भारतीय मानक
Indian Standard

IS 18690 : 2024

टार और बिटुमिनस सामग्री — संतृप्त, नैफ्थीन एरोमेटिक्स, ध्रुवीय एरोमेटिक्स और एस्फाल्टीन का निर्धारण — परीक्षण पद्धतियाँ

Tar and Bituminous Materials — Determination of Saturates, Naphthene Aromatics, Polar Aromatics and Asphaltenes — Methods of Test

ICS 75.14

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August 2024 **Price Group 5**

Bitumen, Tar and Related Products Sectional Committee, PCD 06

FOREWORD

This Indian Standard 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 Products Division Council.

This standard provides a tool for separation of asphalts or bitumen into four fractions that is, saturates aromatic, resin and asphaltenes. The test methods specified in this standard are mainly applicable for determination of saturates, napthene aromatics, polar aromatics and asphaltene.

Considerable assistances have been taken from ASTM D 4124 – 09 (2018) 'Standard test method for separation of asphalt into four fractions' and research done by Indian Institute of Petroleum, Dehradun.

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 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

TAR AND BITUMINOUS MATERIALS — DETERMINATION OF SATURATES, NAPHTHENE AROMATICS, POLAR AROMATICS AND ASPHALTENES — METHODS OF TEST

1 SCOPE

This standard prescribes method for separation and determination of asphalt or bitumen into four fractions saturates aromatics, resins, and asphaltenes. These test method, can also be employed for petroleum products asphalt, bitumen, vacuum gas oil (VGO), lubricating oils, and other related products.

2 REFERENCE

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

IS No.	*Title*
IS 10511 : 2024	Determination of asphaltene in bitumen by precipitation with *n*-heptane

3 TERMINOLOGY

3.1 Petroleum Product — Bitumen, petroleum-derived asphalt, vacuum gas oil (VGO), and lubricating oils are defined as petroleum products.

3.2 Asphaltene — The matter insoluble in *n*-alkane (*n*-heptane) and branched alkanes (isooctane) and, in some cases, under the standard test conditions.

3.3 Maltene — Matter soluble in *n*-alkane under the standard test conditions is maltene (petrolene).

3.4 Saturates — The material on discharge in *n*-alkane and desorbed on activated adsorbent (alumina) under the standard test conditions are termed as saturates.

3.5 Naphthene Aromatics (Cyclo Saturates) — The material obtained after removing saturates and permeating in *n*-alkane and desorbed by toluene on activated adsorbent (alumina) under the standard test conditions is termed as naphthene aromatics.

3.6 Polar Aromatics (Resins) — The material obtained after removal of saturates and naphthene aromatics and permeate in *n*-alkane and desorbed by trichloroethylene on activated adsorbent (alumina) under the standard test conditions is termed as polar aromatics.

4 APPARATUS

4.1 Reflux Unit — for asphaltenes separation from petroleum products (*see* Fig. 1)

4.2 Glass Column — having dimension 75 cm × 1.5 cm

4.3 General Laboratory Glass Ware

4.4 Balance — capable to weigh with least count 0.001 g

5 REAGENTS

5.1 Alumina (Aluminium oxide) — CG-20 chromatographic grade

5.2 *n*-Heptane — HPLC grade

5.3 Toluene — HPLC grade

5.4 Methanol — HPLC grade

5.5 Trichloroethylene — ACS grade (boiling point 86.5 °C to 87.5 °C)

5.6 Iso-octane — HPLC grade

6 PROCEDURE

The test procedure is subdivided into two parts, that is, separation of petroleum product into asphaltene and maltene (petrolene).

6.1 Separation of Petroleum Product into Asphaltenes and Maltene (Petrolene)

The asphaltenes content of bitumen is the percentage by mass of wax-free material insoluble in *n*-heptane but soluble in benzene or toluene (heat if needed). The material is dissolved in *n*-heptane, and insoluble

material consisting of asphaltenes and waxy substances is separated by filtration through a fine filter paper. The waxy constituents are extracted under hot reflux with *n*-heptane, and the asphaltenes are isolated by extraction with benzene or toluene.

NOTE — 42 Whatman filter paper is suitable for filtration.

6.1.1 Before starting the procedure, the petroleum product sample should be made free from foreign materials by filtering it using sieve no. 600 microns, if required.

6.1.2 Follow the test method as prescribed in IS 10511 for the determination of asphaltenes in bitumen by precipitation with normal heptane.

6.2 Separation of Maltene into Saturates, Naphthene Aromatics and Polar Aromatics

6.2.1 Before separating maltene into three defined fractions, the chromatographic glass column (burette) is gently packed with activated alumina (*see* Fig. 2. Ensure that column is packed with alumina ½ inchbelow the top of the cylindrical mouth of the column. Also, ensure that before filling alumina, put some dry and clean cotton at the bottom of the column.

6.2.2 Fill about 110 g the calcined alumina into the chromatographic column with the help of a small funnel and use a rubber cork to gently tap the external wall of the glass column to settle and pack the alumina uniformly.

NOTE — Alumina should be calcined at 425 °C for 16 h to drive off all chemically combined moisture or water, and the same should be stored in an evacuated desiccator in the airtight bottle for 5 h.

6.2.3 Pour 70 ml of *n*-heptane to prewet the column and collect the eluting solvent in the graduated cylinder to ensure the volume of the eluting solvent. Adjust the flow rate of eluting solvent to 3.0 ml/min. Once the flow rate is adjusted, put 100 ml graduate cylinder to collect the excess solvent before pouring the sample into the column. Also, ensure the column temperature 25 °C for maintaining the standard flow rate throughout the experiment.

6.2.4 Take nearly (1.000 ± 0.001) g concentrated maltene obtained in section **6.1** and pour it into the chromatographic glass column with funnel. Sample size on the given size of chromatographic column is mentioned in Table 1.

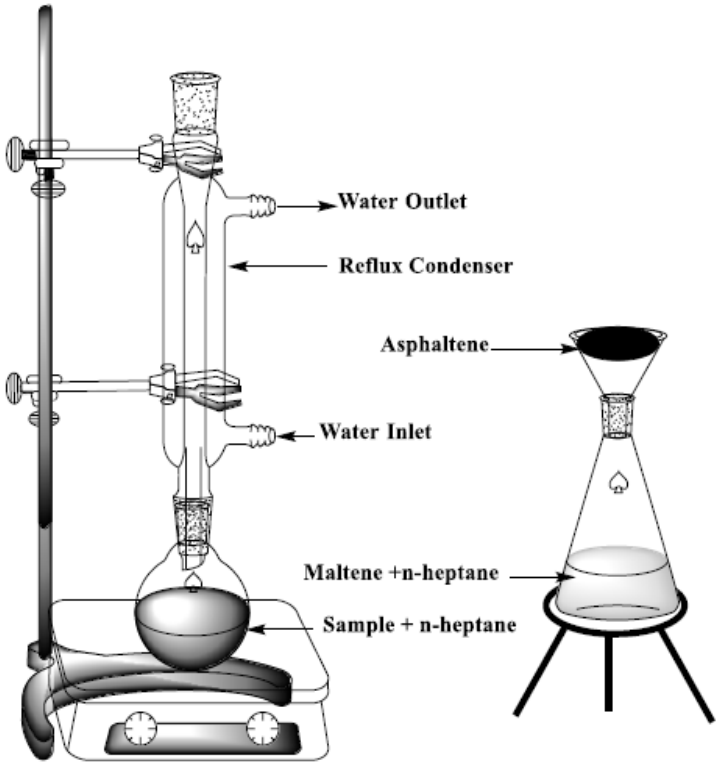

FIG. 1 SCHEMATIC FOR SEPARATION OF ASPHALTENE FRACTION FROM THE SAMPLE

Table 1 Reference Table for Sample Size, the Volume of the Eluting Solvent, and Cut Point

(*Clause* 6.2.4)

Sl No.	Eluting Agent	Volume	Eluted Fraction	Volume Recovered
		ml		ml (Approx)
(1)	(2)	(3)	(4)	(5)
i)	*n*-Heptane	150	Saturates (S)	183
ii)	Toluene	33		—
iii)	Toluene	67	Naphthene aromatics (NA)	142
iv)	Methanol/Toluene	75		—
v)	Trichloroethylene	150	Polar aromatics (PA)	150

NOTE — Solvent volumes mentioned in Table 1 are for a 1.0 g sample size (mass of maltene)

6.2.5 When all the standard conditions are stabilized, like prewetting of the column, flow rate, and temperature, pour the concentrated maltene sample (1.000 ± 0.001 g) to be separated into three defined fractions into the column with the help of a funnel and rinse down completely the flask containing remaining maltene sample with 10 ml of *n*-heptane onto the column. Replace the 100 ml graduated cylinder with a 500 ml Erlenmeyer flask to collect the eluting solvent containing saturates (the first defined fraction of maltene). Add 150 ml *n*-heptane and 33 ml toluene and collect the volume of eluting solvent (150 ml + 33 ml = 183 ml) in the Erlenmeyer flask and mentioned it as saturates (S).

NOTE — Eluate containing saturates fraction is transparent or clear in color.

6.2.6 After eluting the 183 ml (150 ml *n*-heptane + 33 ml toluene), the cut point is made between saturates and naphthene aromatics by observing the movement of fluorescent band shined in the column. This band rises upward as the second eluting solvent (toluene) is entered into the column. Before adding the second eluting solvent, replace the 500 ml Erlenmeyer flask (containing saturates fraction) by another 250 ml graduated cylinder to collect the second defined SARA fraction (naphthene aromatics).

6.2.7 After recovering saturates, add 67 ml of toluene and add 75 ml methanol/toluene (50 : 50) subsequently. Hence, further adjust the cut point between naphthene aromatics and polar aromatics to collect the naphthene aromatics while the fluorescent band is approximately 2 inch below the top of the alumina bed. Note and collect 142 ml (67 ml + 75 ml) in the 250 ml graduated cylinder and mentioned as naphthene aromatics. Before adding the next eluant solvent, replace this 250 ml graduated cylinder containing naphthene aromatics (NA) by another 250 ml graduated glass cylinder to collect the last fraction of maltene.

NOTE — Eluate containing the naphthene aromatics is light yellow in color.

6.2.8 To elute the last remained fraction (polar aromatics), add 150 ml of trichloroethylene into the column. As the solvent reaches from top to bottom of the column, it carries all polar aromatics with itself and collect the same 150 ml solvent in a 250 ml graduated cylinder and this eluted solvent containing the last fraction of maltene and treated as polar aromatics.

NOTES

1 The cut point between the naphthene and polar aromatics is quite noticeable in comparison of the cut point between the saturates and naphthene aromatics. This is because in case of the separation between naphthene aromatics and polar aromatics the appearance of dark band (polar aromatics) moving upwards in column and goes down when trichloroethylene is added onto the column.

2 The confirmation of cut points between the fractions naphthene aromatics and polar aromatics may be further checked and analyzed with the use of ultra-violet (UV) spectroscopy. Peak absorbance at 350.35 nm is corresponds to naphthene aromatics and peak absorbance at 400.34 nm is corresponds to polar aromatics. The sharpness in peak intensity confirms the accuracy and precession in cut points separation between naphthene and polar aromatics also.

6.2.9 Collect all three eluated specified fractions — Saturates (*see* **6.2.5**), naphthene aromatics (*see* **6.2.7**), and polar aromatics (*see* **6.2.8**) separately. Heat the samples in an inert nitrogen environment to drive off the solvent from each eluated fraction.

6.2.10 Dry each fraction to a constant mass on an oil bath rotating evaporator (22 cm Hg vacuum, 120 °C, 30 min to 60 min). After allowing samples to cool, compute the mass percent of all three defined fractions as given in the formula (*see* **7**), and then note the sample mass by subtracting the vial mass.

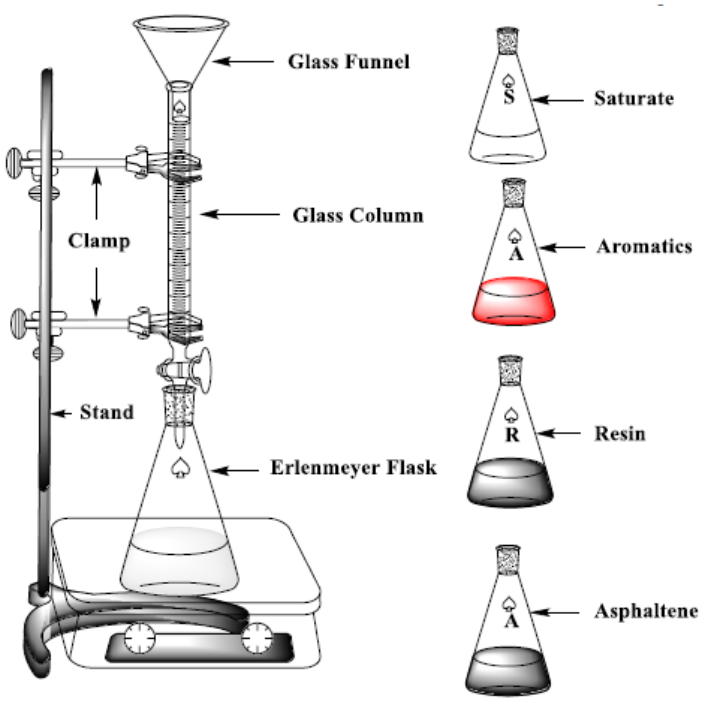

FIG. 2 SCHEMATIC FOR COLUMN CHROMATOGRAPHY FOR SEPARATION OF MALTENE FRACTIONS

7 CALCULATION

Sum of percentage of S + NA + PA +AT = 100

where

$$\text{Saturate } (S), \text{ percent by mass} = \frac{\text{Mass of saturates (g)}}{\text{Sample Mass (g)}} \times 100$$

$$\text{Naphthene aromatics } (NA), \text{ percent by mass} = \frac{\text{Mass of naphthene (g)}}{\text{Sample mass (g)}} \times 100$$

$$\text{Polar aromatics (PA), percent by mass} = \frac{\text{Mass of polar aromatics (g)}}{\text{Sample mass (g)}} \times 100$$

$$\text{Asphaltene } (AT), \text{ percent by mass} = \frac{\text{Mass of asphaltene (g)}}{\text{Sample mass (g)}} \times 100$$

8 REPORT

Report mass percentage of each fraction to the nearest 0.1 percent mass.

9 REPEATABILITY

Duplicate test results by the same operator should not differ by more than ± 2 percent.

10 PRECAUTION

Most of the chemicals used in this test method are toxic and flammable. So, all precautionary measures should be taken as per IS 10511. Along with this reference should be made to material safety data sheets available from the chemical supplier.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

Organization	*Represenative(s)*
CSIR - Central Road Research Institute, New Delhi	PROF MANORANJAN PARIDA **(*Chairperson*)**
Airports Authority of India, New Delhi	SHRI VINOD KUMAR SHARMA SHRI SUPRIO GOSH (*Alternate*)
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Member Secretary
SHRI HARI MOHAN MEENA
SCIENTIST 'C'/DEPUTY DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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