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BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

भारतीय मानक मसौदा

टार और बिटुमिनस सामग्री — ट्राइक्लोरोइथिलीन एवं टॉल्युईन में घुलनशीलता का निधार्रण — परीक्षण पद्धतियाँ

(IS 1216 *का तीसरा पुनरीक्षण*)

Draft *Indian Standard*

TAR AND BITUMINOUS MATERIALS — DETERMINATION OF SOLUBILITY IN TRICHLOROETHYLENE OR TOLUENE — METHODS OF TEST

(*Third Revision of* IS 1216)

(ICS 75.140)

Bitumen, Tar and Related Products Sectional Committee, PCD 06	Last date for comments: **25 October 2025**

FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1958 and subsequently revised in 1978. The first revision was published as series of 22 standards in the form of a booklet, as given 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IS 1214 : 1978	Determination of matter insoluble in benzene (Withdrawn due to toxic nature of benzene)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.

The second revision was brought out to formulate individual standard on determination of solubility in trichloroethylene and the following major changes were made:

a) Use of carbon disulphide was removed from the test method due to its carcinogenicity; and
b) Precautions for use of trichloroethylene were incorporated.

This revision has been brought out to keep pace with the latest technological developments and international practices. In this revision, the following major changes have been made:

a) Title has been modified;
b) Use of toluene as an alternative solvent to trichloroethylene to mitigate hazards;
c) Procedure for preparation of the filtering assembly has been modified using the glass microfiber filter pad and Gooch crucible; and
d) Asbestos from the filtration assembly has been removed.

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*)’.

1 SCOPE

This standard prescribes the test method for determining the solubility of asphaltic bitumen and native asphalts in trichloroethylene or toluene.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were 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 edition of these standards:

IS No.	*Title*
IS 245 : 2020	Specification for trichloroethylene, technical (*fourth revision*)
IS 334 : 2023	Glossary of terms relating to bitumen and tar (*fourth revision*)
IS 537 : 2025	Toluene — Specification (*third revision*)
IS 1211 : 2022	Methods for testing tar and bituminous materials — Determination of water content — Dean and Stark method (*second revision*)

3 TERMINOLOGY

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

4 METHOD A (FOR ASPHALTIC BITUMEN)

4.1 Apparatus

4.1.1 *Balance* — capable to weigh with the least count of 0.001 g

4.1.2 *Oven* — capable of maintaining a temperature of (110 ± 5) ºC

4.1.3 *Gooch Crucible*

4.1.4 *Glass Microfiber Filter Pad* — capable of retention of 1.5 µm particles

4.1.5 *Filter Flask, with heavy walls and side tube* — 250 ml to 1 000 ml capacity

4.1.6 *Filter Tube*

4.1.7 *Rubber Connector* — to secure the crucible onto the filter tube

4.1.8 *Conical Glass Flask* — 200 ml capacity

4.2 Solvent

4.2.1 *Trichloroethylene* — *see* IS 245

4.2.2 *Toluene* — *see* IS 537

4.3 Preparation of Material

If the sample contains water, heat it to a temperature not exceeding 130 °C until the water has been removed, stirring constantly, when possible, during heating. Perform this operation as expeditiously as possible.

4.4 Preparation of the Filtering Assembly

FIG. 1 illustrates the construction of a typical filtering apparatus. Seat the glass microfiber filter pad into the Gooch crucible firmly. Moisten the filter pad with the solvent, and place the crucible in an oven at (110 ± 5) ºC for 15 min. After cooling the assembly in a desiccator for (30 ± 5) min, the mass of the crucible should be determined to the nearest 0.001 g. Insert the filter tube into the stopper of the filtering flask and set the Gooch crucible in the filter tube using the rubber connector. Connect the filtering flask to the suction pump.

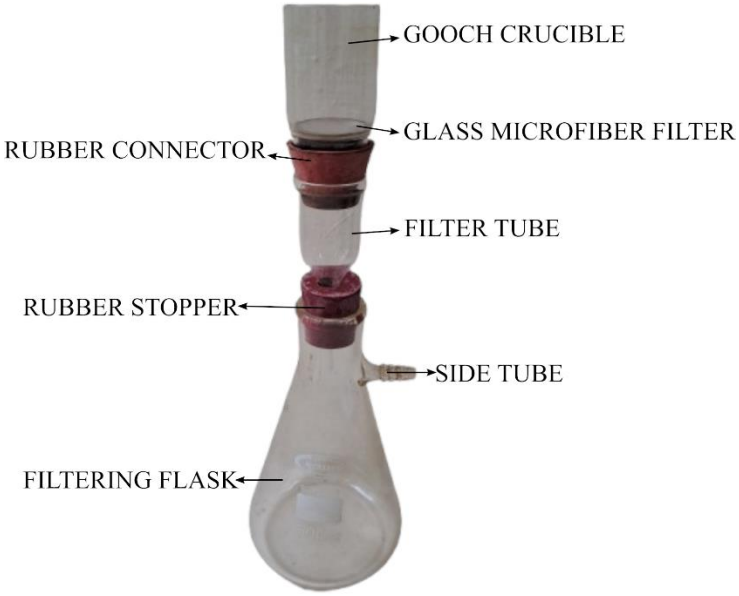

FIG. *1* FILTERING ASSEMBLY

4.5 Procedure

Weigh about 2 g of the dry sample to the nearest 0.001 g into a 200 ml conical flask and add 100 ml of trichloroethylene (*see* **4.2.1**) or toluene (*see* **4.2.2**). Stir the contents of the flask periodically until no large particles of the sample are observed. Allow it to stand loosely corked for a period of one hour.

> NOTE — In case of using toluene, the solution in the flask may be placed in a water bath of temperature not above 60 ºC for faster dissolution.

Filter the contents of the flask through the Gooch crucible prepared as described under **4.4**. For faster filtration process, light suction may be applied at the side tube. Moisten the filter pad with trichloroethylene/toluene before commencing. The filtrate shall be quite clear. Transfer the insoluble matter remaining in the flask to the crucible by washing out the flask with a stream of trichloroethylene/toluene from a wash bottle. Wash the sample retained in the crucible with successive small amounts of trichloroethylene/toluene until a filtrate is obtained which is not discoloured. Allow the crucible to dry in air for 30 min, after which place it in an oven at (100 ± 5) ºC for one hour. Allow the crucible to cool in a desiccator for 30 min and then weigh it to the nearest 0.001 g.

4.6 Calculation

Calculate the matter soluble in trichloroethylene/toluene as follows:

$$\text{Matter soluble in trichloroethylene/toluene, percent} = \frac{W_1 - W_2}{W_1} \times 100$$

where

W_1 = weight, in g, of dry sample taken for the test, and
W_2 = weight, in g, of insoluble material retained in the Gooch crucible.

4.7 Report

Report the result obtained in **4.6** to the nearest 0.05 percent as the matter soluble in trichloroethylene/toluene of the dry material.

4.8 Precision

Test results shall not differ from the mean by more than the following:

Sl No.	*Matter Soluble in Trichloroethylene/Toluene*	*Repeatability*	*Reproducibility*
(1)	(2)	(3)	(4)
i)	Below 98 percent	0.5	1.0
ii)	98 to 100 percent	0.1	0.2

5 METHOD B (FOR NATIVE ASPHALTS)

5.1 Apparatus

5.1.1 *Glass Tap Funnel* — approximately 100 mm diameter, the stem fitted with a tap and the top ground flat.

5.1.2 *Glass Plate* — to cover the funnel, about 120 mm in diameter, with a hole of about 16 mm diameter in the centre.

5.1.3 *Glass Funnel* — smaller than that described under **5.1.1** with its stem passing through a cork placed in the hole in the glass plate, the lower end of the stem reaching approximately half-way down the tap funnel.

5.1.4 *Filter Papers* — two, Whatman No. 5 or equivalent filter papers, 185 mm in diameter, dried in a suitable oven, cooled in a desiccator, counterpoised and folded together as shown in Fig. 2.

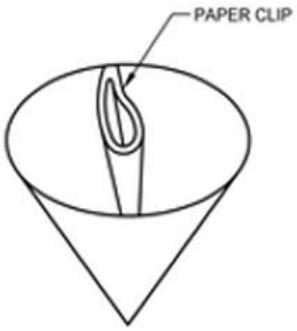

FIG. 2 FOLDED FILTER PAPER

5.2 Solvent and Material

5.2.1 *Trichloroethylene — see* IS 245

5.2.2 *Toluene — see* IS 537

5.2.3 *Cement*

Suitable for sealing the glass plate to the funnel. It may be prepared with 10 g of gelatin, 80 ml of water and 20 g of glycerine.

5.3 Preparation of Sample

5.3.1 If the sample contains water, heat a 100 g portion at a temperature not exceeding 130 °C, the material being constantly stirred when possible, until the rate of loss in weight of the sample does not exceed 0.1 g during a 15 min period of heating.

5.3.2 If the loss in weight on drying exceeds 0.1 percent, determine the water content of another sample of the material in accordance with IS 1211.

5.4 Procedure

Dry the two filter papers (*see* **5.1.4**) in an oven at 100 °C to 110 °C, cool in a desiccator, counterpoise, fold together as illustrated in Fig. 2 and place in the tap funnel. Weigh about 2 g of the dry sample to the nearest 0.001 g, as prepared in **5.3**, in the filter papers and secure the cover to the funnel by means of the cement (*see* **5.2.3**). Now add trichloroethylene/toluene through the small funnel until the filter paper is about half filled and then allow it to stand for 30 min. Draw the solution off through the tap. Place a watch glass on the small funnel to minimize evaporation. Close the tap and introduce a further quantity of trichloroethylene/toluene through the small funnel and again draw the solution off after 30 min. Repeat this procedure until the solvent drawn off is no longer discoloured. When the filtration is completed, remove the cover and allow the filter papers to dry in air for 30 min before placing them in a ventilated air oven at 100 ºC to 110 ºC for one hour. Then place the filter papers in a desiccator and when cool, weigh the inner filter paper and contents, using the outer filter paper as a counterpoise.

5.5 Calculation

Calculate the solubility of the dry material as follows:

$$\text{Matter soluble in trichloroethylene/toluene, percent by weight} = \left(\frac{W_1 - W_2}{W_1} \times 100\right) \times \frac{100}{100 + W_3}$$

where

W_1 = mass, in g, of the dry sample taken for the test;
W_2 = mass, in g, of recovered insoluble matter; and
W_3 = water content, in g, of sample, if determined.

5.6 Report

Report the result obtained in **5.5** to the nearest 0.05 percent as the matter soluble in trichloroethylene/toluene.

5.7 Precision

Test results shall not differ from the mean by more than the following:

Repeatability	0.5
Reproducibility	0.5

6 PRECAUTIONS

6.1 Barriers

Handling of trichloroethylene shall be done in fume cupboard with ventilation.

6.2 Personal Protective Equipment (PPE)

Chemical gloves, eye goggles, face shield, spill proof clothing, chemically resistant safety shoes shall be provided.

6.3 Safety shower nearby and eye wash in immediate vicinity shall be provided.

NOTE — It is strongly recommended that the user reviews the safety data sheet (SDS) of these materials and setup the operating procedure, PPE and barriers accordingly.