Sliding Plate Micro-viscometer Test Sliding Plate Micro-viscometer Test

Lab Test Reference : 104
British Standard Reference : ASTM ANSI D 3570-77

Principal Apparatus

i. Viscosity Test Plates
ii. Film Forming Device
iii. Water Bath
iv. Temperature Control Unit
v. Milli-volt Recorder
vi. Charts for Recorder
vii. Calibrated Timer
ix. Film Forming Spacers
x. Film Thickness Feeler Gauge (102)um

1. General Description of Test Procedure

1.1 The viscosities of Asphaltic types of materials are measured in absolute units with the Seta Sliding Plate Microviscometer.

1.2 The working range is from 10^2 - 10^{11} poise and the principle of operation is used on the classical concept of viscosity ie shearing the sample between two parallel flat plates.

1.3 Since constant or variable rate of shear may be accurately determined, the instrument is equally applicable to materials having Newtonian or Non-Newtonian flow properties.

1.4 The Microviscometer is designed for operation in a constant temperature water bath but may also be used in air if desired.

1.5 An electrically conductive bath liquid is required to complete the circuit of the servo system, which is used to measure the displacement of the plate during the viscosity determination, but if no liquid is to be used, it will be necessary to attach a fine wire between the viscometer frame and the vertical pulling wire. Ordinary tape water is suitable for the former application.

1.6 The movement of the sample plate during a viscosity determination is followed by a simple resistance contact servo system, housed in the top compartment.

1.7 The reversible servo motor rotates an insulated metric micrometer, which maintains a high resistance contact (about 0.5 megohm) between itself and the silver plated contact fastened to the pulling wire of the sample holder. The electrical circuit is completed through the water bath.

1.8 The application of a weight to the balance arm causes an upward movement of the sample plate and its holder, which presses the metal contact plate against the micrometer point.

1.9 The resulting decrease in resistance is enough to momentarily energise the servo motor and the micrometer is backed off 1-2 microns.

1.10 Constant contact pressure between the micrometer point and the contact plate is, therefore, maintained.

1.11 The variation of shear stress due to the effect of this contact pressure in the servo mechanism is immeasurable over the range of viscosities covered.

2. Assembly of Instrument

2.1 The apparatus should be carefully cleaned with a soft cloth or paper tissue, giving particular attention to the balance knife edges and to the agate "V" blocks cemented to the viscometer shelf.

2.2 The viscometer should be assembled inserting the front end of the balance beam marked "F" (see Fig 2) through the back of the viscometer, so that the centre knife edge rests on the agate blocks on the viscometer shelf.

2.3 Great care should be taken to prevent damage to the knife edges.

2.4 Carefully place the weight hangers on the end of the knife edges, and attach the pulling wire of the plate holder assembly by means of the hook.

2.5 No additional weights should be applied to the weight hanger until the sample has been put in place.

2.6 The viscometer has four adjustable feet for levelling and some adjustment may be made prior to the test.

3. Balance Weight

3.1 The weight carried is used to establish balance beam equilibrium by compensation for the weight of the plate holder assembly, and one average glass test plate.

3.2 The balance weight has been adjusted for balance beam equilibrium, with 10cms (12gm) of water above the viscosity plate. (The weight of the assembly will change approximately 0.02 grammes for each centimetre change in weight of water level).

3. Extreme accuracy in establishing equilibrium is necessary only when low loads are involved when measuring low viscosity materials, and such small adjustments can conveniently be made by the insertion of small weight in the cavity provided in the base of weight carrier.

4. Constant Temperature Bath

4.1 This bath is a 45 litre capacity in which the Microviscometer is stood with the water level adjusted to a level approximately 12mm above the plate assembly.

4.2 The control unit incorporates a contact thermometer, operating a small heater through a relay, which gives adequate control for all testing purposes.

4.3 If it is found that there is too much turbulence in the water bath, then the pitch of the stirring propellers may be easily reduced.

5. Operation and Controls

5.1 Three controls will be found on the front panel of the Microviscometer:-

(1) Right Hand supply switch

(2) Left Hand Mircometer Raise and Lower switch. This is used to bring the mircometer into contact with the contact plate (where the motor will automatically stop), and also for the calibration by bringing the Mircometer to the desired position.

(3) The Centre Range Control Knob which is exposed by removal of the finger tight knurled cap. By rotating the exposed shaft, the record deflection can be made to correspond to an even number of divisions on the micrometer, thus facilitating calculations. Range adjustment is described in detail under that heading.

5.2 A power supply of capacity indicated by the name plate on the instrument, should be connected and an efficient earth provided. Switch on the supply switch, and allow at least one minute for the electronic equipment to warm up.

6. To Position Mircometer

6.1 The special light action switch on the left hand side of the panel is used to bring the micrometer in contact with the contact plate, at the start of the viscosity test. The "up" position raises the Micrometer away from the contract plate, and the "down" position moves the point down towards the contact plate.

Warning: There are no warning stops on the micrometer, and care should be taken not to run off the scale.

7. Range Adjustment and Calibration of Recorder

7.1 The range adjustment in the centre of the panel is used to adjust the voltage across the single turn potentiometer, so as to calibrate the millivolt recorder, by proper range adjustment, each division on the recorder chart will correspond to five micron (0.005mm) movement of the viscosity plate. This simplifies calculation of the viscosity.

7.2 This adjustment is made as follows:-

7.3 Switch on the viscometer and recorder, and allow to warm up for approximately one minute: move the micrometer by means of the "up-down" switch until a reading of 45 divisions is obtained.

7.4 Adjust recorder zero to give a reading of five divisions of the chart.

7.5 Rotate Micrometer by means of switch until a reading of five divisions is obtained, and adjust range control until a reading of 90 divisions is obtained in the recorder.

7.6 When this adjustment is made, rotation of the micrometer through 40 division will cause the chart pen to traverse 80 divisions.

7.7 If the viscometer is run so that the Micrometer travels across the zero mark, then the recorder pen will automatically traverse the chart and commence recording at the other end.

7.8 After adjustment, repeat the procedure to check calibration.

7.9 This adjustment makes each division on the chart equal to a plate movement of (0.005mm) or 5 microns.

7.10 If it is found that the instrument cannot be calibrated with the range of adjustment on the panel, further adjustment may be made by using the "calibrate control" on the Varian recorder.

7.11 This control is adjusted with a screwdriver, and is accessible through a hole at the rear of the recorder.

7.12 This control should be checked every few days.

7.13 The reference mercury cell and mercury cell in the Microviscometer give an essentially constant voltage output throughout their rated life of 2000 hrs, but the output decreases rapidly thereafter.

7.14 It should be noted that with the recorder connected to the instrument, the mercury cells are constantly discharging, even though the mains witch is off.

For maximum life, therefore, the recorder should be disconnected from the microviscometer and mercury cell housing in the microviscometer should be unscrewed a few turns to break the contact

8. Setting the Gap on the Film Former

8.1 The film forming device shown in Fig 3 has been developed for making the 100 mircon film.

8.2 To prepare the film using the film former, the following method is used:

8.3 The two glass plates plus a 0.0004 inch (102 microns) thickness gauge are placed on block "3", then the spacer is placed over block "3" so that the spacer feet are resting on the base.

8.4 Block "1" is inserted in the spacer until it rests on the plates; then the clamp is tightened.

8.5 The asphalt film which is to be formed will be as thick as the thickness gauge used.

8.6 After the space between the blocks is set, the thickness gauge and the galss plates are removed.

9. Sample Preparation

9.1 The Test should be performed in duplicate.

9.2 To prepare a sample for making a viscosity measurement, first clean the pair of test plates with benzene, dry and weigh on an analytical balance to the nearest milligramme. Record the weight as P_w (mg).

9.3 The thickness of the Plates will be checked by using the mounted dial gauge at each corner and in the centre. Record these measurements and calculate the average thickness as P_t (um).

9.4 A drop of sample is placed on one of the plates, and both plates are then warmed, on the hot plate, sufficiently to melt the sample.

9.5 Alternatively, if desired, the sample can be melted by placing under an infra-red source for a short time. This will melt the asphalt sufficiently to allow preparation of the film and leave the glass plates cool enough for easy handling.

9.6 The temperature must now be raised to within 25^{o}C of the flash point of the binder.

9.7 The plates are then inserted between the blocks.

9.8 The weight of the spacer assembly will press out the excess binder, and when the legs come to rest on the base, a film o f the desired thickness will remain between the glass plates.

9.9 Uniformity of the thickness may be checked by viewing the film in transmitted light.

9.10 On most asphalts, the film is transparent up to 100 microns.

9.11 The thickness of the sample will be checked by using the mounted dial gauge at each corner and in the centre and the difference will not be greater than 0.0025mm.

9.12 Calculate the average thickness and record this as S_t (mg).

9.13 After collection of the film, allow the plates to cool, and scrape excess asphalt from the edge with a razor blade, and carefully clean the edges with a benzene moistened cloth.

9.14 Weigh the prepared plates on the balance to the nearest milligramme. Record the weight S_w (mg).

9.15 As the specific gravity of most asphalts is approximately one, the film thickness is centimetres may be calculated by dividing the weight of the film in grammes by 6, ie the area of the plates in square centimetres.

9.16 For materials where the gravity varies appreciably from one, the volume of the asphalt is calculated from the weight and specific gravity.

9.17 Prepared plates should be cured in air for between 50 and 70 mins before testing, but non- Newtonian asphalts tend to develop structure on standing.

9.18 The resultant change in viscosity is slight, for most paving asphalts, if prepared plates are tested within one day.

9.19 Such asphalts can be reconditioned by heating the plate above the softening point of the asphalt, for approximately 30 seconds.

9.20 Calculations:-

For film thickness < 250 um

$$T = \frac{Sw-Pw}{6 \times 10^{-4}}$$

For film thickness > 250 um

$T = S_t - P_t$ (um)

9.21 Place the prepared plates into the preheated constant temperature tank for a period of 20 min.

10. Viscometer Measurement

10.1 The chart recorder should be switched on at this stage an d should be allowed to warm up before use for about 5 min.

10.2 Check that the leads from the Microviscometer are connected to the millivolt recorder, and that the black and red plugs are inserted in the appropriate sockets.

10.3 The cooled plates are now clamped in the viscometer, which should previously have been allowed to come to temperature in the constant temperature bath.

10.4 The plates are clamped into position as follows:-

10.5 Remove the sliding plate holder assembly from the water by the rod attached to the top of the holder.

10.6 Open the sliding plate holder by squeezing the assembly in a direction parallel to the rod, so that these springs are compressed.

10.7 Insert the assembled test plates, so that the second plate is towards the fixed plate holder on the base of the viscometer.

10.8 Open the fixed plate holder to receive the second plate, by moving the rod adjacent to the left-hand pillar outwards and away from the pillar.

10.9 Holding the sliding plate assembly by its rods, insert the second test plate into the fixed plate holder, making sure it is positioned against the shoulder of the fixed plate holder before releasing the locking rod.

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10.10 After the plates are clamped, the beam should be correctly centred, the weight hanger placed in position, and the pulling wire hook placed over the weight hanger, before attaching to the plate rod by means of the screwed coupling.

10.11 The coupling should be so adjusted that the balance beam is in a level position.

10.12 Position the contact plate in line with the micrometer, and drive the micrometer in the appropriate direction by means of the "raise/lower" switch.

10.13 When the micrometer point is driven down against the contact plate, the motor will automatically stop.

10.14 For bath temperature between 10 to 60^{o}C, testing may be commenced after approximately 5 mins.

10.15 Testing is started by the application of a weight to the carrier.

10.16 Record the loads applied as L_m (g).

10.17 It is not necessary to position the pen at the start of the test, because the instrument has been designed so that w hen the pen reaches the right hand edge of the chart, it shifts to the left hand edge and continues its trace.

10.18 When making viscosity determinations with the recorder, a load is applied and allowed to remain while the pen traces approximately 100 microns.

10.19 Additional weights may then be removed or added, and a determination made at different shear rates.

10.20 The chart may be conveniently used for making notes eg weights applied.

10.21 After measurement at three to five shear rates the chart may be removed, and a straight line drawn through the trace of each of the separate loads, and produced to establish the necessary data for calculation of the viscosity.

10.22 The viscosity should be determined for three or more shear rates, as most asphalts exhibit some non-Newtonian characteristics.

10.23 Inspection of the calculated viscosities will show whether the asphalt is of this type.

10.24 The viscosity at the desired shear rate may be found by interpolation from the curve obtained by plotting the log of the shear rate in reciprocal seconds against the log of the viscosity in poise.

10.25 The shear rate will depend on the load applied and some suggested loads and chart speeds for different grades of asphalts are shown on the following table:-

Penetration at 25°	50	20	300	100	50	20
Viscosity in poise at 77°F			$7*10^4$	$7.4*10^5$	$3.2*10^6$	$2.3.*10^7$
Range of Loads in Grams						
5°C			500	-	-	-
25°C			20-200	100-	500-	-
60°C			0.1-1.0	0.2-55	0.3-10	0.6-20
Chart Speed/Inches/Hour						
5°C			40	4	4	4
25°C			40	40	40	40
60 C			40	40	40	40

10.26 Re-positioning of the contact point may be necessary in between determintions, at different loads, owing to the recovery of the stress system.

10.27 The Plates could be used for determination at more than one temperature if they are dried, warmed and returned to their original position.

10.28 Viscosities at different temperatures may, therefore, by determined without preparing a fresh sample. The highest temperature (60°C) should be used last as some asphalts tend to strip from the glass plates after the surface has been exposed at this temperature.

10.29 Plates should be cleaned after determination by warming, separating and wiping and dissolving the sample off with benzene.

11. Calculations

11.1 The displacement velocity is read off the chart recorder and recorded as S_C (um.s).

11.2 The viscosity of the Newtonian liquids may be calculated from the following formula

The rate of Shear is then equal to:-

Rate of Shear, Y (gamma) = S_C / T(s^{-1})

T(TAU) = Shearing Stress

$$= \frac{L_m \text{ (Load in grammes} * 98.1}{\text{Area of Plate in sq cm}} \quad \text{(Pa)}$$

N (eta) = T (tau) / Y (gamma * 10 poise

11.3 These equations apply when

Area plate = 6 sq cms

Density of Bitumen = 1.00

Chart Speed = 2.54 cm/90 secs

When the chart speed is 2.54 cm/900 secs the values for rate of shear must be divided by 10.

11.4 An estimate of the present penetration of the bitumen may be calculated from the following formula.

11.5 Where the viscosity n(eta) < $2.2 * 10^6$

Penetration = $5.305 * 10^4 * n(eta)^{-0.465}$

11.6 Where the viscosity n(eta) > $2.2 * 10^6$

Penetration = $1.69 * 10^4 * n(eta)^{-0.385}$

12. Reporting Test Results

12.1 The following information shall be included on the test report:-

1. Laboratory Sample Number
2. Advice Number
3. Test Method
4. Scheme Name
5. Location
6. Description
7. Source
8. Date Supplies
9. Date Tested
10. Method of Recovery of Binder
11. Viscosity in Poises
12. Estimated Penetration

PRIVATE MALLORY MERCURY LT ('A' BATTERIES - CAUTIONtc \l 1 "MALLORY MERCURY LT ('A' BATTERIES - CAUTION"

DO NOT HEAT THE BATTERIES

DO NOT DISPOSE OF IN FIRE ETC

APPENDIX 'A'

Calculation to determine the constant 0.667 to simplify the calculations for rate of shear

Therefore using the following equation:-

$$\frac{dv}{dr} = \frac{\text{Chart division *cms per division* chart speed in ins/sec}_4}{\text{Chart movement in inches* film thickness in microns* 10}}$$

Each division on the record chart will correspond to 5 microns.

Therefore

y division = $y * 5 * 10^{-4}$

As there are 20 divisions/inch on the chart, the following calculations gives centimetre per division

$$\frac{2.54}{20} = 0.127 \text{ cms/div}$$

As chart speed is 40"/hr, the speed in inches per second

$$= \frac{40}{3600} \text{ seconds}$$

$$\text{Speed inches per cm} = \frac{40 * 2.54}{3600}$$

$$= 0.0111 * 2.54$$

To determine weight of sample calculated at density of 1.00

= film thickness (T) * area (6) * density (1) = weight (W)

Therefore T * 6 = W Hence $T = \frac{W}{6}$

$$\text{Therefore } \frac{dv}{dr} = \frac{Y * 5 * 10^{-4} * 10^3}{Z * \frac{(0.127)}{(0.011 * 2.54)} * \frac{W}{6}}$$

$$\text{Simplifying } \frac{dv}{dr} = \frac{Y * 5 * 0.011 * 2.45 * 6 * 10^3}{Z * 0.127 * W * 10^4}$$

$$= \frac{Y * 0.667}{Z * W}$$

$$\text{Rate of Shear} = \frac{0.667}{W * \tan A} \quad \sec^{-1}$$

Maintenance

Periodic inspection of working parts of the apparatus should be made, and micrometer point and contact plate should be cleaned daily with a soft cloth.

The agate "V" blocks and knife edges should also be cleaned regularly to remove grease and dirt.

Fault Finding

A.If the micrometer does not move when contact is made with the contract plate the microviscometer is in a water bath:-

1. Check that the electrical connection to the supply is correctly made and that the supply switch is on.

2. Check the internal fan.

3. Complete the circuit using a wire contact the body of the discometer with the micrometer. If this causes the micrometer to move then there is too much resistance through the pulling assembly. Clean the length adjuster, and contact plate surface.

4. If the micrometer does not move complete the circuit above, replace the Thyratron valve 2021 or equivalent. See fig 1.

Access to this valve is obtained by lifting the cover after removing the crews from the back and sides of the housing.

B. If the micrometer moves when in contact with the plate, but the movement is not recorded when the recorder is properly connected:-

1. Ensure that the recorder is plugged in correctly to the viscometer.

2. Replace the RM3 reference mercury cell, which supplies the reference voltage through the potentiometer in the viscometer. To replace this cell, unscrew the cap on the bottom of the viscometer chassis, remove the old cell, and insert viscometer chassis, remove the old cell, and insert the new one with the small contact downwards.

3. If no movement takes place after the above check, then the recorder should be inspected. Operating instructions for the Varian C.10 are enclosed with the instrument.

C. If the apparatus operates, but the plotted or recorded results show a wavy line, then the contact point may not be properly centred, and/or the contact plate may not be level. If after checking and adjustment difficulty still persists, then the micrometer should be returned to the manufacturer for repair or replacement of the point.

D. If the knife edges on the balance beam become damaged or lose their edge, then the bead should be returned to the manufacturer for repair or replacement.