भारतीय मानक Indian Standard

> टार और बिटुमिनस सामग्री के परीक्षण की विधियाँ — नेफ़थलीन का निर्धारण (दूसरा पुनरीक्षण)

Methods for Testing Tar and Bituminous Materials — Determination of Naphthalene

(Second Revision)

ICS 75.14

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भारतीय मानक ब्यूरो BUREAU OF INDIAN STANDARDS मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002 MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI - 110002 www.bis.gov.in www.standardsbis.in

Price Group 5

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Product Division Council.

This standard was first published in 1958 as 'Methods for testing tar and bituminous materials — Determination of naphthalene'. The first revision was carried out in 1978 'Methods for testing tar and bituminous materials' was published as series of 22 standards in the form of a booklet, as listed below:

IS No.	Title
IS 1201 : 1978	Sampling
IS 1202 : 1978	Determination of specific gravity
IS 1203 : 1978	Determination of penetration
IS 1204 : 1978	Determination of residue of specified penetration
IS 1205 : 1978	Determination of softening point
IS 1206	Determination of viscosity:
(Part 1): 1978	Industrial viscosity
(Part 2) : 1978	Absolute viscosity
(Part 3) : 1978	Kinematic viscosity
IS 1207 : 1978	Determination of equiviscous temperature (EVT)
IS 1208 : 1978	Determination of ductility
IS 1209 : 1978	Determination of flash point and fire point
IS 1210 : 1978	Float test
IS 1211 : 1978	Determination of water content dean and stark method
IS 1212 : 1978	Determination of loss on heating
IS 1213 : 1978	Distillation test
IS 1214 : 1978	Determination of matter insoluble in benzene (withdrawn due to toxic nature of
	benzene)
IS 1215 : 1978	Determination of matter insoluble in toluene
IS 1216 : 1978	Determination of solubility in carbon disulphide or trichloroethylene
IS 1217 : 1978	Determination of mineral matter ash
IS 1218 : 1978	Determination of phenols
IS 1219 : 1978	Determination of naphthalene
IS 1220 : 1978	Determination of volatile matter content

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian standards separately for each test so as to make it user friendly.

This revision has been taken up to keep pace with the latest technological development and international practices. In this revision no major changes have been made.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

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 : 2022 'Rule for rounding off numerical values (*second revision*)'.

Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS — DETERMINATION OF NAPHTHALENE

(Second Revision)

1 SCOPE

1.1 This standard prescribes the method for determination of naphthalene in road tar.

1.2 This determination is necessary only in respect of the naphthalene deposited at 15.5 °C from solution in the total distillate up to 270 °C.

2 REFERENCES

The standards contain provisions which through reference in the text constitute provisions of this standard, at the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	Title
IS 265 : 2021	Hydrochloric acid —
IS 334 : 2023	Specification (<i>fifth revision</i>) Glossary of terms relating to
	bitumen and tar
	(fourth revision)
IS 1213 : 2020	Methods for testing tar and
	bituminous materials —
	Distillation test (second
	revision)
IS 1218 : 2024	Methods for testing tar and
	bituminous materials —
	Determination of phenols
	(first revision)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 334 shall apply.

4 PROCESS

The distillation fraction of tar distilling up to 270 °C left after the removal of phenols is cooled to 15.5 °C \pm 0.5 °C and the solid separated and weighed.

5 APPARATUS

5.1 Crystallizing-Point Apparatus — see 7.1

5.2 Buchner Funnel and Flask

- **5.3 Hand Screw Press**
- 5.4 Thermometer
- 5.5 Blotting Paper
- 5.6 A Filter Pump

6 PROCEDURE

6.1 Collect the distillate recovered up to 270 °C according to Method B of IS 1213. This shall be washed free from phenols (*see* IS 1218). Cool the residue to 15.5 °C \pm 0.5 °C and maintain it with occasional stirring within these limits of temperature for 2 h. Naphthalene crystallizes out.

6.2 Transfer the cooled material to a cold Buchner funnel fitted with a rapid filter proper, and rapidly remove as much oil as possible using a filter pump. Place the crude naphthalene between sheets of blotting paper and press in a hand screw press. Remove the oily margins of the cake, and again press separately. Conduct the operations, starting with the transfer to the Buchner funnel to the preliminary pressing, with utmost rapidity.

6.3 Weigh the pressed naphthalene and calculate the percentage by weight in the original tar.

6.4 Determine the corrected wet crystallizing-point of the pressed naphthalene by the method described in $\underline{7}$.

7 CORRECTED WET CRYSTALLIZING POINT

7.1 Apparatus

A crystallizing-point apparatus of the shape, dimensions and tolerances given in <u>Fig. 1</u>, consisting of the following.

7.1.1 Outer Glass Test Tube

It serves as an air jacket, shall be weighed with lead shots or similar loading material. It is provided with a cork through which the inner tube, as described in 7.1.2, is held in position.

IS 1219 : 2024

7.1.2 Inner Glass Test Tube

Approximately 25 mm in diameter, closed by means of a cork which carries a stirrer in the form of a loop of glass with a glass stem and a standard thermometer (see 7.1.4) placed centrally within the tube and the glass loop and the bottom of the bulb being about 10 mm from the bottom of the inner tube. The cork is so fixed that the immersion mark on the thermometer is level with the top of the cork.

7.1.3 Cooling Bath

A glass beaker of 1 000 ml capacity and 150 mm in height with the level of the liquid at least as high as the level of the sample in the inner tube.

7.1.4 *Thermometer* — With the following dimensions and characteristics:

Range	65 °C to 90 °C	
Graduation at each	0.1 °C	
Longer lines at each	0.5 °C and 1.0 °C	
Fully figured at each	2 °C	
Immersion	100 mm	
Maximum overall length	400 mm	
Maximum length of main scale	215 mm	
Maximum bulb length	12 mm	
Maximum distance from bottom of bulb to top of contraction chamber	25 mm	
Limit of accuracy	0.05 °C	

7.2 Material for Test

While the difference between the crystallizing point of dry naphthalene and of naphthalene. Containing a small amount of water is appreciable, the lowering of the crystallizing-point ceases when a minimum of two percent of water is present. Therefore, if the samples should contain two percent or more of water, it shall be examined as received. If the sample should contain a smaller percentage of water, 0.5 ml of water shall be placed in the inner tube of the crystallizing-point apparatus before the sample is introduced as described under <u>7.3</u>. If the test is carried out in the presence of two percent or more of water, the addition of 0.85 °C to the observed crystallizing-point gives the crystallizing-point of the material on the dry basis.

7.3 Procedure

Place about 40 g of the material for test in a loosely stoppered conical flask which has been warmed in a boiling water bath to 85 °C. Mix the contents thoroughly and pour about 20 g into the warmed inner tube of the crystallizing-point apparatus. Place the tube in its jacket and assemble the apparatus as shown in Fig. 1 with the bath 6 °C to 8 °C below the expected crystallizing-point. Do not heat the water during the subsequent operations. Take the bath thermometer readings at intervals of half a min, starting when the temperature has fallen to 81 °C. Stir the contents of the inner tube continuously.



FIG. 1 CRYSTALLIZING-POINT-ASSEMBLY OF APPARATUS

7.4 Results

7.4.1 The crystallizing-point corresponds to the first five consecutive readings during which the temperature remains constant within 0.05 °C. Super cooling may occur, in which case the five readings may be observed after the temperature rise. A temperature rise of 1 °C shall be regarded as the maximum allowable.

7.4.2 If a constant temperature within 0.05 °C is not obtained over five readings, take six final readings commencing with the first of two successive readings within 0.05 °C. Plot the readings on graph paper against time intervals, and draw a straight line to lit evenly between the first and second, and between the fifth and the sixth of the six points just mentioned. Produce the line backwards until it intersects the earlier portion of the curve.

7.5 Report

7.5.1 Report the constant temperature within 0.05 °C (see 7.4.1) or the point of intersection as found in 7.4.2, as the crystallizing-point.

7.5.2 Report as corrected wet crystallizing-point the value obtained after adding 0.85 °C to the observed crystallizing-point.

7.5.3 The corrected wet crystallizing-point shall be not lower than 70 °C. If it is found to be below 70 °C, the indication is that the removal of the excessive oil as prescribed in <u>6.2</u> has not been carried out satisfactorily and the determination has

to be repeated.

8 PRECISION

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

Repeatability0.1 °CReproducibility0.8 °C

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

Organization

Represenative(s)

CSIR - Central Road Research Institute, New Delhi

Airports Authority of India, New Delhi

Bharat Petroleum Corporation Limited Corporate Research & Development Centre, Greater Noida

Bharat Petroleum Corporation Limited, Mumbai

Birla Institute of Technology and Science, Pilani

Central Public Works Department, New Delhi

Chennai Petroleum Corporation Limited, Chennai

CSIR - Central Building Research Institute, Roorkee

CSIR - Central Road Research Institute, New Delhi

CSIR - Indian Institute of Petroleum, Dehradun

Dilip Buildcon Limited, Bhopal

Directorate General of Quality Assurance, Ministry of Defence, Kanpur

GP Global Asphalt Private Limited, New Delhi

Highways Research Station, Chennai

Hindalco Industries Limited, Mumbai

Hindustan Colas Private Limited, Mumbai

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Hindustan Petroleum Corporation Limited, Mumbai

Indian Institute of Technology Delhi, New Delhi

Indian Institute of Technology Guwahati, Guwahati

Indian Institute of Technology Madras, Chennai

Indian Institute of Technology Roorkee, Roorkee

Indian Oil Corporation Limited - Refineries and Pipelines Division, New Delhi

Indian Oil Corporation (R and D Centre), Faridabad

Indian Road Congress, New Delhi

Indian Oil Total Private Limited, Mumbai

Mangalore Refinery and Petro Chemical Limited, Mangalore

Ministry of Road Transport and Highways, New Delhi

National Highways Authority of India, New Delhi

National Institute of Technology, Warangal

National Test House, Kolkata

Nayara Energy Limited, Mumbai

Ooms Polymer Modified Bitumen Private Limited, Gurugram

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Member Secretary Shri Hari Mohan Meena Scientist 'C'/Deputy Director (Petroleum, Coal and Related Products), BIS this Page has been intertionally left blank

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Amendments Issued Since Publication

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