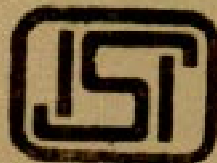


IS : 9381 • 1979

Indian Standard

METHODS FOR TESTING
TAR AND BITUMINOUS MATERIALS:
DETERMINATION OF FRAASS BREAKING
POINT OF BITUMEN

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**METHODS FOR TESTING
TAR AND BITUMINOUS MATERIALS:
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(Continued on page 9)

Indian Standard
METHODS FOR TESTING
TAR AND BITUMINOUS MATERIALS:
DETERMINATION OF FRAASS BREAKING
POINT OF BITUMEN
0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 December 1979, after the draft finalized by the Bitumen and Tar Products Sectional Committee had been approved by the Civil Engineering Division Council and Petroleum, Coal and Related Products Division Council.

0.2 A compilation of methods of test for testing tar and bituminous materials for their various characteristics has already been prepared (see IS : 1201-1978 to IS : 1220-1978*). However, it has now been felt necessary to establish a method of test to determine the behaviour of bituminous materials under low temperatures and to find out whether these products would stand the low temperature or not. Accordingly, this standard has been prepared to cover the method of test for the determination of FRAASS breaking point of solid and semi-solid bitumens.

0.3 The Sectional Committee responsible for the preparation of this standard has taken into consideration the views of producers, consumers and technologists and has related the standard to the manufacturing and trade practices followed in the country in this field. Due weightage has also been given to the need for international co-ordination among standards prevailing in different countries of the world. These considerations have led the Sectional Committee to derive assistance from the publications of Institute of Petroleum, United Kingdom.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

*Methods for testing tar and bituminous materials (*first revision*).

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard covers the method of test for the determination of FRAASS breaking point of solid and semi-solid bitumens.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions and those given in IS : 334-1965* shall apply.

2.1 FRAASS Breaking Point — It is the temperature at which bitumen first becomes brittle as indicated by the appearance of cracks when a thin film of the bitumen on a metal plaque is cooled and flexed in accordance with the specified condition.

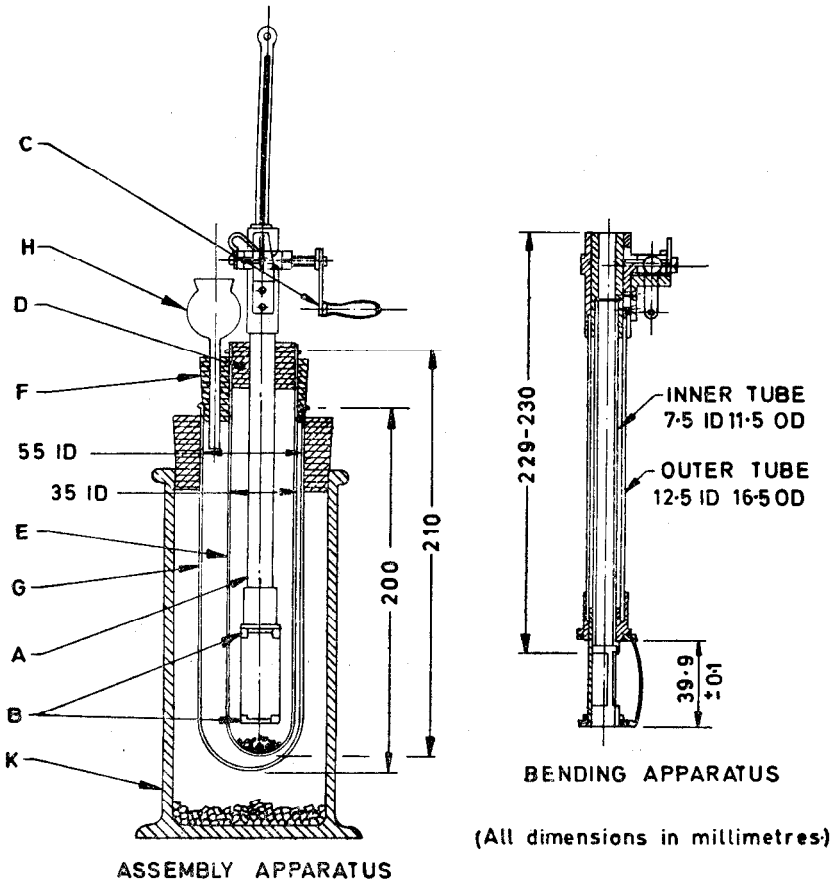
3. APPARATUS

3.1 Bending Apparatus — It consists of two concentric tubes (*A*) of insulating materials, such as pyrex glass or porcelain, at the lower end of which steel clips or jaws (*B*) are tightly fixed. The part of the inner tube which lies between the two clips is slotted so that the bulb of the thermometer, which fits into the inner tube, is exposed. By rotating a handle (*C*) which operates a cone-and-peg mechanism the inner tube may be moved up and down relative to the outer tube, thereby varying the distance between the clips. Ten to twelve rotations of the handle shall decrease the distance between the clips by 3.5 ± 0.2 mm from maximum of 39.9 ± 1 mm (*see* Fig. 1).

3.2 Plaque — It is made of springy steel 41 ± 0.05 mm long, 20 ± 0.2 mm wide and 0.15 ± 0.02 mm thick. The plaque shall be kept flat when not in use.

3.3 Cooling Apparatus — It consists of a wide test-tube (*E*) held eccentrically in a second wider test tube (*G*) by means of a rubber bung which also holds a small funnel (*H*). The larger test-tube is, in turn, held by a large rubber or cork bung in an outer cylinder (*K*). The test-tube (*E*) and the cylinder (*K*) contain a small quantity of calcium chloride or anhydrous. The bending apparatus is supported in (*E*) by means of a rubber bung (*D*). The wider test-tube (*G*) and the outer cylinder (*K*) may be replaced by an unsilvered vacuum flask of suitable dimensions (*see* Fig. 1).

*Glossary of terms relating to bitumen and tar (*revised*).



- | | |
|-------------------------|---------------------|
| A - CONCENTRIC TUBE | F - RUBBER BUNG |
| B - STEEL CLIPS OR JAWS | G - WIDER TEST-TUBE |
| C - ROTATING HANDLE | H - FUNNEL |
| D - RUBBER BUNG | K - CYLINDER |
| E - WIDE TEST-TUBE | |

FIG. 1 APPARATUS FOR FRAASS BREAKING POINT TEST

3.4 Thermometer — A thermometer graduated in Celsius degrees and conforming to the following requirements shall be used:

<i>Characteristic</i>	<i>Requirement</i>
Range	− 38°C to + 30°C
Graduation	0.5°C
Immersion	250 mm
Overall length	370 ± 10 mm
Stem diameter	6.0 to 7.0 mm
Bulb shape	Cylindrical
Bulb length	10 to 16 mm
Bulb diameter	Not greater than stem
Distance of bottom of bulb to the lowest graduation	Not less than 60 mm
Longer graduation line at each	1°C and 5°C
Scale error not to exceed	± 0.5°C

3.5 Plate and Stand — It consists of a metal heating plate approximately 5 mm thick with an iron baffle plate, 1 to 2 mm thick, at a distance of about 50 mm below its upper surface. The plates which are of suitable shape and size, are supported on a stand equipped with levelling screws.

4. PROCEDURE

4.1 Preparation of Test Sample — Soften the material to the pouring consistency at a temperature not more than 60°C for tars and pitches and not more than 90°C for bituminous materials above the respective approximate softening point and stir it thoroughly until it is homogeneous and is free from air bubbles and water. Prepare the convenient number of plaques as given in 4.1.1 and 4.1.2.

4.1.1 For Materials of Softening Point Below 70°C — Place an amount of the sample corresponding to 0.40 ± 0.01 ml in the unheated liquid or solid state on a clean tared plaques. For normal bitumens of specific gravity 1.03 ± 0.4 at 27°C, a weight of 0.4 ± 0.01 g may be used. Place the plaque on heating plate and heat the baffle plate continuously until the bitumen just flows. Manipulate the plaque by hand, replacing on the heating plate, if necessary, until the plaque is uniformly coated. Obtain the final smooth film by replacing the plaque on the heating plate for a short time. Reweigh the plaque when it has cooled.

4.1.2 For Materials of Softening Point Above 70°C — Place an amount of the sample corresponding to 0.40 ± 0.01 ml in the unheated state on a clean tared plaque. The amount of bitumen shall be the same as given in 4.1.1. Place the plaque on the heating plate until the bitumen is soft enough to mould yet not soft enough to stick to the fingers. A rough uneven film is obtained by moulding the bitumen, reheating, if necessary. Obtain the final smooth film by replacing the plaque on the heating plate for a short time. Reweigh the plaque when it has cooled.

For conveniently conducting the test and to make measuring of bitumen more accurate, the prescribed quality of bitumen for each test may either be weighed directly on the steel strip or extruded from a small press shown in Fig. 2. The mould of the press is cylindrical in form, measuring 20 mm in diameter by 20 mm in height, having a slit in the bottom 20 mm long by 0.5 mm wide through which is extruded a 0.4-ml strip of standard dimension.

4.1.3 If necessary, remove bubbles from the surface of the film by shock cooling the heated bitumen and subsequent reheating. The sudden cooling can conveniently be achieved by pressing the heated plaque on the powdered solid carbon dioxide. Protect the plaque from dust and allow it to stand for one to four hours before testing.

4.2 Fill the annular space between wide test-tube (*E*) and eccentric test-tube (*G*) to about half its height with acetone. Place the plaque between the clips of the bending apparatus, bending the plaque gently to do so, and mount the bending apparatus in the wide test-tube (*E*). Add solid carbon dioxide through the funnel to the acetone at such a rate that the temperature falls at a rate of 1°C per minute. Commencing at a temperature of at least 10°C above the expected breaking point, bend the plaque once every minute by turning the handle at a rate of one rev/sec until it is checked and then turning it backwards at the same speed. Record the temperature at which one or more cracks appear on bending as the breaking point.

5. REPORTING

5.1 Calculate the mean of three determinations which lie between a range of 3°C, and report it to the nearest 1°C as the breaking point.

6. PRECISION

6.1 The results of duplicate tests shall not differ by more than the following:

Repeatability

2°C

Reproducibility

Not established

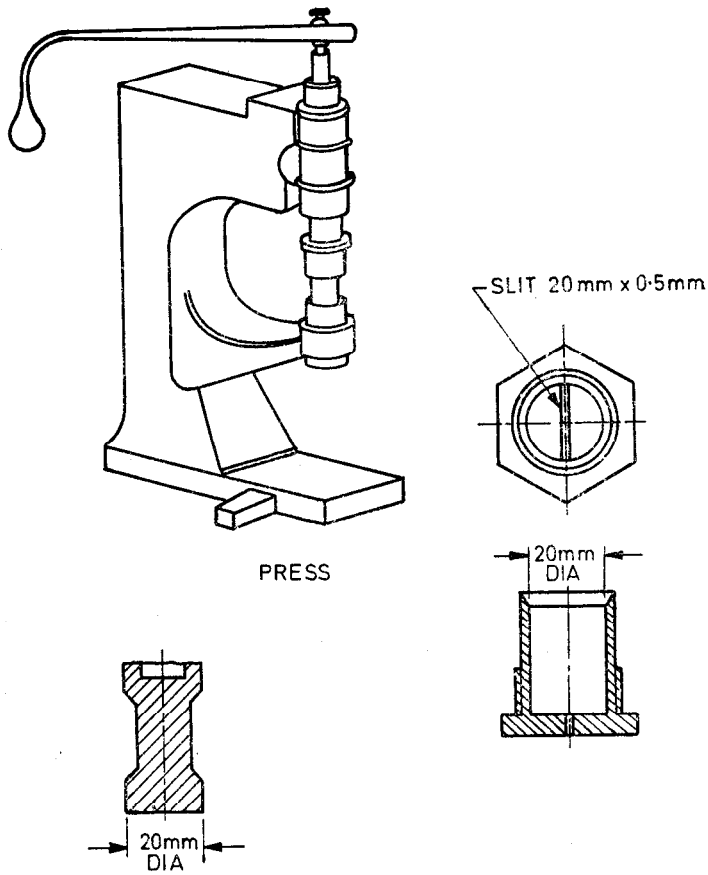


FIG. 2 MOULD OF FRAASS BREAKING POINT APPARATUS

7. PRECAUTIONS

- 7.1 Rate of cooling should be strictly adhered to.
- 7.2 The apparatus should be protected from draught of air.

(Continued from page 2)

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INDIAN STANDARDS
ON
BITUMEN AND TAR PRODUCTS

IS:

- 73-1961 Paving bitumen (*revised*)
- 212-1961 Crude coal tar for general use (*revised*)
- 215-1961 Road tar (*revised*)
- 216-1961 Coal tar pitch (*revised*)
- 217-1961 Cutback bitumen (*revised*)
- 218-1961 Creosote and anthracene oil for use as wood preservatives (*revised*)
- 334-1965 Glossary of terms relating to bitumen and tar (*revised*)
- 454-1961 Digboi type cutback bitumen (*revised*)
- 702-1961 Industrial bitumen (*revised*)
- 1201-1978 Methods for testing tar and bituminous materials: Sampling (*first revision*)
- 1202-1978 Methods for testing tar and bituminous materials: Determination of specific gravity (*first revision*)
- 1203-1978 Methods for testing tar and bituminous materials: Determination of penetration (*first revision*)
- 1204-1978 Methods for testing tar and bituminous materials: Determination of residue of specified penetration
- 1205-1978 Methods for testing tar and bituminous materials: Determination of softening point (*first revision*)
- 1206 (Part I)-1978 Methods for testing tar and bituminous materials: Determination of viscosity Part I — Industrial viscosity (*first revision*)
- 1206 (Part II)-1978 Methods for testing tar and bituminous materials: Determination of viscosity Part II — Absolute viscosity (*first revision*)
- 1206 (Part III)-1978 Methods for testing tar and bituminous materials: Determination of viscosity Part III — Kinematic viscosity (*first revision*)
- 1207-1978 Methods for testing tar and bituminous materials: Determination of equivalent Temperature (EVT) (*first revision*)
- 1208-1978 Methods for testing tar and bituminous materials: Determination of ductility (*first revision*)
- 1209-1978 Methods for testing tar and bituminous materials: Determination of flash point and fire point (*first revision*)
- 1210-1978 Methods for testing tar and bituminous materials: Float test (*first revision*)
- 1211-1978 Methods for testing tar and bituminous materials: Determination of water content (Dean and stark method) (*first revision*)
- 1212-1978 Methods for testing tar and bituminous materials: Determination of loss on heating
- 1213-1978 Methods for testing tar and bituminous materials: Distillation test (*first revision*)
- 1214-1978 Methods for testing tar and bituminous materials: Determination of matter insoluble in benzene (*first revision*)
- 1215-1978 Methods for testing tar and bituminous materials: Determination of matter insoluble in toluene
- 1216-1978 Methods for testing tar and bituminous materials: Determination of solubility in carbon disulphide or tri-chloroethylene (*first revision*)
- 1217-1978 Methods for testing tar and bituminous materials: Determination of mineral matter (ash) (*first revision*)
- 1218-1978 Methods for testing tar and bituminous materials: Determination of Phenols (*first revision*)
- 1219-1978 Methods for testing tar and bituminous materials: Determination of naphthalene (*first revision*)
- 1220-1978 Methods for testing tar and bituminous materials: Determination of volatile matter content
- 3117-1965 Bitumen emulsion for roads (anionic type)
- 8887-1978 Bitumen emulsion for roads (cationic type)

AMENDMENT NO. 1 JUNE 2000
TO
IS 9381 : 1979 METHODS FOR TESTING TAR AND
BITUMINOUS MATERIALS: DETERMINATION OF
FRAASS BREAKING POINT OF BITUMEN

(*Page 4, clause 1.1*) — Insert the following note after clause 1.1:

'NOTE — Any other instrumental method simulating the manual method may be employed.
However, this method shall be the referee method.'

(PCD 6)

Reprography Unit, BIS, New Delhi, India