ON THE MEASUREMENT OF THE ABSOLUTE VISCOSITY OF SOME REFRIGERANTS

ON THE MEASUREMENT OF THE ABSOLUTE VISCOSITY OF SOME REFRIGERANTS

By

SURESH CHANDRA ASRANI
B.E.

A Thesis
Submitted to the Faculty of Graduate
Studies in Partial Fulfilment of the
Requirements for the Degree
Master of Engineering
McMaster University,
Hamilton, Ontario.

MASTER OF ENGINEERING (1969)
(Mechanical Engineering)

McMASTER UNIVERSITY
Hamilton, Ontario.

TITLE: ON THE MEASUREMENT OF THE ABSOLUTE VISCOSITY OF SOME REFRIGERANTS.

AUTHOR: SURESH CHANDRA ASRANI
B.E. (Birla Institute of Technology & Science)

SUPERVISOR: Dr. B. Latto

NUMBER OF PAGES: xi, 125

SCOPE AND CONTENTS:

Using a series capillary viscometer, absolute viscosity of two halogenated hydrocarbon referigerants, R-13 and R-500 in vapour phase, has been measured at atmospheric pressure and in the temperature range -53.25 to 87.19°C and -28.64 to 87.38°C respectively. The estimated maximum error is of the order of ± 1.64%. The experimental results have been compared with the previous data.

Viscosity values have been correlated with polynomials of degree 1 to 5 and an approximate form of Sutherland equation $\eta = A_1\sqrt{T} + B_1$.

Intermolecular force constants have been computed for R-13 by the collision theory approach, and using these constants viscosity coefficients have been reported for a temperature range of 200 to 1650°K.

ACKNOWLEDGEMENT

The author is thankful to Dr. B. Latto for his guidance. Many thanks are also due to the persons who offered assistance.

The author wishes to thank DuPont of Canada, National Bureau of Standards and Thermophysical data centre for supplying technical information.

The experimental work was supported by the National Research Council, Grant No. A2937.

TABLE OF CONTENTS

Particulars Page

9 Appendices

LIST OF FIGURES

LIST OF TABLES

NOMENCLATURE

Symbol	Description
A, A_1	Constants
B	Constant
C	Constant
C	Centigrade temperature scale
D	Constant
E	Intermolecular potential field
F	Fahrenheit temperature scale
K	Kelvin temperature scale
L	Length
Q	Volumetric flow rate
$\overline{Q}$	Mass flow rate
R	Reynold number
R-	Refrigerant
T	Absolute temperature in °K
T^*	Reduced temperature = $\frac{kT}{\varepsilon}$
V	Volume
W	Weight
$W^{(2)}(2)$	Hirschfelder, Bird, Spotz integral
X	Development length
a	Radius of capillary tube
b	Boiling properties

d	Diameter of pipe
g	Acceleration due to gravity
h	Height of mercury column
k	Boltzmann constant
$\bar{k}$	Empirical constant
m	Kinetic Energy Correction constant
$\bar{m}$	Reduced mass
n	Couette constant
P	Pressure
ΔP	Pressure difference
r	Radial distance
r_o	Low velocity collision diameter
s	Stress
t	Temperature
v	Specific volume
x	Distance
Ω	Chapman & Cowling integral
Σ	Sum
α	Coefficient of linear expansion
δ	Small increment
δ	Reduced dipole energy
ε	Maximum energy of attraction
$\bar{\varepsilon}$	Coefficient of slip correction
η	Dynamic viscosity
γ	Kinematic viscosity

θ	Time
θ	Angle
π	Constant
ρ	Density
ρ_e	Resistivity
χ	Angle
μ	Dipole moment
ζ	Orientation factor
ϕ	Angle

I INTRODUCTION

The viscosity, which could be termed as the internal friction of the fluid flow, is an important parameter in the majority of flow problems. Therefore, the economic design of any fluid flow device requires accurate values of the coefficient of viscosity.

The importance and popularity of air conditioning and refrigeration need not be overemphasized. However, very little actual research work has been done to determine the thermophysical properties of the refrigerants. Some work has been done on the correlation of viscosity data, most of which is based on the secondary measurements*. After a comprehensive literature survey, it was apparent that there was very little primary absolute viscosity data available. The majority of correlations were based on relatively scant and rather inaccurate secondary data. With this in mind, the temperature dependence of viscosity for two refrigerants, R-13 & R-500, in vapour phase with an improved accuracy using a primary method, has been obtained.

A viscometer with two capillaries in series was designed and used for this purpose. The theory of the capillary viscometer has been quite satisfactorily developed and consequently the absolute determination of viscosity was possible. This approach avoids the inaccuracy due to the

---

* Secondary measurements are obtained using an apparatus calibrated with a fluid of known viscosity whereas primary measurements are absolute measurements based on fundamental principles.

calibration of a secondary type of apparatus. The use of two capillary tubes in series eliminates the need of applying kinetic energy correction and Couette correction and thus avoids the error due to the uncertainity of the correction constants.

The viscometer used in the present work has an advantage that the capillary tubes can be replaced and that it can accomodate a wide range of the capillary tube sizes and permit determination over a wide temperature range.

Absolute viscosity of the following refrigerant vapours was determined at atmospheric pressure and the temperature ranges indicated:

R-13	-53.25 to 87.19°C
R-500	-28.64 to 87.38°C

the viscosity values were compared and correlated with other available data and intermolecular force constants were computed for R-13 using the viscosity values obtained in this work.

The viscosity of Nitrogen (99.996%)was determined at atmospheric pressure and in the temperature range of -12.13°C to 77.18°C,in order to check the reliability and accuracy of the apparatus.

2 LITERATURE SURVEY

The research on the viscosity of the refrigerants started nearly 35 years ago, but very little attention was paid to it in the early years; although, in the last 15 years more research has been done, still very few absolute viscosity data are available. A brief description of the work done so far is given here. The reader is referred to a master thesis by Al-saloum [16] and a paper by Latto and Al-saloum [17] for a comprehensive study of a number of refrigerants in the vapour and liquid phases.

One of the first papers on the viscosity of halogenated hydro-carbon refrigerants was by Hovorka and Geiger [1] in 1933, who measured viscosity of R-113 in the liquid phase, in the temperature range -30° to 9°C, using a rolling ball viscometer. No error analysis was reported.

In 1936, Awbery and Griffiths [2] measured the viscosity of R-12 in the liquid phase and in the temperature range -15° to 30°C, using a falling plug viscometer. The calibrating fluids that were used were benzene, pentane, ether and water and a variation in the calibration constant was observed. The experimental data were correlated using an equation of the form $\ln \eta = A/T + C$. No error estimate was reported.

Benning and Markwood [3], (1939) measured the viscosity of R-11, R-12, R-21 and R-22 in the vapour phase, in the temperature range 0° to 50°C and a pressure range of 1 to 9 atmospheres. Also, viscosity of R-11, R-12 and R-113 was measured in the liquid phase,

Numbers in parentheses refer to references.

the measurements in this case were made at saturation pressure and in the temperature range -30° to 60°C. A rolling ball viscometer inclined at 80° to the horizontal was used with air as the calibrating fluid for the vapour phase measurements . For the liquid phase, water and chloroform were used as calibrating liquids. Variation of calibration constant with temperature was neglected. The viscosity of calibrating fluid at a temperature t was calculated as $\eta_t = (\theta/\theta_{20})\,\eta_{20}$; where θ is time and subscript 20 is for the value at 20°C. It was assumed that $(\rho_b - \rho)/(\rho_b - \rho_c) = 1$; where ρ_b is the density of ball, ρ is the density of test fluid and ρ_c is the density of calibrating fluid. Therefore, the viscosity was calculated from the relation $\eta = (\theta/\theta_c) . \eta_c$. The experimental data were fitted to an equation of the form $\eta = A\sqrt{T} - B$ for the vapour phase and for the liquid phase the data were correlated using equation of the form $\eta = A\,e^{k/T}$, but an empirical relation of the form $\eta = \frac{c}{(A+t)^a}$ proved to be more satisfactory in correlating the results since the first equation is only suitable for correlating constant pressure viscosity data.

In 1945 Graham [4] measured the viscosity of R- C 381, both in the liquid and the vapour phases. He used a rolling ball viscometer and calibrated it using air and distilled water. The measurements were carried out at the saturation pressure and in the temperature range 0 to 71.5°C for the liquid region whereas for the vapour region the viscosity was measured at atmospheric pressure, in the temperature range 3° to 97°C. A tenfold increase in pressure resulted in 20% increase in the vapour viscosity.

Coughlin [5], (1953) measured the viscosity of R-22, R-23 and R-115 in the vapour phase, using a rolling ball viscometer. The measurements were made at atmospheric pressure and in the temperature range -36 to 60°C. The accuracy of the measurements was reported to be ± 0.4% to ± 0.8%.

Makita [6], (1954) designed a rolling ball viscometer in which the tube could be inclined at several inclinations. He measured the viscosity of R-12, R-21, R-22 in the vapour phase, in the temperature range 25° to 200°C and a pressure range of 1 to 18.39 atmospheres. The viscometer was calibrated using dry air and the variation in the calibration constant was neglected. Makita compared his results with Chapman and Cowling's collision theory and fitted equations of the form $\eta = AT^{1/2} e^{-D/T}$ and $\ln \eta = k \ln P + c$ to his results, where A, D, k and c are constants and P is vapour pressure of the liquid refrigerants. He observed that the viscosity of all the refrigerant vapours increased with increase in pressure.

In 1955 Wellman [7] measured viscosity of R-12 R-13, R-14 in the vapour phase, using a rolling ball viscometer. The measurements were carried out in the temperature range 3 to 97°C and a pressure range of 1 to 20 atmospheres. Only one measurement was made for R-114 at 30 •2°C and atmospheric pressure. Air was used as calibrating fluid and an increase of 3% in the calibration constant for an increase of 100°C in temperature was observed but with pressure no variation was detected. The data were correlated using an equation of the form $\eta \times 10^{-4} = A\sqrt{T} - B$ and the results were compared with the work of other researchers. An assessed error of ± 2.73% was reported.

Kinser [8], (1956) measured the viscosity of R-11, R-12, R-21, R-113, R-114, R-12B1 in the liquid phase, using a rolling ball viscometer (Wellman's design). The measurements were made at atmospheric pressure in the temperature range -70°C to the saturation temperature. The viscometer was calibrated using ethyl ether and a variation in the calibration constant was detected. The results were compared with other available data. The accuracy of the data were thought to be ± 4.21%. Kinser suggested that the primary method should be tried for better results.

In 1957 Lilios [9] used a rolling ball viscometer to measure the viscosity of R-12, R-12B1, R-113, R-13B1, R-C318 in the liquid phase. He also used a capillary viscometer for the measurement of the viscosity of R-12B2, R-114B2, R-12B1, R-12 in the liquid phase. The measurements were made at the atmospheric pressure, in the temperature range -70°C to the saturation temperature. The viscometer was calibrated using ethyl ether and kinetic energy corrections were neglected for the capillary viscometer. It was found that the results with the capillary viscometer were 10% lower than those obtained using rolling ball viscometer. Lilios recommended the use of capillary viscometer.

McCullum [10], (1958), measured viscosity of R-11, R-21, R-23, R-114, R-115, in the vapour phase. A rolling ball viscometer was used for the measurement of the viscosity at atmospheric pressure, in the temperature range 90° to 200°C. Nitrogen gas was used as the calibrating fluid, and the calibration constant was found to decrease by 3% for an increase in temperature of 100°C (Wellman predicted an increase in the calibration constant). The experimental data were correlated using an equation of the for $\eta = A\sqrt{T} + B$. The accuracy of the results was estimated to be ± 2.2%.

Kamien [11], (1959) used Wellman's viscometer to measure the viscosity of R-12, R-13, R-14, R-21, R-22, R-23, R-114, R-115, R-C318 in the vapour phase, in the temperature range 30° to 90°C and a pressure range of 1 to 12 atmospheres. The viscometer was calibrated using air at atmospheric pressure and nitrogen for higher pressures. The data were correlated using an equation of the form $\eta = A\sqrt{T} + B$. The results were compared with other available data. It was observed that the viscosity increased with increasing pressures, but only appreciably at and over 6 atmospheres.

In 1959 Tsui [12] measured the viscosity of R-11, R12, R-13, R-14, R-21, R-22, R-23, R-114, R-115, R-13B1, R-C318 in the vapour phase. He used a rolling ball viscometer which was a modification of Wellman's design. The measurements were made in the pressure range 1 to 20 atmospheres and in the temperature range 90° to 150°C. Nitrogen gas was employed as the calibrating fluid and a variation in the calibration constant was observed with both pressure and temperature. With pressure the calibration constant was found to decrease as the pressure increased whilst with temperature, the calibration constant increased with an increase in temperature. The accuracy of the result was assessed to be from ± 1.59% to 6.6%. The experimental data were correlated using an equation of the form $\eta = A\sqrt{T} + B$. The results were compared with those of other researchers.

Willbers [13], (1961) measured the viscosity of R-12, R-13, R-14, R-22, R-23, R-13B1, R-115, R-C318 in the vapour phase using a rolling ball viscometer. Viscosity values were obtained in the pressure range of 1 to 12 atwospheres and temperature -40° to 15°C. Nitrogen gas was employed as the calibrating fluid and variation of the calibration constant was observed with (a) viscometer tube inclination (b) pressure and (c) temperature. The results were compared with other available data. The accuracy of the results was thought to be ± 1.5%.

Riley [14], (1963) measured the viscosity of R-11 and R-12 in the liquid phase. He used Ostwald type capillary viscometer. The measurements were made at atmospheric pressure, in the temperature range -11° to 20°C. Riley gave the reason for using a capillary viscometer that at the low temperatures the density of rolling ball became less than that of the fluid being tested. He calibrated the viscometer using ethyl ether. A polynomial of the form $\eta = \sum_{1}^{n} a_{n-1} t^{n}$ was fitted to the results using the least square method, where a is constant, t is temperature and n is the degree of polymonial. The results were compared with other available data. The maximum error in the results was estimated to be ± 5%.

Eisele [15], (1965) measured the viscosity of R-11 , R-12 and R-22 in the liquid phase. He used a Cannon-Ubbelohde type capillary viscometer. The measurements were made at the saturation pressure and in the temperature range -95° to 90°F. The viscometer was already calibrated but he checked the calibration using ethyl ether and observed a difference of 1.4%, however he used the calibration constant supplied by the manufacturer. Eisele fitted the following equation to his results:

$$\eta = A\sqrt{T} + B$$

$$\text{and } \gamma = \gamma_b \exp\left(\frac{T_b}{T} - 1\right)$$

where γ is kinematic viscosity

and b is boiling properties.

The results were compared with other available data. An error of 0.7% to 1.5% was estimated.

In 1968 Al-saloum [16] measured viscosity of R-12 and R-22 in the vapour phase using a series capillary viscometer. The viscosity was measured at atmospheric pressure in the temperature range -40° to 92°C. The viscometer was checked for its reliability using nitrogen gas. Polynomials and an equation of the form $\eta = A\sqrt{T} + B$ were fitted to the results. The results were compared with other available data. The error was estimated to be $\pm$ 3.1%. Al-saloum has presented a comprehensive literature survey of the work done so far in this field.

It is apparent from the preceeding survey that very little absolute determination of the viscosity of refrigerants has been completed and that there is great degree of uncertainity and divergence of the data obtained from different sources. The major cause of the lack of agreement may be due to the inaccuracy in the viscosity data for the calibrating fluids and the variation of the calibration constant.

The author is of the opinion that there is still need for more absolute viscosity determination of the halogenated hydrocarbon refrigerants, specially in the low temperature range and at high pressures; and more specially, for some of the newer commercially important refrigerants, eg. R-500, R-501, R-502 etc., on which there is apparently no information.

3 THEORY

For fully developed laminar flow through smooth circular pipes of uniform diameter, Poiseuille observed experimentally that the dynamic viscosity is directly proportional to ;the pressure gradient along the length of the pipe and the fourth power of the diameter of the pipe, and inversely proportional to the volume flow rate. That is ,

$$\eta = \frac{\pi a^4}{8Q} \cdot \frac{dp}{dL} \qquad (3.1)$$

where η is the dynamic viscosity,

a is the radius of the pipe,

Q is the volume flow rate,

and $\frac{dp}{dL}$ is the pressure gradient along the length of the pipe.

(For a comprehensive alternative theoretical development of equation 3.1, see appendix 9.1).

Except at the entrance, where the flow is not fully developed, pressure gradient is constant for laminar flow. However, since the pressure is measured at the ends of the pipe, a correction, known as kinetic energy correction or end correction has to be applied to take into account the flow development at the entrance of the pipe. Thus the modified Poiseuille equation can be stated as ,

$$\eta = \frac{\pi a^4}{8Q} \quad \frac{\Delta p}{L} - \frac{mQ}{8\pi L} \qquad (3.2)$$

where $\frac{\Delta p}{L}$ is the pressure gradient (since $\frac{dp}{dL}$ is constant) and m is the kinetic energy correction factor.

Many attempts have been made to obtain a universal value of 'm'. The most reliable value is considered to be that given by Boussinesq, who found 'm' to be 1.12 , (see Barr [18])

When the fluid enters the pipe from a reservoir, it flows in a converging stream and similarly, at the exit end the stream lines diverge. Consequently, some energy is spent in overcoming the viscous resistance due to the velocity difference in the adjacent layers of the converging and diverging streams. A correction for this, called Couette correction can be applied such that the length of the pipe is hypothetically increased from L to L+na, where n is the couette correction constant, the value of which, as found experimentally by different workers, ranges from 0 to 6. (See Barr [18])

After applying the Couette correction, equation becomes,

$$\eta = \frac{\pi a^4}{8Q} \times \frac{\Delta p}{(L + na)} - \frac{mQ}{8\pi(L + na)} \qquad (3.3)$$

Furthermore, considering thermal expansion of the pipe material, assuming the coefficient of thermal expansion to be isotropic, the final equation can be written as,

$$\eta = \frac{\pi a^4 \Delta p(1 + 3\alpha t)}{8Q(L + na)} - \frac{mQ}{8\pi(1 + \alpha t)(L + na)} \quad (3.4)$$

since $(1 + \alpha t)^4/(1 + \alpha t) \approx (1 + 3\alpha t)$ (3.5)

Where α is the linear coefficient of thermal expansion and t is the temperature

Equation 3.4 is the basic equation for a single capillary tube viscometer, where the capillary tube is a smooth circular pipe of uniform diameter. In order to determine the viscosity, the values of 'm' and 'n' must be known.

The problem of the uncertainity of the values of constants 'm' and 'n' can be avoided by the use of two capillary tubes of similar shape and bore diameter in series. In this way the correction can be eliminated; that is, if L_1, L_2, Δp_1, Δp_2 refer to the lengths and pressure drops for the two capillary tubes of unequal lengths where,

$$\Delta p_1 = \Delta p_{v1} + \Delta p_{D1} \quad (3.6a)$$

$$\Delta p_2 = \Delta p_{v1} + \Delta p_{D2} \quad (3.6b)$$

Δp_v is the pressure drop due to the viscous forces and Δp_D is due to the flow irregularities at the ends.

If the shape of the ends of the capillary tubes is the same and the tubes are of the similar cross section and bore size, the assumption that $\Delta p_{D1} = \Delta p_{D2}$ would be reasonable. Thus subtracting pressure Δp_2 across the small capillary tube of length L_2 from that of large capillary tube of length L_1

would give the pressure drop due to the viscous forces in the length ($L_1 - L_2$).

The final equation for the two capillaries in series - viscometer, for 'm_1'='m_2' and 'n_1'='n_2', can be given as (see appendix 9.1)

$$\eta = \frac{\pi a^4(1 + 3\alpha t)}{8\bar{Q}(L_1 - L_2)}\left[\Delta p_1 \rho_1 - \left(\frac{a_2}{a_1}\right)^4 \Delta p_2 \rho_2\right] \qquad (3.7)$$

Above subscripts 1 and 2 refer to the large and small capillary tubes respectively, $\bar{Q}$ is the mass flow rate and ρ is the density of the test gas.

One of the assumptions in deriving Poiseuille equation is that there is no slip at the wall of the pipe. This is not strictly true, and a correction for this can be applied to the working equation. Some work has been done on this aspect of flow. Most reliable seems to be by Knudsen, who developed a semi-empirical relation of the form

$$\eta_t = \eta \cdot \left(1 + \frac{8\bar{\varepsilon}}{d}\right) \qquad (3.8)$$

where η_t is the true viscosity

η is the viscosity calculated from equation 3.7

$\bar{\varepsilon}$ is the slip correction factor

and d is the diameter of the pipe.

Again, $$\bar{\varepsilon} = \frac{2.128\ \bar{k}\ \eta}{\sqrt{\rho \bar{p}}} \qquad (3.9)$$

Where p is the absolute pressure of the system

ρ is the density of the fluid

and $\bar{k}$ is the empirical constant depending upon fluid

In the present research, the slip correction is neglected since Latto [20] showed that for steam and the majority of gases at atmospheric pressure, the correction is negligible; furthermore, the value of the empirical constants $\bar{k}$ were not available for the vapours investigated.

4 DESIGN CONSIDERATIONS

Poiseuille equation can only be used if the flow is laminar, that is, the Reynold number is below the critical limit of about 2000. Therefore, R, the Reynold number is the main parameter for the design consideration of the apparatus.

Mass flow rate and hence, the capillary tube diameter directly affect R. Also the pressure drops across the tubes depend upon the flow rates. Therefore, R was calculated assuming different capillary tube diameters. Corresponding mass flow rates were also calculated to select the most suitable flow measuring device.

Though the effect of the velocity development at the entrance is eliminated by using two capillary tubes in series, still it is desirable to keep the ratio $\frac{X}{L}$ below a certain limit, X and L being the development length and the total length respectively.

From the Poiseuille equation

$$\eta = \frac{\pi a^4}{8Q} \frac{dp}{dL} = \frac{\pi a^4 \rho}{8\bar{Q}} \cdot \frac{dp}{dL}$$

Where $\bar{Q}$ is the mass flow rate

$$\text{Therefore, } \bar{Q} = \frac{\pi a^4 \rho}{8\eta} \frac{dp}{dL} \qquad (4.1)$$

$$\text{and } R = \frac{\rho v d}{\eta} = \frac{2a\rho}{\eta} \frac{\bar{Q}}{\pi a^2 \rho}, \text{ or } R = \frac{\rho a^3}{4\eta^2} \frac{dp}{dL} \qquad (4.2)$$

$$\text{and } X = .0288 \times R \times d = \frac{.0288}{2} \frac{\rho a^4}{\eta^2} \frac{dp}{dL} \qquad (4.3)$$

(see [18])

It is clear from the relations 4.1, 4.2, and 4.3 that the mass flow rate $\bar{Q}$, Reynold number R and the development length X, all depend upon the capillary tube diameter, pressure gradient, dynamic viscosity and the density of the test fluid. Furthermore, the dynamic viscosity and density are functions of temperature. (The variation of capillary size due to the thermal expansion and contraction was negligibly small, since the coefficient of linear expansion for glass is of the order of 3.3×10^{-6} per °C)

The available data for the specific volume v and the viscosity η were correlated using the equations of the form

$$v = A \frac{t - 32}{1.8} + B$$

$$\eta \times 10^4 = C\sqrt{T} + D$$

Where T is temperature in °K

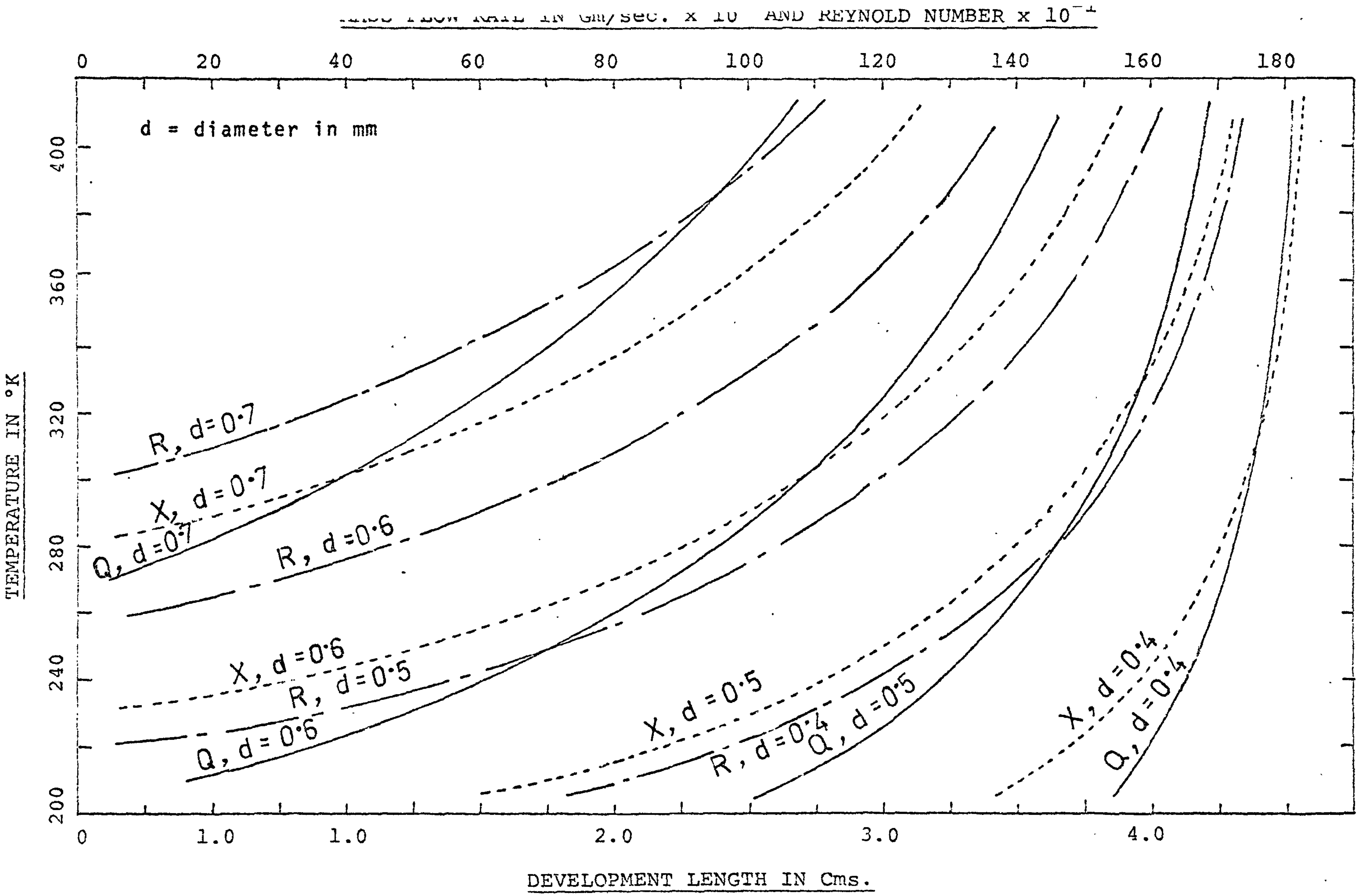
t is temperature in °F

and A, B, C, D are constants.

The specific volume of test gases was taken from the 'Properties of commonly used refrigerants'. For the viscosity values of R-13 and R-500, secondary and estimated values respectively, were used as an approximation.

A pressure drop of 30 cm of water for 100 cm of capillary length was assumed.

A graph was plotted for each gas, showing variation of Q, R, & X with the temperature for different capillary bore sizes to facilitate the selection of a suitable capillary tube.



REYNOLD NUMBER, MASS FLOW RATE & DEVELOPMENT LENGTH Vs TEMPERATURE FOR R-13

FIG. 1

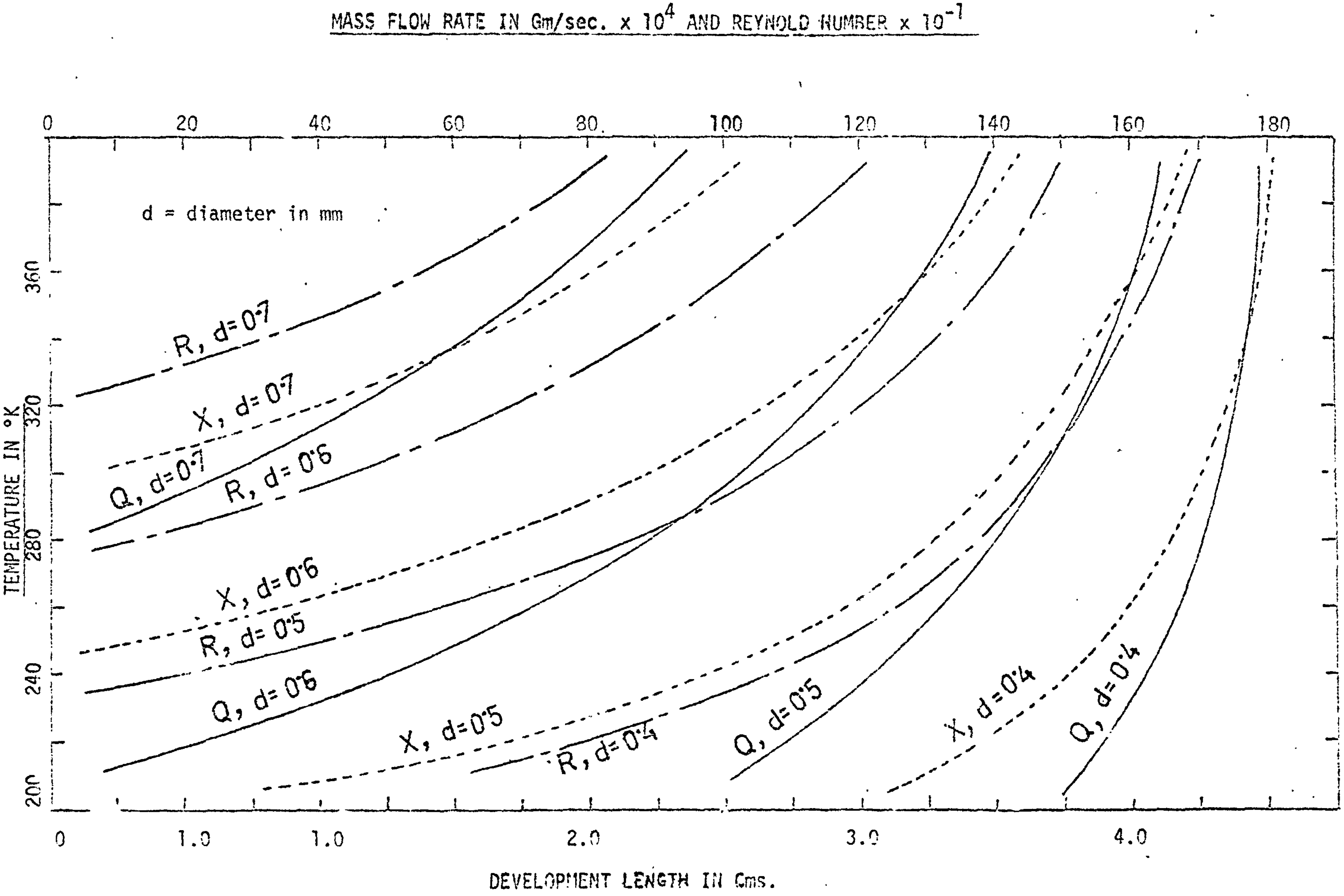


Fig. 2 REYNOLD NUMBER, MASS FLOW RATE & DEVELOPMENT LENGTH Vs TEMPERATURE FOR R-500

5 Description of the apparatus

The viscometer was designed and developed at McMaster University by Latto and the author. The description of the various parts of the apparatus is given below in the following sections:

i The viscometer
ii Pressure measurement
iii Temperature control and measurement
iv Flow measurement
v Assembly of the apparatus.

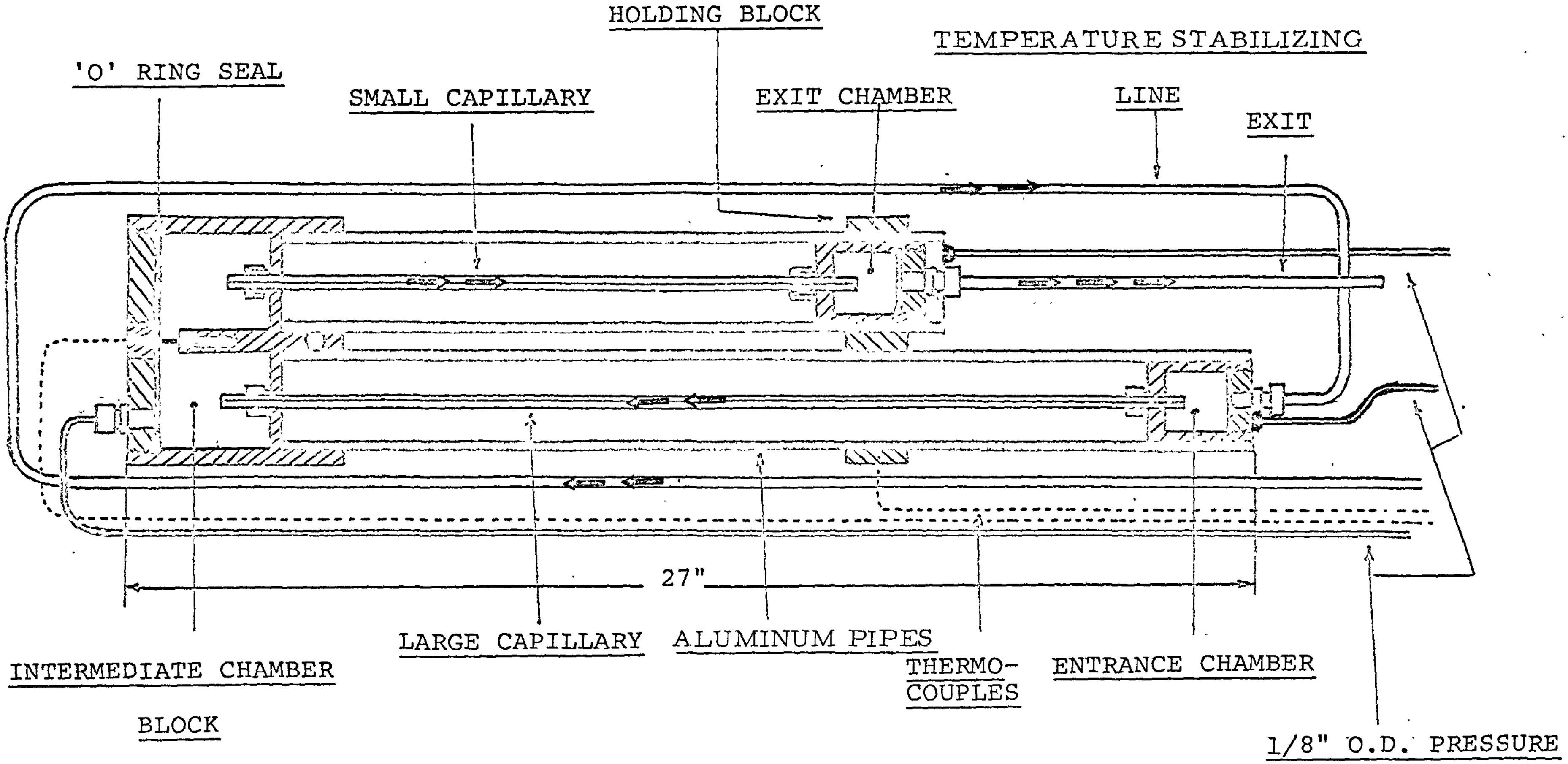
i The viscometer

The viscometer essentially consisted of (see fig. 3)

An entrance chamber
An intermediate chamber block
An exit chamber
And two glass capillary tubes.

The test gas was supplied to the entrance chamber through a temperature stablizing 'U' tube. The stablizing tube was a 1/4" O.D. copper tube and was connected to the entrance chamber with a 'Swagelok'* fitting. The entrance and exit chambers, which were similar in shape and size, were machined out of an aluminium block. 'Swagelok' type fittings, which were an integral part of the chambers, were used for the capillary tube connections. (see fig. 4)

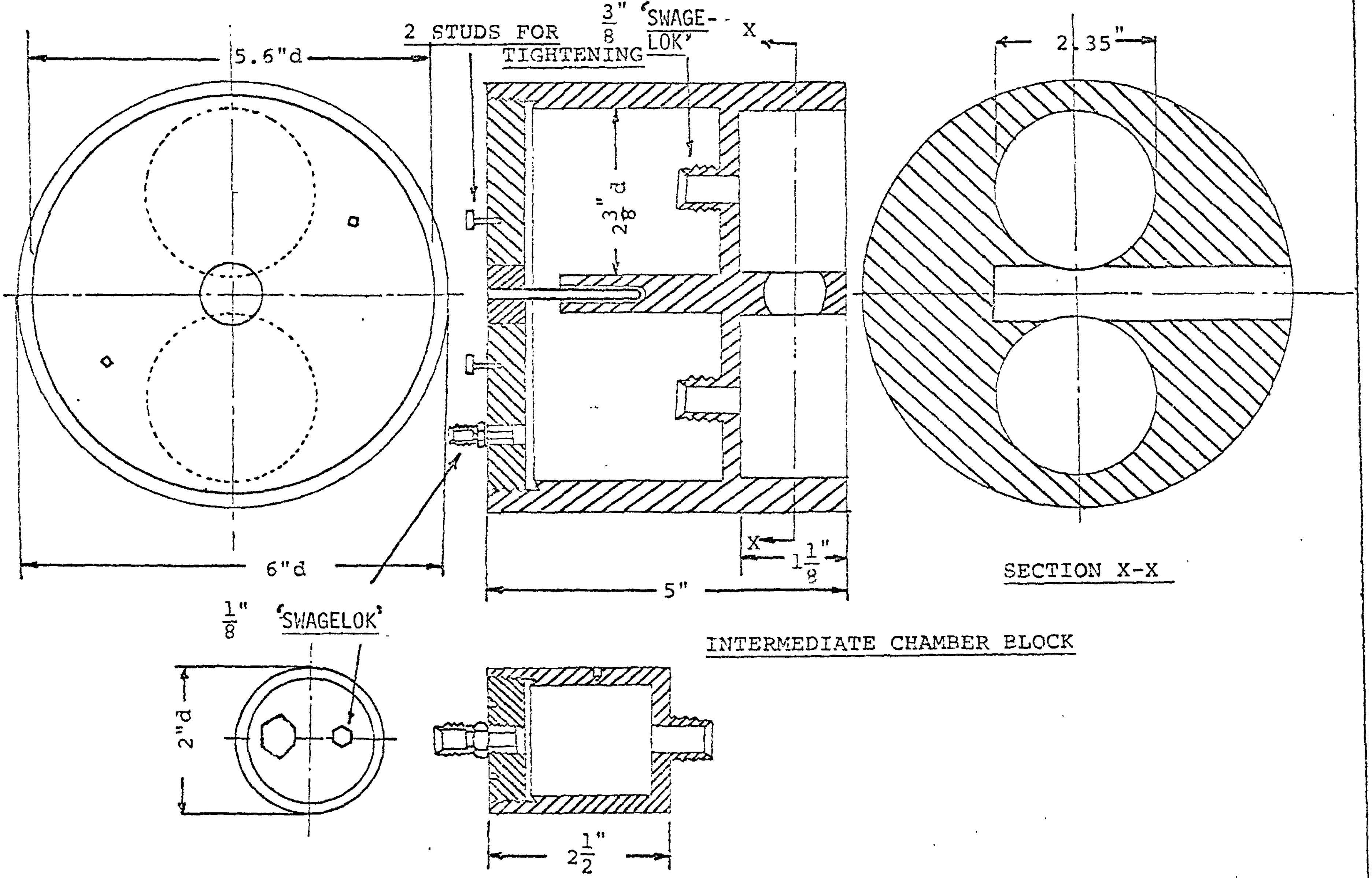
* A trade name for a threaded joint.



SCALE 1"=4"

VISCOMETER ASSEMBLY

FIG. 3



SECTION X-X

INTERMEDIATE CHAMBER BLOCK

FIG. 4

ENTRANCE / EXIT CHAMBER

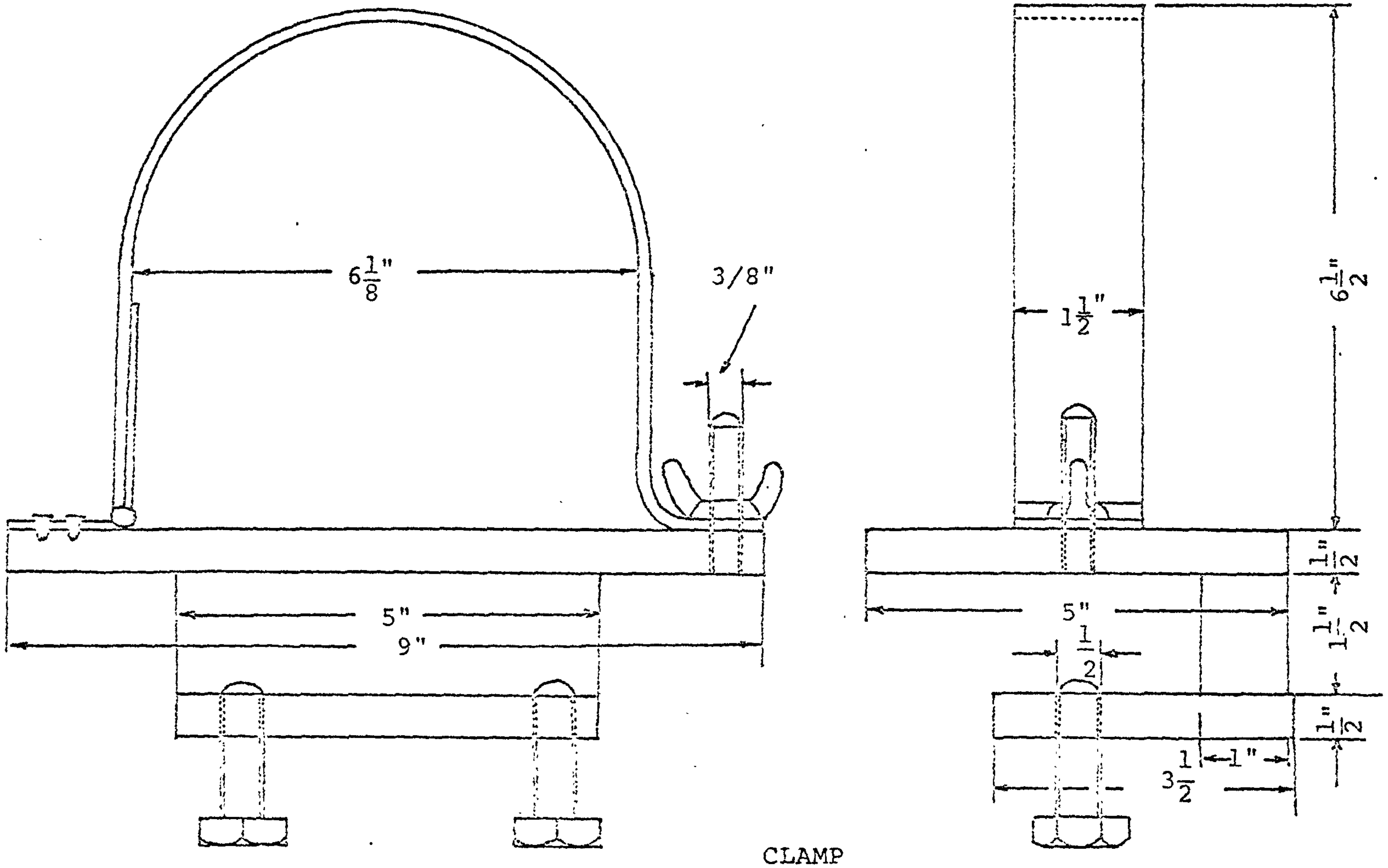
SCALE HALF FULL SIZE

The end covers, which were threaded, had two 'Swagelok' fittings; one 1/8" and the other 1/4" for pressure tappings and entrance/exit of the test gas respectively. 'O' ring seals were used on all the end covers.

The intermediate chamber block served two purposes; one end formed the intermediate chamber and the other held the aluminium alignment tubes. (see fig. 3) The intermediate chamber was further divided into two sections where the ends of upstream and downstream capillary tubes held. The end cover was threaded and was tightened with the aid of two studs provided. A 1/8" O.D. copper tube pressure tap was connected to the end cover using 'Swagelok' fitting. A thin tubular thermocouple pocket was welded at the center of the end cover which was located in the corresponding hole provided in the main block. (see fig.4) The other end of the intermediate block held two aluminium alignment tubes which could be locked using a locking device. The opposite end of the tubes were carefully held and locked in a holding block. The alignment tubes held all the three chambers together such that no undue stresses were created in the glass capillary tubes. The inner surface of the alignment tubes was machined to allow a smooth sliding fit of the exit and entrance chambers. Grub screws were used to lock the two chambers into position.

Fluid leakage was one the main problems to be handled. A slight leakage would cause large errors in the experimental results, since the flow rates used were very small. Leakage was most likely to occur at the following points; (i) Where capillary tubes were connected to the chambers, (ii) Where connections were made for the pressure taps, the test gas entrance and exit tubes and (iii) At the end covers. 'Swagelok' connections were exclusively used and teflon tape was used for all the threaded joints. Special teflon seals were used to hold the tubes in the 'Swagelok' fittings. This was alright for the copper tubes, however, for the glass capillary tubes, there was danger of breakage due to overtightening of the 'Swagelok' nut. Therefore, Silicon seal (General Electric) was used for all the joints. To avoid leakage past the threaded joints of the end covers, 'O' rings were used. For the intermediate chamber teflon tape was used and the end cover was tightened by holding the chamber in a specially designed clamp. (see fig. 5)

Before assembling the viscometer, the three chambers were thoroughly cleaned by rinsing with methanol and then with acetone. The capillary tubes, which were already thoroughly cleaned for the calibration purposes, were again cleaned with chromic acid and then rinsed with water followed by methanol and then acetone. In order to assemble the viscometer, the aluminium alignment tubes were held in



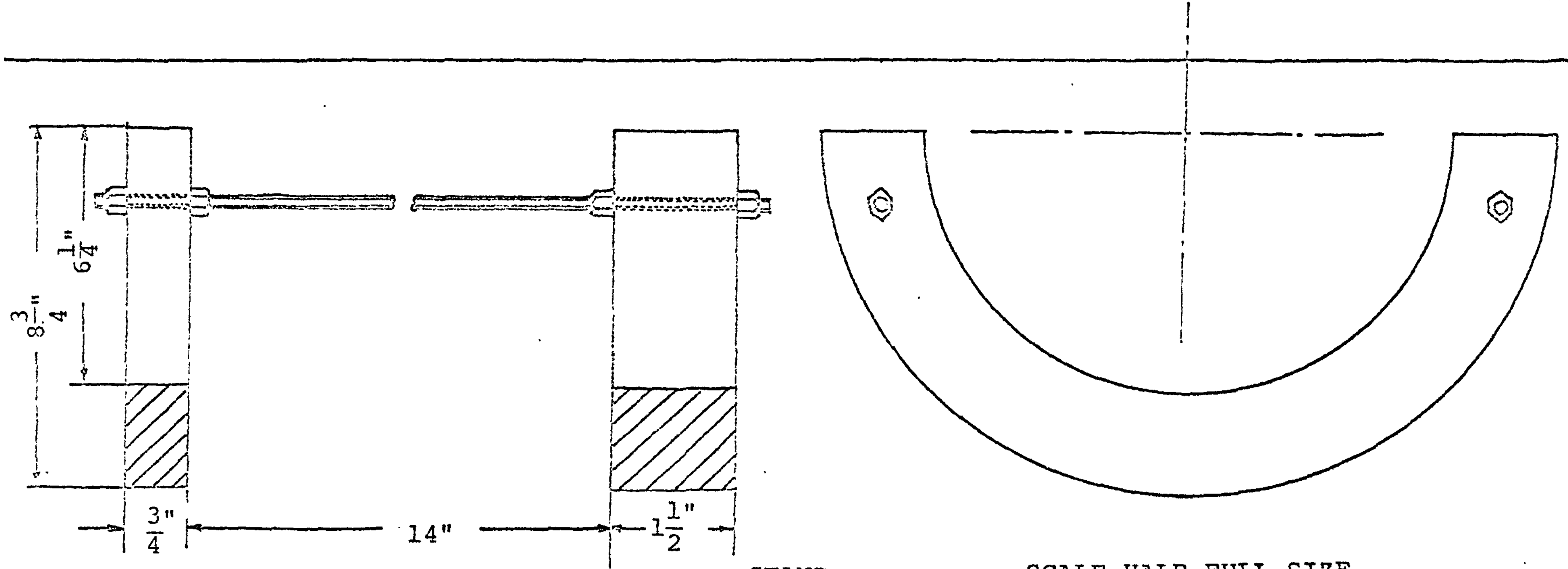
CLAMP

FIG.5

SCALE HALF FULL SIZE

position in the intermediate chamberblock and the holding block. The end held in the intermediate chamber block was locked while the opposite end, held in the holding block, was kept loose to enable the chambers to slide easily. The assembly was then held vertically and the exit and entance chambers, having capillary tubes already connected, were introduced very carefully. The opposite ends of the capillary tubes came out through the corresponding 'Swagelok' fittings in the intermediate chamber. The exit and entrance chambers were then held in position with grub screws. The alignment tubes were then locked in position in the holding block whilst the 'Swagelok' holding capillary tubes were tightened. The assembly was held in the clamp (see fig. 5) and the end covers were tightened. Two studs were provided in the intermediate chamber end cover for tightening purposes whilst two blind holes 1/8" deep were provided in the end covers of the exit and entrance chambers for the same purpose.

The assembled viscometer was installed in a 9" diameter and 36" long heat exchange tube of glass, (M/s QVF- Quick Visible Flow). A stand was made, as shown in fig. 6 , which supported the viscometer in the heat exchange glass tube. Two, eight bolts cast iron flanges were held in position by the fibre belt packings, to which two steel cover plates were bolted. The cover plates had 'Swagelok' fittings for



STAND SCALE HALF FULL SIZE

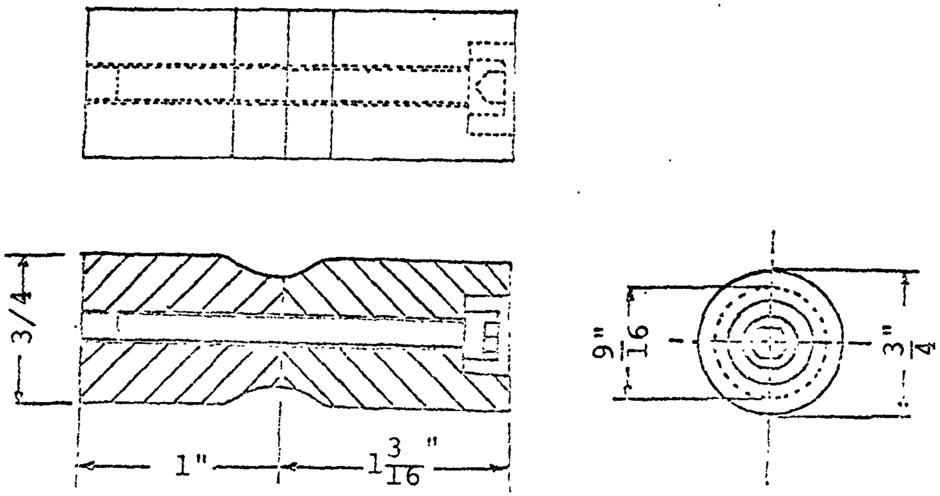


FIG. 6 LOCKING DEVICE SCALE FULL SIZE

the various copper tubes and thermocouple wires to pass through them. For ease in the assembly, all the leads were taken out through one of the cover plates. To avoid any leakage of heat exchange fluid past the cover plates, a rubber ring seal was glued to the cover plate with contact cement ,(Bull dog adhesive, Canadian adhesive co.) .

The heat exchange glass tube was housed in a wooden box which was filled with polystyrene foam for heat insulation .

The thermocouples were put in pockets; one in the end cover of the intermediate chamber block and the other in the holding block. (see fig. 3) They were held in the position by thin pieces of wood and molten wax. The wires were taken out through the 'Swagelok' fittings in the steel cover plate. The pressure tappings (1/8" O.D. copper tubes) were connected to tygon tubing by using an adapter of 1/8"-1/4" size. The tygon tubing were then connected to the manometer.

For the fluid leakage test, the apparatus was slightly pressurised with nitrogen to about 30" of oil. The drop in the pressure due to leakage was observed using the manometer-cathetometer arrangement. A dilute soap solution was used to detect the points of leakage in the viscometer. The viscometer was dismentled and then re-assembled after applying Silicon seal (General Electric.) and was again

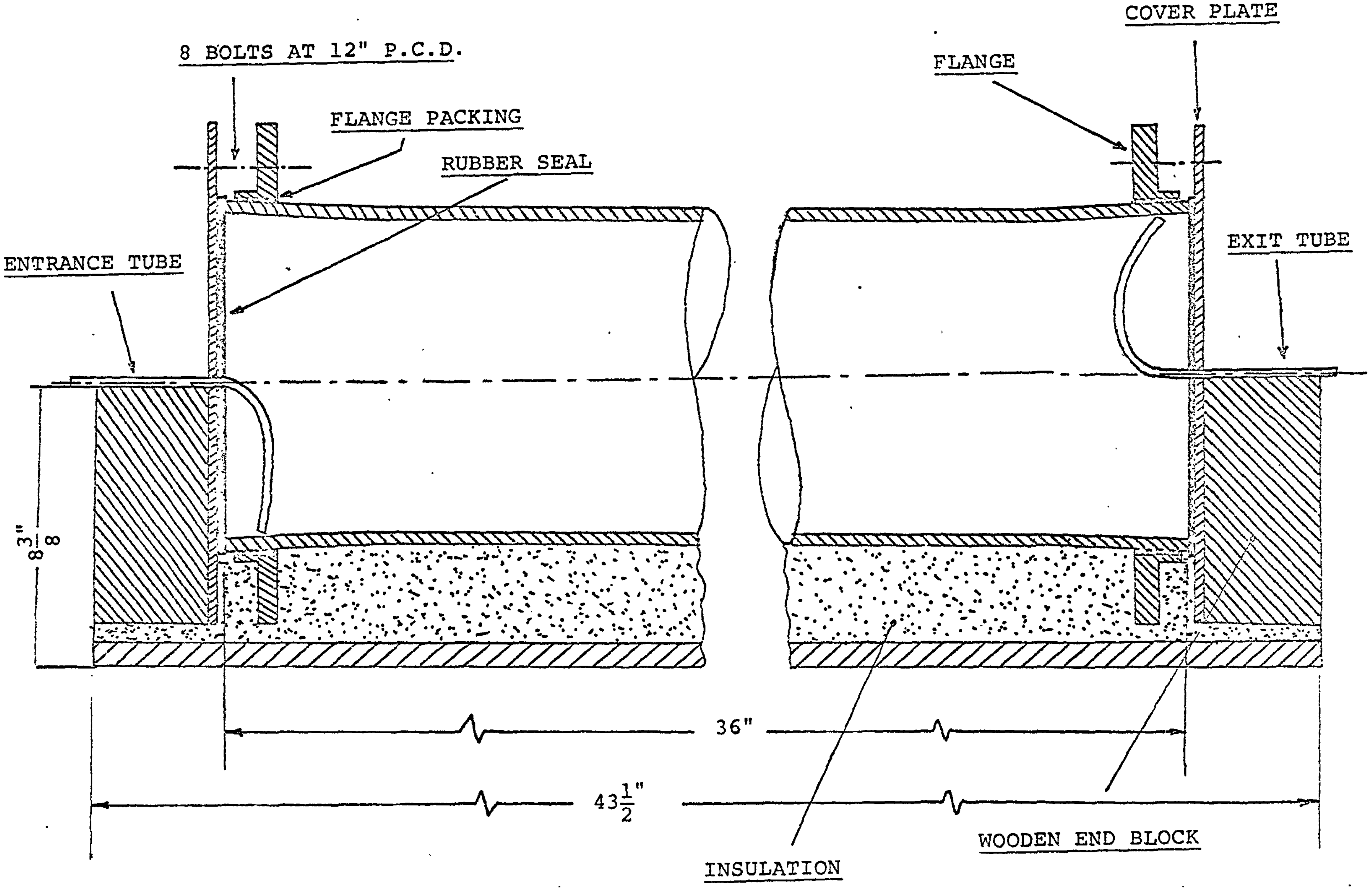
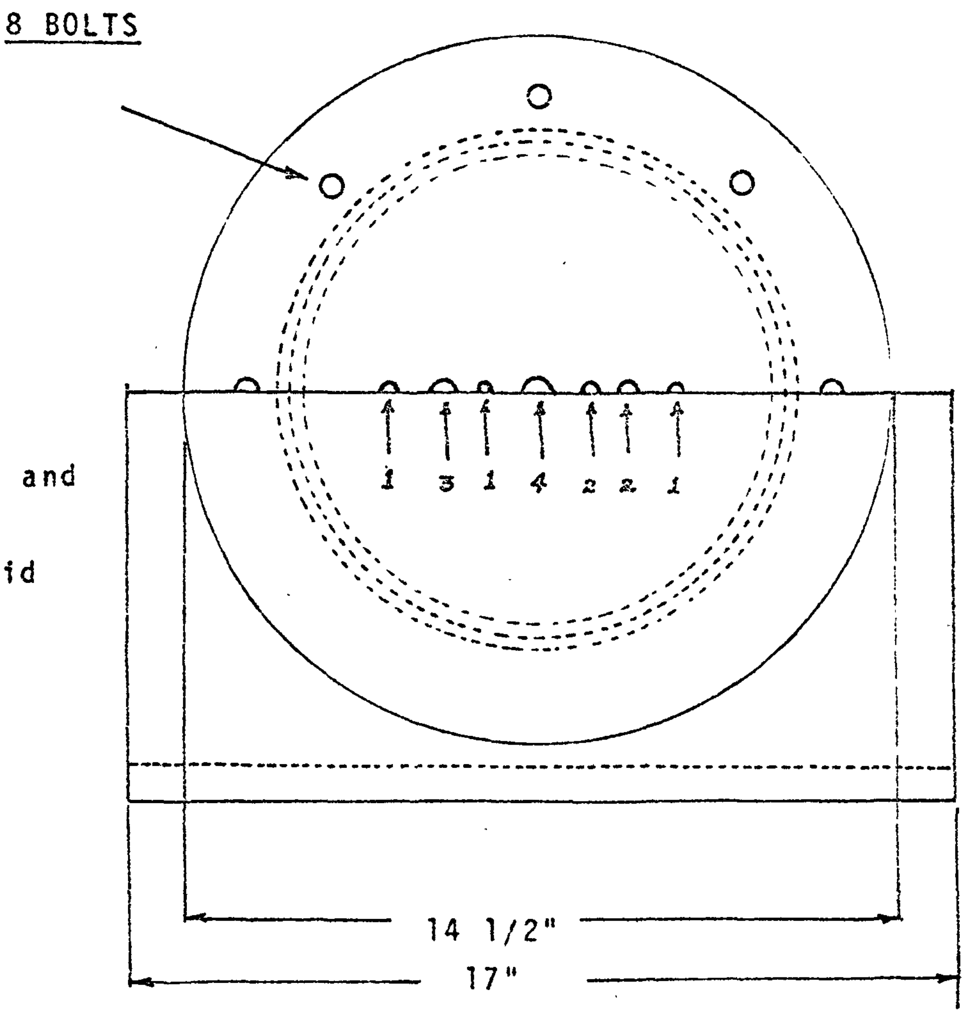


FIG. 7

HEAT EXCHANGE GLASS TUBE AND THE INSULATION BOX

SCALE 1" = 4"



1. Pressure Leads
2. Test-gas Entrance and Exit Tubes
3. Thermocouple
4. Heat Exchange Fluid Exit Tube

FIG. 8

HEAT EXCHANGE GLASS TUBE - COVER PLATE

SCALE 1" = 4"

leak tested. This procedure was repeated until the viscometer was leak proof.

ii Pressure measurement

A three limb[*] differential oil[**] in glass manometer was used for measuring the pressure differences across the capillary tubes. Three-way stop cocks were provided to connect each of the manometer limbs to the apparatus and to the atmosphere. Red gauge oil was used as the manometer fluid for two reasons:

(i) It increased the sensitivity of the manometer, since its specific gravity was .826.

(ii) Distilled water was found to diffuse into the viscometer, therefore, oil, being a less volatile liquid, was used for this purpose.

In order to ascertain if there was any leakage past the various joints in the manometer and the associated connections, the apparatus was pressurized and the manometer then isolated, any change in oil level indicated leakage in the manometer system. All the glass to glass surfaces were coated with 'Dow Corning high vacuum grease-silicon lubricant'. No leakage was observed after the use of the grease.

The manometer oil levels were measured using

---

[*] An extra, fourth limb was for temperature measurement.

[**] Specific gravity .826.

a cathetometer capable of measuring to 0.001 cm. The cathetometer stand was levelled by means of three screws and spirit level provided. Also before each manometer reading was taken, the cathetometer telescope was levelled by observing the spirit level and adjusting a screw provided for this purpose.

It is worthwhile to note that a pressure transducer was tried for pressure measurement. A special valve was designed and manufactured which would make it possible to use only one transducer to read all the three required pressures. (see fig.37) After amplification the signal from the transducer was fed to the chart recorder. This method proved to be incapable of producing the required high degree of accuracy i.e. of the order of 1 mm of so. The potentiometer-Galvanometer set up, which was used for temperature measurement, was also tried for the measurement of the output signal from the transducer, but it proved to be too sensitive. Ultimately it was decided to use the manometer-cathetometer set up which in the author's opinion, though inconvenient to use, was one of the most accurate and sensitive devices available at that time. The detailed description of the use of the pressure transducer is given in the appendix 9.6.

iii Temperature control and measurement

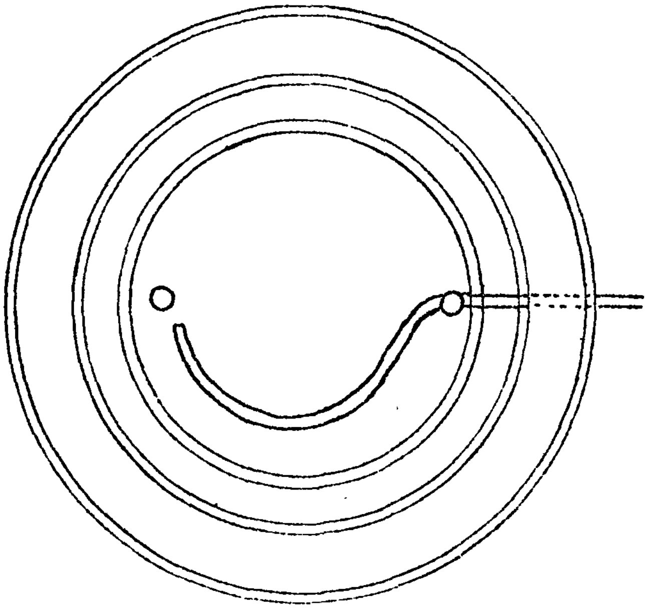
The heat exchange fluids were circulated through the heat exchanger. The following heat exchange fluids were used for the temperature ranges indicated:

(i) Water, for temperatures from atmospheric temperature to approximately 90°C

(ii) Methanol, for temperatures from atmospheric temperature to approximately -60°C.

The water was heated in a thermostatically controlled heater with stirrer ('Porta temp' from M/s Precision Scientific Co.) and was circulated using two miniature pumps.

The methanol (Freezing point -97.8°C) was cooled with dry ice (Melting point -56.6°C at 5.2 atms., -66°C measured by the author in the laboratory at the atmospheric conditions) and liquid nitrogen (Boiling point -194°C), and was circulated by an immersion pump. The liquid nitrogen was discharged from a large dewar flask by supplying dry and compressed air to the space above liquid nitrogen. The displaced liquid nitrogen was circulated through the copper coils in the Methanol-heat exchanger. (see fig. 9) The supply of liquid nitrogen could be controlled by adjusting the compressed air supply and/or by throttling liquid nitrogen.



PLAN - PUMP AND COVER REMOVED

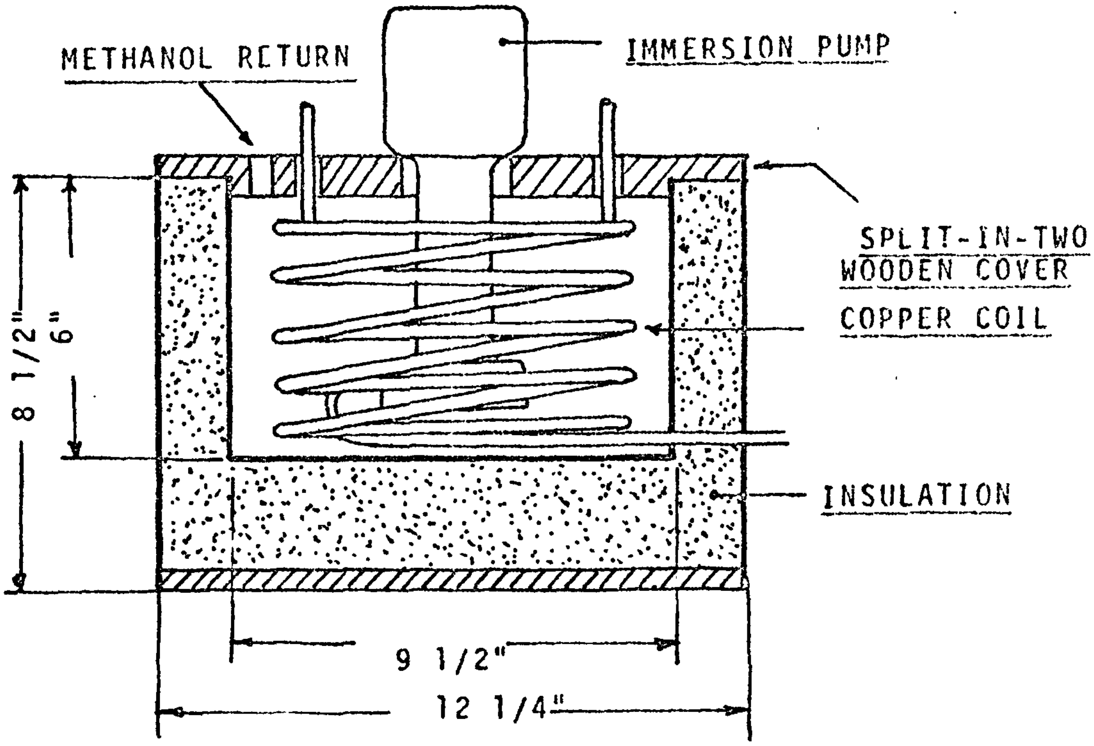


FIG. 9 METHANOL HEAT EXCHANGER SCALE 1" = 4"

Two thermocouples, one on each end, were used to measure the temperature of the viscometer. Two more thermocouples, one for measuring the temperature of the manometer oil and other for the temperature of the flow meter water jacket, were also used. All the thermocouples were from the same reel of Copper-Constantan wire (Thermo-Electric co.) and were calibrated against a standard copper-constantan thermocouple. (see appendix9.4) The cold junction was kept at 0°C by placing it in a ice-water mixture. The thermocouple emf was measured using a Potentiometer-Galvanometer set up consisting of ;

(i) Potentiometer, 'Guildline instruments' - capable of measuring $1x10^{-7}$ volts (.003°C)

(ii) Photocell amplifier, 'Guildline instruments'.

(iii) Null galvanometer, 'Guildline instruments'

(iv) Standard cell, 'Guildline instruments' (voltage 1.01859 at 20°C)

and (v) 6 volt battery.

A 12 point selector switch was employed for switching desirable thermocouple into the circuit.

All the cold junctions were inserted into long glass tubes and were embedded in wax.

iv Flow measurement

In order to keep the flow laminar, and thus keep the development length and the pressure drop in the capillary tubes small, the flow rates were maintained as small as possible.

For such small flow rates, the use of conventional commercial flow meters was not possible. Therefore, a number of special flow measuring devices were examined and ultimately the soap film type flow meter was selected and used. This simple flow measurement device, though not new (see Barr [18]) is perhaps one of the most accurate methods of measuring flow rates.

The flow meter, as shown in fig.10 ,essentially consisted of a precision graduated glass tube (burette) of uniform diameter having a cup filled with soap solution. A water jacket surrounds the burette. The test gas enters the burette through a temperature stablizing tube. A soap film is raised in the burette by pressing the rubber bulb. This film acts as a leak proof, pressure tight, weightless piston membrane, which indicates flow rates when timed with a stop watch.

v Assembly of the apparatus

The assembled viscometer was placed over its stand inside the heat exchange glass tube. All the copper tubes and the thermocouple wires were brought out of the glass tube through the appropriate 'Swagelok' fittings in the cover plate and the cover plates were bolted to the flanges.

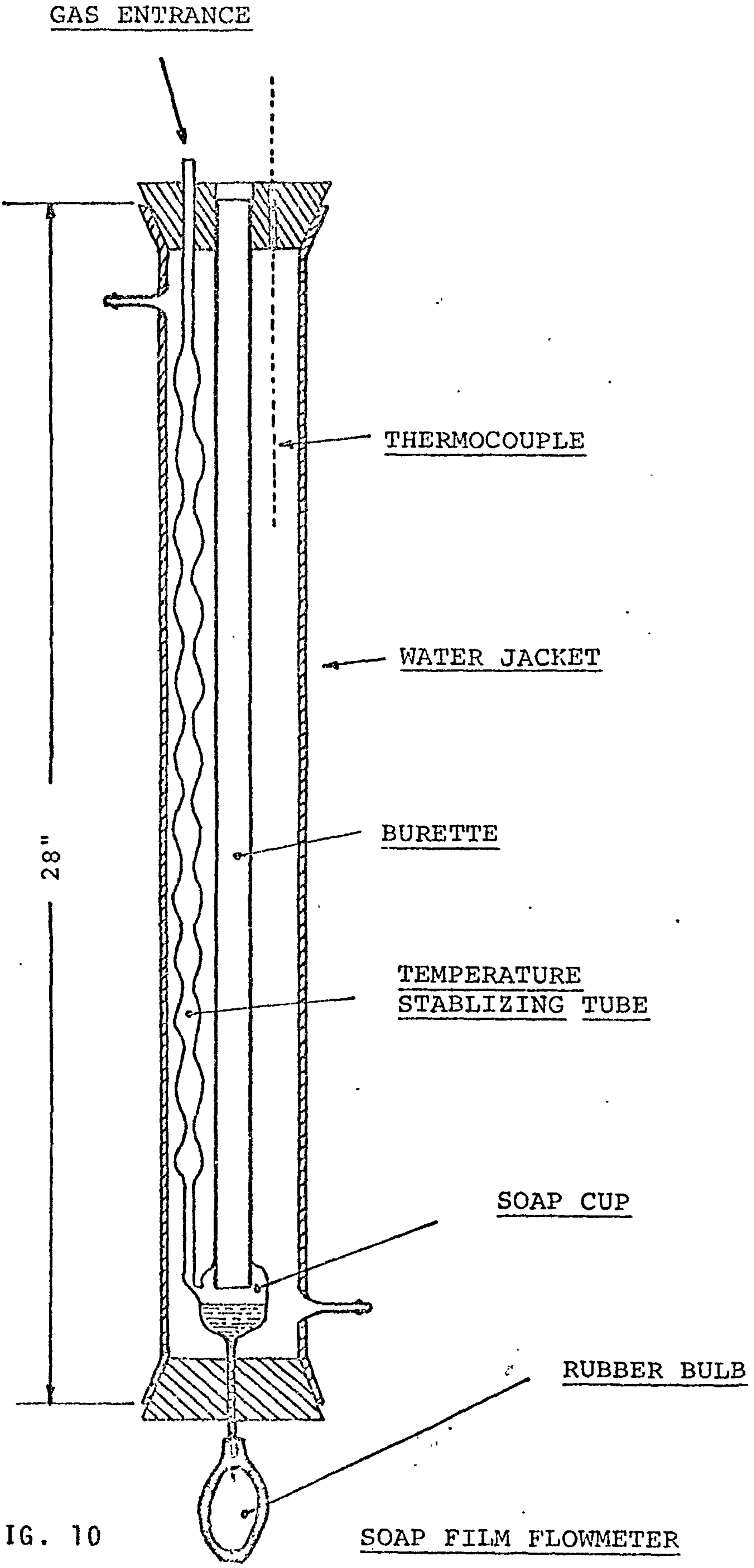
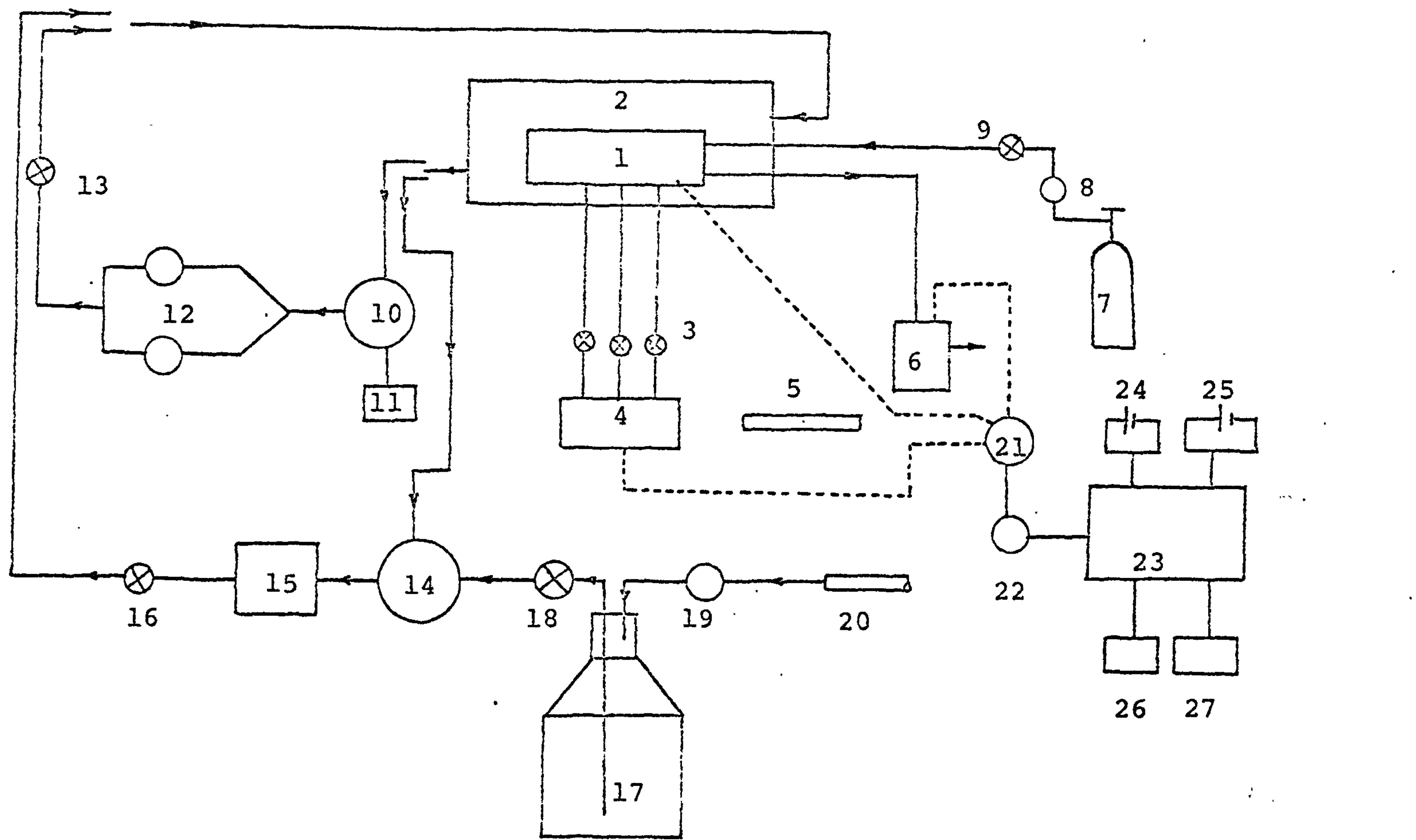


FIG. 10 SOAP FILM FLOWMETER SCALE 1" = 4"

After fixing the side wooden blocks, the insulation box was closed. The pressure taps were connected to the manometer and the supply tube to the entrance chamber was connected to the test gas cylinder through the pressure regulators and needle valve. The outlet from the exit chamber was connected to the flow meter. The flow diagram of the whole set up is shown in fig. 11.



Dotted lines indicate thermocouples.

FIG. 11 FLOW DIAGRAM

LEGEND OF FLOW DIAGRAM

(1) Viscometer

(2) Viscometer heat exchange glass pipe

(3) Three-way manometer stop cocks

(4) Three limb differential red gauge oil manometer

(5) Cathetomter

(6) Soap film flow meter and water jacket

(7) Test gas bottle

(8) High and low pressure regulators

(9) Needle valve

(10) Constant temperature water bath

(11) Heating unit (heater, stirrer & thermostatic control)

(12) Two centrifugal pumps, 'Little Giant'- 1/3 hp.

(13) Water control valve

(14) Methanol heat exchanger

(15) Immersion pump, 'Little Giant'

(16) Methanol control valve

(17) Liquid nitrogen dewar

(18) Liquid nitrogen control valve

(19) Air regulator, 'Watts' model (M-0125)

(20) 20 psi Air supply line

(21) Thermocouple cold junction

(22) Thermocouple selector switch

(23) Potentiometer, 'Guildline ' Model (9461A)

(24) Standard cell, 1.01859 volts at 20°C, 'Guildline '

(25) Six volt battery

(26) Photo-cell galvenometer amplifier, 'Guildline '

(27) Galvanometer, 'Guildline '

6 PROCEDURE

To test the reliability of the apparatus, the viscosity of highly purified nitrogen (99.996% pure), the viscosity of which is known to better than 2% in the temperature range of -100 to 150°C, was determined over a temperature range of -12.13 to 77.18°C. It should be noted that this test with nitrogen was not a calibration but merely a check of the apparatus.

Nitrogen gas was fed to the viscometer through a standard pressure regulator (Matheson Automatic Regulator #1L) where pressure pertubations were damped out.

The gas was then allowed to flow continuously through the apparatus at reduced flow rates, i.e. less than for a normal run, for several hours in order to remove air or moisture etc. from the system.

For controlling the temperature of the viscometer, the heat exchange fluids (see chapter 5,'Description of the apparatus') were circulated through the heat exchanger and when the desired temperature was reached and thermal equilibrium achieved, the reading was taken.

The manometer reading was, firstly, noted and then the temperature readings were taken. The flow rate was measured two to three times during a test and then the manometer and temperature readings were repeated to check any instability, after which the flow rate was again measured. It was noted that during all the tests, the maximum temperature gradient along the entire length of the viscometer was less than 1°F. The barometer pressure was measured during each test.

After the test with one gas was completed, the above procedure was repeated for other gases tested.

7 Result and discussion

To test the reliability of the apparatus, viscosity of nitrogen gas was measured in the temperature range of -12.13°C to 77.18°C and is tabulated in the table 1 , together with recommended viscosity values given in the 'Thermophysical Properties Research Center Data Book', which are quoted to be accurate to ±2%. This table also shows the percentage deviation of the results obtained in this work from the TPRC [21] values. The percentage deviation indicates that the viscosity values are generally lower than the recommended values, but are within the recommended deviation. It is worthwhile noting that the TPRC data was correlated using 20 sets of different data.

Table 2 shows the viscosity of R-13 in the temperature range of -53.25°C to 87.19°C. Polynomials of 1 to 5 degrees were fitted in the data and 2nd degree equation was found to be most suitable, (see fig. 12). The polynomial fitted is:

$$\eta = 6.43206\mathrm{x}10^{-6}\mathrm{x}T^{2} + 1.05245\mathrm{x}10^{-3}\mathrm{x}T + 5.5777\mathrm{x}10^{-1}$$

Standard deviation=$1.37167\mathrm{x}10^{-2}$,

Maximum % deviation=1.58147%,

Maximum deviation=$2.01599\mathrm{x}10^{-4}$ cp

Mean deviation=$2.46998\mathrm{x}10^{-16}$ cp

Fig. 13 shows the scatter of the experimental results from the fitted polynomial. All the data points lie within ± 1.58% .

TABLE 1

VISCOSITY OF NITROGEN GAS

Temp. °K	Viscosity Poise x 10^4		PERCENTAGE-
	This work	TPRC	DEVIATION
261.015	1.5881	1.5980	-0.618
264.755	1.6203	1.6175	+0.173
266.970	1.6106	1.6300	-1.190
272.048	1.6473	1.6510	-0.224
278.790	1.6826	1.6820	+0.035
282.300	1.6788	1.6990	-1.180
296.150	1.7693	1.7633	+0.330
296.225	1.7548	1.7635	-0.493
297.390	1.7525	1.7685	-0.904
301.470	1.7716	1.7870	-0.862
305.590	1.7862	1.8058	-1.080
308.670	1.7850	1.8200	-1.920
310.150	1.7972	1.8262	-1.580
319.640	1.8527	1.8680	-0.819
331.780	1.9260	1.9226	+0.177
344.310	1.9669	1.9750	-0.410
350.330	2.0033	2.0008	+0.125

TABLE 2

VISCOSITY OF R-13

Temp. °K	Viscosity Poise x 10^4
219.896	1.0982
224.980	1.1102
229.685	1.1420
235.784	1.1724
243.040	1.2084
248.900	1.2316
261.518	1.2684
266.466	1.2748
275.535	1.3349
276.424	1.3282
283.537	1.3556
284.960	1.3824
292.475	1.4244
295.975	1.4290
299.000	1.4438
308.912	1.5068
318.917	1.5510
329.034	1.6084
335.990	1.6554
349.090	1.7094
360.346	1.7559

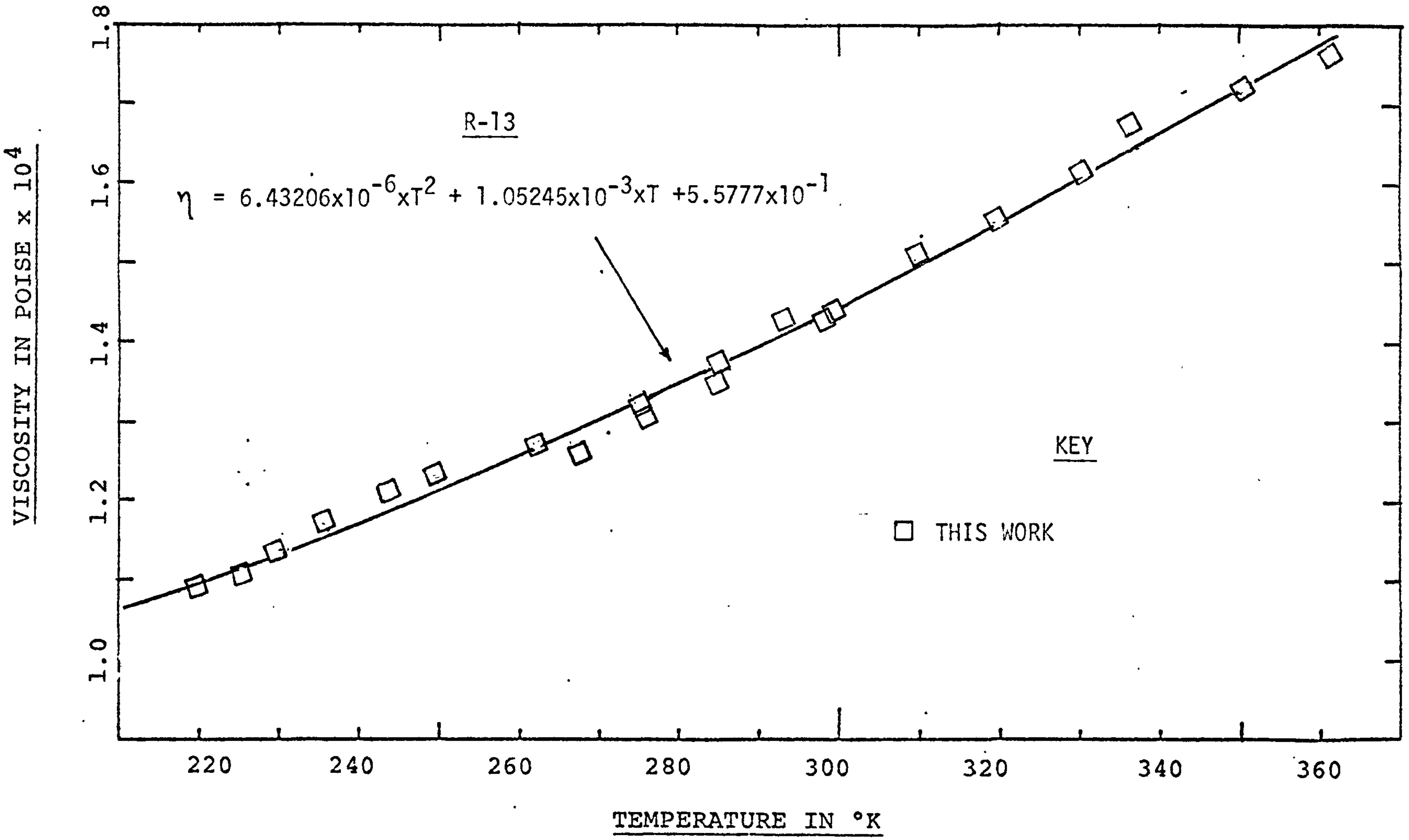
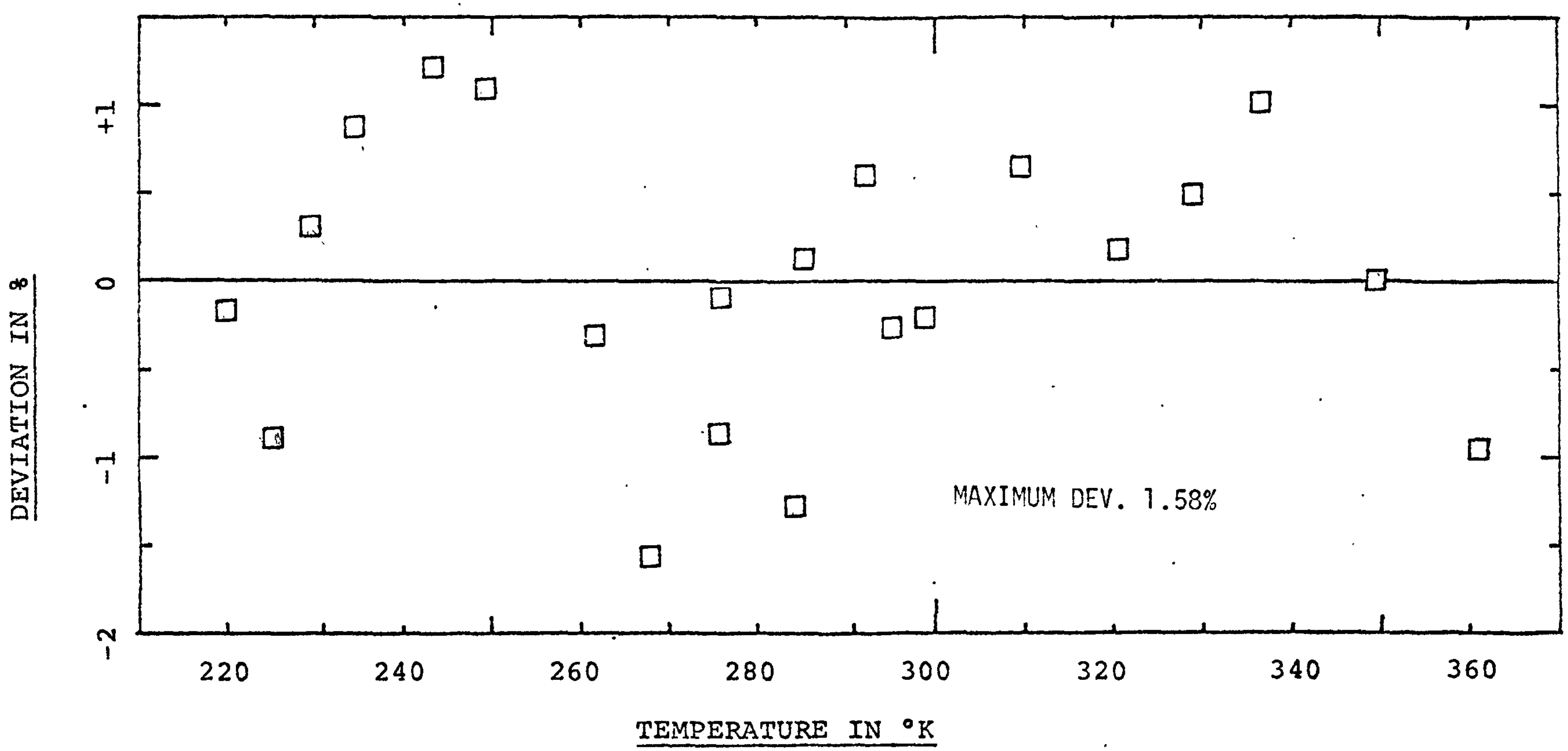


FIG. 12 VISCOSITY OF R-13 Vs TEMPERATURE WHEN FITTED TO 2nd DEGREE POLYNOMIAL.

DEVIATION FROM $\eta = 6.43206\times10^{-6}\times T^2 + 1.05245\times10^{-3}\times T + 5.5777\times10^{-1}$



DEVIATION OF EXPERIMENTAL RESULTS FOR R-13 FROM THE FITTED 2nd DEGREE POLYNOMIAL.

FIG. 13

A 2nd order polynomial was also found suitable for the combined result of this work and the work by previous researchers. The plot in fig.14 shows a great deal of scatter from the following fitted equation;

$$\eta = -1.8506\text{x}10^{-6}\text{x}T^{2} + 5.6674\text{x}10^{-3}\text{x}T - 7.7134\text{x}10^{-2}$$

Standard deviation=$2.560198\text{x}10^{-2}$

Maximum % deviation=4.673%

Maximum deviation=$6.2669\text{x}10^{-4}$ cp

Mean deviation=$1.577\text{x}10^{-16}$ cp

A equation of the form, $\eta = A_1\sqrt{T}°K + B_1$ where A_1 & B_1 are constants, was also fitted to the R-13 viscosity values. This equation is an approximation of Sutherland's equation, which is given by,

$$\eta = \frac{AT^{3/2}}{(B+CT)}$$; where A,B,C are constants.

The constants for the fitted equation are:

$A_1=1.5919\text{x}10^{-1}$, $B_1= -1.2902$

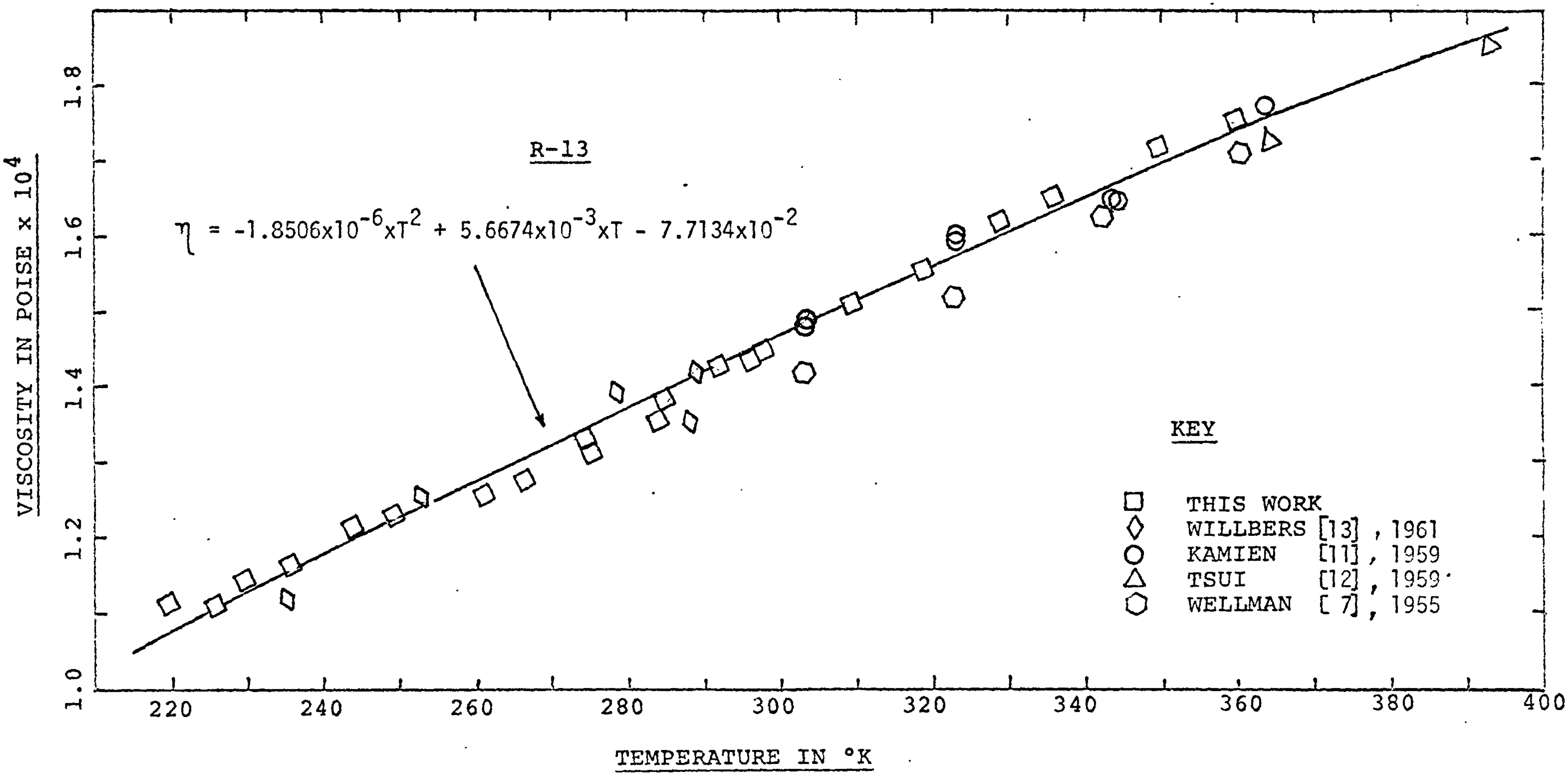
Standard deviation=$1.5327\text{x}10^{-2}$

Maximum % deviation=2.64028%

Maximum deviation=$3.4767\text{x}10^{-4}$ cp

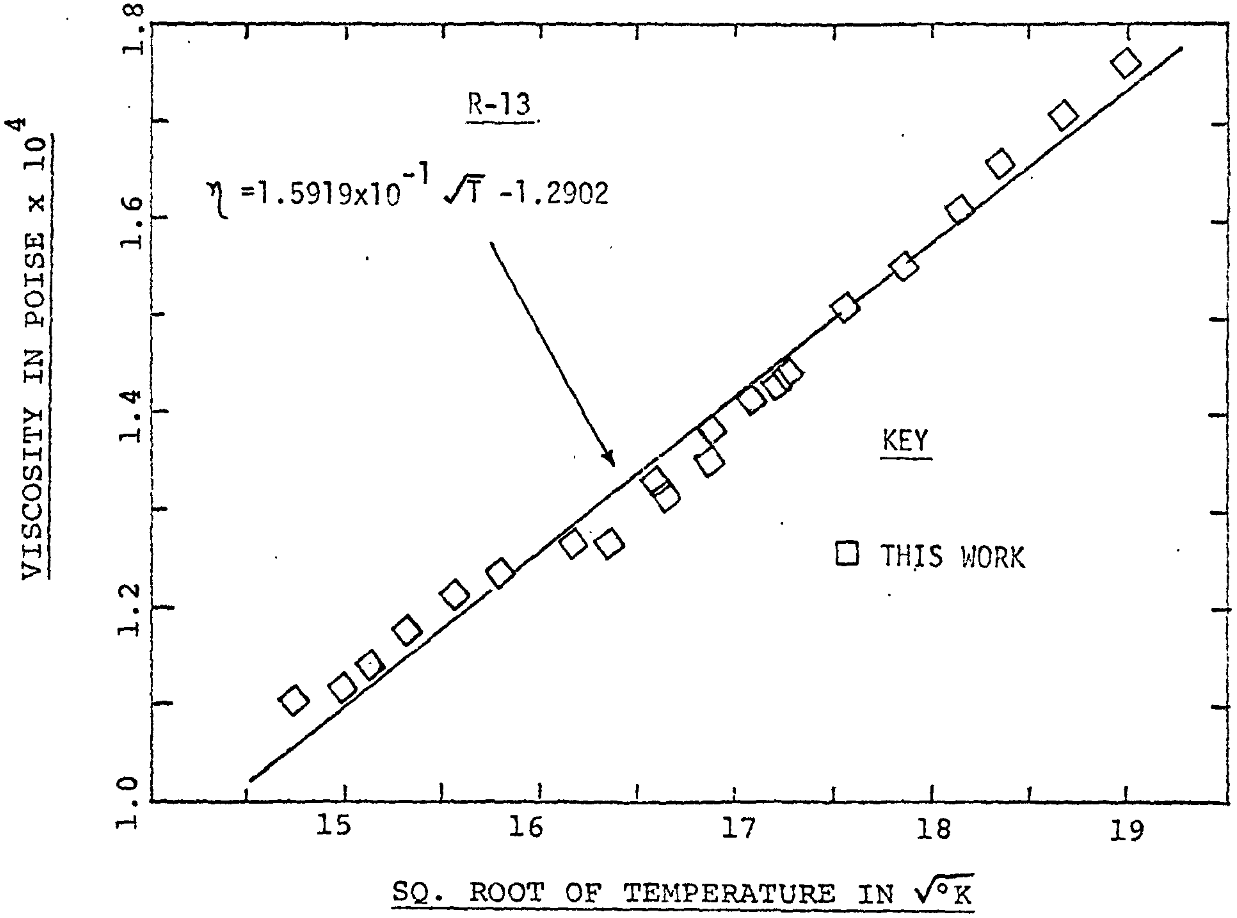
Mean deviation=$1.59026\text{x}10^{-16}$ cp

The plot of the above mentioned equation is shown in fig. 15 . Fig.16 shows the deviation plot.



2nd DEGREE POLYNOMIAL; VISCOSITY Vs TEMPERATURE, COMBINED RESULTS.

Fig. 14



VISCOSITY OF R-13 Vs TEMPERATURE IN $\sqrt{°K}$

WHEN FITTED TO $\eta = A_1\sqrt{T} + B_1$

FIG. 15

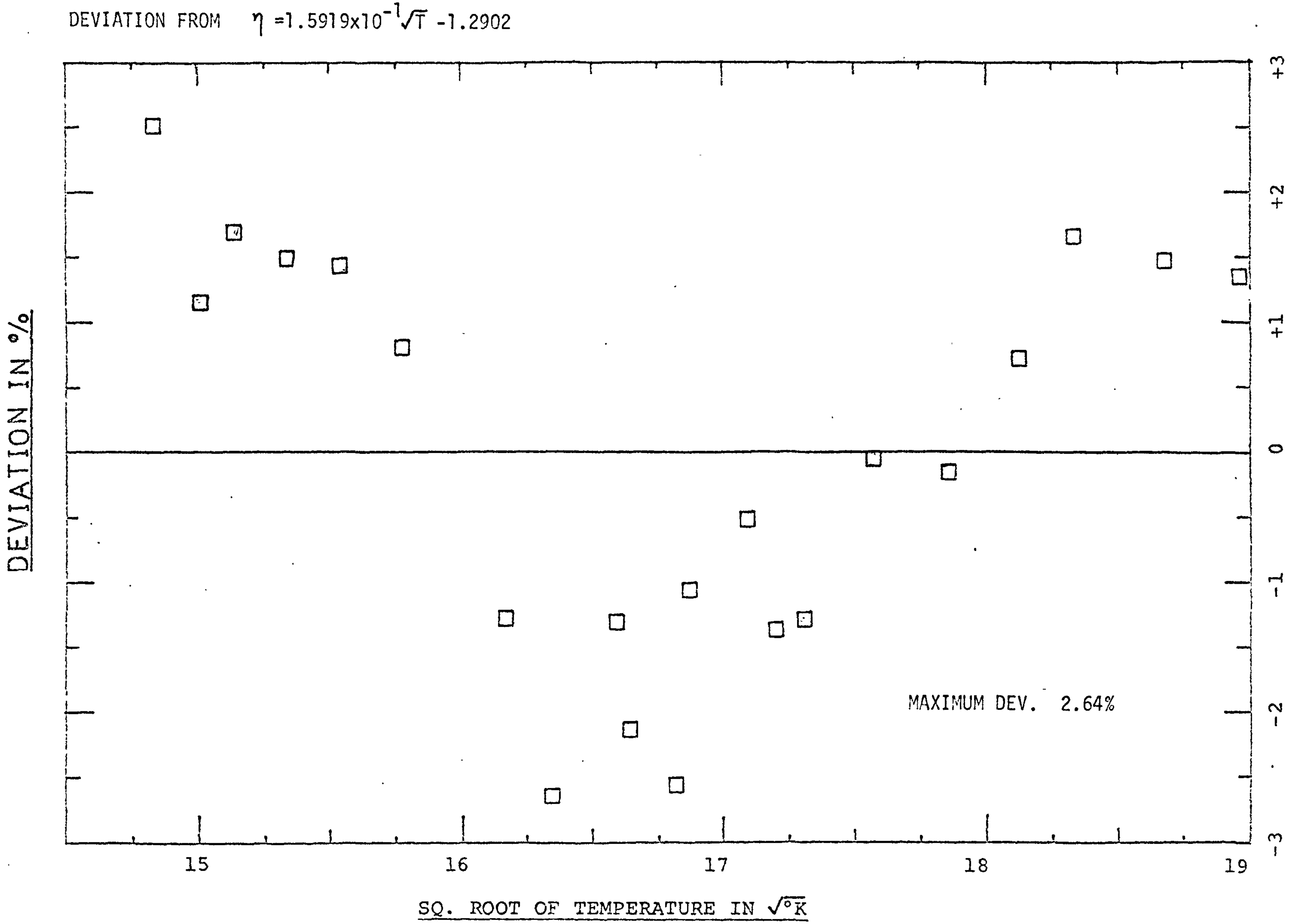


FIG.16 DEVIATION OF EXPERIMENTAL RESULTS FOR R-13 FROM THE FITTED EQUATION $\eta = A_1\sqrt{T} + B_1$

A combined plot of the available experimental data and the results of this work is shown in fig.17. Constants for the approximate Sutherland's equation in this case are:

$A_1 = 1.57618 \times 10^{-1}$, $B_1 = -1.26915$

Standard deviation=1.8999×10^{-2}

Maximum % deviation=4.98%

Maximum deviation=6.68×10^{-4} cp

Mean deviation=8.717×10^{-16} cp

Table 3 shows the viscosity coefficient of R-500 in the temperature range of -28.64° to 87.38°C. Polynomials of 1 to 5 degrees were fitted in the data and 4th degree equation was found to be most suitable. The results are shown in fig.18 . The resultant polynomial fitted is,

$$\eta = -3.44123 \times 10^{-9} \times T^4 + 4.2545 \times 10^{-6} \times T^3 - 1.9641 \times 10^{-3} \times T^2 + 4.05348 \times 10^{-1} \times T - 30.6097$$

Standard deviation=1.6064×10^{-2}

Maximum % deviation=2.048%

Maximum deviation=2.4334×10^{-4} cp

Mean deviation= 8.287×10^{-15} cp

Fig. 19 shows the scatter of the experimental values from the fitted polynomial; all the points lie within ±2.048%. Excepting one point all other lie within ±0.95%

Comparision of the viscosity values obtained in this

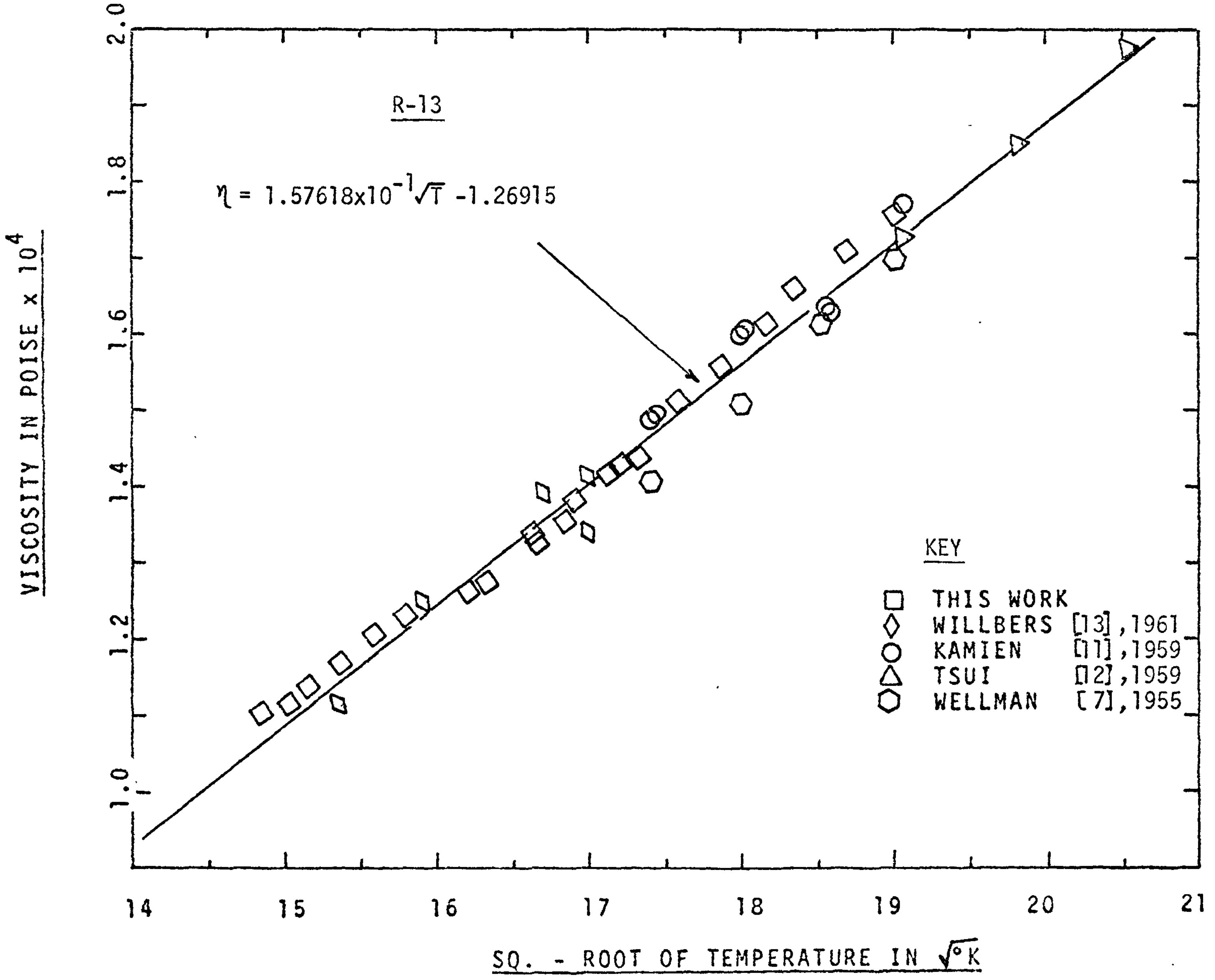


FIG. 17 EQUATION $\eta = A_1\sqrt{T} + B_1$; VISCOSITY V_s SQ.-ROOT OF TEMPERATURE, COMBINED RESULTS.

TABLE 3

VISCOSITY OF R-500

Temp. °K	Viscosity Poise x 10^4
244.507	0.9720
252.320	1.0120
257.997	1.0451
267.966	1.0884
275.237	1.1343
282.330	1.1632
293.132	1.1932
297.990	1.1882
305.345	1.2457
313.864	1.2821
321.680	1.3102
328.894	1.3472
339.130	1.3797
349.283	1.4221
360.537	1.4660

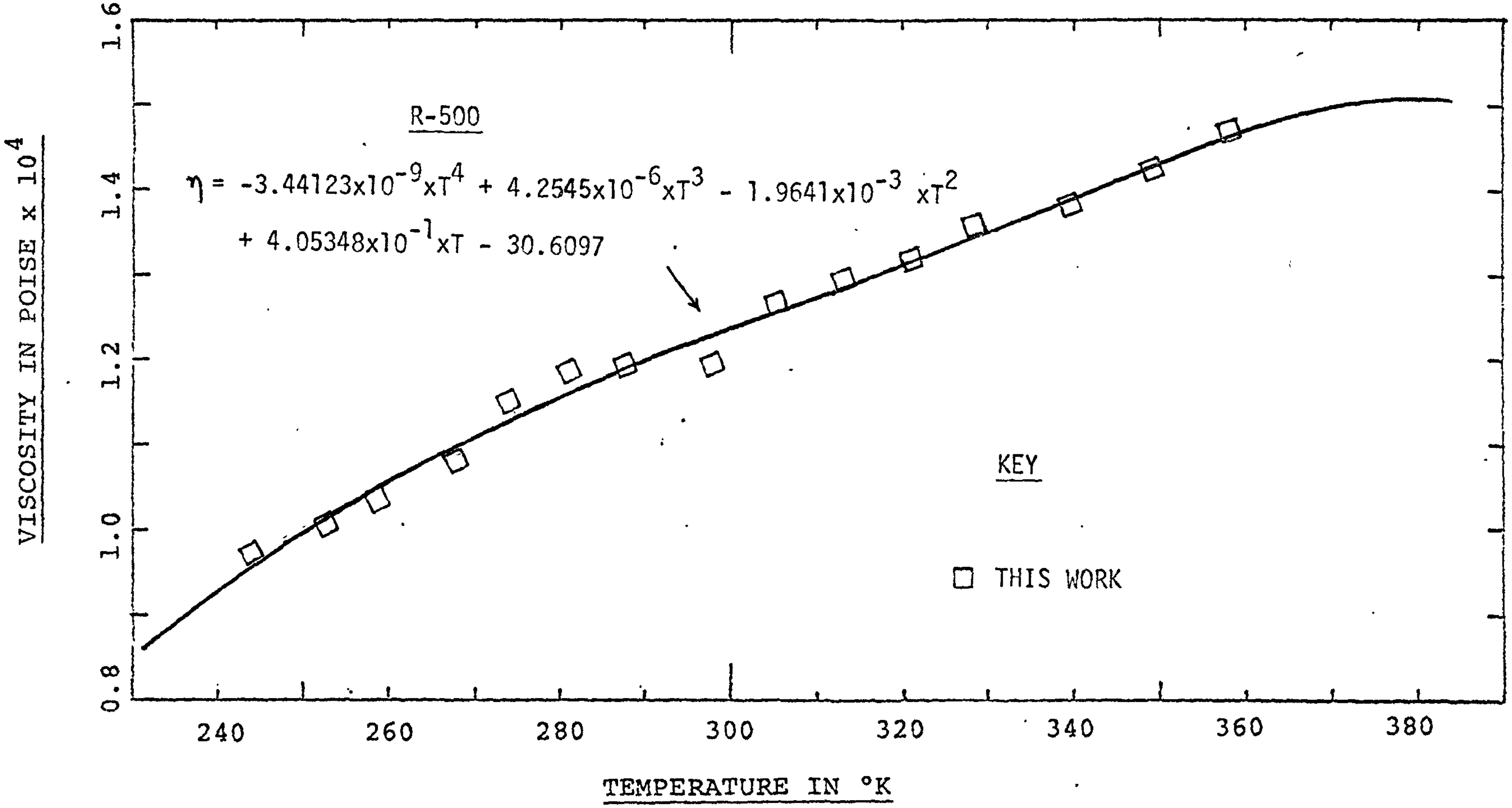
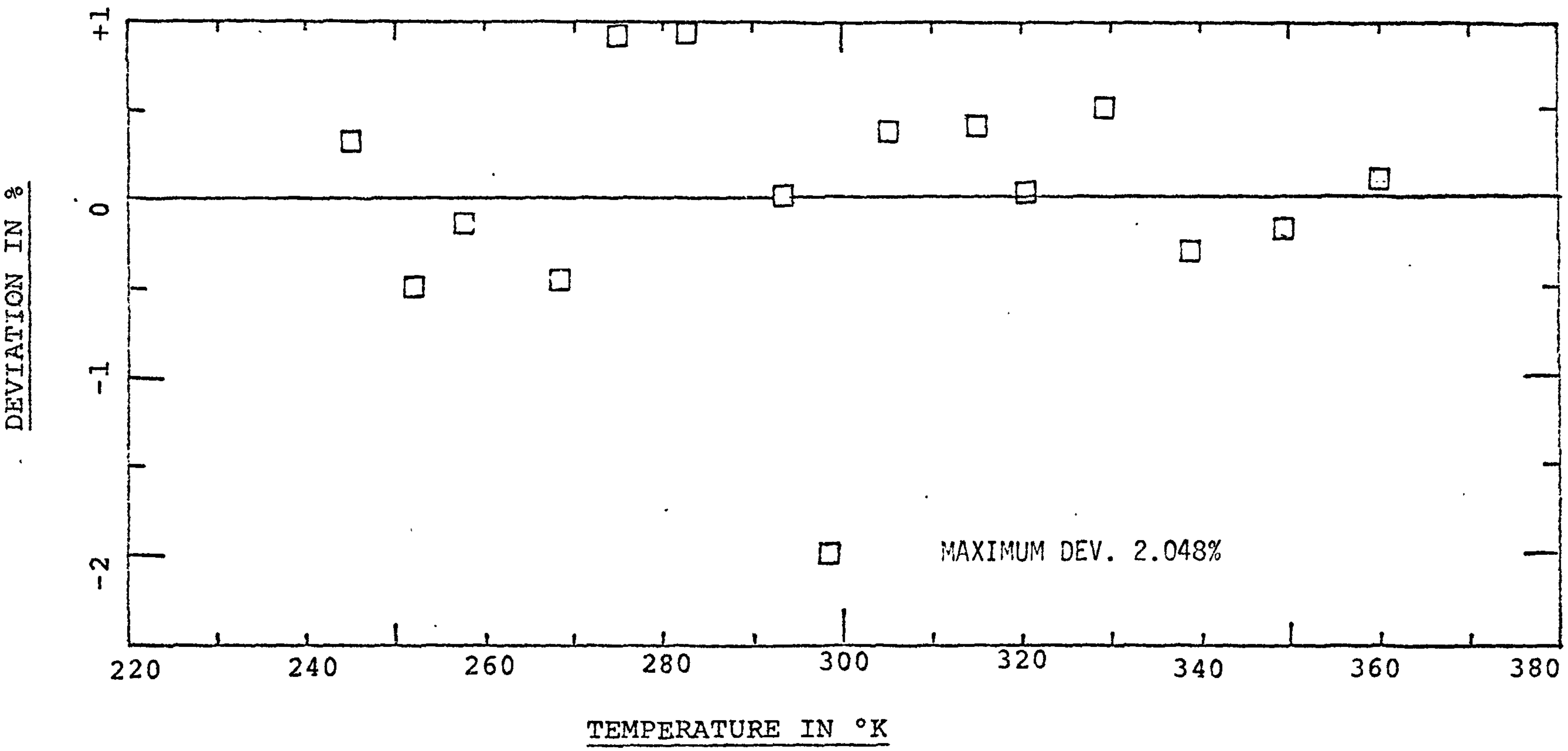


Fig. 18 VISCOSITY OF R-500 Vs TEMPERATURE WHEN FITTED TO 4th DEGREE POLYNOMIAL

DEVIATION FROM $\eta = -3.44123\text{x}10^{-9}\text{x}T^4 + 4.2545\text{x}10^{-6}\text{x}T^3 - 1.9641\text{x}10^{-3}\text{x}T^2 + 4.05348\text{x}10^{-1}\text{x}T - 30.6097$



DEVIATION OF EXPERIMENTAL RESULTS FOR R-500 FROM

THE FITTED 4th DEGREE POLYNOMIAL

FIG. 19

work with those shown in the Du Pont's publication [24], under the heading 'Estimated Viscosity of Freon-500' is shown in the fig. 20.

Approximate Sutherland's equation was fitted into the results of this work, the plot of which is shown in fig. 21. The constants for the fitted equation are;

$A_1= 1.4458\mathrm{x}10^{-1}$, $B_1=-1.28058$

Standard deviation=$8.1748\mathrm{x}10^{-2}$

Maximum % deviation=2.27023%

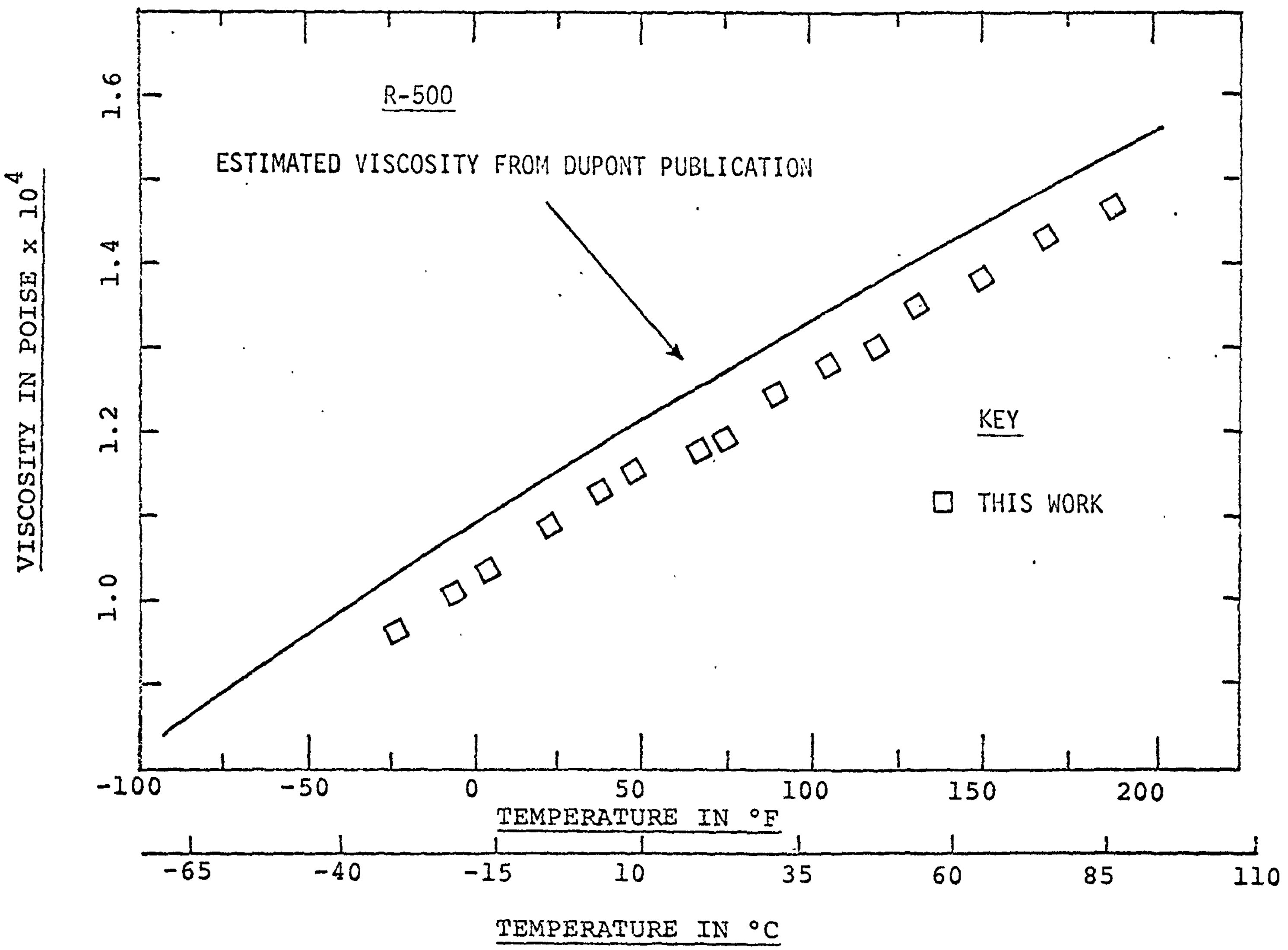
Maximum deviation=$2.6975\mathrm{x}10^{-4}$ cp

Mean deviation=$3.8132\mathrm{x}10^{-16}$ cp

Fig. 22 shows the deviation plot showing scatter of the experimental results from the fitted Sutherland type equation. All the points lie within ±2.27% and excepting one point, all other lie within ±1.43%

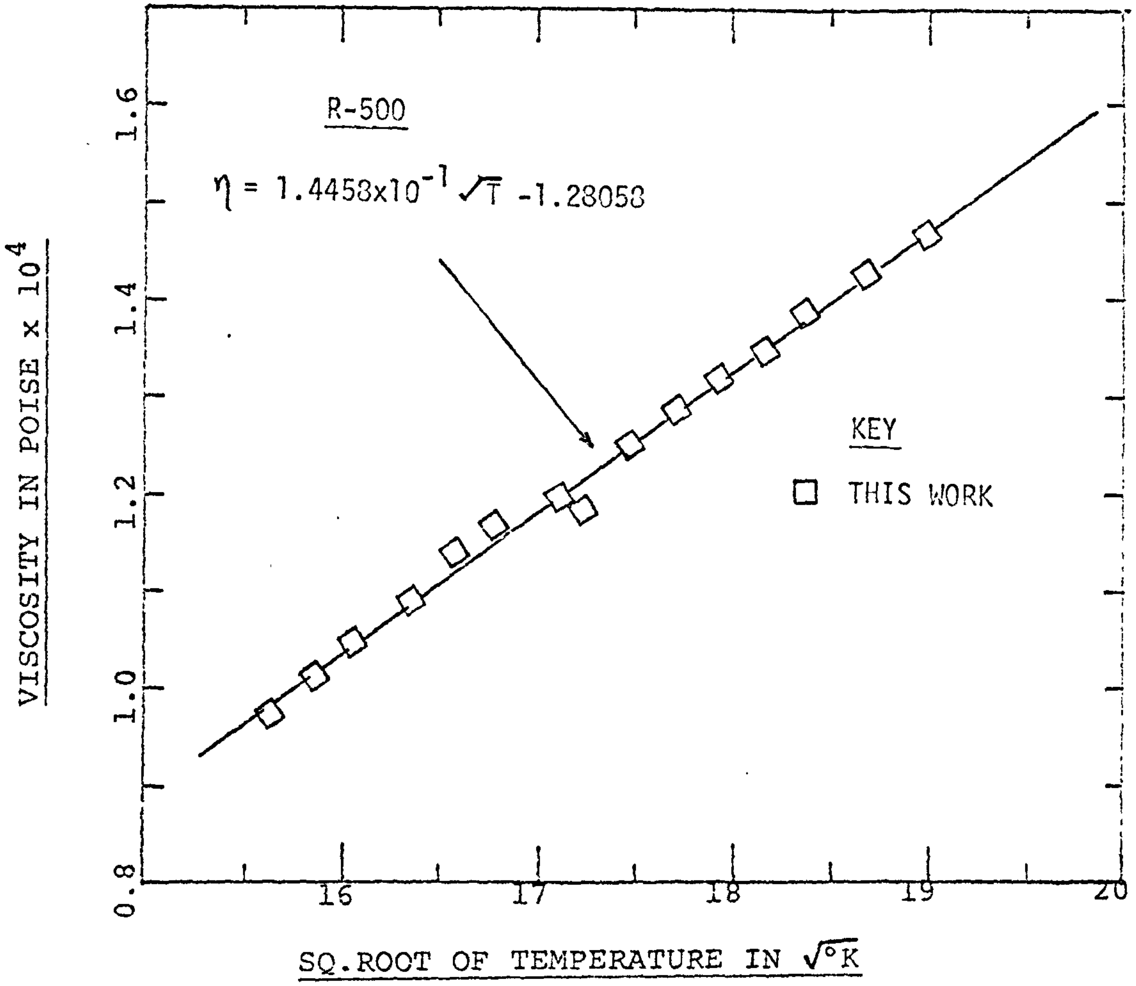
Though the approximate form of Sutherland's equation is a semi-empirical relation derived from the kinetic theory of gases, the polynomial are merely statistical correlations of the experimental data and so the selection of the suitable polynomial has only the statistical basis. Therefore, it was thought desirable to correlate the data using a more rigorous theory.

Viscosity of many gases have been correlated using collision theory. Although, many mathematical models have



COMPARISION OF EXPERIMENTAL RESULTS FOR R-500 WITH THE ESTIMATED VISCOSITY REPORTED IN DUPONT PUBLICATION RT-41

FIG. 20



VISCOSITY OF R-500 Vs TEMPERATURE IN $\sqrt{^\circ K}$

WHEN FITTED TO $\eta = A_1 \sqrt{T} + B_1$

FIG. 21

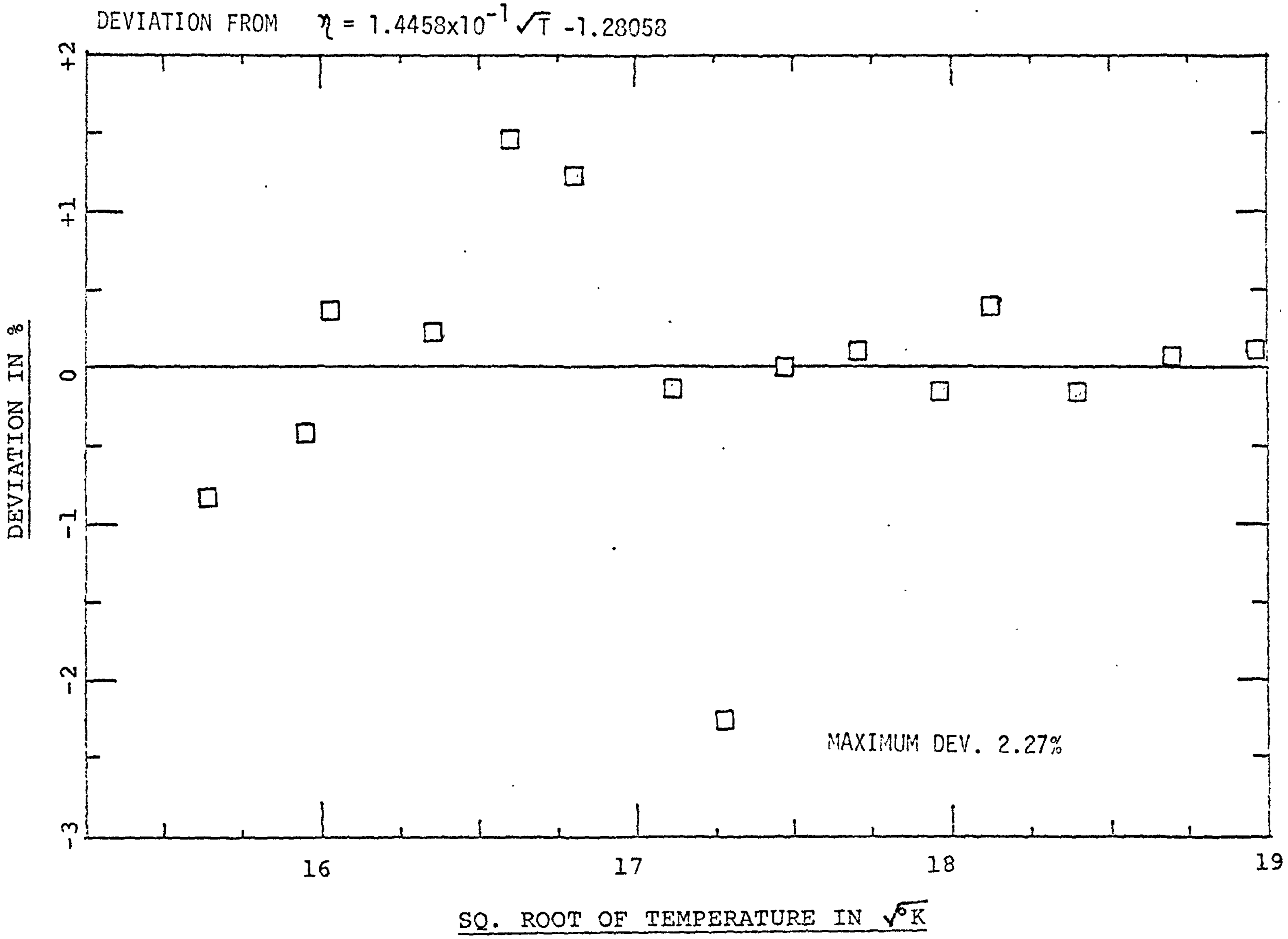


FIG .22 DEVIATION OF EXPERIMENTAL RESULTS FOR R-500 FROM THE EQUATION $\eta = A_1 \sqrt{T} + B_1$

been developed, the Lennard-Jones (L-J 6-12) model has been found most satisfactory for the non-polar gases. For polar gases, however, stockmayer's modification of this model has been quite useful in correlating the viscosity values (for more details see appendix 9.2).

$$E(r) = 4\varepsilon[(\frac{r_o}{r})^{12} - (\frac{r_o}{r})^6] \tag{7.1}$$

$$E(r) = 4\varepsilon[(\frac{r_o}{r})^{12} - (\frac{r_o}{r})^6 - \delta(\frac{r_o}{r})^3] \tag{7.2}$$

where $E(r)$ is Intermolecular potential field

ε is Maximum energy of attraction

r_o is Low velocity collision diameter

r is Radial separation between the colliding molecules

and δ is Reduced dipole energy.

Equation 7.1 is Lennard Jones potential for non-polar gases and equation 7.2 is Stockmayer potential for polar gases, modified by Krieger for end on collision such that

$$\delta = \frac{\mu^2}{2\varepsilon r_o{}^3} \tag{7.3}$$

where μ is Dipole moment.

Chapman and Cowling correlated transport properties with intermolecular potential in terms of a set of integrals $\Omega^{(l,s)}$. They used the potentials given in equations 7.1 and 7.2.

On the basis of Chapman and Cowling's integrals, Hirschfelder, Bird and Spotz [26] expressed viscosity as

$$\eta = 2.6693 \times 10^{-5} \frac{(MT)^{1/2}}{r_o^2 W^{(2)}(2)} \text{ gms/cm sec.} \qquad (7.4)$$

where M is Molecular weight expressed in mass units.

Functions $W^{(2)}(2)$ were tabulated against reduced temperature T* and δ, T* being $\frac{kT}{\varepsilon}$; where k is Boltzmann constant.

The value of electric dipole moment (μ) for R-13 was obtained from the 'Selected values of electric dipole moments for molecules in the gas phase' an NBS publication [24], given as 0.5 debyes, but the value of μ for R-500 could not be located. Since the constituents of R-500 (CCl_2F_2/CH_3CHF_2; 73.8/26.2% by weight) are both polar, R-500 will be polar gas-mixture. But since a mathematical model for a mixture of two polar gases was not immediately available, it was decided to correlate the experimental results of R-500 assuming it to be non-polar. Therefore, it should be emphasized here that though, the intermolecular force constants were computed for both R-13 and R-500, the constants for R-500 bear no physical significance and are merely empirical constants for the correlation of the experimental results.

Using equations 7.3 and 7.4, intermolecular force constants were computed by cross interpolation. For detail see appendix 9.2.

The experimental data were then compared with the viscosity values calculated from equations 7.3 and 7.4.

Intermolecular force constants and the deviations of experimental results from the collision theory are as follows:

R-13 Polar gas.

$\delta = 0.0355$; $r_o = 4.685$ Angstroms; $\frac{\varepsilon}{k} = 248.792^{\circ}K$

Standard deviation = 1.217×10^{-2}; Maximum % deviation = 2.14%

Maximum deviation = 2.511×10^{-4} cp; Mean deviation = 2.58×10^{-7} cp

R-500; Assumed to be non-polar, (i.e. $\mu = 0$ and therefore, $\delta = 0$)

$r_0 = 4.9864$ Angstroms; $\frac{\varepsilon}{k} = 268.7815^0$K

Standard deviation = 1.26×10^{-2}; maximum deviation = 3.129×10^{-4} cp

Mean deviation = 5.636×10^{-7} cp, maximum % deviation = 3.219%

Figs. 23 and 26 show the comparison of the experimental results with collision theory and Figs. 24 and 27 are the deviation.

A comparison of (a) the polynomial and (b) collision theory, is shown in Figs. 25 and 28 for R-13 and R-500 respectively.

Table 4 shows the viscosity values of R-13 for a wide range of temperature (from 200^{0}K to 1650^{0}K), computed from the collision theory.

Recommended Correlations

Following are the correlations recommended for R-13 and R-500.

R-13

(i) A 2nd degree polynomial

$$\eta = 6.43206 \times 10^{-6} \times T^2 + 1.05245 \times 10^{-3} \times T + 5.5777 \times 10^{-1}$$

(ii) Correlation based on collision theory, i.e. equation 7.3 and 7.4 with intermolecular force constants:

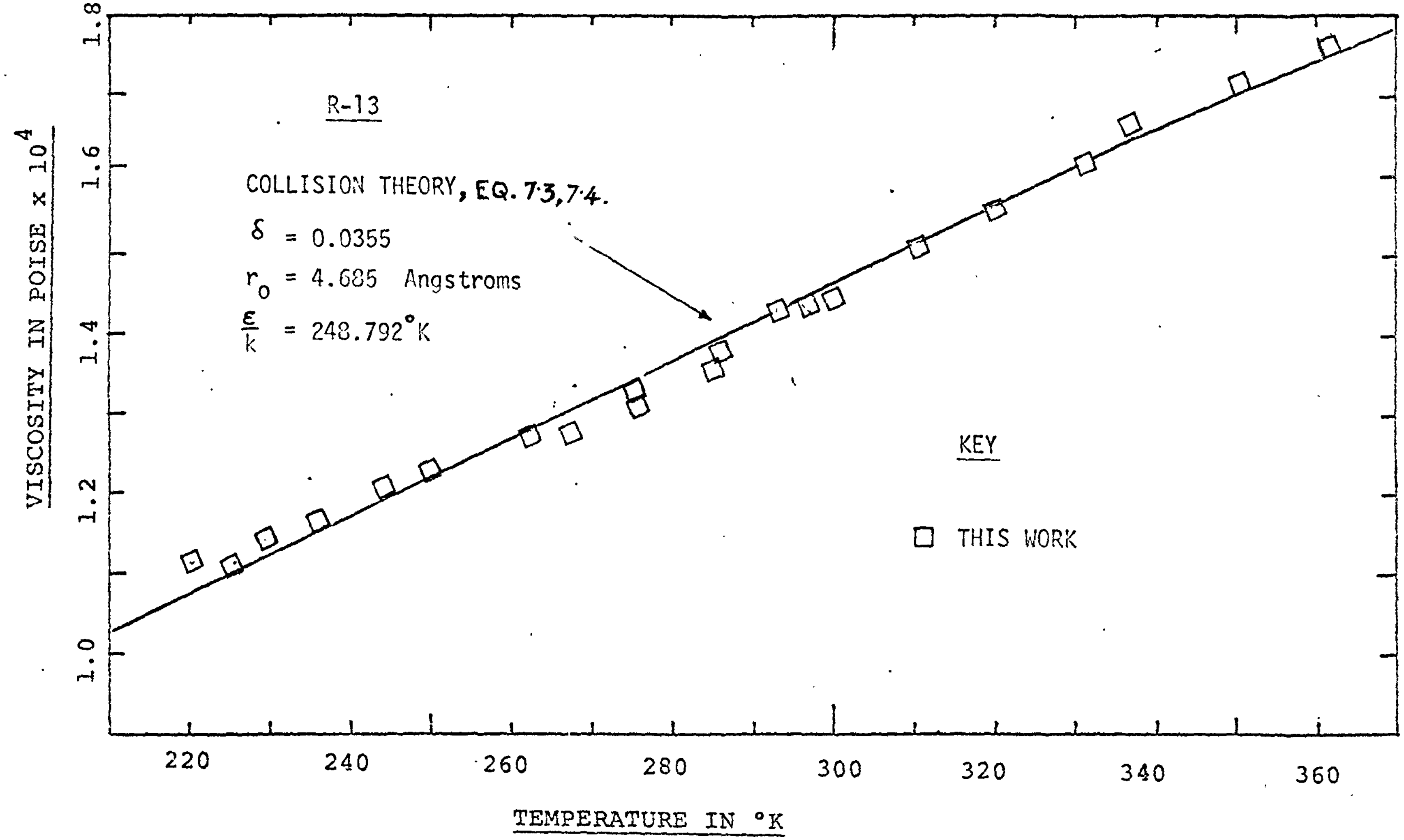
$\delta = 0.0355$; $r_0 = 4.685$ Angstroms; $\frac{\varepsilon}{k} = 248.792^0$K

See table 4 for the generated data.

R-500

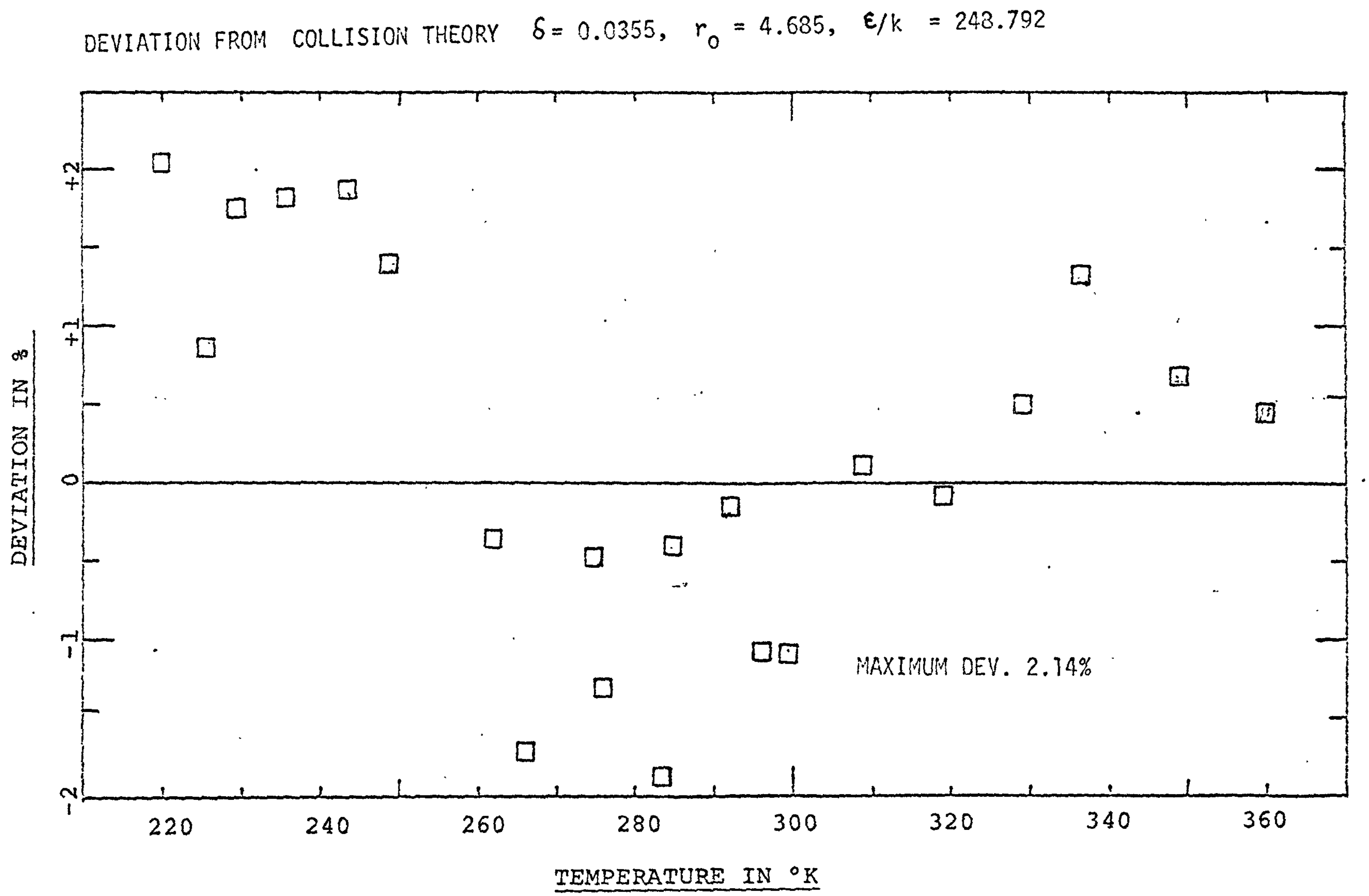
A 4th degree polynomial

$$\eta = -3.44123 \times 10^{-9} \times T^4 + 4.2545 \times 10^{-6} \times T^3 - 1.9641 \times 10^{-3} \times T^2 + 4.05348 \times 10^{-1} \times T - 30.6097$$



VISCOSITY OF R-13 Vs TEMPERATURE; COMPARED WITH COLLISION THEORY.

FIG. 23



DEVIATION OF EXPERIMENTAL RESULTS FOR R-13 FROM THE COLLISION THEORY

FIG. 24

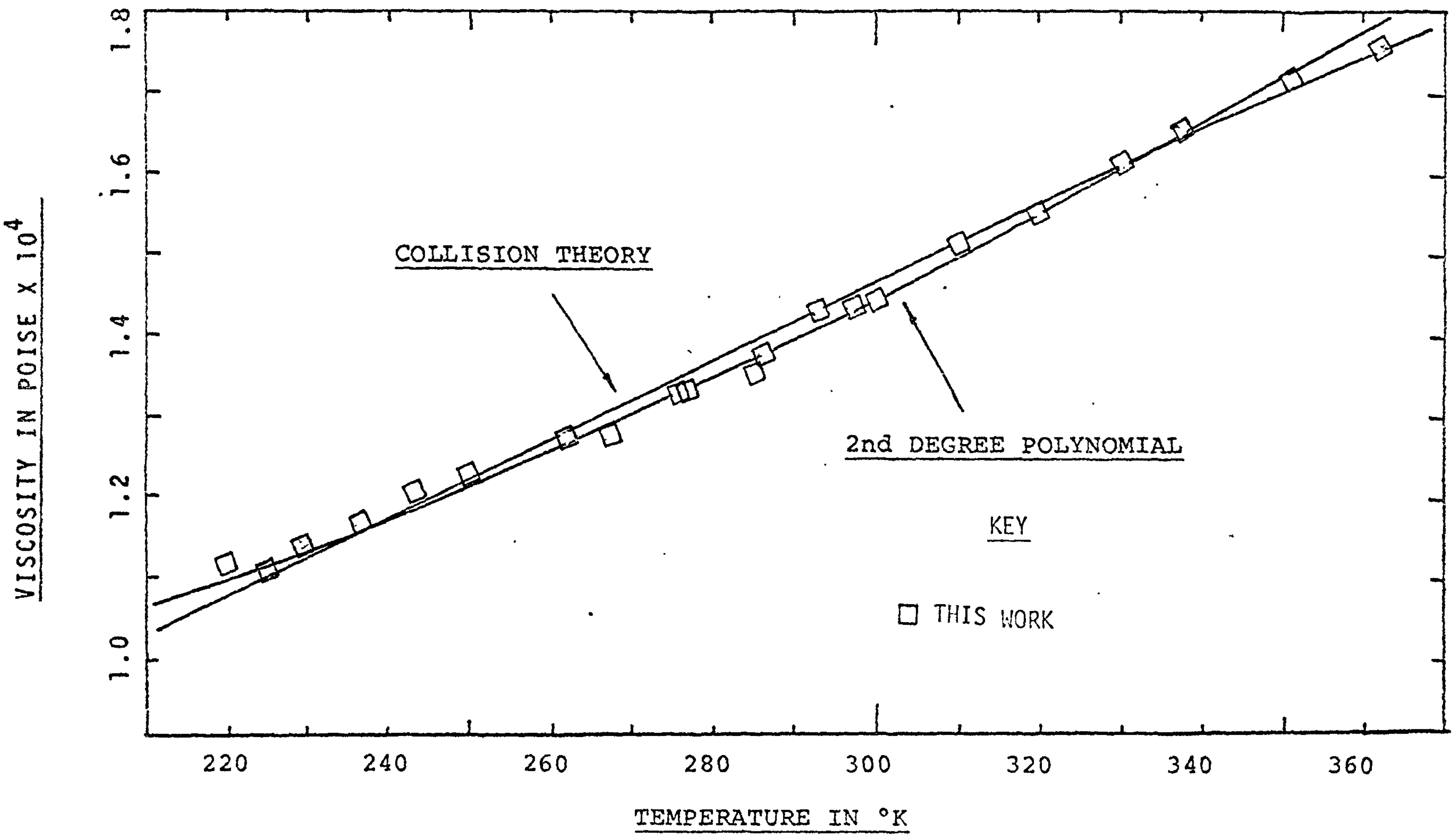


FIG. 25 COMPARISION OF VISCOSITY VALUES FOR R-13 WITH THE COLLISION THEORY AND 2nd DEGREE POLYNOMIAL.

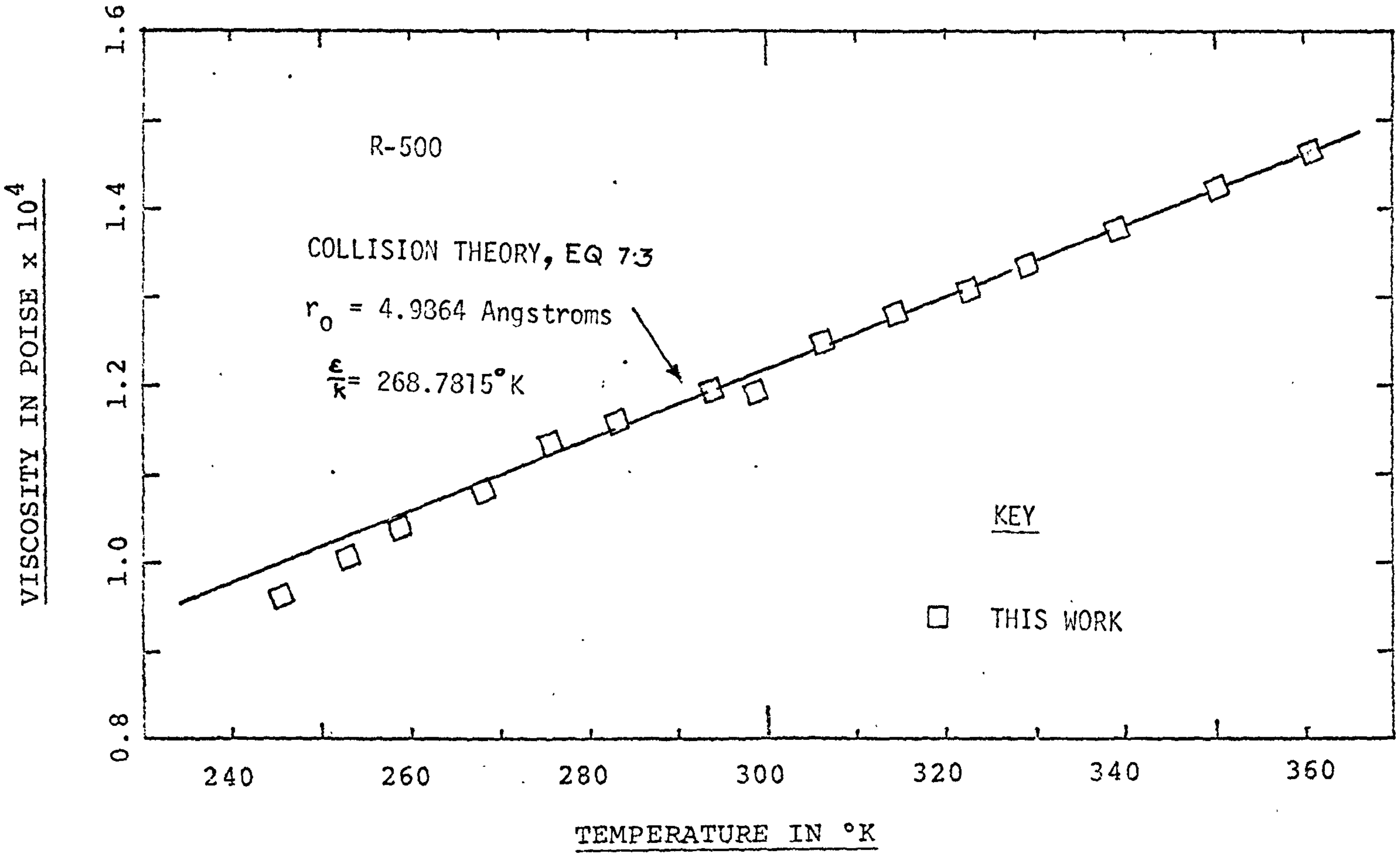
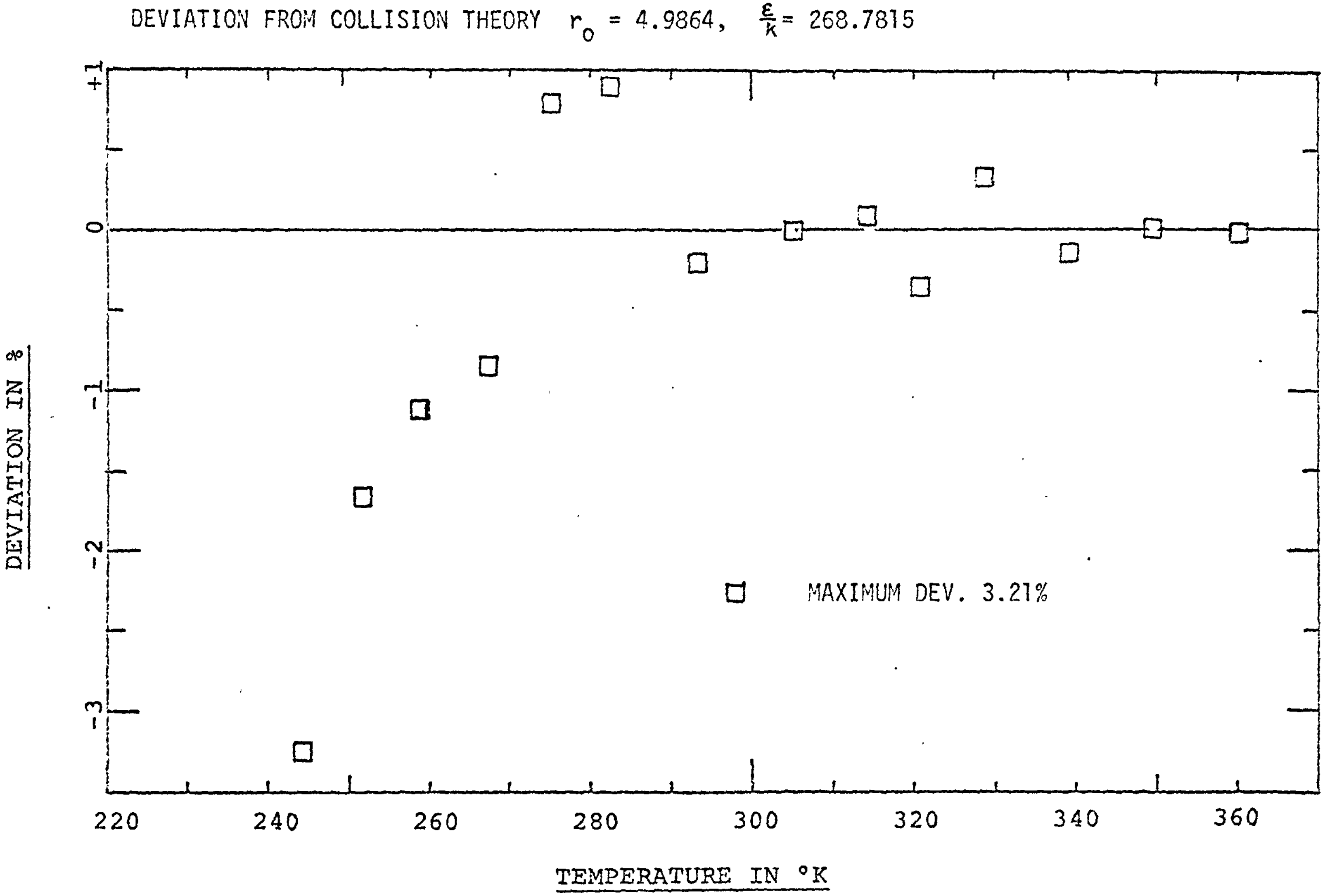


Fig. 26 VISCOSITY OF R-500 Vs TEMPERATURE, COMPARED WITH COLLISION THEORY.



DEVIATION OF EXPERIMENTAL RESULTS FOR R-500 FROM THE COLLISION THEORY

FIG. 27

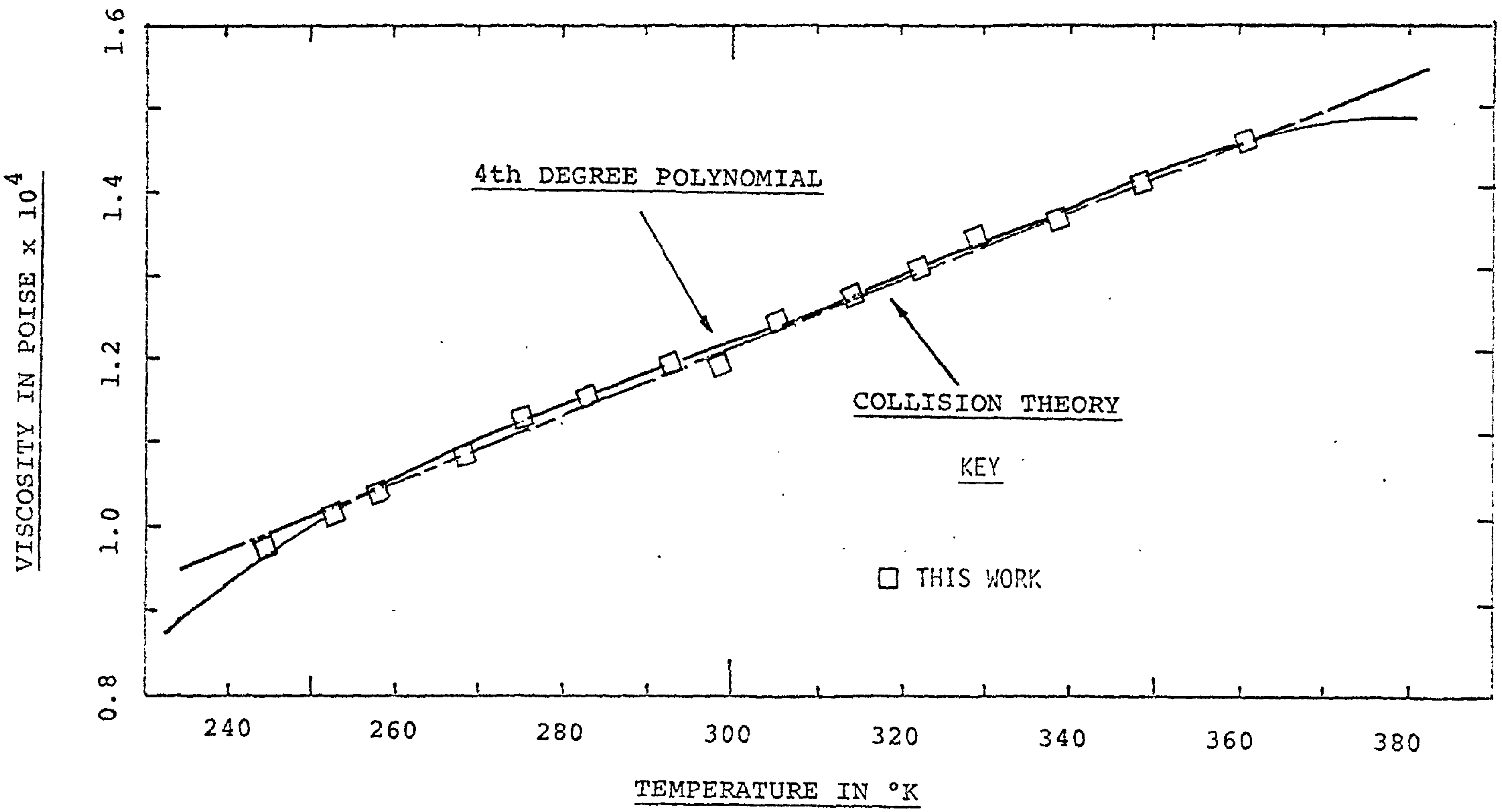


Fig. 28 COMPARISION OF VISCOSITY VALUES OF R-500 WITH THE COLLISION THEORY AND 4th DEGREE POLYNOMIAL

TABLE 4

VISCOSITY OF R-13, CALCULATED USING COLLISION THEORY

Temp. °K	Viscosity Poise x 10^4
200.0	0.9767
210.0	1.0251
220.0	1.0752
230.0	1.1234
240.0	1.1710
250.0	1.2194
260.0	1.2660
270.0	1.3138
280.0	1.3630
290.0	1.4136
300.0	1.4640
310.0	1.5098
320.0	1.5566
330.0	1.6044
340.0	1.6532
350.0	1.7015
360.0	1.7458
370.0	1.7908
380.0	1.8365
390.0	1.8830
400.0	1.9287

Cont'd

Temp. °K	Viscosity Poise x 10^4
450.0	2.1455
500.0	2.3530
550.0	2.6609
600.0	2.7410
650.0	2.9239
700.0	3.1001
750.0	3.2703
800.0	3.4353
850.0	3.5954
900.0	3.7510
950.0	3.9024
1000.0	4.0496
1050.0	4.1880
1100.0	4.3265
1150.0	4.4655
1200.0	4.6049
1250.0	4.7425
1300.0	4.8994
1350.0	4.9962
1400.0	5.1230
1450.0	5.2500
1500.0	5.3752
1550.0	5.4935
1600.0	5.6117
1650.0	5.7298

8. CONCLUSION

A series viscometer was designed and developed for the measurement of the absolute viscosity of fluids at atmospheric pressure and very low temperatures.

The viscosity of R-13 and R-500 was measured at atmospheric pressure and the temperature range - 53.25 to 87.19°C and - 28.64 to 87.38°C, respectively. The overall accuracy of the measured results is estimated to be better than ± 1.64%.

The present data and other available data have been correlated using an nth degree polynomial and an approximate form of the Sutherland equation, $\eta = A_1\sqrt{T} + B_1$, and equations have been recommended together with their estimated accuracy.

The present data have also been correlated using Chapman and Cowling Collision theory, which yielded intermolecular force constants for R-13 and equivalent intermolecular force constants for R-500. A table of recommended data for a wide range of temperature, 200 to 1650°K, has been generated based on the collision theory approach, and are presented.

9. APPENDICES

9.1 EQUATION FOR 'TWO TUBES IN SERIES' VISCOMETER

Considering general equation of motion, the axial component can be written as

$$\rho\left[\frac{\partial v_z}{\partial \theta} + v_r \frac{\partial v_z}{\partial r} + \frac{v_\phi}{r} \cdot \frac{\partial v_z}{\partial \phi} + v_z \frac{\partial v_z}{\partial z}\right]$$

$$= - \frac{\partial p}{\partial z} - \left[\frac{1}{r}\frac{\partial}{\partial r}(r s_{rz}) + \frac{1}{r}\frac{\partial s_{\phi z}}{\partial \phi} + \frac{\partial s_{zz}}{\partial z}\right] + \rho g_c \quad (9.1.1)$$

where ρ is density of fluid

r, z, ϕ are the co-ordinates

v is the velocity

and ρg_c is the body force

For the case considered:

i) flow is assumed to be steady so $\frac{\partial v_z}{\partial \theta} = 0$

ii) no radial and angular components of velocity so v_r and v_ϕ are zero.

iii) no variation of velocity in z direction, i.e. $\frac{\partial v_z}{\partial z} = 0$

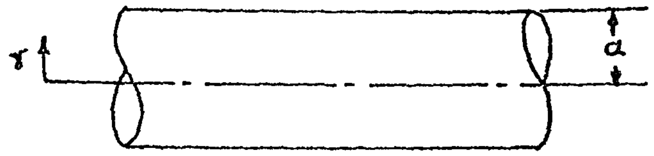
iv) stress components $s_{\phi z}$ and s_{zz} are zero.

Therefore, equation (9.1.1) reduces to

$$0 = - \frac{\partial p}{\partial z} - \left[\frac{1}{r}\frac{\partial}{\partial r}(r s_{rz})\right] + \rho g_c$$

or

$$r\left[\rho g_c - \frac{\partial p}{\partial z}\right] = \frac{\partial}{\partial r}(r s_{rz})$$



Integrating, $\frac{r^2}{2}\ [\rho g_c - \frac{\partial p}{\partial z}] + c = rs_{rz}$

Putting value of $s_{rz} = -\eta[\frac{\partial v_z}{\partial r} + \frac{\partial v_r}{\partial z}]$

But since $v_r = 0$, $\frac{\partial v_r}{\partial z} = 0$ we get

$$-\eta\ \frac{\partial v_z}{\partial r} = \frac{r}{2}\ [\rho g_c - \frac{\partial p}{\partial z}] + c$$

But at $r = 0$, $s_{rz} = 0$, $\frac{\delta v_z}{\delta r} = 0$ and $c = 0$

Integrating again,

$$v_z = \frac{r^2}{4\eta}\ [\rho g_c - \frac{\partial p}{\partial z}] + c_1$$

But at $r = a$, $v_z = 0$ (neglecting slip) we get

$$v_z = [\rho g_c + \frac{\partial p}{\partial z}]\ [\frac{a^2}{4\eta}]\ [1 - (\frac{r}{a})^2]$$

Assuming pressure gradient to be linear, and neglecting body forces;

$$v_z = \frac{\Delta p}{4\eta L}\ (a^2 - r^2) \qquad (9.1.2)$$

Equation (9.1.2) shows that velocity is parabolic function of radius in a pipe flow.

Now, flow passing through the annular area of a ring of radii r and r + dr is

$$dQ = 2\pi r \cdot dr \cdot v_z$$

where v_z is the velocity in z direction.

Therefore, volumetric flow rate will be

$$Q = 2\pi\ \frac{\Delta p}{4\eta L}\ \int_0^a (a^2 r - r^3)$$

$$= \frac{\pi a^4 \Delta p}{8\eta L}$$

This gives $\eta = \frac{\pi a^4}{8Q} \frac{\Delta p}{L}$ (9.1.3)

Equation (9.1.3) is Poiseuille Equation.

The equation (9.1.3) is valid only if the parabolic profile exists in whole of the length 'L' of the tube. However, the fluid is at rest upstream of the entrance, the velocity profile at the entrance is approximately uniform but develops the parabolic profile after some length (the development length). Therefore, taking the case of capillary tube connected between the two chambers, if the pressures are measured in the two chambers, the difference in pressure would be the sum of pressure drops due to viscous forces and due to the velocity development at the entrance, i.e.

$$p_{vis} = p_m - p_d$$

where p_{vis} is pressure drop due to viscous forces
p_m is pressure drop measured
p_d is pressure drop due to velocity development

Boussinesq argued that if the mean rate of discharge were established without friction, giving $v = \frac{Q}{\pi a^2}$ uniform over whole cross-section of the tube, the pressure drop, according to Bernoulli's theorem would be the kinetic energy per unit volume, i.e. $1/2\ \rho v^2$. Due to parabolic profile, having a maximum velocity of 2v, a further loss of 1/2 ρv^2 results. [After comparing the kinetic energy losses per unit volume for both types of velocity profiles.] If friction, i.e. viscous forces, are also taken into account for the development length, there is a further loss of 0.12 ρv^2. The total loss is then 1.12 ρv^2, or expressing m as the numerical factor associated with ρv^2, it is $m\rho v^2$ where m according to Boussinesq is 1.12. A range of values for m have been reported in the literature (see Barr [18]).

The equation now becomes

$$\eta = \frac{\pi a^4}{8QL}\left[\Delta p - m\rho\left(\frac{Q}{\pi a^2}\right)^2\right]$$

$$= \frac{\pi a^4}{8Q}\ \frac{\Delta p}{L} - \frac{m\rho Q}{8\pi L} \qquad (9.1.4)$$

The fluid enters the capillary tube in the form of a converging stream and comes out in a divergent stream. Any differences in velocity between adjacent stream lines in these streams will require the expenditure of energy in overcoming viscous forces. The correction for this, known as 'Couette Correction' can be applied by hypothetically increasing the length of the capillary tube by a factor λ which is dependent on the bore size and the shape of the ends of the capillary tube. The value of n = λ/a for different shapes, as found out by different workers, lies between 0 to 6.

After applying the correction

$$\eta = \frac{\pi a^4 \Delta p}{8Q(L + na)} - \frac{m\rho Q}{8\pi(L + na)} \qquad (9.1.5)$$

It is to be noted that the effect of both the corrections is to lower the viscosity value.

Allowing for the thermal expansion or contraction of the capillary tubes, equation (9.1.5) becomes

$$\eta = \frac{\pi[a(1 + \alpha t)]^4 \Delta p}{8Q(L + na)(1 + \alpha t)}$$

$$- \frac{m\rho Q}{8\pi(L + na)(1 + \alpha t)} \qquad (9.1.6)$$

where α is the coefficient of thermal expansion, assuming it to be isotropic.

Neglecting α^2, α^3 etc., the higher power of α, which would be very small since α itself is very small, we get

$$\eta = \frac{\pi a^4 \Delta p(1 + 3\alpha t)}{8Q(L + na)} - \frac{m\rho Q}{8\pi(L + na)(1 + \alpha t)} \qquad (9.1.7)$$

Equation (9.1.7) is the representative equation of single capillary tube viscometer.

Let subscripts l and s represent large and small capillary tubes.

Then

$$\eta = \frac{\pi a_i^4 \Delta p_i (1 + 3\alpha t)}{8Q(L_i + n_i a_i)} - \frac{m_i Q \rho}{8\pi(1 + \alpha t)(L_i + n_i a_i)}$$

where $i = l, s$.

$$m_i = \frac{8\pi(1 + \alpha t)(L_i + n_i a_i)}{Q\rho} \left[\frac{\pi a_i^4 \Delta p_i (1 + 3\alpha t)}{8Q(L_i + n_i a_i)} - \eta\right]$$

Substituting this for m_l in the equation for small tube, assuming $m_l = m_s$

$$\eta = \frac{\pi a_s^4 \Delta p_s (1 + 3\alpha t)}{8Q(L_s + n_s a_s)} - \frac{8\pi(1 + \alpha t)(L_l + n_l a_l)}{Q\rho} \times$$

$$\left[\frac{\pi a_l^4 \Delta p_l (1 + 3\alpha t)}{8Q(L_l + n_l a_l)} - \eta\right] \times \frac{Q\rho}{8\pi(1 + \alpha t)(L_s + n_s a_s)}$$

$$\text{or} \quad \eta \left[1 - \frac{L_l + n_l a_l}{L_s + n_s a_s}\right] = \frac{\pi a_s^4 \Delta p_s (1 + 3\alpha t)}{8Q(L_s + n_s a_s)}$$

$$- \frac{L_l + n_l a_l}{L_s + n_s a_s} \; \frac{\pi a_l^4 \Delta p_l (1 + 3\alpha t)}{8Q(L_l + n_l a_l)}$$

$$\text{or} \quad \eta \left[(L_s + n_s a_s) - (L_l + n_l a_l)\right] = \frac{\pi a_s^4 \Delta p_s (1 + 3\alpha t)}{8Q}$$

$$- \frac{\pi a_l^4 \Delta p_l (1 + 3\alpha t)}{8Q}$$

Again, assuming $n_l a_l = n_s a_s$ because of similar shape and size of the ends.

$$\eta = \frac{\pi a_l^4 (1 + 3\alpha t)}{8Q(L_l - L_s)} \left[\Delta p_l - \left(\frac{a_s}{a_l}\right)^4 \Delta p_s\right]$$

Since the gases are very pressure sensitive, either $\Delta p_l, \Delta p_s$ and Q should be measured at the same state or allowances should be made for this in the working equation.

Thus, the final equation becomes

$$\eta = \frac{\pi a_1^4(1 + 3\alpha t)}{8Q\rho_f\ (L_1 - L_s)} [\Delta p_1 \rho_1 - (\frac{a_s}{a_1})^4\ \Delta p_s \rho_s]$$

where ρ_f, ρ_1, ρ_s are densities of test gas at flow meter, large capillary conditions and small capillary conditions, respectively.

Generalizing and putting $L_1 - L_s = L$

$$\eta = \frac{\pi a_1^4(1 + 3\alpha t)}{8Q\rho (L)}\ [\Delta p_1 \rho_1 - (\frac{a_2}{a_1})^4\ \Delta p_2 \rho_2]$$

where 1 and 2 represent the large and small capillary tubes.

9.2 CORRELATING THE RESULTS USING COLLISION THEORY

Many attempts have been made to correlate viscosity and other transport properties using some form of the kinetic theory of the gases. A few mathematical models using a collision theory approach have been put forward in order to find variation of viscosity with temperature. A detailed description of the mathematical models that have been used, is given in 'Molecular theory of gases and liquids' [25]. The Lennard-Jones (L-J 6-12) model has been proven to be one of the best mathematical models for describing the behaviour of the non-polar gases. The 'L-J 6-12' model assumes that the fluid is composed of rigid spherical molecules, which attract each other according to 6th power of their separation and repel according to 12th power of their separation That is;

$$E(r) = 4\varepsilon\left[\left(\frac{r_o}{r}\right)^{12} - \left(\frac{r_o}{r}\right)^{6}\right] \qquad (9.2.1)$$

where $E(r)$ is the intermolecular potential field

r_o is the low velocity collision diameter

and ε is the maximum energy of attraction

For non-polar gas, $\frac{\varepsilon}{k}$ and r_o can very precisely be obtained from the knowledge of viscosity at two different temperatures. For polar gases; the gases consisting of rigid spherical molecules, each having a point dipole at its centre, Stockmayer suggested the following potential,

$$E(r) = 4\varepsilon\left[\left(\frac{r_o}{r}\right)^{12} - \left(\frac{r_o}{r}\right)^{6} - \frac{\mu^2 \zeta}{r^3}\right] \qquad (9.2.2)$$

and $\zeta = 2 \cos \theta_1 \cos \theta_2 - \sin \theta_1 \sin \theta_2 \cos \phi$ (9.2.3)

where μ is electric dipole moment

ζ is orientation factor

and θ_1, θ_2 are angles of inclination of the two dipole axes to the intermolecular axis

ϕ is azimuthal angle between the two dipole axes,

Transport coefficients have been expressed by Chapman & Cowling in terms of a set of integrals $\Omega^{(1,s)}$. For collision between molecules of type i and j, these integrals are defined as:

$$\Omega_{ij}^{(1,s)} = \sqrt{\frac{2\pi kT}{\bar{m}_{ij}}} \cdot \int_0^\infty \int_0^\infty e^{-\gamma_{ij}^2} (1 + \cos^1 \chi) b \, db \, d\gamma_{ij}$$

where $\bar{m}_{ij}$ is reduced mass of the colliding molecules i and j

γ_{ij} is reduced initial speed of the colliding molecules

χ is the angle by which the molecules are deflected in the centre of gravity coordinate system

b is the impact parameter

k is Boltzmann's constant

T is temperature in °K

To evaluate $\Omega^{(1,s)}$, χ should be known. But χ is a function of g_{ij} (initial relative velocity) and b such that,

$$\chi(b,g) = \pi - 2b \cdot \int_{r_m}^{\infty} \frac{dr/r^2}{\left[1 - \left(\frac{E}{1/2\ \mu g^2}\right) - \frac{b^2}{r^2}\right]^{\frac{1}{2}}} \qquad (9.2.5)$$

Thus $\Omega^{(1,s)}$ has clear relation with the energy of interaction, E(r).

In very simplified form, Chapman & Cowling expressed viscosity as follows,

$$\eta = \frac{5kT}{8\Omega} \qquad (9.2.6)$$

where Ω is Chapman & Cowling collision integral $\Omega^{(1,s)}$ which is a function of temperature, ϵ and r_o.

Hirchfelder, Bird & Spotz [26] calculated functions $W^{(2)}(2)$, where

$$W^{(2)}(2) = \frac{1}{r_o^2}\left(\frac{\bar{m}}{2\pi kT}\right)^{1/2} \qquad (9.2.7)$$

$$\text{or} \quad \Omega = W^{(2)}(2).r_o.\left[\frac{2\pi kT}{\bar{m}}\right]^{1/2} \qquad (9.2.8)$$

Therefore, viscosity in terms of $W^{(2)}(2)$ function,

$$\eta = \frac{5kT}{8\ W^{(2)}(2)r_o^2} \cdot \left[\frac{\bar{m}}{2\pi kT}\right]^{1/2} \qquad (9.2.9)$$

or, writing in more convenient form,

$$\eta = 2.6693 \times 10^{-5}\ \frac{(MT)^{1/2}}{r_o^2 W^{(2)}(2)}\ \text{gms/cm sec.} \qquad (9.2.10)$$

where M is molecular weight in the mass units.

and r_o is low velocity collision diameter in Angstrom units.

Krieger [27] suggested a procedure for calculating the intermolecular force constants of polar gases. The constants of R-13 were computed using the procedure given in his report.

Krieger simplified equation 9.2.2 by putting $\zeta = 2$ which means that the dipoles are in an attractive end on position and maintain their minimum attractive energy value of $-\frac{2\mu^2}{r^3}$ throughout their encounter.

Therefore, $$E(r) = 4\varepsilon[(\frac{r_o}{r})^{12} - (\frac{r_o}{r})^6 - \delta(\frac{r_o}{r})^3] \qquad (9.2.11)$$

and $$\delta = \frac{\mu^2}{2\varepsilon r_o^3} \qquad (9.2.12)$$

where δ, the reduced dipole energy is a pure number and δ is equal to $2\sqrt{t}$, t being the Rowlinson reduced dipole energy.

The parameters ε/k and r_o can be determined from the experimental measurements of viscosity at two temperatures. Since the parameters ε/k, r_o and are inter-related by means of equation 9.2.12(k being Boltzmann's constant), they may be determined simultaneously by cross interpolation over the tabulated values of $W^{(2)}(2)$ for different T^* and δ, T^* being kT/ε.

For R-13, the value of the dipole moment is known. A brief description of the procedure followed is given below:

(i) Viscosity data were correlated by the approximate Sutherland equation; $\eta = A_1\sqrt{T} + B_1$

(ii) Using the viscosity values at two temperatures the values of $W^{(2)}(2)$ were obtained from equation 9.2.10 for a series of arbitrary values of r_o from 2.5 to 6.5 at equal intervals of 0.1 and δ of 0.0, 0.25, 0.5 & 0.75.

(iii) Values of T^* were obtained from these $W^{(2)}(2)$.

(iv) By cross interpolation, the value of r_o was computed for which ε/k was the same at both the temperatures, for all 4 values of δ. Thus 4 values of r_o were obtained.

(v) From these values of r_o and ε/k, values of δ obtained using equation 9.2.12.

(vi) The calculated values of δ were compared with the 4 values of assumed δ to determine the place of the calculated δ among the 4 δ values.

(vii) δ and r_o were simultaneously optimized for minimum standard deviation for the experimental viscosity values.

For R-500, the value of μ was not immediately available, although, μ for CCl_2F_2 and CH_3CHF_2, its constituents, was 0.51 and 2.27 debey respectively. There does not appear to be a mathematical model which could describe a mixture of two polar gases, consequently, for the purpose of correlation R-500 was assumed to be a non-polar gas. Therefore, the value of intermolecular force parameters r_o and ε/k computed in this work do not have any physical significance other than that these were used to correlate the data. The procedure adopted can be summerised as follows:

(i) Viscosity data were correlated by the approximate Sutherland equation.

(ii) Using the viscosity values at two temperatures, the value of $W^{(2)}(2)$ were obtained from equation 9·2·10 for a series of arbitrary values of r_o from 2.5 to 6.5 at an equal interval of 0.1.

(iii) Values of T^* were obtained from these $W^{(2)}(2)$.

(iv) By cross interpolation, the value of r_o was computed, for which ϵ/k was the same at both the temperatures.

(v) The above mentioned procedure was repeated (step 2 to 4) for different pairs of temperatures.

(vi) The value of r_o was selected which was more near to the assumed value.

(vii) r_o was optimized reducing the interval.

9.3 CALIBRATION OF CAPILLARY TUBES

The accurate determination of the diameter of the bore of the tubes and its variation along the length of the capillary tube was very important since,

(i) the diameter is raised to the fourth power in the working equation

and (ii) one of the assumptions in deriving the Hagen-Poiseuille equation is that the diameter of the pipe should be uniform along the entire length of the pipe.

There are various methods of measuring the diameter of the bore and its variation with the length of the tube. They can be summerised as follows;

(1) A small mercury pellet is passed through the capillary tube and its variation in length is observed at different points throughout the length of the tube. This variation in the length of the pellet gives the variation in the diameter of the bore of the tube. The entire tube is then filled with mercury, forming long thread. The mean diameter can be calculated from the weight of the mercury, given its temperature.

(1A) Combining the results of the small pellet and the long thread of mercury graphically, an average diameter can be calculated.

(2) The capillary tube is filled with mercury and the

electric resistance of the mercury thread is then measured and from this, by the formula $r_e = \rho_e L / \pi r^2$

average diameter can be calculated.

Where r_e is electric resistance,

ρ_e is resistivity of the mercury,

L is length of the mercury thread

and r is the mean radius of the tube bore.

(3) Examining the capillary tube with X- rays.

Latto [20] tried all these methods and recommended (1) method as apparently the most suitable. The gravimetric method was, therefore, used for the determination of the mean diameter. Also (1A) was tried and the results were found to be comparable. The procedure is outlined below:

The capillary tubes were thoroughly cleaned so that the mercury pellet would not stick to the wall of the tubes. Concentrated nitric acid was passed through the tubes, which were then rinsed with water. To remove any organic material, the tubes were rinsed with concentrated chromic acid and then with water. Finally tubes were rinsed with methyl alcohol and then acetone. Dry air was passed through the tubes to dry them.

Triple distilled mercury was cleaned thoroughly by agitating it with carbon tetrachloride to remove any grease etc. and then was passed through dilute nitric acid.

A small mercury pellet was introduced into the capillary tube and then small pieces of tygon tubing were connected to each end of the capillary tube. The capillary tube was then clamped in a special 'V' block placed in a container filled with glycerine. This was to avoid any optical refraction effects while observing mercury pellet through the cathetometer. The refractive indices of glass and glycerine are nearly the same (refractive index of glass 1.48 - 1.55, that of glycerine 1.4746)

The length and the position of the mercury pellet were observed using the cathetometer. The motion of the pellet, from one point to another in the entire length of the capillary tube was regulated by squeezing the tygon tubing and thus compressing the air in the capillary tube. At each position, the cathetometer scale readings were noted. Each new position was such that it overlapped the previous one.

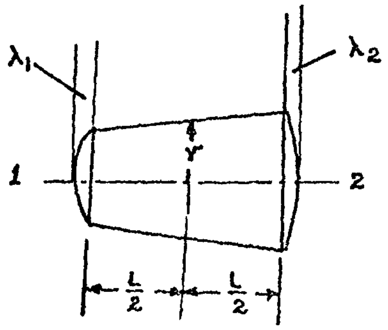
Table (5) shows the equivalent lengths (L_e), the variation in L_e also indicates the variation in the bore size.

The volume of the pellet = $\pi a_1^2 Le_1$

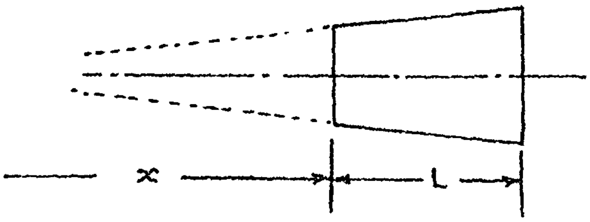
where a_1 and L_{e1} are the radius and equivalent length at a given point. If at another, the equivalent length is

L_{e2}, which is more than L_{e1}, a_2, the radius would be

$$a_2^2 = \frac{Le_1}{Le_2} \quad a_1^2$$



PELLET



FRUSTUM OF CONE

The mercury pellet, with magnified variation in diameter is shown in the figure above.

Volume of the pellet = Volume of the frustum of cone (V_1)
+ volume of the two end caps ($2 V_2$)

$$V = V_1 + V_2 + V_2$$

Volume of the frustum of cone

$$V_1 = \frac{\pi}{3}(L+x)\ (r_2^2) - \frac{\pi}{3}\ (x)\ (r_1^2)$$

since the pellet is very small, we can say,

$$r_2 = r + \delta r$$

$$\&\ r_1 = r - \delta r$$

$$\text{so } V_1 = \frac{\pi}{3}[(L+x)\ (r^2 + (\delta r)^2 + 2r\,\delta r) - x(r^2 + (\delta r)^2 - 2r\,\delta r)]$$

$$= \frac{\pi}{3}[(r^2 + (\delta r)^2 + 2r\,\delta r)L + 4xr\,\delta r]$$

again, $\frac{x}{L+x} = \frac{r-\delta r}{r+\delta r}$, so $x = \frac{r-\delta r}{r+\delta r}\ (L+x)$

or $x\,[1 - \frac{r - \delta r}{r + \delta r}] = \frac{r - \delta r}{r + \delta r}\,L$

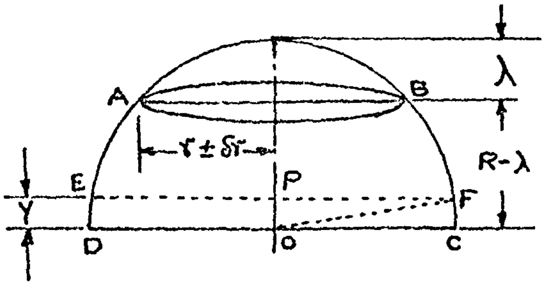
or $x = \frac{r - \delta r}{2\,\delta r}\,L$

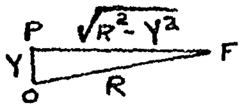
Putting this, we have

$$V_1 = \frac{\pi}{3}\,[(r^2 + (\delta r)^2 + 2r\,\delta r)L + 2rL(r - \delta r)]$$

$$= \frac{\pi}{3}\,[(3r^2 + (\delta r)^2L]$$

$$V_1 = \pi r^2 L\,[1 + \frac{(\delta r)^2}{3r^2}]$$





V_2 = volume of cap = volume of hemisphere - volume of portion ABCD

$$= 2/3\,\pi R^3 - \int_0^{R-\lambda} \pi(R^2 - Y^2)\,dY$$

Considering a very thin disc of thickness dY in the portion ABCD at a distance Y from DC, i.e. EF.

The volume of the disc is $\pi(R^2 - Y^2)\;dY$

Therefore, total volume of ABCD is $\int_0^{R-\lambda} \pi(R^2 - Y^2)\,dY$

so $$V_2 = \frac{2}{3}\,\pi R^3 - \pi[R^2(R - \lambda) - (\frac{R - \lambda}{3})^3] \qquad (I)$$

$$= \frac{2}{3}\,\pi R^3 - \pi[R^3 - \lambda R^2 - \frac{R^3 - \lambda^3 + 3R\lambda^2 - 3R^2\lambda}{3}]$$

$$= \pi[R\lambda^2 - \frac{\lambda^3}{3}] \qquad (9.3.1)$$

But $R^2 = (r \pm \delta r)^2 + (R - \lambda)^2$

or $R = \frac{(r \pm \delta r)^2 + \lambda^2}{2\lambda}$

So volume of both the caps

$$2V_2 = \pi\left[\frac{(r - \delta r)^2 + \lambda_1^2}{2\lambda_1}\lambda_1^2 - \frac{\lambda_1^3}{3} + \frac{(r + \delta r)^2 + \lambda_2^2}{2\lambda_2}\lambda_2^2 - \frac{\lambda_2^3}{3}\right]$$

<u>Total Volume of the Pellet</u>

$$V = V_1 + 2V_2 = \pi r^2 L\left[1 + \frac{(\delta r)^2}{3r^2}\right] + \pi\left[\frac{(r - \delta r)^2 + \lambda_1^2}{2}\lambda_1 + \frac{(r + \delta r)^2 + \lambda_2^2}{2}\lambda_2 - \frac{\lambda_1^3 + \lambda_2^3}{3}\right]$$

$$= \pi r^2 L\left[1 + \frac{(\delta r)^2}{3r^2}\right] + \pi\left[\frac{r^2}{2}(\lambda_1 + \lambda_2) + \frac{(\delta r)^2}{2}(\lambda_1 + \lambda_2) + (\lambda_2 - \lambda_1)(\delta r) + \frac{\lambda_1^3 + \lambda^3}{2} - \frac{\lambda_1^3 + \lambda_2^3}{3}\right]$$

or, $$V = \pi r^2 L\left[1 + \frac{(\delta r)^2}{3r^2}\right] + \pi r^2\left[\frac{\lambda_1 + \lambda_2}{2} + \frac{(\delta r)^2}{2r^2}(\lambda_1 + \lambda_2) + (\lambda_2 - \lambda_1)\frac{\delta r}{r^2} + \frac{\lambda_1^3 + \lambda_2^3}{6r^2}\right]$$

In the above equation, the quantities $(\frac{\delta r}{r})^2$, $(\lambda_2 - \lambda_1) \cdot \frac{\delta r}{r^2}$ and $\frac{\lambda_1^3 + \lambda_2^3}{6r^2}$ are very small since δr and λ_1 and λ_2 are very small.

Therefore, $V = \pi r^2 L + \pi r^2 \frac{\lambda_1 + \lambda_2}{2}$

$$= \pi r^2 \ [L + \frac{\lambda_1 + \lambda_2}{2} \qquad (9.3.2)$$

Comparing this volume of a cylinder,

Le = equivalent length of the pellet $= L + \frac{\lambda_1 + \lambda_2}{2}$

$$= L + \lambda$$

$$\underline{Le = L + \lambda}$$

Fisher's Method as Described in [1A]

Poiseuille eqn. $\eta = \frac{\pi a^4}{8Q} \cdot \frac{dp}{dL}$

or $$\frac{8Q\eta}{\pi} \int_0^L \frac{dL}{a^4} = \int_0^L dp$$

where a is varying along length.

From small pellet $V = \pi Le r^2$ or $\frac{1}{r^2} = \frac{\pi Le}{V}$ (I)

where V is volume of small pellet

r is average radius of pellet.

Also, $V_1 = \int_0^L \pi r^2 dL$ where V_1 = volume of mercury thread filling length L.

$$= V \int_0^L \frac{dL}{Le} . \qquad (II)$$

From I and II

$$\int_0^L \frac{dL}{r^4} = \int_0^L \frac{\pi^2 Le^2}{\left(\frac{V_1}{\int_0^L \frac{dL}{Le}}\right)^2}$$

or $$\int_0^L \frac{dL}{r^4} = \frac{\pi^2}{V_1^2} \cdot [\int_0^L \frac{dL}{Le}]^2 \cdot \int_0^L Le^2 \ dL .$$

Table 5

Small capillary tube

POSITION IN CM.	L in CM.	Le in CM.	$\frac{1}{Le}$ in CM^{-1}	Le^2 in CM^2	TEMP. in °C
79.479	2.837	2.845	0.3515	8.094	
76.636	2.834	2.842	0.3518	8.077	
73.759	2.832	2.841	0.3520	8.071	28.0
70.896	2.830	2.839	0.3522	8.060	
68.039	2.825	2.834	0.3528	8.031	
65.195	2.830	2.839	0.3522	8.060	
62.349	2.824	2.835	0.3527	8.037	
59.473	2.822	2.835	0.3527	8.037	28.5
56.629	2.822	2.833	0.3530	8.026	
53.791	2.826	2.835	0.3527	8.037	
50.940	2.829	2.837	0.3525	8.048	
48.103	2.824	2.834	0.3528	8.031	
45.267	2.824	2.833	0.3530	8.026	
42.417	2.826	2.833	0.3530	8.026	
39.579	2.821	2.830	0.3533	8.009	28.5
36.730	2.819	2.829	0.3535	8.003	
33.878	2.822	2.831	0.3532	8.014	
31.033	2.826	2.835	0.3527	8.037	
28.182	2.816	2.825	0.3540	7.981	

Table 6

Large capillary tube

POSITION in cm.	L in cm	Le in cm	$\frac{1}{Le}$ in cm	Le^2 in cm^2	TEMP. in °C
73.274	2.691	2.701	0.3702	7.295	
70.534	2.689	2.700	0.3704	7.290	
67.843	2.693	2.704	0.3698	7.311	
65.119	2.687	2.696	0.3709	7.268	
62.423	2.671	2.681	0.3730	7.188	28.5
59.717	2.670	2.680	0.3731	7.182	
56.991	2.687	2.695	0.3710	7.263	
54.286	2.682	2.689	0.3719	7.231	
51.583	2.682	2.689	0.3719	7.231	
48.886	2.679	2.687	0.3721	7.220	
46.184	2.680	2.689	0.3719	7.231	29.5
43.477	2.674	2.684	0.3726	7.204	
40.782	2.675	2.685	0.3724	7.209	
38.078	2.674	2.683	0.3727	7.198	
35.385	2.672	2.679	0.3733	7.177	
32.695	2.678	2.687	0.3721	7.220	30.0
29.992	2.671	2.680	0.3731	7.182	
27.317	2.670	2.679	0.3733	7.177	
24.610	2.669	2.678	0.3734	7.172	

Average diameter of the capillary tubes;

Large tube:

(i) By gravimetric method;

Weight of mercury thread = 1.4940 gms at temp. 28.5°C

Length of the mercury thread = 56.23 cms.

Average bore diameter = 0.5001165542 mm.

(ii) By Fisher's method, using graphical integration;

Average bore diameter = 0.500539 mm.

Small tube

(i) By gravimetric method;

Weight of mercury thread = 1.5325 gms at temp.28.5°C

Length of mercury thread = 57.235 cms.

Average bore diameter = 0.502052776 mm.

(ii) By Fisher's method, using graphical integration;

Average bore diameter = 0.5022685 mm.

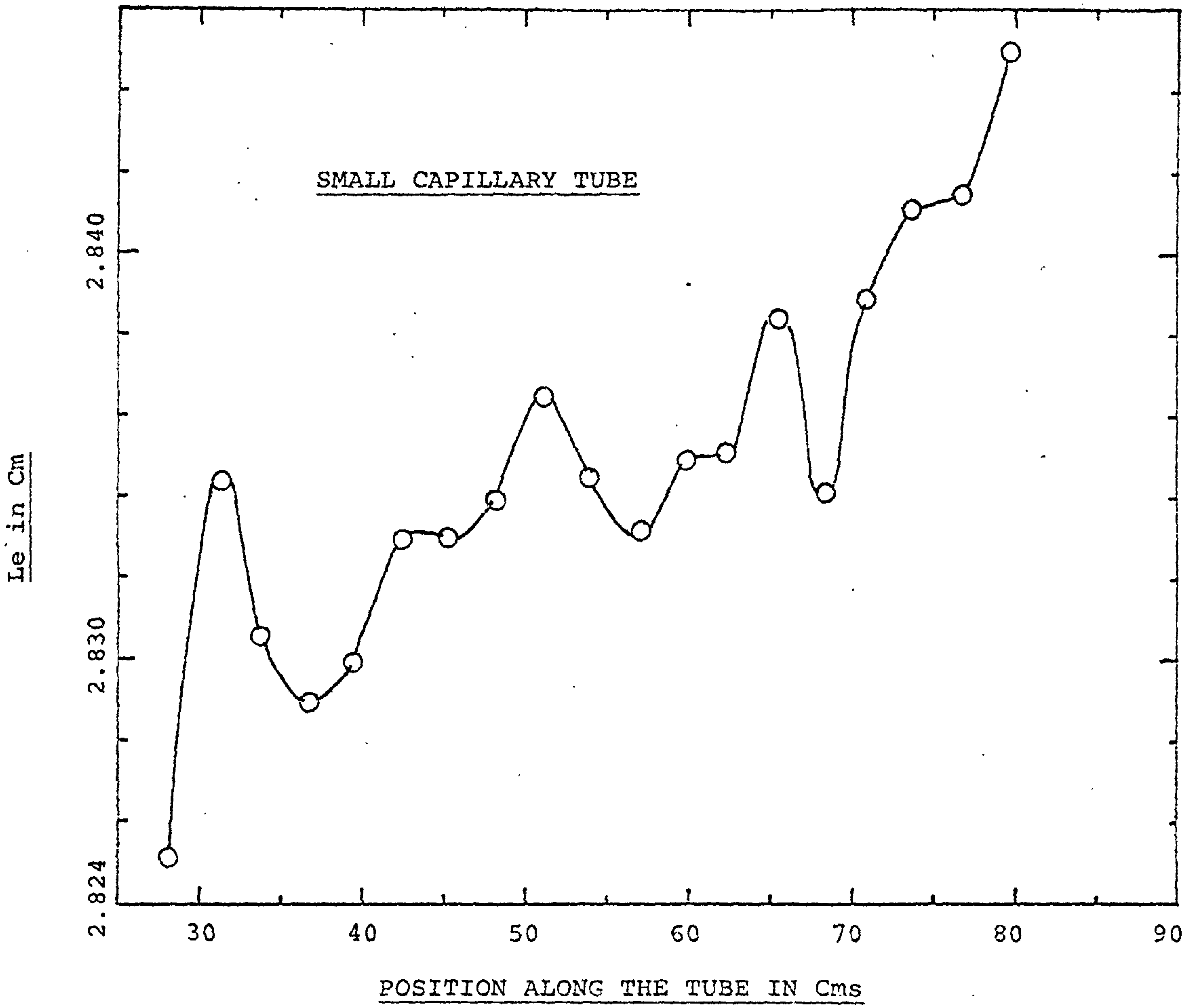


FIG. 29 CAPILLARY TUBE CALIBRATION.

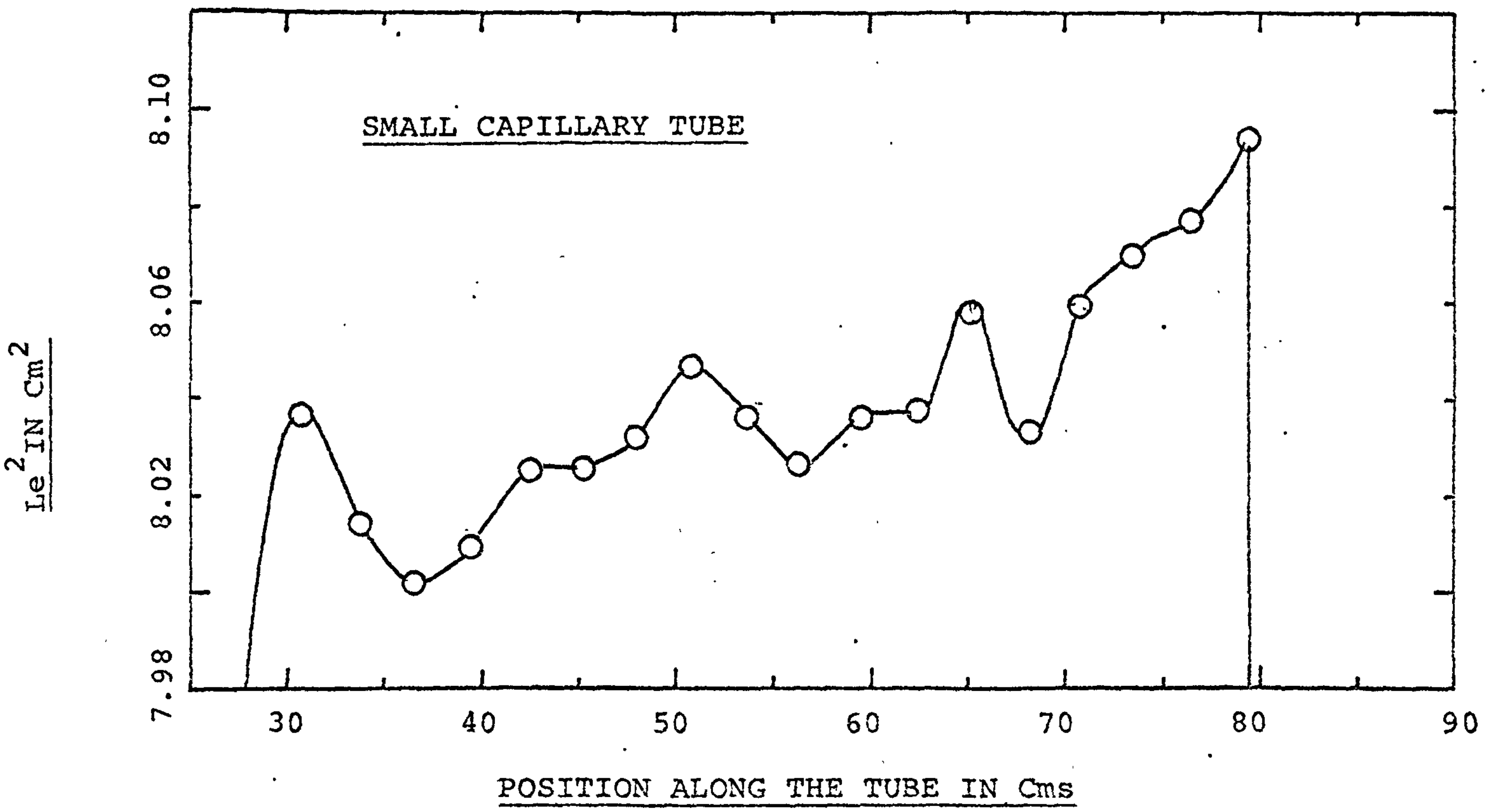
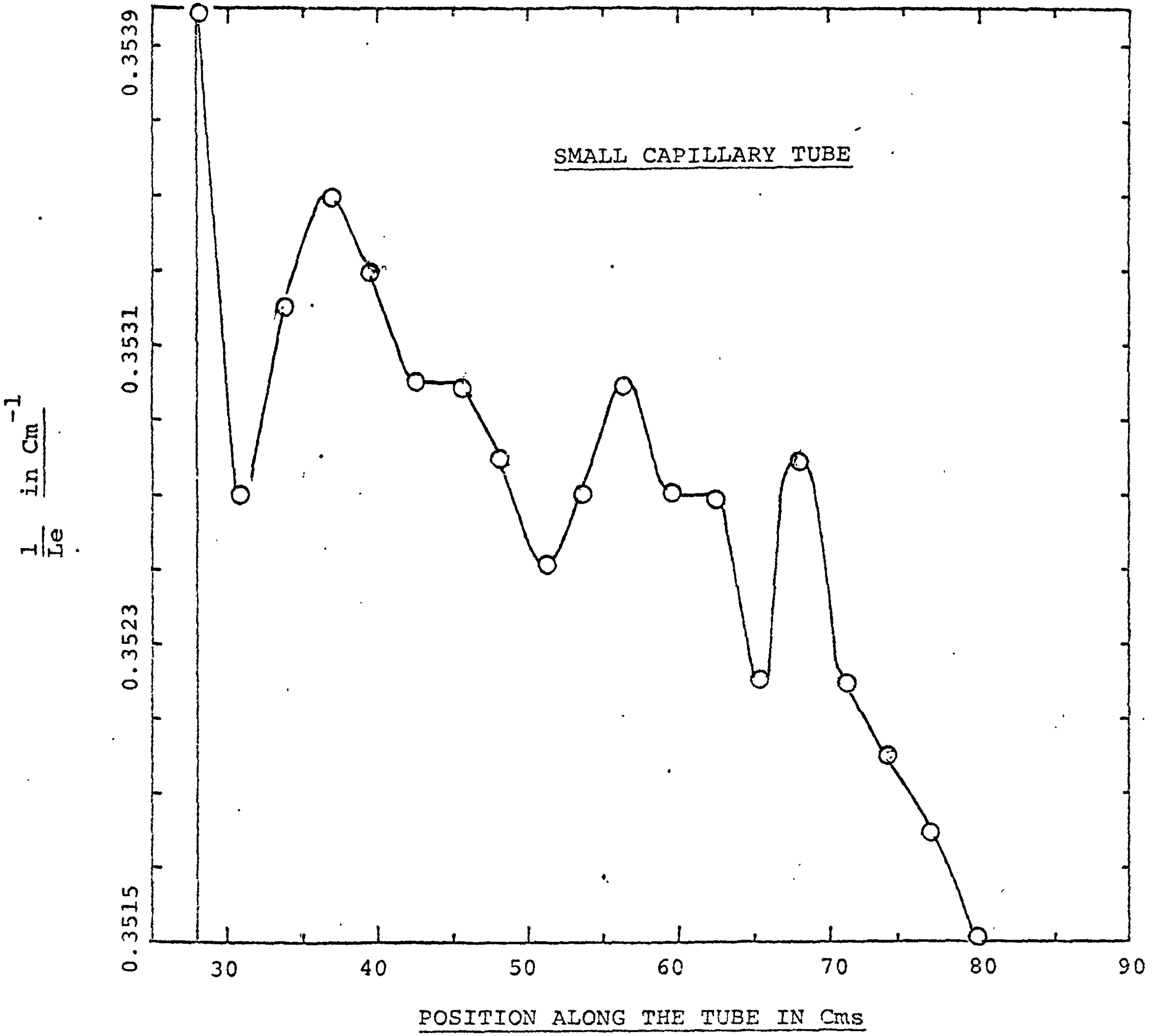
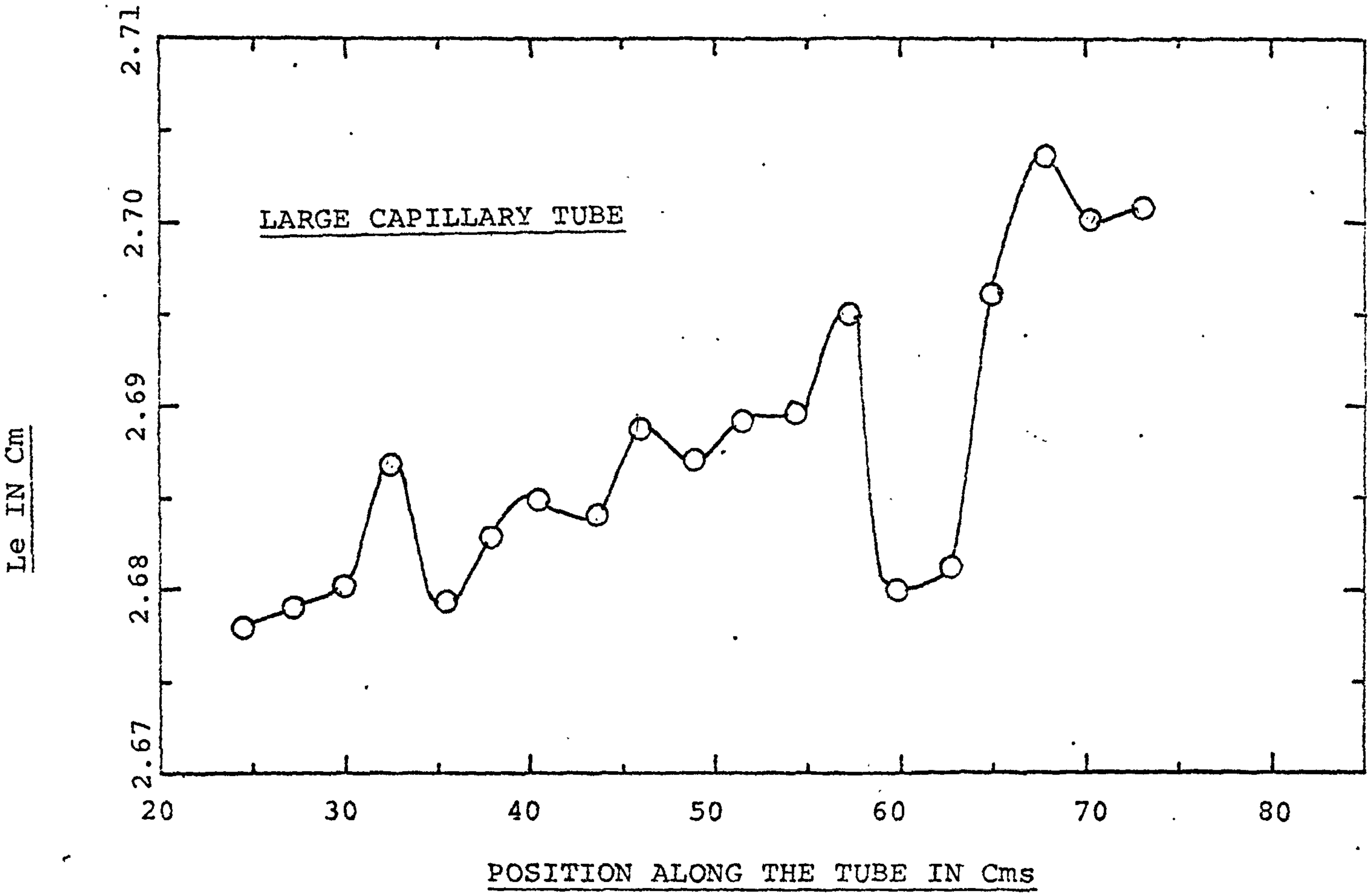


FIG. 30 CAPILLARY TUBE CALIBRATION.

FIG. 31

CAPILLARY TUBE CALIBRATION.





CAPILLARY TUBE CALIBRATION.

FIG. 32

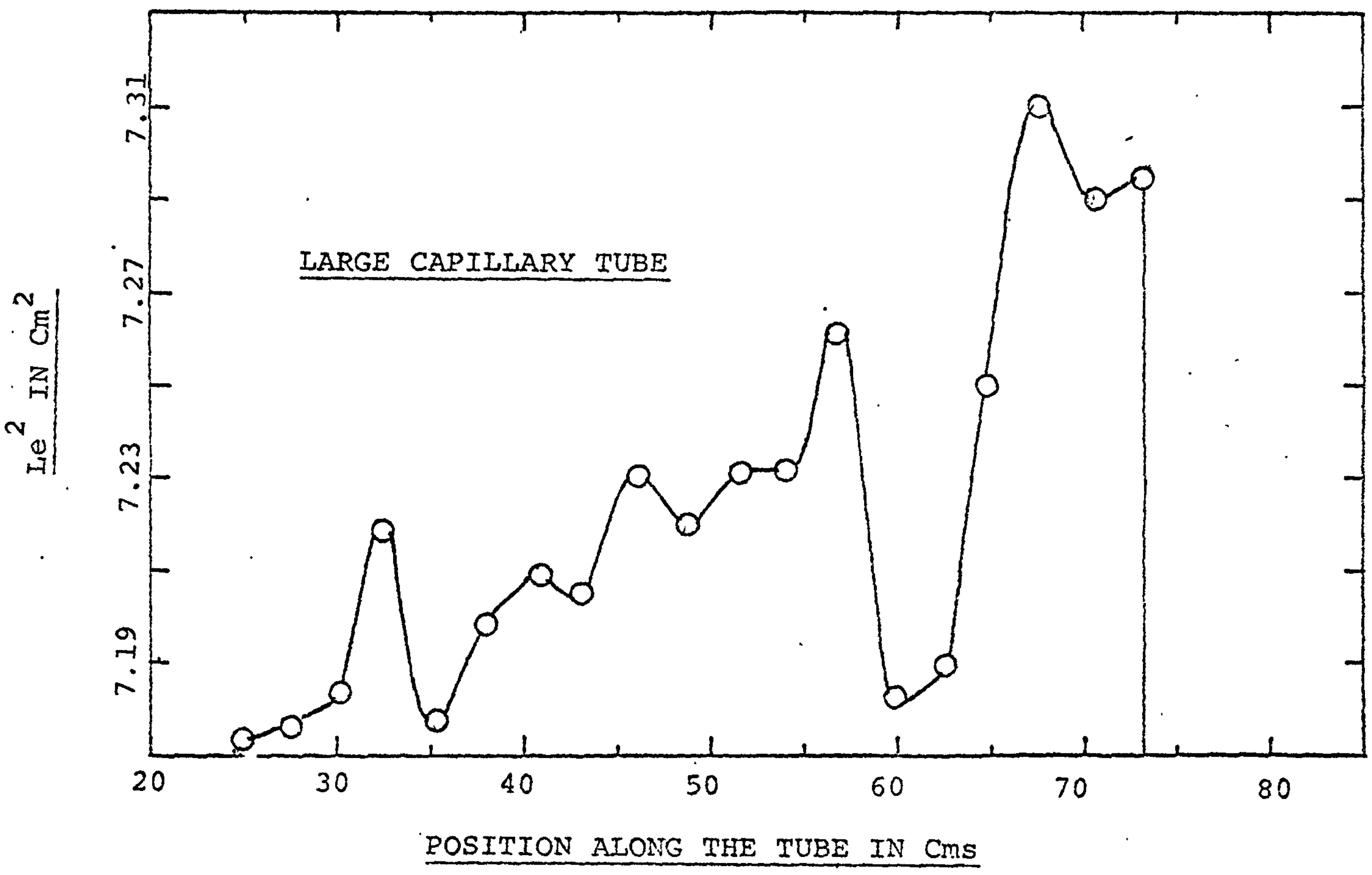
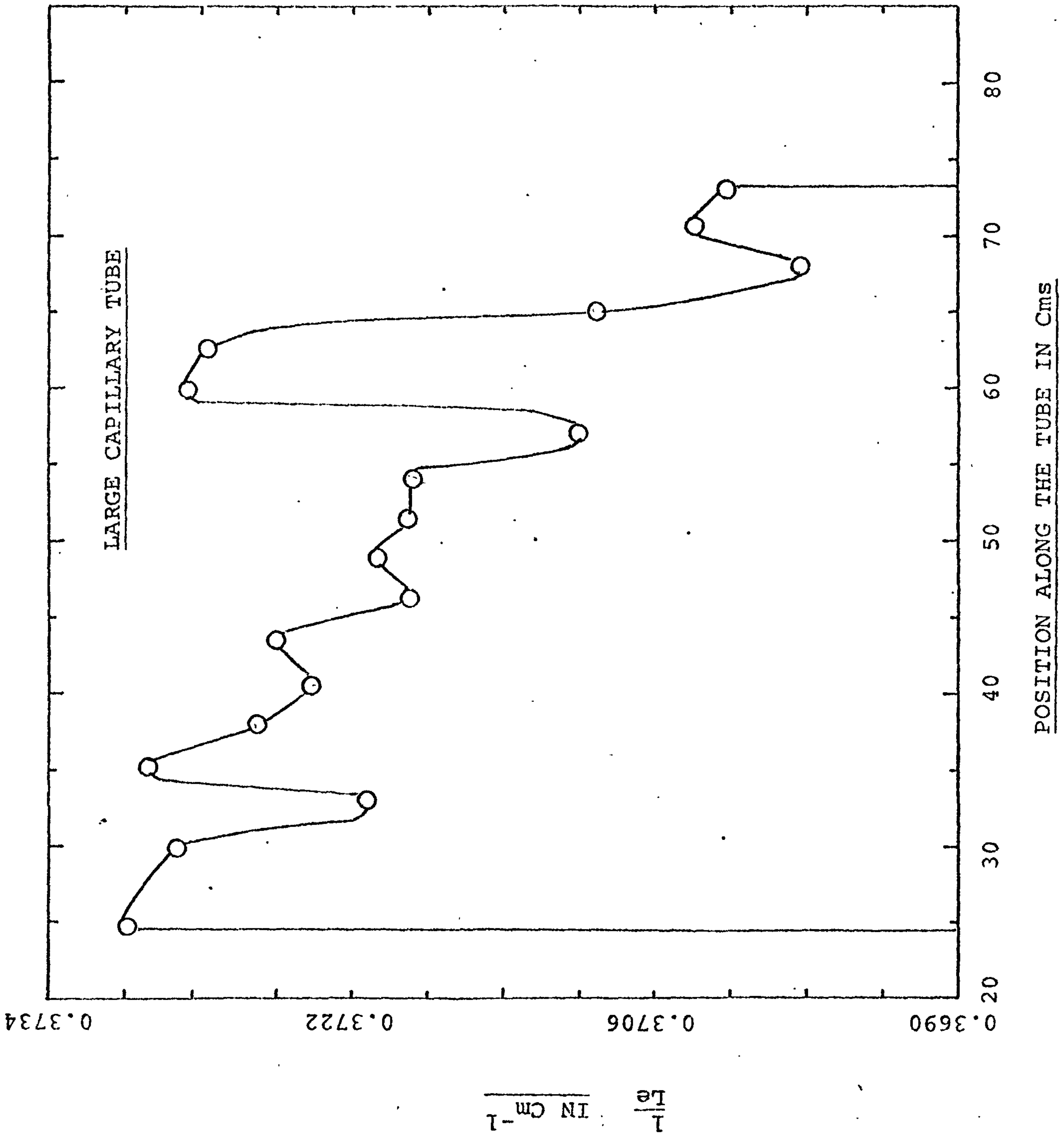


Fig. 33 CAPILLARY TUBE CALIBRATION.



CALIBRATION OF CAPILLARY TUBE

FIG. 34

9.4 THERMOCOUPLE CALIBRATION

To measure the temperature of the test gas as accurate as possible, three copper-constantan thermocouples were calibrated over a temperature range of approximately -99°C to 89°C. The calibration was done against a copper-constantan ' Thermo Electric ' thermocouple, No. 705, which itself had been calibrated by Thermo electric (Canada) ltd, against two standard NBS (National Bureau Of Standards) thermocouples, namely, APH SSP-992/1 and APH-1421. The deviation of the thermocouple 705 from the standard tables, when calibrated against the said thermocouples, is shown below:

Calibration of standard thermocouple No. 705 against NBS thermocouples APH SSP-992/1 & APH-1421

Reference junction temperature = 32°F

THERMOCOUPLE	UNCERTAINITY	TEMPERATURE	DEVIATION FROM TABLES IN mv
APH SSP-992/1	0.1°C	-112°F	+0.014
" "	"	-100°F	+0.021
" "	"	- 50°F	+0.009
APH-1421	0.06°C	+ 50°F	+0.009
" "	"	+100°F	+0.012
" "	"	+150°F	+0.010

General procedure to calibrate thermocouples is to compare emf. of standard thermocouple with the thermocouples at some physical phase change temperatures, viz; temperature of ice and water mixture, temperature of boiling water, temperature of liquid and solid mercury mixture, sublimation temperature of solid carbon dioxide etc. Since a special type of heat exchanger was readily available (see fig.35), instead of physical phase change temperatures, the calibration was performed at some arbitrarily chosen temperatures.

The heat exchanger consisted of;

(i) a central cylindrical copper block capable of holding four glass test tubes.

(ii) a copper cylindrical jacket which surrounds the cylindrical copper block.

(iii) An outside aluminium cylindrical jacket and insulation to avoid excessive heat losses.

One standard and three other thermocouples were inserted into glass test tubes containing oil to improve heat conduction. For the 'hot region', constant temperature hot water was ciculated from the same ' twin pump - constant temperature bath' as mentioned in the chapter 5, Description of the apparatus. When equilibrium was achieved and there was no drift of the galvanometer pointer, the reading was taken. Similarily, for the 'cold region', controlled and regulated liquid nitrogen was circulated. Four sets of readings were taken in each region. The measurements are shown in the table 7 . Fig.36 shows the calibration curve.

TABLE 7

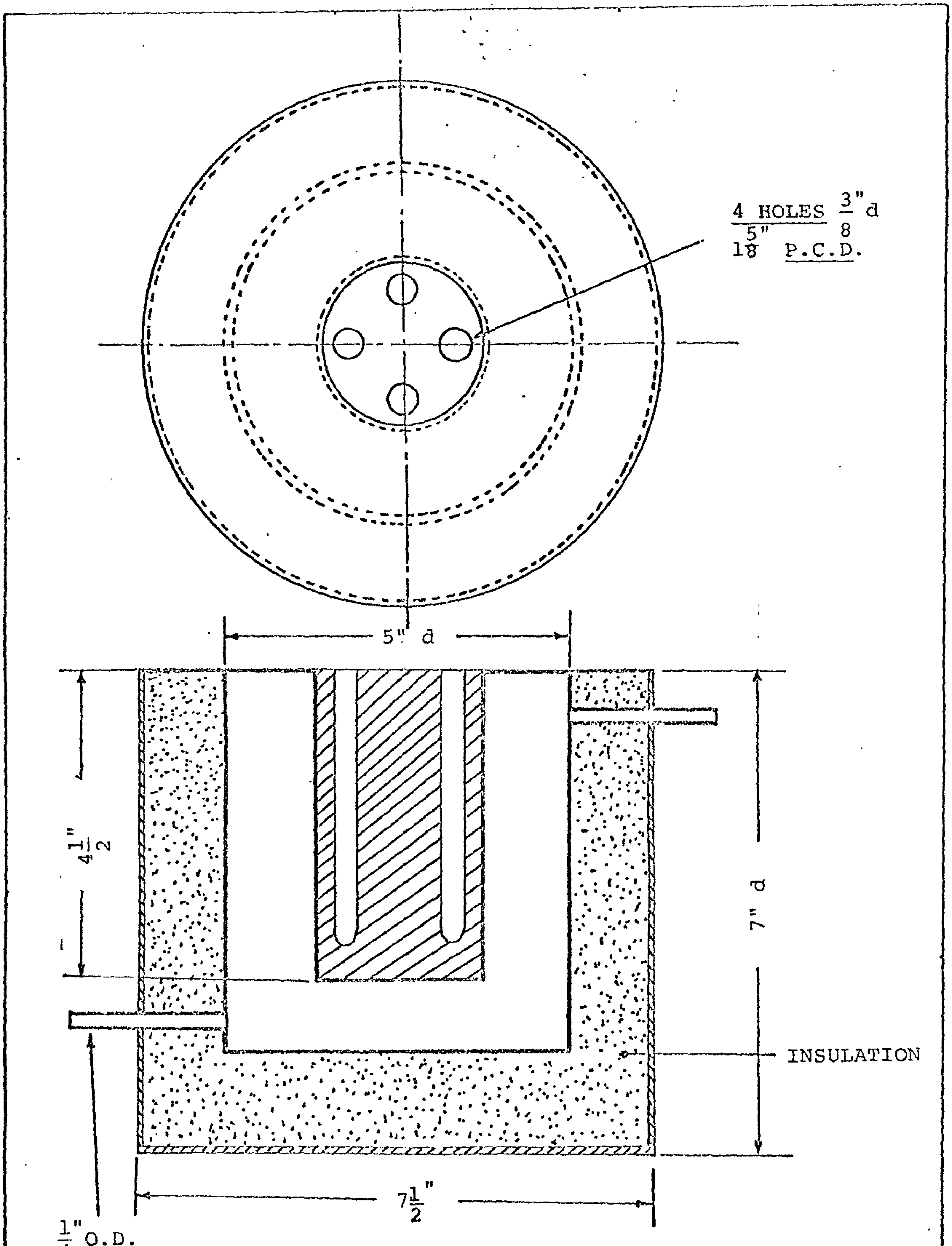
Thermocouple calibration

'Hot region'

Thermocouple Number	Emf. in mv 1st set	2nd set	3rd set	4th set
standard	1.2838	1.7684	2.6297	3.7942
1	1.2638	1.7407	2.5882	3.7395
2	1.2671	1.7436	2.5941	3.7430
3	1.2660	1.3443	2.6013	3.7470

'Cold region'

Thermocouple Number	Emf. in mv 1st set	2nd set	3rd set	4th set
standard	+0.0213	-1.0917	-2.0730	-3.3260
1	-0.0028	-1.0870	-2.0663	-3.3071
2	-0.0014	-1.0870	-2.0670	-3.3050
3	-0.0037	-1.1067	-2.0680	-3.3270



SCALE HALF FULL SIZE

THERMOCOUPLE CALIBRATION HEAT EXCHANGER

FIG. 35

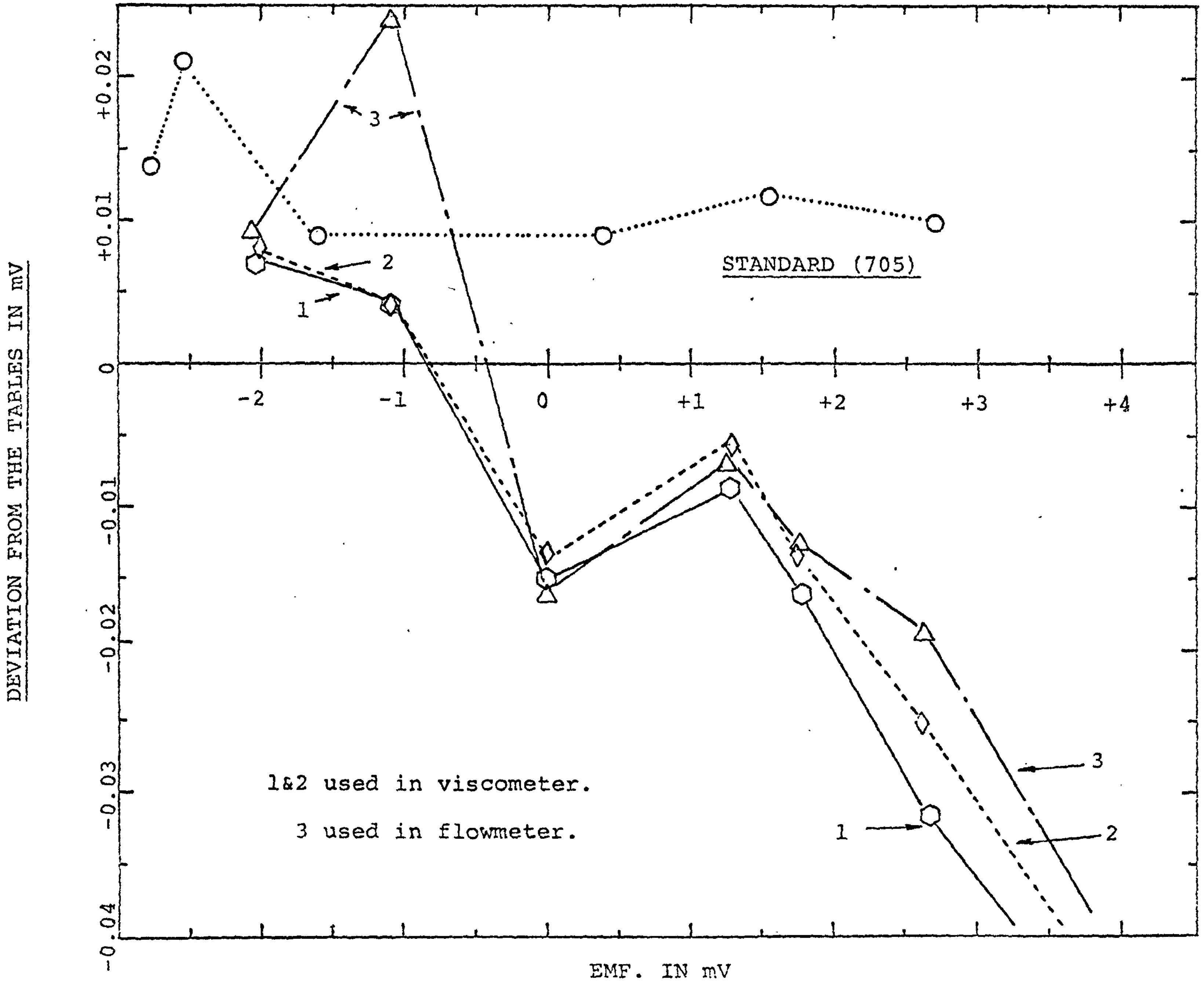


FIG. 36 THERMOCOUPLE CALIBRATION.

9.5 CALIBRATION OF THE FLOW METER

The flow meter was removed from the water jacket, cleaned thoroughly and held upside down. (see fig.10) The lower open end of the burette was closed with a rubber plug. A thin tygon tubing was inserted into the burette and was held near the 100 mark of the graduations. Water was introduced first upto the 0 mark and then accurately measured volumes of water were introduced using a 10 ml. pipette. After each 10 ml. of water, the water level in the burette was noted. Great care was taken to avoid wetting of the burette wall while introducing water.

It was observed that 90 cc of water showed as 90.3 cc by the flow meter burette. The above procedure was repeated three times and the results were found with in 0.05 cc.

The pipette was checked for its accuracy. 10 cc of water was weighed on a 'Mettler' balance capable of reading to 0.0005 gms. It was found to be 9.9865 gms. at 22°C.This was repeated twice and the weight agreed within 0.003 gms. When this weight of water was compared with density given in the 'Handbook of Chemistry and Physics,' 45th edition [28], it was found to be 0.095% more.

Taking these factors into account, the volume of flow meter burette was found 0.23% more; i.e. 100 cc of actual volume was shown as 100.23 cc.

9.6 PRESSURE MEASUREMENT WITH THE PRESSURE TRANSDUCER

For the pressure measurement, a transducer (Scan Co. # PDCR-4 ± 0.2 psi - 5 vac S/N 482, from M/s Scanivalve inc.) was tried. For convenience, consistency and economy, a special 3 way valve was designed and developed as shown in the fig. 37. This valve was capable of connecting the three pressure taps individually to the transducer element.

The valve consisted mainly of a connector having a radial slot which connected one of the pressure leads to the transducer. The mating surfaces were precision ground and honed so that they were perfectly flat, thus avoiding any leakage. The valve was operated manually. The teflon packing underneath the connector was for tight but smooth operation. The transducer seat was machined as shown in fig. 37 , such that it fitted snugly into the housing. A teflon ring seal and a nut were used to avoid any possible leakage.

An electric connector, which could pass through the nut, was used to connect transducer to the oscillator amplifier, the output from which was fed to the chart recorder. It was observed that the recorder was unable to detect any small variation in the pressure (of the order of 1 mm of water). Actually this was the main criteria for selecting this set up, that this arrangement would indicate the pertubation in the system and thus indicate the system stability.

When this was not found satisfactory, A Potentiometer-Galvanometer arrangement (used for the temperature measurement) was tried instead of the chart recorder. But unfortunately, this arrangement proved to be too sensitive and a continuous drift was observed.

A digital integrating voltmeter could have responded better, but due to unavailablity of this at that time, it was decided to use manometer-cathetometer combination for pressure measurement. Though inconvenient, it was one of the most suitable and sensitive device available.

Red gauge oil was used as the manometer fluid. The density of red gauge oil was measured using a density bottle and a 'Metler' balance. The bottle was thoroughly cleaned and dried and filled carefully with the oil from the manometer. The density found at 27°C was .82545 gms/cc, however, a value of .826 gms/cc, recommended by the supplier, was used throughout the calculations, neglecting the variation with temperature.

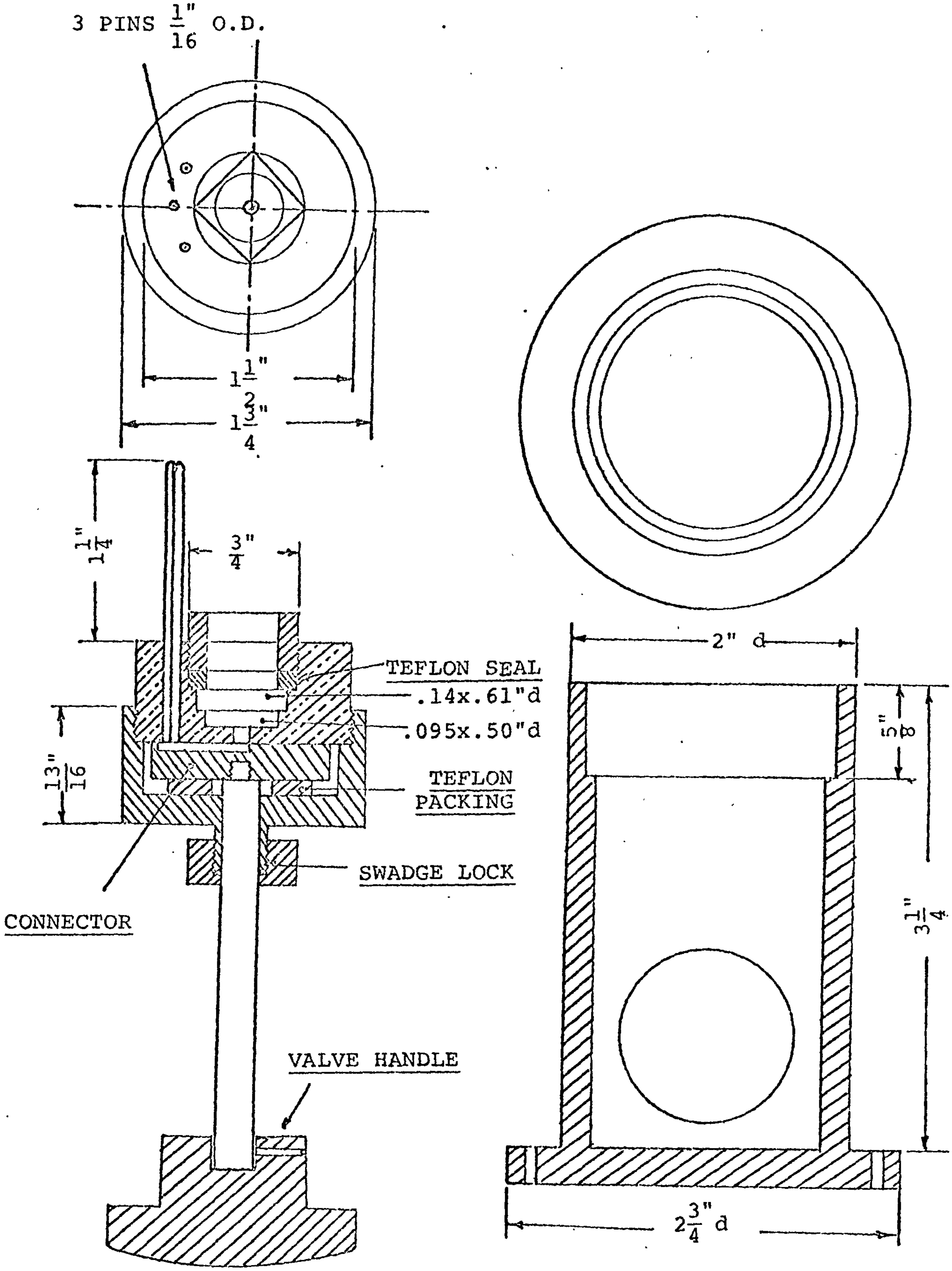


FIG. 37 TRANSDUCER VALVE SCALE FULL SIZE

9.7 ERROR ESTIMATION

The working equation for Series Capillary Viscometer is,

$$\eta = \frac{\pi\ a_1^4\ (1+3\alpha t)}{8Q\rho_f(L_1-L_2)}\ [\Delta p_1\rho_1\ -\ (\frac{a_2}{a_1})^4\ \ \Delta p_2\rho_2] \qquad (3.7)$$

Neglecting the effect due to the expansion and the contraction of the glass capillary tube material which is very small, we get

$$\eta = \frac{\pi\ a_1^4}{8Q\rho_f(L_1-L_2)}\ [\Delta p_1\rho_1\ -\ (\frac{a_2}{a_1})^4\ \ \Delta p_2\rho_2] \qquad (3.7a)$$

The 'most probable' error can be written as

$$d\eta = \sqrt{\Sigma(\frac{\delta\eta}{\delta x}\cdot dx)^2} \qquad (9.7.1)$$

By differentiating equation 3.7a and expressing it in the form of 9.7.1, we get

$$d\eta = [(\frac{4\pi a_1^3\Delta p_1\rho_1}{8Q\rho_f(L_1-L_2)}\cdot da_1)^2 + (\frac{4\pi a_2^3\Delta p_2\rho_2}{8Q\rho_f(L_1-L_2)}\cdot da_2)^2 +$$

$$(\frac{\pi a_1^4\rho_1}{8Q\rho_f(L_1-L_2)}\cdot d(\Delta p_1))^2 + (\frac{\pi a_2^4\rho_2}{8Q\rho_f(L_1-L_2)}\cdot d(\Delta p_2))^2 +$$

$$(\frac{\pi\ a_1^4\Delta p_1}{8Q\rho_f(L_1-L_2)}\cdot d\rho_1)^2 + (\frac{\pi\ a_2^4\Delta p_2}{8Q\rho_f(L_1-L_2)}\cdot d\rho_2)^2 +$$

$$(\frac{\pi\ a_1^4[\Delta p_1\rho_1\ -\ (\frac{a_2}{a_1})^4\ \ \Delta p_2\rho_2]}{8\rho_f(L_1-L_2)}\cdot\frac{dQ}{Q^2})^2 +$$

$$\left(\frac{\pi\, a_1^4\,[\Delta p_1\rho_1 - (\frac{a_2}{a_1})^4\,\Delta p_2\rho_2]}{8Q(L_1-L_2)} \cdot \frac{d\rho_f}{\rho_f^2}\right)^2 + \left(\frac{\pi\, a_1^4\,[\Delta p_1\rho_1 - (\frac{a_2}{a_1})^4\,\Delta p_2\rho_2]}{8Q\rho_f} \cdot \frac{dL}{(L_1-L_2)^2}\right)^2\Bigg]^{1/2} \qquad 9.7.2$$

The viscosity of a fluid using a single capillary viscometer can be given as, (neglecting thermal expansion).

$$\eta = \frac{\pi a^4\Delta p}{8Q(L+na)} - \frac{m\rho Q}{8\pi(L+na)} \qquad (3.4)$$

Neglecting the end conditions and modifying equation 3.4 for gases, it becomes,

$$\eta = \frac{\pi a^4\Delta p\rho}{8Q\rho_f L} \qquad (3.4a)$$

Dividing equation 9.7.2 by equation 3.4a, we get

$$\frac{d\eta}{\eta} = \Bigg[\left(\frac{L_1}{L_1-L_2} \times 4\,\frac{da_1}{a_1}\right)^2 + \left(\frac{L_2}{L_1-L_2} \times 4\,\frac{da_2}{a_2}\right)^2 + \left(\frac{L_1}{L_1-L_2}\;\frac{d(\Delta p_1)}{\Delta p_1}\right)^2$$

$$+ \left(\frac{L_2}{L_1-L_2} \cdot \frac{d\,(\Delta p_2)}{\Delta p_2}\right)^2 + \left(\frac{L_1}{L_1-L_2} \cdot \frac{d\rho_1}{\rho_1}\right)^2 + \left(\frac{L_2}{L_1-L_2} \cdot \frac{d\rho_2}{\rho_2}\right)^2$$

$$+ \left(\frac{dQ}{Q}\right)^2 + \left(\frac{d\rho_f}{\rho_f}\right)^2 + \left(\frac{dL}{L_1-L_2}\right)^2\Bigg]^{1/2} \qquad (9.7.3)$$

Although equation 9.7.3 is approximate, it is thought to be accurate enough for the assessment of the accuracy of the experimental results.

Evaluation of the terms on right hand side of equation 9.7.3 is as follows:

Capillary tube radius term

The bore size of the capillary tube was calculated by gravimetric method using the equation,

$$a^2 = \frac{W_m}{\pi Le \rho_m}$$

where W_m is the mass of the mercury pellet
Le is the length of the thread
ρ_m is the density of mercury
and a is the average radius of the bore of the capillary tube.

Expressing this in the form of equation 9.7.1,we get

$$2a\,da = \left[\left(\frac{dW_m}{\pi Le \rho_m}\right)^2 + \left(\frac{W_m dLe}{\pi Le^2 \rho_m}\right)^2 + \left(\frac{W_m d\rho_m}{\pi Le \rho_m^2}\right)^2 \right]^{1/2}$$

or

$$2\,\frac{da}{a} = \left[\left(\frac{dW_m}{W_m}\right)^2 + \left(\frac{dLe}{Le}\right)^2 + \left(\frac{d\rho_m}{\rho_m}\right)^2 \right]^{1/2}$$

(i) The mass of the long mercury pellet was measured using a 'Mettler' balance which was capable of measuring 160 gms with an accuracy of ± 0.001 gm. The mass of mercury thread was of the order of 1.5 gms. Therefore,

$$\frac{dW_m}{W_m} = \frac{0.001}{1.5} \times 100 = \pm\ 0.067\%.$$

(ii) The length of the mercury thread Le was measured using a cathetometer. The smallest division on the Cathetometer Scale was 0.001 cm, therefore, the error in measuring a length of 60 cms, allowing for the fact that two readings of scale were required , would be

$$\frac{dLe}{Le} = 2 \times \frac{0.001}{60} \times 100 = 0.0033\% .$$

(iii) The density of mercury was obtained from the 'Handbook of Chemistry and Physics' [28]. The values were reported to be better than 0.1%. Since no variation in temeprature was observed while measuring the mass of mercury thread,the error introduced due to the density of mercury would be negligible.

Therefore,

$$2\frac{da}{a} = [(0.067)^2 + (0.0033)^2]^{1/2}$$

or

$$\frac{da}{a} = \pm 0.034\%.$$

It should be mentioned here that $\frac{da}{a}$ calculated here is the uncertainty in the average diameter of the capillary tube. The variation of the diameter along the tubes, which were 0.15% and 0.24% for short and long tube respectively, have already been considered in calculating average diameter using Fisher's method (see appendix 9.7.3). Though the diameter calculated using Fisher's method and gravimetric method compare very satisfactory, the value obtained from gravimetric method was thought to be more reliable and therefore, was used in calculation for the viscosity.

Pressure term

$$\text{Since } \Delta p = \rho_o g\ h$$

where Δp is the pressure drop

ρ_o is the density of manometer oil

h is the height of oil in the manometer

and g is acceleration due to gravity.

Therefore, we can write

$$\frac{d(\Delta p)}{\Delta p} = [(\frac{d\rho_o}{\rho_o})^2 + (\frac{dg}{g})^2 + (\frac{dh}{h})^2]^{1/2}$$

(i) The accuracy of the density for the manometer oil can be assumed to be better than ± 0.25%.

(ii) The value of acceleration due to gravity used, was obtained from the Department of Energy, Mines and Resources. The accuracy was quoted to be ± 0.0005%.

(iii) The oil level in the manometer was read using the cathetometer capable of reading ± 0.001 cm. But assuming that the maniscus could only be located to ± 0.005 cm, the maximum error in the measurement of height 'h',for the smallest height of 10 cms allowing for the fact that two readings were required, would be

$$\frac{dh}{h} = 2 \times \frac{0.005}{10} \times 100 = \pm\ 0.1\%$$

Thus,

$$\frac{d(\Delta p)}{\Delta p} = [(0.25)^2 + (0.0005)^2 + (0.1)^2]^{\frac{1}{2}}$$

$$\text{or} \quad \frac{d(\Delta p)}{\Delta p} = \pm\ 0.27\%.$$

Density of the test gas term

The density of the test gas was taken from the 'Properties of Commonly Used Refrigerants' and was reported to be better than ± 0.1%.

Volumetric flow rate term

Volumetric flow rates were measured by timing the flow of a given volume of the test gas

$$\text{That is,} \quad Q = \frac{v}{t}$$

where Q is the volume flow rate

v is the known volume between two fixed points

t is the time taken by the soap film to travel the distance.

Therefore, by the similar method as before, we get

$$\frac{dQ}{Q} = [(\frac{dv}{v})^2 + (\frac{dt}{t})^2]^{1/2}.$$

The accuracy of the burette graduation of the flowmeter was better than ± 0.1%. Therefore, between the fixed points, $\frac{dv}{v} = \pm\ 0.1\%$ or better.

An 'Excelsior Park' stop watch was used for the timing and was assessed to have an accuracy of better than ± 0.01%.

Thus,

$$\frac{dQ}{Q} = [(0.01)^2 + (0.1)^2]^{\frac{1}{2}}$$

$$\text{or} \quad \frac{dQ}{Q} \simeq \pm\ 0.1\%.$$

Length term

The maximum error in measuring the length of the capillary tubes using cathetometer would be for the short tube of length 41.369 cms. The error would, therefore be

$$\frac{dL}{L} \simeq 2 \times \frac{0.001}{41} \times 100 \simeq \pm\ 0.005\%.$$

Summarizing, the errors are as follows:

$$\frac{da}{a} = \pm 0.034\%$$

$$\frac{d(\Delta p)}{p} = \pm 0.27\%$$

$$\frac{d\ \rho}{\rho} = \pm 0.1\%$$

$$\frac{dQ}{Q} = \pm 0.1\%$$

$$\frac{dL}{L} = \pm 0.005\%$$

Substituting these values in equation 9.7.3 we get,

$$\frac{d\eta}{\eta} = [(\frac{56.23}{14.861} \times 4 \times 0.034)^2 + (\frac{41.369}{14.861} \times 4 \times 0.034)^2 +$$

$$(\frac{56.23}{14.861} \times 0.27)^2 + (\frac{41.369}{14.861} \quad 0.27)^2 + (\frac{56.23}{14.861} \times 0.1)^2$$

$$+ (\frac{41.369}{14.861} \times 0.1)^2 + (0.1)^2 + (0.1)^2$$

$$+ (\frac{41.369}{14.861} \times 0.005)^2]^{1/2}$$

$$= \pm 1.515\%$$

Allowance for Temperature Gradient

The above approach is true only if there was no temperature gradient along the length of the capillary tube, however in the actual case a maximum of 0.5°C temperature gradient was observed during the test runs. Assuming a simple correlation for the variation of viscosity with temperature, $\eta = A_1 \sqrt{T} + B_1$; where A_1 and B_1 are constants and T is the temperature in absolute centigrade scale. Taking logarithms of the equation and then differentiating, we get,

$$\frac{d\eta}{\eta} = \frac{1}{2} \times \frac{\delta T}{T}$$

Therefore, for a minimum temperature of 200°K

$$\frac{\delta\eta}{\eta} = \frac{1}{2} \times \frac{0.5}{200} \times 100 = \pm 0.125\%$$

This results in an overall maximum assessed error of ±1.64%.

Thus , the maximum possible error in the experimental values of viscosity is ±1.64%.

9.8 SPECIMEN CALCULATION

A specimen calculation of the viscosity of R-500 at 24.8°C, chosen arbitrarily, is illustrated here.

Test gas: R-500; Date: 7.5.69; Apprx. Temp.: 25°C

Length of large capillary tube, $L_1 = 56.23$ cms.

Length of small capillary tube, $L_2 = 41.369$ cms.

Therefore, $L = L_1 - L_2 = 14.861$ cms.

Diameter of large capillary tube, $d_1 = 0.5001165542$ mm

Diameter of small capillary tube, $d_2 = 0.5020527780$ mm

Commencing with the flow equation,

$$\eta = \frac{\pi d_1^4 (1+3\alpha t)}{128 \bar{Q} L} \cdot [\Delta P_1 \rho_1 - (\frac{d_2}{d_1})^4 \Delta P_2 \rho_2]$$

The value of the coefficient of thermal expansion, α for glass is 3.3×10^{-6} per °C at 20°C. Therefore, correction for the thermal expansion of the capillary tubes would be $3 \times 3.3 \times 10^{-6} \times (24.84-20.0)$

Thermocouple emf. measured for different thermocouples was

viscometer, No. 1 = 0.9779 mv

viscometer, No. 2 = 0.9719 mv

flow meter, No. 3 = 1.0794 mv

manometer, No. 4 = 1.0855 mv

After applying the individual corrections, the corrected emf. were

No. 1 = 0.9879 mv

No. 2 = 0.9789 mv

No. 3 = 1.0884 mv

No. 4 = 1.0945 mv

From the tables of thermocouple properties [29], thus,

Average viscometer temperature = 24.84°C = 76.71 °F

Flowmeter temperature = 27.41°C = 81.34°F

Manometer temperature = 27.57°C

And the temperature difference between the two ends of the viscometer = 0.22°C.

P_a, the atmospheric pressure was 75.4 cms of mercury and the manometer readings were,

Exit limb = 93.12 cms of oil

Intermediate limb = 71.22 cms of oil

Entrance limb = 41.62 cms of oil

Therefore, $\Delta P_1 = 71.22 - 41.62 = 29.60$ cms of oil

and $\Delta P_2 = 93.12 - 71.22 = 21.60$ cms of oil.

The mean pressure in the upstream capillary tube was,

$$P_{1m} = P_a + z.\ (\Delta P_2 + 1/2\ \Delta P_1\ ) = 77.637 \text{ cms of Hg}$$

and the mean pressure in the downstream capillary tube,

$$P_{2m} = P_a + z.\ 1/2\ \Delta P_2 = 76.067 \text{ cms of Hg}$$

$$\text{where } z = \frac{\text{Density of manometric oil}}{\text{Density of mercury}}$$

The specific volumes of R-500 at the temperature 76.71°F and the pressure of 77.637 cms of Hg, and 76.067 cms of Hg; the mean conditions in the capillary tubes were, as obtained after interpolation for both temperature and pressure, from [22]

$v_1 = 3.7967$ cu ft / lb

$v_2 = 3.8751$ cu ft / lb

Similarly, the specific volume of R-500 at the flow meter conditions, i.e. atmospheric pressure of 75.4 cms of Hg and temperature of 81.34°F was 3.9449 cu ft / lb

The volumetric flow rate was 0.569 cc/sec. After applying the correction, the corrected volumetric flow rate became 1.0023 x 0.569 cc/sec.

The value of 'g', the acceleration due to gravity, as obtained from the department of Energy, Mines and Resources was 980.3941 cm / $\sec^2$.

Substituting all these in the flow equation,

$$\eta = \frac{\pi \times (0.5001165542)^4 \times 10^{-4} \times (1 + 9.9 \times 10^{-6} \times 4.84)}{128 \times 1.0023 \times 0.569 \times 14.861}$$

$$\times 980.3941 \times \left[\frac{29.6}{3.7967} - \left(\frac{0.5001165542}{0.5020527780}\right)^4 \times \frac{21.6}{3.8751}\right]$$

This gives, $\eta = 1.188237 \times 10^{-4}$ gm / cm sec.

Therefore, the viscosity of R-500 at atmospheric pressure and temperature of 24.84°C is 1.188237×10^{-4} Poise.

10 REFERENCES

[1] Hovorka,F. and F.E. Geiger, ' Thermodynamic properties of Trifluorotrichloroethane and Difluorotetrachloroethane' J. Am. Chem. Soc., 55, 47-60, 1933.

[2] Awbery, J.H. and E. Griffiths, 'The viscosity of some liquid refrigerants', Phy. Soc. Proc., 48, 372, 1936.

[3] Benning, A.F. and W.H. Markwood, 'The viscosity of Freon refrigerants', April, 1939, Refrigerating Engineering.

[4] Graham,D.P., 'Properties of PF-1 (Octafluorocyclobutane)', JLR-69-8, No.1, Serial No. 18958, DuPont de Nemours.

[5] Coughlin,J. 'The vapour viscosity of refrigerants', Ph.D. thesis in mechanical Engineering, Purdue Univ., 1953.

[6] Makita,T. 'The viscosity of Freon under pressure', Rev. Phy. Chem. of Japan, 24, No.2, 1954.

[7] Wellman,E.J. 'Viscosity determination of several fluorinated hydrocarbon vapours with a rolling ball viscometer', Ph.D. thesis in Mech. Eng., Purdue Univ., 1955

[8] Kinser,R.E. 'Viscosity of several fluorinated hydrocarbon compounds in vapour phase', MSc thesis in Mech. Eng.,Purdue Univ.,1956.

[9] Lillios,N. 'The viscosity determination of several liquid refrigerants at atmospheric pressure', MSc thesis in Mech. Eng. Purdue Univ., 1957

[10] McCullum, R.G. 'High temperature viscosity measurement of fluorinated hydrocarbon compounds in vapour phase', MSc.

thesis in Mech. Eng.,Purdue Univ., 1958.

[11] Kamien,C.Z. 'Effect of pressure and temperature on the viscosity of refrigerants in vapour phase', ASHRAE Trans., 65, 663-674, 1959.

[12] Tsui,C.Y., 'Viscosity measurement of several fluorinated hydrocarbon vapours at elevated pressure and temperatures', MSc thesis in Mech. Eng., Purdue Univ., 1959.

[13] Willbers,O.J., 'Viscosity measurement of severalfluorinated hydrocarbon vapours at low temperature',MSc thesis in Mech.Eng. Purdue Univ., 1961.

[14] Riley,V.J., 'The viscosity of liquid Freon-11 and Freon-12 at temperature to -110°C', MSc thesis in Mech.Eng., Purdue Univ., 1963.

[15] Eisele,E.H., 'The determination of dynamic viscosity of several Freon compounds at temperatures in the range 200 to -200°F', MSc thesis in Mech. Eng., Purdue Univ., 1965.

[16] Al-Saloum,A.J.M., 'Determination of the absolute viscosity of some refrigerants',MSc thesis in Mech. Eng., McMaster Univ., 1968.

[17] Latto,B. and Al-Saloum,A.J.M., 'Extensive viscosity correlations for halogenated hydrocarbons (Freons) in liquid and vapour phases', Under publication.

[18] Barr,G. 'A monograph of viscometery', Oxford University Press, 1931

[19] Knudsen 'Annalytik Physik (4), 28, 75, 1909.

[20] Latto,B., 'The viscosity of steam at atmospheric pressure', Tech. report No. 16, Univ. of Glasgow, U.K., 1965.

[21]'Thermophysical properties research centre data book', Vol. 2, Purdue Univ. 1966

[22] 'Properties of commenly used refrigerants', Air conditioning & refrigeration institute, 1957.

[23] 'Freon-500 Refrigerant', A DuPont Publication No. RT-41.

[24] 'Selected values of electric dipole moments for molecules in the gas phase' NBS Publication.

[25] Hirschfelder, Curtiss, Bird, 'Molecular theory of gases and liquids', John Wiley & Sons, 1964.

[26] Hirschfelder, Bird & Spotz, The transport properties of gases and gaseous mixtures', J. Chem. Phy., 16, 968, 1948

[27] Krieger, A Glasgow univ. report

[28] Hand Book of C hemistry and Physics, 45th Edition, Chemical rubber publication., 1964-65.

[29] Conversion tables for thermocouples, Leeds & Northrup Co.

[30] Hillsenrath,J., 'Table of thermodynamic & transport properties of Air --------------- Nitrogen etc.' Pergamon Press, 1960.