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भारतीय मानक मसौदा

रबर उद्योग के लिए अघुलनशील (अनाकार) सल्फर — विशिष्टि

(IS14127 का *पहला पुनरीक्षण*)

Draft Indian Standard

INSOLUBLE (AMORPHOUS) SULPHUR FOR RUBBER INDUSTRY — SPECIFICATION

(*First Revision* of IS 14127)

(ICS No. 83.060)

Rubber and Rubber Products Sectional Committee, PCD 13	Last date for receipt of comment is **28 June 2025**

FOREWORD

(*Formal clauses will be added later*)

Insoluble (amorphous) sulphur is a key ingredient in the rubber industry due to its non-blooming nature and uniform dispersion in rubber compounds. It transforms into the active vulcanizing form upon curing without affecting the tack of uncured rubber. This standard classifies insoluble sulphur into two grades based on oil treatment: Grade 1 (oil-treated) and Grade 2 (non-oil-treated).

Recognizing the distinct properties and applications of insoluble sulphur, this standard has been formulated separately from IS 8854:1994 ‘Sulphur for Rubber Industry’ to specifically address its technical requirements and industry usage.

This standard was first published in 1995.The first revision has been brought out to bring the standard in the latest style and format of Indian standard and to update the cross- referred standards in the standard

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: 2022 ‘Rules for rounding off numerical values (*second revision*)’. 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

This draft standard prescribes the requirements and methods of sampling and test for insoluble (amorphous) sulphur for rubber industry.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All 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 460 (Part 1) : 2020	Test sieves — Specification Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 6655 : 1972	Methods of test for sulphur
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1

3 GRADES

3.1 Grade 1 - Oil treated.

3.2 Grade 2 - Without oil treated.

4 REQUIREMENTS

4.1 The material shall be bright yellow powder and free from impurities.

4.2 The material shall comply with the requirements given in Table 1 when tested according to the procedures given in col (5) of Table 1.

Table 1 Requirements of Insoluble (Amorphous) Sulphur for Rubber Industry
(Clauses 4.2 *and* 6.2*)*

Sl No.	**Characteristics**	**Requirements**		**Method of Test, Ref to**
(1)	(2)	Grade 1 (3)	Grade 2 (4)	(5)
i)	Acidity (H_2SO_4) percent, *Max*	0.05	0.05	3.4 of IS 6655
ii)	Loss in mass, percent, *Max*	0.55	0.55	3.2 of IS 6655
iii)	Residue On ignition, percent, *Max*	0.20	0.30	3.3 of IS 6655

iv)	Fineness as residue (wet) on 63 mesh, percent, *Max* [1)]100 mesh, percent, *Max* 125 mesh, percent, *Max* 180 mesh, percent, *Max*	0.1 0.50 0.2 0.02	2.0 - 0.2 0.02	3 of IS 7086 (Part 1)	
v)	Total sulphur, percent, *Min*	78.5	99.0	3.1 of IS 6655	
vi)	Insoluble sulphur, percent, *Min* (on total sample)	90	90	Annex A	
vii)	Thermal reversion percent, *Max*	30	30	Annex B	
viii)	a) Oil content, percent b) Oil+ Binder content	19.0±1.5 20.0±1.5	 -	Annex C	

[1)] Residue must not contain hard particles and unmilled particles > 0.500 mm

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in packages as agreed to between the purchaser and the supplier.

NOTE — The material can be supplied in 20 kg or 25 kg net weight bags. The packing can be either in HDPE bags or laminated paper bags capable of preventing sun light / moisture absorption during transit, storage and handling. The supply can be either as loose bags or bags stacked in pallets or packed in carton/cardboard boxes. In case of pallets, the maximum weight shall be 1250 Kg. The bags shall be suitably stacked, strapped and stretch wrapped. The base of the pallet should be of good quality material and shall not damage during transit, storage and handling. The pallets or cartons/cardboard boxes shall be identified with all relevant details given under "Material Identification".

5.2 Marking

5.2.1 Each package shall be marked with the following:

a) Material highly inflammable,
b) Name and grade of the material,
c) Indication of source of manufacture,
d) Net mass of the material,
e) Month and year of the manufacture, and
f) Lot/batch number.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the rules and regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 Representative samples of the material shall he drawn as prescribed in IS 7086 (Part 1).

6.2 Number of Tests

Test for all characteristics of the material given in Table 1 shall be conducted on composite sample.

6.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample satisfy the requirements.

7 HANDLING AND SAFETY

The material shall be handled as given in National Safety Council Data Sheet No. 1-592 Rev. 83 and 1-612 Rev. 84.

8 TEST METHODS

8.1 Tests shall be conducted according to the methods prescribed in col (5) of Table 1.

8.2 Quality of Reagents

Unless otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE – 'Pure chemicals' shell mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[*Table* 1, *Item* (vi)]

DETERMINATION OF PERCENTAGE OF INSOLUBLE SULPHUR

A-1 PRINCIPLE

A-1.1 The test sample is titrated with solvent to dissolve the soluble sulphur and any oil. The insoluble sulphur is filtered off, dried and weighed.

Two procedures are specified, viz:

a) Toluene - Reference procedure
b) carbon disulphide - Alternative procedure

NOTE -The two procedures may give different results.

A-2 TOLUENE (REFERENCE PROCEDURE)

A-2.1 Reagents

Toluene - Reagent grade.

A-2.2 Procedure

Accurately weigh to 0.001 g approximately 2 g (W) of the test sample in a 400-ml beaker. Add 200 ml of toluene (**A-2.1**) and a magnetic stirrer. Cover with a clock glass and stir for 30 minutes. Filter through a tared sintered ISO porosity P40 glass filter crucible (W_2). Remove the toluene by suction. Wash three times with toluene (20 ml).

NOTE - The sample must never be sucked completely dry before the last washing because reversion can occur due to the icing of the insoluble sulphur.

Completely remove the toluene by suction, and dry the glass crucible in a drying oven in a fume cupboard for 1 h at 80^0C, cool in a desiccator and weigh (W_1)

A-2.3 Calculation and Expression of Results

Calculate the percentage insoluble sulphur using the following equation :

$$\text{Percent insoluble sulphur (on total sample)} = \frac{(W1-W2)\times 100}{W}$$

where

W_2= the mass in grams of the glass crucible,

W_1= the mass in grams of the glass crucible plus residue of insoluble sulphur, and

W = the mass in grams of the test portion.

$$\text{Percent insoluble sulphur (on the sulphur portion)} = \frac{\text{Percent insoluble sulphur x100 ((On total sample)}}{\text{Percent totsl sulphur (on total sample)}}$$

A-3 CARBON DISULPHIDE ALTERNATIVE PROCEDURE

A-3.1 Reagents

Carbon disulphide - Reagents grade (see Note).

NOTE - Carbon disulphide is a very noxious, toxic and inflammable solvent; therefore take special safety precautions, including the use of a fume hood.

A-3.2 Procedure

Accurately weigh to 0.001 g approximately 5 g (W) of test sample in a 250-ml beaker. Add 100 cc of carbon disulphide and a magnetic stirrer. Cover with a clock glass and stir for 30 minutes, filter through a P40 porosity tared glass filter crucible (W_2). Remove the carbon disulphide by very light suction. Wash three times with carbon disulphide (20 ml).

NOTE - The sample must never be sucked completely dry before the last washing because reversion can occur due to icing of the insoluble sulphur.

Remove completely the carbon disulphide by suction, and dry the glass crucible in a drying oven for 1 h at 80^0C. Cool in a desiccator and weigh (W_1).

A-3.3 Calculation and Expression of Results

Calculate the percentage insoluble sulphur using the following equation :

$$\text{Percent insoluble sulphur (on total sample)} = \frac{(W1-W2)\times 100}{W}$$

where

W_2 = the mass in grams of the glass crucible,

W_1 = the mass in grams of the glass crucible plus residuce, and

W = the mass in grams of the test portion.

$$\text{Percent insoluble sulphur (on the sulphur portion)} = \frac{\text{Percent insoluble sulphur x 100 (on total sample)}}{Percent\ total\ sulphur\ (on\ total\ sample)}$$

ANNEX B
[Table 1, Item (vii)]

DETERMINATION OF THERMAL REVERSION OF INSOLUBLE SULPHUR

B- 1 PRINCIPLE

The test sample is kept in a paraffinic oil at 105 °C for a fixed time and the amount of insoluble sulphur after the treatment is measured.

B-2 TOLUENE – REFERENCE PROCEDURE (METHOD 1)

B-2.1 Apparatus

B-2.1.1 Oil oath with thermostatic control accurate to $\pm$ 0.2 °C

B-2.1.2 Thermometer accurate to 0.2 °C between 100 °C and 110 °C.

B-2.1.3 *Drying Oven*

B-2.2 Reagents

B-2.2.1 Paraffinic oil, viscous pharmaceutical grade, dynamic viscosity greater than 120 cP.

B-2.2.2 *Toluene - Reagent grade.*

B-2.3 PROCEDURE

Pour 20 ml of the paraffinic oil (**B-2.2.1**) into a 100 ml beaker, taking care not to wet the walls of the beaker.

Put the beaker into the thermostatic bath (**B-2.1.1**) kept at such temperature that the oil in the beaker can be maintained at 105^0C, control the temperature of the oil in the beaker with the thermometer (**B-2.1.2**), when its temperature is 105^0C, add cautiously approximately 1 g weighed to 0.001 g (W) of the test sample to the hot paraffinic oil and start a timing device. Stir continuously for exactly 15 minutes, then take the beaker out of the bath. Cool the paraffinic oil -sulphur mixture to room temperature in a cold water bath stirring vigorously. When cold, add 30 cc of toluene (**B-2.2.2**) and stir well. Decant the paraffinic oil/solvent mixture through a tared sintered porosity P40 glass filter crucible (W_2) using a suction flask; wash the sulphur three times with 35 cc portions of toluene and allow to stand for 15 minutes after each wash before decanting. Using a spatula, crush any lumps of sulphur that may be found in the beaker; transfer all the insoluble sulphur quantitatively into the sintered glass crucible and wash 10 times with small amount of toluene.

NOTE - The sample must never he sucked completely dry before the last washing because reversion can occur due to the icing of the insoluble sulphur.

Completely remove all the toluene by suction, and finish the drying by placing the glass crucible in the oven in a fume cupboard at 80^0C for 1 h. Cool in a desiccator and weigh (W_1)

B-2.4 CALCULATION AND EXPRESSION OF RESULTS

Calculate the percentage thermal reversion of the insoluble sulphur using the following equation :

$$\text{Percent reversion} = S_W - \frac{W1-W2}{W} \times 100 \times \frac{100}{SW}$$

where

W_2 = the mass in grams of the glass crucible,

W_1 = the mass in grams of the glass crucible plus residue,

W = the mass in grams of the test portion, and

S_W = percent initial insoluble sulphur.

B-5 CARBON DISULPHIDE – ALTERNATIVE PROCEDURE (METHOD 2)

B-5.1 Reagents

B-5.1.1 Paraffinic oil. viscous pharmaceutical grade, dynamic viscosity greater than 120 cP.

B-5.1.2 *Carbon Disulphide* - Reagent grade (see Note).

NOTE - Carbon disulphide is a very noxious, toxic and inflammable solvent, therefore, take special safety precautions, including the use of a fume hood.

B-5.2 Procedure

The procedure is exactly the same as the reference procedure except that carbon disulphide is used instead of toluene.

ANNEX C
[*Table* 1, *Item* (viii)]

DETERMINATION OF OIL CONTENT OF OIL TREATED SULPHUR

C- 1 PRINCIPLE

C-1.1 Oil is extracted from the sample by using a solvent and the solvent is then evaporated off and the mass of residual oil is determined. Two procedures are specified, viz :

a) light petroleum for rhombic soluble sulphur, and
b) sulphur saturated hexane for insoluble sulphur.

C-2 LIGHT PETROLEUM PROCEDURE (METHOD 1)

C-2.1 Reagents

C-2.1.1 Light petroleum 35-70 with a residue on evaporation of not greater than 0.001 g/100 ml.

C-2.3 Procedure

Weigh about 10 g (W_3) of test sample accurately on 0.001 g into a wide mouthed 250 cc conical flask, to which add by pipette, exactly 100 cc of light petroleum (**C-2.1.1**). Stopper and allow to stand at room temperature for 30 minutes swirling the flask every 5 minutes. Decant 75-ml oil the solution into a 100 cc beaker ensuring that no particles of sulphur are present and pipette 50-ml of this solution into another 100-ml beaker. Evaporate off the solvent from this 50-ml of solution by placing the beaker in a water bath in a fume cupboard. Remove the beaker from the water bath and allow to cool to room temperature. Extract the residue with 2 ml of light petroleum, measuring the solvent from a burette and adding it in such a way that the sides of the beaker are washed down. Swirl the beaker gently to disperse the residue with the solvent. Decant the solution into a tared (W_2) 50-ml beaker.

Repeat the extraction twice collecting all the washings in the 50-ml beaker. Evaporate off the solvent in a water bath and then in an oven at 80°C for 1 h in a fume cupboard. Cool in a desiccator and then weigh immediately (W_1).

C-2.4 Calculation and Expression of Results

Calculate the percentage oil content using the following equation:

$$\text{Percent oil} = \frac{2(W1 - W2) \times 100}{W3} - \quad 0.25$$

Where

W_2 = the mass of the tared beaker in grams,

W_1 = the mass in grams of the tared beaker plus oil,

0.25 = the concentration due to the solubility of sulphur in light petroleum, and

W_3 = the mass in grams of the test portion.

C-3 SULPHUR SATURATED HEXANE SOLUTION METHOD (METHOD II)

C-3.1 Reagents

C-3.1.1 A solution of sulphur saturated hexane prepared by adding 60 g of rhombic soluble sulphur to 4 litres of hexane mixed well and allowed to stand for 24 h before use to allow excess sulphur to fall to the bottom of the vessel. Use only the clear supernatant liquid.

C-3.2 Procedure

Weigh 5 $\pm$ 0.1 g of test sample (W) into a 250-ml conical flask to which add 25 cc of clear reagent solution (**C-3.1.1**). Maintain constant temperature throughout this stage of the procedure. Shake for 10 minutes and then filter through a tared (W_3) sintered glass filter crucible and wash the residue with 100 ml of the clear reagent solution (**C-3.1.1**). Wash the residue twice with 25 ml of ethyl alcohol.

Dry the filter crucible plus residue for 1 h at 80 ^{0}C in an oven in a fume cupboard, cool in a desiccator and weigh (W_3).

C-3.3 Calculation and Expression of Results

Calculate the percentage oil content using the following equation :

$$\text{Percent oil} = \frac{(W + W1 - W2) \times 100}{W}$$

where

W_1 = mass, in g, of the glass crucible,

W_2 = mass, in g, of the glass crucible plus residue, and

W = mass, in g, of the test portion.