UNIVERSITY OF HAIL
Department of Civil Engineering

Asphalt Lab Manual
CE 440
Highway and Airport Materials-Lab Manual

Introduction:

Asphalt Lab: This lab serves the Elective Course- CE 440: “Highway and Airport Materials”. In this lab, students can perform experiments on asphalt including specific gravity of semi-solid bituminous material, loss on heating of oil and asphaltic compound, penetration of bituminous material, softening point of bituminous material, Flash & Fire Points of Bituminous Material (Cleveland Open Cup Method) etc.

EXP. 1: DETERMINATION OF FLAKINESS INDEX

1 GENERAL

This method is based on the classification of aggregate particles as flaky when they have a thickness (smallest dimension) of less than 0.6 of their nominal size, this size being taken as the mean of the limiting sieve apertures used for determining the size fraction in which the particle occurs. The flakiness index of an aggregate sample is found by separating the flaky particles and expressing their mass as a percentage of the mass of the sample tested. The test is not applicable to material passing a 6.30 mm BS test sieve or retained on a 63.0 mm BS test sieve.

2 APPARATUS

The following apparatus is required.

2.1 A metal thickness gauge of the pattern shown in figure 1.1 or special sieves having elongated apertures shown in figure 1.2. The width of the apertures and the thickness of the sheet used in the gauge or sieve shall be as specified in the following figures.

Dimensions are in mm (Tolerances are given on essential dimensions as shown)

Figure 1.1 Thickness gauge

Figure 1.2 Sieves having elongated apertures

2.2 BS test sieves as shown in Table 1 (see also 7.1.3.1 of BS 812 Part 1:1975).
2.3 A balance accurate to 0.5% of the mass of the test sample.

3 SAMPLE FOR TEST

The sample for this test shall be taken in accordance with clause 5 of this part of this standard. It shall comply with the appropriate minimum mass given in Table 1.1, for sieve analysis with due allowance for later rejection of the particles retained on a 63.0 mm BS test sieve and passing 6.30 mm BS test sieve. The sample shall be taken from the laboratory sample by quartering or by means of a sample divider as described in 5.2.4 of BS 812 Part 1. Before testing it shall be brought to a dry condition in accordance with 5.1.4 of BS 812 Part 1.

Table 1.1 Dimensions of thickness gauges

Aggregate size fraction		Thickness gauge Width of slot of (Average x 0.6)	Minimum mass for subdivision
BS test sieve nominal aperture size			
100% passing	100% retained		
mm	mm	mm	Kg
63.0	50.0	33.9 ±0.3	50
50.0	37.5	26.3 ±0.3	35
37.5	28.0	19.7 ±0.3	15
28.0	20.0	14.4 ±0.15	5
20.0	14.0	10.2 ±0.15	2
14.0	10.0	7.2 ±0.10	1
10.0	6.30	4.9 ±0.1	0.5

4 PROCEDURE

Carry out a sieve analysis in accordance with BS 812: section 105.1using the sieve given in Table 1.1.
Discard all aggregate retained on the 63.0 mm BS test sieve and all aggregate passing the 6.30 mm BS test sieve.
Then weigh each of the individual size fractions retained on the sieves, other than the 63 mm BS test sieve and store them in separate trays with their size marked on the trays.

NOTE: *Where the mass of any size fraction is considered to be excessive, i.e. more than the appropriate mass given in Table 1. Provided that the mass of the sub-divided fraction is not less than half the appropriate mass given in Table4.1. Under such circumstances the rest of the procedure should be suitably modified and the appropriate correction factor applied to determine the mass of flaky particles that would have been obtained had the whole of the original size fraction been gauged.*

From the sums of the masses of the fractions in the trays (M_1), calculate the individual percentage retained on each of the various sieves. Discard any fraction of which the mass is, 5% of less of mass M_1. Record the mass remaining (M_2).

Gauge each fraction by one of the following procedures:

(a) Using the gauge: select the thickness gauge appropriate to the size-fraction under test (see Table 1) and gauge each particle separately by hand, or

(b) Using the special sieves: select the special sieve appropriate for the size-fraction under test. Place the whole of the size-fraction into the sieve which shall then be shaken until the majority of flaky particles have passed through the slots. Then gauge the particles retained individually by hand.

Combine and weigh all the particles passing the gauges or special sieves (M_3).

4.5 CALCULATIONS AND REPORTING

Flakiness index = $M_3/M_2 \times 100$

The flakiness index shall be reported to the nearest whole number. The sieve analysisobtained in this test shall also be reported.

Worksheet: Determination of the Flakiness Index

Name: **Student ID**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Test Method : Bs 812 (part 1) Clause 7.3 & 7.4

Sieve size (mm)	**Gauge size used, (mm)**	**Wt. of the material retained (gm)**	**Percent of the material retained**	**Check if greater than 5% (ok/ not ok)**	**Flaky Particles (amount passed) gm**
63.0	-	x	x		
50.0	33.9				
37.5	26.3				
28.0	19.7				
20.0	14.4				
14.0	10.2				
10.0	7.2				
6.3	4.9				
		M_1 =	**M_2 =**		**M_3 =**

Calculation:

Flakiness index (F.I) = M_3/M_2 x 100% = % (to the nearest whole number)

EXP. 2: DETERMINATION OF ELONGATION INDEX

1 GENERAL

This method is based on the classification of aggregate particles as elongated when they have a length (greatest dimension) of more than 1.8 of their nominal size, this size being taken as the mean of the limiting sieve apertures used for determining the size fraction in which the particle occurs.

The elongation index of an aggregate sample is found by separating the elongated particles and expressing their mass as a percentage, of the mass of the sample tested. The test is not applicable to material passing a 6.30 mm BS test sieve or retained on a 50 mm BS test sieve.

2 APPARATUS

The following apparatus is required.

2.1 A metal length gauge of the pattern shown in figure 2.1. The gauge lengths shall be those specified in the length gauge column of table 2.1.

2.2 BS test sieves as shown in Table 2.1 as appropriate (see also 7.1.3.1 of BS Part 1:1975).

2.3 A balance accurate to 0.5% of the mass of the test sample.

3 SAMPLE FOR TEST

The sample for this test shall be taken in accordance with Table 2.1. It shall comply with the appropriate minimum mass given in Table 5 of BS 812 Part 1:1975, for sieve analysis, with due allowance for later rejection of particles retained on a 50.0 mm BS test sieve and passing a 6.30 mm BS test sieve. The sample shall be taken from the laboratory sample by quartering or by means of a sample divider as described in 5.2.4 of BS 812 Part 1:1975. Before testing it shall be brought to a dry condition in accordance with 7.1.4 of BS 812 Part 1:1975.

Table 2.1 Dimensions of length gauges

Aggregate size fraction		Length gauge Gap between pins (Average x 1.8)	Minimum mass for subdivision
BS test sieve nominal aperture size			
100% passing	100% retained		
mm	Mm	Mm	Kg
63.0	50.0	-	50
50.0	37.5	78.0 ± 0.3	35
37.5	28.0	59.0 ± 0.3	15
28.0	20.0	43.2 ± 0.3	5
20.0	14.0	30.6 ± 0.3	2
14.0	10.0	21.6 ± 0.2	1
10.0	6.30	14.7 ± 0.2	0.5

4 PROCEDURE

Carry out a sieve analysis in accordance with BS 812: section 105.2using the sieves shown in Table 2.1.

Discard all aggregate retained on the 50.0 mm BS test sieve and all aggregate passing the 6.30 mm BS test sieve.

Weigh and store each of the individual size-fractions retained on the other sieves in separate trays with their size marked on the tray.

Dimensions are in mm (Tolerances are given on essential dimensions as shown)

Figure 2.1 Metal length gauge

From the sum of the masses of the fractions in the trays (M_1), calculate the individual percentages retained on each of the various sieves. Discard any fraction whose mass is 5% or less of mass M_1. Record the mass remaining (M_2).

Select the length gauge appropriate to the size-fraction under test (see Table 2.1) and gauge each particle separately by hand. Elongated particles are those whose greatest dimension prevents them from passing through the gauge. Combine and weigh all elongated particles (M_3).

5.5CALCULATIONS AND REPORTING

Elongation index = $M_3/M_2 \times 100$

The elongation index shall be reported to the nearest whole number. The sieve analysis obtained in this test shall also be reported.

Worksheet
Determination of the Elongation Index

Name : **Student ID**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Test Method : Bs 812 (part 1) Clause 7.3 & 7.4

Sieve size (mm)	**Gauge size used, (mm)**	**Wt. of the material retained (gm)**	**Percent of the material retained**	**Check if greater than 5% (ok/ not ok)**	**Elongated Particles (amount retained) gm**
50.0	-	**x**	**x**		
37.5	78.0				
28.0	59.0				
20.0	43.2				
14.0	30.6				
10.0	21.6				
6.3	14.7				
		M_1 =	**M_2 =**		**M_3 =**

Calculation:

Elongation index (E.I) = M_3/M_2 x 100% = % (to the nearest whole number)

EXP. 3 DETERMINATION OF ANGULARITY NUMBER

1 GENERAL

The angularity number is determined from the proportion of voids in a sample of aggregate after compaction in the specified manner. This property is used mainly in the design of mix proportions and in research.

Angularity or absence of rounding of the particles of an aggregate is a property which is of importance because it affects the ease of handling of a mixture of aggregate and binder (e.g. the workability of concrete) or the stability of mixtures that rely on the interlocking of the particles. The least angular (most rounded) aggregates are found to have about 33% voids and the angularity number is defined as the amount by which the percentage of voids exceeds 33. The angularity number ranges from 0 to about 12.

Since considerably more effort is used than in the test for bulk density and voids the percentage of voids will be different. Weaker aggregates may be crushed during compaction and the results will be anomalous if this method is applied to any aggregate which breaks down during the test.

2 SAMPLING

The sample for this test shall be taken in accordance with clause 5 of this part of this standard.

3 APPARATUS

The following apparatus is required:

3.1 A metal cylinder closed at one end , of about 0.003 m^3 volume, the diameter and height of which should be approximately equal (e.g. 150 mm and 150 mm).

The cylinder shall be made from metal of a thickness not less than 3 mm and shall be of sufficient rigidity to retain its shape under rough usage.

3.2 A straight metal tamping rod of circular cross section 16 mm in diameter and 600 mm long rounded at one end.

3.3 A balance or scale of capacity 10 kg, accurate to 1 g.

3.4 A metal scoop approximately 200 mm x 120 mm x. 50 mm (i.e. about 1 liter heaped capacity).

3.5 BS perforated plate test sieves from 20.0 mm to 5.0 mm aperture size (but see note to section 3.5.5(a)).

4 CALIBRATION OF THE CYLINDER

The cylinder shall be calibrated by determining to the nearest gram the mass of water at $20^0 \pm 2^0$C required to fill it up so that no meniscus is present above the rim of the container (mass C).

5 PREPARATION OF THE TEST SAMPLE

The sets sample shall be prepared as follows.

a) The amount of aggregate available shall be sufficient to provide, after separation on the appropriate pair of sieves, at least 10 kg of the predominant size as determined by sieve analysis on the BS test sieves.

The test sample shall consist of aggregate retained between the appropriate pair of BS test sieves from the following list:

20.0 mm and 14.0 mm; 14.0 mm and 10.0 mm; 10.0 mm and 6.3 mm; 6.3 mm and 5.0 mm.

Note: In testing aggregates larger than 20.0 mm the volume of the cylinder should be greater than 0.003 m^3, but for aggregates smaller than 5.0 mm a smaller cylinder may be used. The procedures should be the same as with the 0.003 m^3 cylinder, except that the amount of compactive effort (mass) should be proportioned to the volume of the cylinder used.

b) The aggregate to be tested shall be dried for at least 24 hr in shallow trays in a well ventilated oven at a temperature of 105 ± 5^0C, cooled in an air tight container and tested.

6 TEST PROCEDURE

Fill the scoop and heap it to overflowing with the aggregate, which shall be placed in the cylinder by allowing it to slide gently off the scoop from the least height possible.

Subject the aggregate in the cylinder to 100 blows of the tamping rod at a rate of about two blows per second. Apply each blow by holding the rod vertical with its rounded end 50 mmabove the surface of the aggregate and releasing it so that it falls freely. Do not apply any force to the rod. Evenly distribute the 100 blows over the surface of the aggregate.

Repeat the process of filling and tamping exactly as described above with a second and third layer of aggregate; the third layer shall contain just sufficient aggregate to fill the cylinder level with the top edge before tamping.

After the third layer of aggregate has been tamped, fill the cylinder to overflowing, and strike off the aggregate level with the top, using the tamping rod as a straight-edge.

Then add individual pieces of aggregate and roll them in, to the surface by rolling the tamping rod across the upper edge of the cylinder, and continue this finishing process as, long as the aggregate does not lift the rod off the edge of the cylinder on either side. Do not push in or otherwise force down the aggregate, and apply no downward pressure to the tamping rod, which shall roll in contact with the metal on both sides of the cylinder. Then weigh the aggregate in the cylinder to the nearest 5 g.

Make three separate determinations and calculate the mean mass of aggregate in the cylinder (mass M). If the result of any one determination differs from the mean by more than 25 g, three additional determinations shall immediately be made on the same material, and the determinations calculated (mass M).

7 CALCULATIONS

The angularity number of the aggregate shall be calculated from the equation:

Angularity number $= 67 - \frac{100\ \mathrm{M}}{\mathrm{CG}}$

Where,

M is the mean mass of aggregate in the cylinder (g);
C is the mass of water required to fill the cylinder (g);
G is the relative density on an oven-dried basis of the aggregate determined in accordance with clause 5 of part 2 of this standard.

8 REPORTING OF RESULTS

The angularity number shall be reported to the nearest whole number.

Worksheet
Determination of Angularity Number

Name : **Student ID**:

Type of material : Brick Chips/ Stone Chips/ Gravels
Test Method : Bs 812; Part 1 Clause: 7.5 & BS 812, Part 2 Clause: 5

Serial No.	Mass of Aggregate in Cylinder (gm)	Mean Mass of Aggregate in the Cylinder, (M) (gm)	Relative Density of the Aggregate (Oven dry basis) (G)	Water Required to Fill the Cylinder, (C) (gm)	Angularity Number (A.N)
1					
2					
3					

Calculation:

Angularity Number (A.N) = $67-\frac{100\ M}{CG}=$ **(to the nearest whole number)**

EXP. 4: AGGREGATE IMPACT VALUE

1 GENERAL

The aggregate impact value gives a relative measure of the resistance of an aggregate to “sudden shock or impact”, which in some aggregates differs from its resistance to a slowly applied compressive load. With aggregate of aggregate impact value (AIV) higher than 30 the result may be anomalous. Also, aggregate sizes larger than 14 mm are not appropriate to the aggregate impact test.

The standard aggregate impact test shall be made on aggregate passing a 14. mm BS test sieve and retained on a 10.0 mm BS test sieve. If required, or if the standard size is not available, smaller sizes may be tested but owing to the non-homogeneity of aggregates the results are not likely to be the same as those obtained from the standard size. In general, the smaller sizes of aggregate will give a lower impact value but the relationship between the values obtained with different sizes may vary from one aggregate to another.

2 APPARATUS

The following apparatus is required.

1.2.1 An impact testing machine of the general form and complying with the followings,

(a) Total mass not more than 60 kg or less than 45 kg.
The machine shall have a circular metal base weighing between 22 kg and 30 kg., with a plane lower surface of not less than 300 mm diameter, and shall be supported on a level and plane concrete or stone block or floor at least 450 mm thick. The machine shall be prevented from rocking either by fixing it to the block or floor or by supporting it on a level and plane metal plate cost into the surface of the block or floor.

(b) A cylinder steel cup having an internal diameter of 102 mm and an internal depth of 50 mm. The walls shall be not less than 6 mm thick and the inner surfaces shall be case hardened. The cup shall be rigidly fastened at the center of the base and be easily removed for emptying.

(c) A metal hammer weighing 13.5 kg to 14.0 kg the lower end of which shall be cylindrical in shape, 100.00 mm diameter and 50 mm long, with a 1.5 mm chamfer at the lower edge, and case hardened. The hammer shall slide freely between vertical guides so arranged that the lower (cylindrical) part of the hammer is above and concentric with the cup.

(d) Means or raising the hammer and allowing it to fall freely between the vertical guides from a height of 380±5 mm on to the test sample in the cup, and means for adjusting the height of fall within 5 mm.

Figure 4.1 Aggregate Impact Test machine

(e) Means for supporting the hammer whilst fastening or removing the cup.

NOTE: Some means for automatically recording the number of blow is desirable.

1.2.2 BS test sieves of aperture size 14.0 mm, 10.0 mm and 2.36 mm for a standard test.

2.3 A cylindrical metal measure of sufficient rigidity to retain its form under rough usage and with an internal diameter of 75 ± 1 mm and an internal depth of 50± 1 mm.

2.4 A straight metal tamping rod of circular cross section, 10 mm diameter, 230 mm long, rounded at one edge.

2.5 A balance of capacity not less than 500 gm, and accurate to 0.1 gm.

3 PREPARATION OF THE TEST SAMPLE

The material for the standard test shall consist of aggregate passing a 14.0 mm BS test sieve and retained on a 10.00 mm BS test sieve and shall be thoroughly separated on these sieves before testing. For smaller sizes the aggregate shall be prepared in a similar manner using the appropriate sieves given in Table 1. The quantity of aggregate sieved out shall be sufficient for two tests.

The aggregate shall be tested in a surface dry condition. If dried by heating, the period of drying shall not exceed 4 h, the temperature shall not exceed 110^0c and the samples shall be cooled to room temperature before testing.

The measure shall be filled about one third full with the aggregate by means of a scoop, the aggregate being discharged from a height not exceeding 50mm above the top of the container. The aggregate shall then be tamped with 25 blows of the rounded end of the tamping rod, each blows being given by allowing the tamping rod to fall freely from a height of about 50 mm above the surface of the aggregate and the blows being evenly distributed over the surface. A further similar quantity of aggregate shall be added in the same manner and a further tamping of 25 times and the surplus aggregate removed by rolling the tamping rod across, and in contact with, the top of the container, any aggregate which impedes its progress being removed by hand and aggregate being added to fill any obvious depressions. The net mass of aggregates in the measure shall be recorded (mass A) and the same mass used for the second test.

4 TEST PROCEDURE

Rest the impact machine, without wedging or packing, upon the level plate, black or floor, so that it is rigid and the hammer guide columns are vertical. Fix the cup firmly in position on the base of the machine and place the whole of the test sample in it and compact by a single tamping of 25 strokes of the taming rod as above.

Adjust the height of the hammer so that its lower face is 380± 5 mm above the upper surface of the aggregate in the cup and then allow it to fall freely on to the aggregate. Subject the test sample to a total of 15 such blows, each being delivered act an interval of not less than 1 s. No adjustment for hammer height is required after the first blow.

Then remove the crushed aggregate by holding the cup over a clean tray and hammering on the outside with a suitable rubber mallet until the sample particle s are sufficiently disturbed to enable the mass of the sample to fall freely on to the tray. Transfer fine particles adhering to the inside of the cup and the underside of the hammer to the tray by means of a stiff bristle brush. Sieve the whole of the sample in the tray, for the standard test, on the 2.36 mm BS test sieve until no further significant amount in 1 min. When testing sizes smaller than the standard separate the fines on the appropriate sieve given in the ‘for separating fines’ column in table 4.1.

Weigh the fraction passing and retained on the sieve to an accurately of 0.1 gm (mass B and mass C respectively) and if the total mass B+C is less than the initial mass (mass A) by more than 1 gm, discard the result and make afresh test.
Repeat the whole procedure starting from the beginning using a second sample of the same mass as the first sample.

Table 4.1 Particulars of BS test sieves for testing standard and non standard sizes of aggregates

Sample size	Nominal aperture sizes of BS test sieves complying with the requirements of BS410			
	for sample preparation		for separating fines	
	passing			
Non-standard	mm	mm	mm	μ
	28.0	20.0	5.00	-
	20.0	14.0	3.35	-
.Standard	14.0	10.0	2.36	-
Non-standard	10.0	6.30	1.70	-
	6.30	5.00	1.18	-
	5.00	3.35	-	85
	3.35	2.36	-	60

NOTE: Aggregate sizes larger than 14.0 mm are not appropriate to the aggregate impact test.

5 CALCULATIONS

The ratio of the mass of fines formed to the total sample mass in each test shall be expressed as a percentage, the result being recorded to the first decimal place.

Percentage fines: B/A × 100

Where,
A is the a mss of surface dry sample, gm
B is the fraction passing the sieve for separating the fines, gm

6 REPORTING OF RESULTS

The mean of the two results shall be reported to the nearest whole number as the aggregate impact value.

Determination of Aggregate Impact Value

Name : **Student ID**:

Type of material : Brick Chips/ Stone Chips/ Gravels/ Boulder/ Rock
Sample Size : 14 mm to 10 mm
Test Method : Bs 812 (part 3) 1975

Test No **Data**	**1**	**2**
Wt. of Sample (Surface Dry), A gm		
Wt. of materials retained on 2.36 mm sieve, C gm		
Wt. of materials passing 2.36 mm sieve, B gm		
Aggregate Impact Value (%) = B/A x 100% **(to the first decimal place)**		
Average Aggregate Impact Value (AIV) = **(to the nearest whole number)**		

Calculation:

EXP. 5: SPECIFIC GRAVITY OF SEMI-SOLID BITUMINOUS MATERIAL

AASHTO DESIGNATION : T 228-93

(ASTM DESIGNATION : D 70-76)

1. SCOPE

1.1 This method covers the determination of the specific gravity of semi-solid bituminous materials, asphalt cements, and soil tar pitches by use of a pycnometer.

2. SPECIFIC GRAVITY

2.1 The specific gravity of semi-solid bituminous materials, asphalt cements, and soft tar pitches shall be expressed as the ratio of the mass of a given volume of the material at 25^{0}C(77^{0}F) or at 15.6˚C (60˚F) to that of an equal volume of water at the same temperature, and shall be expressed thus:

Specific gravity, 25/25˚C (77/77^{0}F) or 15.6/15.6˚C (60/60˚F)

3. APPARATUS

3.1 Pycnometer, glass, consisting of a cylindrical or conical vessel carefully ground to receive an accurately fitting glass stopper 22 to 26 mm in diameter. The stopper shall be provided with a hole 1.0 to 2.0 mm in diameter, centrally located in reference to the vertical axis. The top surface of the stopper shall be smooth and substantially plane and the lower surface shall be concave in order to allow all air to escape through the bore. The height of the concave section shall be 4.0 to 18.0 mm at the centre. The stoppered pycnometer shall have a capacity of 24 to 30 ml, and shall weigh not more than 40 g.

3.2 Water Bath- Constant temperature, capable of maintaining the temperature within 0.1^{0}C (0.2^{0}F) of the test temperature.

3.3 Thermometers- Calibrated liquid-in-glass of suitable range with graduations at least every 0.2°F (0.1°C) and a maximum scale error of 0.2°F (0.1°C) as prescribed in ASTM specification on El. Thermometers commonly used are 63°F or 63°C. Any other thermometer of equal accuracy may be used.

NOTE-1: Other ASTM thermometers (such as the ASTM 17°C) which have sub-divisions and scale errors equal to or smaller than those specified for the ASTM 63^0C and 63^0F may also be used.
3.4 Balance - a balance conforming to the requirements of M 231, Class B.

4. MATERIALS

4.1 Distilled Water - Freshly boiled and cooled distilled water shall be used to fill the pycnometer and the beaker.
NOTE-2: For the purpose of this lest, freshly boiled and cooled distilled, aemineralized or deionized water may be used.

5. PREPARATION OF EQUIPMENT

5.1 Partially fill a 600 ml or larger Griffin low-form beaker with freshly boiled and cooled distilled water to a level that will allow the top of the pycnometer to be immersed to a depth of not less than 40 mm.

5.2 Partially immerse the beaker in the water bath to a depth sufficient to allow the bottom of the beaker to be immersed to a depth of not less than 100 mm, while the top of the beaker is above the water level of the bath. Clamp the beaker in place.

5.3 Maintain the temperature of the water bath within 0.1°C(0.2°F) of the test temperature.

6. CALIBRATION OF PYCNOMETER

6.1 Thoroughly clean, dry, and weigh the pycnometer to the nearest 1 mg. Designate this mass as "A".

6.2 Fill the pycnometer with freshly boiled distilled water at test temperature and place the stopper in the pycnometer. Do not allow any air bubbles to remain in the pycnometer.

6.3 Allow the pycnometer to remain in the water for a period of not less than 30 min. Remove the pycnometer, immediately dry the top of the stopper with one stroke of a dry towel (Note 3), then quickly dry the remaining outside area of the pycnometer and weigh to the nearest 1 mg. Designate the mass of the pycnometer plus water as "B".

Note-3: Do not re-dry the top of the stopper even if a small droplet of water forms due to expansion. If the top is dried at the instant of removing the pycnometer from the water, the proper mass of the contents at the test temperature will be recorded. If moisture condenses on the pycnometer during weighing, quickly re-dry the outside of the pycnometer (excluding the top) before recording the mass.

Note-4: Calibration should be done at the specific temperature. A pycnometer calibrated at one temperature cannot be used at a different temperature without recalibration, at that temperature.

Table 5.1: Precision of specific Gravity Data for Semi-Solid Bituminous Materials

	Deg. C (deg. F)	Single-Operator			Multi-laboratory		
		Degrees of Freedom	(IS)	(D2S)	Degrees of Freedom	(LS)	(D2S)
Asphalt	15.6(60)	54	0.0011	0.0032	24	0.0018	0.0051
	25.0(77)	54	0.00080	0.0023	24	0.0024	0.0068
Soft tar pitch	15.6(60)	72	0.0013	0.0038	27	0.0029	0.0083
	25.0(77)	72	0.00083	0.0023	27	0.0017	0.0048
Pooled	15.6(60)	114	0.0013	0.0035	51	0.0024	0.0067
	25.0(77)	114	0.00082	0.0023	51	0.0019	0.0053

7. PROCEDURE

7.1 Preparation of Sample - Heat the sample with care, stirring to prevent local overheating, until the sample has become sufficiently fluid to pour. In no case should the temperature be raised to more than 56^0C (100°F) above the expected softening point for tar, or to more than 111°C (200°F) above the expected softening point for asphalt. Do not heat for more than 30 minutes over a flame or hot plate or for more than 2 hours in an oven, and avoid incorporating air bubbles in the sample.

7.2Pour enough sample into the clean, dry, warmed pycnometer to fill it about three-fourth to its capacity. Take precautions to keep the material from touching the sides of the pycnometer above the final level, and to prevent the inclusion of air bubbles (Note 5). Allow the pycnometer and its contents to cool to ambient temperature for a period of not less than 40 minutes, and weigh with the stopper to the nearest 1 mg. Designate the mass of the pycnometer plus sample as “C”.

NOTE-5: If any air bubbles are inadvertently included, remove by brushing the surface of the asphalt in the pycnometer with a high "soft" flame of a Bunsen burner. In order to avoid overheating, do not allow the flame to remain in contact with the asphalt more than a few seconds at any one time.

7.3 Fill the pycnometer with freshly boiled distilled water at test temperature and place the stopper in the pycnometer. Do not allow any air bubbles to remain in the pycnometer.

7.4 Allow the pycnometer to remain in the water bath for a period of not less than 30 minutes. Remove the pycnometer from the bath. Dry and weigh using the same technique as that employed in Section 6.3. Designate this mass of pycnometer plus sample plus water as “D”.

8. CALCULATIONS

8.1 Calculate the specific gravity to the nearest third decimal as follows:

$$\text{Specific gravity} = \frac{(C-A)}{[(B-A)-(D-C)]}$$

Where :

A = mass of pycnometer (plus stopper)
B = mass of pycnometer filled with water
C = mass of pycnometer partially filled with asphalt, and
D = mass of pycnometer plus asphalt plus water

9. REPORT

9.1 Report the specific gravity to the nearest third decimal at 25/25°C (77°F) or $15.6/15.6^0$C (60/60°F).

Average Aggregate Impact Value (AIV):

EXP. 6: LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUND

AASHTO DESIGNATION : T 47-83 (1993)

(ASTM DESIGNATION : D 6-80)

1. SCOPE

1.1 This method covers the determination of the loss in mass (exclusive of water) of oil and asphaltic compounds when heated as herein after prescribed.

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:
E1 Specification for ASTM Thermometers
E145 Specification for gravity-convection and forced-ventilation ovens

3. APPARATUS

3.1 Oven: The oven shall be electrically heated and shall conform to the performance requirements of ASTM specification E 145, for Gravity-Convection and Forced-Ventilation Ovens. It shall comply with the following requirements.

3.1.1 Construction: The oven shall be rectangular with minimum interior dimensions of 330 mm (13 in.) in each direction. The oven shall have in front a tightly fitting hinged door,which shall provide a clear opening substantially the same as the interior height and width of the oven. The door may contain a window with dimensions of a least 100 by 100 mm (4 by 4 in.), and with two sheets of glass separated by an air space, through which a vertical thermometer located in the oven, may be read without opening the door, or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily. The oven shall be adequately ventilated by convection currents of air and for this purpose shall be provided with openings for the entrance of air and for the exit of heated air and vapors.

3.1.2 Rotating Shelf: The oven shall be provided with a circular metal shelf having a minimum diameter of 250 mm (9.8 in.). The shelf shall suspend by a vertical shaft and centered with respect to the horizontal interior dimensions. The shelf shall be provided with a mechanical means for rotating it at the rate of 5 to 6 rpm. The shelf shall be vertically located as close to the center of the oven as permitted by compliance with the requirements regarding to thermometer placement.

3.2 Thermometer: An ASTM Loss on Heating Thermometer graduated in Celsius degrees, having a range from 155 to 170°C, and conforming to the requirements for Thermometer as prescribed in the ASTM Specification E1, for ASTM Thermometers.

3.3 Container: The container in which the sample is to be tested shall be of metal or glass, cylindrical in shape, and shall have a flat bottom. It's inside diameter and depth shall be 55mm (2.17 in.) and 35 mm(1.38 in.) respectively.

4. PROCEDURE

4.1 First test the material under examination for water and if water is present, remove it by suitable methods of dehydration before subjecting the material to the loss on heating test, or obtain another sample that is free from water.

4.2 Place 50.0±0.5 gm of the sample of the water free material in a container, cool the sample to room temperature and weigh to the nearest 0.01 gm. Bring the oven to a temperatun of 163°C(325°F) and place the container with the weighed sample on recesses if the recommended shelf is used. Close the oven and rotate the shelf during the entire test at a rate of 5 to 6 rpm. Maintain the temperature at 163 ± 1°C (325±1.8°F) for 5 hrs, start counting the time when the temperature reaches 162°C, and in no case shall the total time that a sample is in the oven be more than 5h and 15 min. At the conclusion of the heating period, remove the sample from the oven, cool to room temperature, and weigh to the nearest 0.01 gm.

5. CALCULATIONS

5.1 Calculate the percentage loss to the nearest seconddecimal as follows:

$$\% \text{ loss} = [(A-B)/A'] * 100$$

Where, A= initial weight of the container plus sample
B = final weight of the container plus sample after heating
A′ = initial weight of the sample

6. PRECAUTIONS

6.1 Under ordinary circumstances a number of samples having about the same degree volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. When extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven to check the accuracy of result. Samples showing evidences of foaming during the test shall be rejected.

7. Reproducibility of Results

7.1 Up to 5 percent loss in mass the results obtained may be considered as correct within 0.5. Above 5 percent loss in mass, the numerical limit of error increases 0.01 for every 0.5 percent increase in loss by volatilization as follows (Table 6.1):

Table 6.1: Numerical limit of error for loss above 5 percent.

Volatilization Loss (%)	Numerical Correction	True Volatilization Loss, (%)
5.0	± 0.50	4.50 to 5.55
5.5	± 0.51	4.99 to 6.01
6.0	± 0.52	5.48 to 6.52
10.0	± 0.60	9.40 to 10.60
15.0	± 0.70	14.30 to 15.70
25.0	± 0.90	24.10 to 25.90
40.0	± 1.20	38.80 to 41.20

EXP 7: PENETRATION OF BITUMINOUS MATERIAL

1. SCOPE

1.1 This test method covers determination of the penetration of semi-solid and solid bituminous materials. Materials having penetrations below 350 can be tested by the standard apparatus and procedure described. Materials having penetrations between 350 and 500 can be determined using the special apparatus and modifications given in Section 9.3.

1.2 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. REFERENCED DOCUMENTS

2.1 AASHTO standards:

T 53 Softening Point of Asphalt (Bitumen) and Tar in Ethylene Glycol (Ring-and-Ball)

2.2 ASTM Standards:

C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials
E1 Specification for ASTM Thermometers
E77 Method for Verification and Calibration of Liquid in Glass Thermometers.

2.3 ANSI Standard

B 46.1 Surface Texture
IP Standard Thermometers

3. DESCRIPTION OF TERM

3.1 The penetration of a bituminous material is the distance in tenths of a millimeter that a standard needle penetrates vertically into a sample of the material under fixed conditions of temperature, load and time.

4. SUMMARY OF METHOD

4.1 The sample is melted and cooled under controlled conditions. The penetration is measured with a penetrometer by means of which a standard needle is applied to the sample under specific conditions.

5. SIGNIFICANCE AND USE

5.1 The penetration test is used as a measure of consistency. Higher values of penetration indicate softer consistency.

6. APPARATUS

6.1 Penetration Apparatus - Any apparatus permitting movements of the spindle without appreciable friction and which is accurately calibrated to yield results in accordance With the description of the term penetration (see section 3.1) will be acceptable. The surface on which the sample container rests shall be flat and the axis of the plunger shall be at approximately 90 degrees to this surface. The spindle shall be detachable without the use of special toots, for checking its mass. When the needle is mounted in a ferrule, the mass of the moving spindle shall be 47.5 ± 0.05 g. Regardless of the type of mounting of the needle, the total mass of the needle and spindle assembly shall be 50.0 ±g. Weights of 50.0 ± 0.05 g and 100.0 ± 0.05 g shall be provided for total loads of 100 g and 200 g (0.9 N and 2 N), depending upon the conditions of test to be applied.

NOTE: 1- The detachability of the spindle prescribed here is intended to apply to penetration apparatus acquired after January 1, 1976. Penetration apparatus acquired before January 1, 1976, may conform either to this standard or to the previous standard (T. 49-74).

6.2 Needle- The needle, Figure 7.1, shall be made from fully hardened and tempered stainless steel, grade 440 C or equal HRC 54 to 60. It shall be approximately 50 mm (2 in.) in length and 1.00 to 1.02 mm (0.039 to 0.040 in) in diameter. It shall be symmetrically tapered at one end to a conewhose angle shall be within the range of 8.7 to 9.7 deg over the entire length from full needle diameter, and whose axis shall be coincident with the needle axis within 0.0127 mm (0.005 in.) maximum run out (total indicator reading).

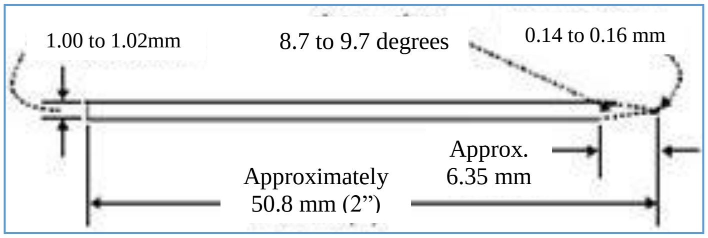

Figure 7.1: Needle for penetration test

After tapering, the point shall be ground off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm (0.0055 to 0.0063 in) in diameter. The truncation shall be square with the needle axis within 2 degree, and the edge shall be sharp and free from burrs.

6.2.1 When the surface texture of the tapered cone surface is measured in accordance with American National Standards Institute Standard B46.1, the surface roughness height shall be 0.2 to 0.3 μm (8 to 12 μ in.) arithmetic average.

6.2.2 The exposed length of the needle when mounted in the chuck of the penetration apparatus or in a ferrule shall be approximately 40 to 45 mm (1.57 to 1.77 in). When the needle is mounted in a ferrule, the ferrule shall be a cylindrical rod, 3.20 ± 0.05 mm (0.126 ± 0.002 in) in diameter and approximately 38 mm (1.5 in) long, made of stainless steel or brass, in which the needle shall be rigidly and coaxially mounted. The weight of the ferrule needle assembly shall be 2.50 ± 0.05 g. (A drill hole is permissible at the end of the ferrule to control weight). Individual identification markings shall be placed on the ferrule of each needle; the same markings shall not be repeated by a manufacturer within a 3 year period.
NOTE 2- The manufacturer or commercial laboratories will certify the test needles for conformance to the permissible variations.

6.3 Container- A container, in which the sample is tested, made of metal or glass cylindrical in shape, and having a flat bottom. The container to be used for materials having a penetration of 200 or less shall have a nominal capacity of 3 oz (90 ml). Its inside dimensions shall be essentially as follows: 55 mm (2.17 in) in diameter and 35 mm (1.38 in) in depth. The container to be used for materials having a penetration over 200 shall have a nominal capacity of 6 oz (175 ml). Its inside dimensions shall be essentially as follows: 70 mm (2.75 in) in diameter and 45 mm (1.77 in) in depth.
NOTE 3- Containers known as tin boxes or as seamless ointment boxes may be obtained in dimensions conforming to the above requirements.

6.4 Water Bath - A water bath maintained at a temperature varying not more than 0.1°C (0.2°F) from the temperature of the test. The volume of water shall not be less than 10 liters. The bath shall have a perforated shelf supported in a position not less than 50 mm from the bottom of the bath and not less than 100 mm below the liquid level in the bath. The water in the bath shall be substantially free from oil and slime or other organic growth. Brine may be used in the water bath for determinations at low temperatures. If penetration tests are to be made without removing the sample from the bath, a shelf strong enough to support the penetration apparatus shall be provided.
NOTE 4- The use of distilled, demineralized or deionized water is recommended for the bath. Care should be taken to avoid contamination of the bath water by surface active agents, release agents or other chemicals as their presence may affect the penetration values obtained.

6.5 Transfer Dish for Container- When used; the transfer dish for the container shall be a cylinder with a flat bottom made of glass, metal or plastic. It shall be provided with some means which will ensure a firm bearing and prevent rocking of the container. It shall have a minimum inside diameter of 90 mm (3.5 in) and a minimum depth above the bottom bearing of 55 mm (2.17 in).
NOTE 5- A magnetic strip in the bottom of the transfer dish may be used to prevent the ointment tin from rocking.

6.6 Thermometers for Water Bath- Calibrated Liquid-in-glass thermometers of suitable range with subdivisions and maximum scale error of 0.1°C (0.2°F) or any other thermometric device of equal accuracy, precision, and sensitivity shall be used.

The following thermometers conforming to the requirements of ASTM Specification E1, ASTM Thermometers are required:

6.6.1 For tests at 25°C (77°F) use an ASTM Saybolt Viscosity Thermometer 17°C (or 17°F) having a range of 19 to 27°C (66 to 80°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.2 For tests at 0°C (32°F) and 4°C (39.2°F) use ASTM Precision Thermometer 63°C (or 63°F) having a range of -8 to + 32°C (18 to 89°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.3 For tests at 46.1°C (115°F) use ASTM Precision Thermometer 64°C (or 64°F) having a range of 25 to 55°C (77 to 131°F). The thermometer shall be immersed in the bath 150 ± 15 mm.

6.6.4 Since the accuracy of the test results is dependent upon closely controlled temperature conditions, the thermometer used for the water bath should be calibrated by ASTM E77, Inspection, Test and standardization of Etched Stem Liquid-in-Glass thermometers.

6.7 Timing Device- For hand-operated penetrometers any convenient timing device such as an electric timer, a stop watch, or other spring-activated device may be used provided it is graduated in 0.1 second or less and is accurate to within ± 0.1 second for a 60 second interval. An audible second counter adjusted to provide 1 beat each 0.5 second may also be used. The time for a count interval must be 5±0.1 second. Any automatic timing device attached to a penetrometer must be accurately calibrated to provide the desired test interval within ± 0.1 second.

6.8 Heater- An oven or hot plate, heated by electricity or gas, shall be provided for heating samples.

7. PREPARATION OF SAMPLE

7.1 Heat the sample with care to prevent local overheating until it has become fluid. Then with constant stirring, raise the temperature of the asphalt sample not more than 100°C or l80°F above its expected softening point or the tar pitch sample not more than 56°C or 100°F above its softening point determined in accordance with the Method of test for Softening Point of Bituminous Materials (Ring and Ball Method), T 53. Avoid the inclusion of air bubbles. To reach the pouring temperature, do not heat the softened sample more than 30 minutes.

Then pour it into the sample container to a depth such that, when cooled to the temperature of test the depth of the sample is at least 10 mm greater than the depth to which the needle is expected to penetrate. Pour separate samples for each variation in test conditions.

7.2 Loosely cover each container and its contents as a protection against dust, and allow to cool in an atmosphere at a temperature not higher than 30°C or 86°F and not lower than 20°C or 68°F for not less than $1\frac{1}{2}$hours nor more than 2 hours when the sample is in a 175 ml (6 oz) container and for not less than 1 nor more than $1\frac{1}{2}$hours when the sample is in a 90 ml (3 oz) container. Then place the sample in the water bath maintained at the prescribed temperature of test, along with the transfer dish if used, and allow it to remain for not less than $1\frac{1}{2}$hours nor more than 2 hours when the sample is in the 175 ml (6 oz) container, and for not less than 1 nor more than $1\frac{1}{2}$hours when the sample is in a 90 ml (3 oz) container.

8. TEST CONDITIONS

8.1 Where the conditions of test are not specifically mentioned, the temperature, load, and time are understood to be 25°C (77°F), 100 g, 5 second, respectively.

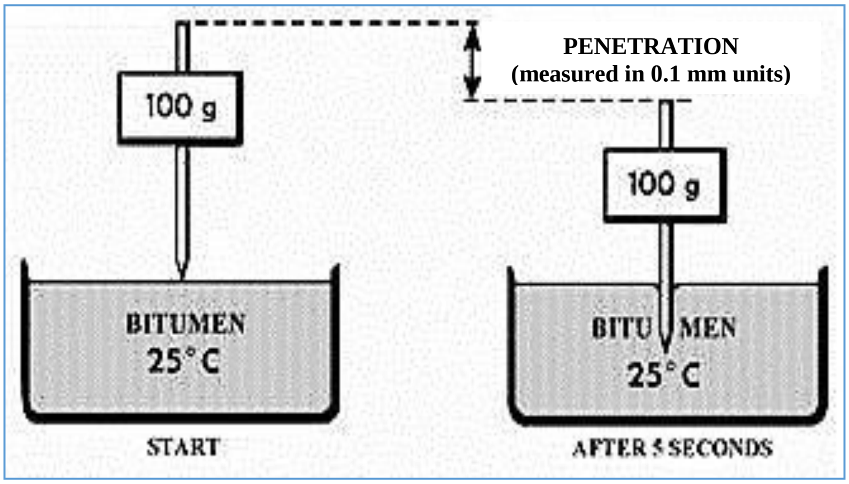

Figure 7.2: Penetration test

Other conditions of temperature, load and time may be used for special testing, such as:

Table 7.1: Temperature and Time for different loads of penetration needle assembly.

Temperature	Load, g	Time
0˚C/(32˚F)	200	60
4˚C/(39.2˚F)	200	60
46.1^0C/(115^0F)	50	5

In such cases, the specific conditions of test shall be reported.

9. PROCEDURES

9.1 Examine the needle holder and guide to establish the absence of water and other extraneous matter. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth, and insert the needle in the penetrometer. Unless otherwise specified, place the 50 g weight above the needle, making the total load of 100 g ± 0.1g for the needle and attachment. If tests are made with the penetration apparatus mounted in the bath, place the sample container directly on the submerged stand of the penetration apparatus. If tests are made with the sample in the bath and the penetration apparatus outside the bath, place the containers on the shelf provided in the bath. In the above procedures the container shall be kept completely submerged during the complete test. If tests aremade using the transfer dish with the penetration apparatus outside the bath, place the sample in a dish filled with water from the bath to a depth to cover completely the sample container. Then place the transfer dish containing the sample on the stand on the penetration apparatus and penetrate immediately. In each case, adjust the needle loaded with the specified weight to just make contact with the surface of the sample. Accomplish this by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light (Note 8). Either note the reading of the dial or bring the pointer to zero. Then quickly release the needle for the specified period of time and adjust the instrument to measure the distance penetrated. Observe the sample container as the needle is applied, and if any movement of the container is noted, ignore the result.

NOTE 6- For certain types of asphalt erratic results are sometimes obtained. When this occurs, pre-treat the needles by immersing them for 5 minutes in a 1 percent solution of olene acid prior to drying and running the test.

NOLL 7- For reference tests, penetrations at temperature other than 25^0C (77^0F) should be made without removing the sample from the bath.

NOTE 8- The positioning of the needle can be materially aided by using an illuminated methyl methacrylate rod.

9.2 Make at least three penetrations at points on the surface of the sample not less than 10 mm (3/8 in) from the side of the container and not less than 10 mm (3/8 in) apart. If the transfer dish is used, return the dish and sample to the water bath after each penetration. Before each test, clean the needle with a clean cloth moistened with toluene or other suitable solvent to remove all adhering bitumen, and then wipe with a clean dry cloth. For penetration values greater than 200, use at least three needles, leaving them in the sample until completion of the penetrations.

9.3 The needles, containers, and other conditions described in this method provide for determinations of penetrations up to 350. However, the method may be used for direct determinations up to 500 provided special containers and needles are used. The container shall be at least 60 mm in depth. The overall volume of material in the container should not exceed 125 ml to permit proper temperature adjustment of the sample.

9.3.1 Specially made needles for such determination shall meet all the requirements of Section 6.2 for dimensions and weight except that the minimum exposed length of the needle shall be 50 mm.
9.3.2 An approximation of the penetration of such high penetration materials may also be obtained by determining the penetration using the standard needle and 6 oz container but with a 50 g loading. The penetration is then calculated by multiplying the result for the 50 g load by the square root of 2. That is:

Penetration under 100g load = (Penetration under 50g load) x 1.414

The report of results obtained by this procedure shall indicate the basis of the test.

10. REPORT

10.1 Report to the nearest whole unit the average of at least three penetrations whose values do not differ by more than the amount shown (table 7.2):

Table 7.2 : Limit of penetration values for different penetration range.

Penetration	0-49	50-149	150-249	≥ 250
Maximum difference between highest and lowest determinations	2	4	6	8

10.1.1 If the appropriate tolerance is exceeded, ignore all results and repeat the test.

EXP. 8: SOFTENING POINT OF BITUMINOUS MATERIAL (RING AND BALL METHOD)

1. SCOPE

1.1 The ring and ball softening point is extensively used to evaluate the consistency of bituminous binders. It is a very simple one, consisting of placing a 3/8 in diameter steel ball on a binder sample placed in a steel ring and immersed in a water bath. Heat is applied to the water and its temperature is raised until a value is reached when the test sample has become sufficiently soft to allow the ball, enveloped in binder to fall down. The water temperature at which this occurs is called the ring and ball softening point.

The softening point is not a melting point; bituminous binders do not melt but instead gradually change from semi-solids to liquids on the application of heat. It is useful for determining the temperature susceptibilities of bitumen which are to be used in thick films, such as in crack fillers. When two bitumen have the same penetration value, the one with the higher softening point is normally less susceptible to temperature changes.

2. REFERENCED DOCUMENTS

2.1 Standards

C 670 Practices for Preparing Precision Statements for Test Methods for Construction materials

E 1 Specification for ASTM Thermometers

T 40 Methods of Sampling Bituminous Materials

T 48 Test Method for Flash and Fire Points by Cleveland Open cup

3. APPARATUS AND MATERIALS

3.1 Ring- A brass ring of 15.875 mm (5/8 in) inside diameter, 6.35 mm (1/4 in) depth and thickness of wallis 2.38 mm (3/32 in). This ring shall be attached in a convenient manner to a brass with (diameter 1.85 mm = 0.072 in).

3.2 Ball - A steel ball 9.53 mm (3/8 in) in diameter having a mass of 3.50 ± 0.05 g.

3.3 Container - A glass vessel, not less than 8.5 cm (3.34 in) in diameter and measuring 10.5 cm (4.13 in.) in depth from the bottom of the flare (a 600 ml beaker, low form, meets this requirement).

Figure 8.1 Ring and Ball apparatus

3.4 Thermometer - ASTM Low Softening point Thermometer having a range of -2 to +80°C or 30°to 180°Fis specified.

4. REAGENTS AND MATERIALS

4.1 Bath liquids:
4.1.1 Freshly boiled distilled water.
4.1.2 USP Glycerin, or
4.1.3 Ethyl Glycol, with a boiling point between 195 and 197°C (383 and 387°F).

5. PREPARATION OF SAMPLE

Melt and thoroughly stir the sample avoiding incorporating air bubbles in the mass and then pour it into the ring. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. Allow the excess material to cool for 1 hr. then cut it off cleanly with a slightly heated knife.

6. PROCEDURE FOR MATERIALS HAVING SOFTENING POINTS 80^{0}C (176^{0}F) OR BELOW

6.1 Fill the glass vessel to a depth of substantially 8.25 cm (3.25 in) with freshly boiled, distilled water at 5°C (41°F).

6.2 Suspend the ring containing the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm (1 in) above the bottom of the glass vessel and its upper surface is 5.08 cm (2 in) below the surface of the water.
6.3 Place the ball in the water but not on the specimen.

6.4 Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm (3/4 in) but not touching the ring. Maintain the temperature of the water at 5°C (41°F) for 15 min.

6.5 With suitable force, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly.

6.6 Apply the heat in such a manner that the temperature of the water is raised 5°C (9°F) each minute.

7. SOFTENING POINT

Report the temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel as the softening point. No correction shall be made for emergent stem of the thermometer.

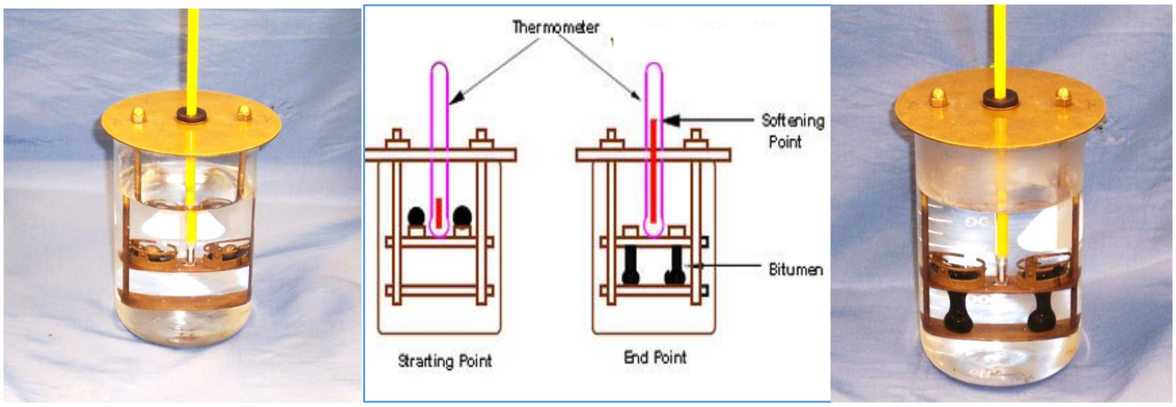

Figure 8.2 Illustration of softening point

8. PERMISSIBLE VARIATION IN RISE OF TEMPERATURE

The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three minutes shall be 0.5°C (0.9°F). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

9. PROCEDURE FOR MATERIALS HAVING SOFTENING POINTS ABOVE 80^{0}C (176^{0}F)

Thermometer- An ASTM high softening point Thermometer having a range of 30 to 200°C or 85 to 392°F is specified Modifications for Hard Materials.
Employ the same procedure as described above except that U.S.P., Glycerin shall be used instead of water, and the starting point of the Glycerin bath shall be 32°C (89.6°F). Bring the bath to this

temperature and thoroughly agitate it, then place the apparatus and specimens in the bath, which shall be maintained, under agitation at the starting temperature for 15 min. In applying the heat, place the ring apparatus of the center of the container and place the burner midway between the center and edge of the beaker away from the specimen.

10. PRECAUTIONS

10.1 The use of freshly boiled distilled water is essential as otherwise air bubbles may form on the specimen and affect the accuracy of the results. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

11. ACCURACY

The limit of accuracy of the test is 0.5°C (0.9°F).

EXP. 9: DUCTILITY OF BITUMINOUS MATERIAL

1. SCOPE

1.1 The ductility of a bituminous material is measured by the distance to which it will elongated before breaking when two ends of a briquette specimen of the material, of the form describe in Section 2, are pulled apart at a specified speed and at a specified temperature. Unless otherwise specified, the test shall be made at a temperature of $77^{\circ}\pm0.9^{\circ}$F ($25^{\circ}\pm0.5^{\circ}$C) and with a speed of 5 cm/min, + 5.0 percent. At other temperatures the speed should be specified.

2. APPARATUS

2.1 Mould - the mould shall be similar in design to that shown in Figure 1. Dimensions shown Figure 1 shall be as given with the permissible variations indicated. The mould shall be made of brass, the ends b and b' being known as clips, and the parts a and a' as sides of the mould. The dimensions of the mould shall be such that, when properly assembled, it will form a briquette specimen having the following dimensions (Table 9.1):

Table 9.1: Dimensions of Ductility Test apparatus.

Total length	7.45 to 7.55 cm
Distance between clips	2.97 to 3.03 cm
Width at mouth of clip	1.98 to 2.02 cm
Width at minimum cross section (halfway between clips)	0.99 to 1.01 cm
Thickness throughout	0.99 to 1.01 cm

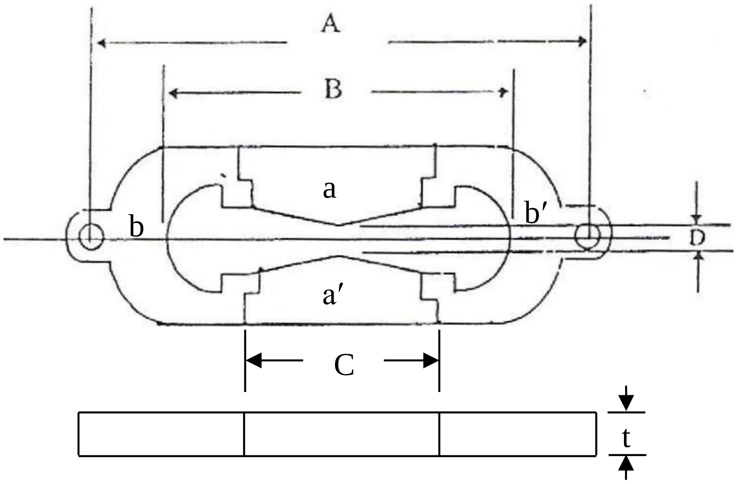

Figure 9.1 Mold for Ductility Test Specimen

Table 9.2: Dimensions of different parts of Ductility Test apparatus shown in Figure 9.1.

A	Distance between centers	111.5 to 113.5 mm
B	Total length of briquette	74.5 to 75.5 mm
D	Width at minimum cross section	9.9 to 10.1 mm
t	Thickness	9.9 to 10.1 mm

2.2 Water Bath - The water bath shall be maintained at the specified test temperature, varying not more than 0.18°F (0.1°C) from this temperature. They volume of water shall be not less than 10 liters, and the specimen shall be immersed to a depth of not less than 10 cm and shall be supported on a perforated shelf not less than 5 cm from the bottom of the bath.

2.3 Testing Machine - For pulling the briquette of bituminous material apart, any apparatus may be used which is so constructed that the specimen will be continuously immersed in water as specified in Section 3.3, while the two clips are pulled apart at a uniform speed, as specified, without under vibration. Figure 2 shows the ductility testing machine.

Figure 9.2: Ductility Testing Machine

2.4 Thermometer - A thermometer having a range as shown below (Table 9.3) and conforming to the requirements prescribed in Specification E 1 for Standard Thermometer.

Table 9.3: Specifics of Standard Thermometer for Ductility Test.

Temperature Range	ASTM Thermometer No
-8 to 32°C	63°C
18 to 89°F	63°F

3. PROCEDURE

3.1 Molding Test Specimen - Heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour. Strain the melted sample through a No. 50 sieve conforming to ASTM Specification E11, for Wire cloth Sieves for Testing Purposes, and, after a thorough stirring pour it into the mold. Assemble the mold on a brass plate and, to prevent the material under test from sticking, thoroughly amalgamate the surface of the plate and interior surface of the sides a and a', Figure 1, of the mold or coat with a mixture of glycerin and dextrin, tale, or china clay. The plate upon which the mold is placed shall be perfectly flat and level so that the bottom surface of the mold will touch it throughout. In filling the mold, take care not to disarrange the parts and thus distort the briquette. In filling, pour the material in a thin stream back and forth from end to end of the mold until the mold is more than level full. Let the mold containing the material cool to room temperature or a period of from 30 to 40 min and then place it in the water bath maintained at the specified temperature of test for 30 min; then cut off the excess bitumen with a hot straight edged putty knife or spatula to make the mold just level full as shown in Figure 9.3.

Figure 9.3: Molding and Cutting of Test Specimen

3.1.1 Caution - Careless handling of mercury will create a definite health hazard. The rules prescribed as follows should be observed at all times:

1. Store the mercury in a closed jug in a cool place.
2. Strictly avoid spilling any mercury.
3. Remove mercury vapors by working under a ventilated hood.
4. Keep amalgamated brass plates and other apparatus at no higher than normal room temperature.

3.2 Keeping Specimen at Standard Temperature - Place the brass plate and mold, with briquette specimen, in the water bath and keep at the specified temperature for a period of from 85 to 95 min. Then remove the briquette from plate, detach the side pieces, and immediately test the briquette.

3.3 Testing - Attach the rings at each end of the clips to the pins or hooks in the testing machine and pull the two clips apart at a uniform speed as specified until the briquetteruptures as shown in Figure 9.4.

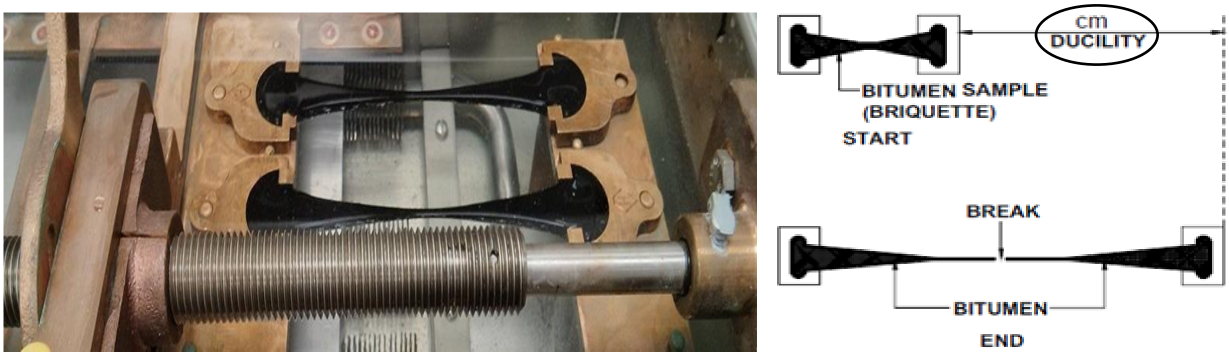

Figure 9.4: Ductility Testing

A variation of + 5 percent from the speed specified will be permissible. Measure the distance in centimeters through which the clips have been pulled to produce rupture. While the test is being made, the water in the tank of the testing machine shall cover the specimen both above and below it by at least 2.5 cm and shall be kept continuously at the temperature specified within +0.9°F (0.5°C).

4. REPORT

4.1 A normal test is one in which the material between the clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross sectional area. Report the average of three normal tests as the ductility of the sample.

4.2 If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal. Adjust the specific gravity of the bath by the addition of either methyl alcohol or sodium chloride so that the bituminous material neither comes to the surface of the water, nor touches the bottom of the bath at any time during the test.

4.3 If a normal tests not obtainable on three terms, report the ductility as being unobtainable under the conditions of the test.

5. PRECAUTIONS

5.1 Owing to possible danger to health if mercury is handled carelessly, the following rules should be observed at all times:

5.1.1 Store the mercury in a closed jug in a cool place.
5.1.2 Strictly avoid spilling any mercury.
5.1.3 Remove mercury vapors by working under a suitable hood with good ventilation.
5.1.4 Keep amalgamated brass plates and other apparatus at not above normal room temperature.

EXP. 10: FLASH AND FIRE POINTS OF BITUMINOUS MATERIAL (CLEVELAND OPEN CUP METHOD)

1. SCOPE AND SIGNIFICANCE

This method describes a test procedure for determining the flash and fire points (Cleveland Open Cup Tester) of all petroleum products except fuel oils and those having an open cup flash below 175^0F. The flash point is the temperature at which a bituminous material, during heating, will evolve vapors that will temporarily ignites or flash when a small flame is brought in contact with them. The fire point is the temperature at which the evolved vapors will ignite and continue to burn.

To make the test, the material is heated in an open cup, and at intervals a small flame is applied near its surface. The lowest temperature at which application of the test flame causes the vapors to ignite is recorded as the flash point while the temperature at which the vapors ignited and burn for at least 5 seconds is recorded as the fire point. The flash and fire point test is purely a safety test. It indicates the maximum temperature to which the material can be safely heated.

NOTE 1- It is the practice in the United Kingdom and in any other countries to use IP Method 35, Flash Point (Open) and Fire Points by Means of the Pensky-Martens Apparatus unless T73. Test flash point by Pensky-Martens Closed Tester is specified. This Method may occasionally be specified for the determination of the fire point of a fuel oil. For the determination of Flash points of fuel oils, use AASHTO T 73 IP 34, T 73 should be used when it is desired to determine the possible presence of small but significant concentrations of lower flash points substances which may escape detection by T 48. T79, Flash Point with Tag Open Cup Apparatus, may be employed if the flash point is below 79˚C (175^0F) as determined by T 48.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards:
T 73 Flash Point by Pensky-Martens Closed Tester
T 79 Flash Point with Tag Open-Cup Tester

2.2 ASTM Standards:
E 1 Specification for ASTM Thermometers

2.3 Other Methods:
IP Method 35 Flash Point(Open) and Fire Point by Means of the pensky-Martens Apparatus.

3. APPARATUS AND MATERIALS

3.1 Cleveland Open Tester - The apparatus consists of the test cup, heating plate, test flame applicator, heater, and support as shown in Figure 10.1.

Figure 10.1: Cleveland Open Cup Tester

3.2 Shield - A shield 18 inch (46 cm) square and 24 in, (61 cm) high, is recommended but not essential.

3.3 Thermometer - ASTM thermometer having a range of 20^0F to 760^0F(-6°C to + 400°C).

4. PROCEDURE

4.1 Support the tester on a level steady table in a draft free room or compartment and shield the spot of the tester from strong light by any suitable means.

4.2 Clean the cup with an appropriate solvent and remove all gums, carbon deposit, and oxide coating from the inside of the cup with fine steel wool until a bright metallic surface is presented.

4.3 Support the thermometer in a vertical position with the bottom of the bulb 1/4 inch (0.635 cm) from the bottom of the cup and above a point halfway between the center and back of the cup.
Note 2- The immersion line engraved on the thermometer will be 5/64 inch (0.20 cm) below level of the rim of the cup when the thermometer is properly positioned.

4.4 Fill the cup at any convenient temperature (Note 3) so that the top of the meniscus is exactly at the filling line. When too much sample has been added to the cup, remove the excess, using a spoon or other suitable device; however, if there is sample on the outside of the apparatus, empty, clean. Destroy any air bubbles appear on the surface of the sample.

Note 3- Viscous samples should be heated until they are reasonably fluid before being poured in to the cup; however, the temperature during heating must not exceed 100°F (65°C) below the probable flash point.

4.5 Light the test flame and adjust it to a diameter of 1/8 to 3/16 in. (0.08 cm).

4.6 Apply heat initially so that the rate of temperature rise of the sample is 25 to 30^0F (13.9 to 16.7°C) per minute. When the sample temperature is approximately 100°F (56^0C) below the anticipated flash point, decrease the heat so that the rate of temperature rise for the last 50^0F (27.8°C) before the flash point is 10 + 1°F (5.5 + 0.6°C) per minute.

4.7 Starting at least 50°F (2.8°C) mark pass the test flame across the center of the cup, at right angles to the diameter which passes through the thermometer. With a smooth, continuous motion apply the flame either in a straight line or along the circumference of a circle having a radius of at least 6 inch (15 cm). The center of the test flame must move in a plane not more than 5/6" inch (0.2 cm) above the plane of the upper edge of the cup. The time consumed in passing the test flame across the cup shall be about 1 sec.

4.8 Record as the flash point the temperature read on the thermometer when a flash appear at any point on the surface of the sample but do not confuse the true flash with the bluish halo that sometimes surrounds the test flame.

4.9 To determine the fire point, continue heating so that the sample temperature increases at rate of 10 ± 1°F (5.5± 0.6^0C) per minute. Continue the application of the test flame at 5^0F (2.8°C) intervals until the vapor ignites and continues to burn for at least 5 sec. Record the temperature at this point as the fire point.

5. CALCULATION AND REPORT

5.1 Observe and record the barometric pressure at the time of the test. When the pressure differs from 760 mm Hg, correct the flash or fire point, or both, by means of the following equations:

- Corrected flash or fire point, or both = F + 0.06(760-P) or

- Corrected flash or fire point, or both = C + 0.03(760-P)

Where:

F = observed flash or fire point, or both, to the nearest 5°F
C = observed flash or fire point, or both, to the nearest 2°C.
P = barometric pressure, mm Hg.