CHAPTER III

RHEOLOGICAL TECHNIQUES - PAST & PRESENT

A. INSTRUMENTS AVAILABLE AND THEIR LIMITATIONS

Efflux Type

The principle of operation is that the time of flow for a given quantity of fluid through an opening is approximately proportional to the viscosity. The instruments included in this type are Engler, Redwood, and Saybolt viscometers. These, however, cannot be used for measurement of viscosity above 1000 centistokes (50).

A-1 Capillary Tube

These instruments include the gravity flow type and the vacuum capillary type of glass viscometers discussed in detail in article 1-C-2. In addition, the Franklin Institute capillary viscometer is suitable for asphalt at high temperatures as the sample is totally enclosed in nitrogen atmosphere to minimize the evaporation losses and oxidation (1). Cannon-Manning pressure viscometers are also available for testing at high shear rate upto 26,000 reciprocal seconds.
A-2 Rotational Viscometers

These comprise the Stormer type, the Rotary type and the Brookfield type (1). In the Stormer viscometer the time of rotation of bob under various loads is determined. It has limited application. The Franklin Institute Rotary viscometer is useful to determine the rheological properties of asphalt over a wider range of shear rates, shear stresses and temperature. The Brookfield viscometer is a valuable tool for fairly rapid routine control for viscosity specification monitoring, but it requires 1000 ml. sample size, continuous stirring for uniform temperature, and preheating of spindles, etc. (50).

A-3 Falling Coaxial Cylinder

Pochettino, (23,(50) in Italy devided this instrument for asphalts. It is inexpensive, easy to operate, and has been used for the rapid determination of viscosities upto hundreds of millions of poises. It is also useful for the evaluation of thixotropy and elasticity of asphalts (51).

A-4 Rotational Coaxial Cylinder

This has been exceptionally useful for
measuring the consistency of asphalts in absolute and evaluating the amount of non-Newtonian flow, elasticity, thixotropy and age-hardening possessed by them. But, the most complex part of the apparatus is the equipment necessary for measuring and recording the torque applied to the stationary element.

A-5 Cone Plate Viscometer

This is now being marketed by the Cannon Instrument Co., U.S.A., for viscosity measurement at 60°F and 39.2°F. It is a very useful tool to study the low temperature viscosity of asphalt cements at shear rates from $10^{-3}$ to $10^{-2}$ sec.$^{-1}$ and requires a very small sample of asphalt for testing. The instrument is operated to minimize thixotropic, elastic and normal stress effects. Two methods of treating the data are attractive. One method requires shifting viscosities determined at various shear rates and temperature to yield a master curve at some reference temperature; the other simply displays the data on a plot of viscosity against temperature as a family of curves, one for each shear rate.
A-6 Sonic and Vibrational Methods

Goetz (50) believes that the Sonic method for determining the modulus of elasticity has a potential value as non-destructive test to follow changes caused by laboratory accelerated weathering. Visco-elastic parameters of small examples of asphalts under vibration can be obtained by Bendix ultraviscosion and Dynatrol viscometer. Magnetic driver, amplifier, and electronic computer are needed for continuous viscosity monitoring.

A-7 Viscosity Measurement from Glass Transition Temperature

These are the temperatures at which flow regimes of polymers and visco-elastic bodies are greatly altered. One of such characteristic temperatures is the glass transition temperature below which visco-elastic substances exhibit glassy, brittle behaviour. The great reduction in the mobility of asphalts below this temperature results in excessive brittleness which may be a critical factor in reducing the durability of asphaltic concrete pavement. The theoretical significance of glass-transition temperature and its relation to temperature flow function needs to be studied (51).
The W.L.F. equation can be used to reduce viscoelastic measurements to a common temperature. The dilatometrically measured glass-transition temperature is an important parameter for asphalts. The temperature-time dependence of asphalt retardation times obeys W.L.F. equation. The glass transition point is the temperature at which a substance shows a marked change in its physical, mechanical and optical properties. One concept of the glass transition temperature is that it is the temperature below which there is insufficient space between the molecules for them to rotate or move except for in-place material to flow, and the elastic modulus is very high. Below this temperature, the substance is characterised mainly by dimensional stability and a tendency to be relatively brittle. Above this temperature creep and rubber-like properties make their appearance. This temperature is the dividing line between plastic and rubber-like properties. The temperature is determined by knowing a sudden change in any of the properties, such as specific volume, specific heat, viscosity, thermal expansion, differential pressure, differential thermal analysis, static dielectric constant, dynamic mechanical properties, beta ray absorption, X-ray diffraction, linear
thermal expansion, nuclear magnetic resonance, and penetration. The penetrometer method is inexpensive apparatus for this purpose (51). The curve of temperature (-20°F to 30°F) versus penetration (0 to 0.03 inches) consists of three portions. The intermediate region is a transition region. The point of intersection of upper and lower portion gives the glass transition temperature. This temperature may increase with the asphaltene content for certain types of asphalts. The difference between the standard reference temperature and the glass transition temperature is very close to 50°C for most asphalts tested. The method of reduced variables needs to be applied to viscosity-rate of shear data for asphalt cements having different physical and chemical properties, to obtain shift factors and composite master curves. Expansion coefficients of asphalts in the glassy range vary from 3.1 - 3.95 x 10^-4 and 6.5 - 6.95 x 10^-4°C^-1 in the fluid range. These values are similar to those found for amorphous polymers. Viscosities calculated from the T_g compare well with experimentally determined viscosities as do experimentally determined values obtained with different type of viscometers (52). The form of W.L.F. equation used is:
\[
\log \eta_{T_1} = \log \eta_{T_2} + \frac{8.86 (T_2 - T_g)}{101.6 + (T_2 - T_s)} - \frac{8.86 (T_1 - T_g)}{101.6 + (T_1 - T_s)}
\]

where \( \eta_{T_1} = \) viscosity in poises at temperature \( T_1 \) in °K.,

\( \eta_{T_2} = \) viscosity in poises at temperature \( T_2 \) in °K,

\( T_s - T_g = C \) in °C.,

where \( T_g = \) glass transition temperature in °K

\( C = \) constant characteristic of each compound.

\( T_s \) is assumed to be temperature at which a given polymer has the same fractional volume as polyisobutylene at 243°K.

The best fit of asphalt viscosities calculated by the W.L.F. equation with experimentally determined viscosities gives values for \( T_s - T_g \) in W.L.F. equation at 60.7°C for Newtonian Californian asphalts, 64.2°C for non-Newtonian Venezuelan asphalts, and 61.9°C for Waxy Canadian asphalts (52). Calculated and experimentally determined low temperature viscosities agree as well as two viscosities obtained experimentally using different types of viscometers.
As repeated by Brodnyan, the dependance of storage and loss moduli on frequency and also the temperature dependance of the asphalts shows that asphalts behave very much like concentrated solutions of high polymers, that is typical visco-elastic bodies. Hence, it has been possible to incorporate much of the work, both theoretical and experimental, in high polymer field into the framework of an asphalt technology (53).

B. EQUIPMENT AND TECHNIQUES USED IN THE RESEARCH PROJECT

B-1 VACUUM CAPILLARY & GRAVITY FLOW VISCOMETERS

In the present investigation, viscosity of asphalt cements was measured by calibrated (i) Cannon vacuum capillary tubes at 30 cm Hg vacuum at 140°F, and (ii) Cannon-Fenske gravity flow viscometers at 275°F. The viscosity of liquid asphalts at 140°F was measured by Cannon vacuum capillary, Zeitfuchs crossarm and Cannon-Fenske reverse flow viscometers. The detailed description of these instruments is presented in article C-2 of chapter I. Figures 1-2, 1-3, 1-4 may also be referred.
B-2 EQUIPMENT FOR TEMPERATURES AND VACUUM CONTROL

B-2-1 CONSTANT TEMPERATURE BATH - MODEL H-1

This instrument was obtained from the Cannon Instrument Company, State College, Pennsylvania (54). The model (Figure 3-1) is designed for ± 0.01°C control of temperature from 68°F to 400°F. Its salient features are as follows:

(i) Control: The temperature is automatically controlled by a mercury glass thermo-regulator with electronic relay which is set to control temperature simply by turning the magnetic cap until the proper position is indicated on a built-in temperature scale. The bath has three thermo-regulators with temperature range of 0°C to 100°C, 100°C to 160°C, and 150°C to 210°C, providing greater sensitivity over the entire operating range of the bath. The circuit operates at only 5 microammperes at 10 volts. Load circuit may be up to 550 watts.

(ii) Capacity: The bath accommodates seven capillary viscometers. The front half of the top cover can be removed to install larger items. Approximately five gallons are required.
Figure 3-1
Constant Temperature Bath with Viscometer and Vacuum Control
(iii) Construction: The bath is contained in a 12-inch diameter by 13\(\frac{1}{2}\) inch high pyrex jar, which is surrounded by a stainless steel reflector, several inches of insulation and a heavy gauge stainless steel cabinet. The heaters, baffle, stirrer, cooling unit and control panel are stainless steel. The unit is quite rugged and corrosion resistant.

(iv) Stirring: This is done by a motor driven impeller located behind a stainless steel baffle which provides a uniform background for viewing instruments. The motor had a down draft cooling to prevent overheating at the high temperatures.

(v) Safety: The bath is viewed through a 1/4 inch safety plate glass window in the front panel and pyrex glass heat shield located between the jar and fluorescent light. The lower part of the stainless cabinet is leak-tight so that in the event of accidental breakage of the jar, all of the bath contents will be contained. Because of grounded electrical circuits, accidental burns are not obtained even at the highest operating temperatures.

The overall cabinet dimensions are 19\(\frac{1}{2}\) inches
wide by 27$\frac{1}{2}$ inches high by 18$\frac{3}{4}$ inches deep.

**Operation of Bath**

The assembly is ready for operation after (a) the motor stirrer is installed in position and the motor plugged into the grounded receptacle on the rear of the bath housing, (b) the thermo-regulator is inserted in the holder and the unit plugged into the receptacle on the rear of the cabinet, and (c) the bath is filled with appropriate fluid such that the heaters are completely immersed in it. Generally, the jar is filled with paraffin oil to a level two inches from the top for operating at 275$^\circ$F or below. The oil supplied with the bath was specially refined with oxidation inhibitor added to reduce the tendency of the oil to darken at high temperature. Silicon fluids are useful for operation beyond 300$^\circ$F. Since silicons are very difficult to remove from capillary, care has to be taken to avoid contamination of the inside of the viscometer with the bath fluid.

The thermo-regulator is set approximately to the control temperature and readjusted when bath reaches the desire temperature for exact control.
All switches are turned on and the variac set at the recommended position for desired control temperature. After a few seconds, the neon light would indicate the control circuit is functioning and that the temperature has started to increase. When the neon light is off, the control heater is off. The rate of heating should be such that the desired temperature be obtained within 30 to 45 minutes. For temperatures below 200°F, water is used for the bath fluid, because of good visibility.

With the control circuit on, the 700 watts preheat heater is automatically thrown on and off when the bath reaches control temperature and a crude control results. It thus eliminates the danger of overshooting if the bath is unattended. For faster heat-up, the variac was turned to position 11, but the bath was watched to avoid overshooting of the control point.

A calibrated thermometer with small scale divisions (A.S.T.M. specifications E-1) was inserted and the thermo-regulator set until thermal stability was obtained. The best control was obtained by setting the variac such that the heat was on for a
short interval of 10 to 20 seconds, and off for a longer period. The plastic covers were placed in the holes not occupied by viscometer tubes to reduce heat loss and prevent dirt from entering the jar.

B-2-2 VACUUM CONTROLLER

Operation: For operating this unit, it is required to set the manometer scale so that zero on the scale is at the top of the mercury meniscus (Figure 3-1). The movable electric probe is set in the manometer to the pressure desired. The automatic control switch is turned on and the hose end at the base of the surge tank is connected to the viscometers, i.e., the vacuum manifold on the bath. The other hose end is connected to the permanent vacuum line in the building. The pressure rate adjust valve is then opened and closed when the desired pressure is reached. It is then opened just a crack so that it simply serves as a throttle to prevent over movement of mercury. The pressure release valve is normally kept closed. The mercury in the manometer makes and breaks an electronic relay circuit which operates the solenoid bleed valve in the vacuum line. A ballast tank in the line smooths out the fluctuations so that the only noticeable
movement in the mercury column is a slight dimpling at the contact surface. The vacuum control range is from 0 to 50 cm. of mercury above the surroundings.

The vacuum manifold containing 3 to 6 toggle valves, is mounted on the bath lid and simplifies vacuum viscosity measurements by providing a rapid connection between the viscometer and the vacuum controller. The viscometer is connected to the manifold after it has been inserted with the bath. The run is initiated with the flip of the toggle valve, and terminated by returning the valve to the initial position. The toggle action of the valve permits rapid connection of viscometer to controlled pressure source, or vent to room.

The Viscometer holders used were made of neoprene rubber drilled with two holes to accommodate the viscometer tubes, and split from one edge through the small hole to the large hole so that the holder opens like a clamp hinging on the uncut rubber between the large hole and adjacent edge.

B-2-3
BLUE-M-CONSTANT TEMPERATURE WATER BATH

The water bath used with micro-viscometer was purchased from the Blue-M Electric Company, Blue Island,
Illinois. The electrical rating of the unit is 1600 watts, 115V single phase 50-60 cycles. Operating temperature is from room to 100°C. Proper thermometer needs to be clipped to the side of the bath. The bath needs to be filled with distilled water completely up to 10 litres capacity, and to immerse the sample in the bath to a depth of not less than 10 cm. The approximate size of bath was 18 x 12 x 12 inches. The dual microtrol and temperature selector switch was set for selected temperatures, by either setting at high or low position. The microtrol knob for temperature control should always be in a vertical position. When temperature reaches the selected value, the appropriate knob is adjusted until the heater pilot light goes out. The bath is allowed to operate for sometime to stabilize. The exclusive features of the bath are efficient agitation, automatic control, self time electromagnet below bottom magnetic wall which periodically pulsates the perforated agitator plate through up and down movement. The D.C. electromagnet energized end and de-energized by means of quality D.C. "packaged power supply" consists of silicon rectifier, capacitor, and timing relay. High quality alloy leaf springs attached to agitator plate allow return when magnet is de-energized. Multi-
perforations in agitator plate provide complete, gentle agitating and circulating action throughout the entire liquid mass irrespective of how heavily the bath is loaded with test specimens. The sectional view is shown in Figure 3-2. Bath is constructed of Type 304 stainless, 18 per cent chromium and 8 per cent nickel. The temperature can be controlled up to ± 0.1°C.

B-2.4 LIMITATIONS

(1) The bath model H-1 is designed for ± 0.01°C control of temperature, however, the A.S.T.M. 47°F and 110 F (specifications E-1) thermometers have graduations to read up to 0.2°F and approximately up to 0.1°F only.

(2) The toggle valves on the vacuum manifold were quite leak proof, but once during cleaning of viscometer tubes with the help of vacuum, the solvent entered the rubber gasket on the seat of the valve and damaged it. After this happened, some valves did not function without leaking of air.

(3) The mercury in the manometer of the vacuum controller did not fall to the zero level many times after the vacuum was destroyed, indicating that some
SECTIONAL VIEW OF THE MAGNI-WHirl
CONSTANT TEMPERATURE BATH

FIG. 3.2
air must be trapped in the system preventing the downward movement of the mercury.

(4) It was noticed that when the preheater is on and the variac is on full resistance, the constant temperature bath should not be left unattended for longer than the required time, determined from experience.

(5) When there is even a slight leakage in the vacuum system, the mercury level in the manometer keeps fluctuating spontaneously, indicating unbalanced relay control. The vacuum tolerance of 0.05 cm. is no longer possible then.

B-3 TEST PROCEDURES FOR VISCOSITY DETERMINATION AT 140°F

The method covered the test procedures (17) for the determination of the kinematic viscosity of cut back asphalts, as suggested under A.S.T.M. designations D2170, 2171-63 T.

The apparatus used in the investigation consisted of (a) Cannon-Fenske, Zeitfuchs crossarm and Cannon-Manning vacuum viscometers; (b) A.S.T.M. kinematic viscosity thermometers having range of 134 to 145°F,
as prescribed in A.S.T.M. specifications E-1. The thermometers are standardized at total immersion, (c) Model H-1 temperature bath as supplied by the Cannon Instrument Co. (precision ± 0.01°C), vacuum control unit (precision ± 0.05 cm.), and (d) timers, as described earlier.

The sample was prepared in such a manner that loss of volatile constituents was minimized. Twenty ml. of samples were poured in a number of dry containers and sealed with air tight closure. The samples of grade RC-3000 were heated in the sealed container in an oven at 145°F for 15 minutes.

The procedure of test varied with the type of instrument used for the study. The bath was maintained at ± .02°F for the test temperature of 140°F. The viscometer sizes were generally so selected that the efflux or flow time of 60 to 400 seconds was obtained. The Cannon-Fenske viscometer was charged by inverting the clean viscometer with the smaller tube submerged in the prepared sample, and applying suction to larger tube to draw the sample in the bulb up to the fill line. Other viscometers were charged by pouring the samples directly up to the fill lines. A period of 15 minutes
was allowed to reach temperature equilibrium for all viscometers. While pouring samples, care was taken to avoid the trapping of air bubbles. The flow of asphalt was started by taking out the rubber stopper in case of Cannon-Fenske viscometer; by applying slight suction to the smaller arm in case of Zeitfuchs crossarm viscometer; by applying 30 cm. Hg vacuum in case of vacuum capillary tubes. The time required to flow between the timing marks was measured to within 0.1 second.

Upon completion of the test, the viscometers were cleaned thoroughly with the help of trichloroethylene and the solvent was finally removed by rinsing with acetone. The tubes were dried by passing a slow stream of filtered dry air through the capillary for two minutes. After 10 observations, each tube was cleaned with chromic acid to remove organic deposits if any. The cleaning of Zeitfuchs crossarm tubes was accomplished when the tubes were held in the bath to minimise the chances of breakage. Other viscometers were held inverted in the ovens for one hour for cleaning. It is felt, however, that Cannon-Fenske and vacuum capillary tubes could also be cleaned with the help of vacuum while they are suspended in the bath, with proper care.
B-4 SLIDING PLATE MICRO-VISCOMETER

The viscosities of asphalt cements at 77°F and 39.2°F were measured by the sliding plate microviscometer purchased from the Hallikainen Instrument Co., California. The instrument requires a very small asphalt-cement sample and operates on the same basic principle as that of Labout and Van Oort's design. In recent years it has been used extensively.

B-5 PRINCIPLES OF MICROVISCOMETRY

Viscosity is a proportionality constant that relates the shear stress to the shear rate. Shear rate, often called the velocity gradient, is the relative change in the velocity of one plane with respect to the distance between it and a datum plane. In the sliding plate microviscometer the velocity of movement for a known thickness of asphalt between two plates is measured when a known force is applied. Shear rate increases faster than shear stress for non-Newtonian asphalts. The relation, \( M = \frac{F}{S^c} \) is well known, where \( F \) is the shearing stress in dynes/cm², \( S \) is the shear rate in seconds⁻¹, and \( M \) is the value of \( F \) when \( S = 1 \), and \( c \) is the coefficient of complex flow. \( C \) gives the
comparison of the degree of non-Newtonian flow of different asphalts. For Newtonian asphalts, $c = 1$. Asphalts with value of $c$ less than one, have low ductility. Minniford described a phenomena called "retarded elasticity" in which there is an apparent viscosity increase at low shear rates followed by a decrease at high shear rates. It is necessary to calculate the viscosity after this initial movement when the rate of shear at a given shear stress is constant. The phenomena known as 'yield point' causes the plot of shear stress–shear rate to deviate from the origin. The correction is significant at viscosity less than about 1000 poises. The viscometer has a working range of $10^2$ to $10^{11}$ poises.

B-6 TESTING PROCEDURE AND OPERATION

B-6-1 Viscosity Plates

These are pyrex polished glass plates 30 x 20 x 6 mm purchased from Corning Glass Company. These dimensions are critical, for they determine the area of the film, that is to be sheared. The inner edges next to the sample must be sharp and free of chips.

B-6-2 Film-forming Procedure

The asphalt film is formed by pressing heated
asphalt drop between heated plates with a film-forming
device. The thickness of film selected was close to 100
microns at 77°F and 300 microns at 39.2°F. Glass plates
are cleaned with benzene. The weight of asphalt film is
determined in grams. The specific gravity is generally
assumed to be unity and the area of film is 6 sq.cm.
The film thickness is equal to weight of asphalt divided
by the product of density of asphalt and area of film.
A spacer device was effective in making films of selected
thickness. 0.06 grams of asphalt was needed for 100
micron film. The viscosity plates were accurately aligned
after cleaning. They were cooled for one hour to 77°F
temperature before testing. Extreme care was taken to
see that no air bubbles were present in the film.

B-6-3 MICROVISCOMETER

This instrument was purchased from Hallikainen
Instrument Company. It is illustrated in Figure 3-3. The
displacement of the sliding plate is followed by resistance
contact servo system. The servomotor drives an insulated
metric micrometer and causes it to maintain a high
resistance contact between the point on the micrometer
and the flag attached to the plate holder as shown in
Figure 3-4. The plates with the sample are clamped in
Figure 3-3
Sliding Plate Microviscometer
SCHEMATIC DIAGRAM OF SLIDING PLATE MICROVISCOMETER

FIG. 3-4
the microviscometer by the spring-loaded clamps. The time allowed for temperature equilibrium was nearly two minutes. The point of the micrometer is driven downward until it makes contact with the flag. Then a load is applied by placing weights on the weight hanger. The subsequent upward movement of the sliding plate moves the flag upward an equal amount and decreases the resistance between the flag and the micrometer. The servomotor then drives the micrometer upward in order to increase the resistance and maintain a constant contact pressure. A more detailed description of operation of the instrument is given in the instrument manual (55).

B-6-4 Recorder

The recording instrument used is a Varian G-10 Graphic Recorder (56). The voltage from a single-turn potentiometer mounted on the microviscometer shaft, is fed to the recorder. There are 100 divisions on the recording chart. Each division corresponds to a 5-micron movement of the sliding plate. Using the range adjustment on the recorder, this 5-micron movement is calibrated. This has to be done once a week during the operation. The movement of the sliding plate is magnified 250 times on the recorder graph. This
magnification permits viscosity runs at four different shear rates with less than 0.5 mm movement of the plate. To determine viscosity at 0.05 seconds\(^{-1}\) shear rate at 77°F, it is necessary to test the sample at several different convenient loads (57). From graph of log shear stress and log shear rate, the viscosity at 0.05 reciprocal seconds shear rate is obtained by interpolation or extrapolation. Each load is continued to be applied until the recorder pen traces 20 divisions, i.e. 0.1 mm total deformation. Similar procedure is repeated for five different suitable loads.

### B-7 COMPUTATION OF VISCOSITY

The following equations or nomograms (supplied by manufacturers) may be used:

\[
\text{Viscosity} = \frac{\text{shear stress}}{\text{shear rate}} = \frac{S_0}{\frac{dv}{dr}}
\]

\[
S_0 = \text{shear stress} = \left(\text{Load in grams} \times 980\right) \frac{1}{\text{Area of plate in g cm}^2}
\]

\[
\text{Shear rate} = \frac{dv}{dr} = \frac{\text{chart divisions} \times \text{cms per division}}{\text{chart speed} \times \text{chart movement (inches)} \times \text{film thickness in microns} \times 10^{-4}}
\]

\[
= \frac{L \times C \times M}{M \times f}
\]
C = recorder calibration, cm./division = 5 x 10^-4 cm. per division.

L = Chart divisions (maximum 100).

The chart speed, N, is constant at 40 inches per hour, or 0.0111 inches per second. The chart movement is measured on the chart with scale.

B-8 SAMPLE CALCULATIONS FOR ONE RUN WITH RECORDER


85-100 penetration asphalt from identification number 6180.

Bath temperature 77°F
Recorder calibration, C = cm./div. 5 x 10^-4
Chart speed, N in inches/second = 0.0111

1. Weight of plates + sample grams = 16.0544
   Weight of plates alone grams = 15.9996
   Weight of asphalt sample grams = 0.0548

2. Film thickness, \( f = \frac{W}{A} \) =
   \( \frac{0.0548}{6} \) = microns 91.3

(density = 1 assumed)
3. Load applied to shear the sample
   \[ = P \text{ grams} = 200 \]

4. Chart movement, \( M \) inch
   measured as shown in Figure 3-5.

5. Shear stress, \( S = \frac{200 \times 980}{6.00} \)
   \[ = \frac{P}{A} = \frac{200 \times 980}{6.00} \]
   \[ = \text{dynes/cm}^2 = 2.266 \times 10^4 \]

6. Shear rate, \( R = \frac{40 \times 5 \times 10^{-4} \times 0.0111}{1 \times 91.3 \times 10^{-4}} \)
   \[ = \frac{L C N}{M f} = \frac{40 \times 5 \times 10^{-4} \times 0.0111}{1 \times 91.3 \times 10^{-4}} \]
   \[ = 2.431 \times 10^{-2} \]

7. Viscosity, \( \eta = \frac{3.266}{2.453} \)
   \[ = \frac{S}{R} = \frac{3.266}{2.453} \text{ poises} = 1.343 \times 10^6 \]

B-9 SOURCES OF ERROR AND LIMITATIONS

The analysis of error includes all variables pertaining to error in viscosity due to testing procedure with the microviscometer, graphic recorder, and other apparatus. To determine the contribution each variable makes to the error in viscosity, method of partial differentiation may be useful. The method defines the probable error of a function as a square root of the sum of the squares of the errors in the variables that comprise the function. The basic
SAMPLE NO.  6180
FILM THICKNESS  91.3 MICRONS
PLATE NO.  1
LOAD  200 GRAMS

FIG. 3.5
RECORD CHART
variables and factors causing error are:

(a) Load  Imbalance of microviscometer and sway of weights, friction of apparatus, calibration of weights.
(b) Area  Imperfect plates, gas voids in film, alignment of plates.
(c) Chart divisions  Limit of error of recorder.
(d) Recorder calibration  Calibration error.
(e) Chart speed  Electronic variations.
(f) Chart movement  Personal error.
(g) Weight of sample  Plate weight loss, balance error.
(h) Specific gravity  Variation of G.
(i) Temperature (Kelvin)  Water bath temperature variation.

It is necessary to assign the maximum positive, negative and probable errors in the variables. Some errors may be due to the limit of error of apparatus, others due to physical and chemical variations in the asphalt tested, and due to personal factors, such as drawing the best line on the chart for finding the chart movement.
\[ \eta = \frac{S}{R} \] is the equation of viscosity.

Hence, \[ \Delta \eta = \frac{\Delta S}{R} - \frac{S \Delta R}{R^2} \]

Now, \( S = \frac{980P}{A} \) and \( R = \frac{LCN}{M \times f} \times 10^4 \)

Hence, \( \Delta S = \frac{980}{A} \Delta P - \frac{980P}{A^2} \times \Delta A \)

\[ \Delta R = \left( \frac{CN \Delta L}{Mf} + \frac{LN \Delta C}{Mf} + \frac{LC \Delta N}{Mf} - \frac{LCN \Delta M}{M^2 f} \right) \times 10^4 \]

Now, \( f = \frac{V}{A} \times 10^4 \) and \( V = \frac{W}{G} \)

\[ \Delta f = \frac{10^4}{A} \times \Delta V - \frac{V \times 10^4}{A^2} \times \Delta Af \]

and \( \Delta V = \frac{\Delta W}{G} - \frac{W \Delta G}{G^2} \)

Errors can be substituted in the formulas derived to obtain the positive, negative and probable errors in viscosity. Errors in sample volume, film thickness, shear stress, shear rate and finally the error in viscosity can be calculated assuming...
reasonable error factors from judgment and (ii) the probable viscosity-temperature relationship. The contribution to error in viscosity of the error in the area of the sample is shown to be very significant, which may even be 80 per cent of the total error in viscosity. In correct alignment should be reduced to 0.005 cm. Griffin (55) recommends using an upper viscosity plate that has lightened to about 3.5 grams by using a thickness of about 3 mm. To reduce the error in the area of the sample tested, it is prime importance to decrease the factor error in strain during the test(58). The variation in bath temperature even of the order of 0.1°C contributes significantly to the error in viscosity determination. The use of recorder may result in 20 per cent less error compared with the error without using it. But, it saves time, simplifies computations, and gives a complete graph showing instantaneous shear rates that enables detection of any anomalous viscosity behaviour of a non-Newtonian asphalt.

B-9-1 LIMITATIONS

Considerable trouble was experienced with the use of this instrument at 39.2°F. The constant temperature bath was used with ice to maintain this temperature.
Although a separate electric stirrer was used for maintaining uniform temperature in the bath, it was required to watch the bath temperature very closely at shorter intervals of time. Use of dry ice would be a better idea to control the temperature, or Magniwhirl refrigerated baths. MR - 3262A or MR - 3210A type of Blue-M Company, would help control the temperature very precisely.

The recorder was first set at 4 inches/hour speed at 39.2°F, but it took extremely long periods of time for reasonable movement of the chart and the samples needed loads beyond 10,000 grams for shear rates up to 0.01 reciprocal seconds. Hence, the recorder was operated at 40 inches/hour speed. But this facilitated measurements to be made at shear rates only between 0.003 to 0.007 seconds$^{-1}$ for loads from 5000 to 10,000 grams for 85-100 as well as 120-150 penetration asphalts for film thickness close to 300 microns. It was necessary to extrapolate the results of viscosity for $\phi 0.001$ and 0.01 seconds$^{-1}$ shear rate. This also resulted in higher values of yield stresses in the sample, for viscosity determination. Consequently, the repeatability worked out to be quite high, at this temperature. Sometimes the sliding plate slipped over the fixed plate at
higher loads during longer intervals of observation periods. The glass plates were subjected to heavy wear and chipping at higher loads. This caused considerable variation in film thickness of the sample during test.

B-10 MODIFICATIONS OF THE USE OF MICROVISCOMETER

B-10-1 USE OF STEEL PLATES

The microviscometer is less suitable at lower temperatures because it is not possible to achieve high shear rates and short test times with the films and loads normally used. To lower the undesirably high resistance to shear, test plates of ground stainless steel are now proposed to reduce the width of the film while maintaining the other dimensions. The revised plates fit the standard instrument, permit the same displacement, and give the desired shear rates without excessive loads. They also offer the further advantages of improved durability compared with glass plates and the possibility of using spacers to insure a film of chosen thickness. A controlled film thickness in turn makes it unnecessary to weigh the test film and possible to plot viscosity versus shear rate directly from the load and the angle of the pen trace on the recorder chart. More precision
and quick operations are possible with this modification. A second modification is replacement of the usual chain suspension by a phosphor bronze ribbon (59). Unexpectedly large deviations can occur, due to deformations of the chain during the experiments. Repeatability as poor as 30-40 per cent may result due to this cause.

The modified steel plates have reduced width but have the same length of 3 cm, so that the maximum allowable displacement could remain unchanged. Room temperature affects the measured viscosity, due to the formation of structure during one hour cooling period.

The steel plates give superior repeatability for both viscosity as well as shear susceptibility. It is also interesting to note that as a result of the compression of the higher ranges of viscosity in the Walther charts, a given error causes a smaller shift in the line at high viscosity than at a lower level. The increase in the log-log function as a result of a 10 per cent error in these measurements is 0.16 per cent at 39.2°F and 0.24 per cent at 77°F. In the present investigation, glass plates were used owing to the difficulty of procuring the precision steel plates in time.
An asphalt film of known thickness may be formed between matched pairs of aluminium or glass plates. One of the plates is displaced at constant pre-selected velocities and the constant shearing force is measured. The shear rates obtained vary from $1$ to $10^{-4}$ seconds$^{-1}$ and viscosities from $10^5$ to $10^{10}$ poises (60). Loading apparatus may be any testing machine of the screw or hydraulic type capable of producing vertical displacements in the test specimen at accuracy of within ± per cent of the rates and loads desired. Maximum load is recorded for each deformation rate used. The total deformation should not exceed 0.6 mm.