Doc: PCD 13 (25111) WC
March 2024

BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

स्टाइरीनेटेड फिनोल – विशिष्टता

(IS 7351 का दूसरा पुनरीक्षण)

Draft Indian Standard

STYRENATED PHENOL — SPECIFICATION

(*Second Revision of* IS 7351)

ICS 71.080.90

Rubber and Rubber Products Sectional Committee, PCD 13	Last date for receipt of comment is **21 May 2024**

FOREWORD

(*Formal clauses will be added later*)

Styrenated phenol are non-staining and non-disclouring antioxidants finding application in white and transparent vulcanizates. In this standard the requirements pertaining to the product obtained by arylating phenol with styrene only is considered. This is essentially a mixture of mono-di-and tri-styrenated phenol with molecular mass ranging from 200 to 400. This product is soluble, in benzene, acetone, ethyl alcohol and petroleum ether and insoluble in water.

This standard was originally published in 1974. The standard was subsequently revised in 1985. In the first revision, the requirements of refractive index and relative density had been modified.

This revision has been undertaken to update the cross-referred standards in the standard and editorial changes. Amendment no.1 issued to previous version of the standard has also been incorporated this revision.

Besides specifying chemical requirements, the standard includes a recommended procedure for evaluating the material by means of a standard compounding and vulcanizing procedure.

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 or significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirement and methods of sampling and test for styrenated phenol intended for use as an antioxidant in rubber compounds.

2 REFERENCES

The following Indian Standards contains provision which, through reference in this 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 agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (fourth revision)
IS 1675 : 1971	Specification for steric acid, technical (first revision)
IS 1683 : 1994	Barytes for rubber industry — Specification (second revision)
IS 3399 : 2013	Zinc oxide for rubber industry — Specification (third revision)
IS 3660 (Part 8) : 2023 / ISO 2393 : 2014	Methods of test for natural rubber : Part 8 Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures (third revision)
IS 4588 : 1986	Specification for rubber, raw, natural (third revision)
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients : Part 1
IS 8851 : 1994	Sulphur for rubber industry — Specification (first revision)
IS 8862 : 1978	Specification for titanium oxide (Anatase type) for rubber industry
IS 8979 : 1997	Tetramethyl thiuram disulphide — Specification (second revision)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a clear viscous liquid not darker than pale amber colour. It shall not contain any visible impurities.

3.2 The material shall comply with the requirements given in Table l when tested according to the methods given in col (4) of Table 1.

Table 1 Requirements for Styrenated Phenol
(*Clause* 3.2)

SL No.	Characteristic	Requirement	Method of Test, (Ref to Annex)
(1)	(2)	(3)	(4)
i)	Refractive index at 27 °C	1.595 to 1.605	A
ii)	Relative density at 27 °C/27 °C	1.079 to 1.085	B
iii)	Viscosity at 27 °C, CPS	2000 to 6000	C
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0.1	D
v)	Ash, percent by mass, *Max*	0.1	E
vi)	Flash point in °C, *Min*	120	F
vii)	Volatile matter at 60 °C, percent by mass, *Max*	1.0	G

3.2.1 All the tests shall be carried out within one month of receipt of the material by the purchaser.

3.3 Compounding

The material when compounded and tested as given in Annex H shall have its performance comparable with that of a sample, previously approved by the purchaser complying with the requirements given in **3.1** and **3.2**.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in 25 kg and 200 kg drums or as agreed to between the purchaser and the supplier.

4.2 Marking

The packages shall be securely sealed and legibly marked with the following information:

a) Manufacturers name and registered trade-mark, if any;
b) Chemical name or trade name of the material;
c) Net man of the package; and
d) The lot and the batch number.

4.2.1 *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.

5 SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Sampling

5.1.1 *Lot*

All the containers in a single consignment manufactured under the same condition shall constitute a lot.

5.1.2 Samples shall be tested for each lot separately for ascertaining the conformity of the material to the requirements of the specification.

5.1.3 The number of containers to be chosen from the lot shall be in accordance with Table 2.

Table 2 Scale of Sampling
(*Clause* 4.1.3)

Lot size	*No. of containers to be chosen*
Up to 50	3
51 to 100	4

6 QUALITY OF REAGENTS

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

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of test or analysis.

ANNEX A
[*Table* 1, *Sl no.*(i)]
DETERMINATION OF REFRACTIVE INDEX

A-1 APPARATUS

A-1.1 Abbe-refractometer of standard make covering a range of 1.300 to 1.700 suitable for reading up to fourth place of decimal and provided with thermostat.

A-2 PROCEDURE

A-2.1 Start the thermostat attached to the apparatus and wait till it attains a constant temperature at (27.0 ± 0.5) °C. Clean the prism with cotton soaked with alcohol and wait till alcohol is evaporated off.

A-2.2 Transfer a drop of styrenated phenol on the prism with the help of a clean, dry glass rod and close the prism carefully. Adjust the eye piece in such a manner that the cross wires coincide with the line of demarcation between bright and dark region. Read the result on the dial to fourth place of decimal with the help of the vernier scale.

A-3 PRECAUTIONS

a) Check occasionally the readings of the instrument with test sample;
b) Temperature should be controlled within the limit of ± 1 °C;
c) Clean the prism with alcohol;
d) Put right quantity of liquid on the prism; and
e) There should be no air bubble in the sample while taking reading.

ANNEX B
[Table 1, *Sl no.*(ii)]
DETERMINATION OF RELATIVE DENSITY

B-1 APPARATUS

B-1.1 Hydrometer, with a range of 1.000 to 1.110.

B-1.2 Measuring Cylinder, 100 ml and Internal diameter 28 mm and internal height 250 mm.

B-l.3 Thermostat

B-2 PROCEDURE

Fill styrenated phenol in a clean, dry, measuring cylinder to 100 ml mark (approximately) and place the cylinder in a thermostat. Start the thermostat and wait till it attains a constant temperature at (27 ± 1) °C. Check that the material has also attained the set temperature. Now allow the clean, dry, hydrometer to be immersed in the liquid and wait till it assumes a steady position. The reading corresponding to the meniscus of the liquid will give the relative density of the sample.

B-3 PRECAUTIONS

a) Avoid any entrapment of air bubbles in the samples;
b) Take care that the hydrometer does not touch the cylinder wall, and
c) While taking reading, position of eye should be on the same level as of the meniscus.

ANNEX C
[*Table* 1, *Sl no.*(iii)]
DETERMINATION OF VISCOSITY

C-1 GENERAL

The measurement of the drag produced upon a given spindle rotating at a definite constant speed while immersed in the material at a given temperature and indicated on a dial produces viscosity reading in centipoise.

C-2 APPARATUS

C-2.1 Brook Field Synchro Electric Viscometer

C-3 PROCEDURE

C-3.1 Support the viscometer on the stand and level. Attach spindle No.3 by holding the upper shaft coupling and turning the spindle. Place sample in as wide a container as possible that will give the proper depth and adjust to (27 ± 1) °C. Lower the instrument so that the spindle is immersed in the liquid to the groove on the spindle. Check levelling and centre spindle by moving container. Set hexagonal knob to the proper speed (rpm) and start motor by snapping toggle switch.

C-3.2 After 8 revolutions to 10 revolutions press down clutch level on the back and shut off motor so that the dial indicator is visible. Keep clutch level depressed to hold the reading. Without releasing the clutch start motor first and then release the clutch. After the second 8 revolutions to 10 revolutions, repeat the steps above to get the check reading. Make several check readings, allowing enough time to attain shear equilibrium between readings. Release clutch after shutting off motor and while spindle is still immersed raise instrument and remove spindle carefully by holding the upper shaft coupling firmly.

Factor for spindle at different speeds:

RPM	*Spindle No.* 3
60	20
30	40
12	100
6	200

Deduct 8 cps from final reading at drag correction.

ANNEX D
[*Table* 1, *Sl no.*(iv)]
DETERMINATION OF ACIDITY

D-1 GENERAL

This method determines the acidity in styrenated phenol. The result is expressed in terms of percent sulphuric acid.

D-2 REAGENTS

D-2.1 Methanol

D-2.2 Potassium Hydroxide Solution, 0.5 N, alcoholic.

D-2.3 Phenolphthalein Indicator

D-3 PROCEDURE

Weigh 100 g of styrenated phenol to the nearest mg in a one litre conical flask and dissolve the contents in 500 ml of methanol thoroughly. Titrate the solution with 0.5 N alcoholic potassium hydroxide using phenolphthalein as indicator.

D-4 CALCULATION

Acidity (as H_2SO_4), percent by mass = 0.0245 × ml of 0.5 N potassium hydroxide

ANNEX E
[*Table* 1, *Sl no.*(v)]
DETERMINATION OF ASH CONTENT

E-1 APPARATUS

E-1.1 Silica crucible

E-1.2 Desiccator

E-1.3 Tripod stand

E-1.4 Bunsen flame

E-2 PROCEDURE

E-2.1 Heat a clean, dry, silica crucible to (550 ± 25) °C for 30 min. Cool in a desiccator and weigh it to the nearest mg. In the crucible weigh to the nearest 0.001 g about 10 g of a representative sample of the material under teat.

E-2.2 Place the crucible in the hole in the asbestos board, place the board on a tripod stand and heat the crucible from below with a Bunsen flame. Heat gently, avoiding ignition until the sample has all volatilized, pyrolyzed or charred. Transfer the crucible to the muffle furnace at (550 ± 25) °C and leave for 30 min or longer until all the carbon has burnt off. Remove from the furnace, cool to room temperature in a desiccator and weigh to the nearest 0.001 g.

E-3 CALCULATION

$$\text{Ash, percent by mass} = \frac{B-A}{M} \times 100$$

where

A = mass in g of the crucible;
B = mass in g of the crucible plus ash; and
M = mass in g of the sample.

ANNEX F
[*Table* 1, *Sl no.*(vi)]
DETERMINATION OF FLASH POINT

F-1 APPARATUS

F-1.1 Automatic Pensky-Martens closed cup tester

F-2 PROCEDURE

Flash point determination is carried out by the Pensky-Marten closed cup method as indicated in **F-2.1.**

F-2.1 Thoroughly clean the cup and accessories taking care to remove any gasoline or naphtha used for cleaning. Fill the cup to the filling mark with the styrenated phenol. After locking in the lid properly, place the cup on the stove. Light the test flame, adjust it to 4 mm diameter. Rate of heating should be such that there is 6 °C temperature rise per minute. The stirrer should run at 90 rpm to 120 rpm. Apply test flame at 104 °C by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame lowered into the vapour space of the cup in one second, left in its lowered position for one second and quickly raised to its high position. Continue to apply the flame after every 20 °C rise in temperature till the flash point is reached. Flash point temperature is the temperature at which the test flame application causes a distinct flash in the interior of the cup.

ANNEX G
[*Table* 1, *Sl no.*(vii)]
DETERMINATION OF VOLATILE MATTER

G-1 Weigh 50 g of the styrenated phenol in a previously weighed evaporation dish. Keep the dish in vacuum at 60 °C and pressure of 40 mm of mercury for 2 h. Record the loss in mass.

Volatile matter, percent by mass = 2 × loss in mass.

ANNEX H
(*Clause* 3.3)
METHOD OF COMPOUND TESTING OF STYRENATED PHENOL

H-1 TEST COMPOUND

H-1.1 As a guidance, the following test compound may be used for testing performance properties of styrenated phenol in rubber compounds:

	Parts by Mass
Natural rubber grade A (*see* IS 4588)	100
Barytes (*see* IS 1683)	60
Zinc oxide (*see* IS 3399)	10

	Parts by Mass
Stearic acid (*see* IS 1675)	0.5
Titanium dioxide (*see* IS 8862)	10
Antioxidant styrenated phenol	2.0
Sulphur (*see* IS 8851)	2.0
Tetramethyl thiuram disulphide (*see* IS 8979)	0.5

H-2 PROCEDURE

H-2.1 Follow the procedure prescribed in IS 3660 (Part 8).

H-3 TESTS

H-3.1 The test given below is recommended for each test sample. The approved sample shall also be tested side by side using the same master batch.

H-3.2 Tensile strength, modulus at 300 percent elongation, elongation at break and hardness at optimum cure at 141 °C shall be carried out in accordance with the method prescribed in the appropriate parts of IS 3400 both before and after ageing at 70 °C for 7 days.

H-4. RESULTS

H-4.1 The tensile strength, modulus at 300 percent elongation and elongation at break, both of the original sample and the aged sample shall be within ±10 percent of the approved sample at the same time.