

A STUDY OF AN INDENTOR VISCOMETER

by 8529

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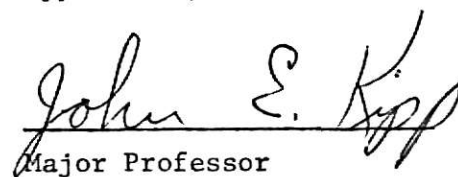
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CHAPTER I

INTRODUCTION

Viscosity is the property of fluid that characterizes its flow behaviour. Flow is the continuous deformation caused by any type of external force. The viscosity of a fluid is a measure of its frictional resistance to the sliding motion of one layer of fluid over another. This resistance forms the basis for the measurement of viscosity of a fluid.

The measurement of the coefficient of viscosity of a fluid is important in both fundamental science and industrial applications. As far as the fundamental science concern, viscosity determines whether the flow of a fluid is a laminar or turbulent. Viscosity also is of great importance in the study of mixing and heat transfer characteristics of a fluid, viscosity has also proved a valuable tool for physical chemistry studies since the viscosity coefficient was found to be influenced by the size, shape and arrangement of the molecules. For industrial applications, viscosity is important in determining the resistance to be overcome when fluids are transported in pipelines, bearing analysis, etc., and determines the nature of the flow in such processes as spraying, extrusion and surface coating. In addition, viscosity measurements are used to check the consistency of products.

Among the various kinds of viscometers that are used to measure the coefficient of viscosity of different fluids in different ranges, there are a very limited number of viscometers which can measure the coefficient of viscosity of fluids in the range from 10^5 to 10^{10} poises.

The indentor viscometer which is described herein may be suitable for the measurements of viscosities in the range of viscosities mentioned above.

Review of Literature:

The review presented here was concerned only with the indentor viscometer, how it was originated, and what progress has been made to prove its validity for absolute measurements of coefficient of viscosity of highly viscous fluids.

In a work done by Goudy and Kirmser(4), in 1967, a theoretical study of the indentation of a viscous fluid by a right circular cylinder was investigated. The assumptions made to solve this problem were that the fluid indented was a Newtonian fluid in a semi-infinite space and that the viscosity of the fluid was so high that all body and inertia forces could be neglected when compared to the viscous forces. Their study using a transform technique to solve the non-linear partial differential equation, gave the initial velocity, pressure and stress at every point in the flow as well as the force required to indent a fluid for a given velocity of indentor. They developed the following expression:

$$F = 8 \mu V a$$

where

F is the force of indentation,

μ is the coefficient of viscosity of the fluid,

V is the velocity of indentation,

a is the radius of indentor.

Using this expression as a basis for constructing a new type of viscometer which could measure the viscosity coefficients of highly viscous fluids, a viscometer, called the indentor viscometer, was designed and fabricated at Kansas State University.

Using this viscometer Sachdeva (8), in 1969, did a study aiming to verify the above expression experimentally. His method of attack was to assume the coefficient of viscosity of a fluid depends on the type of fluid, the radius of indenter, the velocity of indentation, and the force of indentation. He chose three different standard viscosity fluids and assumed a linear model to relate the dependent and independent variables in the form:

$$\mu = c_1 L_1 + c_2 L_2 + c_3 L_3 + c_4 a + c_5 V + c_6 F + c_7 a^2 + c_8 V^2 + c_9 F^2 \\ c_{10} aV + c_{11} aF + c_{12} VF$$

where:

- L_1, L_2, L_3 are parameters that represent the type of fluid used, in other words, if fluid number 2 is used then $L_1 = L_3 = 0$ while $L_2 = 1$
- a is the radius of indenter,
- V is the velocity of indentation,
- F is the force of indentation,
- c_1, \dots, c_{12} are coefficients to be determined experimentally.

Then, using a statistical method based on the criteria of least squares to determine the best approximations to the coefficients c_1, c_2, \dots, c_{12} out of the data obtained from the 81 experiments carried out using three different fluids; three different sizes of indenter; three different indenter weights; and three different depths of indentation, he concluded that the viscosity coefficient depends on the type of the fluid, the radius of indenter and the square of the radius of indenter. This conclusion does not agree with the theoretical conclusion obtained by Goudy and Kirmser (4) in 1967. This disagreement is believed to be due to two factors. First, the fluids used by Sachdeva (8) were not viscous enough to neglect the inertia and body forces in comparison with viscous forces an assumption which was made by Goudy and Kirmser (4).

The viscosity coefficient of the most viscous fluid used by Sachdeva was 590 poises. Secondly, the container in which the fluids were put was not large enough to consider the fluid semi-infinite. This directly contradicts the assumption made by Goudy and Kirmser in their study and causes the boundaries of the fluid to affect the flow characteristics.

Purpose of the Study:

In order to step ahead towards verification of the validity of the indenter viscometer as a measuring device for measuring the coefficients of viscosity of highly viscous fluids a much more viscous fluid than those used by Sachdeva was chosen and a wider container was used. The program of the study presented here can be divided into two parts:

Part I

The well-known falling co-axial cylinder viscometer was used to :

1. check wheather the asphalt was a Newtonian or Non-Newtonian fluid by measuring the rate of shear strain at different shear stresses, keeping the temperature of asphalt constant,
2. measure the viscosity of asphalt at different temperatures.

Part II

1. A dimensional analysis of the problem of indenter viscometer.
2. Deducing the functional relation which relates the dependent and independent dimensionless groups obtained in the previous step through experimentations using the indenter viscometer as a measuring device and making use of the data obtained in part I.

CHAPTER II

PART I

FALLING CO-AXIAL CYLINDER VISCOMETER

Historical Review:

The falling co-axial cylinder viscometer was first described by Segel in 1903, adopted by Pochettino in 1914 for measurements of viscosity of pitch, then in 1932 Mack used it to measure the viscosity of asphaltic bitumens. Up to this time the dimensions of the viscometer were inconveniently large and so has the disadvantage of needing a long time to cool down to the test temperature, it is very difficult to control its temperature and it needs a large quantity of the material whose coefficient of viscosity is to be measured. A study was done by Traxler and Schweyer in 1936 to investigate the effect of dimensions of viscometer on the resulting values of coefficient of viscosity. They concluded that the thickness of the specimen should be less than one half its length.

Puch (7), in 1944, constructed a viscometer which confirmed with the restrictions put by Traxler and Schweyer. The chief merits of the instrument were:

- a. easy to fill and bring down to test temperature in half an hour.
- b. it requires only 12 c.c. of the material whose coefficient of viscosity is to be measured.
- c. It can be used to measure the absolute coefficient of viscosity in

the range from 5×10^4 to 10^{10} poises.

In 1965 the American Society of Testing and Materials, ASTM (1), published a proposed method of test of viscosity of asphalt cements using a falling plunger viscometer. According to this method the following viscometer was constructed at Kansas State University.

Description of Viscometer:

The viscometer as shown in Plate I consists mainly of two co-axial cylinders 4 and 5 between which the specimen 3 is poured. The inner cylinder 4 had the shape of a cup with a cover plate 2. The outer cylinder 5 was a cylindrical ring. Both cylinders were mounted on a moulding base 8 which assures the concentricity of both cylinders. The viscometer was mounted on a stand 9. The moulding base was mounted on a stand ring 9A. while the outer cylinder 5 was fixed inside the stand ring 9B with three tightened screws.

A three leg dial indicator stand 6 was mounted on the outer cylinder 5 such that the plunger of the dial indicator rests on the cover plate 2. When the inner cylinder 4 moved down under the effect of its weight and the weights which may be in it, the dial indicator showed the distance moved by the inner cylinder. The time taken by the inner cylinder to move down one tenth of an inch was measured by an electric stop watch to within 0.1 second.

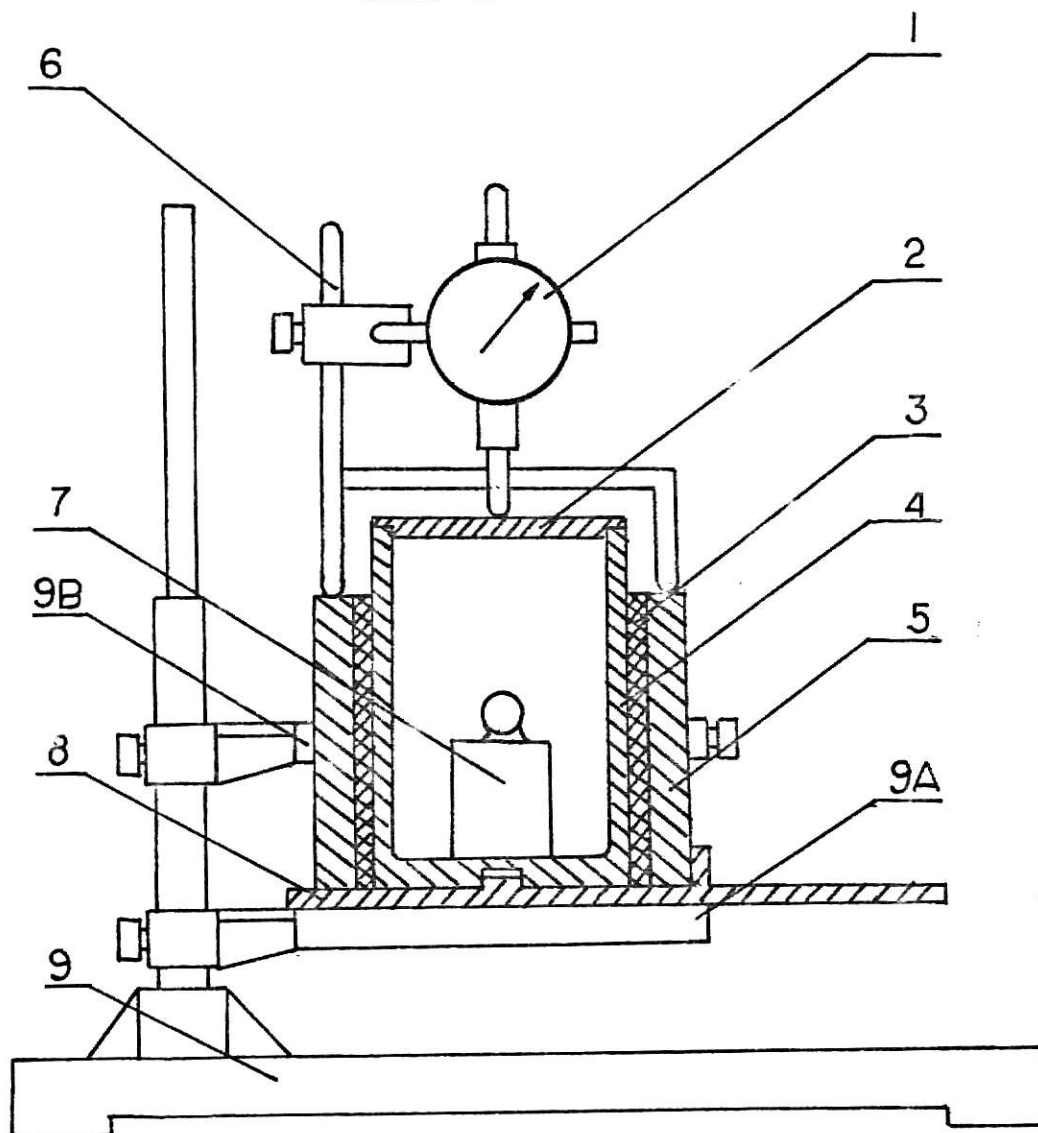
The whole assembly was immersed in a controlled temperature water bath up to a level slightly above the edge of the outer cylinder. When the specimen was left in the water bath to control its temperature, the stand ring 9A was fixed to the stand shaft by a tightened screw, while the screw of the stand ring 9B was loose.

EXPLANATION OF PLATE I

THE FALLING CO-AXIAL CYLINDER VISCOMETER

1. Dial indicator.
2. Cover plate.
3. Fluid specimen.
4. Inner cylinder.
5. Outer cylinder.
6. Dial indicator stand.
7. Additional weight.
8. Moulding base.
9. Stand:
 - 9A. stand ring.
 - 9B. stand ring.

PLATE I



Specifications of Viscometer:

Outer radius of inner cylinder	= a = 1.604 in.
Inner radius of outer cylinder	= b = 1.726 in.
Height of outer cylinder	= L = 3.025 in.
Weight of inner cylinder with cover plate	= 765.5 gm

Theory of Viscometer:

In this section two theoretical approaches will be discussed after which a comparison between them applied to the viscometer used will be presented.

First Theoretical Approach:

As shown in Fig.(2.1), due to symmetry about the cylinder axis, the velocity v of a layer of test fluid at a distance r from the axis of symmetry was a function of r alone.

Since the outer cylinder was fixed while the inner cylinder was moving down with a velocity V , the velocity gradient at distance r from the axis of symmetry can be expressed as $-\frac{dv}{dr}$.

Assuming the fluid to be Newtonian, and applying Newton's law of viscosity Appendix A, hence

$$\tau = -\mu \frac{dv}{dr} \quad (2.1)$$

where:

- τ is the shear stress at distance r from the axis of symmetry,
- μ is the absolute coefficient of viscosity of test fluid.

$$\text{The shearing area at distance } r = 2 \pi r L \quad (2.2)$$

Substituting (2.2) in equation (2.1) the viscous shearing force F can be expressed as :

$$F = -2 \pi r L \mu \frac{dv}{dr} \quad (2.3)$$

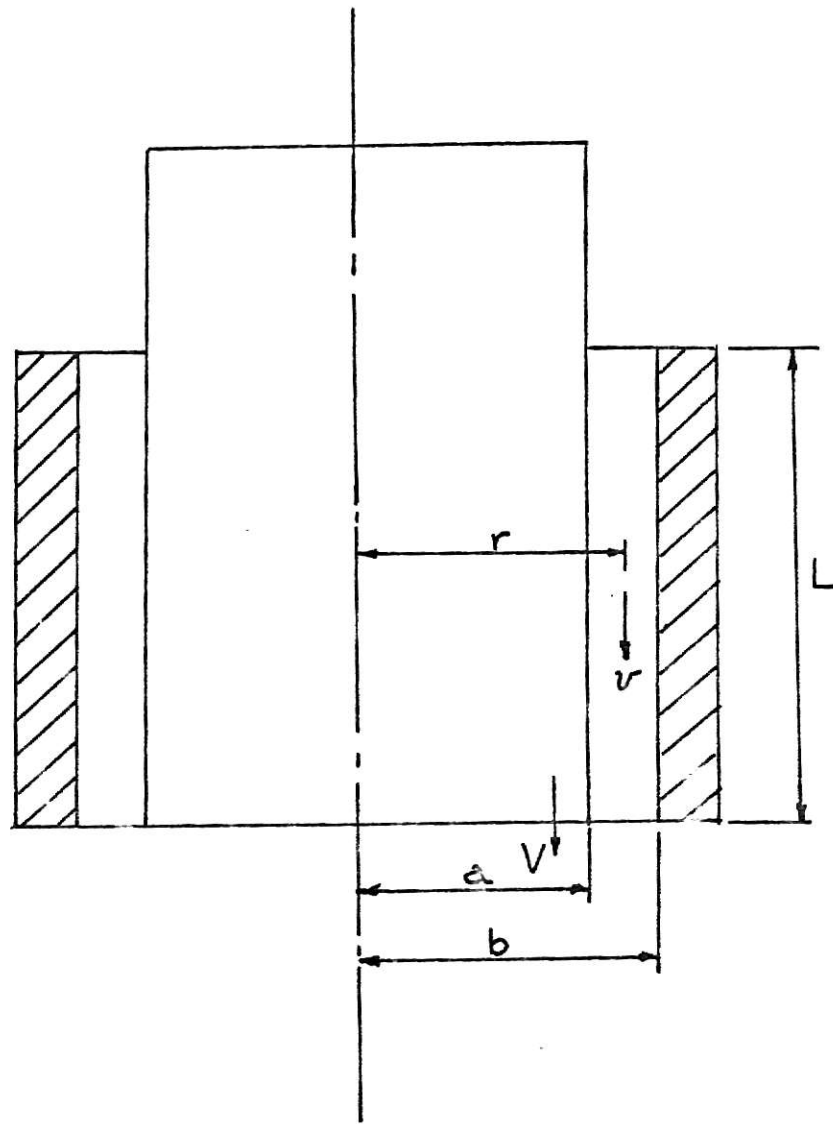


Fig.(2.1) Falling co-axial cylinder viscometer.

Since a state of steady motion exists, the viscous shearing force may be balanced by the total load inside a cylinder of radius r , hence,

$$F = W + \rho g \pi (r^2 - a^2)L - \rho_1 g \pi r^2 \quad (2.4)$$

where:

W is the weight of inner cylinder and its contents,

ρ is the density of test fluid,

a is the radius of inner cylinder,

ρ_1 is the density of bath fluid.

The first two terms in the right hand side of equation (2.4) represent the gravity force of both the inner cylinder and the test fluid, while the third term represents the buoyancy force.

Substituting equation (2.4) in equation (2.3) and rearranging.

$$-dv = \frac{W - \rho g \pi a^2 L}{2 \pi L \mu} \frac{dr}{r} + \frac{g(\rho - \rho_1)}{2 \mu} r dr \quad (2.5)$$

Integrating equation (2.5) and applying the boundary conditions:

$$\text{at } r = b \quad v = 0$$

$$\text{at } r = a \quad v = V$$

equation (2.5) becomes,

$$V = \frac{W - \rho g \pi a^2 L}{2 \pi L \mu} \ln \frac{b}{a} + \frac{g(\rho - \rho_1)(b^2 - a^2)}{4 \mu} \quad (2.6)$$

Assuming,

$$W' = W - \rho_1 g \pi a^2 L \quad (2.7)$$

where:

W' is the effective weight of the inner cylinder.

and substituting equation (2.7) in (2.6) equation (2.6) becomes,

$$V = \frac{1}{\mu} \left[\frac{W' - g \pi a^2 L (\rho - \rho_1)}{2 \pi L} \ln \frac{b}{a} + \frac{g(\rho - \rho_1)(b^2 - a^2)}{4} \right] \quad (2.8)$$

Hence,

$$\mu = \frac{1}{V} \left[\frac{W'}{2 \pi L} \ln \frac{b}{a} + \frac{g(\rho - \rho_1)}{2} \left\{ \frac{b^2 - a^2}{2} - a^2 \ln \frac{b}{a} \right\} \right] \quad (2.9)$$

For the case in which the thickness of the test specimen is small, in other words b is slightly larger than a , and in addition the medium in which the viscometer is immersed has a density which is slightly different from that of the test material, it is obvious that the second term in the right hand side of equation (2.9) can be neglected when compared with the first term, hence equation (2.9) reduces to:

$$\mu = \frac{\ln \frac{b}{a}}{2 \pi L} \frac{W'}{V} = k \frac{W'}{V} \quad (2.10)$$

where:

$$k = \frac{\ln \frac{b}{a}}{2 \pi L} \quad (2.11)$$

k is called the viscometer constant.

Equation (2.10) can be obtained directly if both the weight and buoyant force of the test specimen is neglected.

Second Theoretical Approach:

This approach basically assumes the velocity profile inside the test specimen to be linear, hence on applying Newton's law of viscosity, Appendix A,

$$\tau = \frac{F}{A} = \mu \frac{V}{b - a} \quad (2.12)$$

The shearing area A is assumed to be the mean circumferential area of test specimen, hence

$$A = 2 \pi \frac{a + b}{2} L = \pi (a + b) L \quad (2.13)$$

Substituting equation (2.13) in (2.12) and solving for the viscous shearing force F ,

$$F = \mu \pi (a + b) L \frac{V}{b - a} \quad (2.14)$$

As in the first approach the shearing force can be considered due to the effective weight of both the inner cylinder and the test specimen, hence,

$$F = W + \rho g \pi (b^2 - a^2) L - \rho_1 g \pi b^2 L \quad (2.15)$$

substituting equation (2.7) in (2.15) and rearranging,

$$F = W' + g \pi L (b^2 - a^2) (\rho - \rho_1) \quad (2.16)$$

Inserting (2.16) in (2.14) and solving for the coefficient of viscosity μ equation (2.14) becomes,

$$\mu = \frac{1}{V} \left\{ \frac{W' (b - a)}{\pi L (a + b)} + \frac{g (\rho - \rho_1) (b^2 - a^2) (b - a)}{(b + a)} \right\} \quad (2.17)$$

Following the same argument done for equation (2.9), the second term in the right hand side of equation (2.17) can be safely neglected when compared with the first term, and hence equation (2.17) reduces to :

$$\mu = \frac{b - a}{\pi L (b + a)} \frac{W'}{V} = k' \frac{W'}{V} \quad (2.18)$$

where

k' is the viscometer constant and is given by:

$$k' = \frac{b - a}{\pi L (b + a)} \quad (2.19)$$

Comparison of the Two Approaches Applied to the Viscometer Used:

Comparing the viscometer constants k and k' given by equations (2.11) and (2.19)

$$\frac{k'}{k} = \frac{2 (b - a)}{(b + a) \ln \frac{b}{a}} \quad (2.20)$$

Substitution of the dimensions of the viscometer given previously in equation (2.20), yields,

$$\frac{k'}{k} = \frac{2 \times 0.122}{3.33 \times \ln \frac{1.726}{1.604}} = 1.0135$$

The above result shows that the second approach gives rise to a coefficient of viscosity which is 1.35% higher than that obtained from the first approach.

Because of the simplicity in computation of the rate of shear strain in the second approach, it was decided to use it as a computation scheme for the results obtained in this study.

Specimen Preparation:

1. The viscometer was assembled with a sheet of aluminum foil on the moulding base to prevent adhering the test material to it, and with a conical sieve at the top to help pouring the specimen.
2. The viscometer and a suitable amount of test material were left in an oven whose temperature is controlled at 275 ± 10 °F.
3. The sample was then poured from one spot to prevent formation of air bubbles. Excess material was poured to allow shrinkage on cooling.
4. The viscometer was left again in the controlled temperature oven for another 30 minutes to allow trapped air bubbles to come out.
5. The specimen was then allowed to cool down to the room temperature, and with a heated blade and kerosene the excess asphalt was removed so that the top of the specimen was even with the edge of the outer cylinder.
6. Viscometer was then placed in a controlled water bath.

Test Procedure:

1. The temperature of the water bath was held at the required temperature for at least half an hour before test.
2. The height of the water above the moulding base was measured.

3. The convenient additional weight was put in the inner cylinder.
4. The stand ring 9B was fixed to the stand shaft by tightening its screws.
5. The stand ring 9A was loosened from the stand shaft and turned around to the other side of the viscometer.
6. The inner cylinder was allowed to move down 0.02 inches and then the time required for the inner cylinder to move down 0.1 inches was measured by the electric stop watch.

CHAPTER III

PART II

THE INDENTOR VISCOMETER

Preface:

In the past the so-called penetroviscometers were used for measurements of the coefficient of viscosity of highly viscous fluids. These penetroviscometers were somewhat similar to the indentor viscometer described herein.

These viscometers can be divided into two types. In the first one a needle is forced in a specimen of the material by a given force, the depth of penetration after a certain time of applying a certain force is taken as a measure for the coefficient of viscosity of the fluid. Saan and Koens in 1933 developed an empirical formula to convert the penetration data to absolute coefficient of viscosity. The theory of this method has been studied extensively by Pendelton in 1943. The theory was also verified by experiments carried out using the capillary viscometer and the falling co-axial cylinder viscometer (5).

In the second type the needle is replaced by a hemisphere. Cox in 1943 developed by analogy with an elastic counterpart an expression to give the coefficient of viscosity in terms of the radius of the hemisphere, the radius of the imprint, the load applied and the time of application of the load. An apparatus was constructed and the viscosity of glass measured with it showed good agreement with the results obtained by the other methods (5).

Bikerman (2), in 1947, devised a penetroviscometer for very viscous fluids in which he used a brass rod to penetrate a fluid contained in a slightly larger tube. He analyzed the problem theoretically by writing down the equation of motion and solving it by making the following assumptions:

- a. No pressure gradient in the radial direction, since the tube is slightly larger than the rod the motion in the radial direction can be neglected with respect to the axial one.
- b. The pressure at the lower end of the rod equals the effective weight of the rod divided by the cross-sectional area of the tube.
- c. The pressure gradient in the axial direction was linear.

He developed two equations from which the coefficient of viscosity could be computed as a function of the dimensions of the apparatus and the rate of penetration of the rod through the specimen. He verified the equations he obtained experimentally using a solution of rosin and wood turpentine.

The results obtained by Bikerman can not be applied to the indenter viscometer because none of the assumptions made by Bikerman can be applied to the indenter viscometer.

Dimensional Analysis of the Indenter Viscometer:

The problem of the indenter viscometer can be stated as seeking the force F required to indent the fluid of coefficient of viscosity μ and density ρ placed in a container whose diameter is D and the height of fluid in it is H with an indenter of diameter d and with a velocity of indentation V . All these variables are shown in the schematic drawing of the container and indenter shown in Fig.(3.1). The variables and their dimensions are listed below in table I.

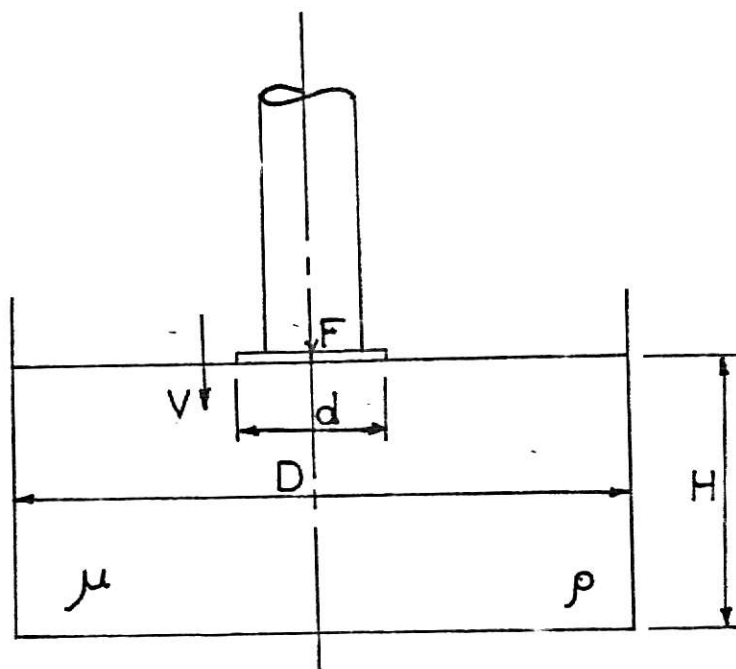


Fig.(3.1) Indentor viscometer model.

TABLE I
DIMENSIONS AND NOTATIONS OF VARIABLES

Variable	Notation	Dimensions
Force of indentation	F	$M L T^{-2}$
Velocity of indentation	V	$L T^{-1}$
Diameter of indenter	d	L
Diameter of container	D	L
Height of fluid in container	H	L
Density of fluid	ρ	$M L^{-3}$
Coefficient of viscosity of fluid	μ	$M L^{-1} T^{-1}$

Considering the force of indentation as the dependent variable, the functional relation between the dependent and independent variables can be assumed in the form :

$$F = K V^a d^b D^c H^e \rho^f \mu^g \quad (3.1)$$

where K is a constant.

Substituting the dimensions of the variables given in table I in equation (3.1).

$$M L T^{-2} = K (L T^{-1})^a L^b L^c L^e (M L^{-3})^f (M L^{-1} T^{-1})^g \quad (3.2)$$

Equating the dimensions of the three main dimensions M, L, and T in both sides of equation (3.2),

$$1 = f + g \quad (3.3.a)$$

$$1 = a + b + c + e - 3f - g \quad (3.3.b)$$

$$-2 = -a - g \quad (3.3.c)$$

From equation (3.3.a)

$$f = 1 - g \quad (3.4.a)$$

From equation (3.3.c)

$$a = 2 - g \quad (3.4.b)$$

Substituting equation (3.4.a) and (3.4.b) in (3.3.b) and solving for b, hence,

$$b = 2 - g - c - e \quad (3.4.c)$$

Inserting equations (3.4.a), (3.4.b) and (3.4.c) in equation (3.1),

$$F = K V^{2-g} d^{2-g-c-e} D^c H^e \rho^{1-g} \mu^g$$

Hence,

$$\frac{F}{\rho V^2 d^2} = K \left(\frac{\mu}{\rho V d} \right)^g \left(\frac{D}{d} \right)^c \left(\frac{H}{d} \right)^e \quad (3.5)$$

The dependent dimensionless group $\frac{F}{\rho V^2 d^2}$ represents the resistance offered by the fluid to the indenter. The first independent dimensionless group $\frac{\mu}{\rho V d}$ is clearly the reciprocal of the Reynold's number. The second and third independent dimensionless groups $\frac{D}{d}$ and $\frac{H}{d}$ represent size ratios.

Fixing the second and third independent dimensionless groups $\frac{D}{d}$ and $\frac{H}{d}$ by fixing the diameter of container and indenter and height of fluid in container, equation (3.5) then reduces to :

$$\frac{F}{\rho V^2 d^2} = K \left(\frac{\mu}{\rho V d} \right)^g \quad (3.6)$$

The above equation shows that if the exponent g is found experimentally to be unity the relation (3.6) reduces to the same form obtained by Goudy and Kirmser (4).

Description of Viscometer:

A diagrammatic sketch of the viscometer set up is shown in plate II.

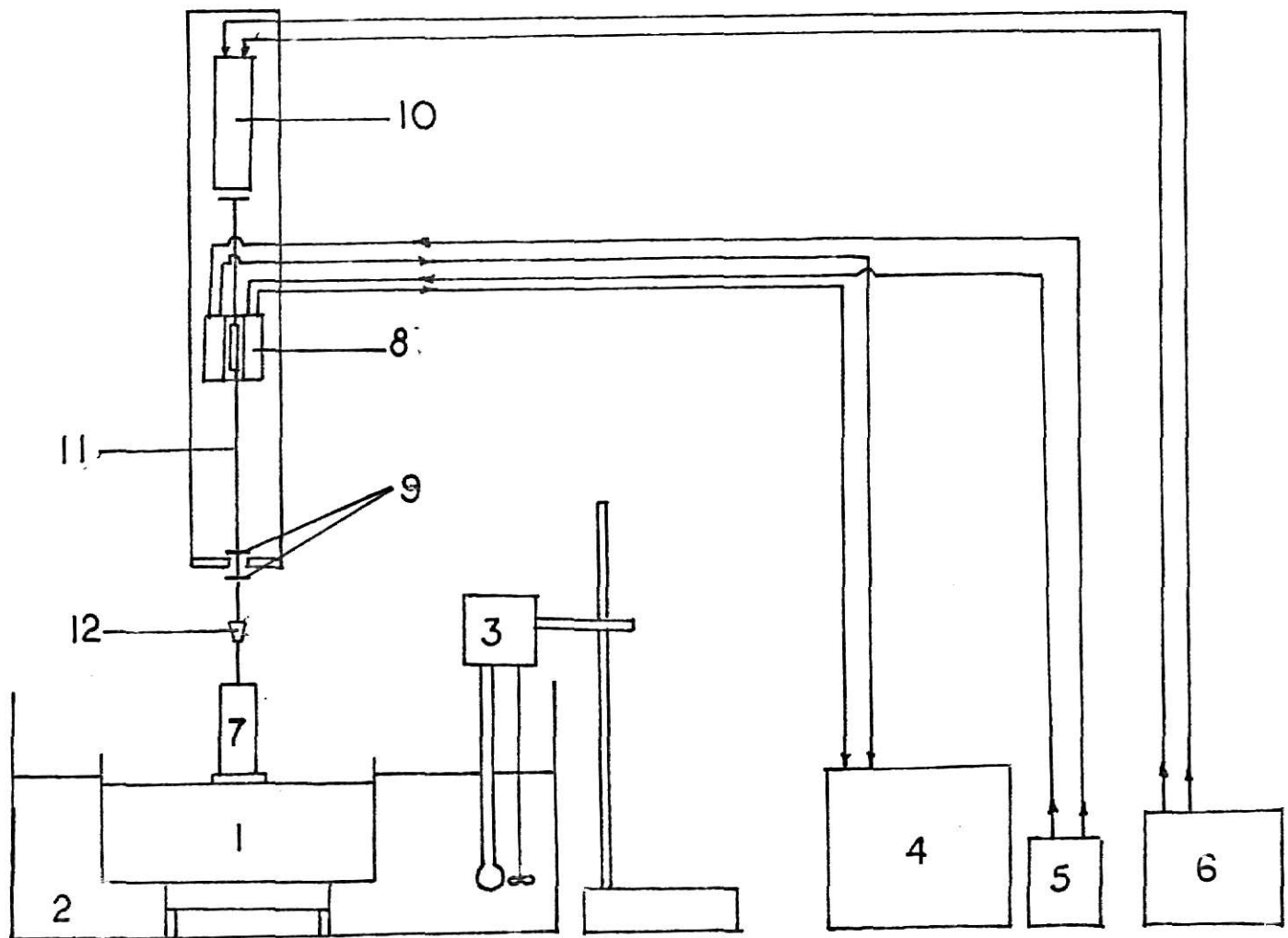
The specimen under test was put in a container 1 which was placed in a water bath 2 whose temperature was controlled by the temperature controller 3.

EXPLANATION OF PLATE II

THE INDENTOR VISCOMETER

1. Fluid specimen.
2. Water bath.
3. Temperature controller.
4. Strip chart recorder.
5. Dry battery.
6. Power supply.
7. Indentor.
8. Displacement transducer.
9. Adjustable stops.
10. Electric magnet.
11. Rod.
12. Chuck.

PLATE II



The indenter 7 was suspended in a rod 11 by a chuck 12. The two adjustable stops 9 were used to adjust the depth of indentation of the indenter. The rod 11 was connected to the core of the displacement D-C transducer 8. The transducer was excited by a 6 volt D-C dry battery 5. The output of the transducer was amplified in a D-C preamplifier and was recorded on the strip chart recorder 4. The indenter was held up by an electric magnet 10 which was excited by a D-C power supply 6. The transducer and the electric magnet were fixed in a vertical plate which was fixed to an adjustable stand. A photograph of the viscometer set up is shown in Fig. (3.2) and a photograph of the stand, electric magnet, transducer and indenter is shown in Fig. (3.3).

Specifications of Equipments:

1. Container 1 : is a pyrex crystalized dish of 190 mm diameter and 100 mm height.
2. Temperature controller 3 : CTC, Circo-Temp, The Chemical Rubber Co.
3. Strip chart recorder 4 : Sanborn strip chart recorder model 7702B with a D-C preamplifier model 8801A.
4. Power supply 6 : D-C power supply NJE corporation model RB 36-2 0-36 VDC 0-2 Amp.
5. Indenter 7 : 2 in. diameter and 456.7 gm weight.
6. Displacement transducer 8 : Hewlett Packard series 7DCDT.

Test Procedure:

1. The strip chart recorder was switched on and left for 10 minutes to warm up.
2. The gain of the preamplifier was adjusted to get a full span (5 cm)

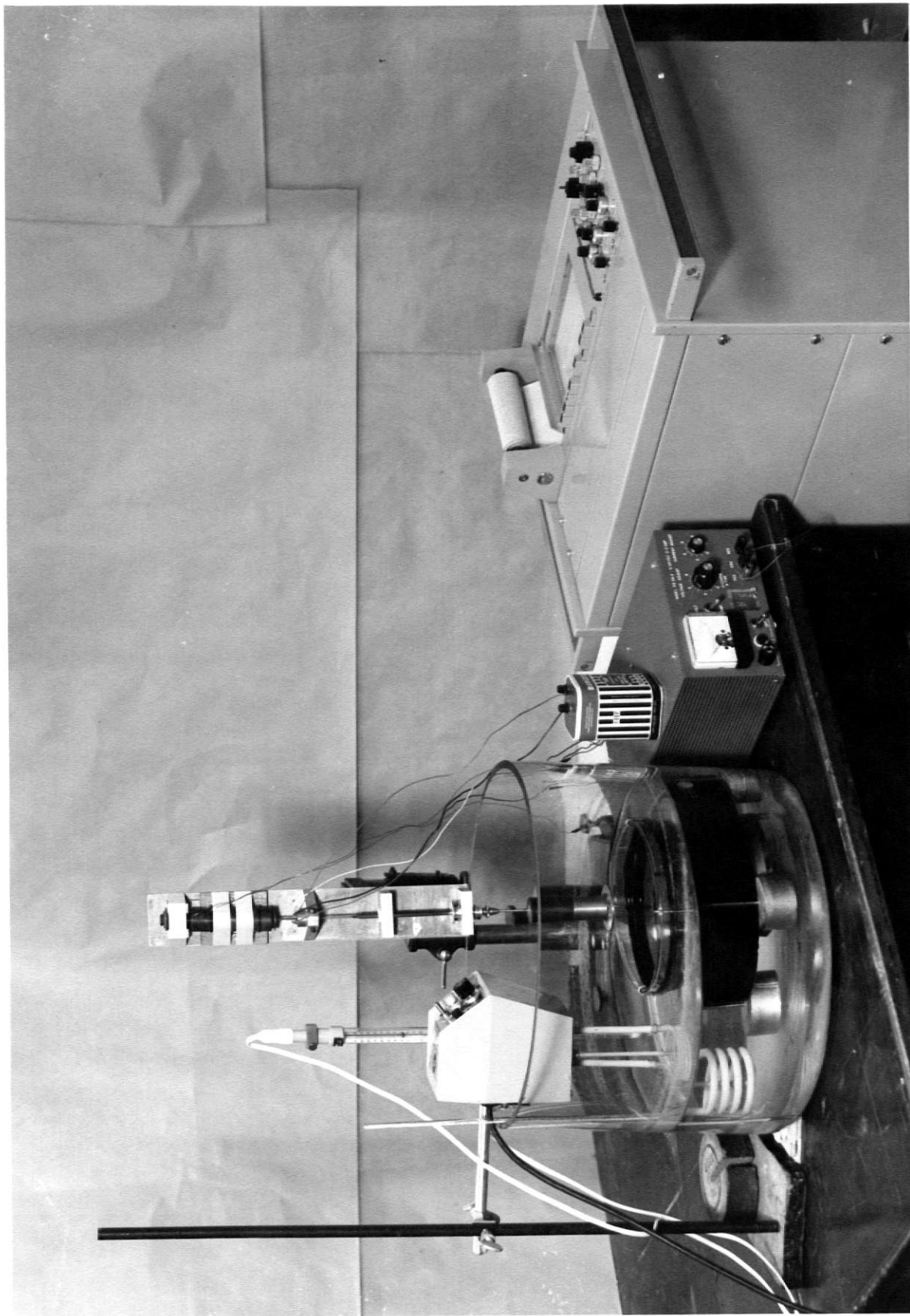


Fig.(3.2) The indenter viscometer set up.

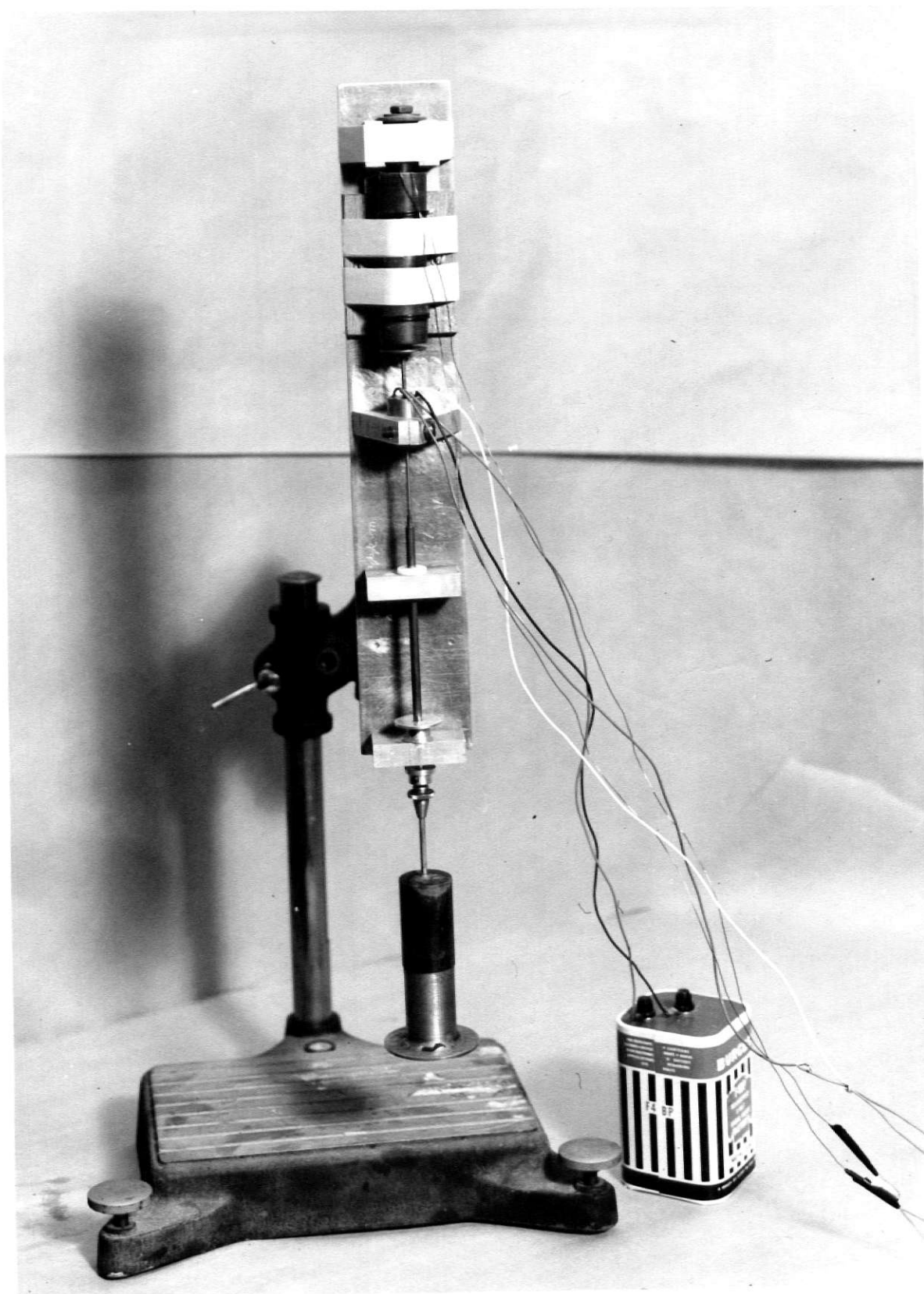


Fig.(3.3) The stand, electric magnet, transducer and indenter assembly.

deflection of the recorder corresponding to the 0.1 in. depth of indentation.

3. The indenter was held up by the electric magnet, centered and adjusted to touch the specimen surface.
4. A suitable chart speed was chosen and the indenter was left to fall down by switching off the power supply unit.
5. A displacement time curve was obtained on the strip chart recorder from which the velocity of indentation was computed.

CHAPTER IV

RESULTS AND DISCUSSIONS

Part I

The Falling Co-axial Cylinder Viscometer Check of Newtonianity of Asphalt:

Newtonian fluid by definition is the fluid for which the shear stress is proportional to the rate of shear strain, Appendix A. This check was done for two types of asphalt obtained from the Phillips Petroleum Company. The first one was specified as 85- 100 penetration number asphalt, the second was 40-60 penetration number asphalt.

To check for Newtonianity the temperature of the specimen was fixed and the rate of shear strain was measured at five different shear stresses. The shear stress was varied by adding additional weights to the inner cylinder of the falling co-axial cylinder viscometer. The additional weights 250, 500, 750 and 1000 grams were chosen to be added to the inner cylinder. The fifth shearing weight was the weight of cylinder alone. The measurements of the rate of shear strain were repeated three time for each shearing weight which made the whole set of experiments consists of 15 experiments. The 15 experiments were carried out in a randomly ordered manner to prevent any repeated error for the same reading.

Figure (4.1) shows the results obtained for 85- 100 penetration number asphalt. The temperature of asphalt for this set of experiments was 84.65°F . The figure shows the shear stress versus rate of shear strain. The results shows a linear relation between the shear stress and the rate of shear strain

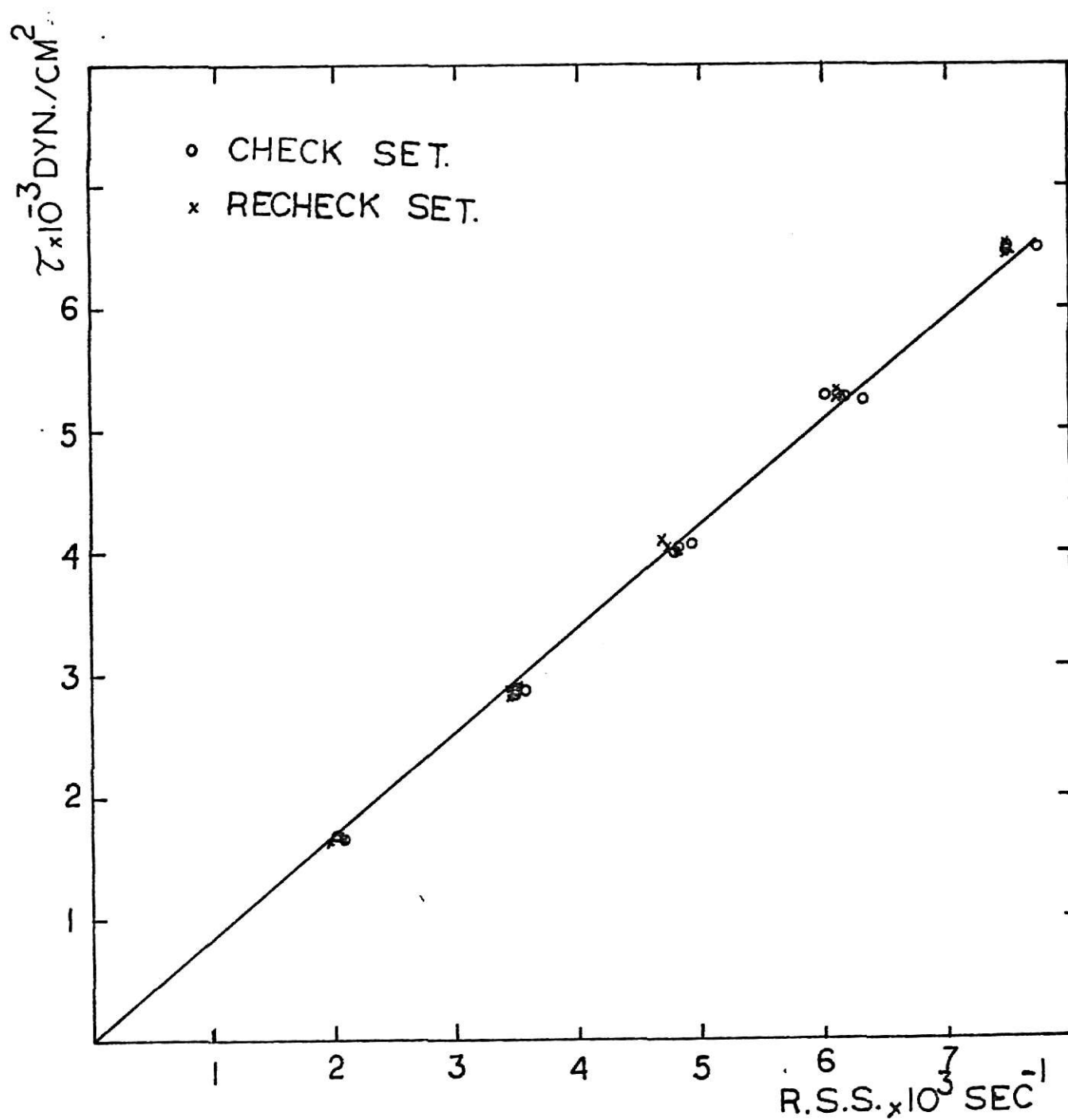


Fig.(4.1) Check of Newtonianity of 85-100 P.N. asphalt.

which means that the 85-100 penetration number asphalt shows a Newtonian behaviour. The whole set of experiments was repeated once more for the same 85-100 penetration number asphalt to check the consistency of the results obtained from the falling co-axial cylinder viscometer and asphalt itself. The results of both sets of experiments are shown in the same figure which shows a good consistent behaviour. The maximum deviation of the obtained experimental points from the straight line relation was of the order of 5%.

Figure (4.2) shows the results obtained for 40-60 penetration number asphalt. The temperature of asphalt for this set was 90 °F. This asphalt shows also a Newtonian behaviour as the first one and as shown from the straight line behaviour shown in Fig. (4.2).

Measurements of Viscosity of Asphalt at Different Temperatures:

The coefficient of viscosity of two types of asphalt was measured at different temperatures. The first one was 60-70 penetration number asphalt, the second was 40-50 penetration number asphalt.

Figure (4.3) shows the coefficient of viscosity of 60-70 penetration number asphalt versus temperature of asphalt drawn on a semi-logarithmic graph paper. The coefficient of viscosity of asphalt was measured at five different temperatures ranging from 80 °F to 100°F. The viscosity- temperature relation for 60-70 penetration number asphalt shows an approximate linear relation as shown in Fig. (4.3). The results also show how sensitive asphalt is to temperature variation, a 5°F drop in temperature causes the viscosity of asphalt to be almost doubled.

Figure (4.4) shows the coefficient of viscosity of 40-50 penetration number asphalt versus temperature of asphalt drawn on a semi-logarithmic graph paper. The coefficient of viscosity of 40-50 penetration number asphalt was

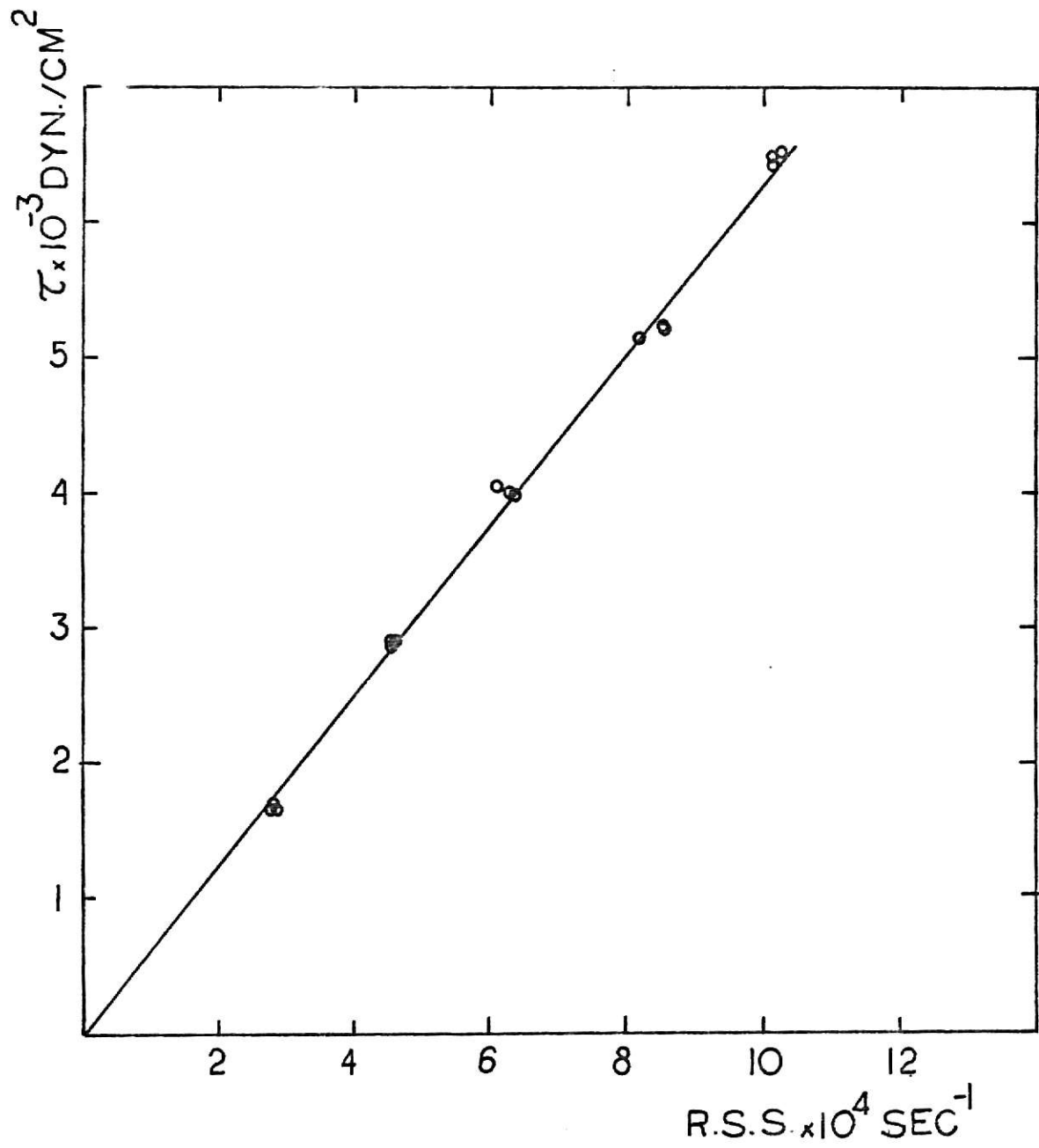


Fig.(4.2) Check of Newtonianity of 40-60 P.N. asphalt.

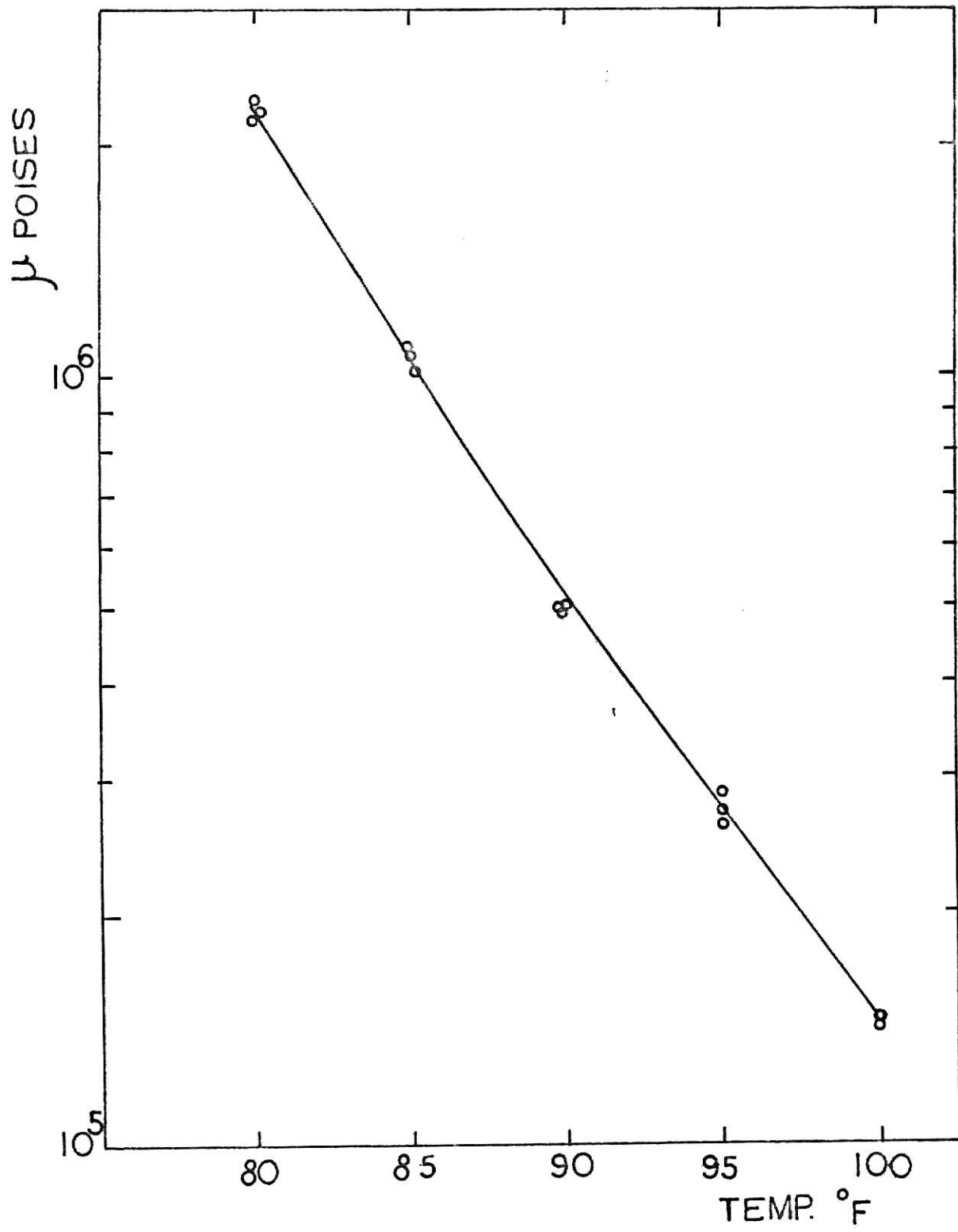


Fig.(4.3) Viscosity coefficient of 60-70 P.N. asphalt.

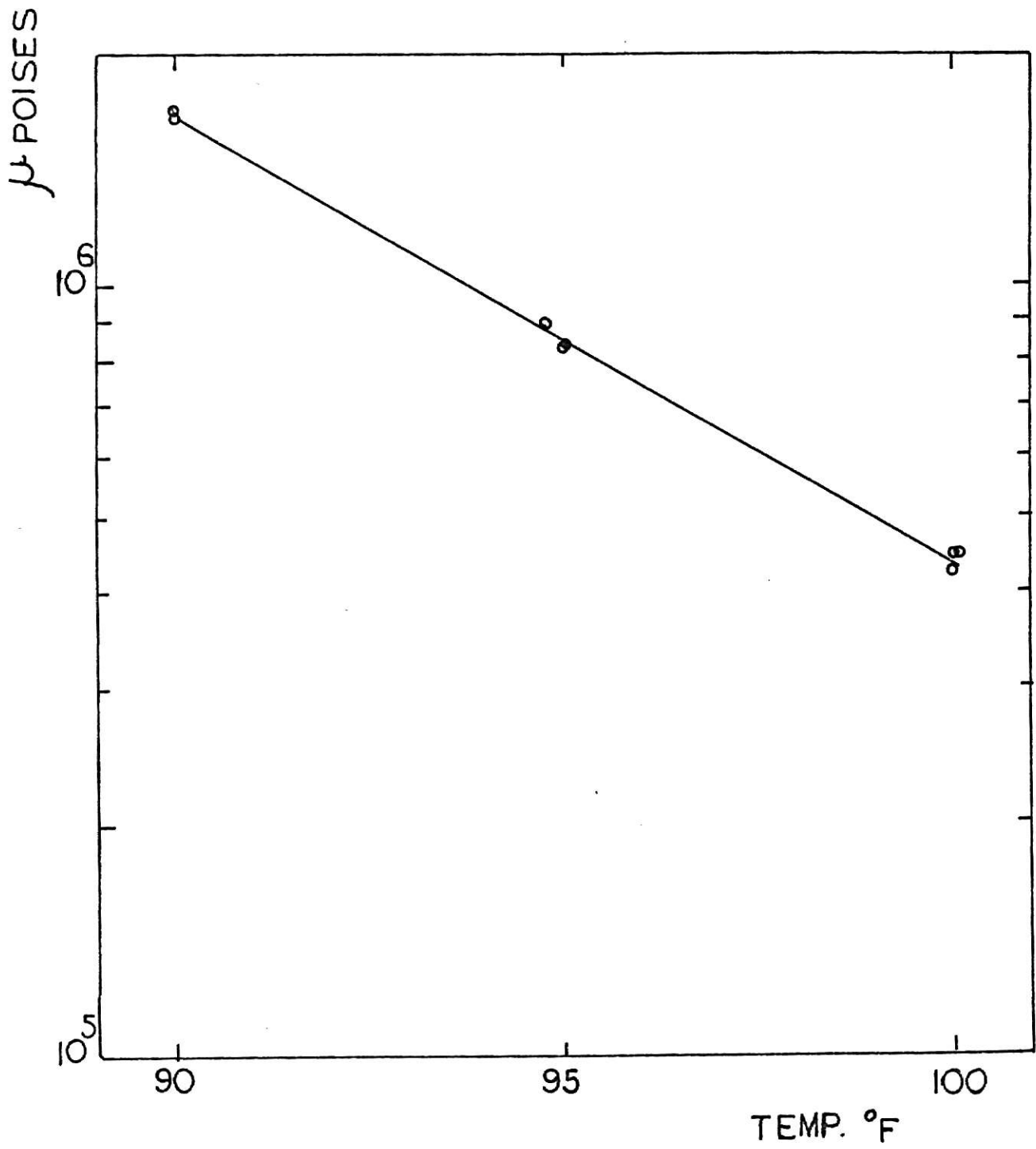


Fig.(4.4) Viscosity coefficient of 40-50 P.N. asphalt.

measured at three different temperatures ranging from 90 °F to 100 °F. The results shows the same general trend as that obtained from 60-70 penetration number asphalt.

The tables of results of this part of the study are shown in Appendix B.

Part II

The Indentor Viscometer:

It has been shown previously in chapter III that if the diameter of both indentor and container is kept constant and also the height of asphalt in container is kept constant the functional relation between the dependent and independent dimensionless groups reduces to:

$$\frac{F}{\rho V^2 d^2} = K \left(\frac{\mu}{\rho V d} \right)^g$$

The aim of the experiments done using the indentor viscometer was to obtain the exponent g and the constant K if the relation between the two dimensionless groups is a linear one when plotted on a logarethmic graph paper.

First, the experiments were tried using a medium weight indentor and 60-70 penetration number asphalt, but the time-displacement curve obtained from the strip chart recorder showed oscillations at the begining of indentation. This oscillations increase with the increase of temperature of asphalt. So a lighter weight indentor was used. The experiments were done on 60-70 penetration number asphalt to measure the velocity of indentation of an indentor of a 2 in. diameter and whose weight with all parts connected to it is 499.435 grams at five different temperatures ranging from 80 °F to 100 °F. Each experiment was repeated three times which made the whole set of experiments. consists of 15 experiments. Also the experiments were carried out in a randomly ordered manner to prevent any possible repeated error for the same

experiment.

The displacement - time curve obtained from the strip chart recorder showed that the indenter during its fall does not actually attain a terminal velocity for the 0.1 in. depth of indentation. The velocity of indentation started with a high value which reduced gradually. Increasing the depth of indentation to 0.15 in. did not cause the indenter to reach a terminal velocity. The velocity of indentation was computed for three different depths of indentation. The first one is the velocity of indentation in the region between 0.02 to 0.05 in. depth of indentation, the second one is in the region between 0.05 to 0.08 in. depth of indentation and the third one is in the region between 0.02 and 0.08 in. depth of indentation. The largest difference between the lowest and the highest velocity of indentation is in the order of 10%. In this study the velocity of indentation was considered to be that in the region from 0.02 to 0.08 in. depth of indentation since it showed an average value between the lowest and the largest velocities. A typical displacement-time curve is shown in Fig. (4.5).

The results obtained by the first set of experiments on the 60-70 penetration number asphalt are shown in Fig. (4.8). Most of the experimental points of this set lie on the straight line denoted by C except some points at temperature of 80 °F and 85 °F. It was noticed during this set of experiments that the indenter did not adhere to asphalt and only made an imprint on the surface of asphalt. This means that the indenter did not penetrate through asphalt and this may be due to the formation of a some sort of a stretched membrane on the surface of asphalt as a result of the repeated process of heating and cooling of asphalt after each experiment. Figure (4.6) shows a photograph of the imprint explained above.

Due to the uncertainty of the results at the temperatures of 80°F and

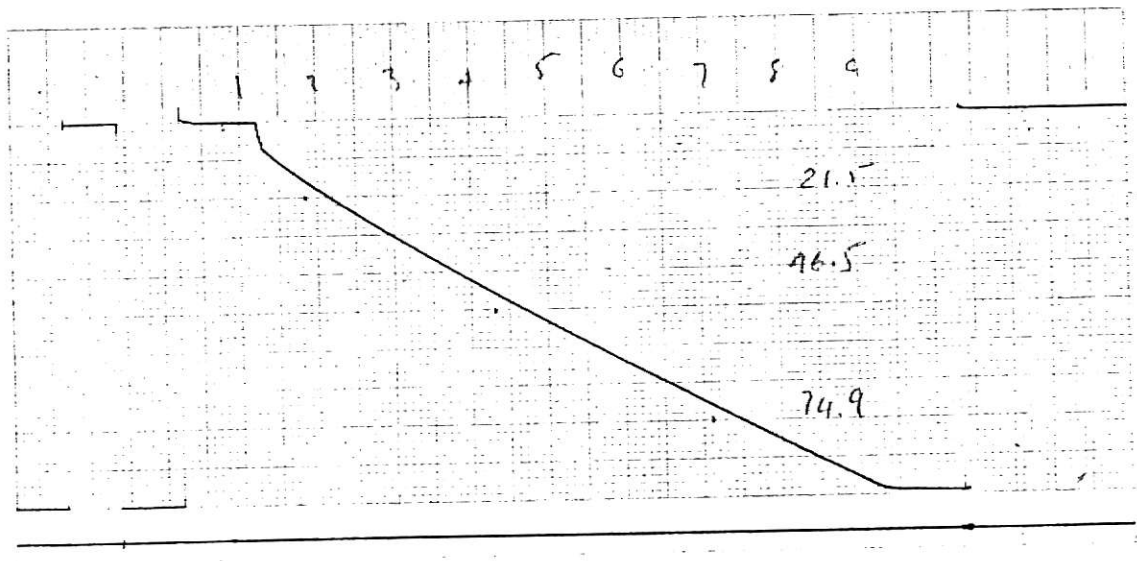
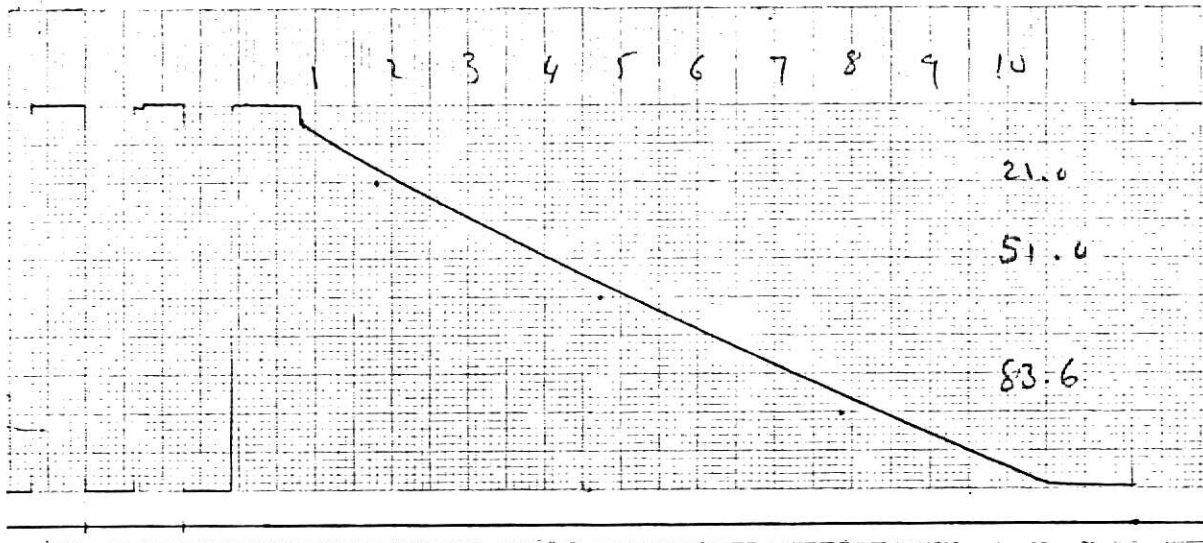


Fig.(4.5) A typical displacement-time Curve.



Fig. (4.6) The imprint of indenter on asphalt.

85 °F, another specimen of the same asphalt was prepared and the same set of experiments was repeated. The results of this set of experiments showed that the first four experimental points lie on the straight line denoted by A in Fig. (4.8). The next five experimental points lie on the straight line denoted by C and the last six experimental points lie on a straight line denoted by B in between the lines A and C. It was noticed that for the first four experimental points, i.e. for the line A, the indenter adhered to the asphalt which means that the indenter was actually penetrating through asphalt and that the stretched membrane surface had not formed yet. This phenomena did not appear for the first few experiments in the first specimen because some preliminary experiments were done on the first specimen to gain experience in operating the viscometer. The viscometer appeared to adhere to asphalt in these few preliminary experiments. This phenomena of adhering is shown in Fig. (4.7), a photograph for the asphalt and indenter after the indenter has been separated from asphalt by a hot blade.

For the next five experiments, i.e. for the line C, the indenter did not adhere to the asphalt which means that the stretched membrane surface was formed. For the last six experiments it was noticed that the weather was relatively hot which made the conditions of cooling of the surface of asphalt slightly different and the stretched membrane surface seemed to be somewhat weaker than that for the line C. It was also noticed that for these six experiments the indenter slightly adhere to the asphalt but only at the edges of the indenter which indicated that the stretched membrane was sheared by the indenter, a phenomena which did not appear for the line C.

From the above discussion it can be seen that the velocity of indentation of asphalt depends on the conditions of cooling of the surface of asphalt.

From Fig. (4.8) the straight lines A,B and C are shown to be parallel



Fig.(4.7) The indenter and asphalt when penetrating.

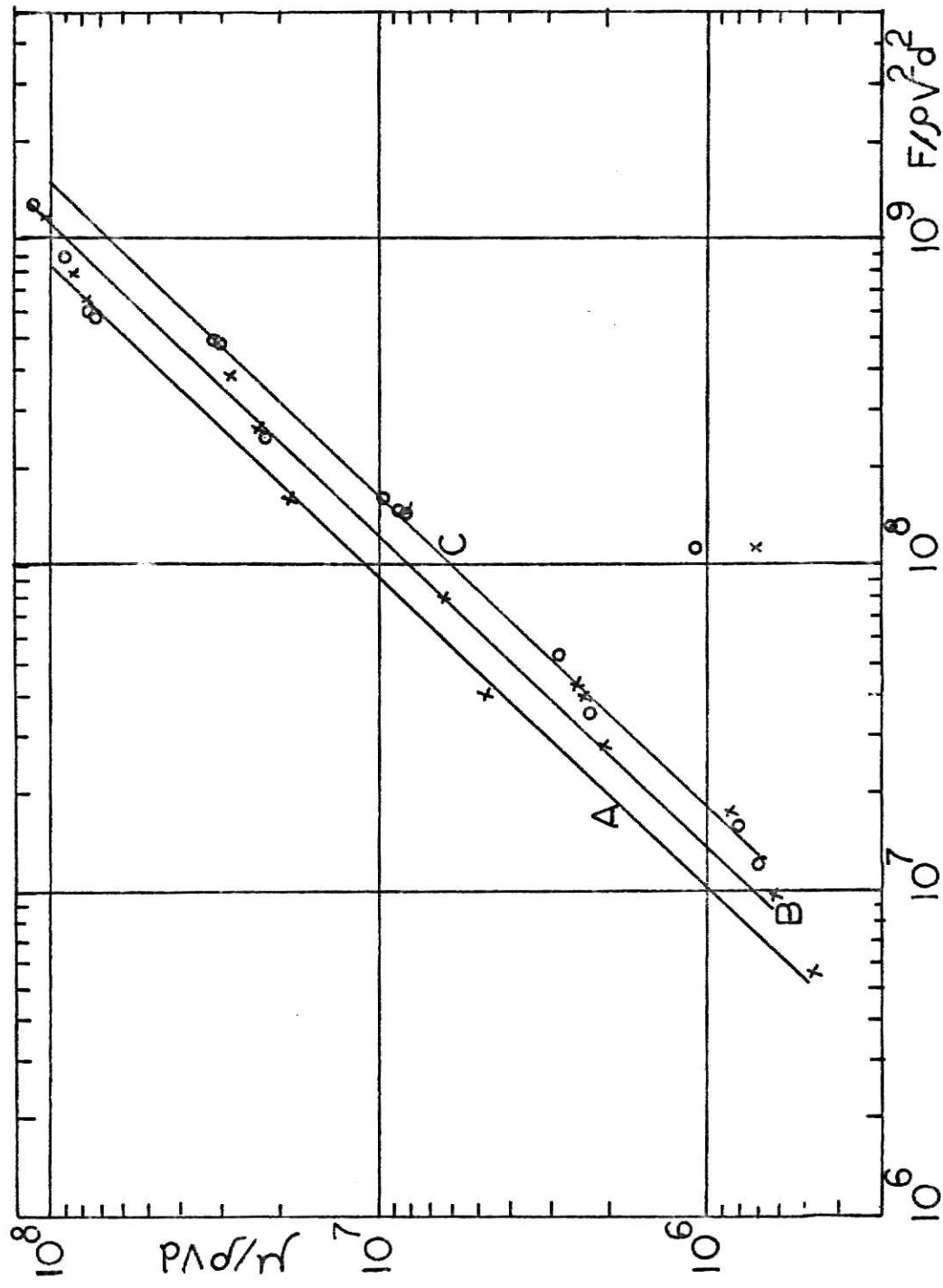


Fig.(4.8) The functional relationship for the indenter viscometer.

which means that they have the same slope, i.e. the same exponent g which is found to be 0.957. But they have different values of the constant K being 18.1 for line A, 24.5 for line B and 32.5 for line C. In other words the constant K depends mainly on the conditions of cooling of the surface of the asphalt.

Secondly, as a check of the validity of the results shown in Fig.(4.8) and the indenter viscometer for measurements of the coefficient of viscosity of asphalt, 40-50 penetration number asphalt was used. The coefficients of viscosity of the 40-50 penetration number asphalt at three different temperatures ranges from 90 °F to 100 °F were measured by the indenter viscometer and were compared with those obtained by the falling co-axial cylinder viscometer shown in Fig.(4.4). The indenter was observed to adhere to asphalt in the first experiment only, so its viscosity was obtained from line A. In the second experiment the indenter was observed to adhere at the edges only, so its viscosity was obtained from line B. For the rest of experiments the viscosity was obtained from line C as the indenter was not observed to adhere to asphalt. Figure (4.9) shows a good agreement for the results obtained by both viscometers.

The tables of results of this part is shown in Appendix B.

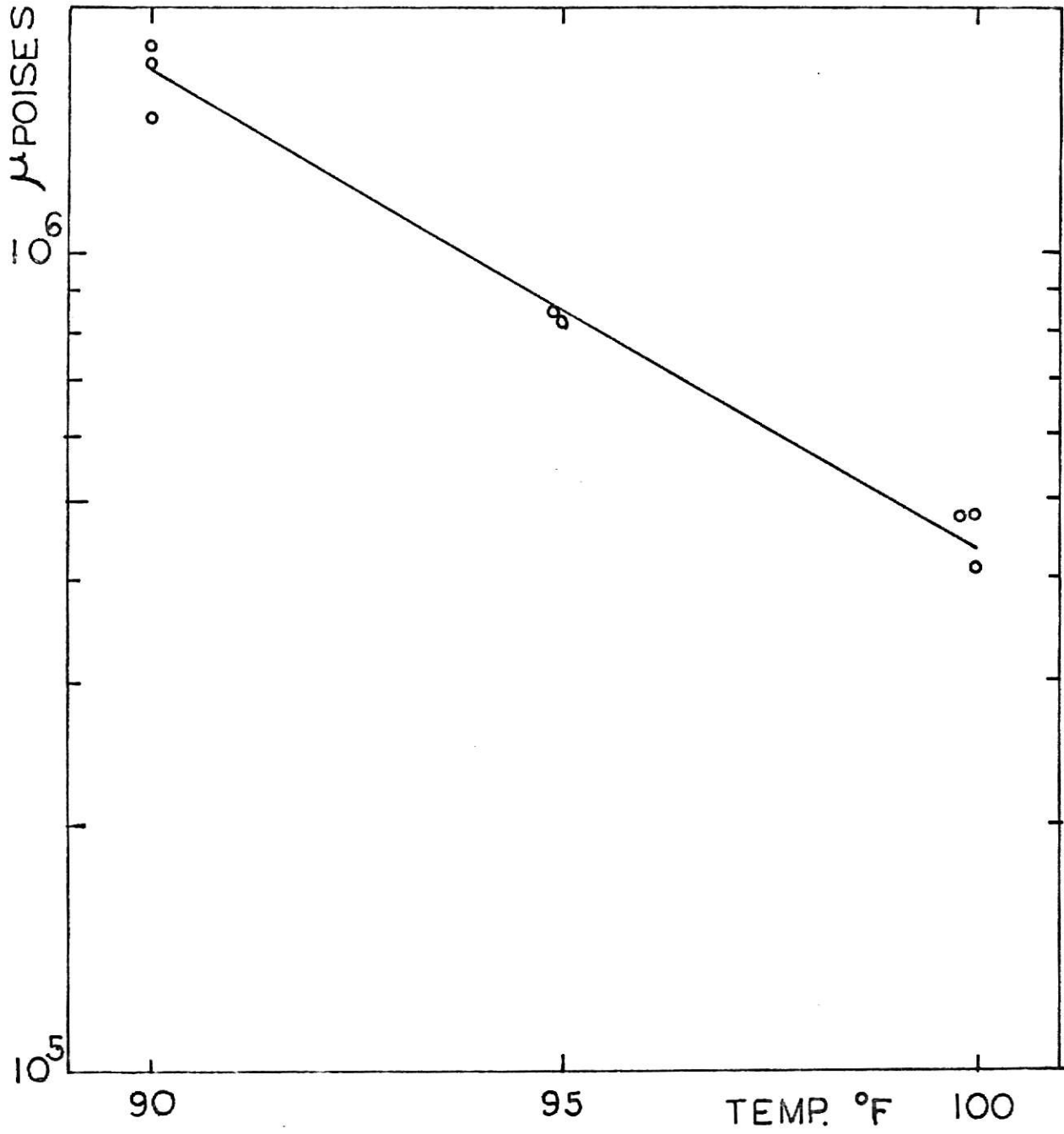


Fig.(4.9) Comparison of viscosity measurements by the indenter viscometer and the falling co-axial cylinder viscometer.

CHAPTER V

CONCLUSIONS

The results discussed in chapter IV show that the falling co-axial cylinder viscometer is indeed a convenient viscometer for measurements of coefficient of viscosity for highly viscous fluids with a good accuracy.

Also it could be concluded that asphalt as a fluid of different grades showed a Newtonian behaviour and it is a highly temperature sensitive fluid, Its coefficient of viscosity almost doubled for a 5 °F decrease in its temperature.

As far as the indentor viscometer concern, the dimensional analysis discussed in chapter III show that for the same indentor diameter, container size and height of asphalt in container,

$$\frac{F}{\rho V^2 d^2} = K \left(\frac{\mu}{\rho V d} \right)^8$$

which means that the resistance offered by a fluid to the indentor is a function of the Reynold's number based on the indentor diameter.

The experimentation done on the indentor viscometer as an apparatus and asphalt as a fluid showed that the functional relationship between the above mentioned dimensionless parameters is in the form:

$$\frac{F}{\rho V^2 d^2} = K \left(\frac{\mu}{\rho V d} \right)^{0.957}$$

where K is a constant found to be a function of the conditions of cooling of the surface of asphalt. The value of K was found to vary between 18.1 to 32.5.

The reason for this is believed to be due to the formation of a stretched membrane surface on the surface of asphalt which is subjected to the uncontrolled environmental atmospheric conditions as a result of the repeated process of heating and cooling of asphalt.

Recommendations for Future Studies:

The indenter viscometer as described in this report needs a thorough theoretical investigation to compare with the experimental data. In addition, the author feels that for another viscous fluid other than asphalt, which does not have the same property of forming an uncontrolled stretched membrane surface when subjected to the repeated process of heating and cooling the results is expected to be more consistent.

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APPENDIXES

APPENDIX A

VISCOSITY, NEWTON'S LAW OF VISCOSITY
AND CLASSIFICATION OF FLUIDS

The idealized model shown in Fig.(A.1) has often been used to derive a quantitative definition of fluid viscosity. The spacing between the two parallel plates of area A is filled with fluid. For a force F applied tangentially to the upper plate to cause it to move with a velocity U relative to the stationary bottom plate, the shear stress will be given by F/A. If the velocity profile between the two parallel plates is linear, the rate of shear strain will be given by U/b. In the absence of gravity and pressure gradient, we can write

$$\tau = \mu \frac{U}{b}$$

where μ is defined as the coefficient of viscosity.

More generally, the rate of shear strain can be defined as $\frac{du}{dx}$ where x is the distance measured normally from the stationary plate. Hence,

$$\tau = \mu \frac{du}{dx}$$

This is the well-known Newton's law of viscosity.

Fluids which behave according to this law are known as "Newtonian Fluids". For these fluids the relationship between the shear stress and the shear strain is one of direct proportionality as shown by A in Fig.(A.2). There are however many materials of great industrial importance like emulsions and concen-

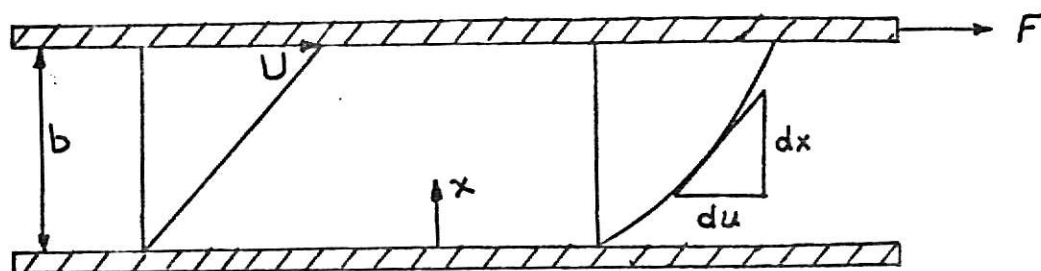


Fig.(A.1) Newton's conceptional model for defining the viscosity of a real fluid.

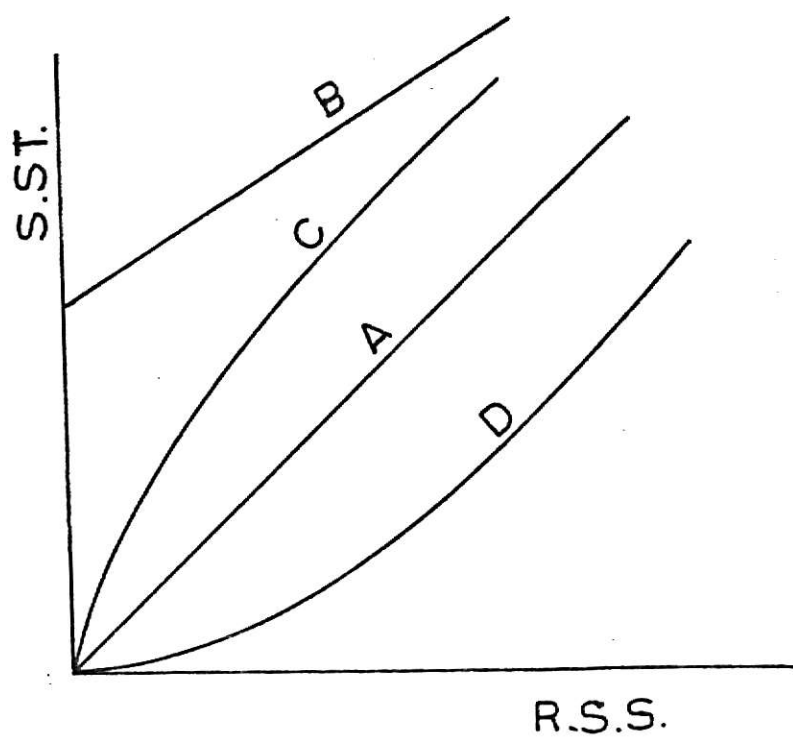


Fig.(A.2) Types of consistency curves.

trated suspensions that are Non-Newtonian as shown by curves C and D in Fig.(A.2). These are sometimes called "Pseudoplastics" or "Power law fluids".

A third type of fluids, which is called "Bingham fluid" or the "Ideal plastic", shows a straight line relationship between shear stress and shear strain but not passing through the origin as shown by line B in Fig.(A.2).

APPENDIX B

TABLES OF RESULTS

TABLE II

CHECK OF NEWTONIANITY OF 85-100 P.N. ASPHALT

Seq.	H	τ	Time	R.S.S. $\times 10^3$
No.	in.	dynes/cm ²	Sec.	Sec. ⁻¹
9	3.18	4056.76	167.0	4.913
13	3.20	1641.60	416.5	1.968
6	3.19	5251.62	133.1	6.167
4	3.15	5277.10	136.8	5.992
2	3.18	6459.20	109.7	7.472
1	3.22	6433.74	109.7	7.472
8	3.31	3974.03	171.2	4.788
11	3.18	2855.54	231.2	3.545
7	3.21	4037.67	170.2	4.816
10	3.21	2836.45	235.7	3.478
3	3.17	6465.60	105.7	7.755
5	3.24	5219.80	129.5	6.330
12	3.19	2849.20	235.0	3.488
15	3.18	1654.30	397.5	2.062
14	3.19	1654.30	395.3	2.074

temperature = 84.65 °F.

TABLE III

RECHECK OF NEWTONIANITY OF 85-100 P.N. ASPHALT

Seq. No.	H in.	τ dynes/cm ²	Time Sec.	R.S.S. $\times 10^3$ Sec. ⁻¹
9	3.14	4082.20	175.8	4.662
13	3.19	1647.96	394.8	2.076
6	3.10	5308.90	134.4	6.099
4	3.15	5277.10	134.4	6.099
2	3.12	6497.40	109.8	7.465
1	3.23	6427.40	110.0	7.452
8	3.21	4037.70	173.6	4.722
11	3.18	2855.50	238.6	8.313
7	3.25	4012.20	170.3	4.813
10	3.10	2906.50	234.0	3.503
3	3.18	6459.20	108.9	7.527
5	3.18	5258.00	134.0	6.144
12	3.28	2791.90	240.0	3.415
15	3.18	1654.30	398.4	2.026
14	3.19	1648.00	396.1	2.069

Temperature = 84.65 °F.

TABLE IV

CHECK OF NEWTONIANITY OF 40-60 P.N. ASPHALT

Seq. No.	H in.	τ dynes/cm ²	Time Sec.	R.S.S.x10 ³ Sec. ⁻¹
9	3.20	4044.0	1339.9	6.117
13	3.20	1641.6	2845.8	2.880
6	3.20	5245.3	958.6	8.551
4	3.23	5226.2	954.5	8.587
2	3.24	6421.0	807.0	10.16
1	3.08	6522.8	800.5	10.24
8	3.28	3993.1	1285.1	6.378
11	3.12	2893.4	1777.5	4.611
7	3.28	3993.1	1288.3	6.362
10	3.17	2861.9	1793.8	4.569
3	3.11	6503.7	811.5	10.10
5	3.38	5130.7	1001.0	8.189
12	3.11	2900.1	1788.1	4.584
15	3.09	1711.6	2918.3	2.809
14	3.20	1641.6	2969.6	2.760

Temperature = 90 °F.

TABLE V
MEASUREMENTS OF VISCOSITY COEFFICIENTS OF 60-70 P.N.
ASPHALT AT DIFFERENT TEMPERATURES

Seq. No.	Temp. ° F	H in.	τ dynes/cm ²	Time Sec.	R.S.S.x10 ³ Sec. ⁻¹	μ x10 ⁵ poises
9	89.9	3.25	1609.8	256.3	3.198	5.034
13	100.0	3.20	1641.6	73.0	11.228	1.462
6	85.0	3.10	1765.2	509.9	1.608	10.604
4	85.1	3.13	1686.1	495.9	1.653	10.200
2	80.1	3.15	1673.4	1075.7	0.762	21.960
1	80.0	3.15	1673.4	1071.8	0.765	21.880
8	90.0	3.19	1648.0	244.1	3.358	4.908
11	95.0	3.20	1641.6	131.7	6.224	2.638
7	90.0	3.18	1654.3	248.1	3.304	5.007
10	95.0	3.18	1654.3	129.3	6.338	2.610
3	80.0	3.18	1654.3	1142.7	0.717	23.06
5	85.0	3.14	1679.8	532.1	1.540	10.91
12	95.0	3.22	1628.9	144.2	5.684	2.866
15	100.0	3.19	1648.0	71.2	11.512	1.432
14	100.0	3.15	1673.4	71.6	11.448	1.448

TABLE VI
MEASUREMENTS OF VISCOSITY COEFFICIENTS OF 40-50 P.N.
ASPHALT AT DIFFERENT TEMPERATURES

Seq. No.	Temp. ° F	H in.	τ dynes/cm ²	Time Sec.	R.S.S. $\times 10^3$ Sec. ⁻¹	$\mu \times 10^{-5}$ poises
13	100.0	3.20	1641.6	211.8	3.8685	4.243
11	98.0	3.10	1710.4	397.6	2.0607	8.300
9	90.0	3.17	1660.7	814.8	1.0060	16.540
10	94.8	3.07	1729.5	421.6	1.9434	8.900
15	100.1	3.12	1697.7	216.5	.7845	4.490
8	90.0	3.07	1729.5	788.2	1.0395	16.640
12	95.0	3.07	1729.5	394.2	2.0785	8.320
14	100.0	3.07	1729.5	212.6	3.8540	4.488
7	90.0	3.20	1641.6	857.7	0.9553	17.18

TABLE VII
THE RELATION BETWEEN THE DIMENSIONLESS GROUPS
FOR THE INDENTOR VISCOMETER
(Specimen 1)

Seq. No.	Temp. °F	Vel.x10 ³ cm/sec	$\frac{F}{\rho V^2 d^2} \times 10^{-7}$	$\frac{\mu}{\rho V d} \times 10^{-5}$
9	90.2	11.3	14.43	85.87
13	100.1	39.5	1.181	6.915
1	80.1	5.76	55.53	713.00
4	85.3	8.66	24.56	220.70
2	79.9	5.56	59.60	762.7
8	90.25	11.4	14.18	83.78
11	94.95	23.2	3.423	22.72
7	90.05	10.7	16.09	94.25
10	95.0	18.5	5.383	28.30
3	80.0	3.77	129.60	1115.00
15	100.05	34.00	1.594	8.09
12	95.00	21.10	4.138	24.80
14	100.00	39.30	1.193	7.00
5	85.20	6.28	46.71	310.20
3'	80.0	4.62	86.30	909.60
6	85.1	6.21	47.80	320.00
2'	79.95	4.61	86.68	911.50

density of asphalt = 1.031 gm/c.c.

TABLE VIII
THE RELATION BETWEEN THE DIMENSIONLESS GROUPS
FOR THE INDENTOR VISCOMETER
(Specimen 2)

Seq. No.	Temp. °F	Vel.x10 ³ cm/sec	$\frac{F}{\rho V^2 d^2} \times 10^{-7}$	$\frac{\mu}{\rho V d} \times 10^{-5}$
9	89.75	21.6	3.948	47.22
13	100.00	57.5	0.557	4.784
6	85.10	10.9	15.54	182.20
1	80.1	5.31	65.34	780.60
11	95.2	20.8	4.358	24.70
4	85.1	7.0	37.60	284.00
2	80.05	4.01	114.60	1043.00
10	95.05	21.8	3.87	24.10
15	100.0	32.7	1.72	84.10
7	90.2	15.2	7.97	63.46
3	80.0	4.89	77.04	859.30
12	95.0	25.8	2.77	20.36
5	85.2	8.41	26.05	231.70
14	99.9	44.7	0.922	6.196
8	90.25	15.2	7.974	62.83

TABLE IX
MEASUREMENT OF VISCOSITY OF 40-50 P.N.
ASPHALT BY THE INDENTOR VISCOMETER

Seq. No.	Temp. °F	Vel. $\times 10^3$ cm/sec	$\frac{F}{\rho V^2 d^2} \times 10^{-7}$	$\mu \times 10^{-5}$ poises
9	90.0	6.15	48.01	18.03
11	95.0	9.83	19.06	8.234
13	100.0	14.30	9.01	4.118
8	90.0	4.25	102.00	14.68
15	100.0	12.2	12.38	4.79
7	90.0	3.73	132.40	17.18
12	94.9	7.21	35.44	8.493
14	99.8	12.2	12.38	4.79

A STUDY OF AN INDENTOR VISCOMETER

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AN ABSTRACT OF A MASTER'S THESIS

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1970

ABSTRACT

The measurement of the coefficient of viscosity of a fluid is of great importance for both science and industry. A very limited number of viscometers which can measure the viscosity coefficients of highly viscous fluids in the range from 10^5 up to 10^{10} poises are available. The indenter viscometer, described in this study, was designed and fabricated at Kansas State University to be used to measure the coefficient of viscosity of highly viscous fluids.

Asphalt, obtained from Phillips Petroleum Company, was used as a fluid whose viscosity was to be measured. The Newtonian behaviour of the asphalt was verified using the well-known falling co-axial cylinder viscometer. The viscosity of asphalt was also measured by the same viscometer at five different temperatures ranging from 80 to 100 °F and the results showed that the viscosity of asphalt was greatly affected by a slight temperature variation.

A dimensional analysis of the indenter viscometer showed that for the same indenter and container diameter and the same height of fluid in container, the dimensionless group representing the resistance offered by the fluid to the indenter is a function of Reynold's number based on the indenter diameter.

The experiments carried out using the indenter viscometer as a measuring device and asphalt as a fluid showed that the functional relation between the two dimensionless groups mentioned above is a linear one when plotted on a logarithmic graph paper. The slope of this line, which is the exponent of the reciprocal of Reynold's number, was found to be 0.957, while the constant of the functional relation was found to depend on the conditions of cooling of the

surface of asphalt. The asphalt was noticed to form a sort of stretched membrane surface as a result of the repeated process of heating and cooling of asphalt after each experiment. Such a surface caused the indenter not to penetrate through asphalt and just made an imprint on the surface of asphalt.

Also the results obtained from the indenter viscometer showed good agreement with those obtained by the falling co-axial cylinder viscometer.