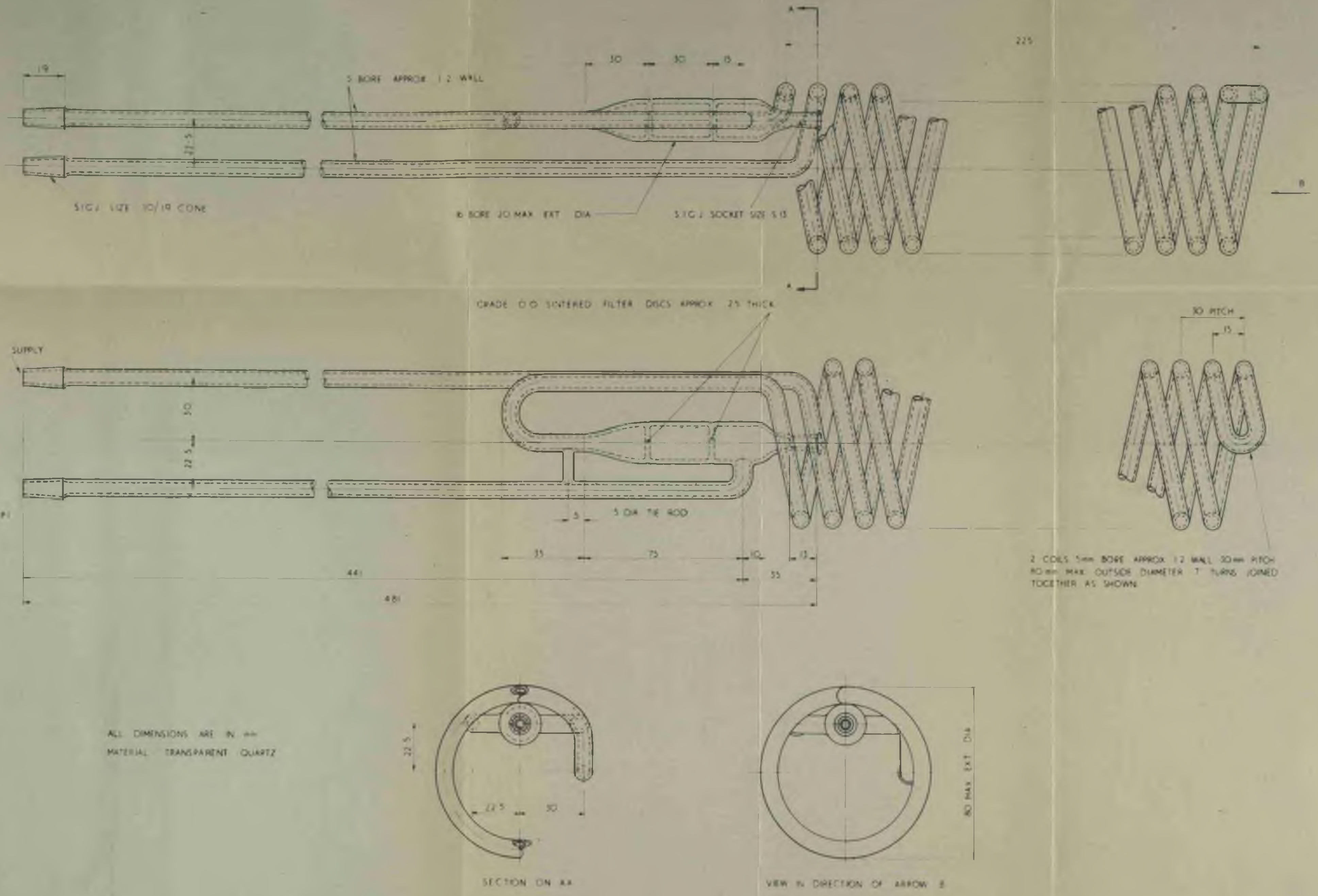
FIG 6.21 ANNULAR INTERMEDIATE CHAMBER



ALL DIMENSIONS ARE IN mm.

EXIT PLENUM CHAMBER FOR SERIES CAPILLARY SYSTEM

FIG 6. 22

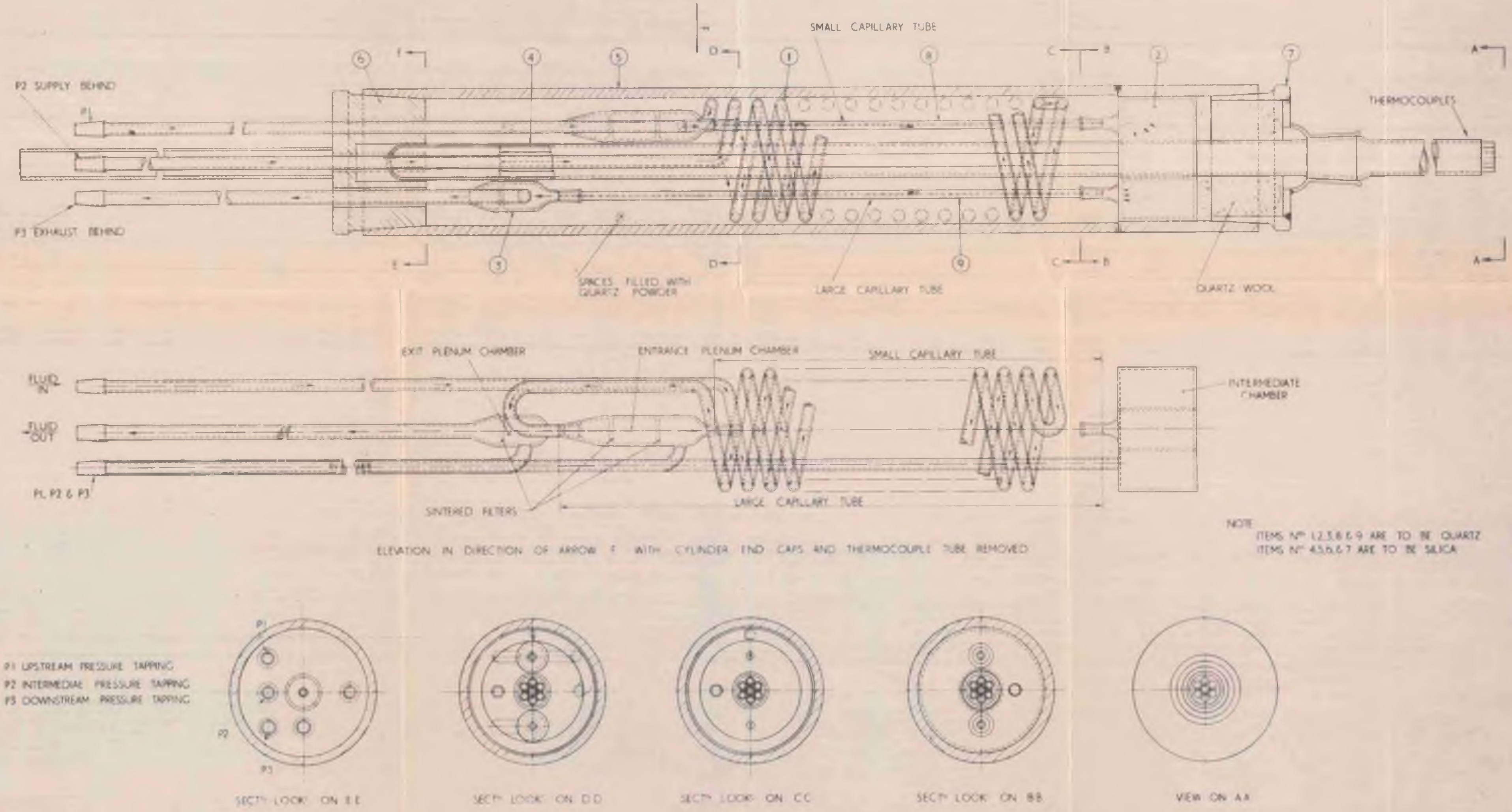


ALL DIMENSIONS ARE IN mm
MATERIAL TRANSPARENT QUARTZ

FIG. 6.23

SPECIAL COIL WITH FILTER TUBE AND BRANCH

DESIGNED BY



P1 UPSTREAM PRESSURE TAPPING
P2 INTERMEDIATE PRESSURE TAPPING
P3 DOWNSTREAM PRESSURE TAPPING

NOTE
ITEMS No 1,2,3,8 & 9 ARE TO BE QUARTZ
ITEMS No 4,5,6 & 7 ARE TO BE SILICA

VISCOMETER CELL ASSEMBLY

DESIGNED BY

FIG. 6.24

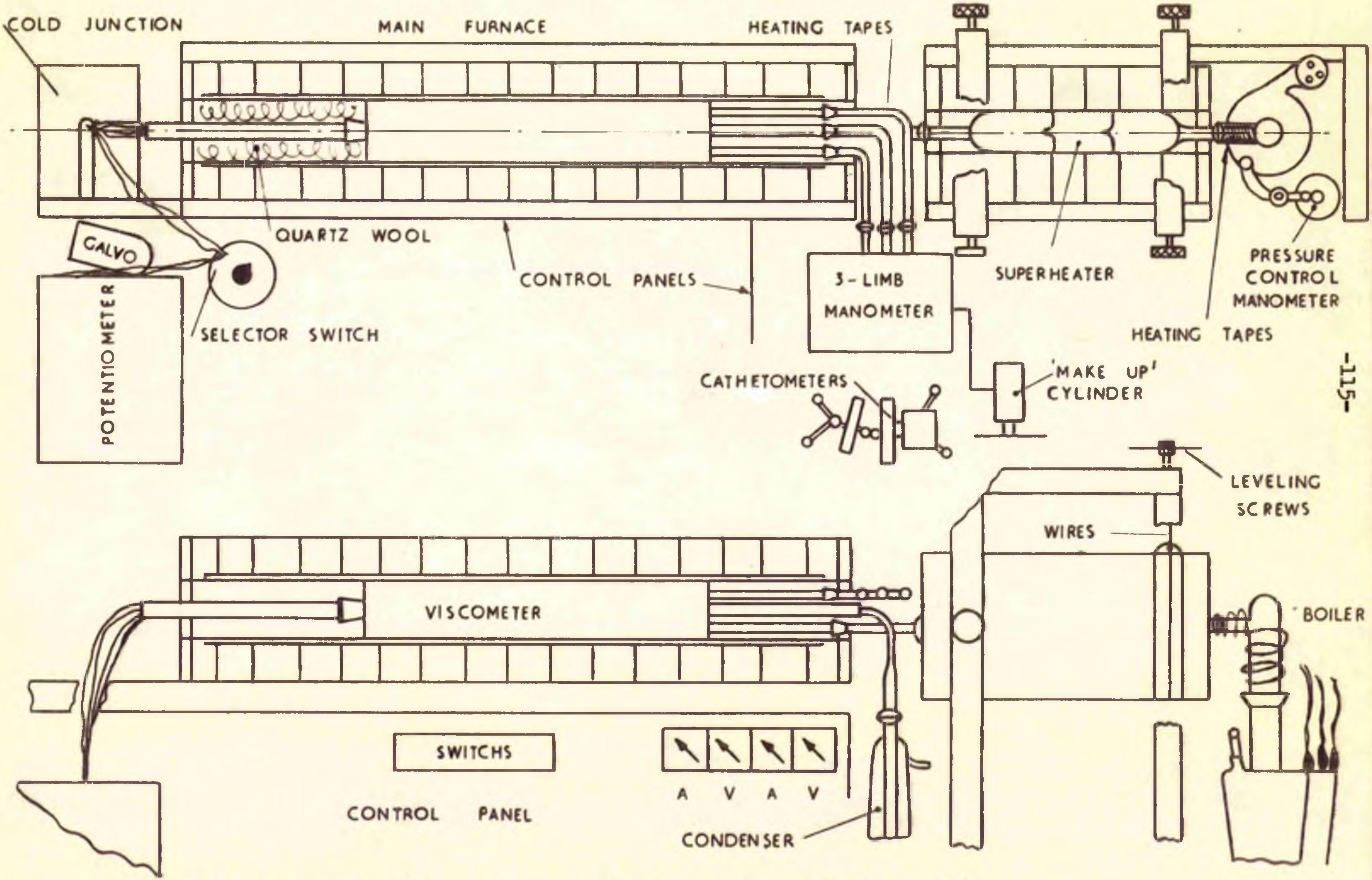


FIG 6.25 LAYOUT OF SERIES CAPILLARY VISCOMETER SYSTEM

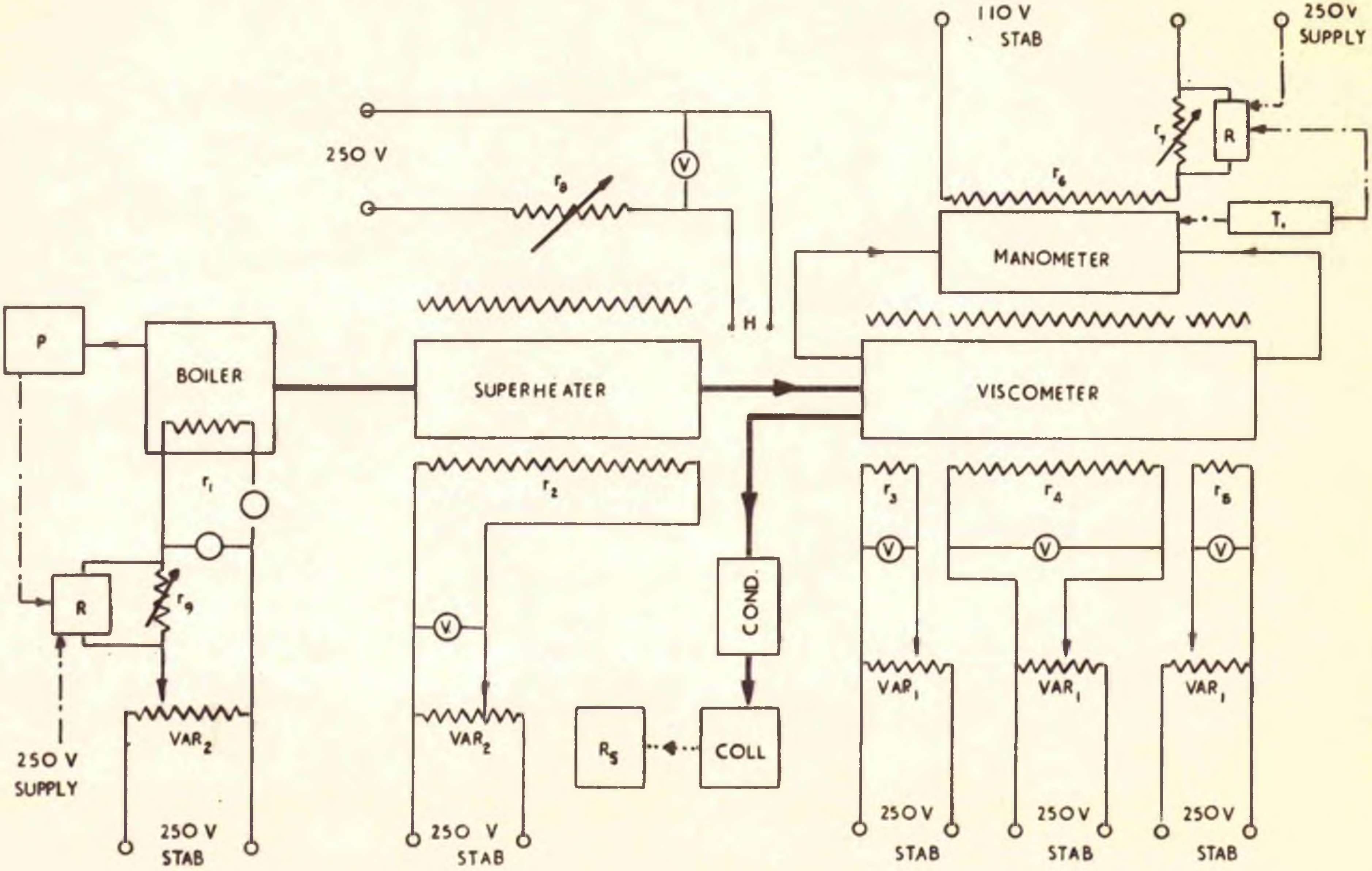


FIG 6.26 STEAM FLOW AND ELECTRICAL CONTROL CIRCUITS FOR SINGLE CAPILLARY SYSTEM

LEGEND FOR SINGLE CAPILLARY CIRCUIT DIAGRAM

r_1 = 80.4 ohms

r_2 = 61.3 "

r_3 = 105.0 "

r_4 = 150.4 "

r_5 = 108.5 "

r_6 = 10.0 "

r_7 = 0 - 20 ohms

r_8 = 0 - 230 ohms

r_9 = 0 - 20 ohms

A - Ammeter

V - Voltmeter

R - Electronic relays

R_s - Stopwatch relay

Var_1 - 2 amp variac

Var_2 - 5 amp variac

T - Contact thermometer

P - Contact manometer

H - Heating Tapes

\- Steam flow

\- Pressure tappings

\- Control circuits

\- Main electrical supply circuits

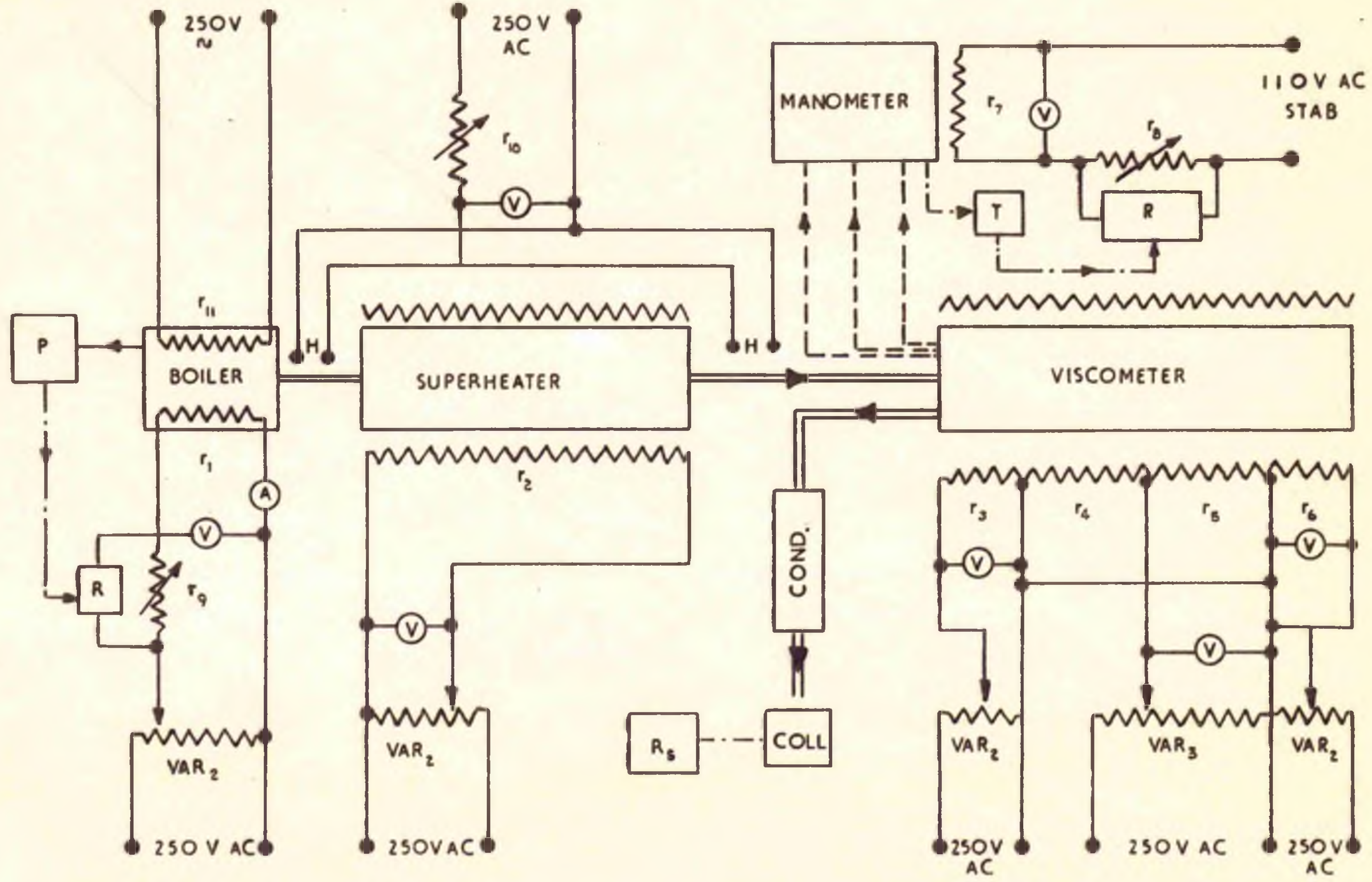


FIG 6. 27 STEAM FLOW AND ELECTRICAL CONTROL CIRCUITS FOR SERIES CAPILLARY SYSTEM

LEGEND FOR SERIES CAPILLARY CIRCUIT DIAGRAM

Resistances

r_1 = 51.6 ohms

r_2 = 52.3 ohms

r_3 = 42.1 ohms

r_4 = 48.6 ohms

r_5 = 47.7 ohms

r_6 = 39.7 ohms

r_7 = 10.0 ohms

r_8 = 0 - 20 ohms

r_9 = 0 - 20 ohms

r_{10} = 0 - 230 ohms

r_{11} = 30.0 ohms

A - Ammeters

V - Voltmeters

R - Electronic relays

R_s - Stopwatch relay

Var_1 - 2 amp variac

Var_2 - 5 amp variac

Var_3 - 15 amp variac

T - Contact thermometer

P - Contact manometer

H - Heating tapes

- Steam flow

- Pressure tappings

- Electrical control circuits

- Main electrical supply circuits

Procedure

The apparatus was initially cleaned; this was done by soaking the individual parts in chromic acid for about half an hour and then washing in distilled water, followed by rinsing in alcohol and then ether. The apparatus was then assembled, the joints being thoroughly flooded with alcohol to ensure that no foreign matter got into the joints. Rubber gloves were worn during assembly to ensure that the surface of the quartz was not contaminated, which could lead to devitrification of the quartz at high temperatures.

The main parts of the viscometer were assembled in a vertical position, that is, all the assembly of the parts was made with the intermediate chamber standing on its end. The capillaries, which were well lubricated with alcohol, were pushed tightly into their respective sockets in the intermediate chamber. The stabilising coil was then slid over the capillaries and the socket on the upstream plenum chamber pushed firmly on to its respective capillary. After which the socket on the downstream chamber was firmly fitted on to the other capillary. Whilst still in a vertical position, the assembled apparatus was lowered into the outer tube. The body was then filled with finely powdered quartz and with quartz wool at the extreme ends, and the end cap fitted. The cell was checked for leaks using a mercury pellet in a vertical drop tube. If the leak check proved successful, the cell was slid into the main furnace and the relevant parts connected up. All the joints were exterior to the furnaces, excepting the four cone joints on the capillaries; it was therefore possible to coat all the joints, excepting those on the capillaries, with Appezone silicon stopcock grease -

which is able to stand temperatures up to 200°C without any appreciable change in its fluidity. Having got thus far, the main leak tests were performed (see appendix (4)).

The furnace, which had been previously calibrated, was then set for a given temperature; usually the lowest intended temperature for a given set of capillaries. Between settings, the furnaces were given approximately 24 hours to settle down. The boiler was filled with demineralised water under a vacuum, the demineralised water having been stored under a vacuum.

The two boiler heaters were switched on and after boiling commenced, the starting heater was switched off and the boiler power adjusted to give a suitable boiler pressure. The boiler control manometer circuit was then switched on and took over the pressure control; coarse control of the power was made at frequent intervals until the system settled down. During the initial stages of boiling a vacuum was applied to the exit end of the system until steam issued into the condenser. This was continued for about ten minutes, in an attempt to remove all the air from the system. The system was actually run for at least four hours before any experimental runs were attempted, in order that the apparatus would be fully stabilised.

After the manometer box had heated up to about 120°C and the control circuit had taken over control of the temperature, the two outer columns of the manometer were zeroed with the manometer vented to the atmosphere. The manometer was then switched to the apparatus and the centre column adjusted using the "make up" cylinder.

The condensate was collected, in previously weighed test tubes, by inserting a test tube under the condenser directly after a water droplet had fallen; it was withdrawn, after a suitable period of time, directly after a droplet had fallen. This ensured that the collection time was the true time for a given mass flow.

The temperature along the axis of the viscometer was checked one or more times during a test run, depending on the time duration of a test. The pressure stability was also checked frequently during each experimental run. The barometric pressure was observed using a Negretti and Zambra direct zero barometer which had been calibrated by N.P.L.

The procedure for the single capillary apparatus was basically the same as that for the series capillary viscometer.

Chapter VII

Assessment of Errors

It is difficult to make an accurate estimation of the true absolute accuracy of the experimental results. The precision and accuracy are definitely allied, but to what extent it is difficult to assess. In the foregoing chapter the word accuracy is used to define the relationship of a value to that of a given absolute standard, whereas the precision is defined as the reproducibility of a given value. It therefore is obvious that one cannot have accuracy without precision, but one can have precision without accuracy. In this chapter both the precision and the accuracy of the experimental work will be estimated.

There are several factors which limit the accuracy of the results obtained for the viscosity of steam using the capillary method. They may be put into four groups, (1) the reliability of the Hagen-Poiseuille equation, (2) the accuracy of the actual measurements, (3) the experimental corrections and (4) the accuracy of subsidiary data required, i.e. properties of the materials used, be they fluid or solid. These four groups will not be discussed separately, but brought together for each assessment.

Commencing with the flow equation for a single capillary,

$$\eta = \frac{\pi d^4 g \Delta P \left[1 + 8 \varepsilon / d \right]}{128 Q I V (1 + 3 \Delta t \alpha)} - \frac{m Q}{8 \pi l (1 + \alpha \Delta t)} \qquad \ldots\ldots\ 7.1$$

and using the "most probable" method to ascertain the overall accuracy and precision of the experimental work, i.e. taking the square root of the sum of the squares of the largest errors in each variable separately the errors will be discussed.

The "most probable" method may be represented by the equation

$$d\eta = \sqrt{\sum\left(\frac{\partial(\eta)}{\partial(x)} \quad d(x)\right)^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 7.2$$

Partially differentiating equation 7.1, and putting the result into the form of equation 7.2, we get

$$d\eta = \sqrt{\begin{aligned}&\left(\frac{\pi 4d^3 g\Delta P\ (d)}{128\ l\ Q\ V}\right)^2 + \left(\frac{\pi g d^4\ d(P)}{128\ l\ Q\ V}\right)^2 + \left(\frac{\pi d^4 \Delta P\ d(g)}{128\ l\ Q\ V}\right)^2 + \left(\frac{\pi d^4 \Delta P g\ d(V)}{128\ l\ Q\ V^2}\right)^2 \\ &+\left(\frac{\pi g d^4 \Delta P}{128\ l\ Q\ V} - \frac{m\ Q}{8\pi\ l}\right)^2 \left(\frac{d(l)}{l}\right)^2 + \left(\frac{Q\ d(m)}{8\pi\ l}\right)^2 + \left(\frac{\pi g d^4 \Delta P}{128\ l\ Q\ V} - \frac{m\ Q}{8\pi\ l}\right)^2 \times \left(\frac{d(Q)}{Q}\right)^2 \\ &+\left(\frac{\pi d^4 g\Delta P}{128\ l\ Q\ V}\right)^2 \quad \left[\frac{8\ d\ (\varepsilon)}{d}\right]^2\end{aligned}} \quad , \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 7.3$$

neglecting the coefficient of linear expansion.

If the above equation is divided by η, an assessment of the accuracy can be made using the resulting equation. For the sake of simplicity, if the denominator is expressed as $\eta = \frac{\pi d^4 g\ \Delta P}{128\ l\ Q\ V}$, i.e. neglecting the end correction, the value of $d(\eta)/\eta$ may be expressed in the approximate form of,

$$d(\eta)/\eta = \sqrt{\begin{aligned}&16\ \left[d(d)/d\right]^2 + \left[d(\Delta P)/\Delta P\right]^2 + \left\{d(g)/g\right\}^2 + \left\{d(V)/V\right\}^2 \\ &\left(\frac{m\ Q}{8\pi\ l\ \eta}\right)^2 \times \left\{d(m)/m\right\}^2 + \left\{d(l)/l\right\}^2 + \left\{d(Q)/Q\right\}^2 + \\ &\left(\frac{8}{(\ d/\varepsilon\ + 8\ )}\right)^2 \times \left\{d(\varepsilon)/\varepsilon\right\}^2\end{aligned}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ 7.4$$

This equation, although only in an approximate form, is thought to be accurate enough for the assessment of the accuracy of the experimental work using the single capillary apparatus.

Using the same procedure for the series capillary system, that is, partially differentiating the flow equation,

$$\eta = \frac{\pi d_1^4 \, g \, (1 + 8 \, \varepsilon / d)}{128 \, (l_1 - l_2) \, Q} \left[\frac{\Delta P_1}{V_1} - (d_2/d_1)^4 \, \frac{\Delta P_2}{V_2} \right] (1 + 3 \alpha \Delta t) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 7.5$$

and making the same approximations, the approximate equation for the error may be expressed as,

$$\left\{ 1 - (l_2/l_1) \right\} \frac{d(\eta)}{\eta} = \sqrt{ \begin{aligned} & \left[d(\Delta P_1)/\Delta P_1 \right]^2 + \left[l_2/l_1 \right]^2 \left[d(\Delta P_2)/\Delta P_2 \right]^2 + \left[1 - \frac{l_2}{l_1} \right]^2 \times \left[d(Q)/Q \right]^2 \\ & + \left[4 d(d_1)/d_1 \right]^2 + \left[4 \, l_2/l_1 \right]^2 \times \left[d(d_2)/d_2 \right]^2 + \left[d(l_1)/l_1 \right]^2 + \\ & \left[l_2/l_1 \right]^2 \times \left[d(l_2/l_2) \right]^2 + \left[d(V_1)/V_1 \right]^2 + \left[l_2/l_1 \right]^2 \left[d(V_2)/V_2 \right]^2 \\ & + \left[1 + \frac{l_2}{l_1} \right]^2 \times \left[d(g)/g \right]^2 + \left[\frac{8 \, d(\varepsilon)/d}{(d/\varepsilon + 8)} \right]^2 \end{aligned} } \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 7.6$$

The following assessments of the individual errors will be used in conjunction with equations 7.4 and 7.6 to estimate the overall accuracy and precision.

An equation of the form,

$$d(\eta)/\eta = \sqrt{ \frac{ \sum \left[\dfrac{\eta_{ex} - \eta_{eq}}{\eta_{eq}} \right] }{n} } \quad \text{at temp. } t^{\circ}$$

will be used to estimate the precision of 'n' experimental results, where η_{eq} and η_{ex} are the viscosities obtained from the fitted curve and the actual experimental result respectively.

Discussion of the Individual Variables

(1) Diameters

The diameters of the capillaries were measured using a gravimetrical method. The accuracy is dependent on several related and unrelated factors. The equation used for obtaining the diameters was,

$$r = \sqrt{\left[\frac{W_m}{\rho_m \pi} + \lambda^3/3 \right] / (L + \lambda)} \; \ldots\ldots \; 7.7$$

where W_m is the weight of the pellet of mercury, L is the length of the pellet between the wetting surfaces, ρ_m is the density of the mercury and λ is the depth of the meniscus.
Since λ is small, λ^3 can be neglected for the purpose of assessment of errors.
Equation 7.7 then becomes,

$$r = \sqrt{\left\{ \frac{W}{\pi \rho L'} \right\}} \text{ , where } L' = (l + \lambda) \text{ and } \lambda = \frac{\lambda_1 + \lambda_2}{2}$$

Hence
$$d(r)/r = \frac{1}{2} \sqrt{[d(W)/W]^2 + [d(\rho)/\rho]^2 + [d(L)/L']^2 + [d(\lambda)/L']^2} \; \ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 7.8$$

The mercury used for the volume measurement was triple distilled and had been stored in a vacuum prior to its use. The values for the density of the mercury were taken from Smithsonian tables, the accuracy of which is thought to be better than 1 in 20000.

The mass measurements were made on a Stanton Unimatic single pan balance having a sensitivity 0.1 mg per vernier division precision - standard deviation of 0.025 mg, accuracy of weights to N.P.L. tolerances (1961), expected errors less than 1 in 200000.

The length measurements were made with a Pye precision cathetometer, having a vernier calibrated to 0.05 mm, with a reproducibility of 0.05 mm. A Swift microscope head, having double hair line was fitted to the cathetometer.

Based on the above data, the accuracy of the measurement of the diameters was about ± 0.0125 %. In the majority of cases the diameter reproducibility was found to be within this tolerance (see appendix (5)).

Length of Capillary

The Pye cathetometer was also used to measure the lengths of the capillaries. The assessed accuracy of the measurements being about ± 0.01 %.

Pressure Drop

The differential pressure measurements depend upon two main factors, they are the height and the density of a mercury column, i.e. $\Delta P = h\,\rho$ and $\rho = \phi(t)$.

The error can be expressed by the equation,

$$d\Delta P/\Delta P = \sqrt{\left[\frac{d(h)}{h}\right]^2 + \left[\frac{d(\rho)}{\rho}\right]^2} \qquad \ldots\ldots\ldots\ldots\ldots\ 7.9$$

The density of the mercury used in the manometer is known to be better than 1 in 10000, over the temperatures used. The density of the mercury obviously depended upon the temperature of the manometer. The temperature varied by about ± 1.5°C, but the accuracy of an individual reading was 0.1°C. The actual pressure drops were measured with a Swift Utilex precision cathetometer, fitted with a Moore and Wright micrometer head (having a sensitivity of 0.004 mm and a range of 25 mm) and a vernier (graduated to read 0.01 mm). The sighting reproducibility of this system is given in appendix (7).

From the above observation it was assessed that the absolute accuracy of the individual differential pressure measurements was 0.1 % for ΔP_2 and 0.05 % for ΔP_1. The expected precision is thought to be 0.27 % to 0.14 % for ΔP_1 and 0.25 % to 0.12 % for ΔP_2, depending on the actual pressure drop being measured.

Mass Flow Rate 'Q'

The measurement of the mass flow rate is obviously dependent upon two main factors, the mass of water collected and the time to collect it. That is, $Q = W/t$ from which we can get

$$d(Q)/Q = \sqrt{\left\{ d(W)/W \right\}^2 + \left\{ d(t)/t \right\}^2} \quad \ldots\ldots\ldots\ldots 7.10$$

The weighing was done on the Stanton Unimatic single pan balance, about which data is given in this chapter under the heading of diameters.

The measurement of the time to collect the condensate was given by a relay and stopwatch manufactured by Messrs. Camerer Cuss (for calibration see appendix (6)), the assessed accuracy for which is better than 1 in 6000. This gives an assessed accuracy for the mass flow rate of better than 0.02 %. This accuracy does not take into account two other factors which are not readily assessable, (1) the uncertainty of collecting droplets of water and (2) the leak at the relevant joints. (1) The experimental scatter obtained when collecting water droplets is of major importance and although attempts were made to even out any scatter in the observed values of the mass flow rates for given conditions, the reprodusibility was not better than ± 0.5 %. It is thought that the timing procedure cannot be blamed for this effect, but that the variation in the size of the droplets and vibrations (causing the droplets to fall before they would normally be expected to do so) were more likely to have been the cause of this experimental scatter. On account of this scatter in the value of the mass flow rate a minimum of five sets of readings were taken at a given temperature. This uncertainty will not be taken into account when assessing the overall accuracy, but it will be used when assessing the overall precision.

(2) Leak tests were carried out on the assembled apparatus, just after assembly and just before dismantling (see appendix (4)). From these tests it can be seen that even when using a pressure in excess of those to be used in the apparatus, the leak should not be greater than 0.1 %.

It is therefore expected that the measurement of the mass flow rate should be better than 0.11 % accurate (i.e. ± 0.055 %).

Temperature Measurement

The temperature inside the viscometer was measured using six platinum-platinum 10 % rhodium thermocouples, all being made from wire from the same reels. One of these couples was calibrated against a N.P.L. thermocouple of the same materials. The uncertainty of the value of the temperature is estimated as ± 0.25°C up to 500°C and ± 0.75°C between 500°C and 1000°C. This uncertainty of the value of the temperature could affect the overall accuracy of the viscosity measurement. If the equation for the viscosity of steam is assumed to be of the form $\eta = a + bt$, where t is the temperature in °C and a and b are constants, then the uncertainty of η can be expressed as

$$d(\eta)/\eta = \frac{0.407\ d(t)}{80.4 + 0.407\ t}\ \%,$$

where the values of a and b are those given by Shifrin (28)

At 500°C, which will be the worst case, the uncertainty in η using the above equation is ± 0.108 %.

Specific Volume of the Steam

The values for the specific volumes of the steam were taken from the V.D.I. Steam Tables 5th edition (1960) (166). It is difficult to assess the accuracy of these values; however, it is thought that they may be better than 0.1 %.

As stated previously, the accuracy of the temperature measurement was approximately ± 0.25° C up to 500°C and ± 0.75°C between 500°C and 1000°C. This uncertainty in the value of the temperature will affect the specific volume of the steam. Making the approximate assumption that for steam $PV = zRT$, then an approximate equation for the effect of the uncertainty in temperature can be expressed in the form,

$$d(V)/V = \sqrt{\left\{ d(P)/P \right\}^2 + \left\{ d(T)/T \right\}^2 + \left\{ d(V')/V' \right\}^2} \quad \ldots\ldots 7.12$$

where $d(V')/V'$ is the uncertainty in the table values,

where P is dependent on the atmospheric pressure and the pressure drop across the capillary.

The atmospheric pressure was measured by a Negretti and Zambra mercury barometer, accuracy ± 0.15 mm Hg., sensitivity of 0.05 mm Hg., calibrated by N.P.L. As stated previously, the accuracy of the pressure drop across the capillaries is 0.27 % in the worst case. Therefore the accuracy of the specific volume values is assessed to be better than ± 0.097 %.

Acceleration due to Gravity g

The value for 'g' at the University of Glasgow taken from a semi-empirical equation is 981.563 cm/sec^2. It is thought that the accuracy of this value is better than 1 in 10000.

Slip

As stated in chapter (V), the actual value of the slip coefficient is uncertain. However, the largest value for the slip correction is of the order of 0.5 % of the viscosity and therefore even an uncertainty of 20 % in the value of the slip correction would mean an uncertainty of only 0.1 % in the value of the viscosity.

Linear Expansion of the Capillary Tubes

The effect of the linear expansion of the capillary tubes in both systems, has been allowed for and amounts to a maximum of about 0.16 % for the quartz tubes and to a maximum of about 0.44 % for the glass tubes. Therefore an uncertainty of 10 % in the value of the coefficient of linear expansion α would only amount to an uncertainty in the value of η of 0.016 % for the quartz tubes and 0.044 % for the glass tubes. The effect of the uncertainty in the value of α may therefore be neglected.

End Correction Constant 'm'

The series capillary system removes the need for a knowledge of an accurate value of the end correction constant 'm'. This is a correct statement, if and only if, the end corrections for both of the capillaries are the same. It is difficult to obtain an accurate value for 'm' (see chapter (V) for a discussion on 'm'). Unfortunately in the work with the glass apparatus, the mass flow at a given temperature was not varied in a methodical manner and it was not possible to use the graphical method to find the viscosity, consequently a value for 'm' was assumed. The uncertainty of 'm' is thought to be about

± 4 %, which would result in an uncertainty of ± 0.4 % in the value of η, in the worst possible case.

The mass flow through the series capillary system was not varied for the first three capillary sizes used, consequently the end correction constants in each capillary could not be compared for equality. However, the mass flow was varied in the majority of the latter series capillary runs. It can be seen from the results and discussion in appendix (11) that the experimental scatter precluded any definite conclusions regarding the value of 'm' and its comparability in the two capillaries. On account of this it was decided that no correction or estimation regarding the uncertainty of the equality of the end corrections for the series system could be made. An estimation of the effect of the uncertainty of the equality of 'm' can be made from the basic flow equations for the two capillaries,

$$\begin{aligned} \eta &= \eta_1 - \left[mQ \right] / (8\pi\, l_1) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ 7.13 \\ \eta &= \eta_2 - \left[mQ \right] / (8\pi\, l_2) \end{aligned}$$

where the suffices 1 and 2 refer to the capillaries (1) and (2) and η_1 and η_2 are the Poiseuille viscosities.

Differentiating the equations for 'm' and re-arranging, we get

$$d(\eta)/\eta = \frac{Q}{16\,\pi\,\eta} \left| \frac{d(m_1)}{l_1} + \frac{d(m_2)}{l_2} \right| \quad \ldots\ldots\ldots\ldots\ 7.14$$

Assuming that the uncertainty in the values of m_1 and m_2 from a given value are the same, i.e. $d(m_1) = d(m_2) = d(m)$, then

$$d(\eta)/\eta = \frac{Q\ d(m)}{16\,\pi\,\eta} \left| \frac{l_1 + l_2}{l_1\ l_2} \right| \quad \ldots\ldots\ldots\ldots\ldots\ldots\ 7.15$$

Making an approximate assumption that the overall uncertainty between the values of 'm' is 4 %, i.e. d(m) equals 2 %, then the worst possible effect that this would have on η would be when Q equalled 0.024 g/sec and η equalled 2.02 10^{-4} g/cm sec, and l_1 and l_2 were approximately 35 mm and 25 mm respectively, making this uncertainty amount to about ± 0.15 %. This uncertainty has not been used when assessing the accuracy of the experimental work since it is only an estimate.

A table giving the assessed accuracies of the results obtained from each assembly, and the estimated overall precision is given on the following page.

Estimation of the Overall Accuracy and Precision of the Experimental Results

Glass Apparatus

Capillary No.	Accuracy
1.	± 0.57 %
2.	± 0.61 %

Quartz Apparatus

Capillary No.	Accuracy
3 a and 4 b	± 0.74 %
2 a and 2 b	± 0.64 %
8 a and 8 b	± 0.64 %
B a and B b	± 0.58 %
A a and A b	± 0.57 %
C a and C b	± 0.57 %

Since, from initial tests, it was obvious that the reproducibility of the mass flow rate could not be better than ± 0.5 %, it was expected that the overall precision or scatter (not accuracy) of the experimental results, should be of the order of ± 1.0 to ± 1.5 %

Chapter Vlll

Results, Discussion of Results and Conclusions

The results listed below have been divided into the following sections,

Results obtained using the single capillary apparatus

Table (1) - Groups 1 to X11 - results obtained using a capillary having a nominal bore of 1 mm.

Table (2) - Groups X111 to XV111 - results obtained using a capillary having a nominal bore of 1.5 mm.

Results obtained using the series capillary quartz apparatus

Table (3) - Groups (1) to (18) - results obtained using capillaries having nominal bores of 1 mm. and a nominal length ratio of 35 : 25 .

Table (4) - Groups (19) to (30) - results obtained using capillaries having nominal bores of 1.3 mm. and a nominal length ratio 35 : 25 .

Table (5) - Groups (31) to (44) - results obtained using capillaries having nominal bores of 1.7 mm. and a nominal length ratio of 35 : 25 .

Table (6) - Groups (45) to (46) - results obtained using capillaries having nominal bores of 1.5 mm. and a nominal length ratio of 37.5 : 23 .

Table (7) - Groups (47) to (58) - results obtained using capillaries having nominal bores of 1 mm. and a nominal length ratio of 37.5 : 23 .

Table (8) - Groups (59) to (65) - results obtained using capillaries having nominal bores of 2.0 mm. and a nominal length ratio of 37.5 : 23 .

Table (1)

Reynolds No. = 1.2744 Q/η (mean value for the tube)

Group	No.	t°C.	Q x10^2 g/sec.	ΔP gf/cm^2	V cm^3/g	η x10^4 poise.
1	1	112.8	0.88434	26.212	1697.7	1.2769
	2	113.1	0.88431	26.231	1699.1	1.2768
	3	113.7	0.87475	26.221	1701.9	1.2907
	4	113.9	0.87076	25.926	1703.1	1.2808
	5	113.8	0.87031	26.020	1702.6	1.2871
11	6	139.3	0.60200	20.038	1817.6	1.3885
	7	139.6	0.60438	20.136	1818.9	1.3885
	8	139.7	0.60687	20.366	1819.1	1.3988
	9	139.9	0.60853	20.122	1817.2	1.3782
	10	139.9	0.60693	19.970	1817.3	1.3711
	11	140.0	0.60602	20.058	1817.7	1.3796
111	12	155.4	0.58126	21.287	1937.4	1.4395
	13	155.6	0.58172	21.371	1938.3	1.4436
	14	155.9	0.58903	21.371	1939.6	1.4275
	15	156.1	0.57358	21.113	1941.1	1.4455
	16	156.3	0.57979	21.252	1941.8	1.4376
	17	156.4	0.57404	21.059	1942.5	1.4392
1V	18	207.0	0.51635	23.538	2103.1	1.6741
	19	207.4	0.51460	23.285	2105.1	1.6598
	20	207.8	0.51337	23.132	2107.0	1.6512
	21	208.0	0.51207	23.137	2108.5	1.6549
	22	208.2	0.51009	23.137	2109.5	1.6611
	23	208.4	0.50891	23.072	2110.4	1.6597
V	24	250.2	0.40740	22.294	2371.3	1.8043
	25	250.3	0.39971	22.117	2371.8	1.8245
	26	250.5	0.39633	21.965	2373.0	1.8286
	27	250.7	0.39501	21.948	2370.8	1.8354
	28	251.2	0.39345	21.913	2373.1	1.8385
	29	251.5	0.39270	21.872	2373.0	1.8385

continued /.

Table (1) cont.

Reynolds No. = 1.2744 Q/η (mean value for the tube)

Group	No.	t°C.	Q x10^2 g/sec	ΔP gf/cm^2	V cm^3/g	η x10^4 poise
V1	30	256.1	0.42387	23.770	2338.6	1.8750
	31	256.1	0.42532	23.768	2338.6	1.8681
	32	256.2	0.42505	23.861	2339.8	1.8759
	33	256.3	0.42433	23.517	2340.4	1.8508
	34	256.3	0.42384	23.535	2340.4	1.8538
	35	256.3	0.42264	23.758	2340.1	1.8786
V11	36	303.1	0.33141	22.210	2547.0	2.0784
	37	303.3	0.32893	22.029	2548.1	2.0765
	38	303.8	0.32740	21.943	2550.5	2.0763
	39	304.2	0.32529	21.870	2552.3	2.0817
	40	304.3	0.32370	21.763	2552.1	2.0821
	41	305.4	0.32378	21.709	2557.2	2.0721
V111	42	342.9	0.32389	25.340	2788.0	2.2219
	43	343.1	0.32307	25.249	2789.0	2.2189
	44	343.2	0.32119	25.161	2791.1	2.2227
	45	343.5	0.31997	24.990	2792.7	2.2148
	46	343.7	0.32026	25.029	2793.7	2.2155
	47	343.8	0.31987	24.977	2795.7	2.2119
	48	344.2	0.30390	23.929	2798.9	2.2307
1X	49	357.8	0.28155	22.612	2777.3	2.2978
	50	357.5	0.27816	22.460	2776.3	2.3118
	51	357.1	0.27531	22.164	2774.9	2.3065
	52	356.8	0.27474	21.966	2773.8	2.2913
	53	356.5	0.26944	21.810	2772.7	2.3221
	54	356.2	0.26952	21.626	2771.6	2.3024
	55	356.0	0.26749	21.540	2770.8	2.3118
	56	356.0	0.26974	21.579	2770.7	2.2938
X	57	400.5	0.23505	21.968	2981.1	2.5027
	58	400.3	0.23616	21.962	2981.2	2.4900
	59	400.2	0.23666	21.962	2980.8	2.4849
	60	399.9	0.23509	21.906	2979.5	2.4966
	61	399.6	0.23548	21.837	2978.3	2.4854
	62	398.8	0.23318	21.575	2975.0	2.4828
X1	63	441.6	0.19929	21.068	3179.9	2.6627
	64	441.4	0.19821	21.139	3178.9	2.6875
	65	441.2	0.19732	20.969	3178.2	2.6785
	66	441.1	0.19640	20.914	3177.9	2.6844
	67	440.9	0.19677	20.895	3177.0	2.6776

Table (1) cont.

Reynolds No. = 1.2744 Q/η (mean value for the tube)

Group	No.	t°C.	Q x10^2 g/sec	ΔP gf/cm^2	V cm^3/g	η x10^4 poise
X1 cont.	68	440.9	0.19741	20.820	3177.1	2.6590
	69	440.9	0.19876	21.092	3176.7	2.6758
X11	70	448.8	0.26283	29.394	3312.9	2.6950
	71	448.9	0.26317	29.414	3313.2	2.6931
	72	449.1	0.26311	29.416	3315.7	2.6918
	73	449.3	0.26297	29.389	3316.6	2.6899
	74	449.4	0.26310	29.387	3320.1	2.6856
	75	449.6	0.26189	29.287	3322.4	2.6871

Table (2)

Reynolds No. = 0.8432 Q/η (mean value for the tube)

Group	No.	t°C.	Q x10^2 g/sec	ΔP gf/cm^2	V cm^3/g	η x10^4 poise
X111	76	173.0	0.11907	9.837	2021.9	1.5190
	77	173.2	0.11976	9.920	2022.7	1.5218
	78	173.3	0.12115	10.045	2023.0	1.5211
	79	173.4	0.12065	10.035	2023.5	1.5267
	80	173.6	0.12089	10.035	2024.5	1.5222
X1V	81	228.6	0.11634	12.152	2262.1	1.7444
	82	228.9	0.11636	12.150	2263.4	1.7427
	83	229.0	0.11541	12.150	2263.9	1.7594
	84	229.0	0.11550	12.154	2263.6	1.7585
	85	229.3	0.11539	12.136	2264.4	1.7581
XV	86	274.7	0.11186	14.058	2457.6	1.9581
	87	274.8	0.11307	14.058	2458.0	1.9331
	88	275.0	0.11286	14.108	2459.9	1.9431
	89	275.1	0.11337	14.187	2461.5	1.9433
	90	275.1	0.10989	13.649	2461.3	1.9330

Continued

Table (2) cont.

Reynolds No. = 0.8432 Q/η (mean value for the tube)

Group	No.	t°C.	Q x10^2 g/sec	ΔP gf/cm^2	V cm^3/g	η x10^4 poise
XV1	91	345.3	0.10779	17.064	2762.5	2.2218
	92	346.1	0.10848	17.207	2767.3	2.2312
	93	346.2	0.10745	17.210	2768.0	2.2453
	94	346.4	0.10738	17.160	2769.0	2.2393
	95	346.7	0.10715	17.187	2770.3	2.2474
	96	346.8	0.10804	17.255	2770.6	2.2353
XV11	97	406.4	0.10678	20.901	3032.8	2.5253
	98	406.6	0.10645	20.818	3033.8	2.5226
	99	406.8	0.10603	20.770	3035.0	2.5265
	100	406.9	0.10616	20.806	3036.5	2.5263
	101	407.0	0.10551	20.679	3037.1	2.5268
	102	407.1	0.10519	20.627	3037.7	2.5282
	103	407.1	0.10496	20.599	3037.7	2.5307
XV111	104	454.3	0.10589	23.792	3229.8	2.7370
	105	454.4	0.10585	23.786	3230.3	2.7368
	106	454.5	0.10561	23.718	3230.9	2.7352
	107	454.5	0.10579	23.693	3230.9	2.7268
	108	454.7	0.10595	23.560	3233.2	2.7039
	109	455.1	0.10560	23.534	3235.0	2.7092

Table (3)

Reynolds No. = 1.2215 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
1	110	126.9	8.5653	26.401	21.121	1802.1	1761.6	1.3027
	111	127.1	8.5818	26.042	20.735	1803.4	1763.4	1.3009
	112	127.3	8.3693	25.416	20.176	1805.9	1766.9	1.3131
	113	127.6	8.1893	25.256	20.196	1806.5	1767.3	1.3048
	114	128.1	8.2585	25.366	20.215	1811.0	1771.9	1.3097
2	115	178.0	7.7731	30.557	24.049	2024.7	1944.6	1.4739
	116	177.9	7.7903	30.556	23.928	2024.2	1944.3	1.4929

Cont/

Table (3) cont.

Reynolds No. = 1.2215 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
2 Cont.	117	178.0	7.7809	30.609	24.074	2024.6	1944.4	1.4774
	118	178.0	7.7265	30.141	23.634	2026.9	1948.1	1.4798
	119	178.0	7.7198	30.340	23.966	2026.6	1947.0	1.4574
3	120	268.4	5.1224	28.850	22.446	2432.1	2372.1	1.8971
	121	269.2	5.1255	28.862	22.379	2435.7	2376.4	1.9113
	122	269.6	5.1371	28.862	22.418	2437.5	2378.1	1.8963
	123	272.7	5.0445	28.818	22.362	2451.2	2391.5	1.9227
	124	273.1	5.0344	28.738	22.309	2453.1	2393.5	1.9180
4	125	330.2	3.9693	27.665	21.435	2711.2	2649.0	2.1383
	126	330.1	3.8928	27.464	21.380	2710.9	2648.1	2.1365
	127	330.1	3.9815	27.474	21.271	2711.1	2648.4	2.1200
	128	330.2	3.8738	27.401	21.298	2711.7	2649.1	2.1509
	129	330.3	3.8616	27.348	21.215	2712.2	2649.7	2.1650
	130	330.6	3.9265	27.340	21.217	2713.6	2651.1	2.1247
	131	330.7	3.8958	27.303	21.087	2714.6	2652.3	2.1652
5	132	367.7	3.7082	29.238	22.514	2863.1	2792.2	2.3163
	133	367.9	3.7519	29.531	22.607	2863.7	2792.2	2.3451
	134	367.1	3.7777	29.557	22.584	2860.1	2788.7	2.3456
	135	367.2	3.7610	29.536	22.588	2860.1	2788.8	2.3491
	136	367.2	3.7802	29.341	22.551	2860.4	2789.3	2.2929
	137	367.2	3.7201	29.290	22.633	2860.4	2789.4	2.2940
	138	367.7	3.6877	29.170	22.580	2862.5	2791.6	2.2933
6	139	411.8	3.2915	29.734	22.816	3045.3	2968.4	2.5150
	140	410.9	3.2547	29.549	22.671	3041.6	2965.2	2.5326
	141	410.9	3.2047	29.286	22.646	3042.4	2966.5	2.4987
	142	4Lo.8	3.1860	29.048	22.448	3042.3	2967.0	2.4985
	143	410.9	3.2501	29.289	22.449	3042.7	2967.1	2.5208
	144	410.4	3.2504	29.452	22.612	3039.8	2963.8	2.5254
7	145	473.8	2.5779	28.077	21.547	3328.4	3249.1	2.7850
	146	474.6	2.5622	28.066	21.615	3332.0	3252.6	2.7727
	147	475.6	2.5721	28.107	21.656	3336.5	3256.8	2.7590
	148	476.2	2.5392	28.122	21.670	3334.7	3254.9	2.7964
	149	476.7	2.5790	28.110	21.671	3336.9	3257.1	2.7412
	150	477.0	2.5398	28.125	21.699	3339.2	3259.3	2.7835
	151	477.0	2.5467	27.952	21.579	3339.2	3259.7	2.7555

Cont./

Table (3) cont.

Reynolds No. = 1.2215 Q/η (mean value for the two tubes)

Group	No.	$t^{\circ}C$	$Q \times 10^3$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	$\eta \times 10^4$ poise
8	152	515.1	2.5188	30.794	23.528	3541.8	3450.3	2.9557
	153	515.1	2.5878	31.036	23.743	3541.3	3449.0	2.8915
	154	515.0	2.5210	30.649	23.529	3541.5	3450.2	2.9049
	155	515.1	2.4742	30.395	23.448	3540.2	3449.5	2.9006
	156	515.9	2.4802	30.135	22.948	3544.2	3454.7	2.9646
	157	516.4	2.4523	30.218	23.136	3545.1	3455.0	2.9331
9	158	600.0	1.9004	28.507	21.747	3925.8	3831.4	3.3017
	159	600.2	1.8807	28.458	21.710	3926.5	3832.6	3.3309
	160	601.0	1.8613	28.556	21.849	3929.9	3835.5	3.3483
	161	601.5	1.8853	28.625	21.891	3935.2	3840.5	3.3132
	162	601.5	1.8972	28.512	21.750	3936.3	3842.2	3.3000
	163	601.6	1.8668	28.341	21.818	3946.6	3842.6	3.2292
10	164	633.0	1.8672	30.676	23.303	4128.5	4023.7	3.4630
	165	633.7	1.8563	30.786	23.439	4131.5	4026.2	3.4732
	166	634.3	1.8697	30.240	22.986	4135.3	4031.9	3.4017
	167	634.9	1.8141	30.190	23.042	4137.8	4034.2	3.4630
	168	634.9	1.8449	30.369	23.059	4137.4	4033.5	3.4686
	169	635.0	1.8339	30.514	23.275	4137.5	4032.9	3.4657
11	170	680.9	1.8815	34.062	26.002	4318.1	4195.8	3.5788
	171	681.3	1.8320	34.056	26.177	4319.9	4197.2	3.6087
	172	681.8	1.8437	33.8576	26.032	4320.6	4198.4	3.5632
	173	682.7	1.8119	33.858	25.979	4324.6	4202.5	3.6418
	174	683.5	1.8389	33.752	25.953	4326.2	4204.0	3.5565
12	175	719.9	1.6858	34.182	26.092	4500.4	4372.4	3.8463
	176	720.6	1.7386	34.048	25.932	4503.9	4376.4	3.7341
	177	721.5	1.7124	34.004	25.809	4508.1	4380.9	3.8164
	178	721.8	1.7097	33.615	25.484	4510.2	4384.4	3.7906
	179	722.4	1.7147	33.581	25.588	4513.1	4387.0	3.7263
	180	722.1	1.6761	33.612	25.615	4511.6	4385.5	3.8152
	181	722.5	1.6973	33.758	25.738	4513.6	4386.9	3.7771
13	182	766.0	1.8243	39.181	29.449	4697.8	4545.7	4.0158
	183	768.0	1.8010	39.273	29.821	4706.6	4553.2	3.9666
	184	770.0	1.8280	39.277	29.544	4716.7	4562.8	3.9914
	185	772.9	1.8206	39.609	29.837	4728.1	4573.2	4.0166

Cont/

Table (3) cont.

Reynolds No. = 1.2215 Q/η (mean value for the two tubes)

Group	No.	t°C	Q $\times10^3$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	$\eta \times10^4$ poise
13 cont.	186	773.7	1.8006	39.652	30.038	4726.7	4571.2	4.0089
	187	775.0	1.8142	39.283	29.509	4733.4	4579.7	4.0227
	188	776.2	1.7643	39.177	29.669	4738.5	4584.5	4.0397
14	189	825.2	1.8708	43.602	32.685	4921.7	4742.9	4.1466
	190	825.6	1.8086	43.162	32.657	4924.6	4746.7	4.1496
	191	826.1	1.7885	43.148	32.644	4931.3	4753.3	4.1906
	192	827.1	1.8028	43.070	32.539	4938.6	4761.0	4.1594
	193	827.6	1.7705	42.978	32.552	4944.7	4767.2	4.1948
	194	828.2	1.8138	43.209	32.463	4946.9	4769.0	4.1976
15	195	875.7	1.7184	45.505	34.474	5224.4	5028.3	4.3117
	196	876.2	1.7272	45.425	34.328	5226.9	5031.3	4.3085
	197	876.4	1.6842	45.198	34.261	5228.3	5033.4	4.3631
	198	877.2	1.6831	45.234	34.258	5225.5	5030.5	4.3808
	199	878.0	1.7073	45.127	34.231	5229.5	5034.6	4.2890
	200	878.2	1.6526	45.019	34.230	5227.6	5033.0	4.3956
	201	878.5	1.7178	44.933	33.797	5227.1	5033.7	4.3403
16	202	908.1	1.6112	45.659	34.457	5354.2	5151.8	4.5443
	203	908.7	1.6102	45.076	33.980	5358.4	5158.6	4.5057
	204	909.3	1.5827	45.117	34.008	5361.0	5161.0	4.5849
	205	909.9	1.6109	45.172	33.942	5358.3	5158.3	4.5473
	206	910.0	1.5993	45.146	34.008	5358.9	5158.6	4.5485
	207	909.7	1.6430	44.869	33.758	5358.2	5159.3	4.4170
17	208	234.8	7.6334	37.457	29.005	2244.6	2172.4	1.7750
	209	234.7	7.7085	37.457	29.005	2244.2	2172.0	1.7580
	210	234.9	7.6629	37.442	29.017	2245.1	2172.9	1.7632
	211	234.7	7.5969	37.361	28.949	2246.2	2174.1	1.7751
	212	234.4	7.6958	37.281	29.029	2244.9	2172.9	1.7265
	213	234.2	7.6566	37.361	29.122	2243.9	2171.8	1.7343
	214	234.0	7.5706	37.042	28.909	2244.0	2172.4	1.7344
18	215	569.1	3.1266	43.451	32.725	3731.4	3595.5	3.2177
	216	567.4	3.1391	43.823	33.150	3725.7	3588.5	3.1990
	217	570.0	3.1499	43.914	33.295	3739.5	3601.3	3.1633
	218	570.8	3.1049	43.769	33.336	3743.3	3605.0	3.1588
	219	570.9	3.1363	43.917	33.337	3733.9	3597.0	3.1749
	220	570.8	3.1914	43.597	33.684	3732.2	3594.0	3.2068

Cont./

Table (4)

Reynolds No. = 0.9666 Q/η (the mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
19	221	201.5	18.931	35.187	26.786	2111.3	2048.5	1.6127
	222	201.6	18.653	35.214	26.787	2111.7	2048.9	1.6413
	223	201.6	18.726	35.240	26.759	2111.7	2048.8	1.6447
	224	201.7	18.696	35.173	26.839	2112.2	2049.3	1.6199
	225	201.8	18.628	35.133	26.799	2112.9	2050.1	1.6254
	226	201.9	18.659	35.160	26.786	2113.4	2050.6	1.6298
	227	201.9	18.637	35.133	26.812	2114.1	2051.2	1.6213
20	228	418.4	12.395	48.834	35.950	3083.3	2958.1	2.4983
	229	418.3	12.413	48.817	35.934	3082.8	2957.7	2.4950
	230	418.2	12.402	48.817	35.894	3082.4	2957.4	2.5052
	231	418.1	12.580	48.819	35.962	3081.9	2956.8	2.4574
	232	418.1	12.414	48.965	35.948	3083.7	2958.4	2.5192
	233	418.1	12.431	48.832	35.869	3083.9	2958.8	2.5062
	234	417.9	12.379	48.803	35.880	3083.1	2958.1	2.5095
21	235	453.4	11.847	51.252	37.211	3265.6	3127.8	2.6771
	236	453.4	11.837	51.385	37.278	3265.4	3127.3	2.6911
	237	453.3	11.896	51.439	37.211	3264.8	3126.8	2.7007
	238	453.3	11.968	51.545	37.278	3264.7	3126.4	2.6916
	239	453.3	11.898	51.612	37.238	3263.3	3125.0	2.7284
	240	453.3	12.040	51.703	37.290	3263.2	3124.7	2.7034
	241	453.3	11.993	51.701	37.275	3263.5	3125.0	2.7161
22	242	553.1	8.4230	48.687	35.501	3703.9	3555.1	3.1374
	243	553.2	8.6231	48.621	35.368	3704.5	3556.1	3.0802
	244	553.3	8.7040	48.623	35.370	3704.9	3556.5	3.0515
	245	553.4	8.6090	48.684	35.322	3705.2	3556.8	3.1096
	246	553.4	8.7550	48.684	35.228	3705.6	3557.0	3.0781
	247	553.4	8.6854	48.655	35.320	3705.4	3557.0	3.1366
23	248	651.7	7.2498	51.608	37.311	4147.7	3971.8	3.5082
	249	651.8	7.3995	51.755	37.299	4148.2	3971.6	3.5424
	250	651.8	7.2737	51.688	37.285	4148.0	3971.9	3.5917
	251	651.9	7.2808	51.701	37.325	4148.4	3972.3	3.5120
	252	652.1	7.4330	51.980	37.365	4149.0	3972.1	3.4951
	253	652.1	7.3770	51.887	37.351	4149.2	3972.5	3.5027
	254	652.2	7.4398	51.806	37.337	4149.8	3973.3	3.4572

Cont./

Table (4) cont.

Reynolds No. = 0.9666 Q/η (mean value for the two tubes)

Group	No.	$t^{\circ}C$	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
24	255	709.5	6.5638	51.519	37.100	4421.8	4235.4	3.6697
	256	709.2	6.5326	51.346	36.993	4420.8	4233.0	3.6724
	257	708.9	6.4309	51.116	36.951	4419.9	4234.8	3.6840
	258	708.8	6.4270	51.377	36.973	4417.2	4231.4	3.7486
	259	708.8	6.5423	51.404	37.014	4417.1	4231.3	3.6791
	260	708.8	6.4975	51.333	36.953	4417.2	4231.7	3.7021
25	261	761.1	5.9060	51.366	37.015	4650.4	4454.8	3.9153
	262	761.4	5.8971	51.669	37.279	4651.5	4455.1	3.8736
	263	761.6	5.8781	51.550	37.014	4652.7	4457.1	3.9285
	264	761.8	5.8459	51.636	36.998	4654.9	4459.1	3.9750
	265	762.0	5.8279	51.583	36.953	4656.0	4460.4	3.9850
	266	762.1	5.8361	51.449	36.975	4656.7	4461.3	3.9369
	267	762.1	5.8742	51.098	36.835	4657.5	4463.2	3.8567
26	268	790.0	4.9084	45.363	32.693	4767.0	4589.8	4.0548
	269	790.2	4.8561	45.267	32.664	4768.1	4591.1	4.0768
	270	790.5	4.9163	45.252	32.637	4769.5	4592.6	4.0299
	271	790.8	4.8560	45.318	32.596	4768.6	4591.6	4,1140
	272	791.0	4.9588	45.241	32.452	4769.6	4593.1	4.0495
	273	791.4	4.8254	44.969	32.328	4772.1	4596.3	4,1138
27	274	842.2	4.9226	49.262	35.457	5025.0	4823.3	4.1477
	275	841.7	4.9145	49.220	35.430	5022.9	4821.5	4.1526
	276	841.7	4.9099	49.977	35.907	5021.0	4816.7	4.2350
	277	842.2	4.8966	49.482	35.679	5024.4	4821.7	4.1676
	278	842.6	4.8983	49.438	35.676	5026.3	4823.6	4.1525
	279	843.0	4.9039	49.5210	35.665	5030.3	4827.4	4.1725
	280	843.0	4.8309	49.387	35.597	5030.6	4828.1	4.2158
	281	842.9	4.8106	49.320	35.491	5030.3	4828.3	4.2468
	282	842.8	4.9145	49.200	35.397	5030.2	4828.7	4.1502
28	283	899.1	4.6415	50.798	36.316	5249.4	5030.4	4.3394
	284	899.1	4.6189	50.864	36.320	5249.2	5030.1	4.3795
	285	899.2	4.5914	50.844	36.515	5249.7	5030.6	4.4008
	286	899.5	4.5916	50.969	36.572	5250.8	5031.1	4.4192
	287	899.5	4.5279	50.492	36.267	5254.2	5036.5	4.5173
	288	899.5	4.5956	50.654	36.255	5253.8	5035.7	4.4169
29	289	922.9	4.5112	52.490	37.459	5358.5	5128.6	4.5895
	290	923.3	4.5882	52.771	37.779	5359.6	5128.1	4.4963
	291	923.7	4.5405	52.690	37.911	5361.6	5129.9	4.4769
	292	923.5	4.5578	52.647	37.630	5360.9	5130.0	4.5343

Table (4) cont.

Reynolds No. = 0.9666 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
29 cont.	293	923.7	4.5537	52.619	37.536	5361.8	5131.2	4.5583
	294	924.1	4.5072	52.577	37.614	5358.5	5127.7	4.5706
	295	924.2	4.5298	52.550	37.653	5359.0	5128.3	4.5272
	296	924.3	4.5298	52.481	37.572	5359.6	5129.2	4.5467
30	297	941.1	4.4894	51.449	36.880	5364.2	5145.2	4.4899
	298	941.0	4.3508	51.380	36.879	5364.2	5145.1	4.6115
	299	941.0	4.3586	51.182	36.707	5364.7	5146.4	4.5962
	300	941.0	4.4442	51.182	36.720	5365.5	5145.3	4.5048
	301	941.2	4.3563	51.114	36.692	5364.4	5146.6	4.5829
	302	941.4	4.2701	51.017	36.663	5365.7	5147.8	4.6528
	303	941.5	4.3848	51.070	36.769	5366.1	5147.9	4.5134
	304	941.7	4.4483	51.278	36.806	5365.8	5147.2	4.5014

Table (5)

Reynolds No. = 0.7443 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
31	305	301.7	2.4092	25.580	18.047	2596.0	2542.3	2.0185
	306	301.7	2.4104	25.609	18.036	2596.0	2542.2	2.0302
	307	301.6	2.4160	25.654	17.933	2595.4	2541.7	2.0738
	308	301.5	2.4295	25.802	17.933	2594.9	2541.0	2.1075
	309	301.4	2.4308	25.828	18.164	2596.3	2539.1	2.0307
	310	301.4	2.4332	25.908	18.177	2593.2	2538.9	2.0580
	311	301.3	2.4444	25.926	18.246	2592.7	2538.3	2.0315
32	312	392.3	2.4413	34.343	23.814	2969.6	2885.6	2.4057
	313	392.4	2.4414	34.276	23.827	2971.6	2889.0	2.3840
	314	392.3	2.4494	34.448	23.879	2970.9	2888.1	2.4070
	315	392.4	2.4814	34.914	24.039	2970.7	2886.9	2.4519
	316	392.5	2.4780	34.969	24.240	2971.1	2887.0	2.4121
	317	392.4	2.4838	34.849	24.226	2970.8	2886.9	2.3795
	318	392.5	2.4753	34.916	24.213	2971.2	2887.1	2.4121

Table (5) cont.

Reynolds No. = 0.7443 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
33	319	490.4	2.3393	43.398	29.544	3433.3	3314.4	2.8342
	320	490.5	2.3372	43.468	29.613	3433.7	3314.5	2.8350
	321	490.4	2.3400	43.467	29.599	3433.2	3314.1	2.8355
	322	490.4	2.3471	43.448	29.631	3433.3	3314.1	2.8140
	323	490.4	2.3351	43.449	29.618	3433.3	3314.1	2.8321
	324	490.4	2.3358	43.411	29.540	3431.8	3312.8	2.8439
	325	490.4	2.3439	43.481	29.542	3431.7	3312.6	2.8502
34	326	600.0	2.2121	53.284	35.714	3906.6	3740.1	3.2996
	327	600.1	2.2130	53.270	35.726	3907.0	3740.5	3.2915
	328	600.0	2.2131	53.216	35.819	3907.1	3740.6	3.2564
	329	600.0	2.2116	53.225	35.736	3907.1	3740.7	3,2810
	330	600.0	2.2068	53.221	35.782	3907.1	3740.7	3.2760
	331	600.0	2.2010	53.020	35.675	3910.2	3744.3	3.2645
	332	600.1	2.1956	52.880	35.683	3910.5	3744.8	3.2391
35	333	698.4	1.8694	54.596	36.446	4321.4	4135.9	3.6534
	334	697.9	1.8706	54.599	36.448	4318.9	4133.8	3.6539
	335	697.8	1.8592	54.451	36.380	4318.8	4133.0	3.6566
	336	697.8	1.8624	54.465	36.328	4318.8	4134.1	3.6701
	337	697.8	1.8624	54.359	36.315	4321.0	4136.2	3.6456
	338	698.0	1.8643	54.503	36.220	4321.6	4136.7	3.7009
	339	698.1	1.8687	54.357	36.233	4322.3	4137.6	3.6532
36	340	802.6	1.4627	52.061	34.565	4838.4	4639.2	4.0531
	341	802.9	1.4641	52.071	34.776	4839.7	4639.9	3.9872
	342	803.1	1.4620	52.112	34.777	4840.5	4640.5	4.0031
	343	803.1	1.4641	52.057	34.523	4840.7	4641.4	4.0389
	344	803.9	1.4625	52.085	34.537	4843.3	4643.9	4.0645
	345	803.9	1.4639	52.107	34.561	4843.3	4643.7	4.0595
	346	803.9	1.4678	52.178	34.563	4843.1	4643.4	4.0487
37	347	855.0	1.3453	51.922	34.406	5041.9	4834.0	4.2389
	348	855.0	1.3491	51.864	34.363	5042.0	4834.4	4.2241
	349	854.9	1.3353	51.862	34.388	5041.4	4833.6	4.2594
	350	854.9	1.3426	51.673	34.147	5043.4	4838.3	4.2610
	351	855.0	1.3321	51.699	34.160	5042.4	4835.7	4.2954
	352	855.0	1.3468	51.688	34.281	5042.5	4835.5	4.2074
	353	855.0	1.3394	51.587	34.276	5042.7	4836.0	4.2034

Table (5) cont.

Reynolds No. = 0.7443 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x 10^4 poise
38	354	906.5	1.3126	54.666	35.934	5267.7	5040.1	4.4451
	355	906.8	1.3102	54.644	36.043	5269.0	5041.2	4.4122
	356	907.0	1.3115	54.605	36.058	5270.0	5042.2	4.3915
	357	907.4	1.3096	54.520	35.828	5272.0	5044.7	4.4439
	358	907.7	1.3050	54.666	35.850	5273.0	5045.4	4.4927
	359	908.0	1.3123	54.601	36.024	5274.5	5046.6	4.3942
	360	908.3	1.3098	54.608	36.028	5275.8	5047.9	4.4027
39	361	951.6	1.2366	55.189	36.225	5482.3	5243.4	4.5895
	362	951.7	1.2376	55.398	36.235	5482.4	5243.1	4.6415
	363	951.8	1.2308	55.243	36.292	5483.0	5243.7	4.6044
	364	952.0	1.2399	55.213	36.303	5483.9	5244.7	4.5582
	365	952.1	1.2332	55.131	36.302	5485.7	5246.6	4.5593
	366	952.1	1.2267	55.184	36.301	5985.6	5246.4	4.5987
	367	952.1	1.2436	55.173	36.316	5485.6	5246.3	4.5284
40	368	1012.4	1.1293	55.475	36.380	5719.9	5469.1	4.8504
	369	1012.4	1.1323	55.502	36.402	5719.9	5468.9	4.8384
	370	1012.5	1.1362	55.476	36.469	5720.4	5469.3	4.7918
	371	1012.6	1.1314	55.568	36.496	5720.5	5469.1	4.8304
	372	1012.8	1.1270	55.542	36.482	5720.6	5470.2	4.8479
	373	1012.9	1.1310	55.399	36.374	5721.8	5473.1	4.8260
	374	1013.4	1.1283	55.343	36.456	5726.2	5475.3	4.7856
41	375	1060.2	1.0352	54.039	35.542	5914.1	5658.9	4.9596
	376	1060.0	1.0334	54.073	35.481	5913.2	5657.9	5.0010
	377	1060.1	1.0340	54.086	35.574	5913.6	5658.1	4.9692
	378	1060.2	1.0397	54.040	35.592	5914.1	5658.9	4.9214
	379	1060.2	1.0362	54.049	35.602	5913.3	5657.8	4.9375
	380	1060.2	1.0313	54.129	35.616	5913.1	5657.3	4.9816
	381	1059.9	1.0305	54.012	35.405	5912.1	5657.3	5.0238
42	382	819.7	1.4573	53.399	35.422	4921.2	4713.8	4.1020
	383	819.8	1.4592	53.480	35.431	4921.4	4713.8	4.1148
	384	819.6	1.4710	53.503	35.429	4920.5	4712.0	4,0875
	385	819.4	1.4520	53.535	35.459	4919.5	4711.8	4.1432
	386	819.2	1.4664	53.329	35.322	4919.1	4712.3	4.0894
	387	819.1	1.4455	53.178	35.346	4919.8	4713.3	4.1003
	388	819.1	1.4399	53.030	35.078	4920.2	4714.2	4.1556

Table (5) cont.

Reynolds No. = 0.7443 Q/η (mean value for the two tubes)

Group	No.	$t^\circ C$	Q $\times 10^{+2}$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η $\times 10^4$ poise
43	389	977.6	1.1677	55.137	36.217	5641.8	5397.9	4.7151
	390	977.9	1.1630	54.986	36.223	5643.6	5400.1	4.6873
	391	978.2	1.1602	54.972	36.169	5644.9	5401.5	4.7108
	392	978.3	1.1676	54.813	36.050	5645.8	5403.2	4.6730
	393	978.5	1.1792	54.801	36.064	5646.8	5404.0	4.6183
	394	979.0	1.1681	54.859	36.085	5648.8	5405.8	4.7606
	395	979.1	1.1644	54.821	36.108	5649.4	5406.3	4.6659
44	396	1036.0	1.0964	55.743	36.506	5836.3	5578.9	4.9344
	397	1037.3	1.1069	55.809	36.506	5841.9	5584.1	4.9030
	398	1039.0	1.1016	55.781	36.458	5849.5	5591.5	4.9338
	399	1039.0	1.0987	55.810	36.440	5845.3	5587.2	4.9581
	400	1038.9	1.0937	55.957	36.639	5843.5	5585.5	4.9608
	401	1038.7	1.0999	55.906	36.641	5843.8	5584.9	5.0118

Table (6)

Reynolds No. = 0.8326 Q/η (mean value for the two tubes)

Group	No.	$t^\circ C$	Q $\times 10^2$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η $\times 10^4$ poise
45	402	294.2	2.0738	34.980	21.829	2597.4	2528.4	2.0044
	403	294.0	2.0662	34.991	21.907	2596.5	2527.4	2.0005
	404	293.8	1.6534	27.886	17.277	2604.7	2549.8	2.0474
	405	293.7	1.6440	27.890	17.280	2604.2	2549.3	2.0598
	406	293.4	1.2636	20.976	12.972	2609.9	2568.7	2.0380
	407	293.1	1.2580	20.783	12.834	2608.7	2567.9	2.0347
46	408	343.9	2.1406	43.169	26.945	2782.6	2690.2	2.2072
	409	343.9	2.1339	43.009	26.860	2782.8	2690.7	2.2035
	410	343.8	1.7208	33.865	21.068	2797.3	2724.9	2.1880
	411	343.8	1.7106	33.852	21.038	2798.1	2725.9	2.2042
	412	344.0	1.3271	25.784	15.800	2810.6	2755.8	2.2364
	413	344.1	1.3227	25.795	15.847	2811.1	2756.2	2.2344

Table (7)

Reynolds No. = 1.2822 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
47	414	147.4	7.3026	34.714	20.080	1943.3	1894.0	1.3902
	415	147.8	7.3165	34.861	20.139	1945.1	1895.6	1.3950
	416	147.9	7.3488	34.868	20.276	1945.6	1895.9	1.3728
	417	148.6	6.0031	28.390	16.305	1950.3	1910.0	1.4066
	418	148.9	5.8954	28.078	16.170	1952.0	1912.1	1.4092
	419	149.4	4.4835	21.212	12.117	1958.1	1928.0	1.4267
48	420	242.0	7.5505	54.605	31.560	2346.9	2251.9	1.7065
	421	242.1	7.3846	54.570	31.403	2350.7	2255.7	1.7550
	422	242.2	7.4764	54.554	31.375	2347.9	2253.1	1.7369
	423	242.6	5.9281	42.833	24.482	2362.5	2288.2	1.7561
	424	243.1	5.9178	42.812	24.538	2364.9	2290.5	1.7482
	425	243.9	4.4836	31.333	17.895	2378.8	2324.4	1.7144
	426	244.2	4.2659	30.879	17.668	2380.7	2327.0	1.7699
49	427	437.0	3.5163	49.440	27.588	3154.5	3037.3	2.6410
	428	437.8	3.5567	49.644	27.643	3157.7	3040.0	2.6274
	429	438.2	3.6036	49.540	27.618	3159.7	3042.0	2.5814
	430	439.3	2.3165	32.593	18.480	3191.1	3113.1	2.6002
	431	439.9	2.3123	32.832	18.613	3193.4	3114.8	2.6219
	432	440.1	2.3216	33.427	18.963	3197.5	3117.2	2.6504
50	433	549.1	2.6629	50.676	28.325	3746.4	3607.5	3.0041
	434	548.7	2.6409	50.602	28.161	3744.7	3606.4	3.0481
	435	548.5	2.6154	50.342	28.018	3744.3	3606.7	3.0630
	436	548.5	2.6005	50.242	27.902	3747.2	3610.0	3.0835
	437	548.6	1.6156	31.365	17.798	3782.3	3695.2	3.0325
	438	548.5	1.6529	31.455	17.870	3781.0	3694.4	2.9685
51	439	660.8	1.8127	44.712	25.105	4198.9	4059.5	3.4738
	440	661.2	1.7895	44.690	25.001	4201.6	4061.5	3.5350
	441	661.7	1.7965	44.775	24.990	4202.8	4063.3	3.5411
	442	663.9	1.8125	44.511	24.937	4208.9	4069.5	3.4633
	443	664,7	1.8103	44.491	24.940	4212.5	4073.4	3.4601
	444	665.4	1.7823	44.487	24.872	4215.7	4076.5	3.5267

Cont./

Table (7) cont.

Reynolds No. = 1.2822 Q/η (mean value for the two tubes)

Group	No.	t^oC	$Q \times 10^3$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	$\eta \times 10^4$ poise
52	445	616.3	2.0491	45.693	25.509	4012.9	3877.1	3.3121
	446	617.2	2.0189	45.706	25.536	4017.0	3880.9	3.3549
	447	617.8	2.0232	45.692	25.575	4019.7	3883.6	3.3345
	448	618.1	1.3464	30.544	17.309	4053.5	3962.0	3.3175
	449	617.0	1.3402	30.538	17.306	4048.5	3957.1	3.3365
	450	616.0	1.3524	30.220	17.060	4044.5	3954.4	3.2971
53	451	532.7	2.4250	45.306	25.429	3720.2	3598.5	2.9713
	452	532.5	2.4503	45.519	25.482	3718.9	3596.7	2.9675
	453	532.4	2.4510	45.386	25.443	3718.6	3596.8	2.9520
	454	531.8	1.6378	30.686	17.253	3741.1	3658.7	3.0076
	455	531.8	1.6330	30.688	17.321	3747.1	3664.6	2.9934
	456	531.7	1.6040	30.636	17.269	3746.7	3664.4	3.0495
54	457	738.0	1.4899	43.389	24.244	4593.8	4446.6	3.7878
	458	738.1	1.4538	42.734	24.081	4595.6	4451.2	3.7734
	459	738.6	1.4755	43.037	24.135	4597.2	4452.0	3.7715
	460	739.1	1.4928	43.010	24.014	4592.6	4447.6	3.7559
	461	739.6	1.4658	42.930	24.060	4595.1	4450.1	3.7927
	462	739.8	1.4384	42.962	24.089	4595.9	4450.8	3.8640
55	463	111.7	7.1060	28.232	16.492	1712.4	1675.4	1.3046
	464	111.4	7.0639	27.855	16.391	1712.2	1676.0	1.2794
	465	111.2	7.0851	27.888	16.312	1710.4	1673.8	1.2916
	466	110.4	8.8213	34.642	20.277	1702.1	1656.7	1.2809
	467	110.3	8.8737	35.169	20.612	1701.2	1655.1	1.2893
	468	110.3	4.8039	19.019	11.206	1715.2	1690.2	1.2946
	469	110.3	5.0019	19.752	11.540	1714.5	1690.4	1.3122
56	470	188.0	7.9632	45.656	26.297	2058.6	1987.7	1.5690
	471	188.1	8.0367	45.645	26.269	2059.1	1988.2	1.5562
	472	188.6	6.9958	39.579	22.898	2066.6	2004.9	1.5432
	473	188.7	7.0526	39.634	22.846	2067.0	2005.3	1.5425
	474	189.1	5.6417	32.142	18.567	2074.8	2024.7	1.5680
	475	189.2	5.7210	32.135	18.570	2075.2	2025.1	1.5445
	476	189.3	4.2412	23.994	13.840	2080.1	2042.7	1.5737
	477	189.4	4.3201	23.976	13.818	2080.6	2043.2	1.5456
	478	189.4	4.2750	23.940	13.834	2080.6	2043.2	1.5526

Cont./

Table (7) cont.

Reynolds No. = 1.2822 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^3 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
57	479	316.9	5.4507	51.321	29.001	2607.9	2508.6	2.0900
	480	316.8	5.5274	51.335	29.022	2607.4	2508.1	2.0602
	481	316.7	4.6148	43.113	24.433	2618.3	2533.4	2.0758
	482	316.7	4.6081	43.151	24.443	2618.3	2533.2	2.0822
	483	316.7	3.8881	36.456	20.718	2627.8	2555.9	2.0843
	484	316.7	3.9145	36.520	20.663	2627.8	2555.8	2.0891
	485	316.8	2.8798	26.857	15.300	2641.6	2588.6	2.0795
	486	316.8	2.9646	27.531	15.642	2640.8	2586.4	2.0787

Table (8)

Reynolds No. = 0.6497 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^2 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
58	487	869.8	1.6183	38.352	24.221	5081.9	4929.3	4.2897
	488	869.8	1.6141	38.417	24.269	5081.8	4928.9	4.3059
	489	869.9	1.6150	38.412	24.247	5082.3	4929.4	4.3081
	490	869.7	1.3969	33.135	21.025	5100.3	4968.2	4.2744
	491	869.5	1.4075	33.188	21.039	5099.3	4967.0	4.2966
	492	868.7	1.1551	27.402	17.288	5114.9	5006.0	4.3391
	493	868.3	1.1579	27.270	17.288	5113.4	5004.9	4.2744
	494	867.3	1.0416	24.468	15.442	5116.1	5018.9	4.3101
	495	867.2	1.0435	24.428	15.469	5115.8	5018.6	4.2713
59	496	962.4	1.5446	42.598	26.829	5474.1	5291.2	4.6243
	497	962.5	1.5545	42.617	26.824	5474.5	5291.6	4.6014
	498	962.4	1.5571	42.613	26.883	5474.1	5291.0	4.5752
	499	961.9	1.3553	37.046	23.434	5491.7	5332.4	4.5702
	500	962.0	1.3507	37.051	23.370	5492.1	5333.0	4.6089
	501	961.8	1.1704	31.966	20.135	5509.3	5372.0	4.6177
	502	961.8	1.1744	31.924	20.160	5507.0	5369.8	4.5777

Cont./

Table (8) cont.

Reynolds No. = 0.6497 Q/η (mean value for the two tubes)

Group	No.	t°C	Q x10^2 g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	η x10^4 poise
59 cont.	503	961.8	1.1742	31.872	20.108	5507.1	5370.2	4.5789
	504	962.2	0.9650	26.139	16.508	5528.6	5416.2	4.5797
	505	963.0	0.9569	26.147	16.553	5532.2	5419.5	4.5975
60	506	1033.8	1.3360	40.262	25.426	5775.5	5591.0	4.7809
	507	1033.8	1.3373	40.265	25.348	5775.5	5591.1	4.8028
	508	1033.7	1.1782	35.656	22.400	5792.5	5629.4	4.8625
	509	1033.6	1.1816	35.547	22.398	5792.4	5629.6	4.8097
	510	1033.5	1.0084	30.356	19.217	5813.3	5674.1	4.7921
	511	1033.7	1.0062	30.291	19.165	5814.4	5675.5	4.7968
	512	1033.9	0.8766	26.229	16.635	5830.2	5709.7	4.7623
	513	1033.9	0.8703	26.140	16.583	5830.4	5710.4	4.7779
	514	1034.0	0.8702	26.141	16.518	5830.9	5711.0	4.8110
61	515	832.6	1.5206	33.440	21.166	4919.3	4790.5	4.1236
	516	832.7	1.5078	33.419	21.161	4919.8	4791.0	4.1527
	517	833.5	1.2786	28.329	17.937	4941.9	4834.0	4.1650
	518	833.7	1.2828	28.326	17.935	4942.8	4833.4	4.1480
	519	834.0	1.2651	28.259	17.828	4944.2	4834.3	4.2195
	520	833.9	1.2796	28.271	17.854	4943.7	4834.8	4.1682
62	521	913.9	1.5153	38.441	24.319	5276.5	5117.2	4.4091
	522	913.8	1.5091	38.398	24.268	5276.2	5117.1	4.4302
	523	913.6	1.5122	38.407	24.266	5275.3	5116.2	4.4253
	524	913.5	1.6760	42.429	26.711	5264.3	5088.9	4.4226
	525	913.6	1.6771	42.663	26.872	5264.2	5087.7	4.4512
	526	913.9	1.3055	33.135	20.980	5299.1	5161.8	4.4189
	527	914.1	1.3123	33.200	20.931	5299.9	5162.5	4.4364
	528	914.1	1.0927	27.689	17.497	5322.8	5208.0	4.4408
	529	914.1	1.0866	27.483	17.408	5323.3	5209.4	4.4153
63	530	1003.8	1.6261	48.045	29.825	5699.0	5486.0	4.8476
	531	1003.7	1.6177	47.830	29.741	5699.1	5487.0	4.8388
	532	1003.5	1.6325	47.614	29.685	5698.8	5487.5	4.7531
	533	1002.7	1.3995	41.191	25.726	5720.0	5537.2	4.8074
	534	1002.6	1.3981	41.109	25.770	5719.8	5537.1	4.7734
	535	1002.4	1.1926	35.029	21.978	5741.1	5585.1	4.7831
	536	1002.7	1.1930	34.883	21.992	5742.8	5587.4	4.7224
	537	1003.6	1.0360	30.334	18.999	5762.7	5627.9	4.7948
	538	1003.6	1.0346	30.332	19.024	5762.7	5627.8	4.7897

cont./

Table (8) cont.

Reynolds No. = 0.6497 Q/η (mean value for the two tubes)

Group	No.	t°C	$Q \times 10^2$ g/sec	ΔP_1 gf/cm^2	ΔP_2 gf/cm^2	V_1 cm^3/g	V_2 cm^3/g	$\eta \times 10^4$ poise
64	539	1070.4	1.4878	47.708	29.681	5930.6	5712.2	5.0379
	540	1070.1	1.4929	47.517	29.687	5929.8	5711.9	4.9666
	541	1070.5	1.3078	41.644	26.135	5950.8	5754.1	4.9450
	542	1070.5	1.3082	41.548	26.036	5949.9	5755.0	4.9484
	543	1070.6	1.1111	35.268	22.159	5976.1	5809.5	4.9438
	544	1070.7	1.1093	35.187	22.128	5976.8	5810.5	4.9324
	545	1071.3	0.9563	30.288	19.061	5994.0	5850.7	4.9381
	546	1071.9	0.9589	30.341	19.167	5996.4	5852.6	4.8995
65	547	1048.6	1.5274	46.834	29.293	5769.6	5570.1	4.9286
	548	1048.4	1.5329	46.886	29.244	5768.6	5569.1	4.9403
	549	1048.1	1.3254	40.274	25.284	5795.1	5615.3	4.8621
	550	1048.0	1.3218	40.245	25.278	5794.7	5615.1	4.8118
	551	1047.9	1.3249	40.244	25.285	5794.3	5614.6	4.8547
	552	1047.3	1.1272	34.356	21.601	5814.6	5656.7	4.8817
	553	1047.1	1.1293	34.321	21.577	5812.6	5654.9	4.8700
	554	1046.4	0.9302	28.237	17.729	5832.4	5701.8	4.8974
	555	1046.4	0.9275	28.237	17.823	5832.4	5701.5	4.8689

DISCUSSION OF RESULTS

From the results obtained from the single capillary apparatus (tables (1) and (2)), graphs of viscosity against temperature and percentage deviation from Shifrin's equation, $\eta = \{.804 + .00407t\} \times 10^{-4}$ poise against temperature were plotted $\{$see figures (8.1) and (8.2)$\}$. Since, in the first instance, the single capillary determinations were only trial runs, it was essential that the results be reasonably comparable with existing well defined low temperature data. For clarity the results were only compared with those of Shifrin (28), Bonilla (22) and Kestin (41), the more recent reliable data in this field of work. The relative value of Shifrin's and Bonilla's work compared with other work in this field has been discussed in Chapter II, and a deviation plot is given in figure (2.1).

It can be observed from figures (8.1) and (8.2) that the experimental points were within the tolerance put forward at the report of the second meeting of the international co-ordinating committee on the properties of steam, Munich, July 1962, i.e. ± 1 % of Shifrin's between 100°C and 300°C, and ± 3 % between 300°C and 900°C, the majority of the individual determinations being within a 2 % band. The actual trend of the curve is to diverge from Shifrin's and Bonilla's, agreeing more closely with the limited data given by Kestin (up to 300°C). If the present work had been based solely on the results from the single capillary apparatus, the atmospheric line would be about 2 % higher than Shifrin's at 400°C. The scatter of the 109 points can be observed to be much less than that obtained by Shifrin over the temperature range of 100°C to 450°C.

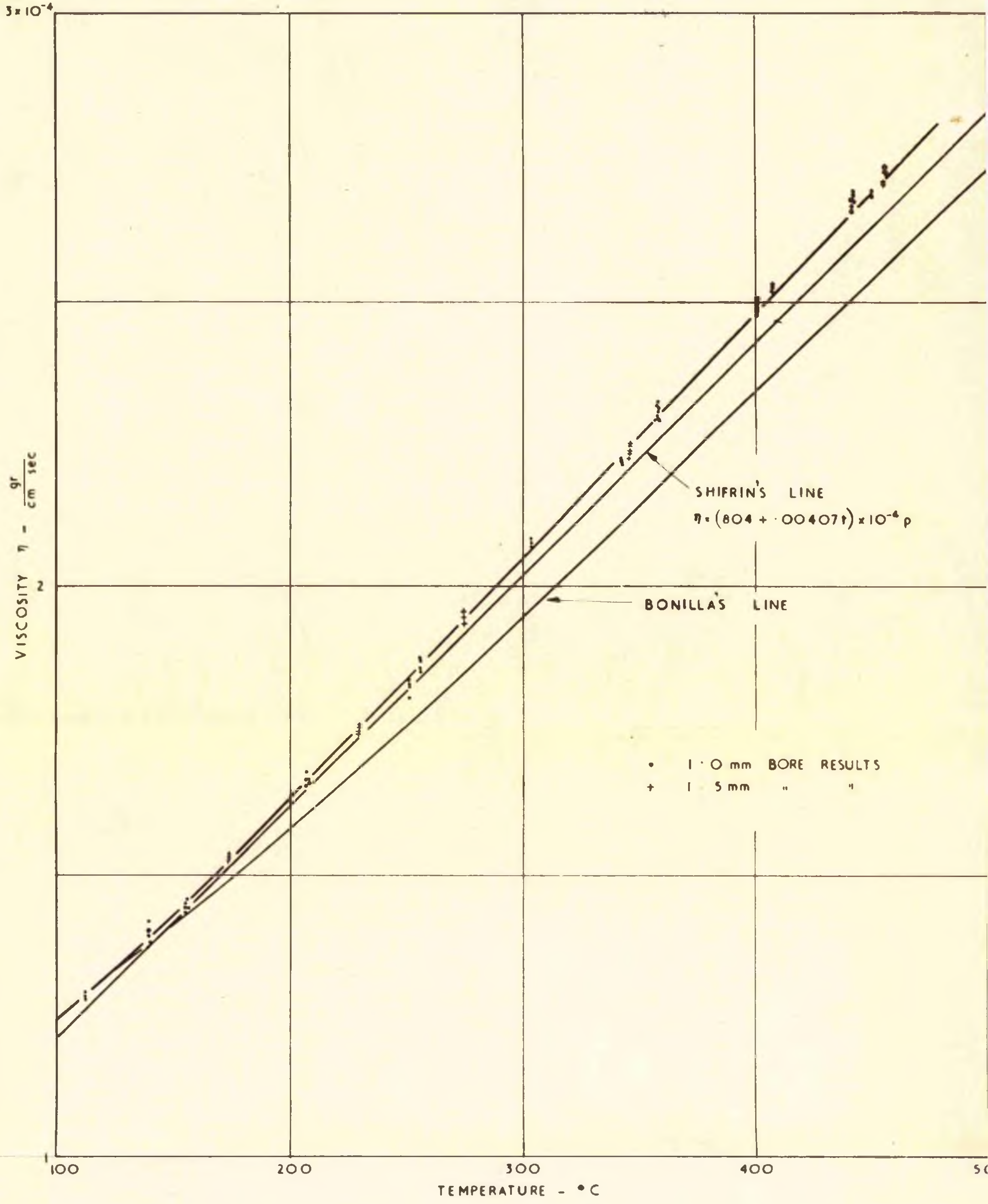


FIG 8. I VISCOSITY ∿ TEMPERATURE °C FROM SINGLE CAPILLARY SYSTEM

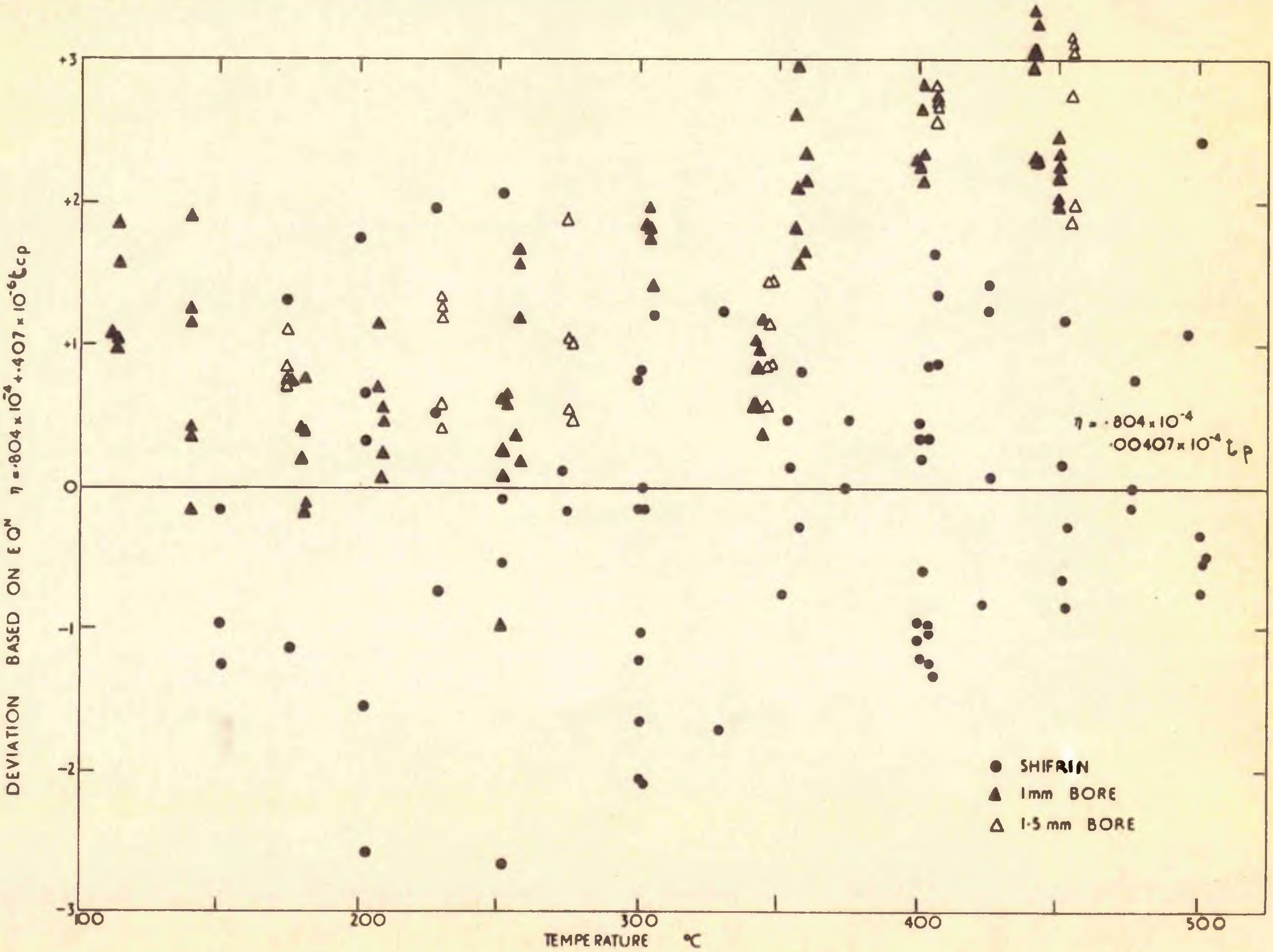


FIG 8.2 DEVIATION PLOT FOR SINGLE CAPILLARY RESULTS AND SHIFRINS RESULTS

Having obtained favourable results from the single capillary glass apparatus, work on the series capillary quartz apparatus was commenced, the anticipated temperature range being 100°C to 1050°C (the safe temperature ceiling for quartz - higher temperatures being likely to produce devitrification of the quartz).

A viscosity against temperature plot for the whole of the results obtained from both apparatus is given in figure (8.3). The curve which is shown on this graph was obtained from a fifth degree polynomial, which has been fitted to the experimental data using the least squares fit method. The curve fitting computation was performed by Hilsenrath of the National Bureau of Standards, U.S.A. (169). In actual fact four polynomials were fitted to the experimental data, 2nd, 3rd, 4th and 5th degree. The fifth degree gives the smallest standard deviation. The procedure adopted by Hilsenrath was to take the mean of each group, since his computer could not take 555 points in one process. The validity of this procedure is open to doubt, but the result appears to be quite satisfactory. The coefficients and their standard deviations for each of the polynomials obtained are given below, together with the overall standard deviation of each equation.

$$\eta = a + bt + ct^2 + dt^3 + et^4 + ft^5.$$

coefficient	standard deviation	
$a = 7.2105 \times 10^{-6}$ poise	1.459×10^{-6} poise	
$b = 4.60299 \times 10^{-5}$ poise	5.810×10^{-9} "	(8.1)
$c = -5.61426 \times 10^{-11}$ poise	4.856×10^{-12} "	

overall standard deviation = 3.078×10^{-6} poise

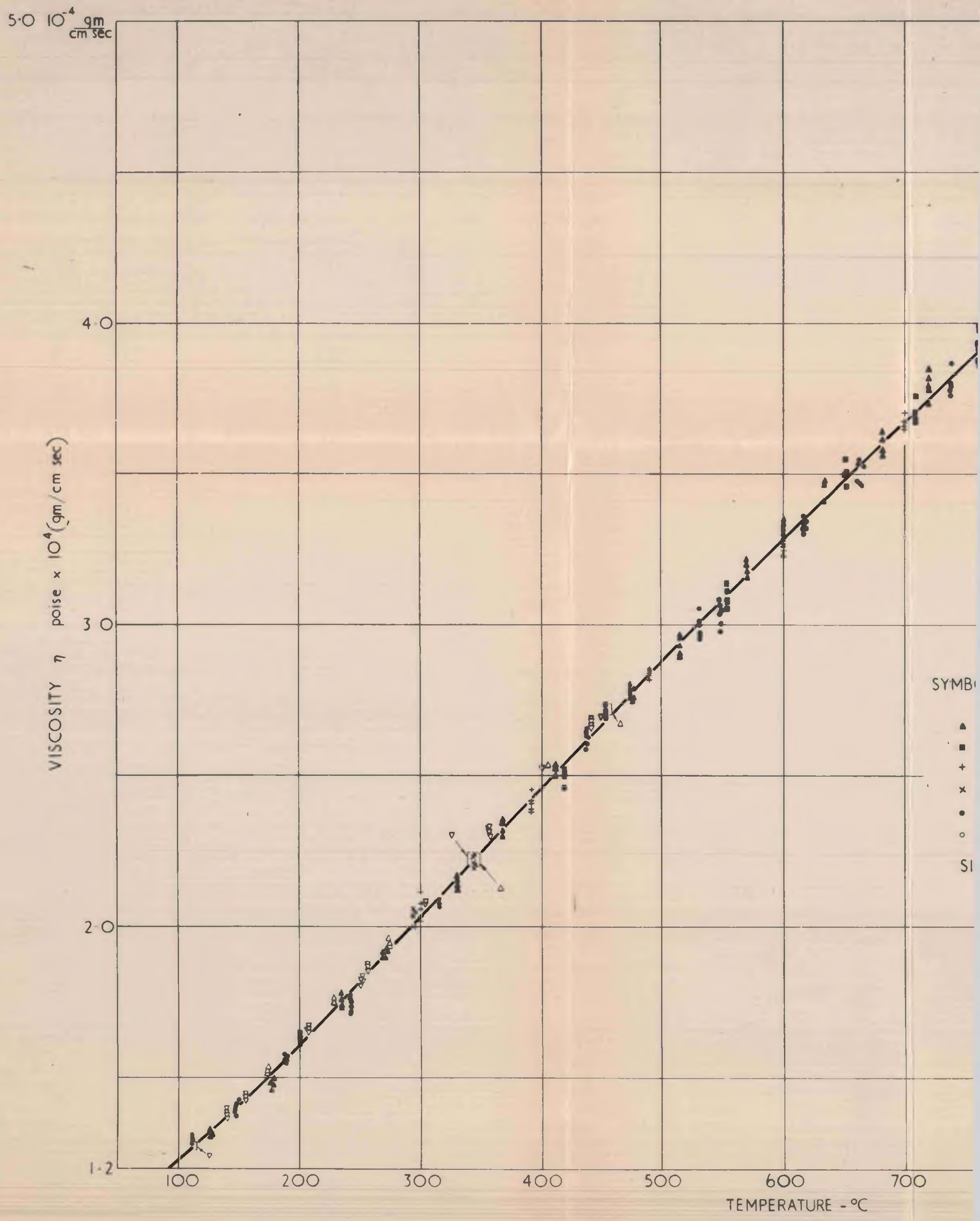


FIG. 8.3

VISCOSITY ∼ TEMPERATURE FOR STEAM AT ATMOS

coefficient	standard deviation	
$a = 7.97762 \times 10^{-5}$ poise	2.444×10^{-6} poise	
$b = 4.02816 \times 10^{-7}$ "	1.618×10^{-8} "	
$c = 5.82647 \times 10^{-11}$ "	3.070×10^{-11} "	(8.2)
$d = -6.53462 \times 10^{-14}$ "	1.734×10^{-14} "	

overall standard deviation = 2.852×10^{-6} poise

coefficient	standard deviation	
$a = 8.30922 \times 10^{-5}$ poise	4.550×10^{-6} poise	
$b = 3.67441 \times 10^{-7}$ "	4.401×10^{-8} "	
$c = 1.74460 \times 10^{-10}$ "	1.379×10^{-10} "	(8.3)
$d = -2.11800 \times 10^{-13}$ "	1.703×10^{-13} "	
$e = 6.22371 \times 10^{-17}$ "	7.199×10^{-17} "	

overall standard deviation = 2.825×10^{-6} poise

coefficient	standard deviation	
$a = 9.50815 \times 10^{-5}$ poise	8.479×10^{-6} poise	
$b = 2.02506 \times 10^{-7}$ "	1.080×10^{-7} "	
$c = 9.43232 \times 10^{-10}$ "	4.806×10^{-10} "	
$d = -1.78368 \times 10^{-12}$ "	9.572×10^{-13} "	(8.4)
$e = 1.51275 \times 10^{-15}$ "	8.724×10^{-16} "	
$f = -4.93862 \times 10^{-19}$ "	2.960×10^{-19} "	

overall standard deviation = 2.825×10^{-6} poise

Overall percentage standard deviation = 1.8 %

The deviation of the experimental points from the fifth degree polynomial is given in figures (8.4) to (8.11). It can be seen from figure (8.4) that more than 95 % of the points lie within a ± 2 % band, thus giving an expected standard deviation of about ± 1.3 % (2nd standard deviation), which compares favourably with the actual calculated value of ± 1.2 %. The plots for the experimental points obtained from each arrangement are given in figures (8.5) to (8.11). Figures (8.5) and (8.6), the results obtained using the single capillary apparatus, show good agreement with the equation (8.4)

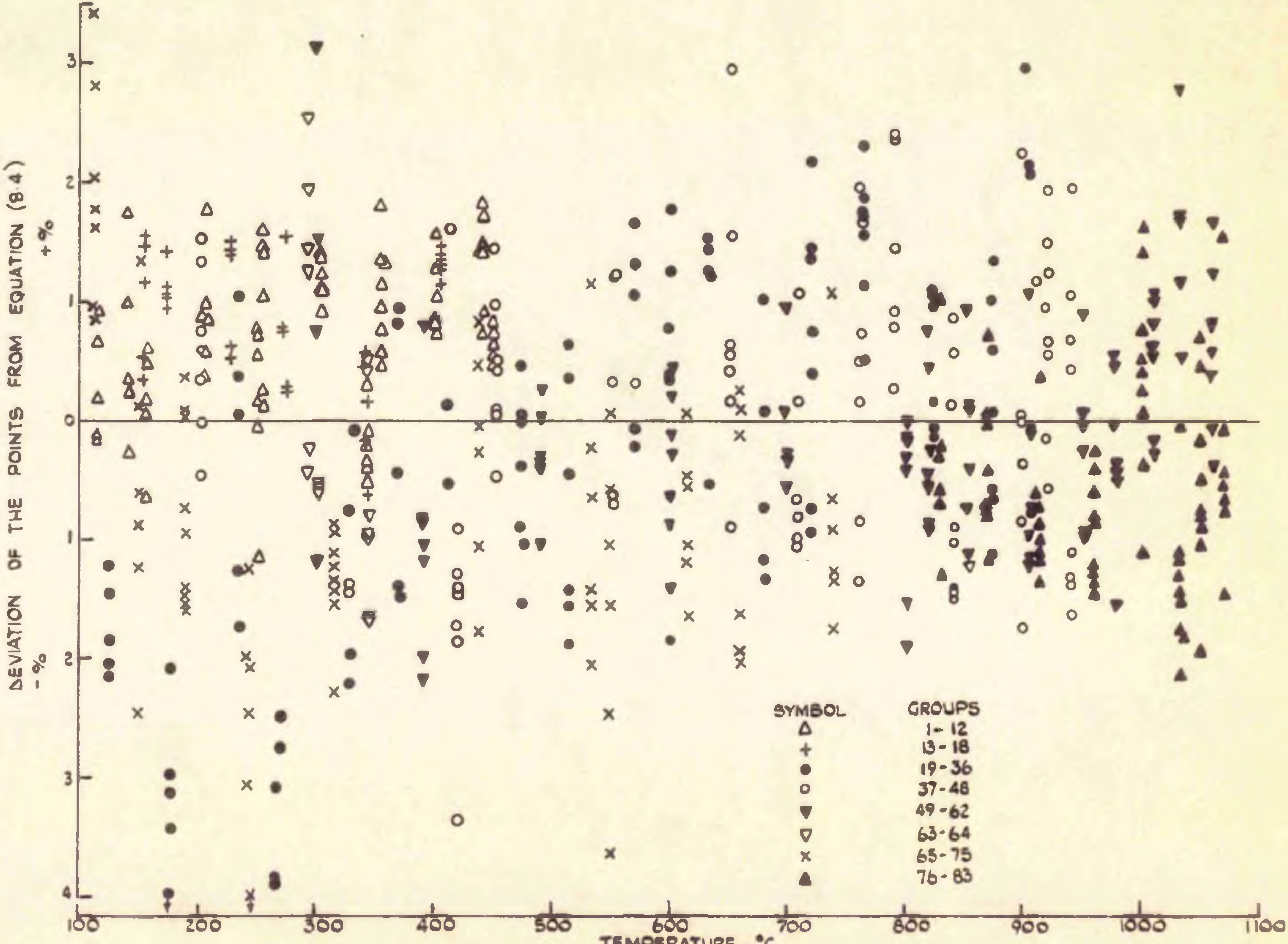


FIG 8·4 MORE THAN 95% OF THE POINTS LIE WITHIN A ± 2% BAND

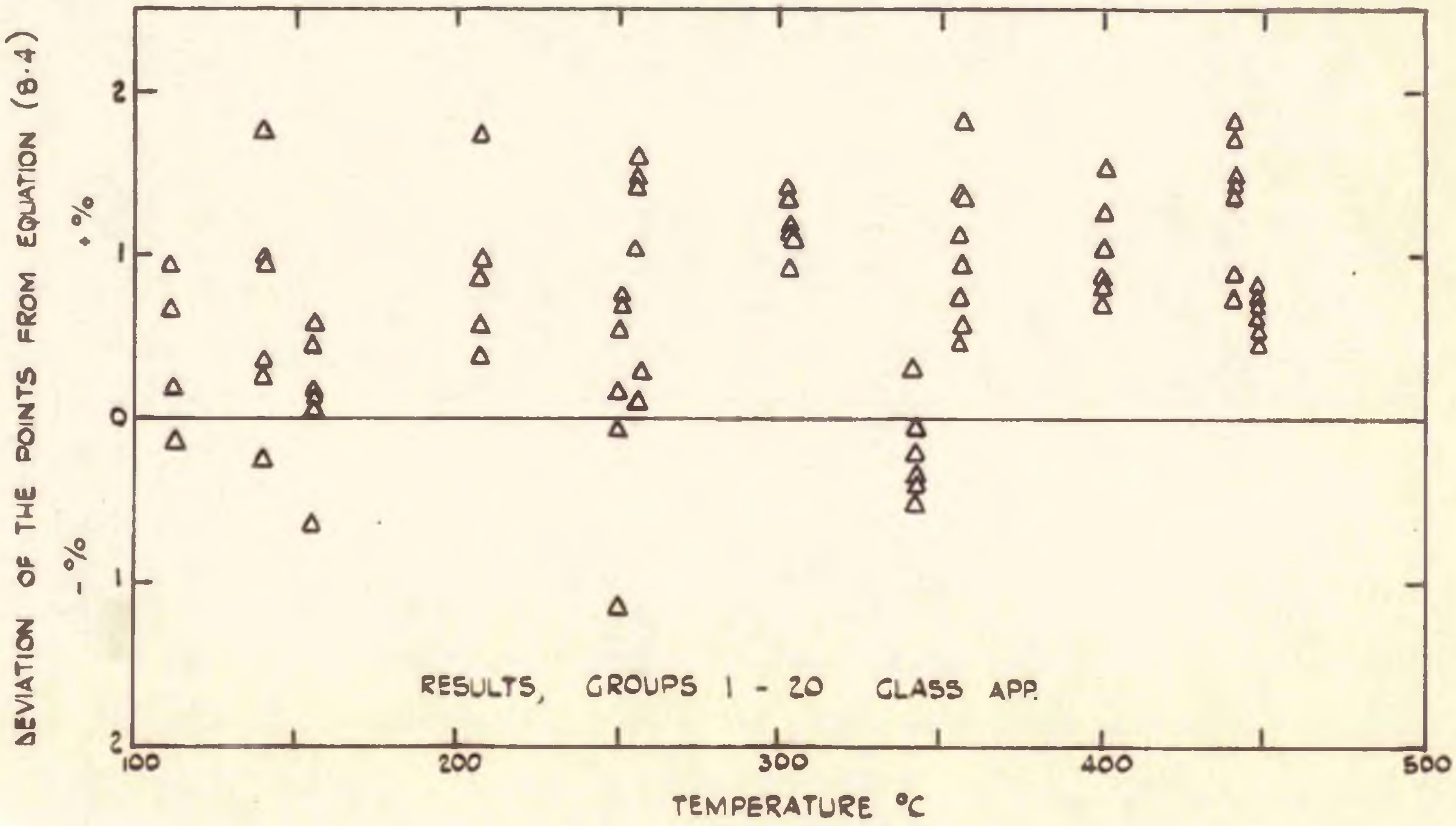


FIG 8·5 ALL POINTS LIE WITHIN ± 2% BAND.

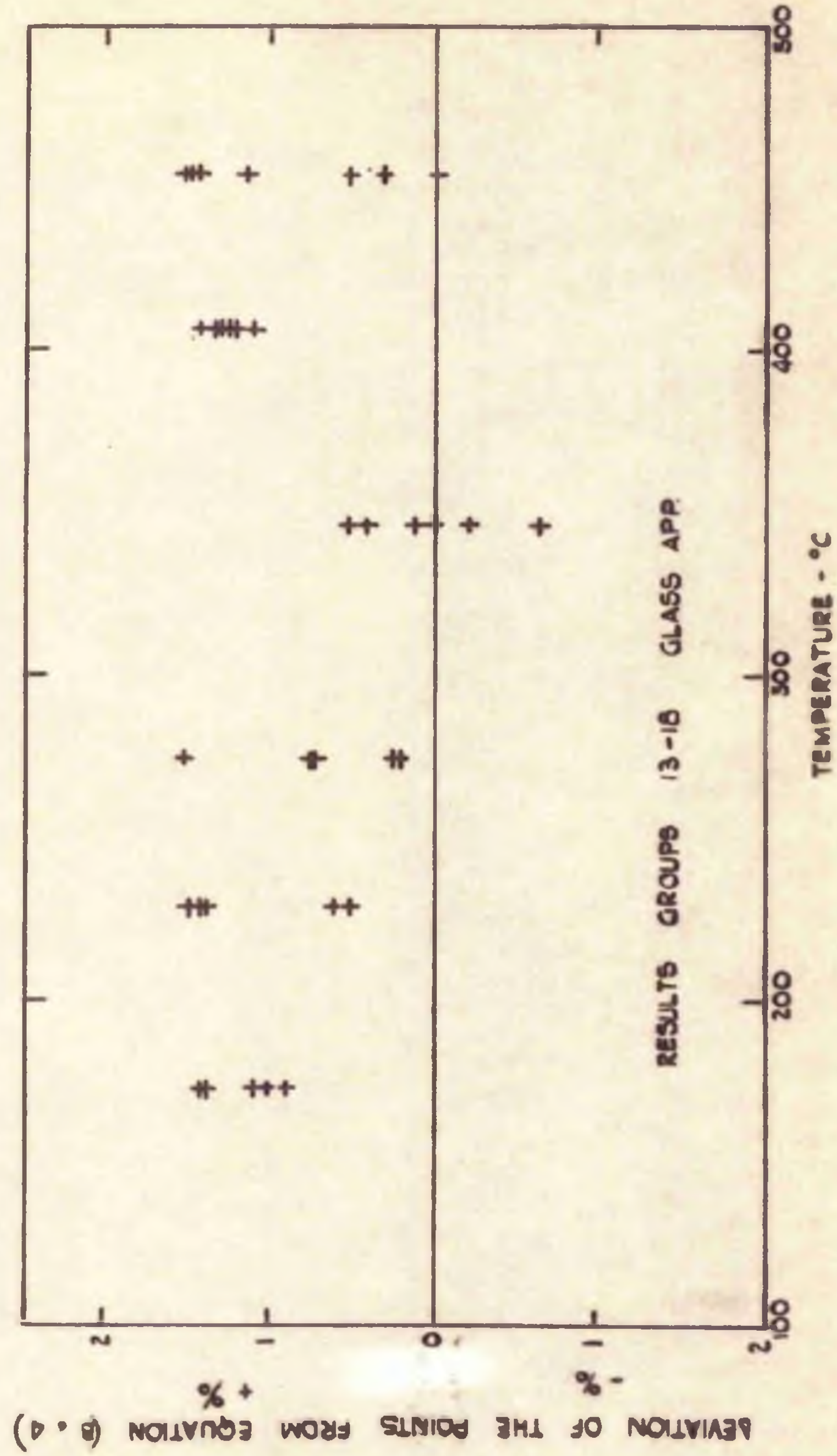


FIG 8.6 ALL POINTS LIE WITHIN ± 2% BAND.

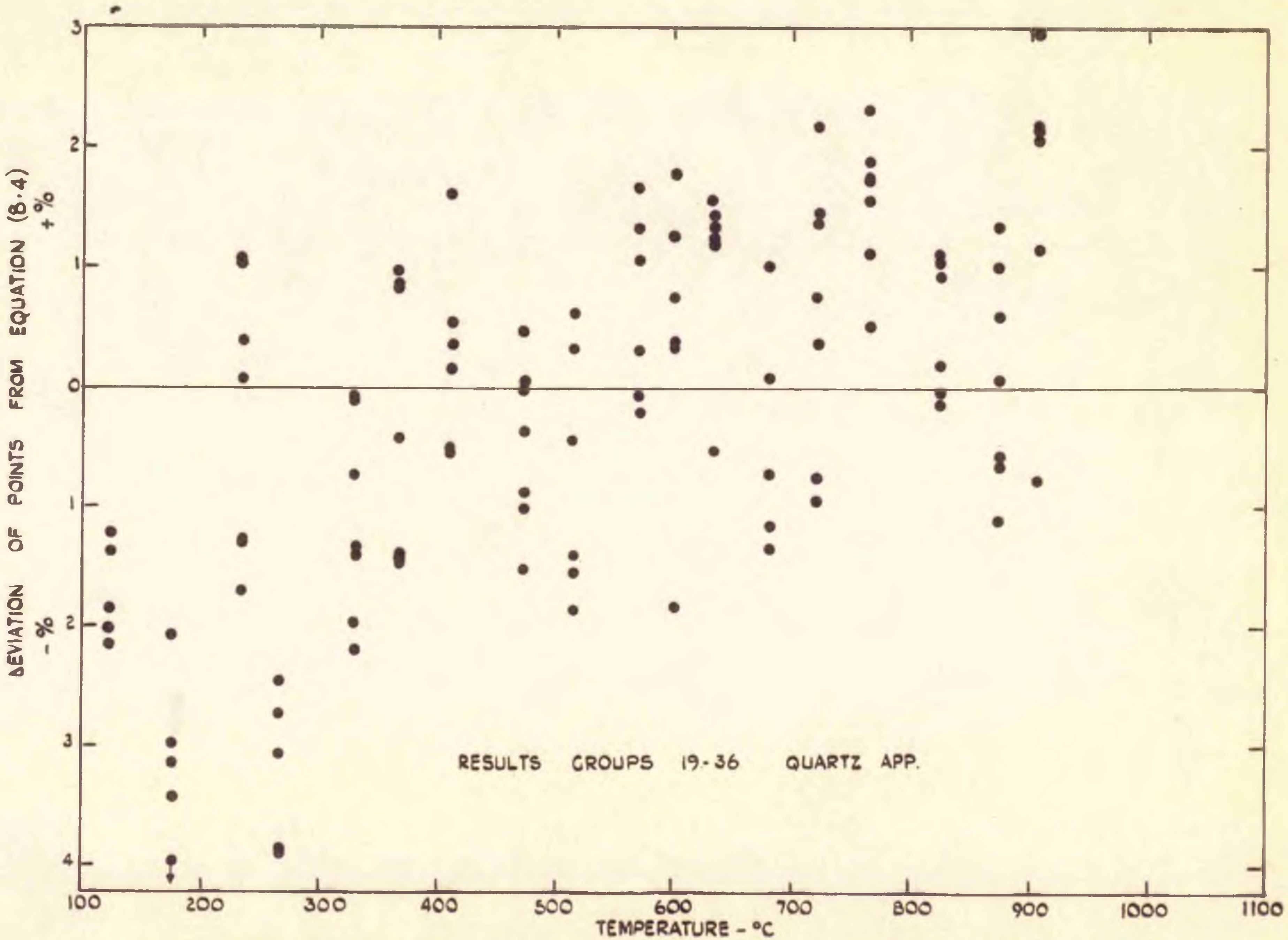


FIG 8·7 MORE THAN 84% OF THE POINTS LIE WITHIN A ± 2% BAND

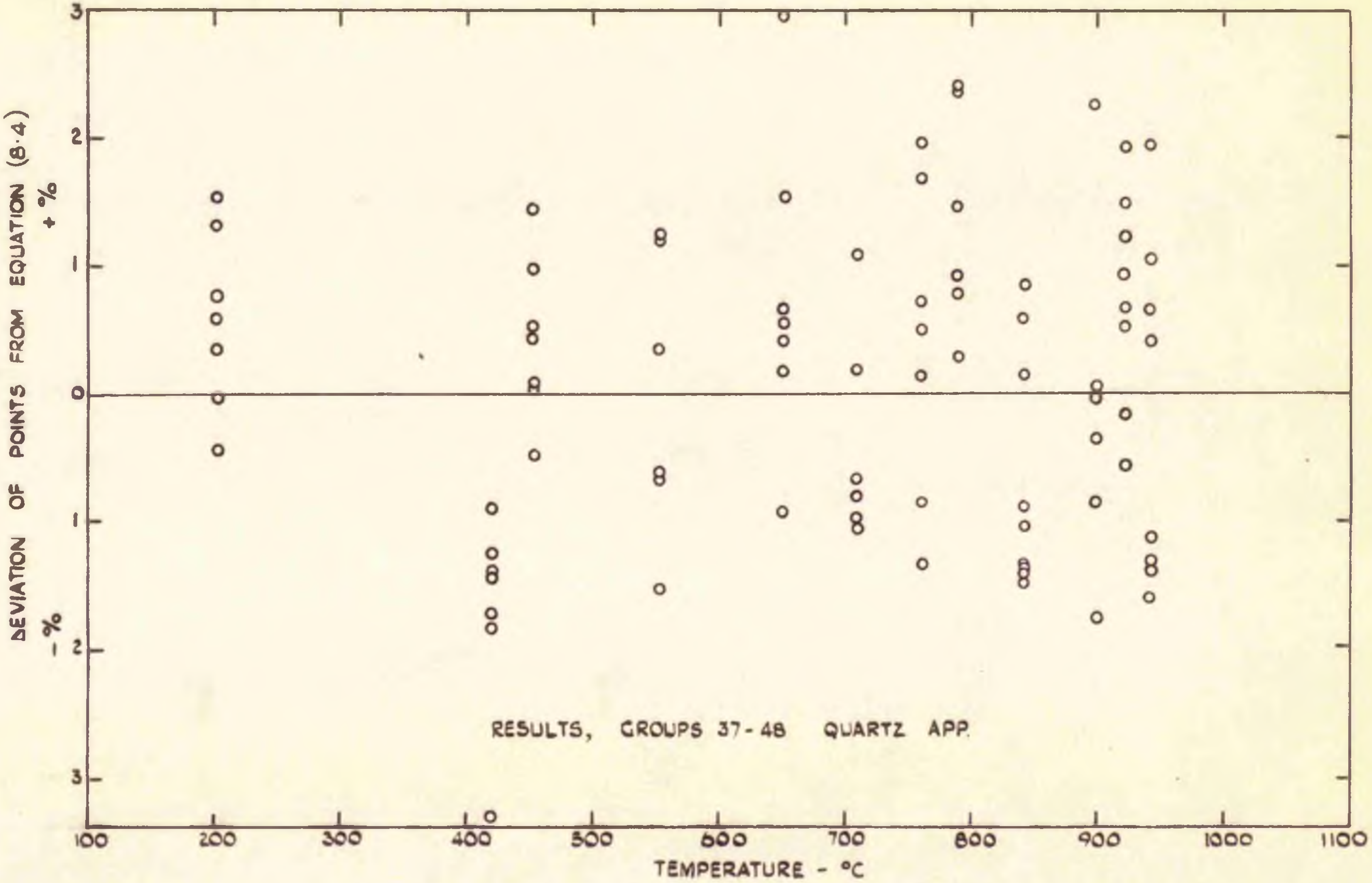


FIG 8·8 "MORE THAN 95% OF THE POINTS LIE WITHIN A ± 2% BAND.

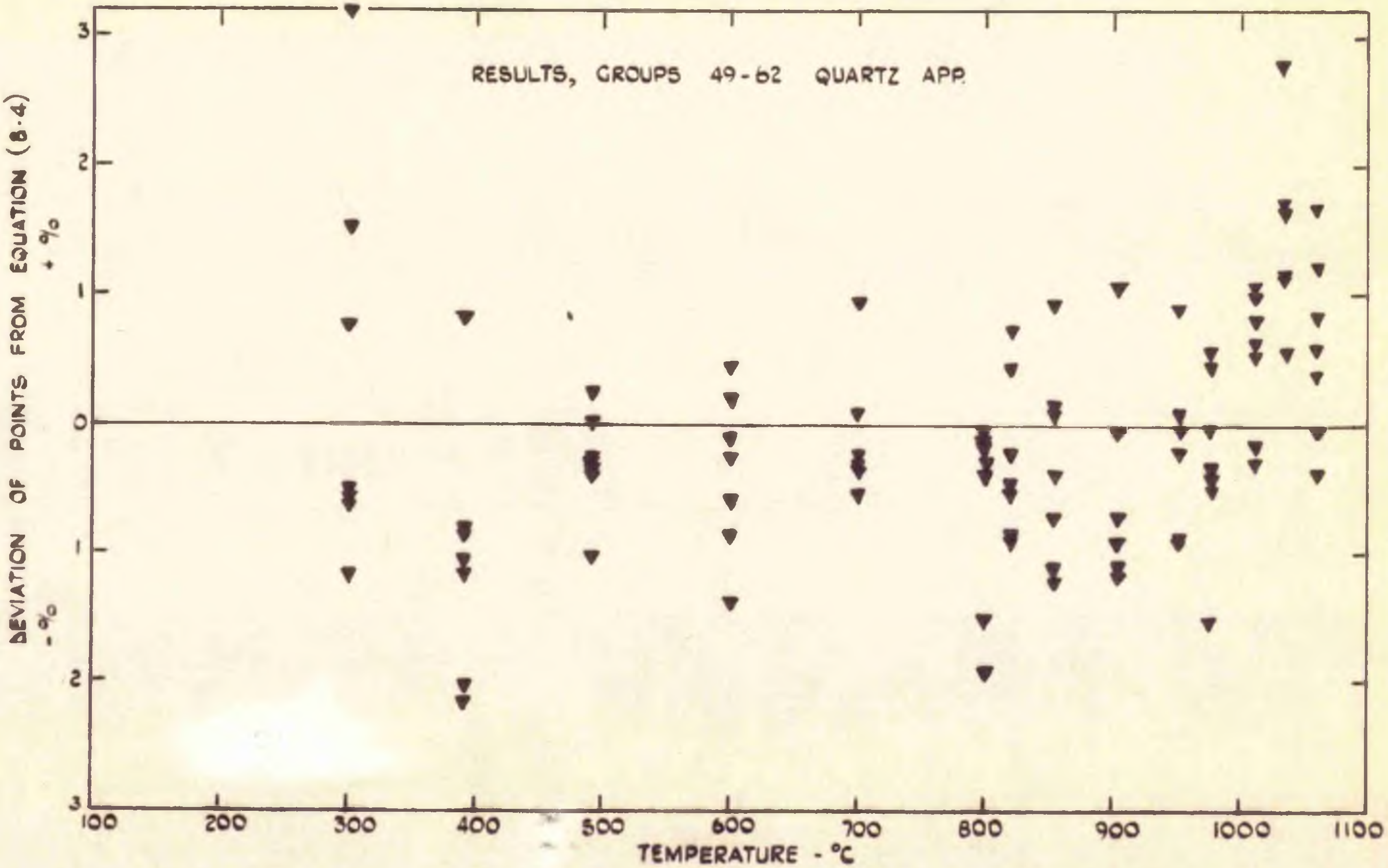


FIG 8.9 MORE THAN 96% OF THE POINTS LIE WITHIN A ±2% BAND

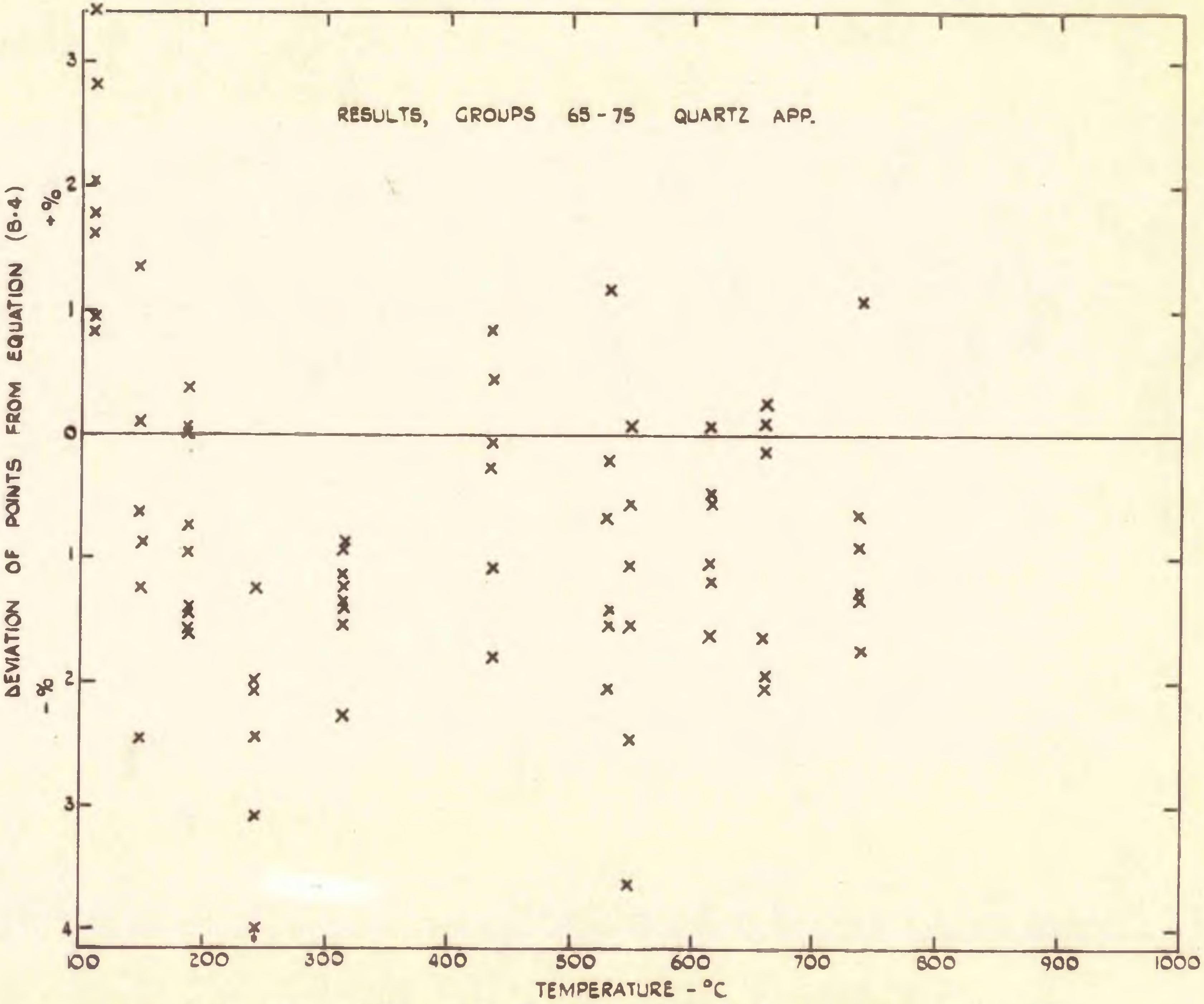


FIG B·10 MORE THAN 81% OF THE POINTS LIE WITHIN A ± 2% BAND

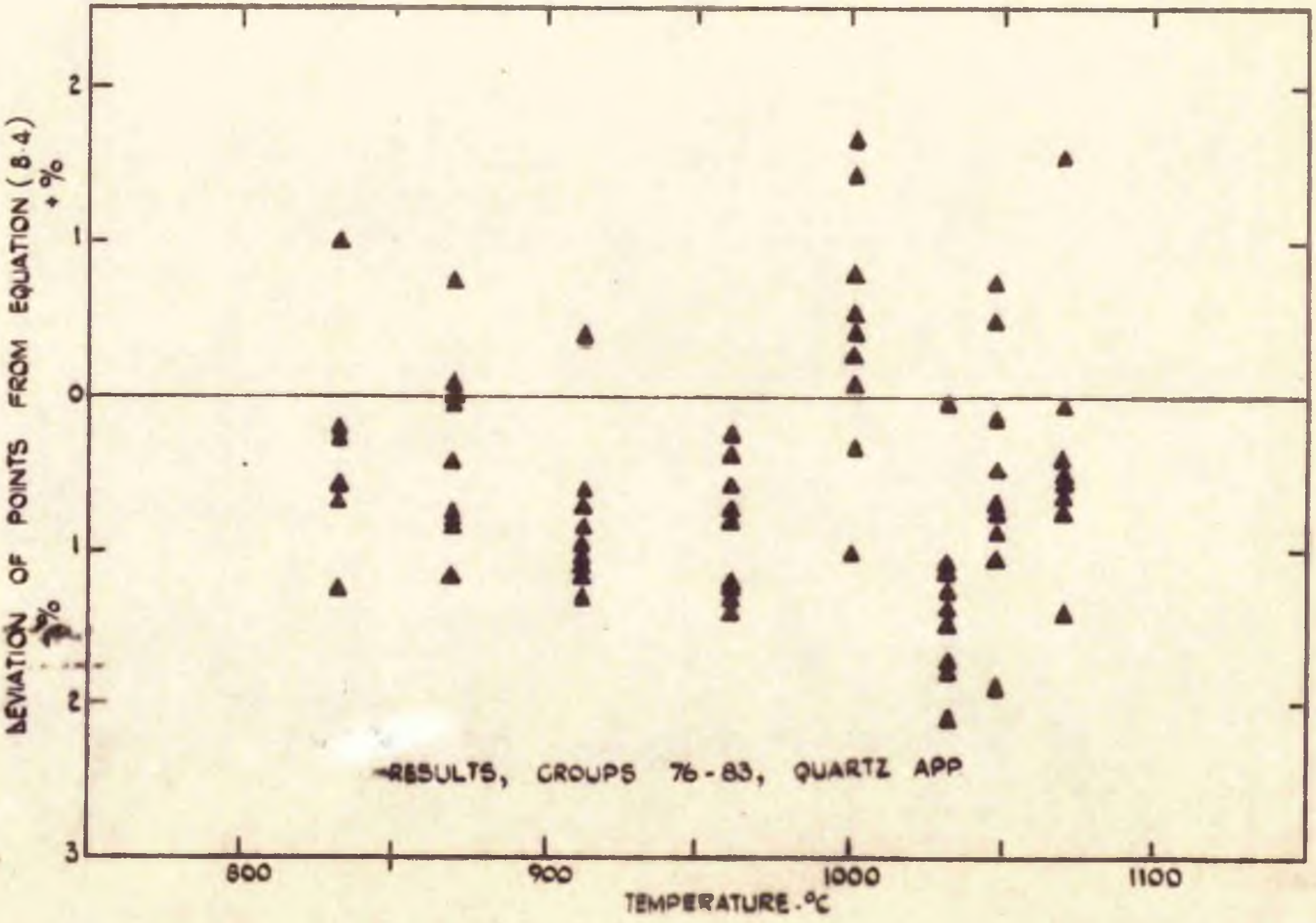


FIG 8·11 MORE THAN 98 % OF THE POINTS LIE WITHIN A ± 2 % BAND all the points lying well within a ± 2 % band. The points obtained from the first series capillary arrangement that was used (figure (8.4)) are not quite so good, only about 84 % of the points lying within a ± 2 % band. However, in all but one of the later arrangements more than 95 % of the points lie within a ± 2 % band. In the penultimate arrangement only 81 % of the points lie within the ± 2 % band. It can be seen that this may be due to the lack of agreement at the lower temperatures, especially at 110°C where the results appear to be inconsistently high. No definite reason for this effect can be given, apart from the fact that this group of results was taken very near the saturation temperature and the steam could have been wet. On the other hand, these results could be correct, tending to make the atmospheric line curve up to meet the saturation line.

A table (table 9) of the recommended values, generated at 50°C intervals between 100°C and 1100°C, has been produced, using equation (8.4), on the following page. A deviation plot comparing the present work (recommended values given in table 9) with values put forward by Bonilla, Keetin and Shifrin is given in figure (8.12). It shows that up to about 300°C both Shifrin and Keetin agree with the present work to within 1 %. At 450°C the present work has its maximum disagreement with both Bonilla and Shifrin, the maximum differences being about -3.1 % and -1.7 % from the two authors respectively. Bonilla and Shifrin agree quite closely on the shape of the curve, but the present work agrees more closely with that of Keetin (who used an entirely different method), inferring more curvature of the atmospheric line between 100°C and 600°C.

Table (9)

Recommended values for the viscosity of steam at atmospheric pressure, based on the results of the present work. (i.e. equation (8.4))

Temperature °C.	Viscosity poise
100°	1.231
150°	1.414
200°	1.613
250°	1.822
300°	2.036
350°	2.251
400°	2.465
450°	2.676
500°	2.883
550°	3.086
600°	3.285
650°	3.481
700°	3.674
750°	3.865
800°	4.053
850°	4.238
900°	4.420
950°	4.595
1000°	4.760
1050°	4.912
1100°	5.045

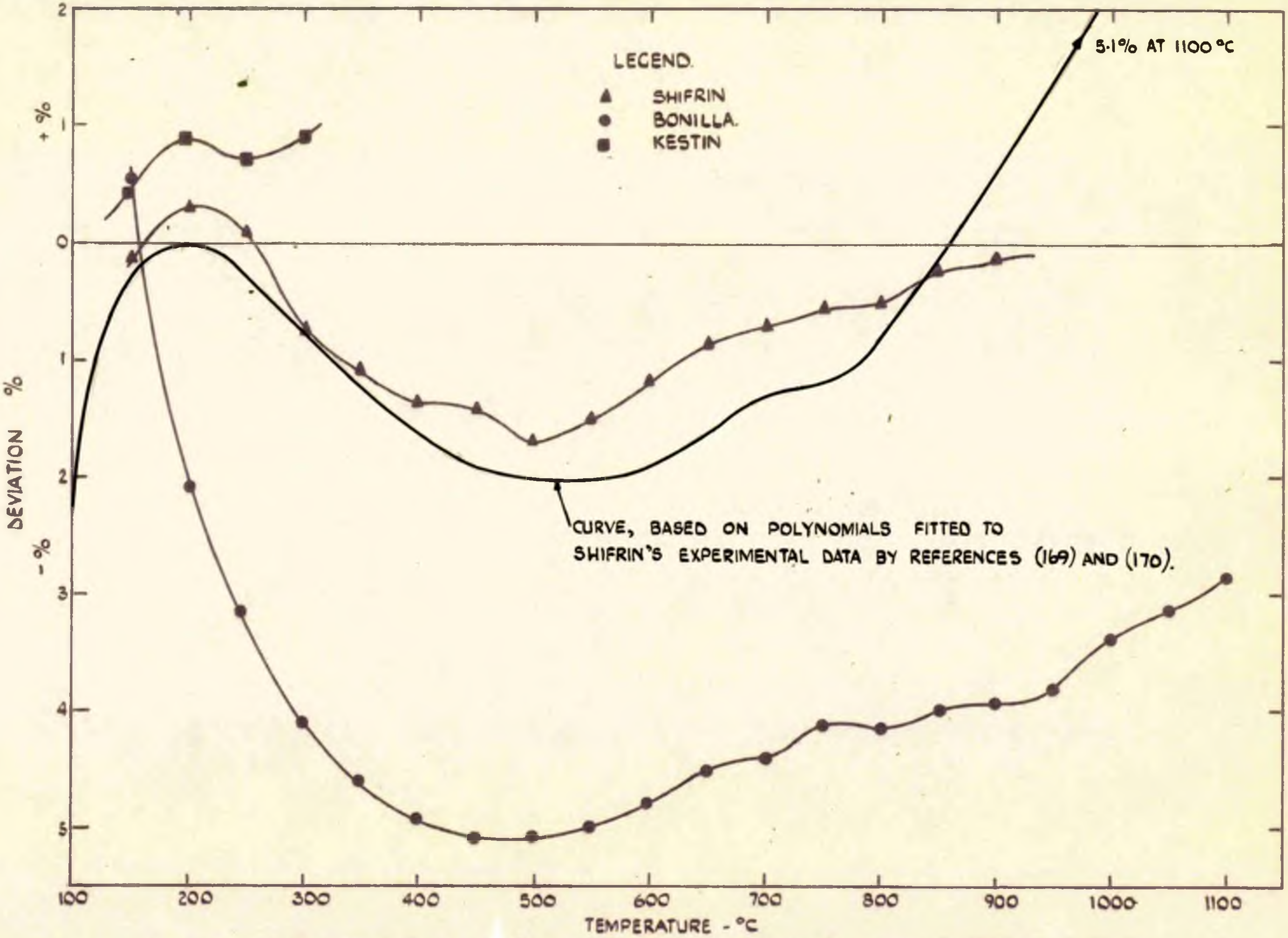


FIG 8·12 DEVIATION OF SHIFRIN'S, BONILLA'S AND KESTIN'S RECOMMENDED VALUES (FOR THE 1 ATM LINE) FROM EQUATION (8.4)

The author does not feel that he is in a position to comment on the reason for the difference between Bonilla's work and the work of the majority of other workers, apart from saying that his work appears to have an inherent error, probably due to a discrepancy in the equation for a spiral capillary. Kestin's work appears to be the most reliable of the workers mentioned.

A graphical determination of the Sutherland constant 'C' using the data given in table (9) was made by means of the equation

$$\eta = \frac{K\,T^{3/2}}{(\,C + T\,)}$$

$$\text{i,e,} \quad T = K\left\{\frac{T^{3/2}}{\eta}\right\} - C$$

The above straight line equation fitted the points quite well between the temperatures of 150°C.and 950°C. Below and above this temperature range the points diverged from the curve. The values for the constants K and C were found to be 25.6×10^{-5} poise/$^{\circ}K^{3/2}$ and 1150°K respectively, which are exactly the same as those put forward by Shifrin.

The author does not agree with Shifrin's conclusion that his curve could quite reliably be extrapolated to 1500°C. without additional high temperature experimental data, since the present work shows a definite increase in the rate of change of the slope of the curve at the higher temperatures, which if extrapolated would be considerably different to the values obtained by extrapolating Shifrin's curve. In actual fact Hilsenrath fitted a 3rd. degree polynomial to Shifrin's experimental data (which was the best fit out of several polynomials) which shows a deviation from the present work at 1100°C of about 5 % which is not exactly indicated by Shifrin's given equation.

It should be noted that at the time of writing this thesis a more detailed analysis of the results was in progress. Several polynomials have been fitted to the experimental data obtained from each individual capillary arrangement, the total number of polynomials being twentythree. The viscosities, using each equation, have been generated at 10°C intervals between 100°C and 1100°C. Examination of this preliminary computation data has shown that in a number of cases (especially when based on the lower temperature or the more sparse data), when extrapolated, showed considerable deviation at the extreme temperatures, although over the measured temperature ranges the polynomials fit very well.

It is intended that a polynomial will be fitted to selected data. After which semi-empirical Sutherland type of equations will be fitted to the experimental data.

These latter computations are being carried out at the Central Electricity Generating Board's Computing Laboratories (170).

CONCLUSIONS

1. The coefficient of the viscosity of steam has been measured at atmospheric pressure over the temperature range 110°C to 1070°C.

2. The results have been compared with the results of previous workers in this field of research and appear to agree quite favourably. A polynomial of the form of

$$\eta = a + bt + ct^2 + dt^3 + et^4 + ft^5$$

and a Sutherland equation of the form of

$$\eta = \frac{KT^{3/2}}{(C + T)}$$

have been fitted to the experimental data.

3. The results confirm Shifrin's assertion that the method of using two capillaries in series to measure the viscosity of vapours and gases at atmospheric pressure can be used with accuracy.

4. Unless a much better precision is obtainable, the method of using two capillaries in series cannot be employed for determining the end correction coefficient 'm' for tubes with any reasonable accuracy.

RECOMMENDATIONS

If the present apparatus is to be used for further work on the measurement of the viscosity of steam and other fluids, it is suggested that several modifications should be made to the system.

The author is of the opinion that for the measurement of the viscosity of steam, the following modifications should be made to improve the accuracy and precision of the results.

1. Increase the ratio of the lengths of the capillaries. The ratio of the length of the two capillaries should be increased, keeping the length of the shorter capillary constant at about 25 cm, and increasing the length of the longer capillary to about 50 cm, thus making the length ratio about 2:1. This would make the differences between the pressure drops across the two capillaries larger than those which were used by the author, which could be more easily measured with a subsequent better precision.

2. Modification to Joints on the Capillaries. Some thought has been given to the matter of the leak, however small it may be, at the joints on the capillaries. It is thought that the leak could be completely stopped by using a specially manufactured capillary having a sudden enlargement of its bore at its ends, making it possible to weld the capillary to the plenum chambers without distortion of the bore at its ends. The thickness of the wall of the tube at the enlargement would have to be thin enough to allow it to be broken without damage to the capillary, and thus make it possible to remove the capillary intact. The author is not certain whether this is a practical proposition, but there has been some discussion with the

manufacturers and it appears that it is not completely out of the question.

3. Improve the Measurement of the Pressure Drop. This could be achieved by using a capacitive or inductive probe method of indicating the position of the mercury surface in the mercury-in-glass manometer. Since the completion of the experimental work, the author has formed the opinion that a capacitive probe method (a probe attached to a micrometer head) would have proved more successful than the optical method of detecting the mercury surface (the method which was used). The capacitive method has the advantages of the visual method without having to rely on the experimenter's ability to use the telescopes.

4. Improvement of the Control and Stability of the Boiler Supply Pressure. This could be achieved by increasing the pressure in the boiler and throttling the steam to the required pressure at the entrance to the superheater and pressure stabiliser. It had been observed that the amplitude of the pressure fluctuations in the boiler was approximately constant whatever the pressure. Therefore, if higher boiler pressures were to be used, the fluctuations in pressure would have less effect. A throttle valve could be fitted into the supply line immediately after the boiler, and by adjusting this valve, the boiler pressure could be kept constant, whatever the mass flow rate through the viscometer. For this modification to be effective the boiler pressure would have to be at least 1 atmosphere gauge, thus requiring modification of the ground glass joints on the boiler, since when boiler pressures of about 0.75 atmosphere

gauge were used, the joints tended to part, even though they had been tightly wired together.

5. **Suggestion for an alternative method of Measuring the Mass Flow Rate.** Since the method of measuring the mass flow rate appeared to be the main cause of the experimental scatter, it might be considered advisable to check more thoroughly a method of measuring the volume flow rate of the steam. Although this would prove quite difficult, if it could be achieved it would certainly improve the precision of the measurement of the mass flow rate.

Modifications required if the Apparatus is to be used for the Measurement of the Viscosity of Gases.

As it is proposed to use the apparatus for the measurement of the viscosity of gases, the system will require some modifications. There does not appear to be any difficulty in supplying the gas to the viscometer at constant pressure. Super pure high pressure gas can be obtained from a gas bottle and then throttled to the required pressure, there being several methods of stabilising the pressure of the gas issuing from the throttle valve.

There are a number of manometers, having a good accuracy and sensitivity, which can be used for gases, since unlike for steam, the manometers do not require to be heated. A manometer using water or some similar low density measuring fluid (such as a Betz manometer) could be used with much better accuracy and sensitivity than the mercury-in-glass type.

Measurement of the Mass Flow Rate

Some work has already been done on the development of a volume flow rate meter for gases. This meter consists of a horizontal 'U' tube, about one metre long, having its ends connected by a smaller vertical 'U' tube (of a larger bore tubing). This smaller 'U' tube is filled with mercury and acts as a reservoir. The gas exhausting from the viscometer enters the meter by a tube at one end of the main loop and pushes a pellet of mercury, which has been displaced from the reservoir, round the loop to the other side of the reservoir, where the entrapped gas escapes. The mercury pellet, on falling into the 'U' tube, displaces a further pellet on the other side of the 'U' tube into the main tube, and the process is repeated. The rate of progress of the pellet along a measured length of the main tube indicates the mass flow rate. This type of meter has the advantage that it can be continuously used. Initially a flat spiral was used instead of the loop for the main tube, but this arrangement could not be manufactured with sufficient precision, whereas the loop arrangement could.

Some success has been achieved with this type of meter, using capacitive 'pick-ups' to indicate the period of time the pellet took to travel along the measured length of tube. Unfortunately slight undulations in the tube caused small pellets to jerk along. The use of very large pellets partially overcomes this problem.

Improvement of the Temperature Control.

A further improvement to the apparatus, whether it be used for gas or steam, would be to introduce platinum resistance control thermometers into the two furnaces, and thereby improve the temperature control and stability of the system.

Appendix (1)

Ground Conical Joint Tests

Several test rigs were set up, the most suitable being as shown in figure (Al.1). A standard ground conical joint attached to the end of a glass tube was connected by a rubber tube to a mercury trap. Attached to the other side of the trap was a precision bore drop tube which had a water jacket. A thermometer, graduated to 0.1°C, indicated the water jacket temperature. The assembled joint, (one of a standard batch) which had been thoroughly cleaned prior to assembly, was put into the furnace and conditions allowed to settle down; the furnace having been set for a given temperature prior to the insertion of the joint. The drop tube, which was in a horizontal position, had a mercury pellet injected into it and was then slowly raised into the vertical position. After the mercury pellet had initially compressed the air in the system, it progressed down the drop tube with what appeared to be a constant velocity. The rate of descent was obtained using a cathetometer and a stopwatch. Several of the joints were checked more than once after they had been dismantled, cleaned and re-assembled.

Graphs of the position of the mercury pellet (which is proportional to the mass of air leaking from the system through the joint) against time are plotted, (see figures (Al.2) and (Al.3)). Although the drop tube temperature remained constant, unfortunately the room temperature did not. A thermometer was strapped to the tubing between the furnace and the water jacket, the temperatures registered by this thermometer are shown on the above mentioned graphs. The variations of the room temperature may have been the cause of the apparent unsteady rate of fall of the pellet.

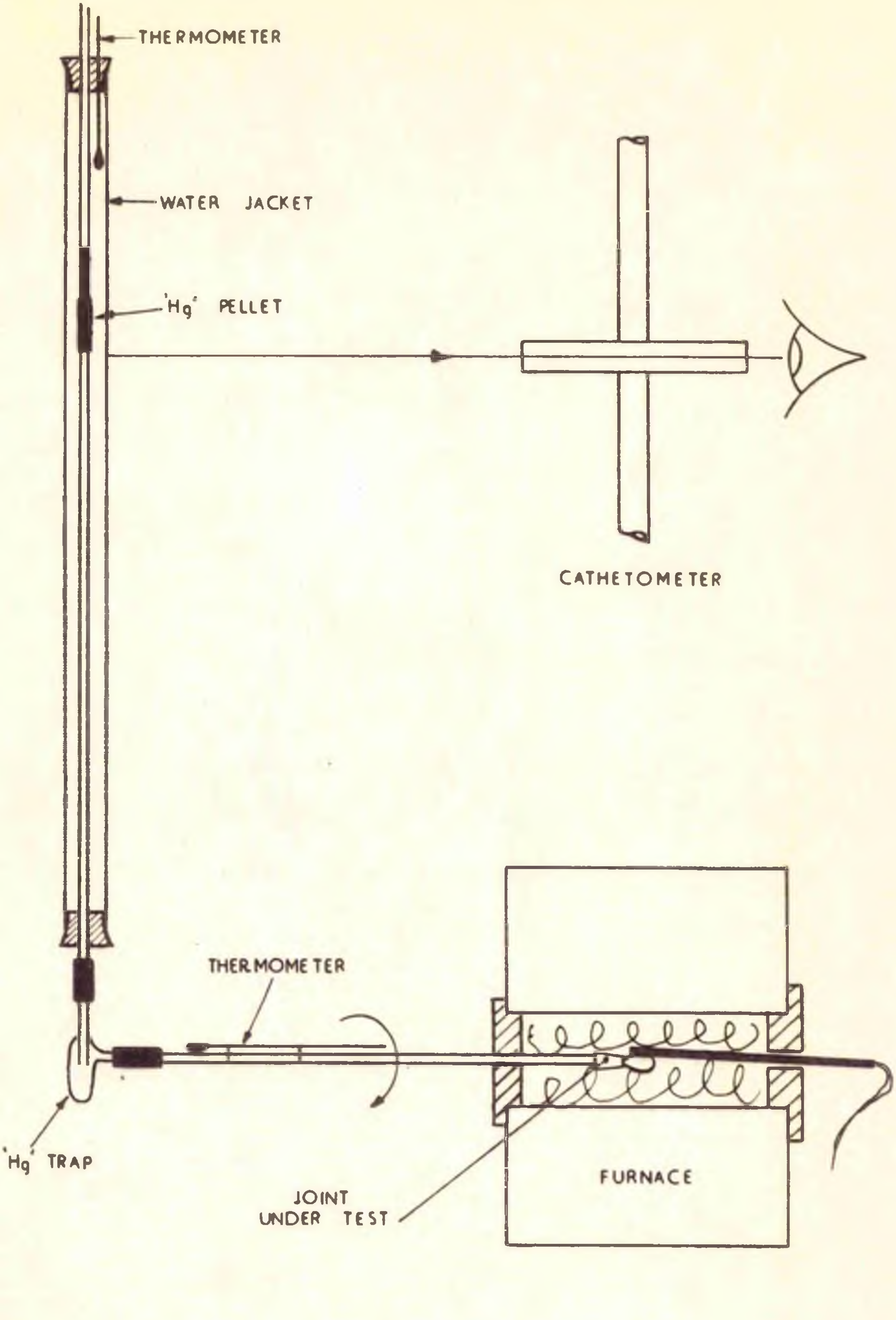


FIG AI. I RIG FOR TESTING CONE JOINTS

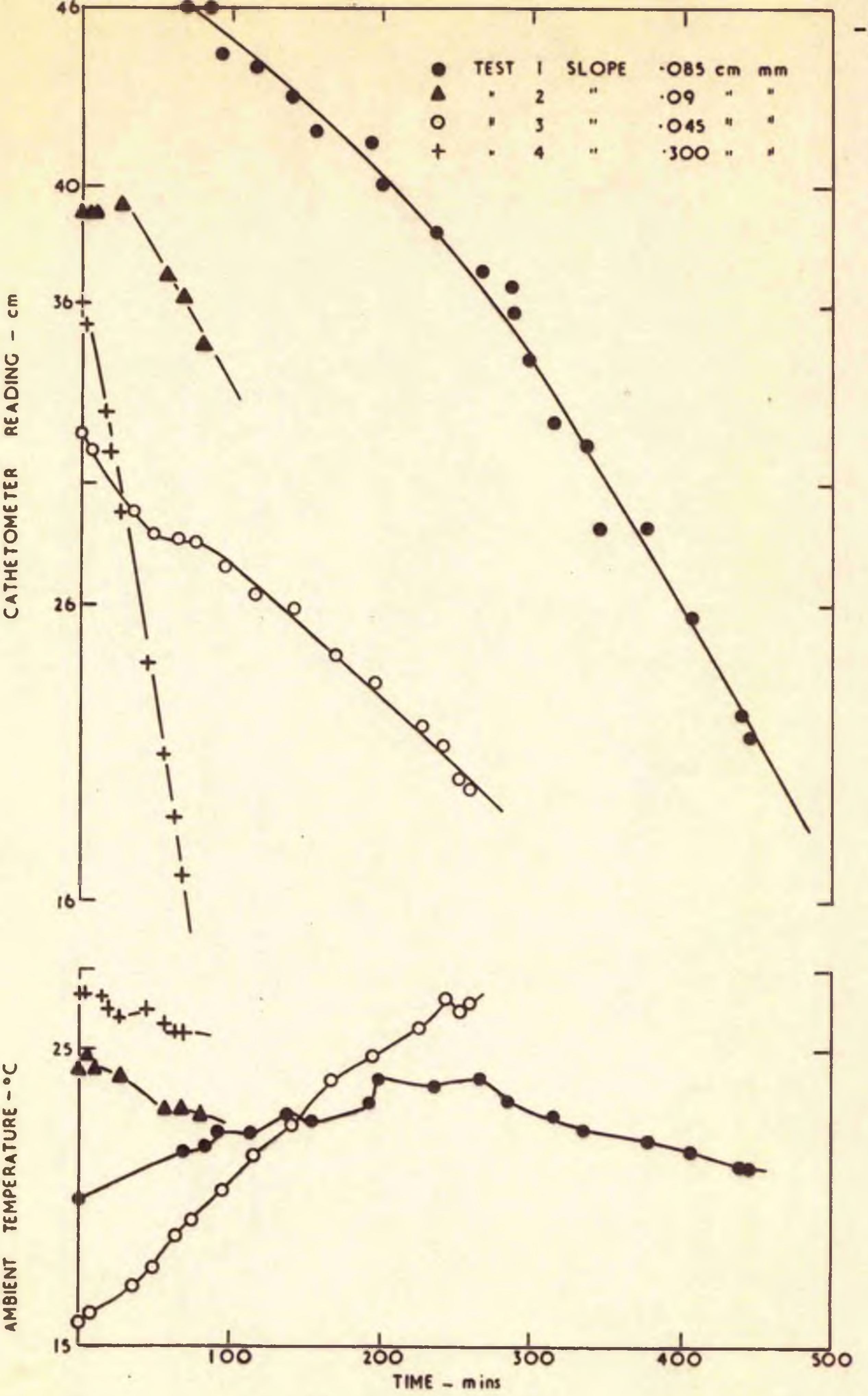

● TEST 1 SLOPE ·085 cm mm
▲ " 2 " ·09 " "
○ " 3 " ·045 " "
+ " 4 " ·300 " "
46
40
36
26
16
CATHETOMETER READING – cm
25
15
AMBIENT TEMPERATURE – °C
100
200
300
400
500
TIME – mins

FIG A1. 2

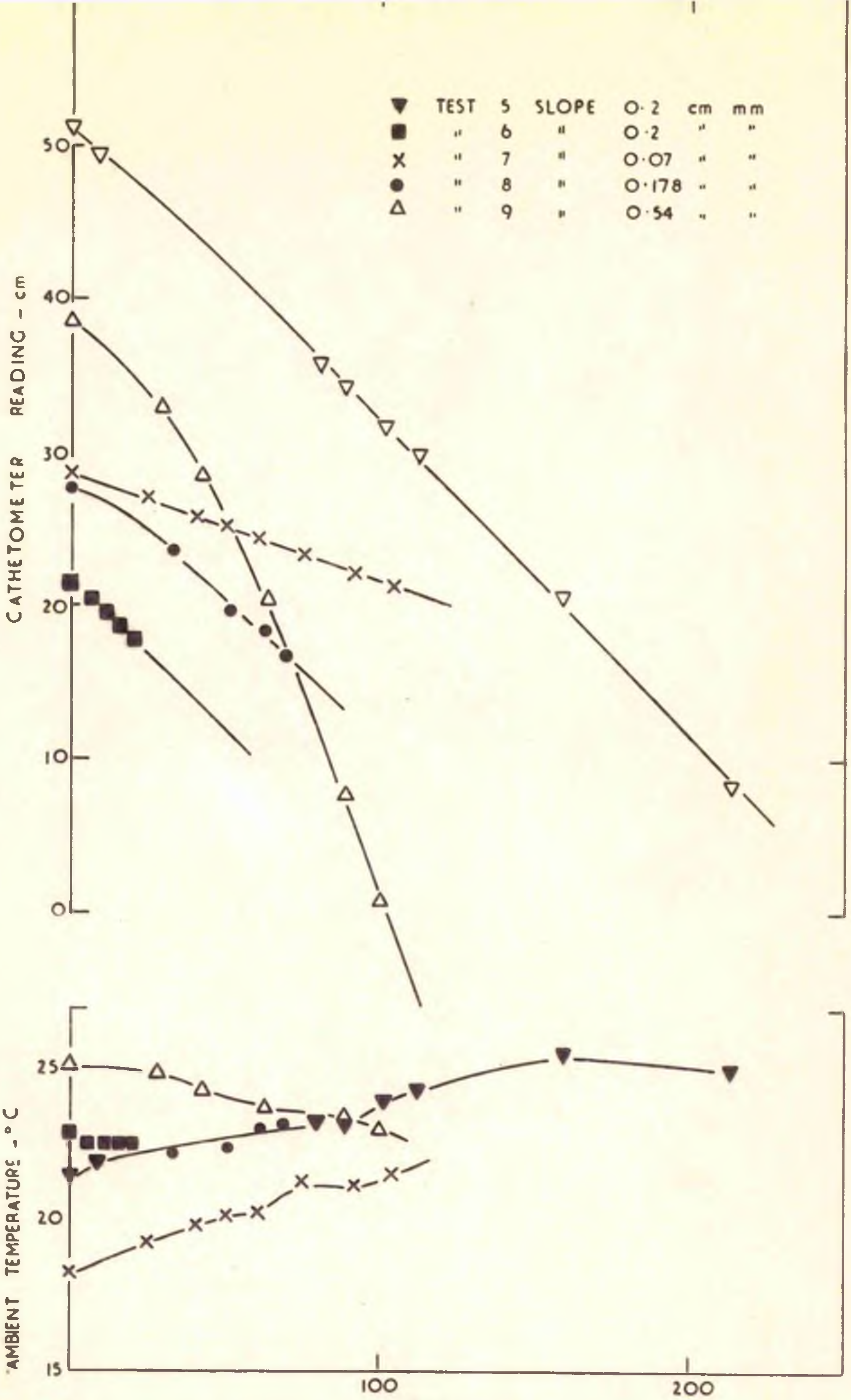

▼ TEST 5 SLOPE 0·2 cm mm
■ " 6 " 0·2 " "
× " 7 " 0·07 " "
● " 8 " 0·178 " "
△ " 9 " 0·54 " "
CATHETOMETER READING – cm
50
40
30
20
10
0
AMBIENT TEMPERATURE – °C
25
20
15
100
200
TIME – mins

FIG A1. 3

Very little can be concluded from the graph on figure (Al.4), apart from the fact that the leakage varied with each assembly and with individual joints. Although no tests were carried out on a given assembly to check the effect of varying the pressure drop across the joint, the author considers that it would be reasonable, at least for this work, to assume that the leakage rate would be proportional to the pressure drop across the joint. Since the joint is akin to a porous plug, it would also be reasonable to assume that the leakage rate is proportional to the viscosity of the fluid escaping through the joint.

Calculation of the maximum leakage rate observed

The drop tube diameter, which was found using a long mercury pellet in the section to be used equalled 2.41 mm. at 20°C.
Maximum leakage rate at 508°C. was 0.0246 cm/min, per cm Hg.,
ΔP = 22.1 cm Hg. i.e. the slope = 0.543 cm/min, the mean drop tube temperature = 24°C, barometric pressure = 755 mm Hg.
Therefore the mass leakage rate =

$$\frac{\pi \; (\; 2.41 \;)^2 \; 10^{-2} \; 0.543 \quad \rho_a}{4} \quad = \quad 0.63 \times 10^{-6} \quad \text{g/sec.}$$

It was concluded that this leakage rate was sufficiently small to permit the use of ground cone joints in the proposed viscometer, provided that the joints were thoroughly cleaned and carefully assembled.

Temperature tests

Several quartz joints, which had been thoroughly cleaned and fitted tightly together, were heated up to temperatures below 1100°C. After cooling and soaking in penetrating oil, the majority of the joints

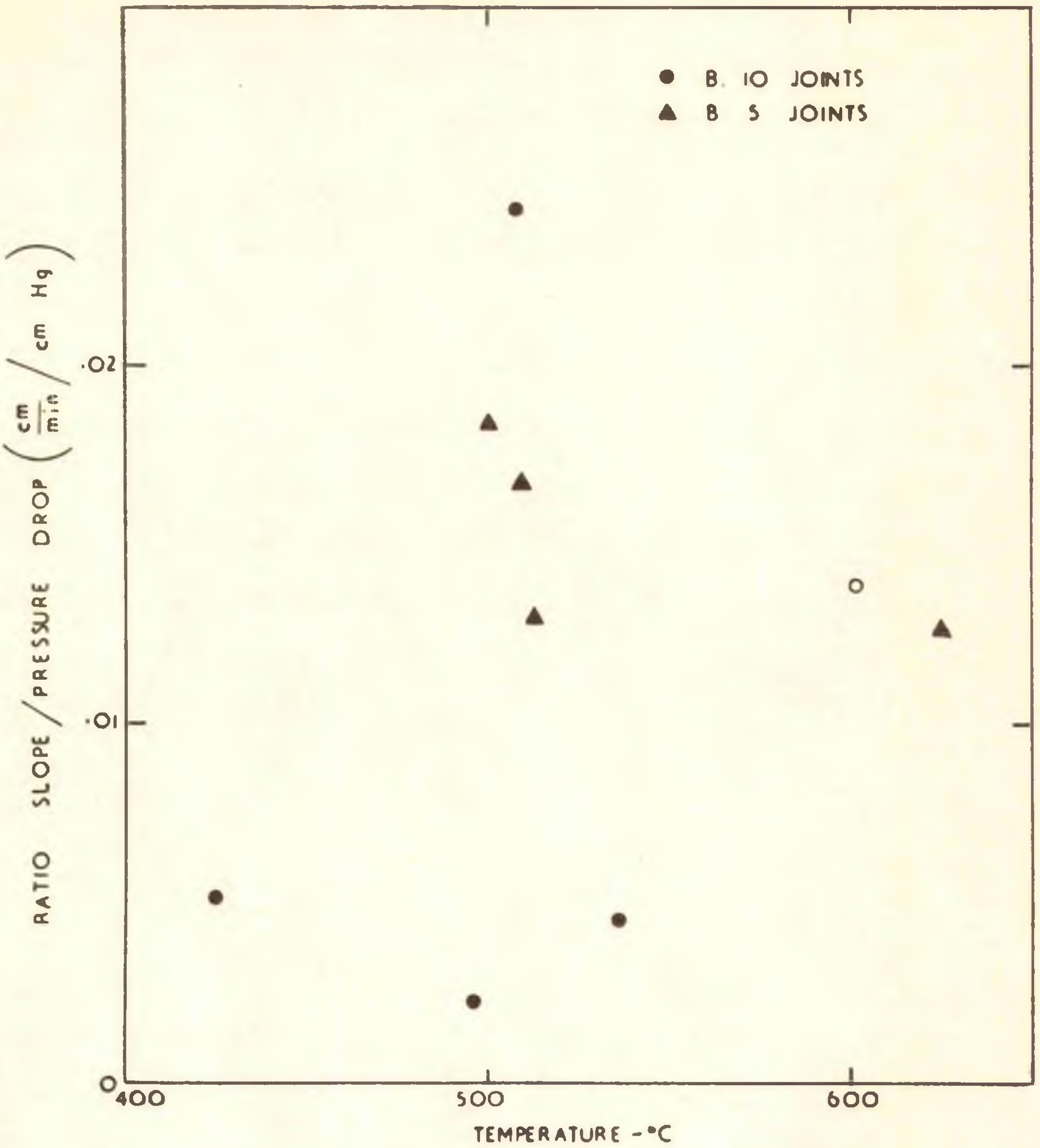


FIG A1. 4 $\frac{\text{SLOPE}}{\text{AP}} \alpha \left(\frac{\text{MASS FLOW}}{\text{AP}}\right) \sim$ TEMPERATURE could be taken apart without breakage, unfortunately some of the joints had to be broken before they could be separated. Two joints were heated up to temperatures in the region of 1400°C., but both of these joints had to be broken to separate them. The joints apperaed to have fused together, which is only to be expected at these high temperatures. A point that was worthwhile noting was that one of the quartz tubes touched the firebricks of the furnace wall and diffused into them, producing a low melting temperature silicon slag. This was an important point to note, since it showed that care should be taken when selecting the materials of the furnaces for the viscometer and the superheater.

From the observations regarding the leak and temperature properties of conical joints, it was decided that it would be quite allowable to use conical joints.

Appendix (2)

Plenum Chamber Tests

The entrance plenum chamber to a capillary is quite important, since it is necessary to establish at the entrance to the capillary, a low and uniform velocity distribution. The obvious solution is to have a large entrance reservoir, with an entrance remote from the exit. This is not practicable with the small space available within the viscometer body, consequently some attention was given to the design of the entrance plenum chambers.

Considering the space that was available, it was decided to assess the performance of a tubular chamber having a 2 cm. bore with a single coarse grade sintered filter across its bore. With water as the working fluid an attempt was made to establish conditions in the chamber similar to those expected in the actual apparatus. To achieve this, the Reynolds numbers in the capillary were made the same as those expected in the apparatus and the ratio of the diameters of the plenum chamber and the capillary were also made approximately the same.

i.e. $$Qw = Qs \left\{ \eta_w / \eta_s \right\} \left\{ d_1 / d_2 \right\} \quad \text{..................A2.1}$$

where Qw and Qs are the mass flow rates for the water and the steam respectively, η_w and η_s are the viscosities of the water and the steam and d_1 and d_2 are the diameters of the capillary tubes using water and steam respectively. From the basic Poiseuille equation for flow, the pressure head required upstream of the capillary was

$$h_w = \frac{128\, \eta_w\, l_w\, Q_w\, (\eta_w/\eta_s)}{\pi\, d^4\, g\, (\rho_w)^2} \quad \text{.......................... A2.2}$$

where l_w is the length of the capillary, ρ_w is the density of the water and $d_1 = d_2 = d$.

A constant head tank was used to control the inlet pressure head, and a dye was used to indicate the flow pattern in the system. Initially potassium permanganate was used as the tracer fluid. Two small jets projecting at right angles to the main stream flow, one on each side of the filter, were initially used to inject the fluid into the main stream. The jet supply tubes were made as small as possible, so as not to disturb the natural flow pattern, and inserted from the extreme ends of the chamber (see figure (A2.1a)).

Under these conditions it was observed that the upstream jet was not effective. That is, it was not possible to see clearly defined stream lines. However, it was possible to see that the jet tubes tended to produce irregularities in the flow patterns. It was also observed that the indicating fluid tended to colour the slower moving fluid and that since it was denser than the water, it fell to the bottom of the chamber.

For the convenience of construction, a pre-plenum chamber was put into the supply tube, which made it possible to put an injection jet into the inlet stream. A continuous stream of dye (a less dense dye, "fluorescene", was used in preference to potassium permanganate) was then injected into the inlet stream. The flow was then as shown in the figure (A2.1b). There was a central faster moving core, which continued to within about 3 mm. of the face of the filter, at this point the flow appeared to be radial and turbulent. Down stream of the filter there was still a slight coring, with the outer slower moving fluid converging to the capillary entrance. It was considered

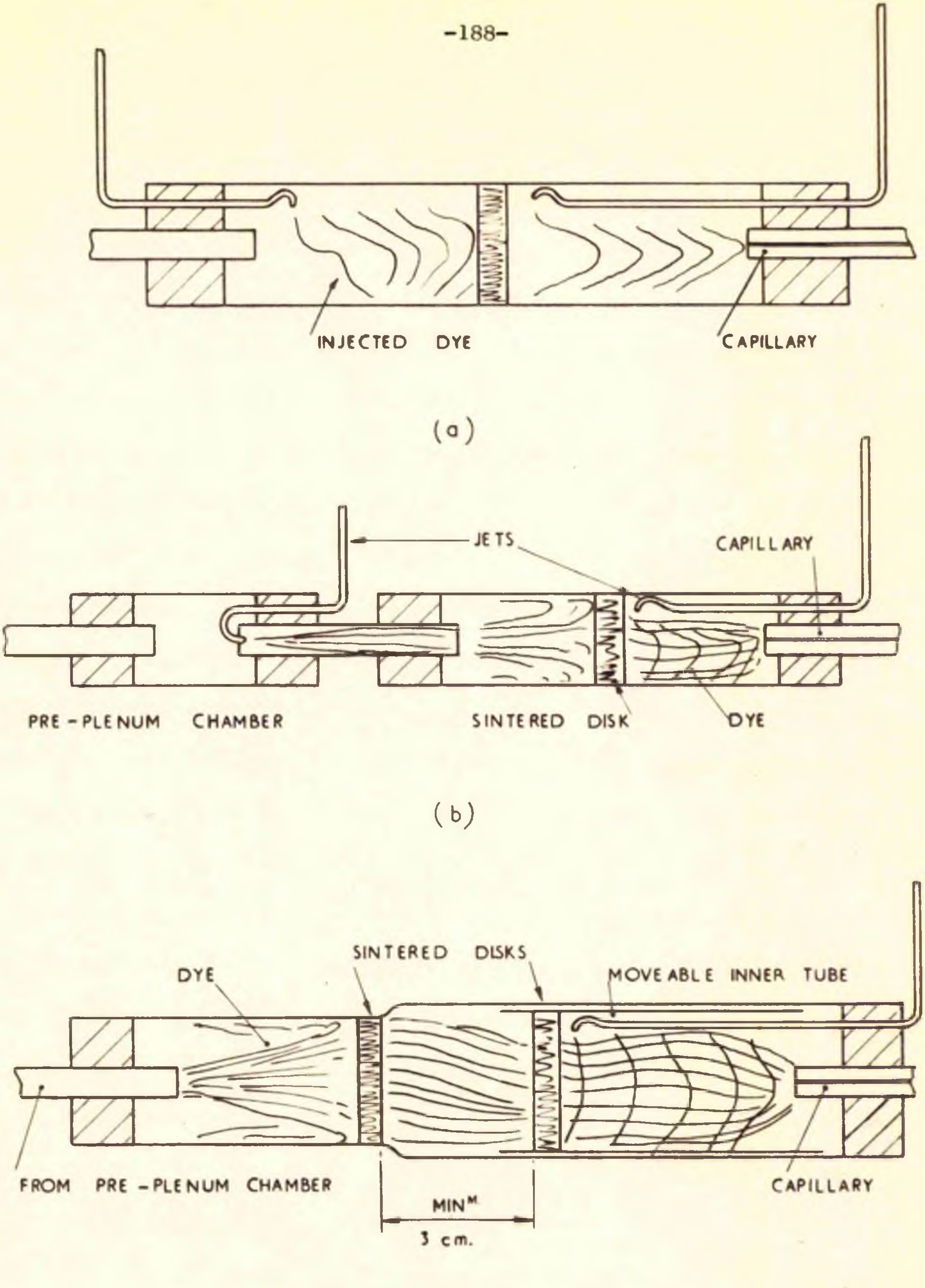


FIG A2.1 FLOW PATTERNS IN ENTRANCE PLENUM CHAMBERS that the downstream conditions could be improved and therefore a two filter system was made in which the second filter was movable in an axial direction, as shown in figure (A2.1e).

As before, the fluid upstream of the filter tended to produce a core of faster moving fluid, with radial turbulence at the filter face. The larger the distance between the filter face and the inlet part, the more pronounced the divergence of the fluid flow from a central coring effect, which was only to be expected. Between the filters the flow was random with only a slight coring. After the second filter, the velocity distribution was very nearly uniform across the tube, except in the vicinity of the jets. The dye indicated that, after the second filter the stream lines tended to converge towards the capillary entrance. It was evident that the distance between the outlet and the second filter had little effect on the flow pattern in that region for the mass flows considered, provided that the distance was greater than 25 mm.

The distance between the filters was varied and observations were made. The apparent minimum distance between the filters, for which they had any reasonable effect on the flow pattern was about 20 mm. When the distance was less than 20 mm, the filters had little or no effect and acted as if there was only one filter.

The velocity of the fluid flow before, between and after the filters was observed for a given mass flow rate. This was done by injecting a small thread of dye at right angles to the axis of flow and timing its progress between measured sections. Several checks were made on the final configuration and the average velocities observed were compared with the theoretical average velocities. Using a 0.8 mm. bore capillary having a length of 7.3 mm. and assuming that the expected mass flow of the steam at 100°C will be 0.1 g/min, for dynamic similarity, the required water flow rate is 0.146 g/sec. The required upstream head will be 11.26 cm. The actual flow rate was about 0.15 g/sec. Therefore the average velocities in the capillary and the plenum chamber were 29.8 cm/sec. and 0.039 cm/sec. respectively: the velocity ratio being 758.

The mean velocities from observations were about 2 cm/sec. and 0.05 cm/sec. before and between the filters respectively. It was difficult to obtain the velocities after the second filter, since the velocity was slow enough to allow the dye to diffuse into the surrounding water before it had passed any measureable distance.

The pressure drop across the filters was also measured for different flow rates of water at 20°C. A graph of ΔP against mass flow rate was plotted from these observations, (see figure (A2.2)). Assuming that the pressure drop is proportional to the product of the mass flow rate and the viscosity of the fluid, the pressure drop across the filters when steam is flowing can be estimated from this graph.

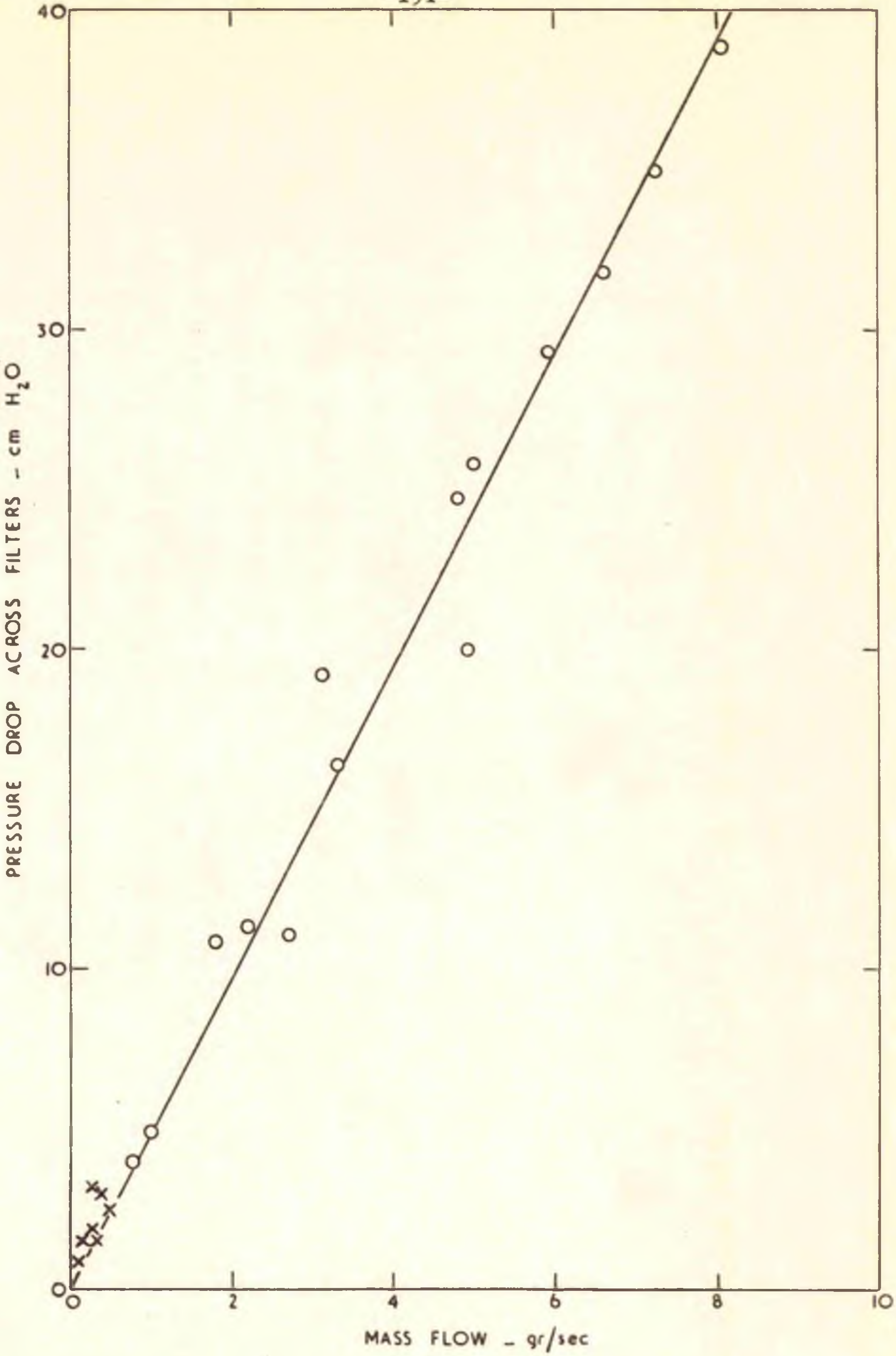
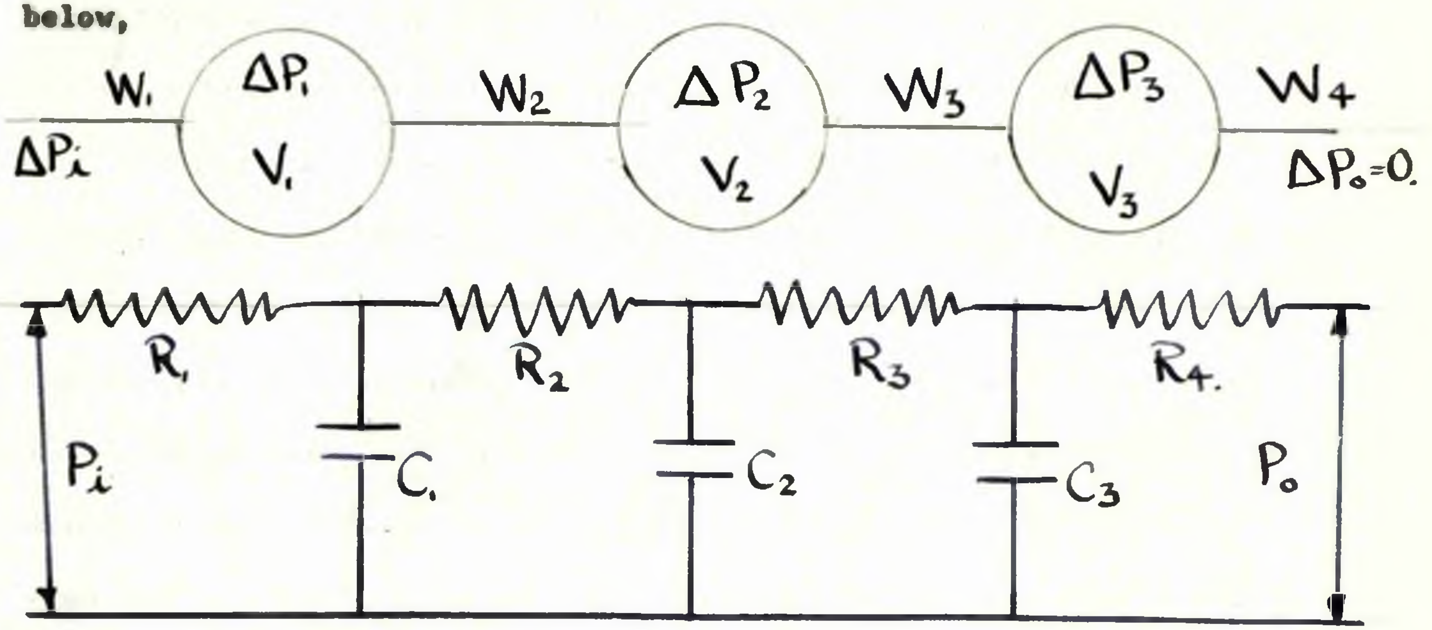


FIG A2. 2 PRESSURE DROP ACROSS FILTERS ~ MASS FLOW
(FILTERS 2cm APART)

Appendix (3)

Superheater Design

The design is based on a pressure stabiliser put forward by Novak (158) for gas chromotography apparatus. Theoretically the stabiliser can be compared with an analagous electrical, capacitive-resistive circuit. The stabiliser is made up of several reservoirs inter-connected by capillary tubes. The electrical analogy is shown below,



if $V_1 = V_2 = V_3 = V$ = the volumes of the reservoirs, and

$R_1 = R_2 = R_3 = R$ = the resistances of the capillaries.

Using the electrical analogy, and assuming that the overpressure at the input is sinusoidal, it can be proved that the ratio of the overpressure at input to the overpressure at the outlet can be expressed by the equation,

$$\frac{Po}{Pi} = \left\{ \frac{R\,T}{w\,WV} \right\}^{n} = \left\{ \frac{R\,T}{2xf\;W\;V} \right\}^{n} \quad \ldots\ldots\ldots\ldots\ldots \text{A3.1}$$

where W is the ratio of the difference between the overpressure at the input and that at the output of a damping unit, having a change of molar flow rate of the gas medium of d(n)/d(t)

i.e.

$$W = \frac{d(\ \Delta Pi - \Delta Po\ )}{d\ \left[\ d(n)/d(t)\ \right]} = \frac{d(\ \Delta Pi - \Delta Po\ )}{d\ \left[\ d(V)/d(t)\ \right]} \ldots\ldots\ldots\ldots \text{A3.2}$$

From the above equations we can get,

$$\frac{d(QPo)}{d(\Delta Pi)} = \frac{\left[\ r^4\ \ Pm\ \right]}{\left[\ 16\ f\ \eta\ v\ l\ \right]} \ldots\ldots\ldots\ldots\ldots\ldots \text{A3.3}$$

where r is the radius of the capillaries, η is the viscosity of the working fluid, f is the frequency of the overpressure oscillations, l is the length of the connecting capillaries and Pm the mean pressure.

But $Pi = Po + \Delta Pi + d(Pi)$ and $Pe = Po + \Delta Po + d(Po)$

Therefore $2Pm = \left[\ Po + \Delta Pi + d(Pi) + Po + \Delta Po + d(Po)\ \right]$

$$= \left[\ \Delta Pi + \Delta Po\ \right] + 2Po + \left[\ d(Pi) + d(Po)\ \right] .$$

It must be appreciated that there is some phase change between the input and the output overpressures, and that d(Pi) is not necessarily in phase with d(Po) and could quite conceivably be anti-phase.

Since it may be assumed that d(Pi) and d(Po) are quite small for the system to be used, equation (A3.3) becomes

$$Ra = \frac{d(Po)}{d(Pi)} = \left\{ \frac{r^4(\ \Delta Pi + \Delta Po + 2Po\ )}{32\ f\ \eta\ v\ l} \right\}^n \ldots\ldots\ldots\ldots\ldots\ldots \text{A3.4}$$

Using three expansions of equal volume and equal capillary resistances, equation (A3.4) may be re-arranged into the form

$$V.l = \left\{ \frac{\left[\ r^4(\ \Delta Pi + \Delta Po + 2Po\ )\ \right]}{32\ f\ \eta^3\ (Ra)^{1/3}} \right\} \ldots\ldots\ldots\ldots\ldots\ldots \text{A.3.5}$$

The optimum conditions for the design of the superheater will be when the temperature in the superheater is about 100°C. This is because the viscosity will be a minimum, while the mass flow rate through the viscometer will be a maximum, thus making the boiler pressure a maximum. Therefore the pressure stabiliser will be least effective at 100°C.

During the boiler tests it was observed that the frequency of the pressure fluctuations was about 1 cycle per second and that the amplitude of the overpressure was about 0.25 mm Hg (gauge). The maximum pressure that can be expected at the entrance to the viscometer (that is the pressure at the exit from the stabiliser), is about 8 cm Hg (gauge). A suitable diameter of 0.35 mm, for the connecting capillaries was obtained by trial and error. It was decided that the ratio of the overpressure at the output to that at the input should be as small as possible and a nominal value of 1 % was taken for this ratio.

At 100°C the viscosity of steam is about $1.25.10^{-4}$ poise, and taking the extreme case of the boiler pressure being 50 cm Hg, then,

$$V.1 = \frac{\left[(0.175)^4 \; 10^{-4}.210.13.5460. \; g \right]}{512 \; 1.25 \; 10^{-4} \; (\; 10^{-3} \;)^{1/3}} = 654 \text{ cm}^4 \quad ..A3.6$$

Letting the length of the capillary be 25 mm, the volume required for each reservoir is 260 cm^3. It was decided to make the reservoirs cylindrical and to limit the maximum outside diameter to 4.5 cm, the internal diameter of the furnace tube. Therefore the required length for each reservoir must be 16.3 cm. For the convenience of manufacture the length of each reservoir was made 13 cm. That is, the produce V x 1 is 207 cm^3, and the ratio d(Po)/d(Pi) is approximately 0.02. With an overpressure amplitude of 0.25 mm at the entrance to the stabiliser the exit overpressure amplitude should be 0.005 mm. Under experimental conditions an amplitude of this magnitude should have very little effect on the stability of the flow.

The dimensions selected reduce the damping effect of the stabiliser at the higher steam mass flow rates, but this should not unduly affect the stability of the conditions in the viscometer. Figure (6.5) gives a drawing of the pressure stabiliser and superheater.

Appendix (4)

Leak Tests

Immediately after the assembly of the viscometer cell, it was heated and evacuated to remove any fluids left in the tubes. Preliminary leak tests were then carried out to check the sealing of the joints at the ends of the capillaries. This was done by observing the rate of fall of a mercury pellet in a 2 mm precision bore drop tube, which was connected to the viscometer cell. The drop tube arrangement and the procedure was basically the same as that used for the joint tests, see appendix (1) and figure (A.1.1). If the preliminary leak tests showed that there was no appreciable leak at these joints, the viscometer was put into its furnace and connected up to the manometer. About 50 % of the initial assemblies proved satisfactory. On some occasions the joints had to be lapped together with a fine paste before an assembly was acceptable.

The main leak tests were carried out after the viscometer cell had been connected up to the manometer; the boiler circuit was blanked off, since it did not matter if the joints in this part of the circuit leaked a little. Unfortunately the compressibility of the air in the combined volumes of the viscometer and the manometer precluded the use of the above mentioned drop tube method for measuring the leaks. This was because the maximum drop tube diameter that can be used with a mercury pellet is about 3 to 4 mm and if a pellet of three or more centimetres length was used with this size of bore drop tube, it either tended to break up or required a drop tube length of at least 250 cm before compression of the air had been accomplished.

An alternative method was employed to check the leaks in the assembled apparatus. This method was to shut off two of the manometer pressure lines and vent their respective columns to atmosphere, connecting the central (third column) to the apparatus. The apparatus was given a small internal pressure, which was larger than that expected under working conditions, displacing the central mercury level. The rate at which the mercury level returned to its equilibrium position is a measure of the leaking rate of the air from the system. Graphs of the rate of the rise of the mercury level for a given initial pressure head are plotted on figures (A4.1) to (A4.8). It was felt that these tests were very reasonable, since at least half the number of the joints were subjected to pressures twice or more times their expected working values (the downstream joints being subjected to as much as 300 % of their working pressure).

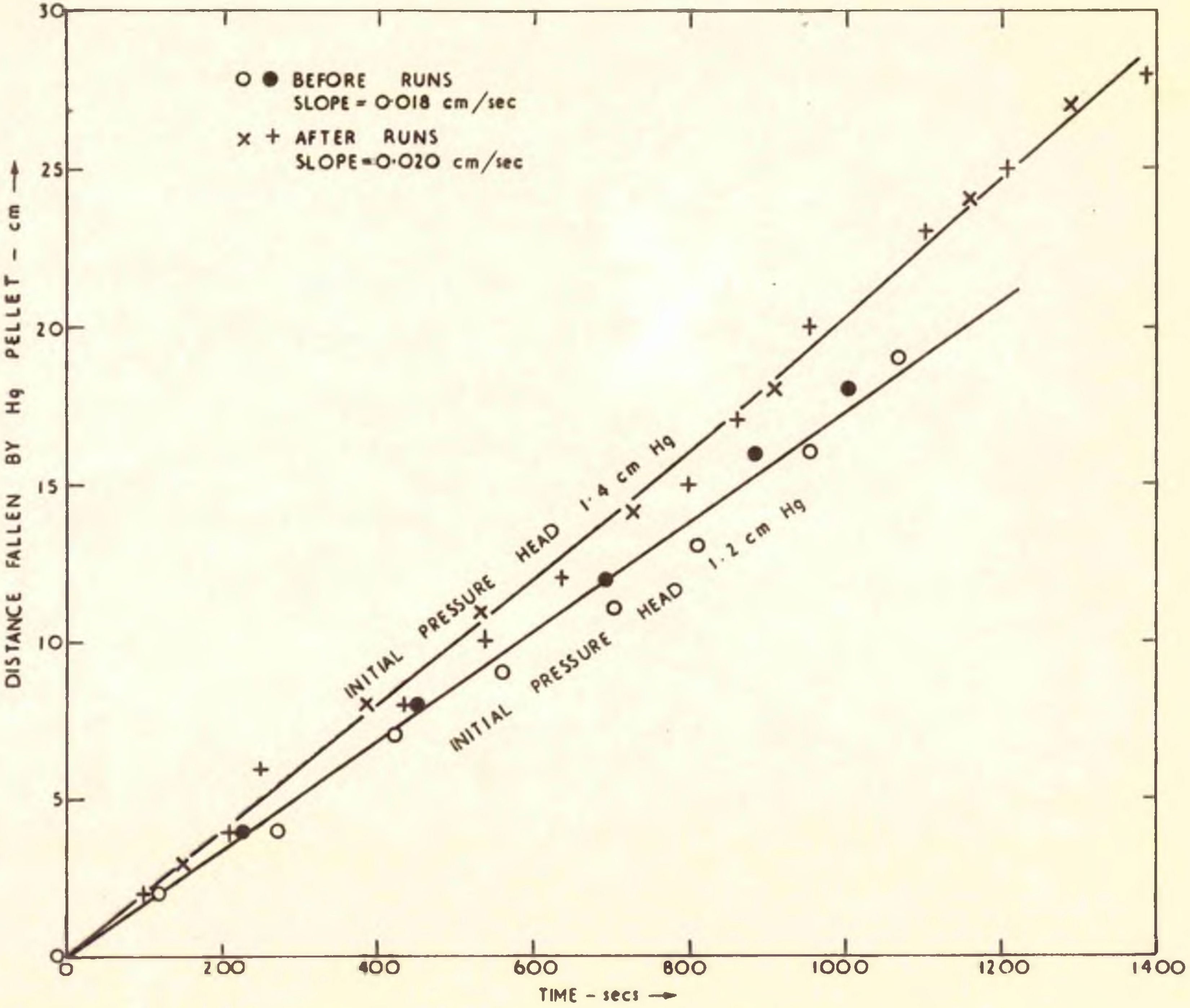


FIG A4.1 LEAK TESTS FOR SINGLE CAPILLARY SYSTEM, USING CAPILLARY N°. 1

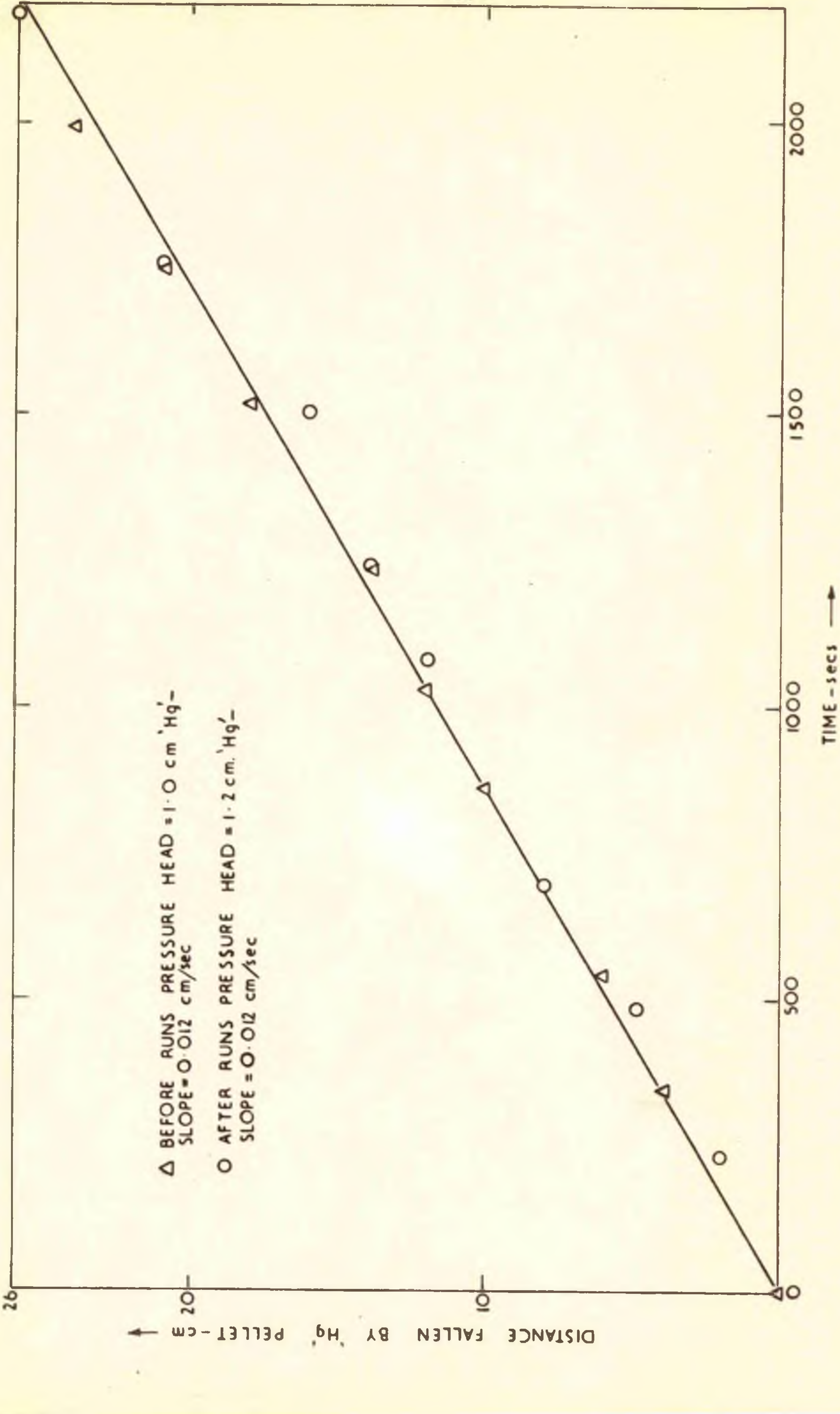


FIG A4.2 LEAK TESTS FOR SINGLE CAPILLARY SYSTEM, USING CAPILLARY N° 2

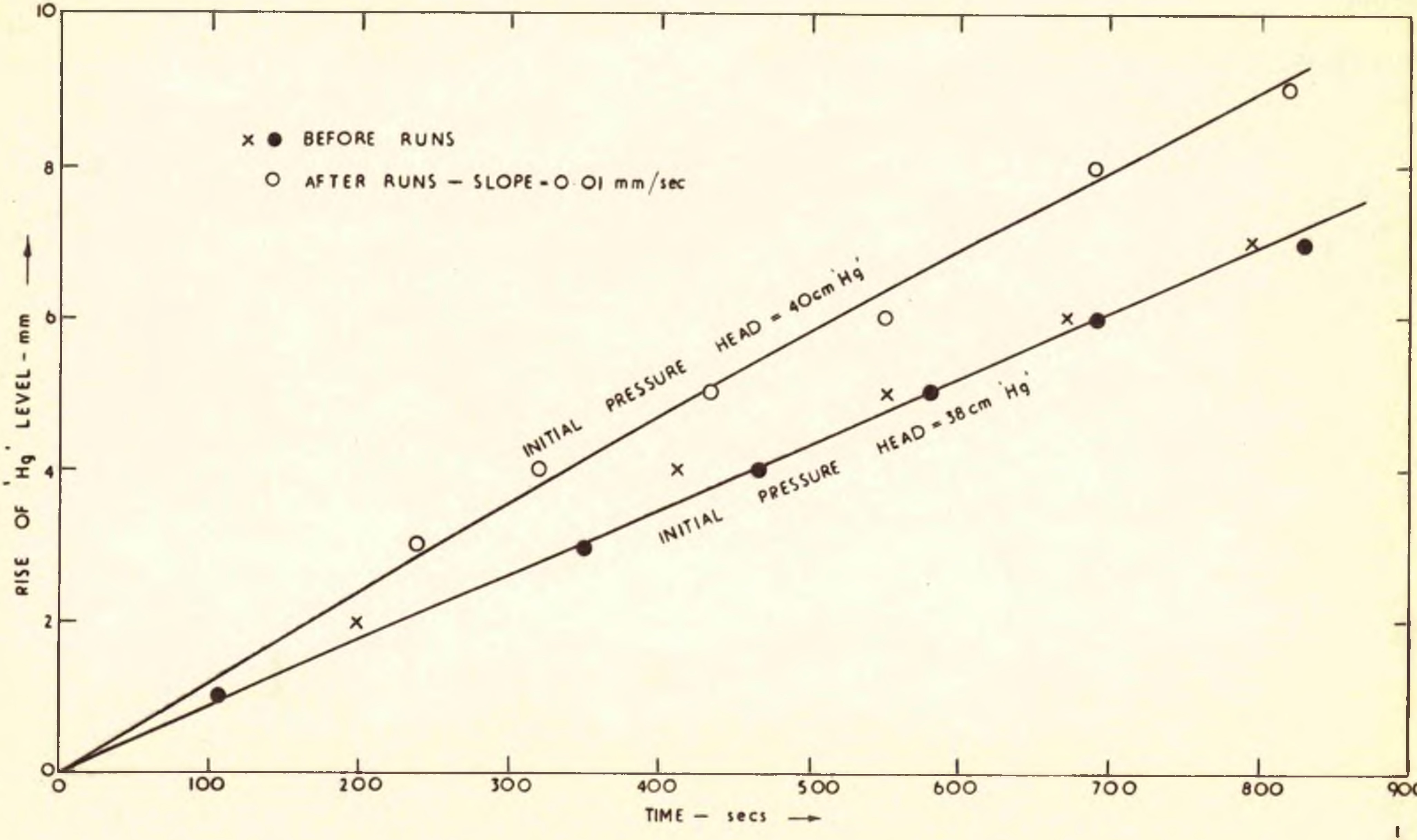


FIG A4.3 LEAK TESTS FOR SERIES CAPILLARY SYSTEM, USING CAPILLARIES N$^{os.}$ 3a AND 4b

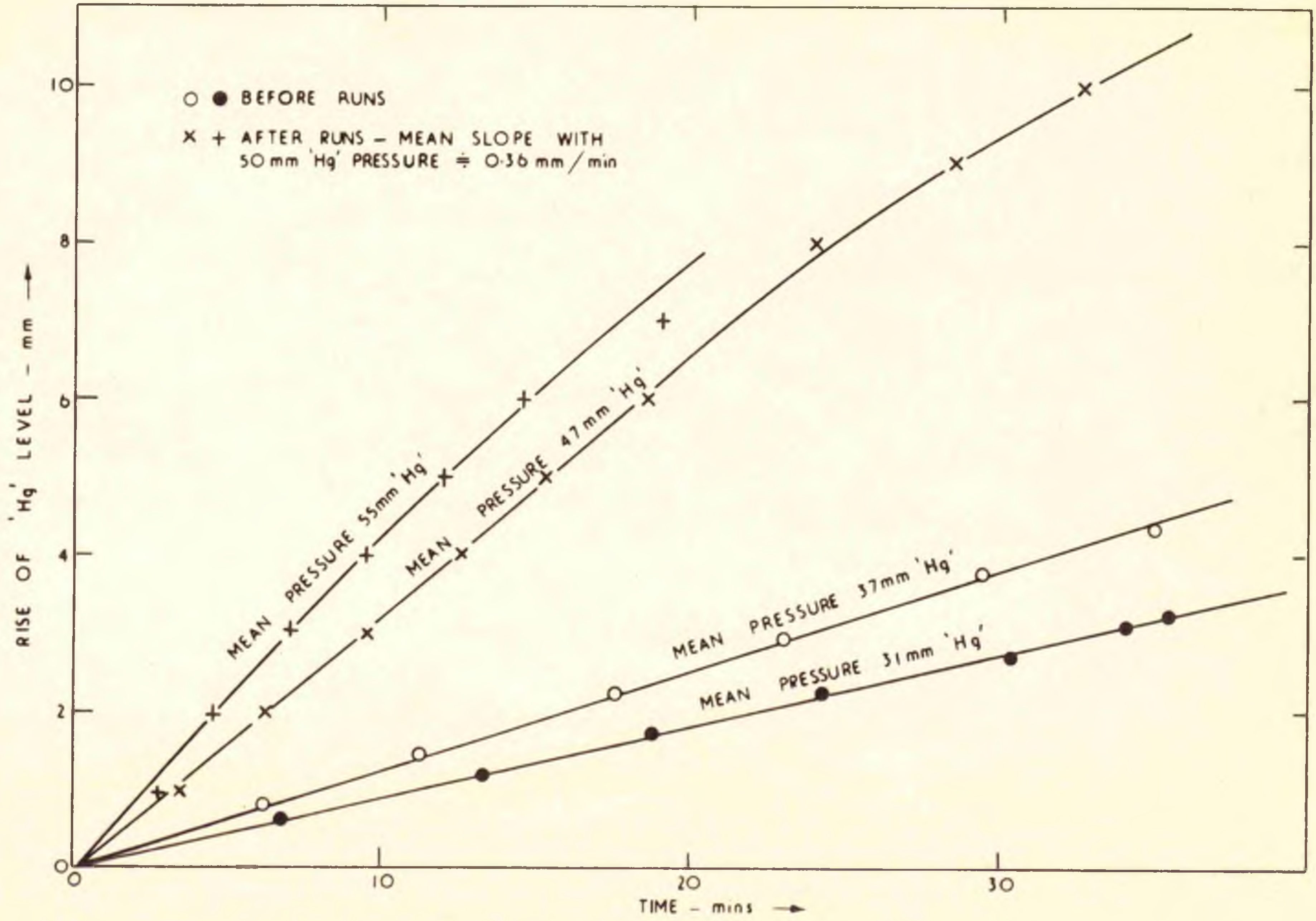
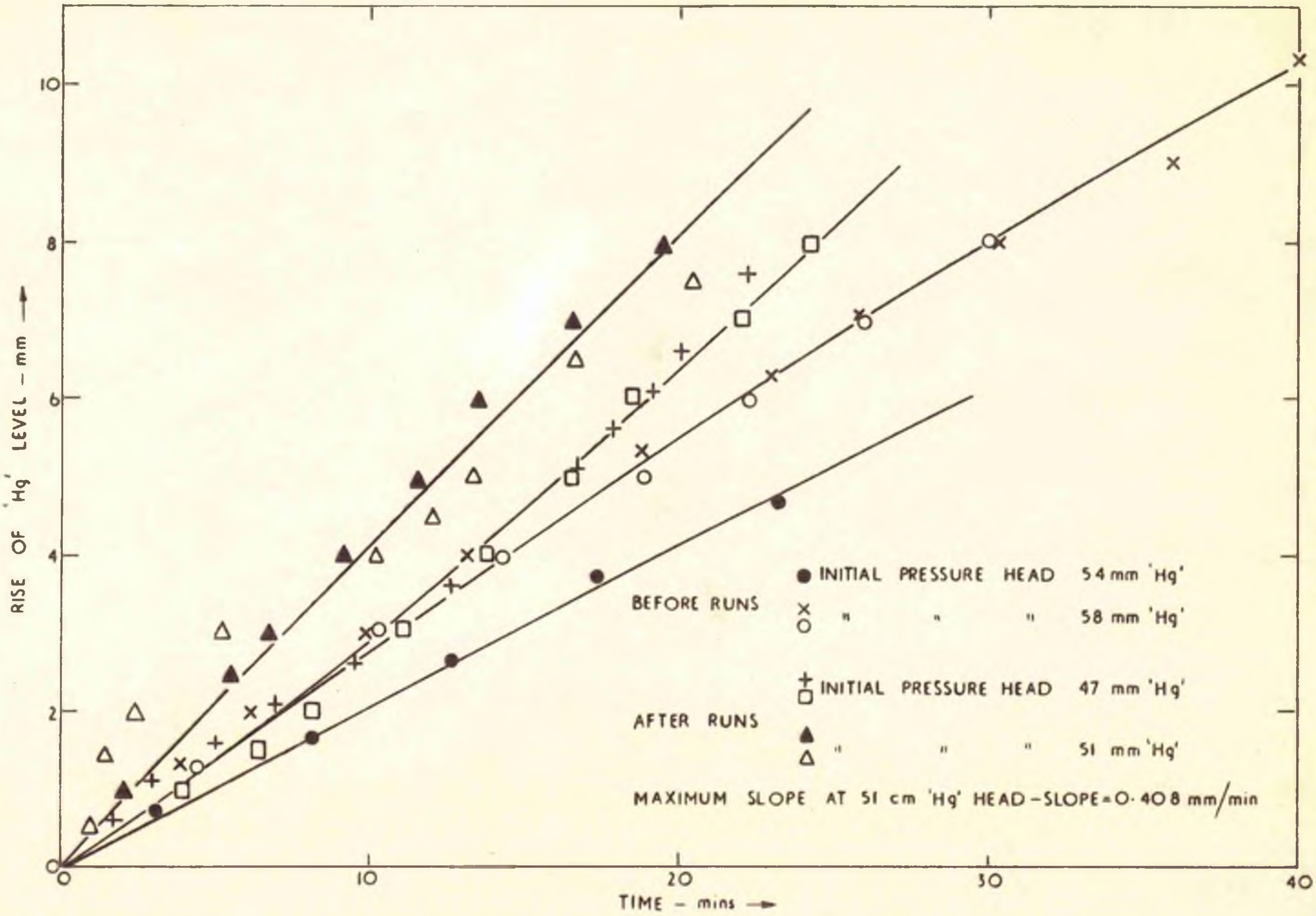


FIG A4.4 LEAK TESTS FOR SERIES CAPILLARY SYSTEM, USING CAPILLARIES N^{os} 2a AND 2b



LEAK TESTS FOR SERIES CAPILLARY SYSTEM, USING CAPILLARIES N$^{os.}$ 8a AND 8b

FIG A4.5

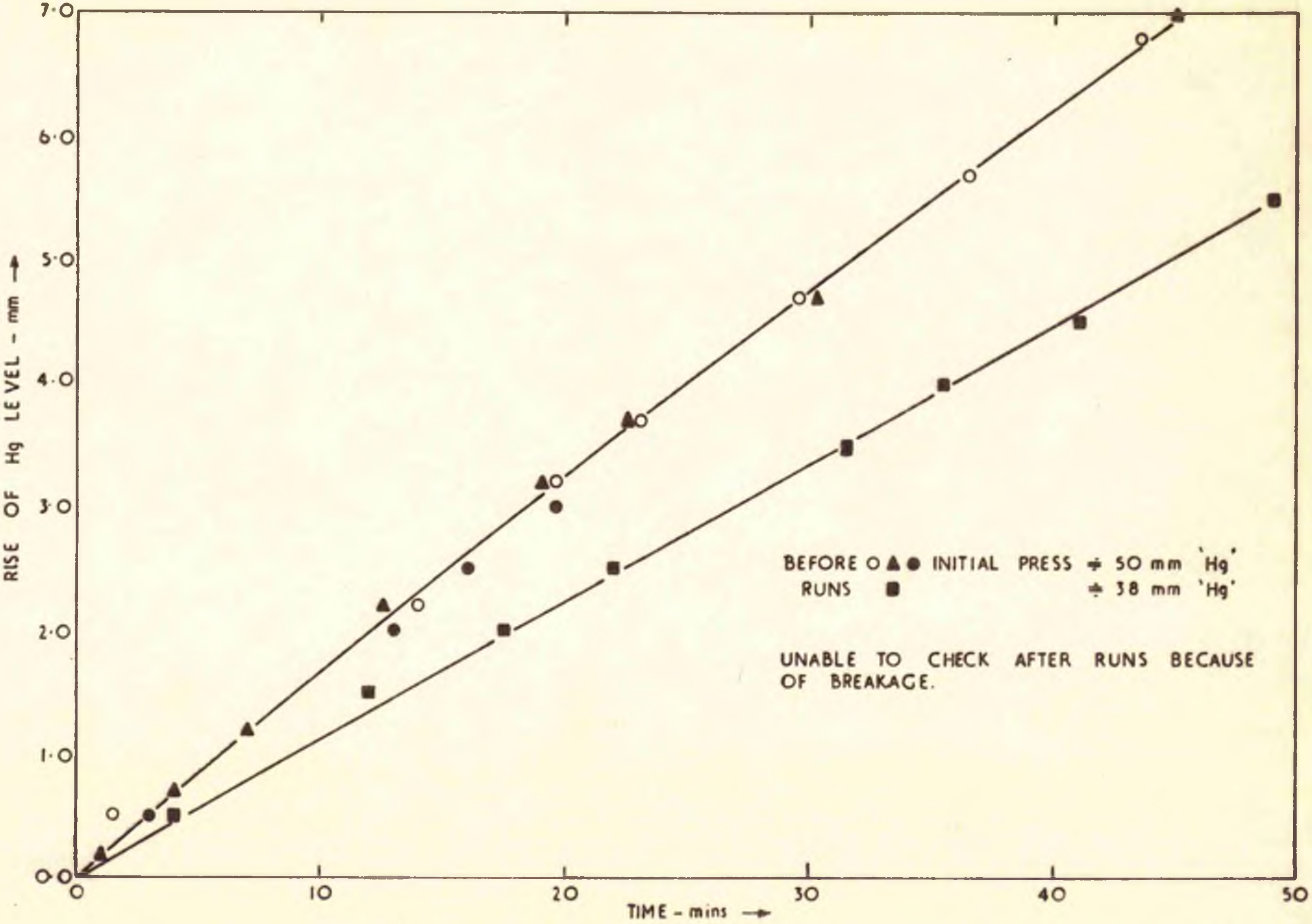


FIG A4.6 LEAK TESTS FOR SERIES SYSTEM USING CAPILLARIES (Ba) & (Bb)

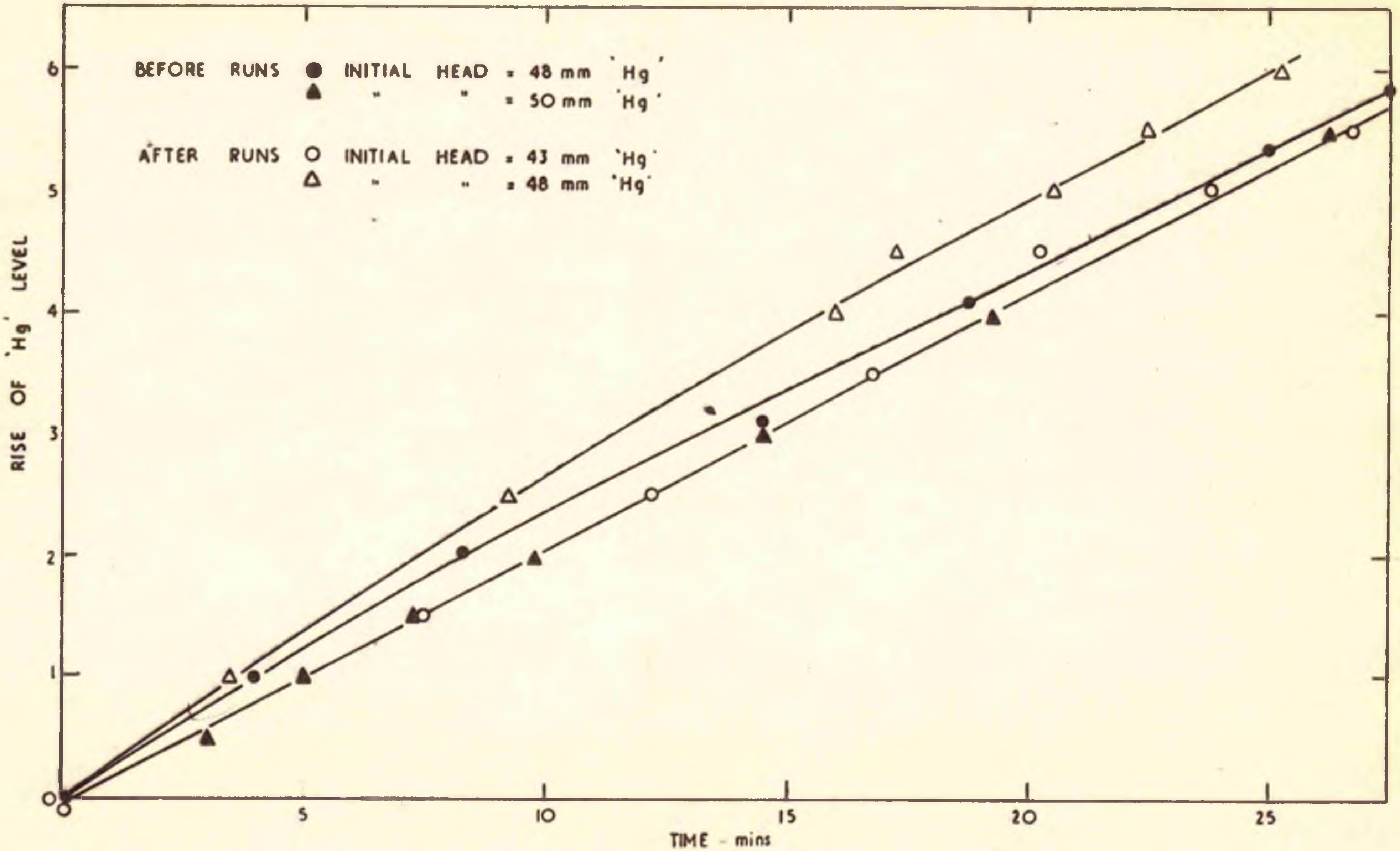


FIG A4.7 LEAK TEST FOR SERIES SYSTEM USING CAPILLARIES A(a) & A(b)

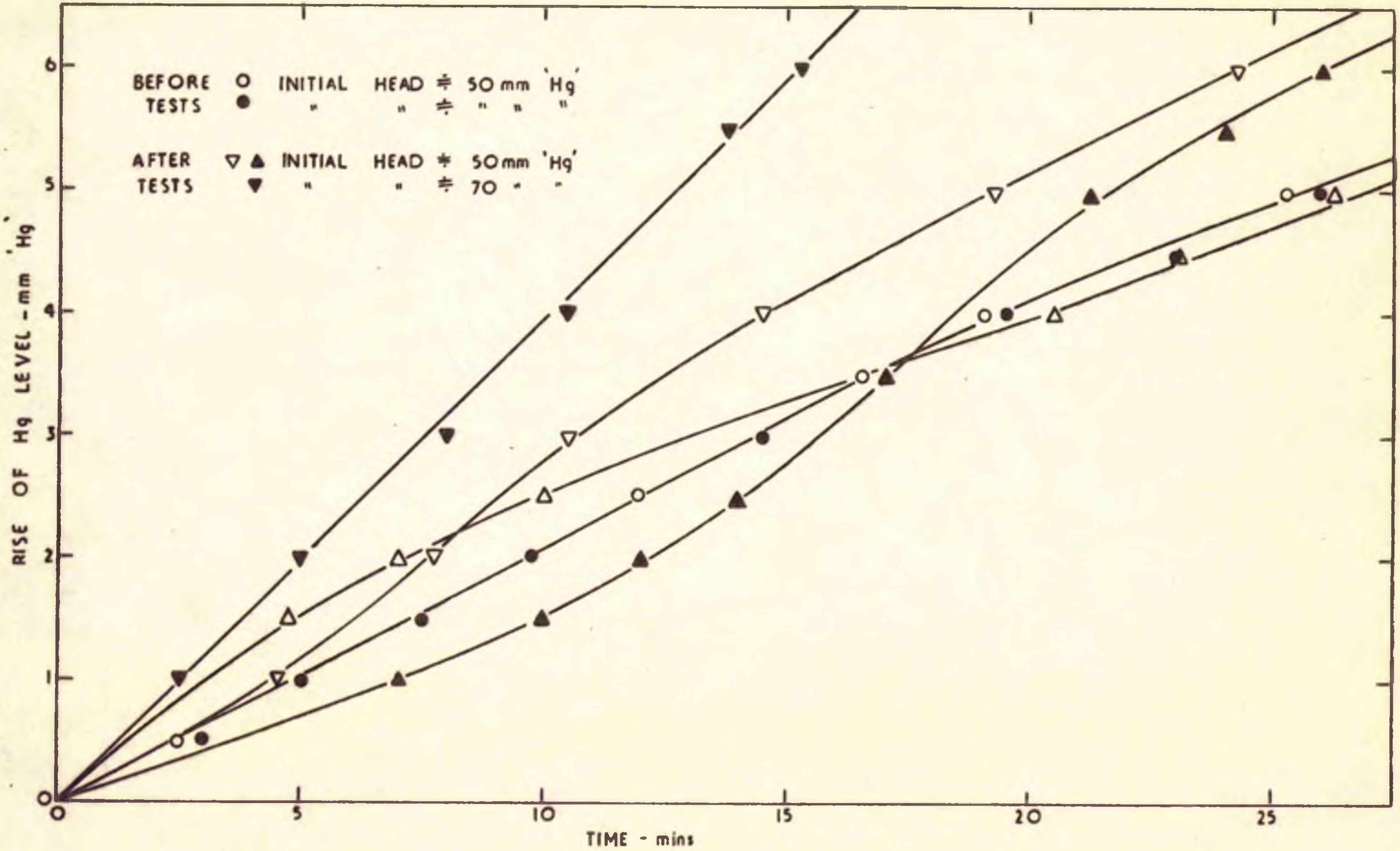


FIG A7.8 LEAK TEST FOR SERIES CAPILLARY SYSTEM USING CAPILLARIES Ca & Cb

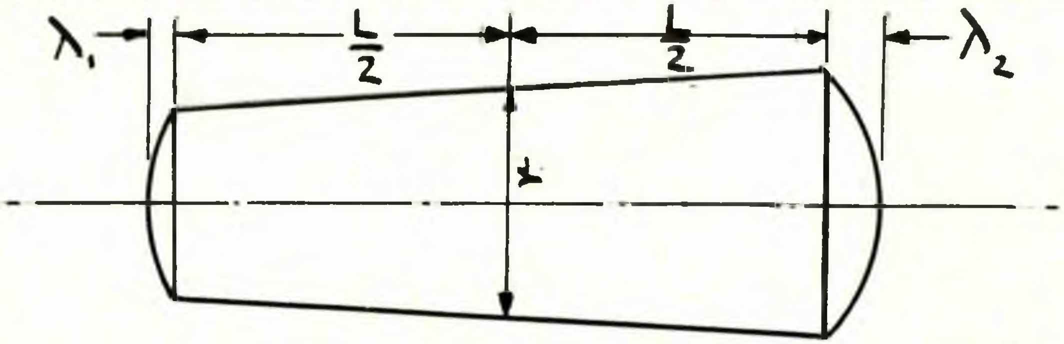
Appendix (5)

Capillary Calibrations

Since the diameter of a capillary is raised to the fourth power, in the flow equation for a capillary viscometer it is essential that the diameter be obtained accurately. It is also essential to have a capillary with a uniform bore with little or no ellipticity or conicality over its length.

The capillaries to be used are transparent and have bores up to 2 mm. It was therefore possible to calibrate them using the "mercury thread" method. The author is not aware of any other method which is as convenient to use or as accurate as this method. There are a number of procedures for calibrating a capillary which are (1) to check the uniformity of the capillary bore by passing a small pellet along the tube and observing its variation in length, after which, putting a thread of mercury along its entire length and obtaining the mean radius gravimetrically; (2) to go through the same procedure as quoted in (1), then combine the results from the small pellet and the large pellet calibrations, using a graphical method to solve for the diameter, and (3) to fill completely the capillary and then measure accurately the electrical resistance of the mercury thread. Two other methods of measuring the diameters of capillaries were tested, with little success. They were, (a) using an X-ray technique and (b) using probes to sense the ends of a mercury pellet. Neither of these methods was sufficiently accurate.

Methods (1) and (2) are very similar but the latter requires an accurate measurement of the small pellet length and its position along the tube. The theory for method (1) is quite straightforward. Consider a pellet of diameter 2r with a length between the wetted surfaces of L and a depth from the top of the meniscus to the wetted surface of λ. For the small pellet calibration the total length of each pellet was assumed to be $\{L + \lambda/2\}$. This is based on the assumption that in a good capillary a small pellet has the form of a right circular cone, capped at each end by a spherical segment, then,



$$V = \pi r^2 \left\{ (1 + \delta r^2/3r^2)L - (\delta r/r)(\lambda_1 - \lambda_2) + (1 + \delta r^2/r^2)\left\{\frac{\lambda_1 + \lambda_2}{2}\right\} + \left\{\frac{\lambda_1^3 + \lambda_2^3}{6r^2}\right\} \right\} \quad \ldots\ldots\ldots\ldots\ A5.1$$

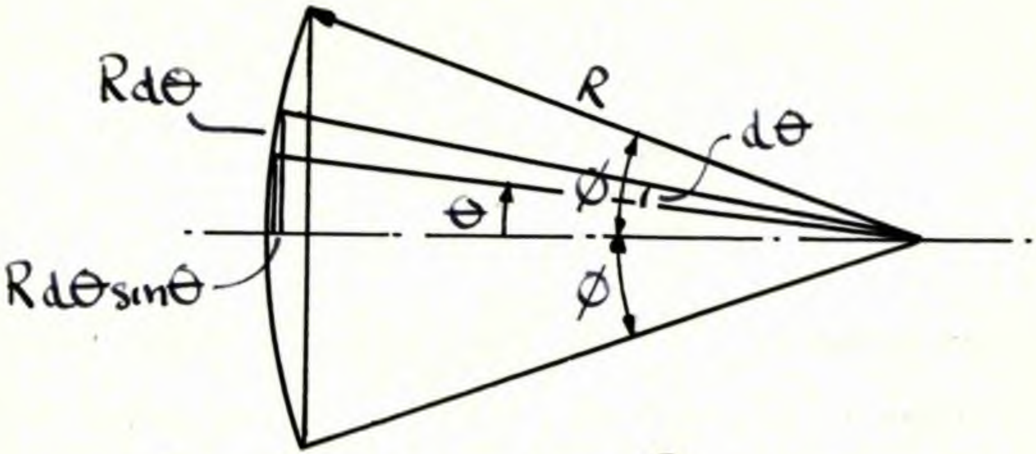
In practice, $\delta r/r$, λ_1^3, λ_2^3 and $(\lambda_1 - \lambda_2)$ are small in comparison with L and may be neglected, hence

$$V = \pi r^2 L + \frac{\pi r^2}{2}(\lambda_1 + \lambda_2) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ A5.2$$

Assuming the volume of an equal cylinder is $V = \pi r^2 L_s$, then,

$$L_s = L + (1/2)\ \{\lambda_1 + \lambda_2\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ A5.3$$

Now considering the large pellet, there are two methods of assessing the radius of the tube. One method is to assume that the meniscus at the ends of the pellet are spherical, of radius R, subtending an angle 2ϕ. That is the volume of each cap is



$$v = \int_0^{\phi} \pi R^3 \sin^3\theta \, d(\theta) = \pi R^3 \left[\frac{\cos^3\phi}{3} - \cos\phi + 2/3 \right] \quad \ldots\ldots \text{A5.4}$$

But $$R = \frac{r^2}{2\lambda} + \frac{\lambda}{2} \quad \ldots\ldots \text{A5.5}$$

Therefore the total volume of the mercury thread is

$$V = \pi R^2 L + 2\pi\left(\frac{r^2}{2\lambda} + \frac{\lambda}{2}\right)^3 \left[\frac{\cos^3\phi}{3} - \cos\phi + 2/3 \right] \quad \ldots\ldots \text{A5.6}$$

where the contact angle is $(90^\circ - \phi)$

This is obviously a difficult equation to use and the contact angle for the "mercury-to-glass" contact should be known. A more simple approach is to say that the volume of the end caps is

$$v = \int_0 \pi y^3 \, d(x). = \pi \left(R\lambda - \frac{\lambda^2}{3} \right) : \quad \ldots\ldots \text{A5.7}$$

but $R = \frac{\lambda}{2} + \frac{r^2}{2\lambda}$, therefore the total volume of the mercury thread is

$$V = \pi \left[(\lambda + L) r^2 - \lambda^3/3 \right] \quad \ldots\ldots \text{A5.8}$$

or $$r^2 = \left[V/\pi + \lambda^3/3 \right] \left[\frac{1}{(L + \lambda)} \right] \quad \ldots\ldots \text{A5.9}$$

Since λ is small λ^3 may be neglected and the above equation becomes

$$r^2 = \left[w/\rho\pi \right] \left[\frac{1}{(L + \lambda)} \right] = \left[W/\rho\pi \right] \left[\frac{1}{L_s} \right] \quad \text{........ A.5.10}$$

where w is the weight of the mercury pellet, ρ its density and $L_s = (L + \lambda)$.

The theory for the second method is based on a semi-graphical solution suggested by Fisher (167) who based his work on a method described by Schultze (168). The total pressure drop along a capillary in which a simple fluid is flowing is given by the equation

$$\int d(P) = \frac{8\ Q\ \eta}{\pi\ g\ \rho} \int_0^L d(L) / r^4$$

, from the basic Poiseuille equation A5.11

Now $V = Ls\ \pi r^2$, where r is the radius of the tube, and L_s and V are the length and the volume of the small pellet.

$$\text{But } V_1 = \int_{L_1}^{L_2} \pi\ r^2\ d(L) = V \int_{L_1}^{L_2} d(L)/Ls \quad \text{.................... A5.12}$$

where V_1 is the volume of a large pellet between two points L_1 and L_2 from the datum end of the capillary.

$$\text{Also, } \int_0^L d(L)/r^4 = \int_0^L \left[\pi Ls/V \right]^2 d(L). = \frac{\pi^2}{V^2} \int_0^L \left[Ls \right]^2 d(L) \quad \text{...A5.13}$$

Using equations (A.5.12) and (A5.13), then

$$\int_0^L d(L) r^4 = \frac{\pi^2}{V_1} \left[\int_{L_1}^{L_2} d(L)/Ls \right]^2 x \int_0^L Ls^2\ d(L) \quad \text{.........A5.14}$$

The integrals $\int_{L_1}^{L_2} d(L)/Ls$ and $\int_0^L Ls^2\ d(L)$ can be obtained from the small pellet calibration. Therefore r^4 in the flow equation may be replaced by $1/\left[\int d(L)/r^4 \right]$. Hanks (59) has defined an error introduced if this substitution is not used, it is

$$\delta = \frac{\left\{\frac{1}{L}\int_0^L d\,(L)/r^4\right\} - \left\{1/r^4\right\}}{\left\{1/r^4\right\}} \quad \ldots\ldots\ldots\ldots \text{A5.15}$$

which can be re-arranged as,

$$\frac{1}{L}\int_0^L d(L)/r^4 = \left\{\frac{1+\delta}{r^4}\right\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{A5.16}$$

With method (3), the electrical method, the capillary has large diameter reservoirs at its ends and the whole system is filled with mercury. Probes are put into the reservoirs and the resistance of the mercury thread in the capillary is compared to a standard resistance. The mean radius is then obtained from the equation

$$r_e = \left\{\frac{\rho e\ L}{A}\right\} = \left\{\frac{\rho e\ L}{\pi\ r^2}\right\} \quad \ldots\ldots\ldots\ldots\ldots\ldots \text{A5.17}$$

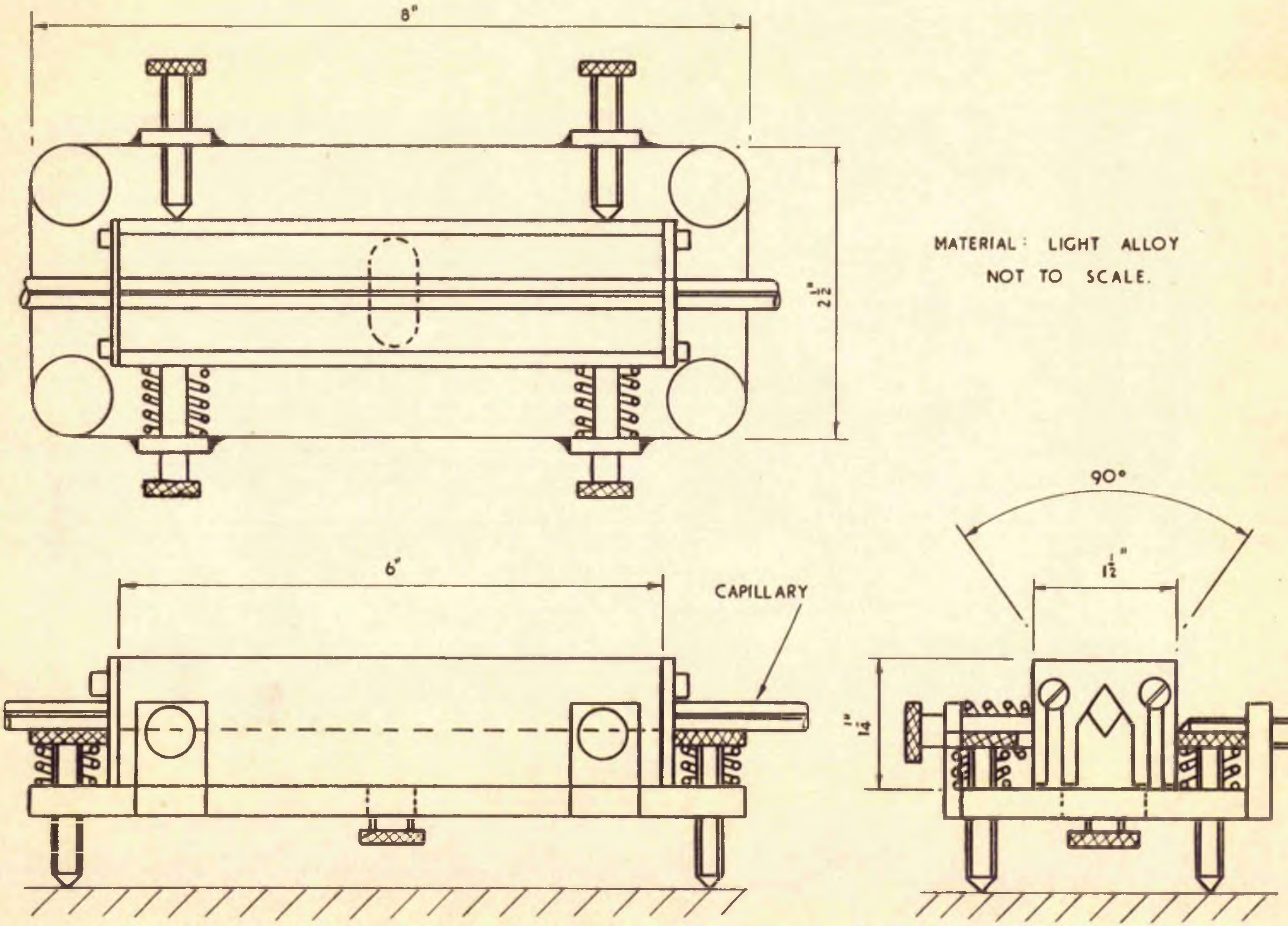
$$\text{or} \quad 1/r^2 = \left\{r_e\ \pi\right\} / \left\{\rho e\ L\right\}, \quad \ldots\ldots\ldots\ldots\ldots\ldots \text{A5.17}$$

where r_e is the electrical resistance, L is the length of the capillary tube, ρe is the resistivity of the mercury and 'r' is the mean radius of the capillary tube.

It was decided that method (1) was quite accurate enough for the proposed project, since the measuring equipment is not accurate enough to justify the use of the more complicated second method. It is also difficult to assess the accuracy of the graphical evaluations of the integrals, $\int_{L_1}^{L_2} d(L)/Ls$ and $\int_0^L Ls^2\, d(L)$. Carr (65) actually compared his capillary diameters, using both the gravimetric and the integration methods. The nominal radius of the capillaries he used was 0.1 mm. and the differences in the radii, obtained using the two methods were only about 0.09 % and 0.03 %. Some workers reckon that method (3) is the most applicable to the flow equation, but the author is of the opinion that the latter method has no obvious advantage over the other two methods, but this is a matter of opinion.

The selection of the capillaries and the procedure for their calibration was as follows. Initially, a large number of capillary tubes of the required nominal bores were calibrated using only the small pellet method. It was then possible to draw up a short list of the capillaries having a tolerable uniform bore. From each of the tubes, two capillaries of the required length were cut and their ends had standard B.5. cones ground on them. They were then thoroughly cleaned by soaking them in chromic acid and rinsing, in turn, with distilled water, alcohol and ether. The capillaries were again calibrated before a final selection was made. The mean diameter of each capillary was obtained several times, before and after use, using the large pellet calibration.

The procedure for calibration was as follows. The capillary, which had been previously cleaned and dried, was put into the jig, see figure (A5.1). A small pellet of "triple distilled" mercury was injected into one end of the capillary and two elbow tubes, having standard B.5. sockets at their ends, were fitted to the capillary. The arrangement, with the end tubes vertical, was immersed in a bath of liquid (the liquid being glycerine for the glass tubes and carbon tetrachloride for the quartz tubes), having the same refractive index as that of the material of the capillary. The length of the pellet was then checked, using a cathetometer manufactured by Messrs. Pye Ltd. The pellet was moved to a new position, such that it overlapped the old position, and once again the length was



MATERIAL: LIGHT ALLOY
NOT TO SCALE.

FIG A5.1 JIG FOR HOLDING CAPILLARIES DURING CALIBRATION measured. This was repeated until the pellet reached the other end of the capillary. The procedure was basically the same for the large pellet calibration, only the pellet was made, as near as possible, as long as the capillary tube. The large pellet was then carefully extracted from the tube and weighed.

It should be noted that the pellets were introduced into the tubes in the presence of air. The author appreciates that although the mercury was thoroughly dried and stored under a vacuum prior to its use, this would not preclude air from being trapped between the capillary wall and the mercury. Attempts were made to introduce the pellets under vacuum conditions, but unfortunately in the majority of cases, the pellets broke up into small beads. A large mercury pellet was successfully introduced into the tube under vacuum conditions and the diameter compared with results from air fillings. The diameters agreed within the expected tolerance of the measuring equipment, which is only to be expected, since the entrapped air, for a smooth bore tube, would be quite small when compared with the sizes of bores measured.

Small pellet calibration graphs for each of the tubes used are given in figures (A5.2) to (A5.15). A table of the diameters obtained from the large pellet calibrations is given on the following page. A table of the lengths of the capillaries is also given on the following page.

Capillary diameters obtained from large pellet calibrations (using equation AS.10)

Capillary No.	Before use.			After use.			Overall mean dia. mm	Overall dev. % ±
	d mm	dm mm	dev. % ±	d mm	dm mm	dev. % ±		
1	0.99920 0.99954	0.99937	0.017	0.99882 0.99886	0.99884	0.002	0.99911	0.027
2	1.51041 1.51035 1.51035	1.51037	0.002	1.50885 1.50989 1.50943 1.51005	1.50956	0.040	1.50996	0.052
Quartz.								
3a.	1.05408 1.05488	1.50448	0.038	1.05504 1.05504	1.05448	0.000	1.05476	0.027
4b.	1.02956 1.02958 1.02964	1.02959	0.004	1.03097 1.03908	1.03097	0.001	1.03028	0.067
2a.	1.32482 1.32488 1.32485	1.32485	0.002	1.32452 1.32469	1.32453	0.001	1.32469	0.012
2b.	1.31024 1.31002	1.31013	0.008	1.30971 1.30999 1.30968	1.30979	0.012	1.30996	0.013

Capillary diameters obtained from large pellet calibrations (using equation AS.10)

Capillary No.	Before use.			After use.			Overall mean dia. mm	Overall dev. % ±
	d mm	dm mm	dev. % ±	d mm	dm mm	dev. % ±		
Quartz.								
8a.	1.70352 1.70321	1.70336	0.009	1.70351 1.70366 1.70343	1.70354	0.007	1.70345	0.005
8b.	1.71804 1.71811 1.71827	1.71814	0.007	1.71838 1.71878 1.71829	1.71848	0.014	1.71831	0.010
Ba.	1.52496 1.52475	1.52485	0.007	broken prior to being dismantled.				
Bb.	1.53365 1.53378 1.53397	1.53380	0.010	1.53388 1.53385	1.53387	0.001	1.53384	0.010
Aa.	0.98462 0.98478 0.98464 0.98475	0.98470	0.008	0.98482 0.98476	0.98480	0.003	0.98475	0.010
Ab.	1.00133 1.00147	1.00140 1.00140	0.007	1.00151 1.00139	1.00145	0.006	1.00142	0.009
Ca.	1.96607 1.96614 1.96643	1.96621	0.009	1.96764 1.96801 1.96807	1.96791	0.011	1.96706	0.051
Cb.	1.95199 1.95208	1.95204	0.002	1.95312 1.95321	1.95317	0.002	1.95260	0.031

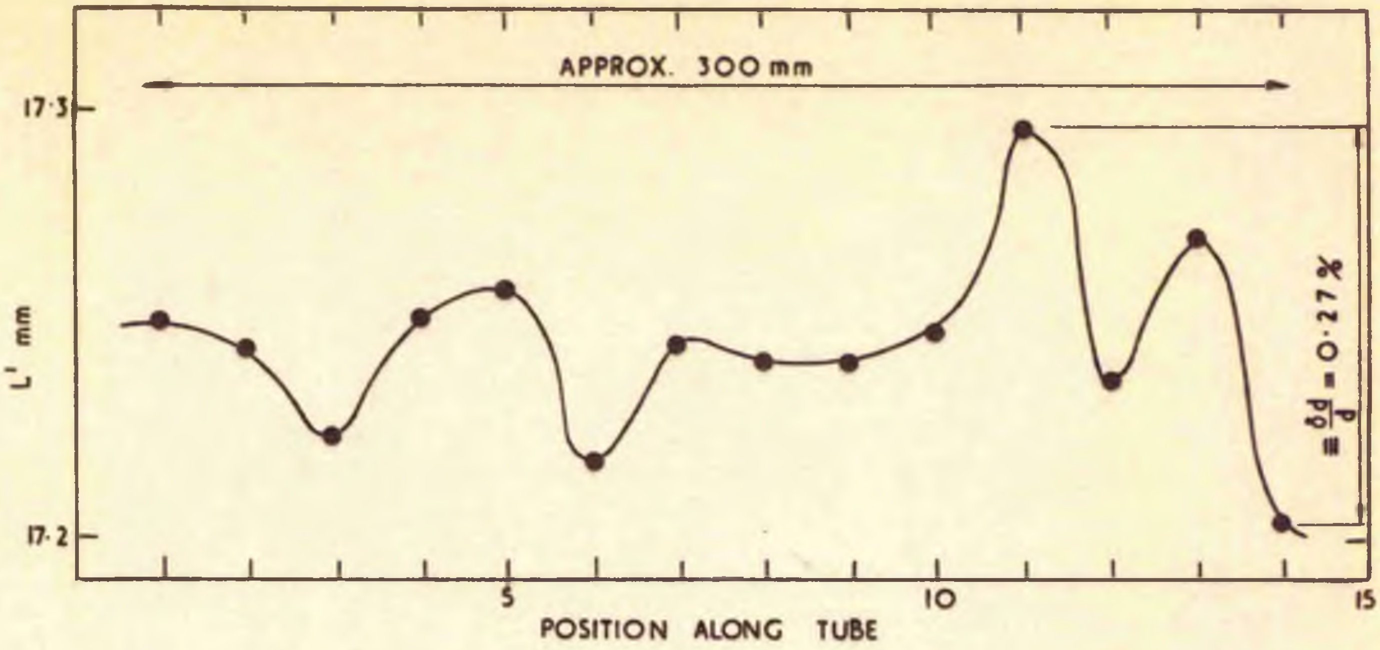
Capillary Lengths

Capillary No.	Length before use cm.	Length after use cm.	Mean length cm.	Dev. ± %
Glass Capillaries				
1.	29.785	29.780	29.783	0.017
2.	30.265	30.260	30.263	0.017
Quartz Capillaries				
3a.	34.975	34.980	34.978	0.014
4b.	25.010	25.000	25.005	0.020
2a.	35.043	35.046	35.045	0.009
2b.	24.926	24.930	24.928	0.016
8a.	35.045	x	35.045	-
8b.	24.595	x	24.595	-
Ba.	37.515	x x	37.515	-
Bb.	23.006	23.004	23.005	0.009
Aa.	37.522	37.525	37.523	0.011
Ab.	22.965	22.960	22.968	0.022
Ca.	37.460	x x x	37.460	-
Cb.	22.990	x x x	22.990	-

x the ends of the capillaries were slightly chipped on dismantling.

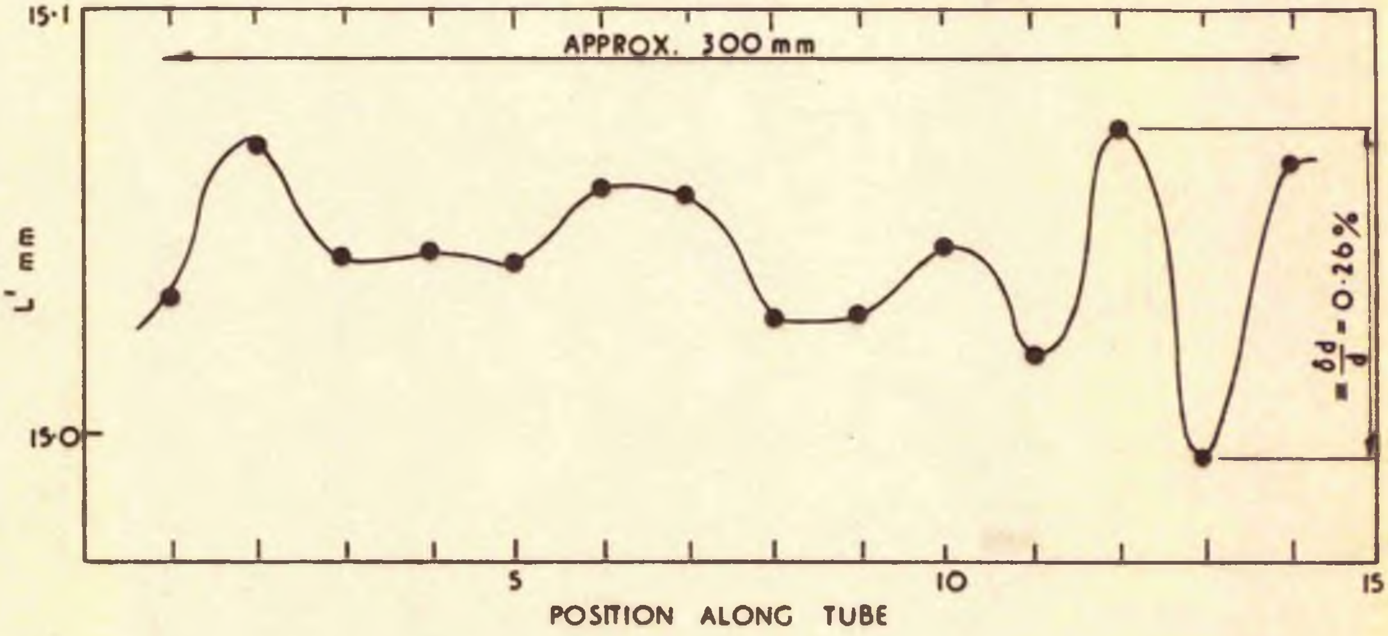
x x the capillaries were broken before being dismantled.

x x x The ends of the capillaries were broken during dismantling.



CAPILLARY TUBE No. 1 (GLASS)

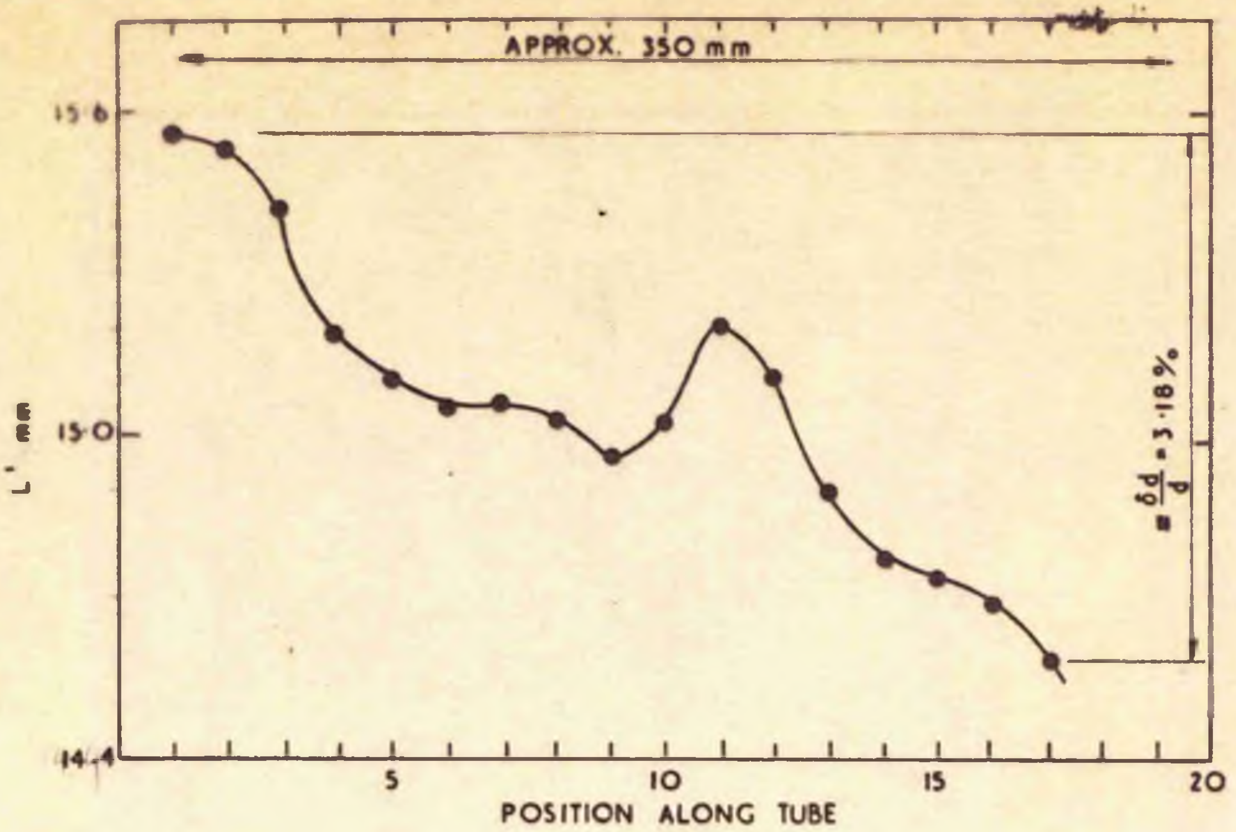
FIG A5. 2



CAPILLARY TUBE No 2 (GLASS)

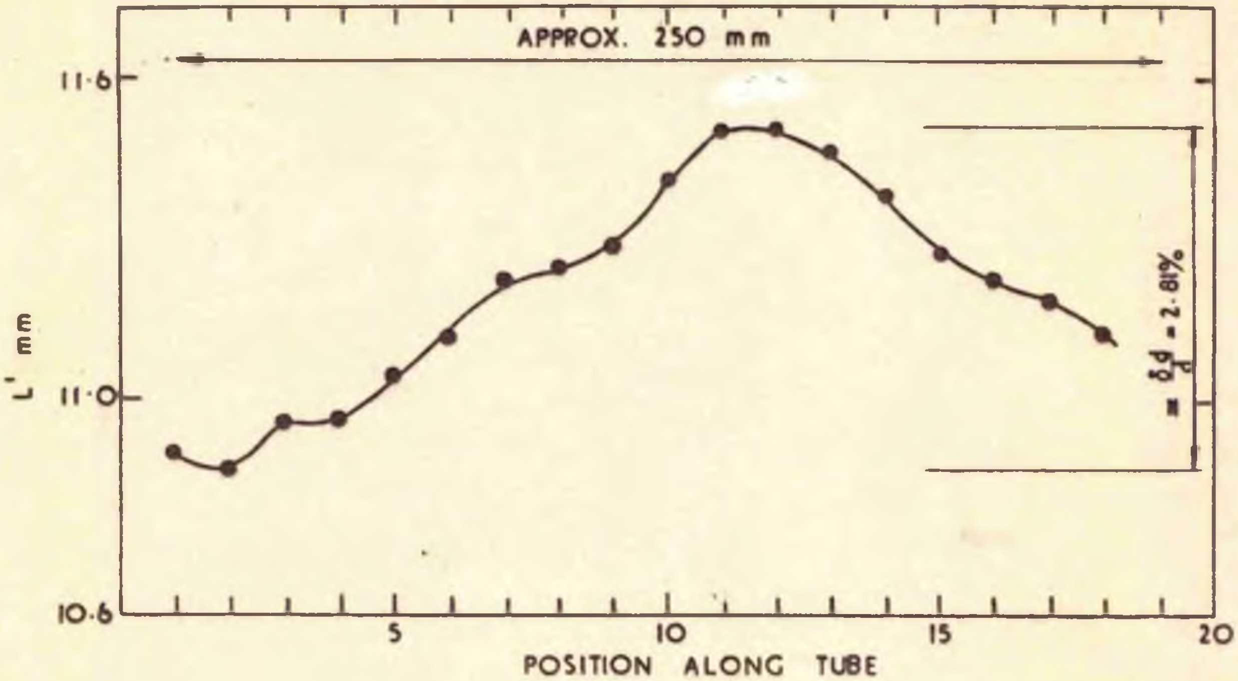
FIG A5. 3

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS



CAPILLARY TUBE N° 3a QUARTZ

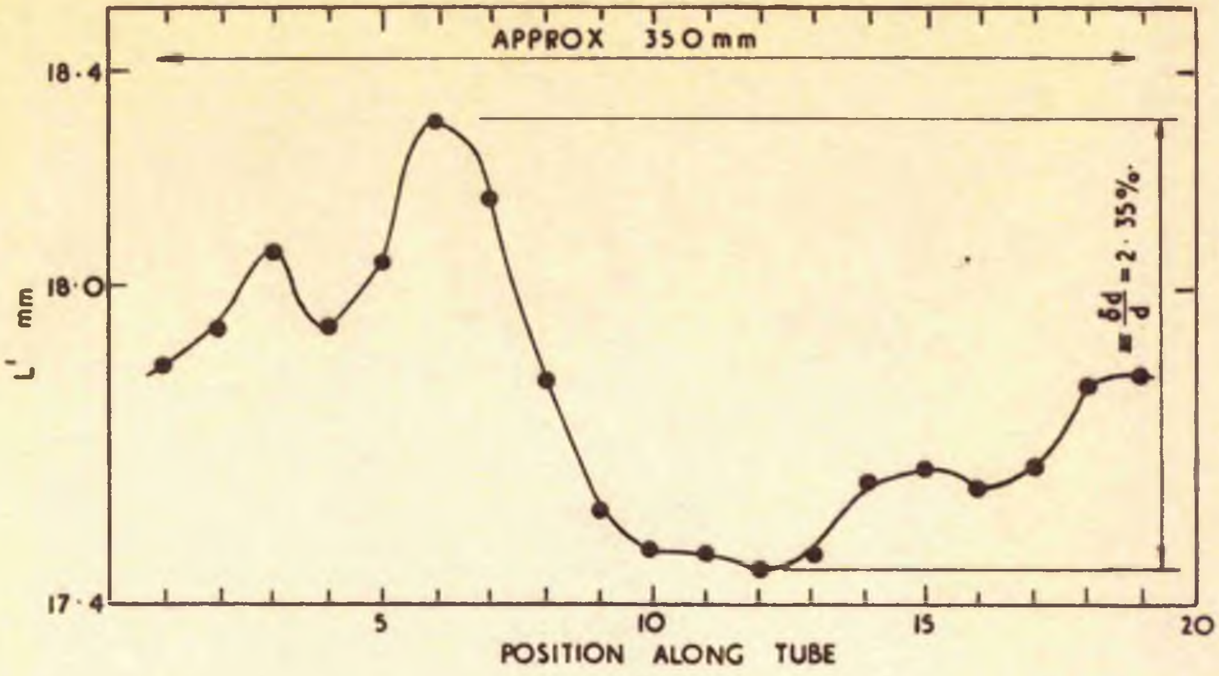
FIG A5. 4



CAPILLARY TUBE N° 4b QUARTZ

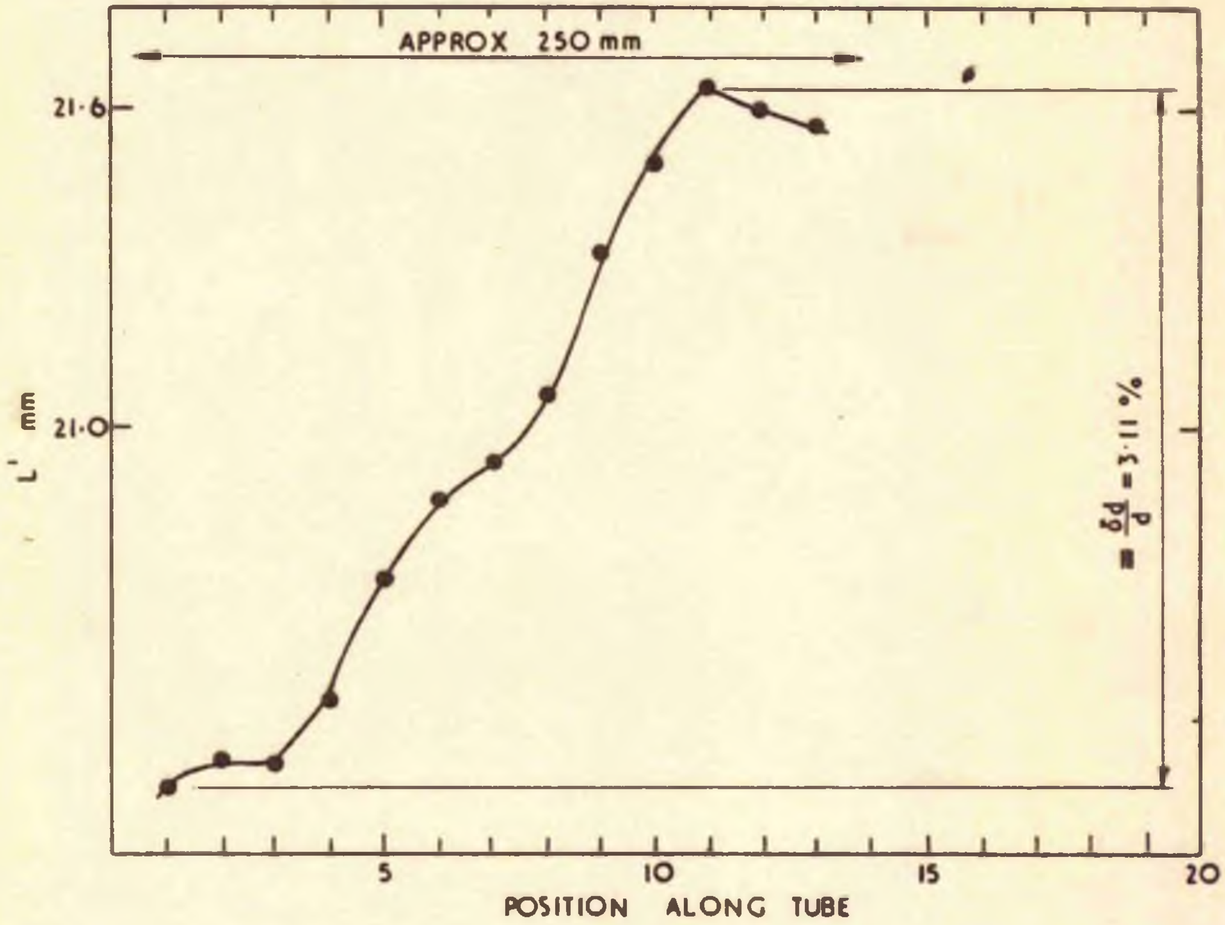
FIG A5. 5

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS



CAPILLARY TUBE N° 2a QUARTZ

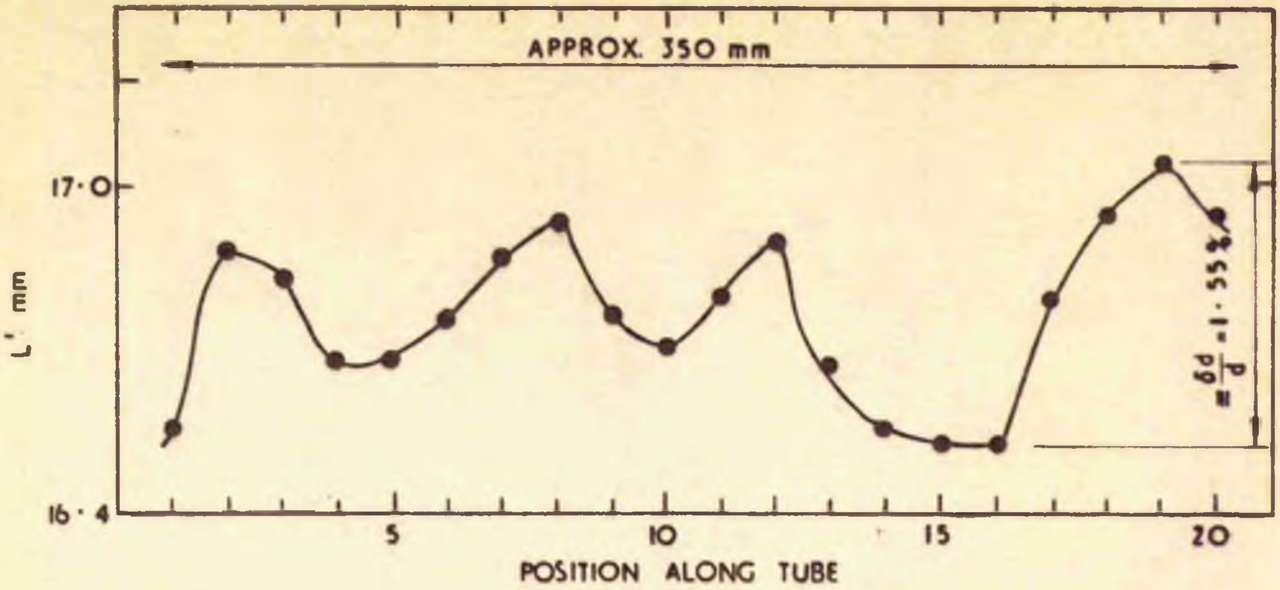
FIG A5. 6



CAPILLARY TUBE N° 2b QUARTZ

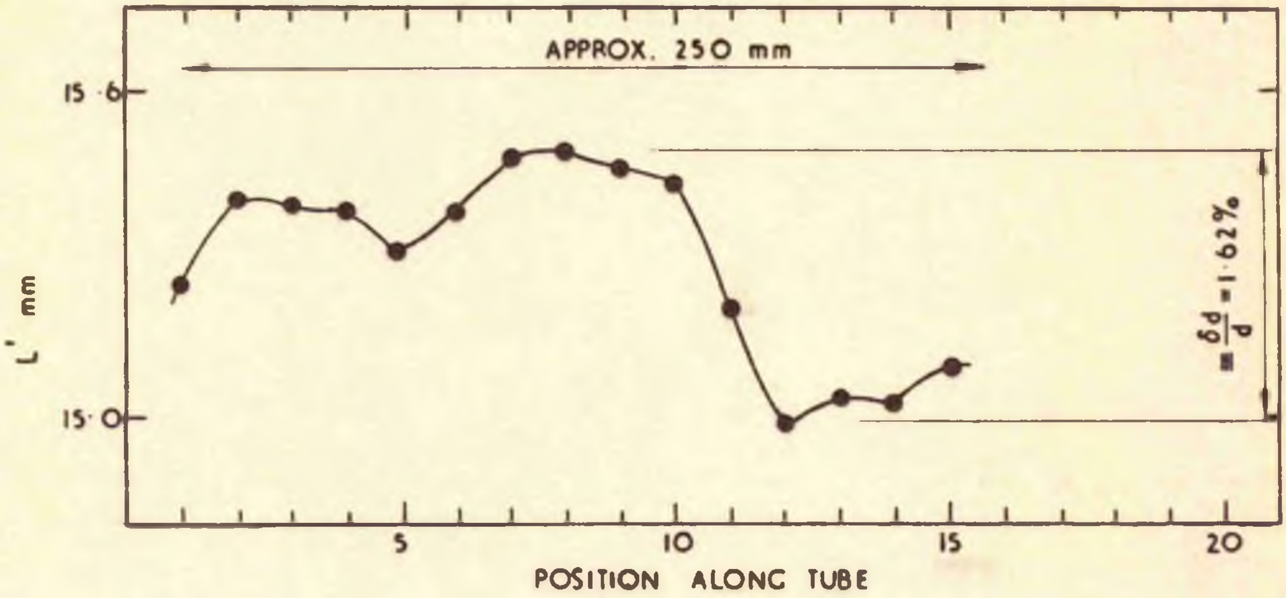
FIG A5. 7

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS



CAPILLARY TUBE N° 8a QUARTZ

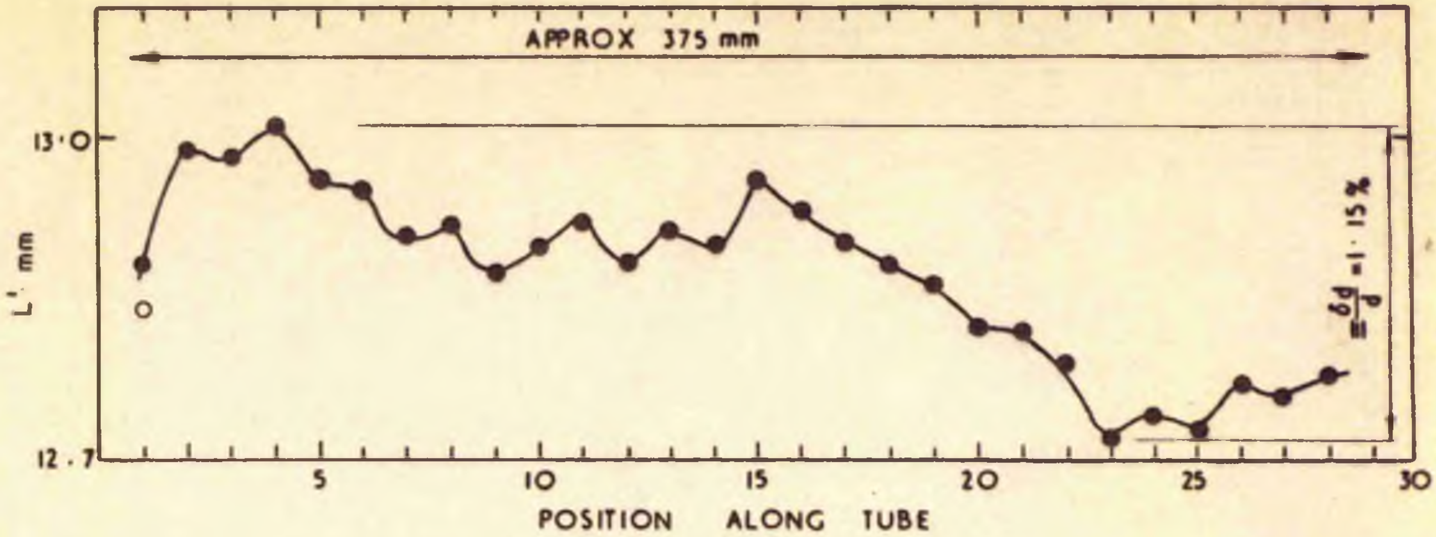
FIG A5. 8



CAPILLARY TUBE N° 8b QUARTZ

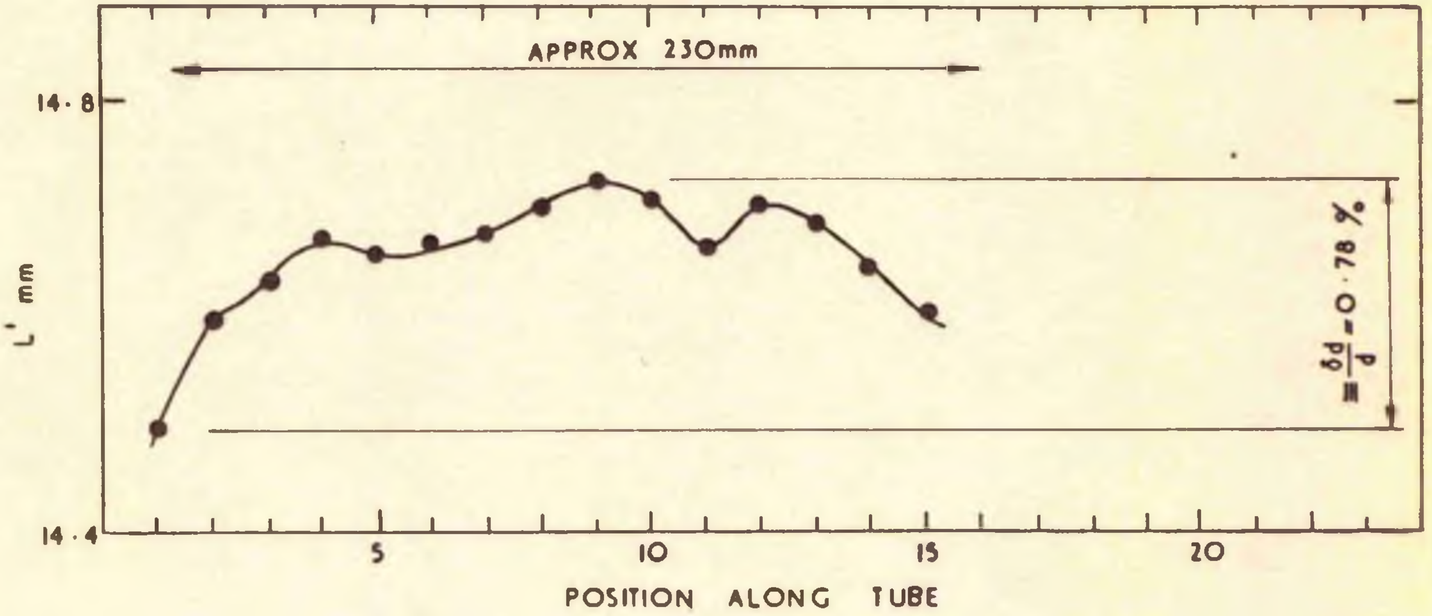
FIG A5. 9

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS



CAPILLARY TUBE N° Ba QUARTZ

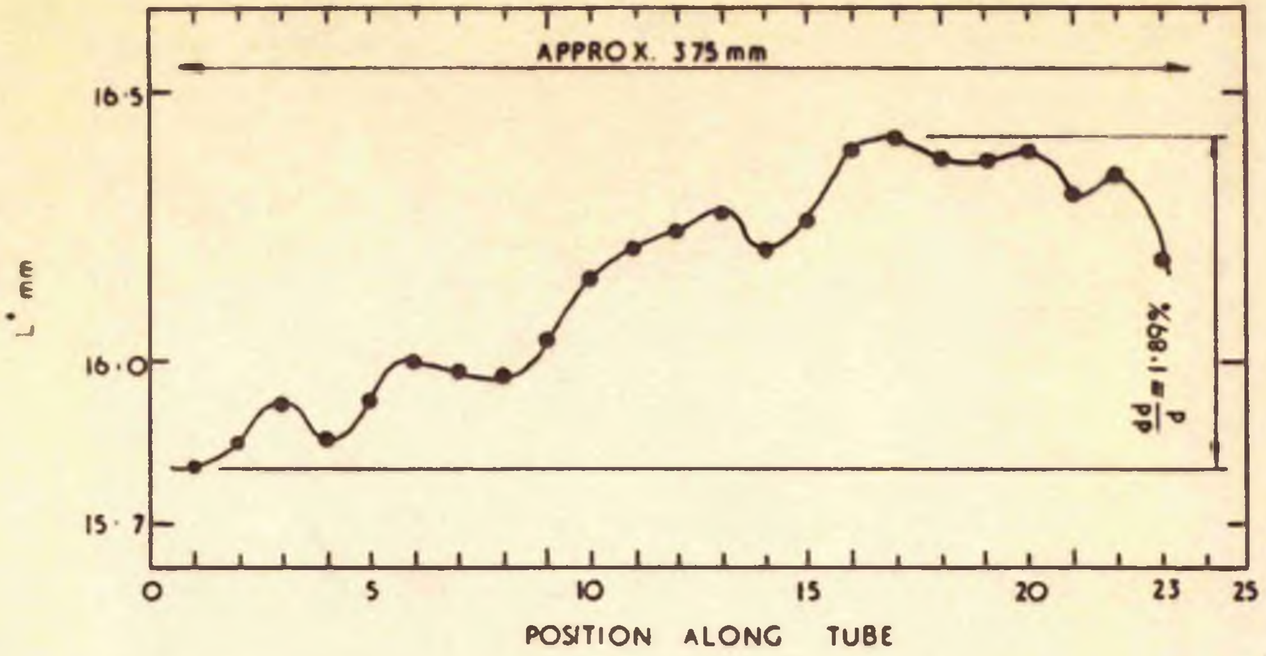
FIG A5. 10



CAPILLARY TUBE N° Bb QUARTZ

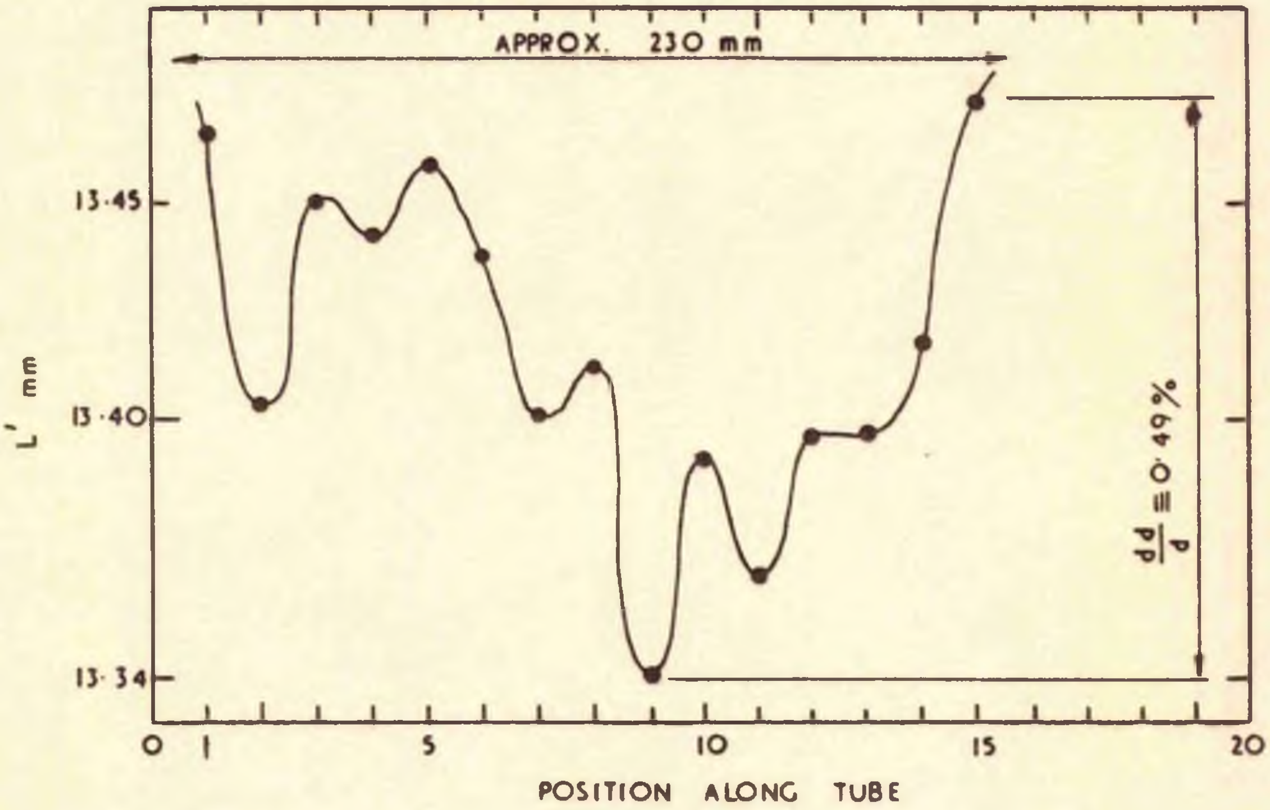
FIG A5. 11

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS



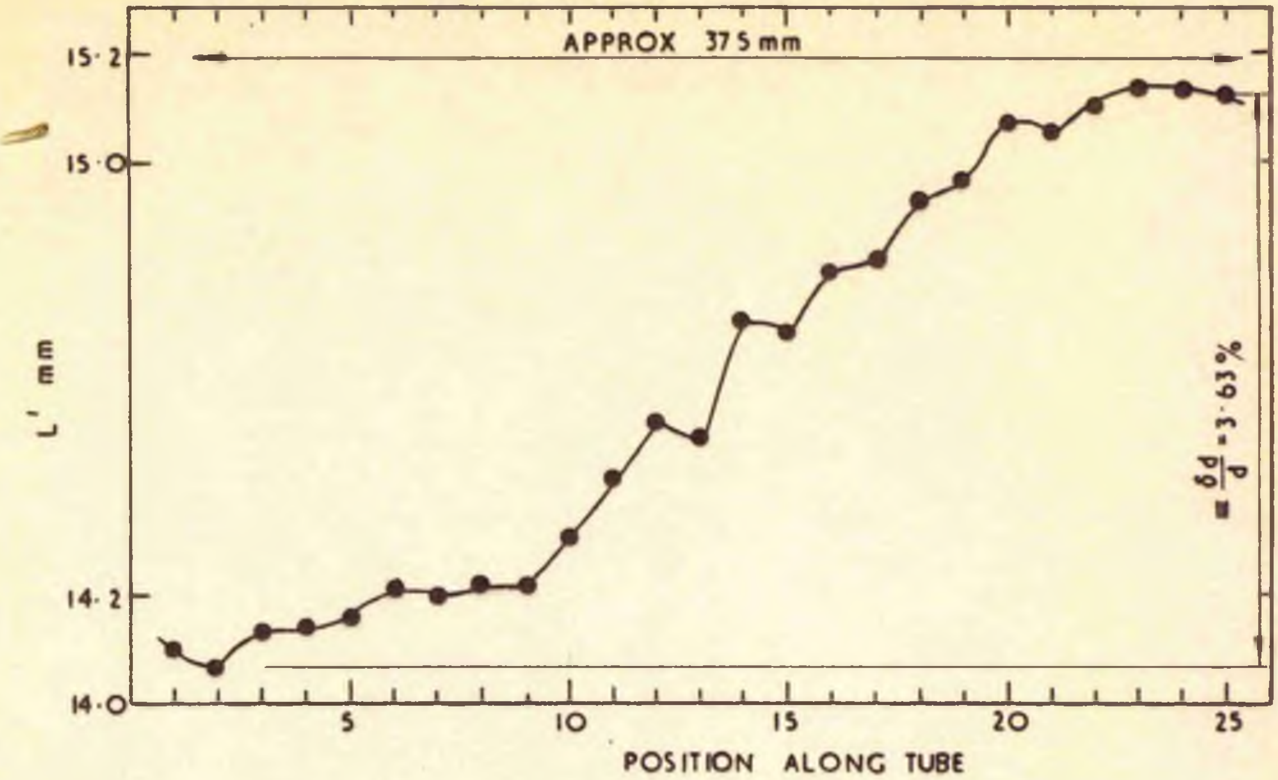
CAPILLARY TUBE N° A(a) QUARTZ

FIG A5. 12



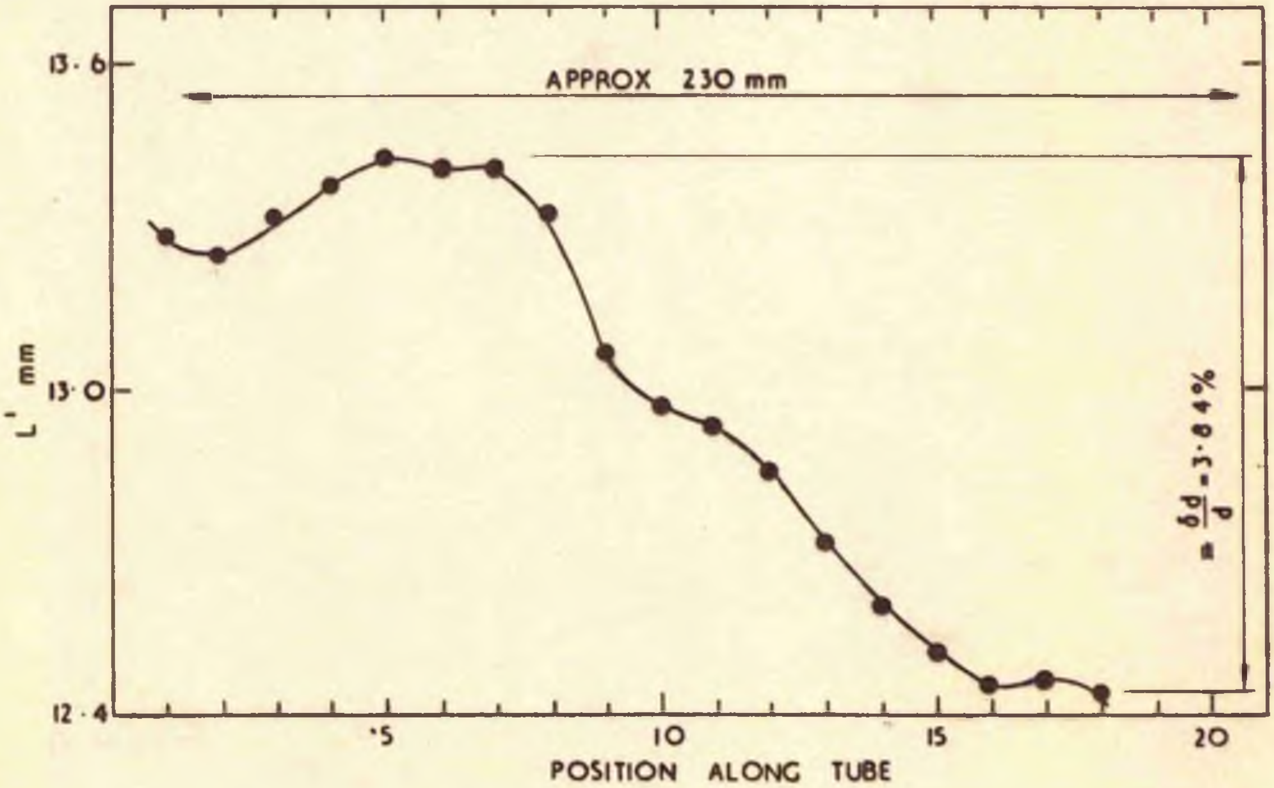
CAPILLARY TUBE N° A(b) QUARTZ

FIG A5. 13



CAPILLARY TUBE N° Ca QUARTZ

FIG A5. 14



CAPILLARY TUBE N° Cb QUARTZ

FIG A5. 15

SMALL "Hg" PELLET CAPILLARY CALIBRATIONS

Appendix (6)

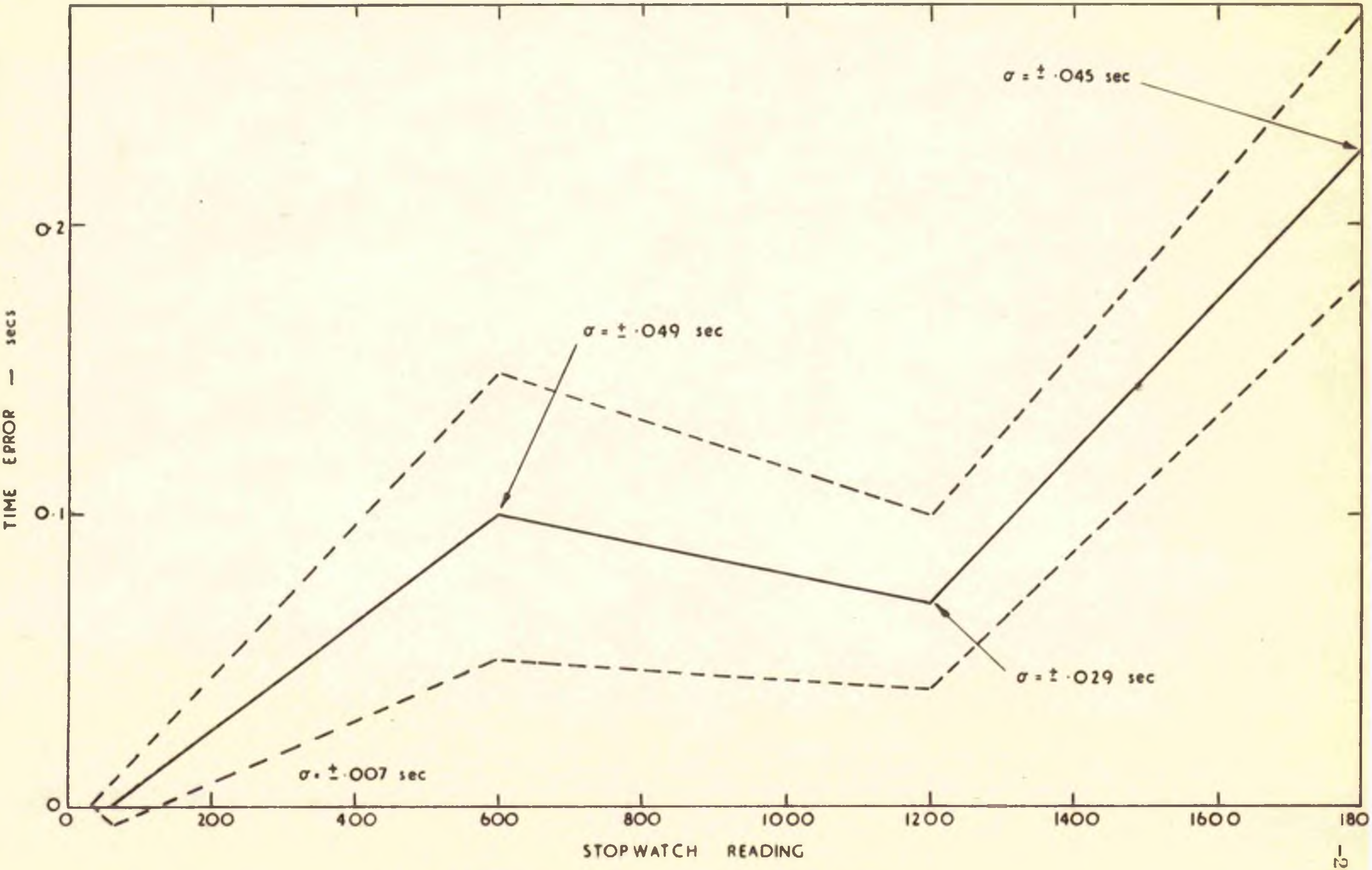
Timing Calibration

As described in chapter (VI), the timing of the condensate collection was done with a time interval meter manufactured by Messrs. Camerer Cuss Ltd. This is a multi-purpose electrical timing relay which operates a Camerer Cuss stopwatch. The stopwatch dial is graduated in 0.1 second divisions, one revolution of the main hand representing 10 seconds. A second small dial is graduated in 10 second divisions, one revolution of this hand representing 10 minutes. The relay was connected to a contact switch in the condensate collection system, such that, immediately the test tube was placed under or removed from under the condenser, the stopwatch was operated.

The manufacturers of the time interval meter state that the variation in the time interval between the closing of the contacts and the operation of the stopwatch is constant at 0.002 seconds.

The timing system was independently checked under working conditions. The signal to operate the interval timer was supplied by a special pulse generator via a secondary relay, the closing of the contacts of the secondary relay starts or stops the interval timer. The tests were carried out with the interval timer set in mode (2) operation, the signal being fed into terminals B and C. Previous tests had shown the variation in the operating time of the secondary relay to be less than 0.001 second on consecutive operations. This variation in the operating time had no effect on the calibration, since the stopwatch can only be read to ± 0.1 second. In about 50 % of the tests the stopwatch was fully wound up prior to its use.

The results of the calibration tests are shown on the graph of timing error against stopwatch reading (see figure (A6.1)). There is no indication on the graph as to whether the stopwatch was fully wound up before a test. It can be seen that the timing error will be of the order of ± 0.008 % at 600 secs., ± 0.002 % at 1200 secs. and ± 0.003 % at 1800 secs. It is possible, though unlikely, that the pulse generator has a constant error, in addition to its known variation with temperature. There have been some preliminary checks which revealed nothing of consequence.



A6.1 ERROR GRAPH FOR STOPWATCH

Appendix (7)

Manometer Precision

The pressure drop across the capillaries was measured using a three limb mercury in glass manometer. Apart from the measurement of the capillary diameters, the observations which have the most effect on the precision, and ultimately on the accuracy of the viscosity determinations, are those of the differential pressure drops. It was therefore obvious that the accuracy of the pressure measuring equipment should be as high as possible. An attempt at assessing the precision of the manometer was made.

The same manometer and associated equipment was used for the single capillary and the series capillary apparatus. The former only required two of the three limbs of the manometer.

The method of measuring the pressure differences is described in chapter (VI). The majority of the pressure drops were expected to lie within the range of 8 mm to 35 mm of mercury and therefore the tests were carried out with this range in mind.

The precision depends on four main factors: (1) the accuracy of the cathetometer calibration, (2) the ability to sight the mercury surface, with the given surface illumination and optical arrangement, (3) the ability to reproduce the 'zero' mercury level in the fixed telescope, and (4) the vertical bearing on the cathetometer.

Dealing with the above four items separately:-

(1) The cathetometer is a Swift Precision Instrument, having a vernier which can be read to 0.01 mm over a length of 200 mm. A Moore and Wright micrometer head is fitted to the instrument. This

can be read to 0.002 mm, but its range is only 25 mm. To make full use of the sensitivity of the micrometer head, it would be necessary to measure both the pressure drops without re-setting the zero on the head. This would mean that pressure drops of the order of 55 mm Hg would be required to be read on the micrometer head. Consequently, only the vernier scale was used to measure the pressure drops in the series capillary system.

(2) The mercury surfaces were illuminated from the rear of the manometer box, through 1/8 in. wide vertical slits in a light alloy sheet, which had a matt black surface, placed behind the manometer columns. The mercury menisci were sighted between two hair lines in telescopes having approximate magnifications of 10:1.

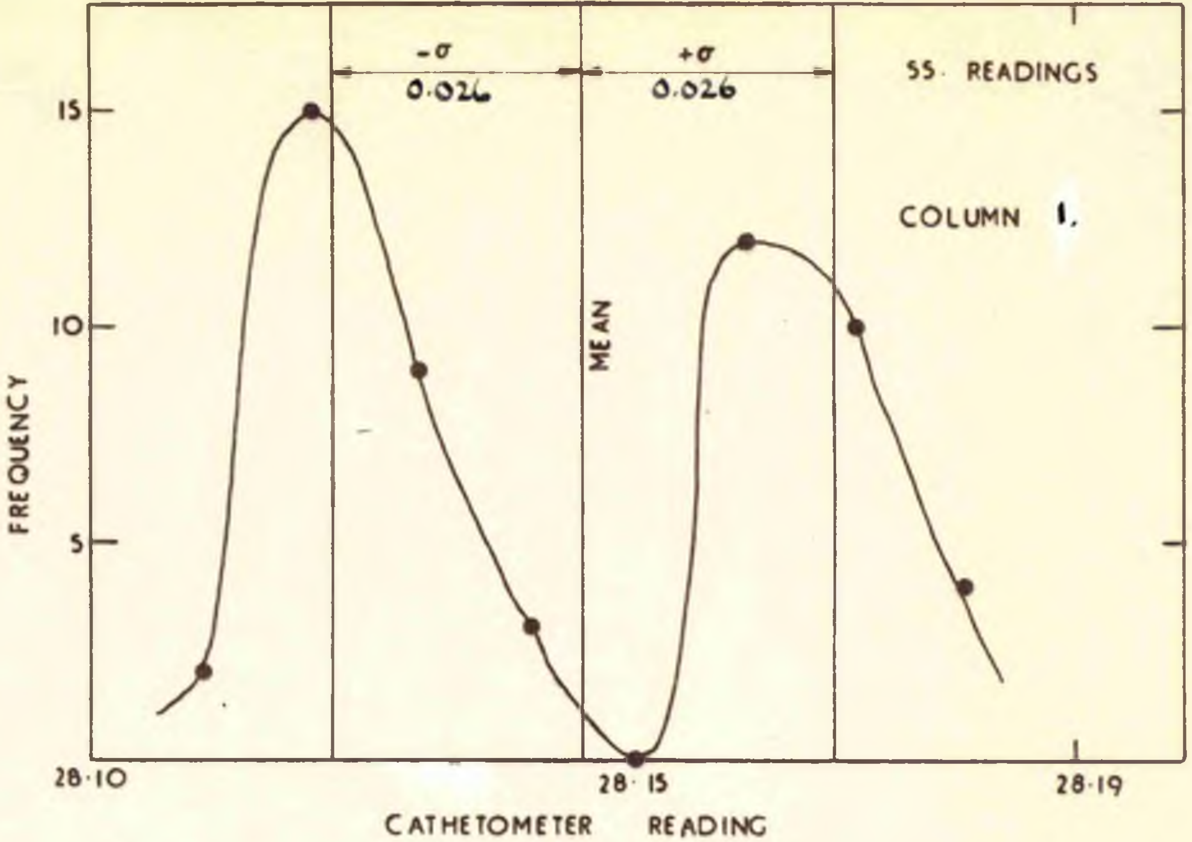
(3) The "make-up" cylinder, (see figure (6.15)), was used to keep the mercury level in the central column constant. The "make-up" cylinder has two control pistons; the coarse piston has a screw thread pitch of 1/9 in., one revolution of which raises the mercury level in a column by about 1.5 mm. The fine control piston has a differential screw thread having an equivalent pitch of 1/112 in. One revolution of this piston raises the mercury level in a column by about 0.0078 mm.

(4) The cathetometer had to be rotated through a small angle of about 20° in order that the two outer columns could be read with the same cathetometer. Only precision tests could indicate the reproducibility of a reading after the cathetometer had been turned through this angle.

The precision tests were carried out in the following manner. The mercury levels were adjusted, using the "make-up" cylinder, to give the correct sighting in the fixed telescope, sighted on the central column of the manometer. All three of the columns were open to the atmosphere. The levels of the mercury in the two outer columns were then measured in turn. Using the "make-up" cylinder, the mercury level in the three columns was altered and the above procedure repeated. This procedure was repeated 33 times for one setting of the fixed telescope; in each case the adjustment of the cathetometer was random. That is, for random adjustment the telescope on the cathetometer was brought into position by sliding it indiscriminately from either direction, upward or downward. The results of these tests can be seen on the frequency graphs for both columns, (see figure (A.7.1.) The standard deviations for columns (1) and (2) were ± 0.0261 mm and ± 0.0223 mm respectively.

The above procedure was followed, but with the adjustment of the telescope being made only in the downwards direction. A frequency graph for the 50 sightings that were made in this way is shown in figure (A7.2). The standard deviations for columns (1) and (2) were ± 0.016 mm and ± 0.0119 mm respectively.

A sighting precision was then carried out, on the two outer columns, in which the cathetometer body was not rotated. The mercury levels were kept constant and the columns were dealt with separately, the telescopes being adjusted only in a downward direction. The sightings were carried out 50 times for each column, the frequency graphs for which are given in figure (A7.3). The standard deviations



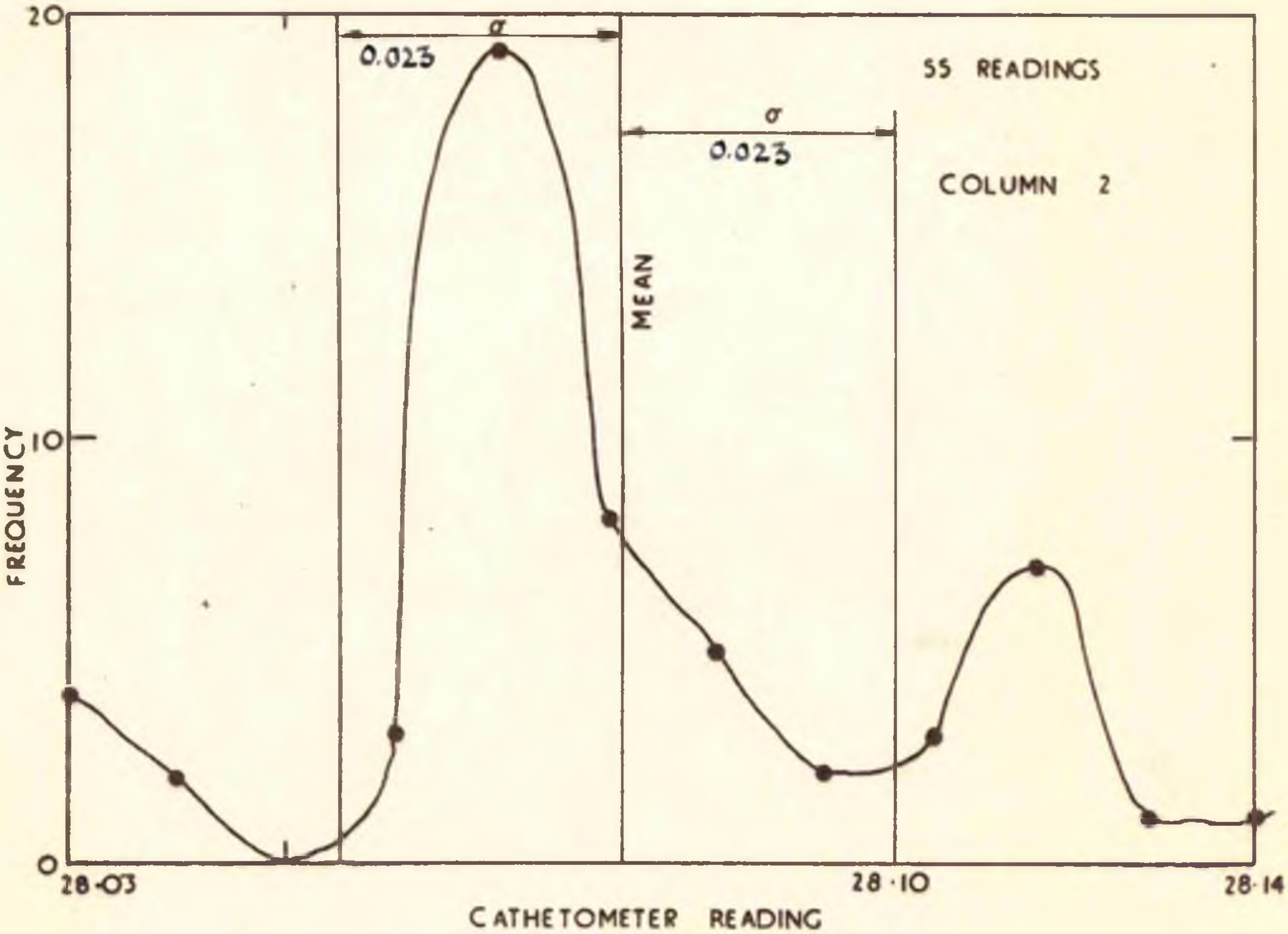


FIG A7.1 ZERO CHECK (RANDOM)

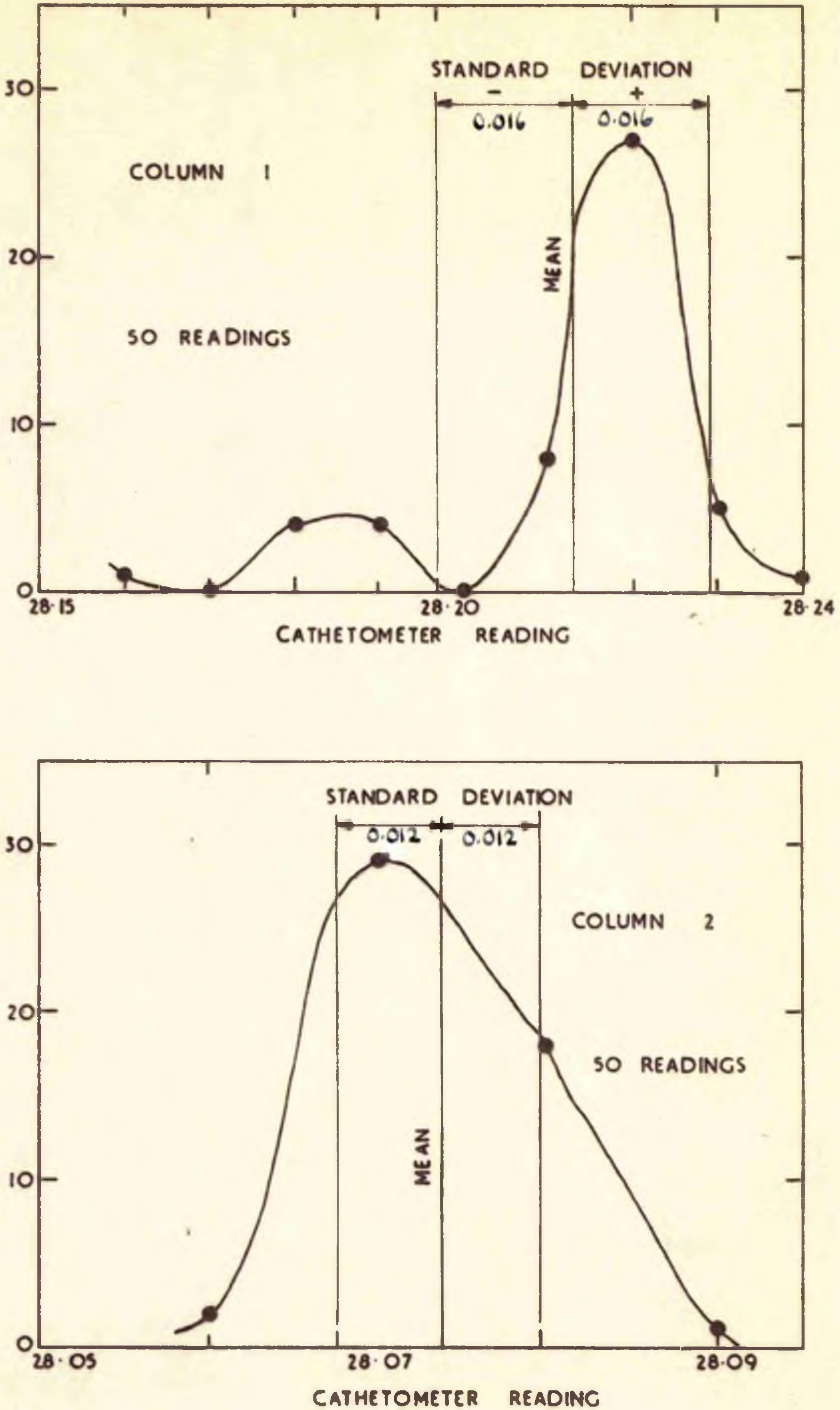


FIG A7. 2 ZERO CHECK — DOWNWARDS ONLY

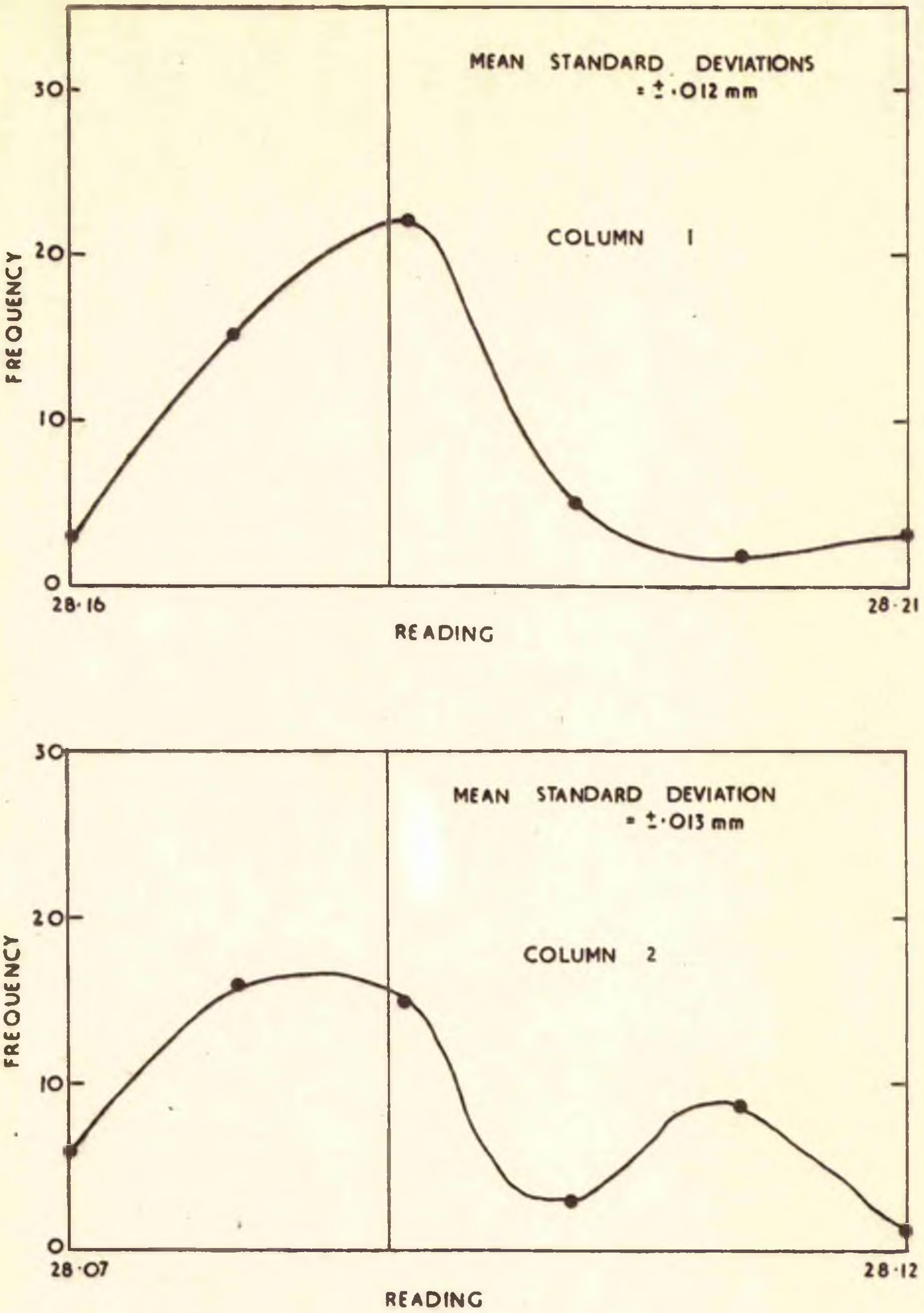


FIG A7. 3 SIGHTING PRECISION

for columns (1) and (2) were ± 0.0116 mm and ± 0.0132 mm respectively.

Therefore the expected precision (not accuracy) for column (1), using downwards readings only, is equal to the sum of the zero precisions plus the sighting precision, which is, ± 0.016 ± 0.0116 = ± 0.0276 mm.; and likewise for column (2), ± 0.0119 ± 0.0132 = ± 0.0251 mm. These precisions will apply to all readings and therefore it is better to use as large pressure drops as possible, in order to make this inherent uncertainty as small as possible. A graph of the percentage precision against the pressure drop measured, for columns (1) and (2), is given on figure (A7.4).

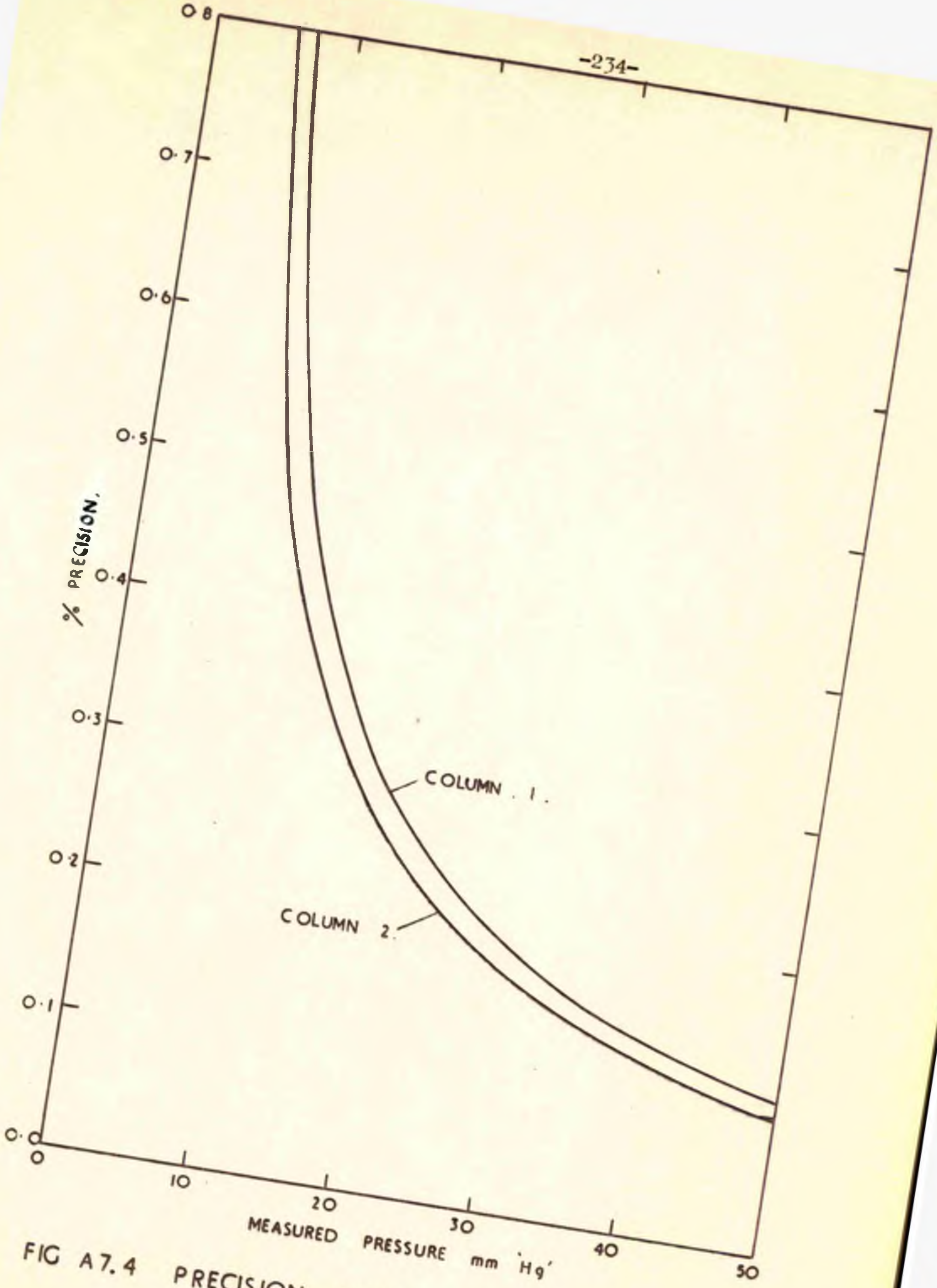


FIG A7.4 PRECISION CURVES FOR PRESSURE MEASUREMENT

Appendix (8)

Specimen Calculation for the Single Capillary Glass Apparatus

The calculation of the viscosity of steam at atmospheric pressure and 173°C (the first determination made using capillary No. 2, the 1.5 mm nominal bore capillary), will be presented in detail in this appendix. Commencing with the flow equation

$$\eta = \frac{\pi\, d^4 g\, \Delta P\, (1 + 3\alpha\, \Delta t)(1 + 8\, \varepsilon/d)}{128\, Q\, f\, l\, V} - \frac{Q\, m\, f}{8\, \pi\, l\, (1 + \alpha\, \Delta t)}$$

For capillary No. 2, d = 1.51048 mm. and l = 302.63 mm. The acceleration due to gravity 'g' at Glasgow University = 981.563 cm/sec^2. The value of the coefficient of linear expansion for the material of the capillaries is 3.3 x 10^{-6} per °C. Assuming a value for the end correction constant 'm' of 1.12, then

$$\eta = \frac{\pi\, (\, 1.51048\,)^4\, 10^{-4}\, 981.563\, \Delta P\, (\, 1 + 9.9\, 10^{-6}\, \Delta t\,)(\, 1 + 8\, \varepsilon/d\,)}{128\quad 30.263\quad Q\, f\, V}$$

$$- \frac{1.12\quad Q\quad f}{8\, \pi\quad 30.263\, (\, 1 + 3.3\, 10^{-6}\, \Delta t\,)}$$

$$= \frac{41.4396\quad 10^{-5}\, \Delta P\, (\, 1 + 9.9\, 10^{-6}\, \Delta t)(\, 1 + 8\, \varepsilon/d\,)}{Q\, f\, V} - \frac{1.47256\, Qf}{(1 + 3.3\, 10^{-6} \Delta t)}$$

where ε is obtained from equation (5.18).

The weight of condensate collected in 822.4 seconds was 9.7920 g. giving the mass flow rate Q as 0.0119076 g/sec. The atmospheric pressure = 757.45 mm. of Hg. at 23°C and, with the N.P.L. correction for the barometer for these conditions, 757.45 - 2.931 = 754.52 mm. of Hg. at 0°C. The density of mercury at 0°c = 13.5955 g/cm^3, therefore

$$Pa = 754.52 \times 13.5955 = 1025.81\ \text{gf/cm}^2.$$

The pressure head between the exit of the capillary and the ambient conditions was 1.87 mm. of Hg. at 113.8°C. The density of mercury at 113.8°C. = 13.3140 g/cm^3, therefore

$$P' = 0.187 \times 13.3140 = 2.4950 \, gf/cm^2.$$

The pressure drop across the capillary = 7.39 mm of Hg. at 117.1°C. The density of mercury at 117.1°C. = 13.3109 g/cm^3, hence the pressure drop across the capillary $\underline{\Delta P = 13.3109 \times 0.739 = 9.8368 \, gf/cm^2}$.

The mean pressure in the capillary = $Pa + P' + \Delta P/2$

$$= 1023.81 + 2.495 + 4.918 = 1033.223 \, gf/cm^2$$

The specific volume of the steam at 173°C and 1033.22 g/cm^3 (interpolated from the 5th edition of the V.D.I. tables (166) = $\underline{2021.86 \, cm^3/g}$.

The buoyancy correction for the weighings 'f'

$$= \frac{(1 - \rho a/\rho wt)}{(1 - \rho a/\rho w)}$$

where $\rho wt = 8 \, g/cm^3$, $\rho w = 0.9973 \, g/cm^3$ and ρa the density of damp air

$$= \frac{(\rho d (H - 0.378 \, p)}{H}$$

The density of dry air $\rho d = 0.001183 \, g/cm^3$ and the (saturated) vapour pressure of water $p = 21.02$ mm of Hg, from which $\rho a = 0.001171 \, g/cm^3$.

Therefore the buoyancy correction $f = \frac{(1 - 0.001171/8)}{(1 - 0.001171/0.9973)} = \underline{1.00103}$

The correction for the thermal expansion of the capillary

$$= (1 + 3.3 \; 10^{-6} \; 173°C.) = (\underline{1.000571})$$

$$\text{and } (1 + 9.9 \; 10^{-6} \; 173°C.) = (\underline{1.001713})$$

The slip correction, $(1 + 8 \, \varepsilon /d) = (1 + 8 \times 0.112 \times 10^{-3}/1.51048)$ $= \underline{1.000594}$, where ε in the above is obtained from equation (5.18), see figure (5.1) for values of ε .

Therefore

$$\eta = \left\{ \frac{41.4396\ 10^{-5}\ 9.8368\ \ 981.563\ \ 1.001713\ \ 1.000594}{0.0119066\ \ 1.00103\ \ 2021.86} \right\}$$

$$- \left\{ \frac{1.47256\ \ 0.0119066\ \ 1.00103}{1.000571} \right\}$$

$$= (\ 1.69442 - 0.17541\ )\ 10^{-4}\ \text{g/cm.sec.}$$

$$= \underline{\underline{1.51901\ \ 10^{-4}\ \ \text{g/cm.sec}}}.$$

The viscosity of steam at atmospheric pressure and 173°C

= 1.519 x 10^{-4} poise.

Appendix (9)

Specimen Calculation for the Series Capillary Quartz Apparatus

The viscosity fo steam at atmospheric pressure and at a temperature of 1070.4°C. (the highest nominal temperature at which measurements were made) will be presented in detail in this appendix. The capillaries used at this temperature were Nos. (Ca) and (Cb), having bores of 1.96706 mm. and 1.95260 mm., and lengths of 374.6 mm. and 229.9 mm. respectively.

Commencing with the flow equation

$$\frac{\pi d_1^4 g(1+3\alpha\Delta t)(1+8\varepsilon/d)}{128(l_1-l_2)Qf}\left\{\left(\frac{\Delta P_1}{V_1}\right)-\left(\frac{[d_1/d_2]^4\Delta P_2}{V_2}\right)\right\}$$

The acceleration due to gravity 'g' at Glasgow University = 981.563 cm/sec^2. The value of the coefficient of thermal expansion α for quartz is 0.54×10^{-6} per °C. Then

$$\frac{\pi(1.96706)^4\times10^{-4}\;981.563\;k_t\;k_s}{128(374.6-229.9)Qf}\left\{\left(\frac{\Delta P_1}{V_1}\right)-\left(\frac{1.95260}{1.96706}\right)^4\left(\frac{\Delta P_2}{V_2}\right)\right\}$$

where k_t is the correction for the thermal expansion of the capillaries and equals $(1+3\times0.54\times10^{-6}\times1070.4°C) = \underline{1.00173}$. k_s is the correction for slip at the walls of the tube and equals $(1+8\varepsilon/d)$ $=(1+8\times0.663\times10^{-3}/1.960) = \underline{1.00265}$. The value of the slip factor ε was obtained from equation (5.18) i.e. $\varepsilon = 0.81\times2.128\frac{\eta}{\sqrt{p\rho}}$, η being taken from Shifrin's data, refer to figure (5.1) for graph of ε against temperature. Therefore

$$\eta = \frac{2.49265 \times 10^{-3} \; k_t \; k_s}{Q \; f} \left\{ \left(\frac{\Delta P_1}{V_1} \right) - \left(\frac{(\; 0.970923 \;) \; \Delta P_2}{V_2} \right) \right\}$$

The weight of condensate collected in 694.0 seconds was 10.3252 g. giving a mass flow rate <u>'Q' of 0.0148778 g/sec.</u>

The atmospheric pressure = 765.30 mm. of Hg. at 24.5°C., with the N.P.L. correction for the barometer at these conditions, it equals 765.30 - 3.152 = 762.15 mm. of Hg. at 0°C. The density of mercury at 0°C. = 13.5955 g/cm^3, therefore Pa = 762.15 x 13.5955 = 1036.18 gf/cm^2 .

The pressure head between the exit of the downstream capillary and the ambient conditions was 5.31 mm. of Hg. at 118.2°C. The density of mercury at 118.2°C. = 13.3083 g/cm^3 , therefore P' = 0.581 x 13.3083 = 7.067 gf/cm^2.

The pressure drops across the downstream and upstream capillaries were ΔP_1 = 35.86 mm. of Hg. at 120°C. and ΔP_2 = 22.31 mm. of Hg. at 120°C. respectively. The density of mercury at 120°C. = 13.3040 g/cm^3, therefore ΔP_1 = 35.86 x 13.3040 <u>= 47.7081 gf/cm^2.</u>

ΔP_2 = 22.31 x 13.3040 <u>= 29.6812 gf/cm^2.</u>

The mean pressuresin the downstream and upstream capillaries were

$P_{1,m}$ = 1036.18 + 7.067 + 47.7081/2 = 1067.10 gf/cm^2. and

$P_{2,m}$ = 1036.18 + 7.067 + 47.7081 + 29.6812/2 = 1105.80 gf/cm^2.

The specific volumes of the steam at a temperature of 1070.4°C. and pressures of 1067.10 gf/cm^2 and 1105.80 gf/cm^2, the mean conditions in the capillaries, were V_1 = 5930.57 cm^3/g and V_2 = 5712.24 cm^3/g z extrapolated from the 5th. edition of the V.D.I. tables (166))

The bouyancy correction 'f' for the weighings = $\dfrac{(\; 1 - \rho a/\rho wt \;)}{(\; 1 - \rho a/\rho w \;)}$

where ρwt is the density of the weights = 8 g/cm^3 (supplied by the manufacturer's of the balance), the density of water at 24.5°C. $\rho w = 0.9972\ g/cm^3$ and ρa is the density of damp air $= \rho d \{ H - 0.378\ p \} / H$. At a pressure of 762.15 mm. of Hg. and a temperature of 24.5°C. the density of dry air = 0.001189 g/cm^3. and the saturated vapour pressure of water = 22.51 mm. of Hg. From which

$$\rho a = \frac{0.001189 \ \{ 762.15 - 0.378 \times 22.51 \}}{762.15} = 0.001172\ g/cm^3$$

$$\text{and} \qquad f = \frac{\{ 1 - 0.001172/8 \}}{\{ 1 - 0.001172/0.9972 \}} = \underline{1.0011}$$

Therefore

$$\eta = \frac{2.49265\ x10^{-3}\ 1.00173\ 1.00265}{0.0145778 \quad 1.0011} \left\{ \left(\frac{47.7081}{5930.57} \right) - \left(\frac{0.970923 \quad 29.6812}{5712.24} \right) \right\}$$

$$\underline{\eta = 5.03793\ x10^{-4}\ g/cm.sec.}$$

<u>The viscosity of steam at atmospheric pressure and a temperature of 1070.4°C. = 5.038 x10^{-4} poise.</u>

Appendix (10)

Working Equations

Glass Apparatus

$$\eta = \frac{\pi d^4 g \Delta P (1 + 3\alpha\Delta t)(1 + 8\varepsilon/d)}{128 \; l \; f \; Q \; V} - \frac{m Q f}{8 \pi l (1 + \alpha\Delta t)}$$

$$\eta = \frac{K \Delta P (1 + 3\alpha\Delta t)(1 + 8\varepsilon/d)}{f Q V} - \frac{k' Q f}{(1 + \alpha\Delta t)}$$

where α is the coefficient of linear expansion for the glass and equals $3.3 \; 10^{-6}$ per °C.

Values for the constants (all values are in gram. cm. sec. units)

Capillary No.	$K \times 10^4$	$k' \times 10^3$
1	0.80715	1.49652
2	4.14397	1.47408

Quartz Apparatus

$$\eta = \frac{\pi d_1^{\,4} g (1 + 3\alpha\Delta t)(1 + 8\varepsilon/d)}{128 (l_1 - l_2) Q f} \left[\left\{ \frac{\Delta P_1}{V_1} \right\} + \left\{ \frac{\{ d_2/d_1 \} \; \Delta P_2}{V_2} \right\} \right]$$

$$\eta = \frac{K_1 \{ 1 + 3\alpha\Delta t \} \{ 1 + 8\varepsilon/d \}}{Q f} \left[\left\{ \frac{\Delta P_1}{V_1} \right\} + \left\{ \frac{K_2 \Delta P_2}{V_2} \right\} \right]$$

where α is the coefficient of linear expansion for the quartz and equals 0.54×10^{-6} per °C.

Values for the constants/.

Values for the constants (all values are in gram. cm. sec. units).

Capillary No.	K_1 x10^3	K_2
3a and 4b.	0.298473	0.910362
2a and 2b.	0.732529	0.956269
8a and 8b.	1.939600	1.035141
Ba and Bb.	0.897794	1.023780
Aa and Ab.	0.155585	1.069510
Ca and Cb.	2.492650	0.970925

Appendix (ii)

End Correction Constant 'm'

An attempt to obtain the value of the end correction constant 'm' for square-cut ended tubes was made using the two capillary apparatus. The original intentions were (1) to determine the value of the viscosity of the steam at given conditions and then feed this data back into the individual flow equations for the two capillaries and hence calculate the value of 'm', or (2) to add the two simultaneous flow equations and 'η' and hence obtain an equation for 'm'. Unfortunately no estimation was made during the initial development stage for the expected precision of those methods. When the actual experimental data was used to determine 'm', it was found that the value obtained varied by as much as 50 % in the extreme cases. A rough estimation of the precision was then made (based on the observed fact that the precision of the mass flow collection was not better than ± 0.5 % in the majority of cases); it predicted that it could be even larger than the experimental value.

It is difficult to make a rigorous estimation of the precision of the two methods for finding 'm', since the accuracy and precision of 'm' depends on the overall value of the end correction $(m\,Q)/(8\pi\, l)$, in the flow equation. It is obvious that for given values of 'm' and 'l', as Q is decreased, the ratio $[mQ/8\pi\, l]/\eta_p$ (where $\eta_p = \{\pi d^4 g\, \Delta P/128 Q l V\}$ also decreases and consequently any experimental scatter in the values of Q or η (the major variables in these equations) will be greatly amplified when finding 'm'.

After three capillary sizes had been used in the series capillary system, it was decided that when possible the mass flow through the capillaries, at given conditions, would be varied in a methodical manner in order that the end correction constant could be checked. That is, the graphical solution for 'm' and 'η' would be attempted using the equation $\eta = \eta_p - mQ/8\pi l$, where η_p is the "Poiseuille viscosity". This was actually attempted for the last three capillary sizes. After the points from several of these sets of results had been plotted, it was apparent that η could be ascertained to within a reasonable tolerance. Unfortunately with the experimental scatter it was impossible to determine even a rough value for 'm', since almost any number of straight lines having different slopes could be fitted to the points and still give reasonable intercepts of the η axis. It was therefore decided to attempt to fit straight line equations to the points using the least squares fit method.

Three graphs of η_p against mkQ are given as examples on figures (A 11.1) to (A 11.3). Straight lines, which are shown on the graphs, were obtained by fitting the data from individual capillaries and both the capillaries together, using the least squares method of curve fitting.

The result of least squares fitting to the data gives, as before, an overall uncertainty in the value of 'm' of about 80 %, which had been previously predicted. The extrapolated value of 'η', neglected the obviously incorrect values, agreed within about ± 2 % in the majority of cases, which is only to be expected with the number

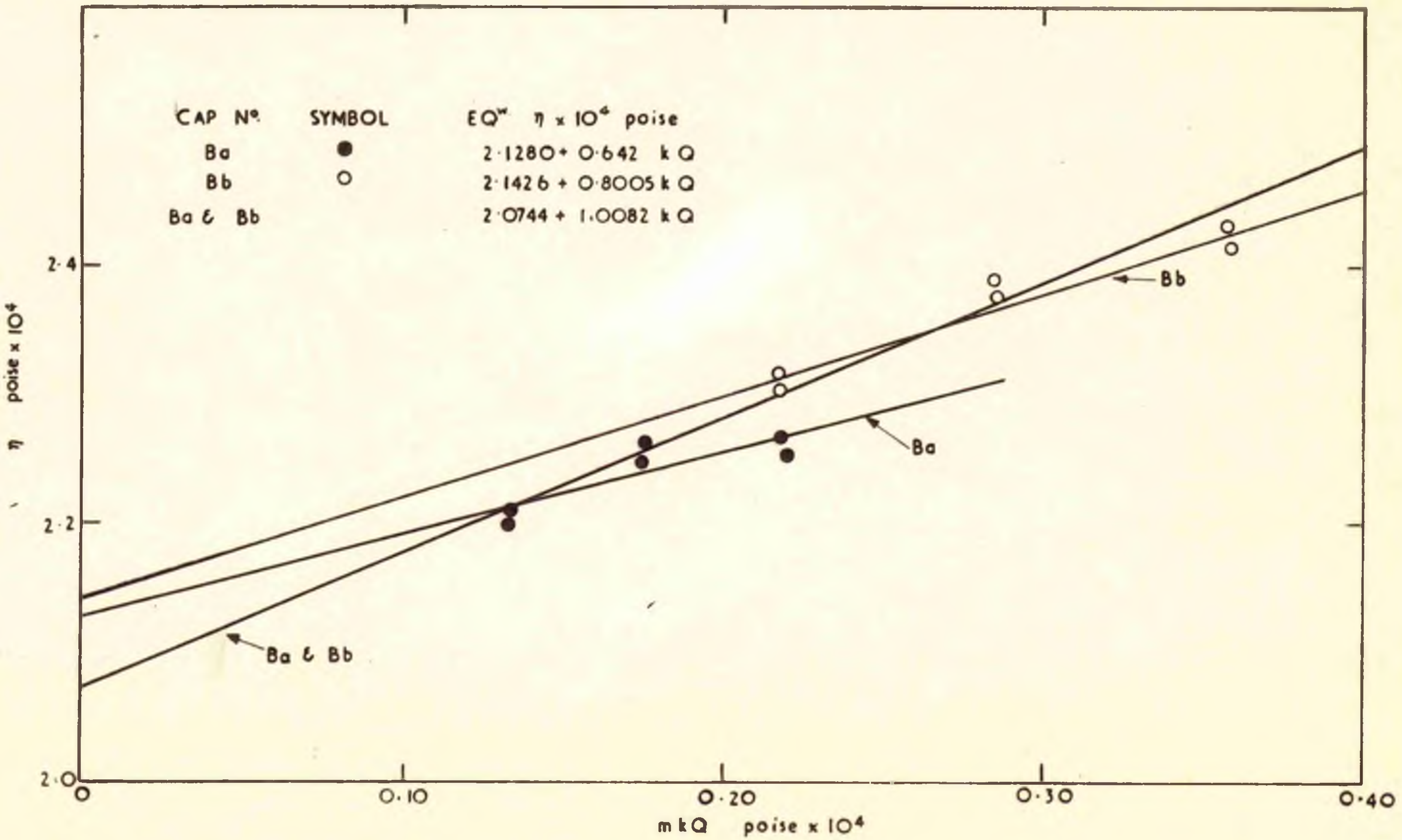


FIG A II. I GROUP 45

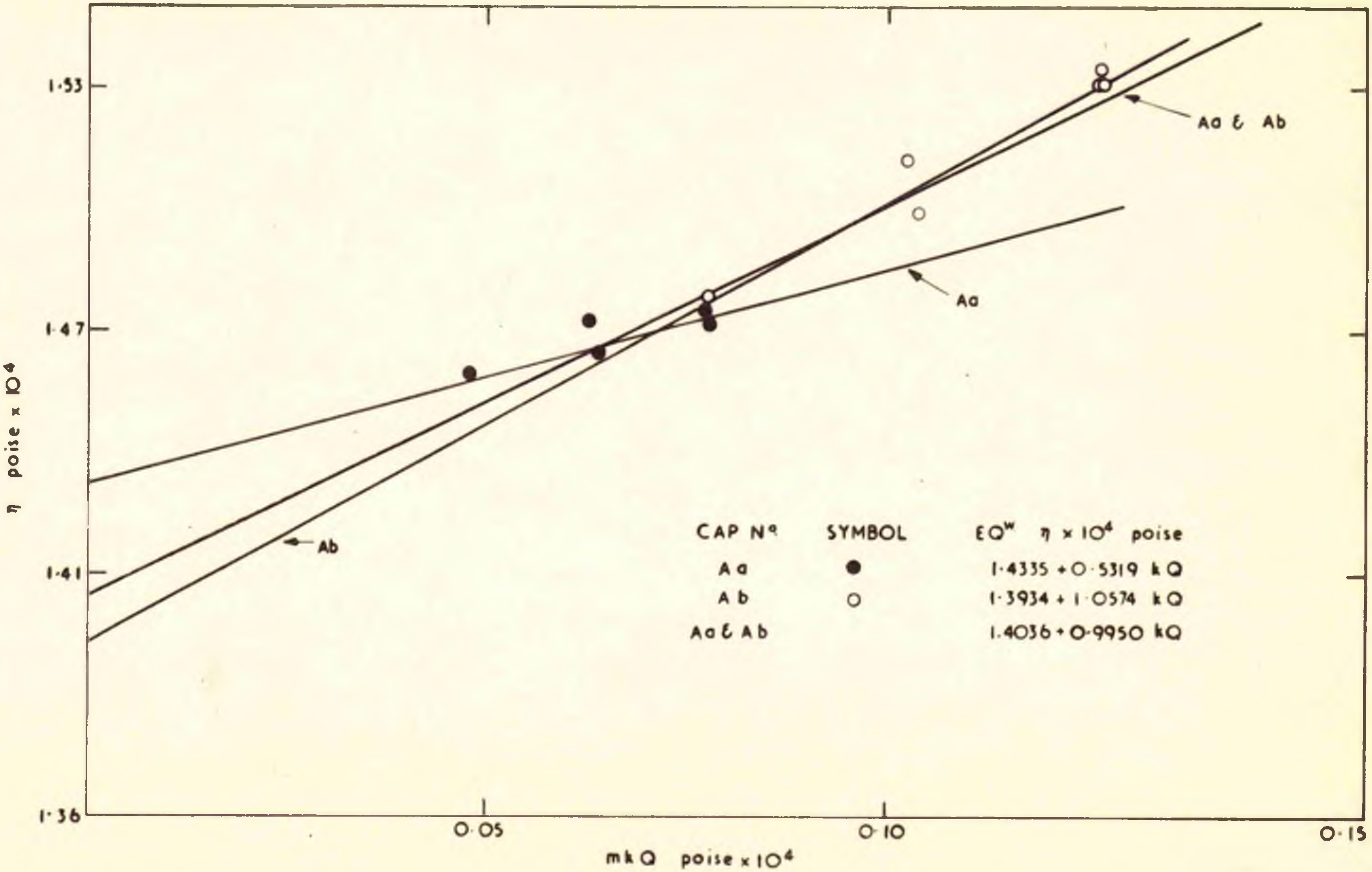


FIG A11.2 GROUP 47

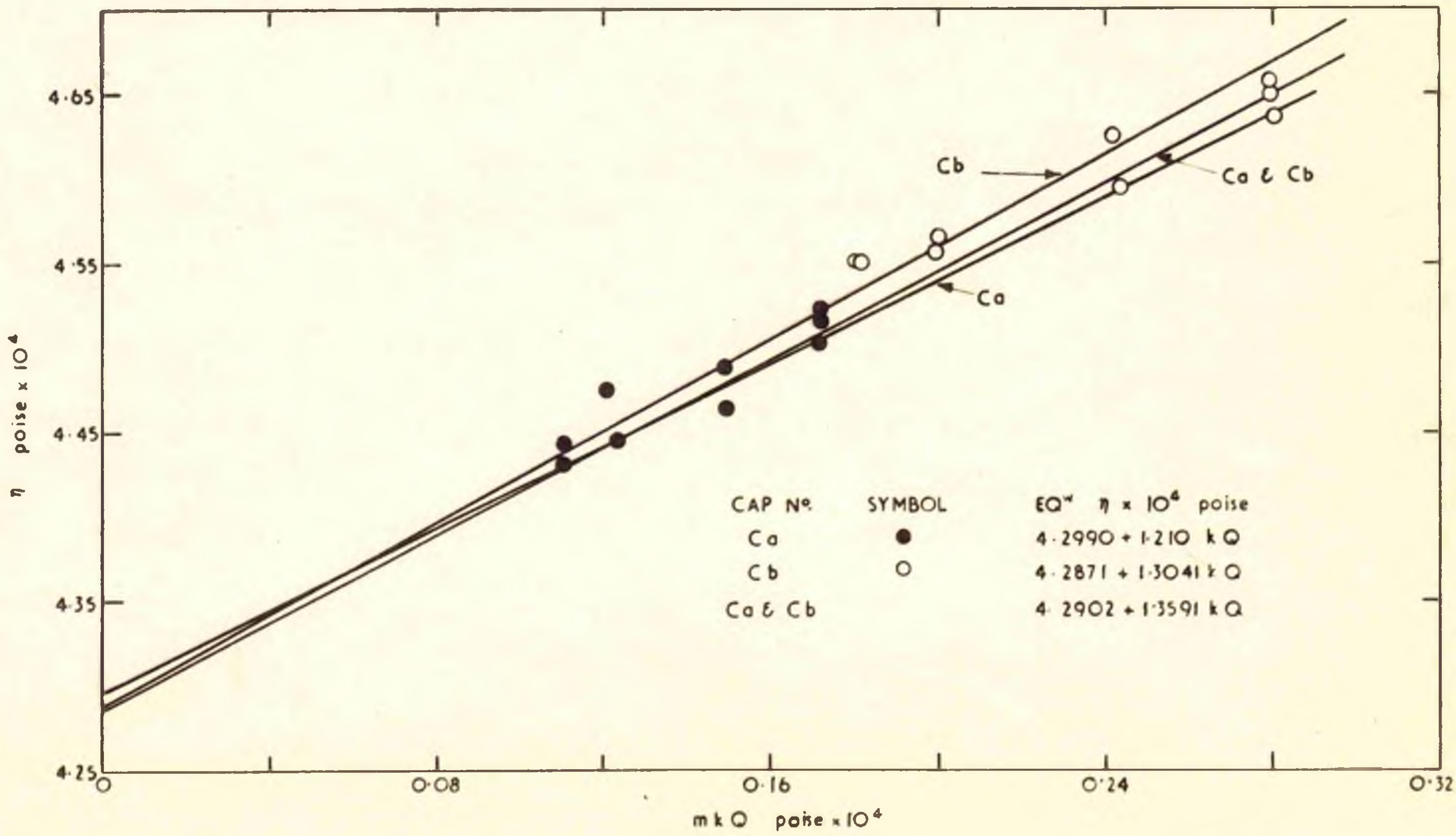


FIG A II. 3 GROUP 5B

of determinations in relation to the experimental scatter.

The overall conclusion was that, with the experimental scatter obtained, unless a much larger number of determinations were made, at a given temperature, very little could be deduced. No recommendation for the value of 'm' could be made. However, it was noticed that, even with the relatively few determinations made at a given temperature, a value for 'η' could be extrapolated with some accuracy.

It was felt that the graphical or semi-graphical methods for obtaining 'η' and 'm' could not be used with the present apparatus, unless the experimental scatter could be reduced and/or a much larger number of individual determinations were made at a given temperature.

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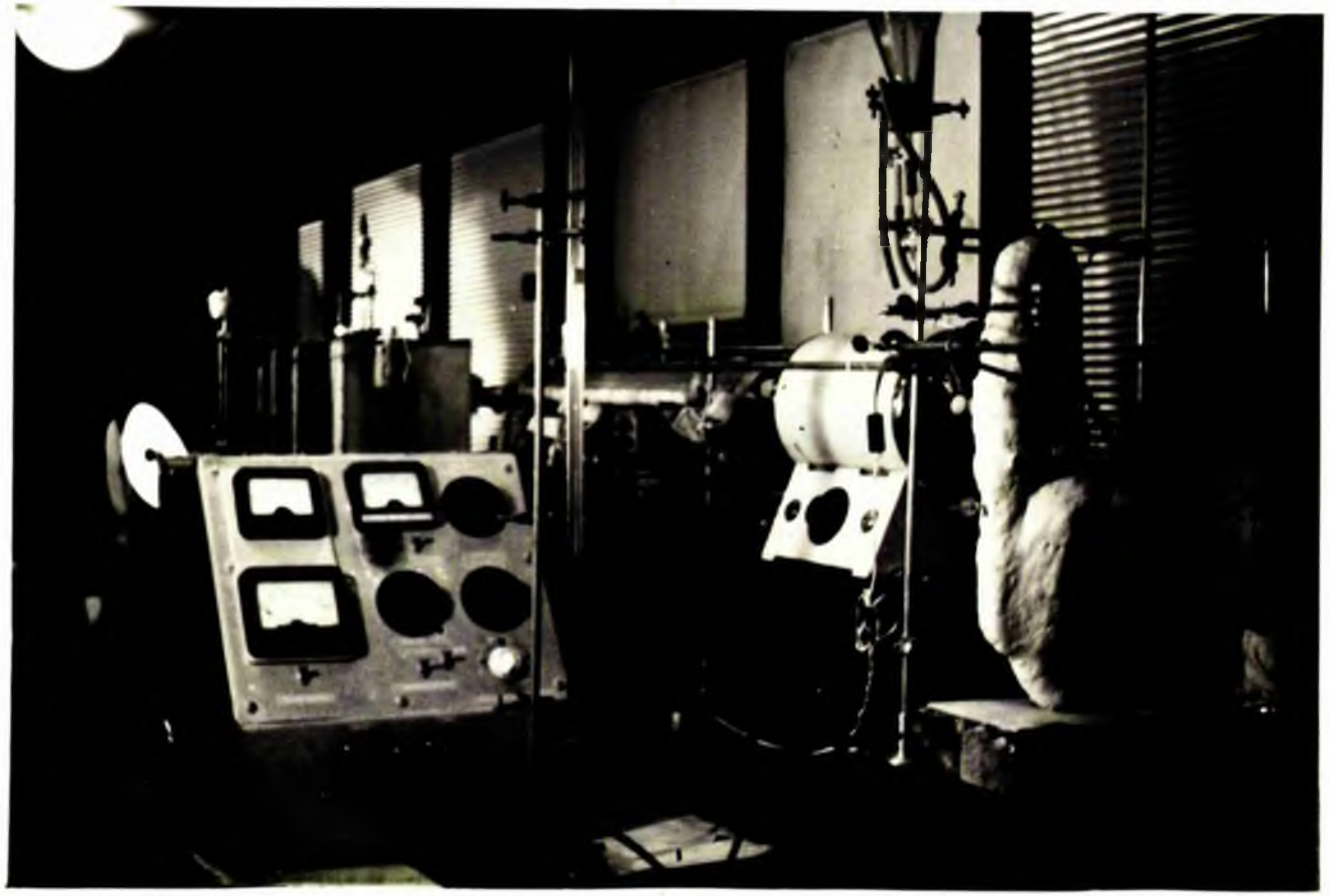
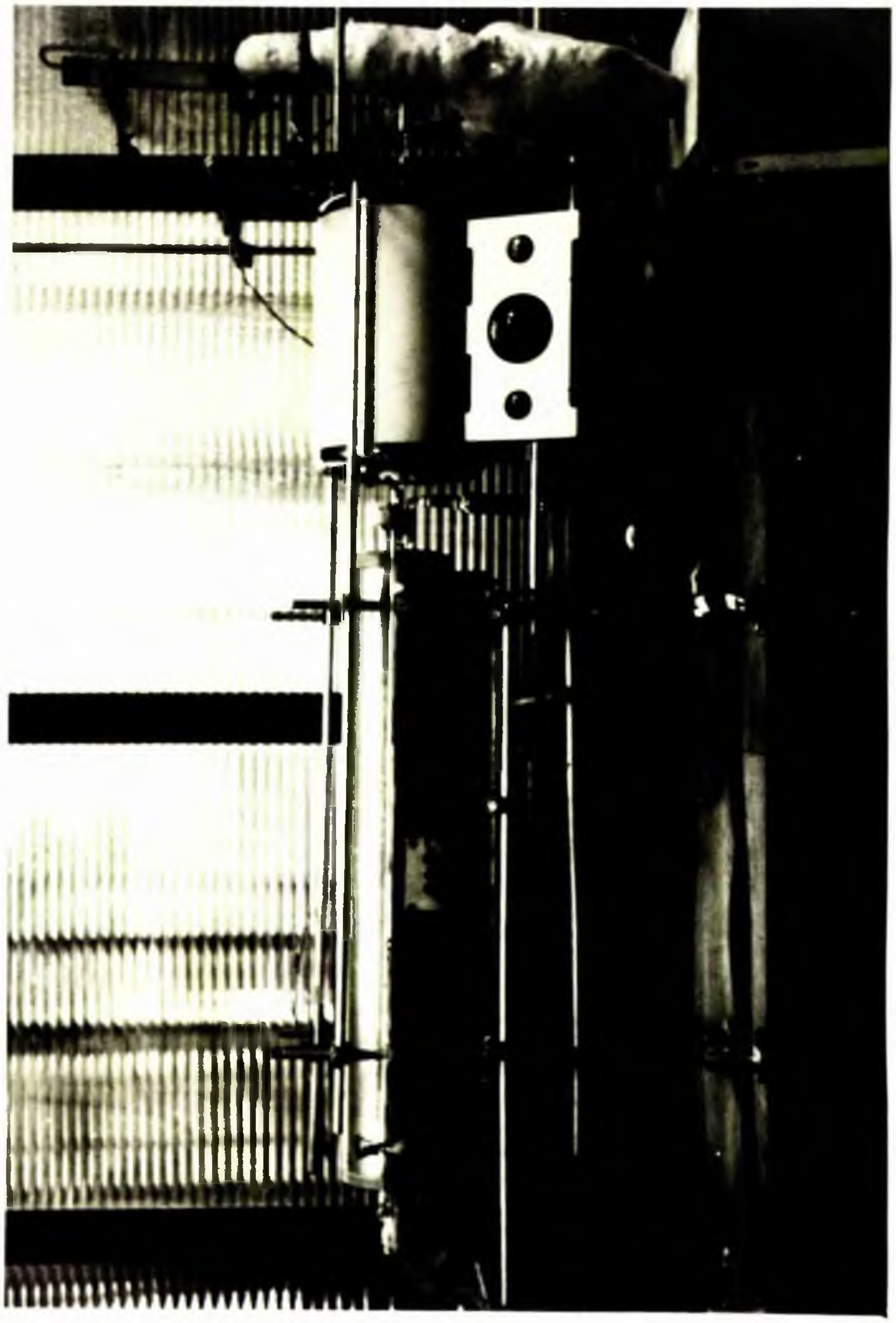
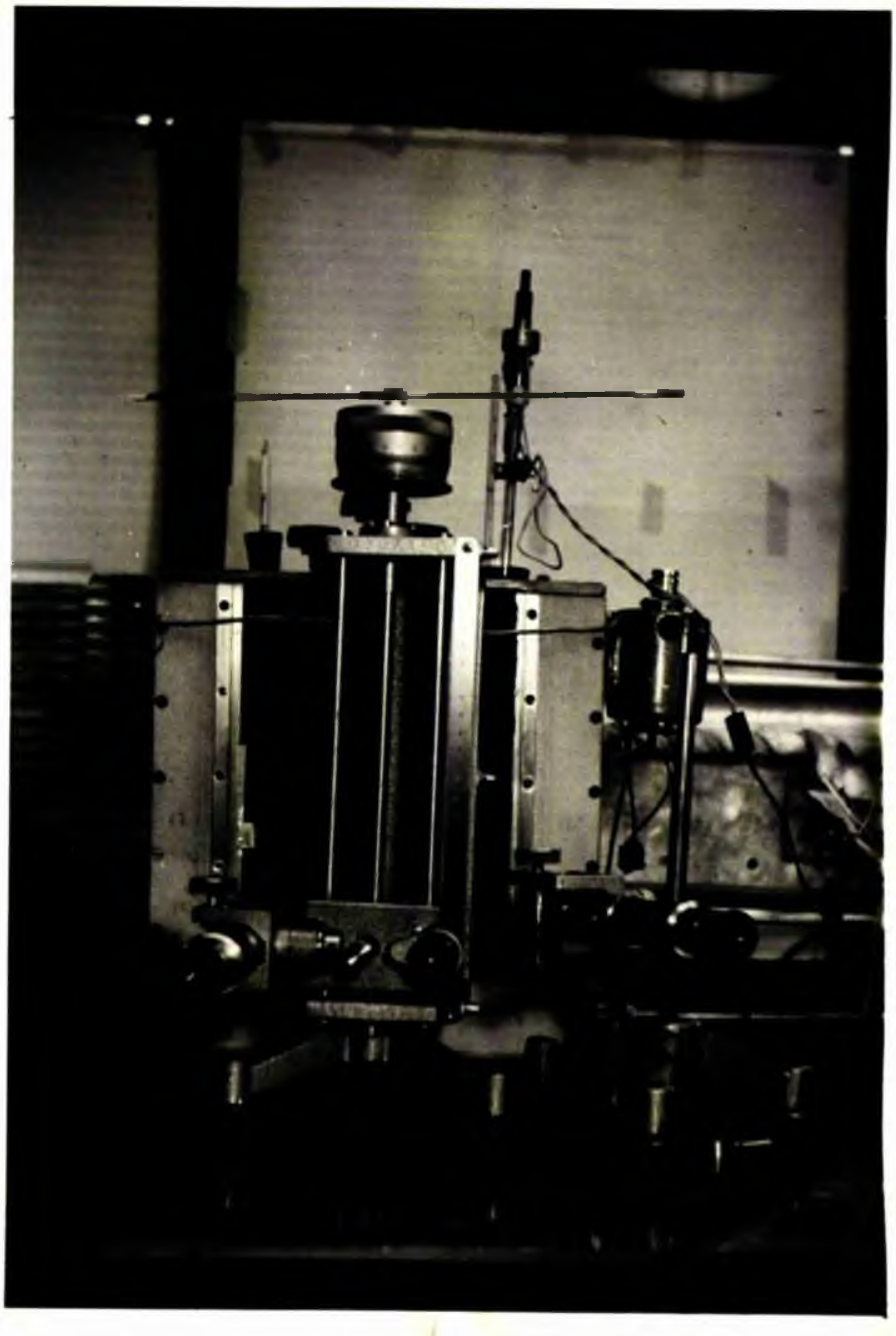


General View of the Single Capillary Glass Apparatus



Single Capillary Glass Apparatus - Auxilliary Equipment Removed

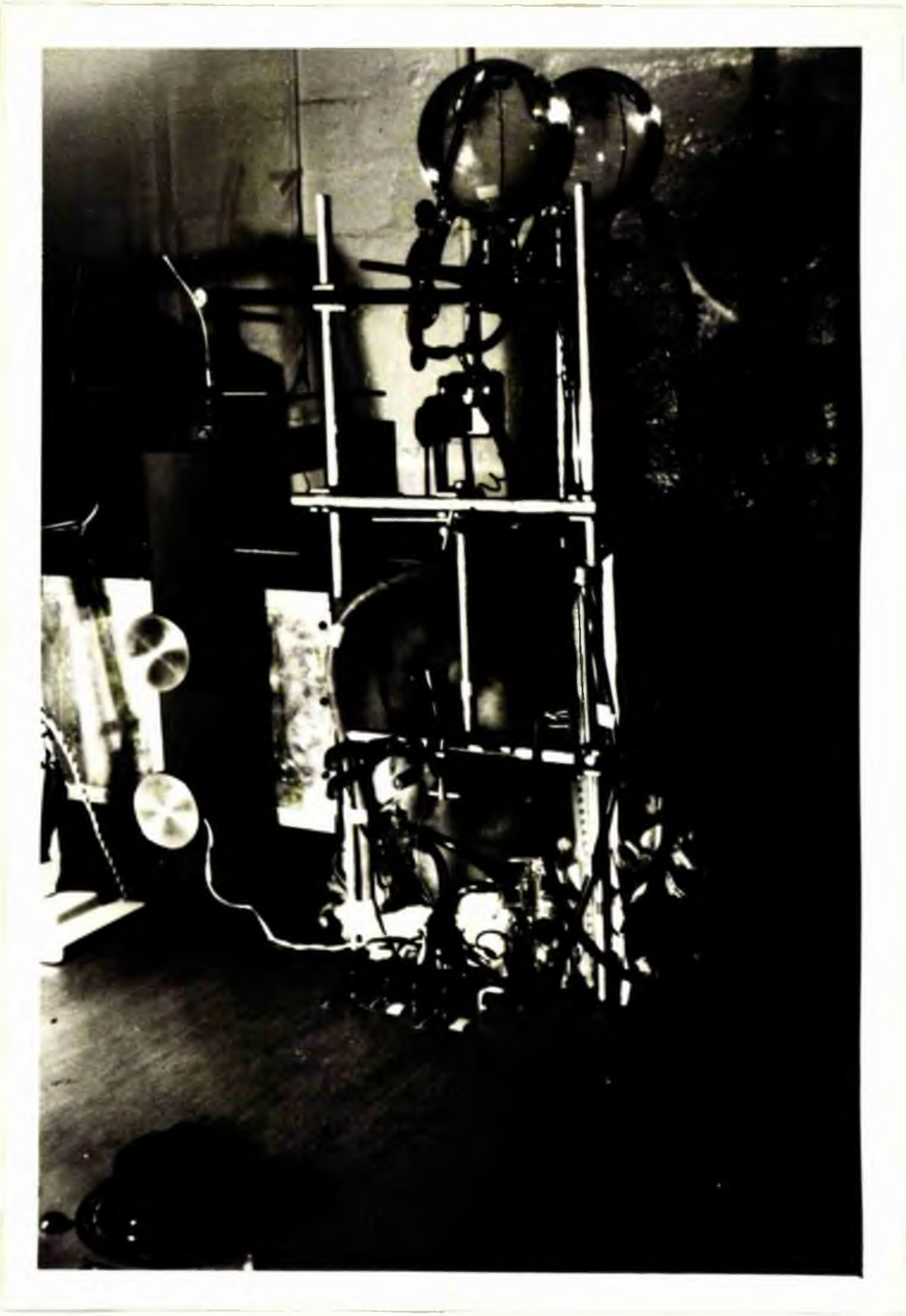


Manometer (Single Capillary Glass Apparatus)

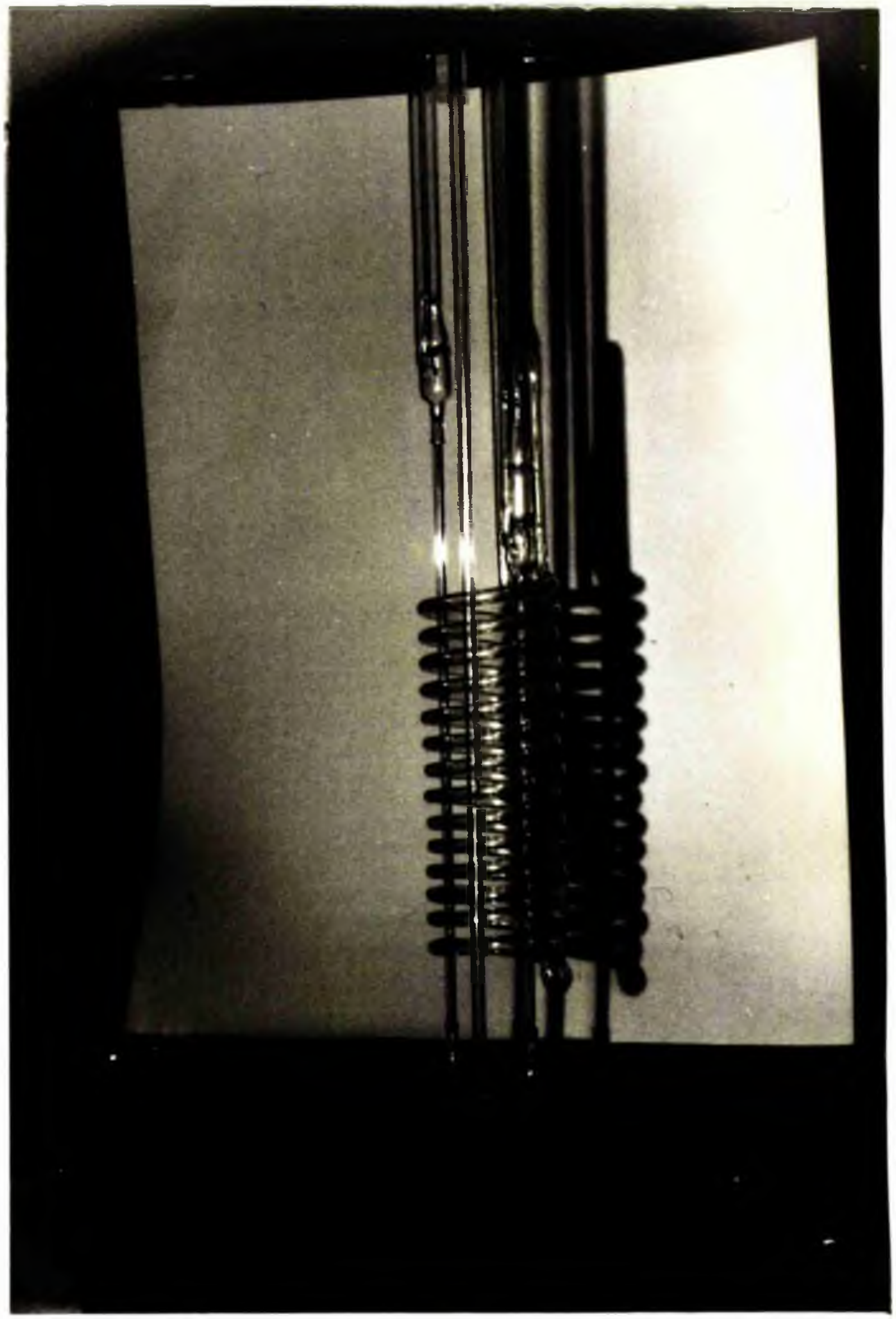
Condensate Collection Arrangement (Single Capillary Glass Apparatus)

Control Panel (Series Capillary Apparatus)

Manometer and Boiler (Series Capillary Apparatus)

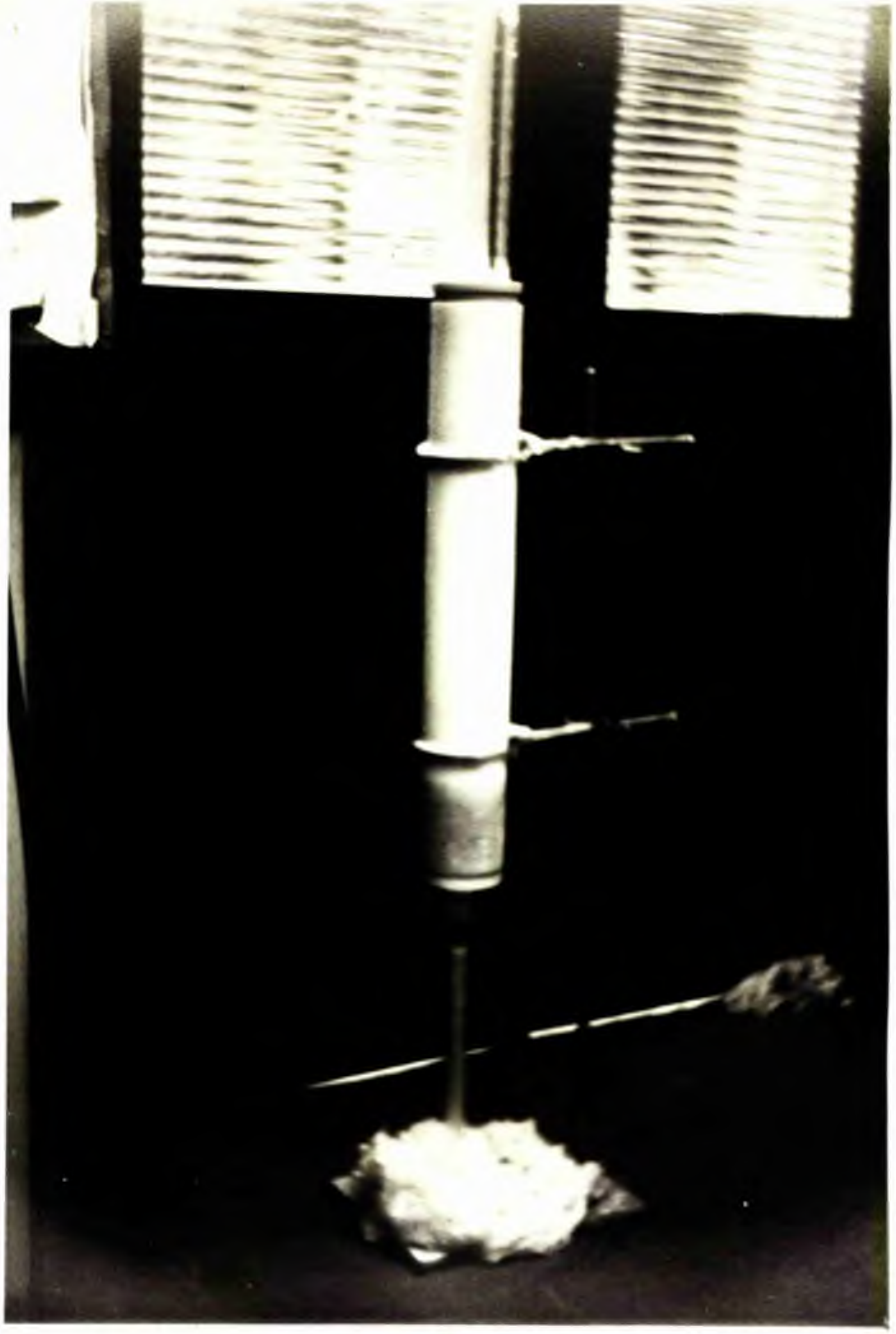


oiler and Distilled Water Storage Vessels (Series Capillary Apparatus)

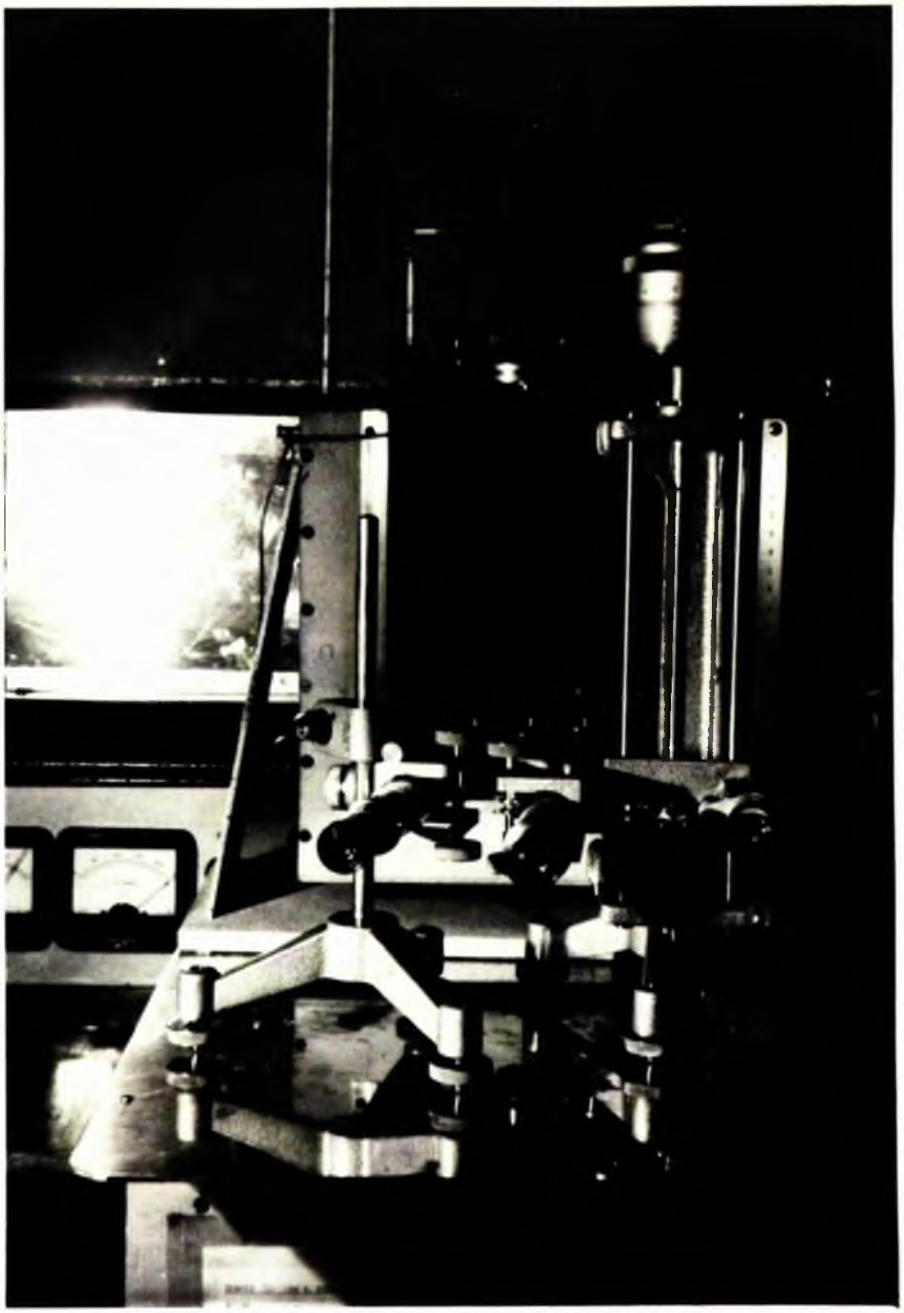


Assembled Viscometer (Series Capillary System)

Component Parts (Series Capillary Viscometer)



Series Capillary Viscometer Cell



Manometer (Series Capillary Apparatus)