IS : 10511 - 1983

(Reaffirmed 1999)

Indian Standard

METHOD FOR DETERMINATION OF ASPHALTENES IN BITUMEN BY PRECIPITATION WITH NORMAL HEPTANE

UDC 665·775·534 [549·883]

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

August 1983

Indian Standard

METHOD FOR DETERMINATION OF ASPHALTENES IN BITUMEN BY PRECIPITATION WITH NORMAL HEPTANE

Bitumen and Tar Production Sectional Committee, BCDC 2

Chairman	*Representing*
PROF C. G. SWAMINATHAN	Central Road Research Institute (CSIR), New Delhi; *and* Indian Roads Congress, New Delhi
Members	
SHRI Y. C. GOKHALE (*Alternate* to Prof C. G. Swaminathan)	Central Road Research Institute (CSIR), New Delhi
SHRI N. SIVAGURU (*Alternate* to Prof C. G. Swaminathan)	Indian Roads Congress, New Delhi
SHRI N. C. CHATTERJEE	National Test House, Calcutta
SHRI J. K. CHARAN	Engineer-in-Chief's Branch, Army Headquarters
LT-COL C. T. CHARI (*Alternate*)	
DEPUTY DIRECTOR (MET)-5	Ministry of Railways
ASSISTANT RESEARCH OFFICER (CHEM)-2, RDSO, LUCKNOW (*Alternate*)	
SHRI G. C. GOSWAMI	Indian Oil Corporation Ltd (Assam Oil Division), New Delhi
SHRI ISWAR CHANDRA (*Alternate*)	
SHRI A. Y. GUPTE	Hindustan Petroleum Corporation Ltd (Marketing Division), Bombay
DR HIMMAT SINGH	Indian Institute of Petroleum (CSIR), Dehra Dun
SHRI J. S. BAHL (*Alternate*)	
SHRI M. B. JAYAWANT	Synthetic Asphalts Limited, Bombay
SHRI V. A. JOLLY	Bharat Petroleum Corporation Ltd (Marketing Division), Bombay
SHRI A. D. NAYAK (*Alternate*)	
SHRI K. L. KAPOOR	Public Works Department, Government of Haryana
SHRI S. C. JAIN (*Alternate*)	
SHRI T. S. KRISHNAMURTHI	Indian Oil Corporation Ltd (Refinery Division), Bombay
SHRI C. V. RAMAMURTHI (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
SHRI S. B. KULKARNI	Indian Oil Corporation Ltd (Marketing Division), Bombay
SHRI S. A. LATHEEF	Highways and Rural Works Department, Government of Tamil Nadu, Madras
SHRI M. R. MALYA	In personal capacity (*3 Panorma, 30 Pali Hill Road, Bombay 400052*)
SHRI C. V. RAMASWAMY	Hindustan Petroleum Corporation (Refinery Division), Bombay
SHRI RANJIT SINGH	Ministry of Defence (R & P)
SHRI P. D. DESHPANDE (*Alternate*)	
DR A. V. R. RAO	National Buildings Organization, New Delhi
DR R. S. RATRA (*Alternate*)	
SHRI T. K. ROY	Shalimar Tar Products (1935) Ltd, Calcutta
SHRI C. H. SAHEBA	Bharat Petroleum Corporation Ltd (Refinery Division), Bombay
SHRI K. R. RAO (*Alternate*)	
SHRI D. K. SEN	Central Fuel Research Institute (CSIR), Dhanbad
SHRI A. K. CHOUDHURI (*Alternate*)	
SHRI N. SIVAGURU	Roads Wing (Ministry of Shipping & Transport)
SHRI R. P. SIKKA (*Alternate*)	
REPRESENTATIVE	Directorate General of Supplies & Disposals, New Delhi
SHRI S. P. GUPTA (*Alternate*)	
SHRI G. Raman, Director (Civ Engg)	Director General, ISI (*Ex-officio Member*)

Secretary
SHRI VIJAY RAJ
Assistant Director (Civ Engg), ISI

Methods of Testing Tar and Bitumen Subcommittee, BCDC 2 : 2

Convener

PROF C. G. SWAMINATHAN	Central Road Research Institute (CSIR), New Delhi

Members

SHRI Y. C. GOKHALE (*Alternate* to Prof C. G. Swaminathan)	
SHRI J. S. BAHL	Indian Institute of Petroleum (CSIR), Dehra Dun
SHRI J. M. NAGPAL (*Alternate*)	
LT-COL C. T. CHARI	Engineer-in-Chief's Branch, Army Headquarters
SHRI G. C. MISHRA (*Alternate*)	
SHRI M. GOPALA KRISHNA	National Test House, Calcutta
SHRI G. C. GOSWAMI	Indian Oil Corporation Ltd (Assam Oil Division), New Delhi
SHRI ISWAR CHANDRA (*Alternate*)	

(*Continued on page* 8)

Indian Standard

METHOD FOR DETERMINATION OF ASPHALTENES IN BITUMEN BY PRECIPITATION WITH NORMAL HEPTANE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 February 1983, 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 series of Indian Standards have been published on methods of tests for testing tar and bituminous materials. This standard is one in the series which covers the method for determination of asphaltenes content in bitumen by precipitation with normal heptane.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

1. SCOPE

1.1 This standard covers the method of test for determination of asphaltenes content in bitumen by precipitation with normal heptane.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition in addition to those given in IS : 334-1982† shall apply.

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

†Glossary of terms relating to bitumen and tar (*second revision*).

2.1 Asphaltene Content — The asphaltene content of bitumen is the percentage by mass of wax free material insoluble in *n*-heptane, but soluble in hot benzene or toluene. The material is dissolved in *n*-heptane and the 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.

3. APPARATUS

3.1 Extraction Apparatus — Extraction apparatus shall consist of an efficient condenser that is, condenser with a coil or double surface, a reflux extractor of the type illustrated in Fig. 1, and a conical fiask. Ground glass joints are to be preferred throughout.

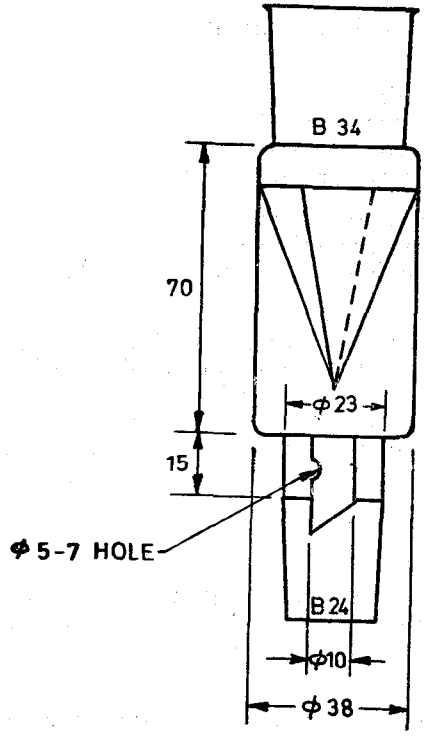

FIG. 1 REFLUX EXTRACTOR

4. SOLVENTS

4.1 ***n*-Heptane** — conforming to the following requirements :

Characteristic	*Requirements*
Density at 20°C g/ml	0·683 80 ± 0·000 15
Refractive index at 20°C	1·387 70 ± 0·000 15
Freezing point, °C	— 90·710 (*Min*)
Distillation : 50% recovered, °C	98·427 ± 0·025
Differential	
80% recovered minus 20% recovered°C	0·020 (*Max*)

4.2 Benzene — Reagent grade; conforming to IS : 1840-1961*.

4.3 Toluene — Reagent grade, conforming to IS : 1839-1961†.

NOTE — The extraction with benzene is not normally recommended because of the high toxicity of this material and it should be done for reference purposes only.

5. PROCEDURE

5.1 Preparation of Sample — If the sample consists of hard bitumen, it shall be ground to powder immediately before the test.

5.2 Preparation of Apparatus — Clean all glass dishes by immersing them in concentrated sulphuric acid or chromic acid mixture for at least 12 hours. Rinse with distilled water and with acetone. Place them in an oven at 100 to 110°C for 30 min, and cool in a dessicator for 30 min before weighing.

5.3 Testing

5.3.1 Weigh to the nearest 0·01 g a quantity of sample not exceeding 5 g and which will give a final weight of asphaltenes not exceeding 0·25 g into a conical flask of suitable capacity. Add *n*-heptane in the ratio of 30 ml to each 1 g of sample and boil the mixture under reflux for one hour. In case the asphaltene content of the sample exceeds 25 percent and the volume of *n*-heptane taken is small, there is danger of bumping during refluxing. In such cases the volume of *n*-heptane may be increased to a maximum dilution ratio of 50 : 1. Bumping is less likely to occur in

*Specification for benzene, reagent grade.

†Specification for toluene, reagent grade.

small flasks. Remove the flask and contents from reflux, cool, close with a ground glass stopper and store in a dark cupboard for $1\frac{1}{2}$ to $2\frac{1}{2}$ hours calculated from time of removal from the reflux.

5.3.2 Without agitation, decant the liquid through a filter paper such as Number 42 Whatman filter paper, of fine porosity and of 11·0 or 12·5 cm diameter, folded as illustrated in Fig. 2 so as to prevent loss of asphaltenes by creeping. Transfer the residue in the flask, as completely as possible, to the filter paper with successive quantities of hot *n*-heptane, using a glass rod, if necessary. Give the flask a final rinse with hot *n*-heptane and pour the rinsings through the filter. Set the flask aside for use, as described in **5.3.4** without washing.

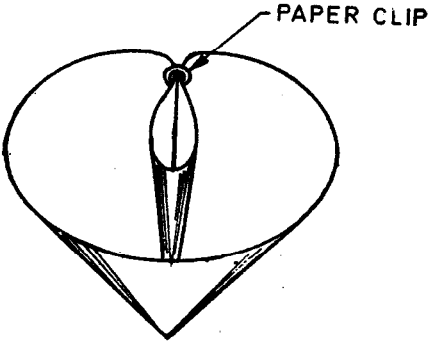

FIG. 2 FOLDED FILTER PAPER

5.3.3 Remove the filter paper and contents from the funnel and place in the extractor. Using another clean flask, reflux with *n*-heptane at a rate of 2-4 drops/sec from the end of the condenser for an extraction period of not less than one hour, or until a few drops of *n*-heptane from the bottom of the extractor leave no residue on evaporation on a glass slide.

5.3.4 Replace the flask with the one set aside as described in **5.3.2**, add 30-60 ml of benzene or toluene and continue refluxing until all the asphaltenes have been dissolved from the paper.

6. PRECAUTION

6.1 Since asphaltenes are very susceptible to oxidation, it is recommended that the procedure specified in the final drying stage be followed strictly, regarding temperature and time.

6.2 Benzene is a highly toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through the skin by contact with the liquid. Use under extraction ventilation, avoid skin contact and wear approved protective gloves.

6.3 Toluene is a toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through the skin by contact with the liquid. Use in adequate ventilation and avoid skin contact.

7. CALCULATION AND REPORTING

7.1 Calculate the asphaltenes content as a percentage by mass on the original sample and report the result to the nearest 0·1 percent.

8. PRECISION

8.1 Repeatability — Duplicate test results by the same operator should not differ by more than 10 percent.

8.2 Reproducibility — The results submitted by each of two laboratories should not differ by more than 20 percent.

(*Continued from page* 2)

Members	*Representing*
SHRI M. B. JAYAWANT	Synthetic Asphalts Limited, Bombay
SHRI B. B. L. KAPOOR	Bharat Petroleum Corporation Ltd (Refinery Division), Bombay
SHRI C. H. SAHEBA (*Alternate*)	
SHRI S. B. KULKARNI	Indian Oil Corporation Ltd (Marketing Division), Bombay
SHRI S. A. LATHEEF	Highways and Rural Works Department, Government of Tamil Nadu, Madras
SHRI M. R. MALYA	In personal capacity (*3 Panorma, 30 Pali Hill Road, Bombay 400052*)
SHRI K. P. NAIR	Indian Oil Corporation Ltd (R & D Centre), New Delhi
SHRI C. V. RAMASWAMY	Hindustan Petroleum Corporation Ltd, Bombay
SHRI A. Y. GUPTE (*Alternate*)	
SHRI T. K. ROY	Shalimar Tar Products (1935) Ltd, Calcutta
SHRI N. SIVAGURU	Roads Wing (Ministry of Shipping & Transport)
SHRI R. P. SIKKA (*Alternate*)	
SHRI T. V. VARGHESE	Madras Refineries Ltd, Madras
SHRI E. S. R. RAO (*Alternate*)	

AMENDMENT NO. 1 JUNE 2000
TO
IS 10511 : 1983 METHOD FOR DETERMINATION OF ASPHALTENES IN BITUMEN BY PRECIPITATION WITH NORMAL HEPTANE

(*Page* 3, *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

AMENDMENT NO. 2 NOVEMBER 2002
TO
IS 10511 : 1983 METHOD FOR DETERMINATION OF ASPHALTENES IN BITUMEN BY PRECIPITATION WITH NORMAL HEPTANE

(*Page* 4, *clause* **2.1**, *line* 3) — Delete 'hot benzene or'.

(*Page* 4, *clause* **2.1**, *line* 7) — Delete 'benzene or'.

(*Page* 5, *clause* **4.2**) — Delete and renumber the subsequent clause.

(*Page* 6, *clause* **5.3.4**, *line* 2) — Delete 'benzene or'.

(*Page* 6, *clause* **5.3.4**) — Insert the following new clauses after **5.3.4**:

'**5.3.5** Transfer the contents of the flask to a tared evaporating dish. Wash out the flask with successive small quantities of toluene not exceeding 30 ml. Remove the toluene by evaporation on a boiling water bath.

5.3.6 Dry the dish and contents in the oven at temperature of 105 ± 5°C for 30 minutes. Cool in a desiccator and weigh.'

(*Page* 7, *clause* **6.2**) — Delete and renumber the subsequent clause.

(*Page* 7, *clause* **7.1**) — Substitute the following for the existing:

'**7.1** Calculate the asphaltenes content, *A* as a percentage by mass on the original sample using the following equation:

$$A = 100\,(M/G)$$

where

M = mass in g of asphaltenes, and
G = mass in g of the test sample.

(PCD 6)

Reprography Unit, BIS, New Delhi, India