BS 2000 :
Part 58 : 1993

Methods of test for

Petroleum and its products

Part 58. Determination of softening point of bitumen – Ring and ball method

(Identical with IP 58/86(89))

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BSI
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Foreword

This British Standard, having been prepared under the direction of the Petroleum Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 February 1993.

This British Standard supersedes BS 2000 : Part 58 : 1988, which is withdrawn.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of `IP Standard methods for analysis and testing of petroleum and related products' which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as Parts of BS 2000.

The numbering of the Parts of BS 2000 follows that of the corresponding methods published in `IP Standard methods for analysis and testing of petroleum and related products'. Under the terms of the agreement between BSI and the Institute of Petroleum, the revised version of BS 2000 : Part 58 will be published by the IP (in `Standard methods for analysis and testing of petroleum and related products' and as a separate publication). BS 2000 : Part 58 : 1993 is thus identical with IP 58/86, which was reapproved in 1989. Square brackets marked in the margin of this IP Standard indicate text that differs from the previous edition.

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Committee reference PTC/13

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IP 58/86 (1989) BS 2000: Part 58: 1993

Determination of softening point of bitumen – Ring and ball method

This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations.

1. SCOPE

1.1. This method describes a procedure for determining the softening point of bitumen.

*NOTE 1: The method can be applied to heterogeneous blends such as polymer-bitumens, but extra care must be taken to obtain a representative sample and to minimise thermal deterioration. Precision data are not available for such materials.

2. DEFINITION

2.1. *Softening Point* – The temperature at which a substance attains a particular degree of softness under specified conditions of test.

3. SUMMARY OF METHOD

3.1. A steel ball of specified mass is placed upon a disk of bitumen contained within a metal ring of specified dimensions. The assembled apparatus is placed in a bath of liquid and the temperature of the liquid is raised at a specified rate. The softening point is the temperature at which the bitumen surrounding the steel ball just touches the base plate of the apparatus.

4. APPARATUS

4.1. *Ball* – a steel ball having a diameter of 9.53 mm and weighing 3.50 ± 0.05 g.

4.2. *Ring* – the tapered ring, made of brass, and conforming to the dimensions shown in Fig. 1, shall be used for referee purposes.

For other purposes either a straight ring or (Fig. 2) a square-shouldered ring (Fig. 3) may be used.

4.3. *Ball Guide* – A convenient form of ball centring guide is shown in Fig. 4.

4.4. *Ring Holder* – of brass or other metal and conforming to the dimensions given in Fig. 5 [Note 2].

4.5. *Bottom Plate* – of brass or other metal and conforming to the dimensions given in Fig. 6 [Note 2].

NOTE 2: In laboratories where many samples are tested it may be found advantageous to use a circular ring holder carrying up to six test sample rings. The distance of the rings from the centre is not necessarily the same as in the standard two-ring holder, but extended tests with such an instrument have shown that it gives results in sufficient practical agreement with those using the standard two-ring instrument to warrant its use for routine operations only.

NOTE 3: Protective metallic plating may be used on any of the above-mentioned rings, ball guides, ring holders or bottom plates, provided that the final dimensions conform to those shown. It may not be used on the steel balls.

4.6. *Thermometer* – conforming to the specification[1] IP 60 C, or IP 61 C, as required.

[1]Appears in volume 2 of this publication. As Appendix A. Specification for Thermometers.

4.7. *Bath* – of heat-resistant glass and conforming to the dimensions given in Fig. 7, the rings being supported in a horizontal position as shown. The bottom of the bulb of the thermometer shall be level with the bottom of the rings and within 10 mm of them but not touching them. A squat form 600 ml beaker is suitable.

4.8. *Bath Liquid* – When testing materials having softening points of 80°C or below, the bath liquid shall be freshly distilled water. For materials of higher softening points, the liquid shall be glycerol [Note 4].

NOTE 4: The volume of liquid needed for a bath of the stated dimensions is approx 550 ml.

4.9. *Stirrer* – To ensure uniform heat distribution throughout the bath, a mechanical stirrer which operates smoothly shall be used. It shall be so placed that the samples are not disturbed when it is in operation.

CAUTION: *If the stirrer is electrically driven ensure that it is safely earthed.*

5. SAMPLE TREATMENT AND TEST-SPECIMEN PREPARATION

5.1. *General* – The importance of taking all possible care in the treatment of the sample in order to prepare the test specimens cannot be over-stressed. Unless it cannot be avoided, the test material shall be heated once only to enable the test specimens to be prepared. The method used to prepare the test specimens will depend very largely upon the condition and volume of the sample received for testing, and on the type of bitumen. The over-riding principles are that:

(*a*) the material shall not undergo any change in its properties during specimen preparation, and

(*b*) the sample shall be sufficiently fluid, at the time that the rings are filled, to keep the risk of the presence of air bubbles in the test specimens to a very low level.

To meet these conditions it is necessary to select the temperature to which the sample shall be heated by considering the properties of the actual material under examination. Thus, for all bitumens used in the production of materials for road pavements, and for industrial grades with softening points below 80°C, the material shall be heated until it is 80 to 90°C above its expected softening point. For industrial grades with softening points above 80°C, it is not possible to define the heating conditions precisely; in

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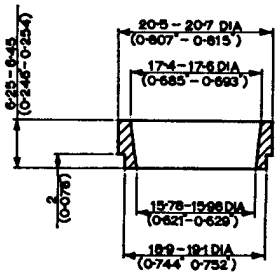

FIG.1 TAPERED RING MATERIAL BRASS

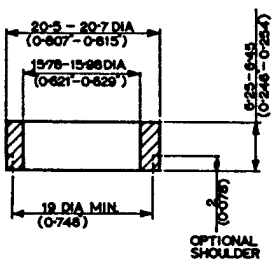

FIG.2 STRAIGHT RING

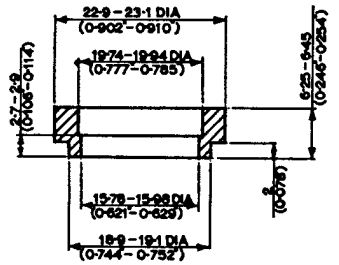

FIG.3 SHOULDERED RING

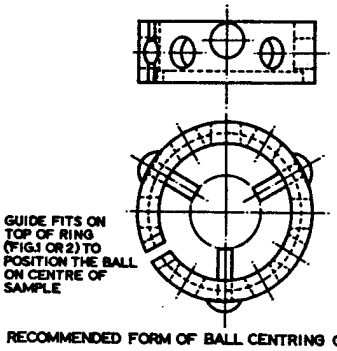

FIG.4 RECOMMENDED FORM OF BALL CENTRING GUIDE

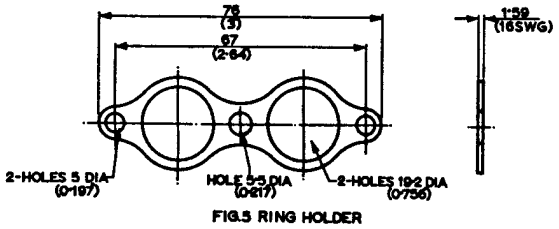

FIG.5 RING HOLDER

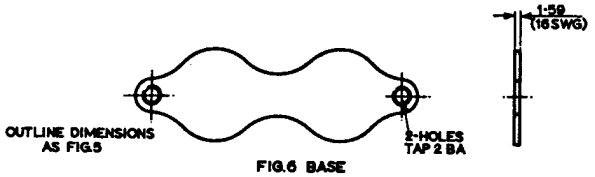

FIG.6 BASE

DIMENSIONS IN MILLIMETRES (INCHES)

such cases the material shall be heated until it reaches the lowest temperature that will ensure that the test specimens are free from air bubbles; in no case shall the temperature of the test material exceed its softening point by more than 120°C.

5.2. *Heating cold samples.*

5.2.1. *Samples of up to 3000 ml* – Ease the lid or other closure of the container in which the sample is received. Then place the container in an oven maintained at a temperature 100 ± 5°C above the expected softening point. For industrial bitumens with softening points above 80°C, use an oven

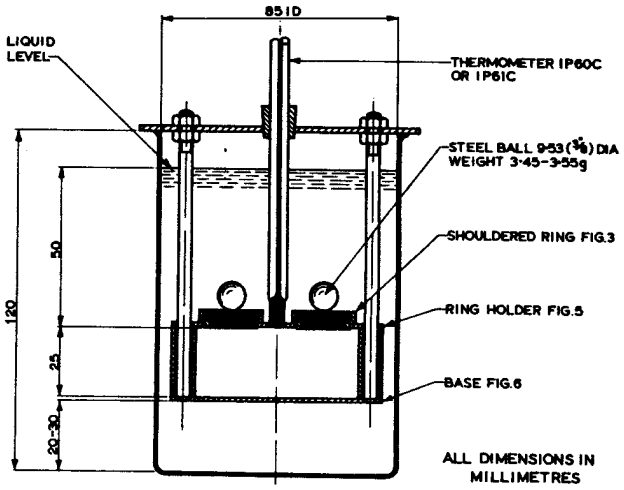

Fig. 7. Assembly of ring-and-ball apparatus for two rings (stirrer not shown).

temperature that will ensure that the bitumen is sufficiently fluid to provide test specimens that are free from air bubbles. Leave the container in the oven for the time indicated in the following Table:

Volume of the sample, ml	Period of heating, hrs
300 ± 200	$1\frac{1}{2} \pm \frac{1}{4}$
750 ± 250	$2 \pm \frac{1}{4}$
1500 ± 500	$2\frac{1}{2} \pm \frac{1}{2}$
2500 ± 500	$3 \pm \frac{1}{2}$

NOTE 5. The oven temperatures and periods of heating given above are intended to achieve the sample temperatures given in 5.1.

5.2.2. Samples of more than 3000 ml and samples received in lump form.

NOTE 6. This procedure may also be used for small samples. Provided there is no reason to suspect the homogeneity of the sample, remove a portion of the sample, eg 350 ± 50 g, if necessary using a warmed 'knife', and transfer to a suitable container. Then, either follow the procedure described in 5.2.1 with a heating period of $1\frac{1}{2} \pm \frac{1}{4}$ hr, or heat the sub-sample over a hot-air bath. If the hot-air bath method is used, stir the material at the earliest opportunity and then frequently to prevent local over-heating; continue the heating until the material is free from air but ensure that the appropriate temperature given in 5.1 is not exceeded.

5.3. *Treatment of very hot samples.*

5.3.1. *General* – Samples taken from delivery tankers or storage tanks and received in the testing laboratory without delay will frequently be at temperatures in excess of the maxima quoted in 5.1. In such cases great care is needed when handling these very hot samples, both because of personal safety and because of the inevitable loss of some of the more volatile constituents of the material to be tested.

5.3.2. *Cooling procedure* – Loosely cover the sample container and allow the sample to cool to the appropriate specimen-preparation temperature specified in 5.1; stir the sample occasionally to prevent the material near the walls of the container from over-cooling.

5.4. *Filling the rings* – With a mixture of equal parts of glycerol and dextrin, lightly coat the upper surface of a small metal plate, eg a brass plate approximately 80 mm × 40 mm × about 1.5 mm thick. Heat two rings to approximately the same temperature as the sample and place them on the

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metal plate, then, without delay, fill the rings with the sample such that there will be an excess above the top of each ring after the test specimens have cooled to room temperature.

NOTE 7: The coating with glycerol and dextrin is made easier if the plate is warmed to about 40°C.

5.5 *Levelling the test specimens* – Allow the filled rings to cool for 30 to 40 min at room temperature, then remove any excess bitumen so that the test specimens are level with the tops of the rings. This operation is easily carried out using a warmed blade.

6. PROCEDURE

6.1. *Start of test* – Start the test not less than ten minutes nor more than eight hours after levelling the specimens.

6.2. *Assembly of apparatus* – Insert the appropriate thermometer through the hole in the top plate and adjust so that the bottom of the thermometer bulb will be level with the bottom of the rings, ie just below (0.4 mm) the bottom of the ring holder. Place the filled rings in the ring holder and the ball-centring guide on the rings.

6.3. *Preparation of the bath.*

6.3.1. *For softening points of 80°C and below* – With the distilled water in the bath at 5 ± 2°C, transfer the assembled apparatus and the steel balls to the bath; ensure that the level of the water is 50 ± 5 mm above the tops of the rings. Maintain the bath temperature at 5 ± 2°C for 15 min, then, using tongs or other suitable implement, place a steel ball on each of the test specimens.

6.3.2. *For softening points above 80°C* – With the glycerol in the bath at 35 ± 2°C, transfer the assembled apparatus and steel balls to the bath; ensure that the level of the glycerol is 50 ± 5 mm above the tops of the rings. Maintain the bath temperature at 35 ± 2°C for 15 min, then, using tongs or other suitable implement, place a steel ball on each of the test specimens.

6.4. *Raising the bath temperature* – Stir the bath liquid and heat so that the temperature of the bath liquid rises at 5 ± 0.5°C per min. There are practical difficulties in obtaining the specified rate immediately heat is applied to the bath, however, if after the first 3 min the rate of temperature rise does not fall within the specified limits, repeat the test. If air bubbles form beneath the specimens, repeat the test.

6.5. *The softening point* – For each test specimen, estimate to the nearest half division the temperature shown by the thermometer at the instant that the bitumen surrounding the ball touches the base plate. Record the temperatures.

6.6. If the differences between the two recorded temperatures exceed 1°C, repeat the test.

7. CALCULATION

7.1. *For paving grades of bitumen* – Calculate the mean of the two recorded softening points and round to the nearest 0.2°C.

7.2. *For industrial grades of bitumen* – Calculate the mean of the two recorded softening points and round to the nearest 0.5°C.

8. REPORT

8.1. Report the mean, obtained as described in 7, as the Softening Point, IP 58.

9. PRECISION

9.1. The following criteria should be used for judging the acceptability of results (95% confidence).

9.1.1. *Paving grades of bitumen* – The following precision values apply to the full range of bitumens from 15 to 450 penetration as specified in BS 3690: Part 1:–

Repeatability 1.0°C
Reproducibility 2.5°C

These precision values (as defined in Appendix E) were obtained by statistical examination of inter-laboratory results, and were first published in 1981.

9.1.2. *Industrial grades of bitumen* – The following precision values apply to the full range of industrial bitumens as specified in BS 3690: Part 2:–

Repeatability 1.5°C
Reproducibility 5.5°C

These precision values (as defined in Appendix E) were obtained by statistical examination of inter-laboratory results, and were first published in 1986.

10. AUTOMATIC APPARATUS

10.1. Automatic apparatus is used in some laboratories for routine, 'non-referee', determinations. It is not possible to write a test method defining an automatic apparatus which would be certain to give results equivalent in level and precision to those obtained by the standard manual method described above. Consequently the use of automatic apparatus is not covered by the standard test which should always be used for referee purposes.

However during the inter-laboratory experiments to estimate the precision of the test for industrial grades, four laboratories determined results obtained by automatic apparatus as well as by the standard manual method. Analysis of these data give the following estimates of precision for the test conducted with automatic apparatus for INDUSTRIAL GRADES ONLY:–

Determinability	0.03 (X – 34)°C
Repeatability	0.03 (X – 34)°C
Reproducibility	0.09 (X – 34)°C

Where X is the average of two results. These values, obtained as described above, were first published in 1986.

Examples of the precision for given values of X are:–

X°C	Determinability and Repeatability, °C	Reproducibility, °C
75	1.2	3.7
80	1.4	4.1
85	1.6	4.6
90	1.7	5.0
95	1.8	5.5
100	2.0	5.9
105	2.1	6.4
110	2.3	6.8
115	2.4	7.3
120	2.6	7.7

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