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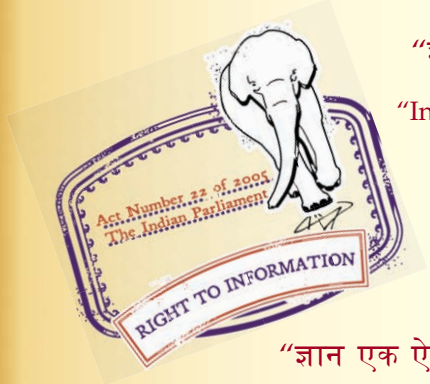

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IS : 9382 - 1979
(Reaffirmed 1999)

Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS: DETERMINATION OF EFFECT OF HEAT AND AIR BY THIN FILM OVEN TEST

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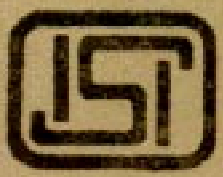

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METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS: DETERMINATION OF EFFECT OF HEAT AND AIR BY THIN FILM OVEN TEST

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(*Continued on page* 8)

Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS: DETERMINATION OF EFFECT OF HEAT AND AIR BY THIN FILM OVEN TEST

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 the Petroleum, Coal and Related Products Division Council.

0.2 A compilation of methods of tests for testing tar and bituminous materials for their 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 for determination of effect of heat and air on bituminous materials when heated to a standard temperature under specified conditions.

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 publication of American Society for Testing and Materials, Philadelphia.

0.4 In reporting the results 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†.

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*Methods for testing tar and bituminous materials (*first revision*).
†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard covers determination of the effect of heat and air on semi-solid bituminous materials by thin film oven test.

2. TERMINOLOGY

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

2.1 Effect of Heat and Air — It is the effect of heat and air on bituminous materials when heated to a standard temperature under specified conditions. The amount of hardening is evaluated from the reduction in penetration expressed as the percentage of the original value.

3. APPARATUS

3.1 Oven — A double walled chamber, rectangular in form, interior dimensions not being less than 290 mm in height from the top of the heating element to the top of the chamber and not less than 300 mm in width and depth. The chamber is provided on one side with a tightly fitted hinged door containing a double glass window, at least 100 mm square, through which the thermometer may be read without opening the door. Ventilation of the oven is provided by means of either one round vent-hole in the top and one in the bottom of the oven, or one round vent-hole near the top and one near the bottom on each of the two sides of the oven, the diameter of each vent-hole being 12 to 16 mm. Heating of the oven shall be done electrically and the temperature is maintained within the limits specified for the test.

3.2 Perforated Metal Shelf — A perforated metal shelf, approximately 250 mm in diameter; and aluminium shelf of the form and dimensions as shown in Fig. 1 of IS : 1212-1978† is recommended. The shelf is placed in the centre of the oven with respect to all dimensions of the interior of the oven, and is suspended by a vertical shaft and provided with mechanical means for rotation at the rate of 5 to 6 rev/min.

NOTE — Ovens of minimum size accommodate two containers. For routine or control operations, larger ovens, having proportionately larger shelves to accommodate a greater number of containers, may be advantageous and are suitable provided they meet the requisite specification requirements.

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

†Methods for testing tar and bituminous materials: Determination of loss on heating (*first revision*).

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

Characteristic	*Requirement*
Liquid	Mercury
Filling above liquid	Nitrogen gas
Temperature range	150 to 170°C
Sub-divisions	0·5°C
Longer graduation lines at each	1°C
Graduations numbered at	0, 155, 163 and 170°C
Immersion	Total
Total length	150 to 154 mm
Bulb length	10 to 15 mm
Bulb diameter	Not larger than stem diameter
Stem diameter	6·0 to 7·0 mm
Distance of bottom of bulb to graduation line:	
at 0°C	33 to 38 mm
at 155°C	70 to 80 mm
Distance of top of thermometer to graduation line at 170°C	20 to 30 mm
Top finish	Glass ring
Scale error at any point when standardized, *Max*	0·5°C

3.3.1 The top of the contraction chamber shall not be more than 41 mm above the bottom of the bulb.

3.3.2 The expansion chamber shall permit heating the thermometer at least 25°C above highest temperature on the scale.

3.3.3 At the ice point, the thermometer shall be graduated from −1°C to +1°C in 0·5°C divisions.

3.3.4 The thermometer shall be supported from the shaft of the metal shelf in a vertical position approximately 20 mm inside the periphery of the shelf with the bottom of the bulb 6 mm above the shelf.

3.4 Container — A cylindrical pan, 140 mm inside diameter and 10 mm deep with a flat bottom. This shall be made of aluminium or stainless steel approximately 0·76 mm and 0·64 mm thick respectively. Fifty millilitres of the sample in this size container give a film thickness of approximately 3·2 mm.

4. PROCEDURE

4.1 Stir and agitate thoroughly the material as received, warm if necessary, to ensure a complete mixture before a portion is removed for the test.

4.2 Heat the container in an oven at 100 to 110°C for 30 minutes, cool and weigh. From its specific gravity at 27/27°C, calculate the weight of the material equivalent to 50·0 ml. Weigh this amount to ± 0·001 g into each of two separate containers.

4.3 At the same time, pour a portion of the sample into the specified container for determination of penetration as described in IS : 1203-1978*.

4.4 Bring the oven to a temperature of 163 ± 1°C and place the sample container in the revolving shelf. Close the oven and rotate the shelf during the entire test at a rate of 5 to 6 rev/min, the temperature being maintained at 163 ± 1°C for 5 hours after the sample has been introduced and the oven has again reached the temperature.

The 5-hour period shall start when the temperature reaches 162°C and in no case shall the total time, during which the sample is in the oven, be more than 5 hour 15 minutes. At the end of the specified heating period remove the container from the oven. If the loss is not being determined, proceed in accordance with **4.6**. If the loss is being determined, cool to room temperature, and weigh to the nearest 0·001 g and calculate the loss due to heating.

4.5 After weighing the two samples, place them on a piece of asbestos cement board. Put the board and the containers on the circular shelf of the oven maintained at 163 ± 1°C. Close the oven and rotate the shelf for 15 minutes, remove the samples and the plate.

4.6 Pour both samples into a 225-g ointment tin. Remove all the material from 140 mm pans by scrapping with a suitable spatula or putty knife. Stir the combined residues thoroughly, placing the 225-g container on a hot plate to maintain the material in a fluid condition, if necessary. Pour the material in a proper container. Determine the penetration of the residue as prescribed in IS : 1203-1978*.

*Methods for testing tar and bituminous materials: Determination of penetration (*first revision*).

5. REPORTING

5.1 Report the value of the original penetration, penetration of the residue and penetration of the residue expressed as the percentage of the original penetration.

5.2 When determined, report the average loss of the material in the two containers as the percent by mass of the original material.

6. PRECISION

6.1 Results in duplicate tests shall not differ by more than the following:

Test	*Repeatability*	*Reproducibility*
Percentage of retained penetration	2·90	8·0
Change in mass percentage not more than 0·4 percent	0·05	0·16
Change in mass percentage greater than 0·4 percent	14 percent of the mean value	40 percent of the mean value

7. PRECAUTION

7.1 Conduct the loss on heating test in duplicate.

7.2 Determine the water content of the sample in accordance with the method for determination of water content specified in IS : 1211-1978*.

7.3 If the sample contains water, test it in that condition.

7.4 Reject the test during which samples show evidence of loss by foaming.

7.5 The pan's bottom should be maintained flat. Results are affected if the bottoms of the pans are not flat. Frequent inspection to eliminate warped or damaged pans is advisable.

*Methods for testing tar and bituminous materials: Determination of water content (Dean and Stark method) (*first revision*).

(*Continued from page* 2)

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AMENDMENT NO. 1 NOVEMBER 2004
TO
IS 9382 : 1979 METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS: DETERMINATION OF EFFECT OF HEAT AND AIR BY THIN FILM OVEN TEST

(*Page* 6, *clause* **4.2**) — Substitute the following for the existing:

'**4.2** Heat the container in an oven at 100° to 110°C for 30 minutes. Cool and weigh. Weigh into the container 50.0 ± 0.5 g of the material.

4.2.1 If the quantitative value of the mass change is desired, cool the samples for the oven test to room temperature and weigh each sample separately to the nearest 0.001 g. If the mass change is not required, allow the samples to cool to room temperature before placing in the oven.'

(*Page* 7, *clause* **6.1**) — Substitute the following for the existing:

6.1 Results in duplicate tests shall not differ by more than the following:

Test	*Repeatability*	*Reproducibily*
Percentage of retained penetration	4.0	8.0
Change in mass percentage, not more than 0.4 percent	0.05	0.16
Change in mass percentage, greater than 0.4 percent	14 percent of the mean value	40 percent of the mean value

NOTE — Percentage retained penetration shall be calculated by the following equation:

$$\text{Percentage retained penetration} = \frac{\text{Penetration before TFOT} - \text{penetration after TFOT} \times 100}{\text{Penetration before TFOT}}$$

(PCD 6)

Reprography Unit, BIS, New Delhi, India